# Welcome to the

# 22<sup>nd</sup> International Conference on Ion Beam Analysis



Opatija, Croatia, June 14 – June 19, 2015

Book of Abstracts: 22nd International Conference on Ion Beam Analysis

22nd International Conference on Ion Beam Analysis June 14 - 19, 2015 - Opatija, Croatia

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# Welcome

On behalf of the local organizing committee, it is our pleasure to welcome you to the 22<sup>nd</sup> International Conference on Ion Beam Analysis. Twenty-two IBA conferences and 42 years since the first one has been organized in 1973 is by itself quite a success. And in spite of that long time period, the IBA flag representing all the countries that previously organized this conference is just like new. Fortunately this also applies to the field of our research, showing how ion beams are still a unique tool in many different application areas from materials to biomedicine, from environment to cultural heritage and others.

This 22<sup>nd</sup> IBA conference is being organized by Ruđer Bošković Institute, the largest research institute in Croatia, that celebrates this year its 65<sup>th</sup> anniversary. Organization would not be possible without generous supports from the Croatian Ministry of Science, Education and Sports, International Atomic Energy Agency and from many sponsors and exhibitors.

In the past the content of the IBA conferences opened an enormous market for users of ion beam accelerators and associated analysis techniques. With more than 230 participants, IBA 2015 is on a good track to continue to do so. This year we have accepted more than 330 abstracts from 42 countries, which will be presented in 22 oral and 2 poster sessions. We are grateful to welcome 6 plenary and 11 invited speakers as well. Scientific sessions will cover all important aspects of the ion beam analysis, from fundamentals of ion-solid interactions, computer programs and simulations, new experimental developments and applications of IBA in different research areas. Conference includes also micro and nanoprobes, accelerator mass spectrometry, complementary techniques as well as related materials modification subjects. On Monday, after Poster session 1, a panel organized by the IAEA on Ion Beam Techniques Roadmap will be held. Peer reviewed papers of the presentations given at the conference will be traditionally published in a special volume of Nuclear Instruments and Methods in Physics Research B. For the second time, with the help from Elsevier, prizes for the best manuscript written by doctoral students and young scientists will be awarded during the conference banquet.

IBA 2015 will be held in Opatija, one of the oldest tourist resorts on the Croatian Adriatic coast. Built mainly at the turn of the 20<sup>th</sup> century with well-maintained public gardens, the illuminated 12-km-long coastal promenade known as the "Lungomare", Opatija has remained in complete harmony with nature until the present days, so we believe that you will enjoy your stay not only because of the rich scientific program. The social program will include conference outing to the Istrian peninsula where visit to the charming little town of Motovun, views at the Roman monuments in Pula and tasting local food will be an excellent opportunity to spend enjoyable moments with colleagues and friends. Some of them we have met already on previous IBA conferences, some of them we will meet for the first time here in Opatija.

Finally, we hope that you will enjoy the conference and all that Opatija can offer.

Iva Bogdanović Radović, Milko Jakšić, Stjepko Fazinić, RBI

# Committees

### Local Organizing Committee

- Iva Bogdanović Radović, Ruđer Bošković Institute, Zagreb Conference chair
- Stjepko Fazinić, Ruđer Bošković Institute, Zagreb Conference co-chair
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- Miguel Ángel Respaldiza Galisteo Centro Nacional de Aceleradores, Spain
- Aliz Simon International Atomic Energy Agency (IAEA), Austria
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- Yongqiang Wang Los Alamos National Laboratory, U.S.A.
- Roger Webb University of Surrey, U.K.
- Isao Yamada Kyoto University, Japan
- Fujia Yang Fudan University, China
- James F. Ziegler United States Naval Academy, U.S.A.

### **Conference Secretary**

Ana Vidoš, Ruđer Bošković Institute, Zagreb

### Locations – map

All sessions will be held in the Congress center Tamaris. Plenary talks, invited talks and contributed talks will be held in the Orhideja hall on the first floor and Mimoza II hall on the second floor. For more details please see the Scientific Program. The poster sessions and coffee breaks will be held in the Mimoza I and III halls together with industrial exhibition. The conference registration will be located in front of the Orhideja hall.



# **Exhibitors and Sponsors**

The exhibitors and sponsors are very important part of every scientific conference. Not only that through the exhibition scientist get acquainted with the latest achievements in the equipment and instrumentation, this support enables also the cost of the registration to be kept to a minimum and provides additional support for the conference events, students and other activities. We are truly grateful to the following sponsors and exhibitors:





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#### Systems for Ion Beam Analysis

- (RBS) Rutherford Backscattering Spectroscopy
- (PIXE)
- Particle Induced X-ray Emission Nuclear Reaction Analysis (NRA)
- Elastic Recoil Detection (ERD)
- . Medium Energy Ionscattering Spectroscopy (MEIS)

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- Geosciences Material sciences
- Biomedicine
- Etc.

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**General Information** 

### **Oral Presentations**

Plenary talks are 45 minutes long (35-40 minutes presentation and 5-10 minutes discussion) and invited talks are 30 minutes long (25 minutes presentation and 5 minutes discussion). Contributed talks are 20 minutes long (15-17 minutes presentation and 3-5 minutes discussion). All the talks should be accompanied by PowerPoint or PDF slides. We will transfer the talk to the conference computer.

### **Projection Capability**

Lecture Halls Orhideja and Mimoza II will be equipped with a PC and LCD projection system. The PC will accept USB drives and will have Microsoft PowerPoint and Adobe Reader. You must have your presentation loaded before the start of your session.

#### Poster Displays

The size allocated for posters is 90 cm (width) by 110 cm (length). There will be two poster sessions, the first one will be held on Monday from 15:30 to 17:30 and the second one on Tuesday from 15:40 to 17:40. Poster presenters should be available at their display during this time. Posters should be set up in the morning of the presenting day and taken down at 20:00 the same day. Please display your poster in the slot assigned with your number. Please contact the conference registration desk if you need assistance.

Awards sponsored by the Croatian Center of Excellence for Advanced Materials and Sensors (CEMS) and ION-TOF for the best student poster will be presented during the conference banquet. Representatives of CEMS, ION-TOF and IBA International Advisory Committee will review student posters during the poster sessions.

### Conference Proceedings

The IBA 2015 conference proceedings will be published as a special volume of the journal "Nuclear Instruments and Methods B: Beam Interactions with Materials and Atoms". All submitted papers must be clearly written in excellent English and contain only original work, which has not been published by or is currently under review for any other journal or conference.

All manuscripts and any supplementary material should be submitted through the Elsevier Editorial System (EES). Papers will be reviewed to the same standards as for the regular NIMB papers. Please contact Iva Bogdanović Radović (<u>iva@irb.hr</u>) for other publication related questions.

#### Internet Access

Free internet access is available in the entire hotel and congress center.

### Registration and Welcome Reception

Registration will be open on Sunday from 14:00 to 18:00 and at 8:00 for other conference days. The welcome reception will be held in front of the Hotel Royal on Sunday, June 14, from 18:00 to 21:00.

#### Morning and Afternoon Breaks

Morning and afternoon breaks with refreshments will be held on the second floor in the Congress center Tamaris Mimoza I and III Halls, within the poster and exhibition area.

#### Lunches

Lunches will be provided in the Restaurant Camellia, Grand Hotel 4 Opatijska cvijeta on Monday, Tuesday and Thursday. On Wednesday, lunch will be provided during the conference outing. Lunches are included in the conference fee.

### International Committee Meeting

A meeting of the IBA International Advisory Committee will be held on Tuesday, June 16, after the poster session. The meeting will be held in the Antica Ostaria de Ugo, Hotel Continental at 19:00.

### **Conference Outing**

The Conference outing will be held on Wednesday, June 17 starting from 11:00. Lunch packages will be provided for all participants. The outing will start by visiting Motovun. At Motovun, IBA participants will have a tour around the old town, each group with their own guides. A short break after the tour will be given for a drink at the central square or to check several small stores with Istrian delicacies, which are located at the city gates.

Then we will visit Pula, the town situated at the southern tip of the Istrian peninsula. The rich itinerary of its three thousand year old history, begins and ends with the Roman amphitheatre. While strolling through Pula-Pola you will come across numerous monuments of Roman architecture: the Triumphal Arch of the Sergi from the 1st century B.C., Hercules' Gate and Twin Gates, the Temple of Augustus, Arena and Small Roman Theatre in the town center. At the end of the Istrian tour, IBA participants will have the opportunity to try a selection of traditional Istrian specialities in a small village near the city of Rovinj.

Additional excursion options are available on request. Please contact the conference registration desk for more information.

### **Conference Banquet**

The conference banquet will be held in the Hotel Royal Ballroom on Thursday, June 18 at 19:00. Banquet is included in the conference fee. We would like to thank High Voltage Engineering Europa B.V. for sponsoring this great event.

#### **Other Details**

We kindly ask participants to stick closely to the allocated duration of oral presentation and to the scheduling for posters. This is particularly important as we have a relatively high attendance and a densely packed program. Please wear your conference badge at all times. We have made special arrangements regarding lunch and refreshments, and your badge needs to be visible at these venues. The Local organizing committee will be pleased to help and advise on travel, touring and other arrangements, or on any problem.

### **Meeting Questions**

If you have any questions, please contact Ana Vidoš or anyone from the local organizing committee. Here are some cell phone numbers that may be useful:

Ana Vidoš <u>avidos@irb.hr</u>, +385 98 480 671 Iva Bogdanović Radović <u>iva@irb.hr</u>, +385 98 939 1685 Milko Jakšić, <u>jaksic@irb.hr</u>, +385 98 1710 330 Stjepko Fazinić, <u>sfazinic@irb.hr</u>, +385 99 317 4866



June 14 - 19, 2015 - Opatija, Croatia

Scientific Program

# 22<sup>nd</sup> International Conference on Ion Beam Analysis (IBA 2015) program

Congress centre Tamaris, Grand Hotel 4 opatijska cvijeta, Opatija

Sunday, June 14	, 2015
14:00 - 18:00	Registration – Congress centre Tamaris
18:00 - 21:00	IBA 2015 Welcome reception in front of the Hotel Royal

Monday, June 15, 2015		
8:00 - 16:00	Registration – Congress centre Tamaris	
8:10 - 8:30	Opening remarks and welcome address	
	Iva Bogdanović Radović – IBA 2015 cha	ir
	Tome Antičić – RBI Director General	
	Ian Vickridge – chair of the IBA Interna	tional Advisory Committee
	Session 1: Plenary session 1 (Orhideja	Hall)
	Chair: Iva Bogdanović Radović	
8:30 – 9:15	PL-1 Marek Rubel, KTH-Royal Institute	of Technology, Stockholm, Sweden,
	"The Role and Application of Ion Beam	Analysis for Studies of Plasma-Facing
	Components in Controlled Fusion Devi	ces"
9:15 – 10:00	PL-2 Andre Vantomme, KU Leuven, Bel	gium,
	"50 years of ion channeling in material	s science – where do we go next?"
10:00 - 10:30	Coffee break (Mimoza I and III Halls)	
	Session 2: Cross sections	Session 3: Channeling
	(Orhideja Hall)	(Mimoza II Hall)
	Chair: Matej Mayer	Chair: Andre Vantomme
10:30 - 11:00	I-1 Alex Gurbich, IPPE, Obninsk,	I-2 Debdulal Kabiraj, Inter University
	Russia,	Accelerator Centre, India,
	"SigmaCalc recent development and	"Study on dependence of structural
	present status of the evaluated	evolution on S <sub>e</sub> sensitivity of
	cross-sections for IBA"	semiconductors by c-RBS"
11:00-11:20	O-1 Wei-Kan Chu, University of	O-4 Daniel J. Silva, IFIMUP and IN-
	Houston, USA,	Institute of Nanoscience and
	"Mott vs Rutherford Scattering:	Nanotechnology, Universidade do
	Wave-Particle Duality of MeV	Porto, Portugal, and KU Leuven,
	Carbon, and Boron Ion Scattering	Belgium
	from Thin Graphite Foil"	"Drawing the geometry of 3d
		transition metal – boron pairs in
		silicon by means of electron emission
		channeling experiments"
11:20-11:40	O-2 Michael Kokkoris, National	O-5 Wenjie Yang, Australian National
	Technical University of Athens,	University, Australia,
	Greece,	"Atom location and ion damage
	"A Review on Benchmarking of	studies of gold hyperdoped silicon via
	Proton Elastic Scattering -	ion implantation followed by pulsed
	Implementation in the cases of <sup>nat</sup> Si, <sup>19</sup> F, <sup>nat</sup> B and <sup>nat</sup> O"	laser melting"

11:40-12:00	O-3 Paraskevi Dimitriou, International Atomic Energy Agency, Austria, "IAEA CRP on Development of a Reference Database for Particle- Induced Gamma-ray Emission (PIGE) spectroscopy"	O-6 Barney L. Doyle, Sandia National Laboratories, USA, "Ion Channeling Revisited"
12:00 - 13:30	Lunch (Restaurant Camellia, Grand Hot	(e) 4 opatijska cvijeta)
	Spectrometry – from keV to MeV (Orhideja Hall) Chair: Roger Webb	(Mimoza II Hall) Chair: Žiga Šmit
13:30-14:00	I-3 Kenji Kimura, Kyoto University, Kyoto, Japan, "Transmission secondary ion mass spectrometry using 5 MeV C <sub>60</sub> <sup>+</sup> ions"	I-4 Thomas Calligaro, Centre for Research and Restoration of the Museums of France, Paris, France, "Ion beam analysis of light elements in cultural heritage materials"
14:00-14:20	O-7 Sofia Gorondy Novak, CEA, France, "HI-ERDA versus SIMS to study helium profiling in pure bcc metals"	O-10 Chris Jeynes, University of Surrey, UK, "Glass deterioration mechanisms using Total-IBA of Rosslyn glass"
14:20-14:40	O-8 Toshio Seki, University of Kyoto, Japan, "Ambient Analysis of Liquid Materials with Wet-SIMS"	O-11 Dubravka Jembrih – Simbürger, Academy of Fine Arts, Austria, "MeV SIMS - a tool to study modern paint materials and their stability"
14:40-15:00		O-12 Caroline Czelusniak, INFN Sezione di Firenze, Italy, "Development of the time-resolved ion beam luminescence technique and its application to the provenance studies of lapis lazuli"
15:00 - 15:30	Coffee break (Mimoza I and III Halls)	
15:30 - 17:30	Poster Session 1 (Mimoza I & III Halls)	
17:30 – 19:00	IAEA Panel on Ion Beam Techniques R Panel co-ordinator: Aliz Simon, IAEA P Massimo Chiari, David Cohen. Ian Vicki	oadmap (Orhideja Hall) anel Members: Nuno Pessoa Barradas, ridge, Roger Webb

Tuesday, June 16, 2015		
8:00 - 16:00	Registration – Congress centre Tamaris	
	Session 6: Plenary session 2 (Orhideja	Hall)
	Chair: Stjepko Fazinić	
8:30 - 9:15	PL-3 Hans Arno Synal, ETH Zurich, Swit	zerland,
	"Progress in Accelerator Mass Spectron	metry"
9:15 - 10:00	PL-4 Jiro Matsuo, Kyoto University, Kyo	oto, Japan,
	"Secondary Ion Mass Spectrometric Ar	alysis of Soft Materials -
	Current Challenges and Future Applica	tions"
10:00 - 10:30	Coffee break (Mimoza I and III Halls)	
	Session 7: Imaging with keV ions	Session 8: Environmental and Earth
	(Orhideja Hall)	Applications
	Chair: Barney Doyle	(Mimoza II Hall)
		Chair: Massimo Chiari
10:30 - 11:00	I-5 Gregor Hlawacek, Helmholtz-	I-6 Silvia Nava, INFN, University of
	Zentrum Dresden-Rossendorf,	Florence, Italy,
	Germany,	"State of the art of IBA analysis of
	"Materials analysis using channeling	high time resolution aerosol
	and ionoluminescence in a helium	samples"
	ion microscope"	
11:00 - 11:20	O-13 Patrick Philipp, Advanced	O-16 Keizo Ishi, Tohoku University,
	Instrumentation for Ion Nano-	Japan,
	Analytics (AINA), Luxembourg,	"Application of micro-ion-beam
	"SIMS on FIB instruments: a powerful	analysis to investigate the
	tool for high-resolution high-	distribution of Cs in silt particles for
	sensitivity analytics at the nano	environmental remediation of
	scale"	Fukushima"
11:20 - 11:40	O-14 Rene Heller, Helmholtz-	O-17 Guy Terwagne, University of
	Zentrum Dresden-Rossendorf,	Namur, Belgium,
	Germany,	"Application of IBA to taphonomy
	"Ion Beam Analysis in the Helium Ion	and paleontology"
	Microscope"	
11:40 - 12:00	O-15 Ernst Jan Vesseur, FEI,	O-18 Mohamad Roumie,
	"Applications and benefits of a Xe	Accelerator Laboratory, Lebanese
	plasma FIB-SEM "	Atomic Energy Commission, National
		Council for Scientific Research,
		Beirut, Lebanon,
		"Evaluation and mapping of PM2.5
		atmospheric aerosols in Arasia region
		using PIXE and gravimetric
		measurements"
12:00 - 13:30	Lunch (Restaurant Camellia, Grand Hot	el 4 opatijska cvijeta)

	Session 9: Experimental developments and novel IBA techniques (Orhideja Hall) Chair: Theva Thevuthasan	Session 10: Stopping and Straggling (Mimoza II Hall) Chair: Nuno Barradas
13:30 - 13:50	O-19 Patrick Reichart, Universität der Bundeswehr, Germany, "Increased Accuracy and Sensitivity in Hydrogen Analysis"	O-24 Peter Sigmund, University of Southern Denmark, Denmark, "Structure in the velocity dependence of heavy-ion energy-loss straggling"
13:50-14:10	O-20 Hao Shen, Institute of Modern Physics, China, "Developments and applications of nuclear microprobes in China"	O-25 David N Jamieson, University of Melbourne, Australia, "Direct imaging of straggled ions for top-down fabrication of Si:P qubits using atom probe tomography"
14:10-14:30	O-21 Jaakko Julin, University of Jyväskylä, Finland, "Advanced pulse processing for Time-of-Flight ERD"	O-26 Moni Behar, Federal University of Rio Grande do Sul, Brazil, "Stopping power study of H and He in Hydropaxyapatite films: A study with implications in ion-beam therapy"
14:30-14:50	O-22 Tilo Reinert, University of North Texas, USA, "Performance Tests of a Scalable Multi-Detector Digital Spectrometer with Eight Channels"	O-27 Pedro Grande, Federal University of Rio Grande do Sul, Brazil, "Neutralization and wake effects on the Coulomb explosion depth profiling"
14:50-15:10	O-23 Martina Schulte-Borchers, ETH Zurich, Switzerland, "Recent progress on the new MeV SIMS setup at ETH Zurich"	O-28 Abdelkader Guesmia, iThemba Labs, South Africa, "Energy loss straggling of Cu, Si, O and C heavy ions through Si <sub>3</sub> N <sub>4</sub> thin foils over a range of energies 0.1–0.6 MeV/n by Time-of-Flight spectrometry"
15:10-15:40	Coffee break (Mimoza I and III Halls)	
15:40-17:40	Poster Session 2 (Mimoza I and III Hall	ls)
19:00-22:00	IBA International Advisory Committee (Antica Ostaria de Ugo, Hotel Contine	: Meeting ntal)

Wednesday, June 17, 2015			
8:00 - 16:00	Registration – Congress centre Tamaris		
	Session 11: Computer Simulations	Session 12: Modification and	
	and Analysis (Orhideja Hall)	Damage (Mimoza II Hall)	
	Chair: Kai Arstila	Chair: Devesh Avashti	
8:30 – 9:00	I-7 Udo von Toussaint, Max-Planck-	O-33 Lionel Thomé, CSNSM Orsay,	
(Orhideja)	Institut für Plasmaphysik, Garching,	CEA Saclay France,	
	Germany,	"Combination of RBS/C and Raman	
8:40-9:00	"Modern data analysis techniques for	techniques for the investigation of	
(Mimoza II)	ion beam analysis"	the SNEEL phenomenon in ion-	
		irradiated materials"	
9:00 - 9:20	O-29 Tiago F. Silva, Instituto de Física	O-34 Philipp Ernst, University of	
	da Universidade de Sao Paulo, Brazil,	Duisburg-Essen, Germany,	
	"MultiSIMNRA: a computational tool	"Graphene to Graphane Transition	
	for self-consistent ion beam analysis	Induced by Highly Charged Ion	
	using SIMNRA"	Irradiation"	
9:20 – 9:40	O-30 Micaela Fonseca, LIBPhys,	O-35 Ettore Vittone, University of	
	Caparica, Portugal,	Torino, Italy,	
	"ERYAProfiling: a code for depth	"A new protocol to evaluate the	
	profiling of light elements in	charge collection efficiency	
	inhomogeneous samples by PIGE"	degradation in semiconductor	
		devices induced by MeV ion beam	
		irradiation"	
9:40 - 10:00	O-31 François Schiettekatte,	O-36 Javier Garcia-Lopez, University	
	Université de Montréal, Canada,	of Sevilla, Spain,	
	"Spectrum simulation of rough and	"A modified drift-diffusion model for	
	nanostructured targets from their 2D	evaluating the carrier lifetimes in	
	and 3D image by Monte Carlo	radiation-damaged semiconductor	
	methods"	detectors"	
10:00 - 10:20	O-32 Marija Čargonja, University of	O-37 Gyorgy Vizkelethy, Sandia	
	Rijeka, Croatia,	National Laboratories, USA,	
	"VIBA-Lab 4.0: Computer program	"Determination of recombination	
	for Simulation and Quantitative	radius in Si for Binary Collision	
	analysis of PIXE, XRF and RBS	Approximation (BCA) codes"	
	Spectra"		
11:00 - 22:00	Conference outing – Istria tour – Moto	ovun & Pula guided tour	

Thursday, June 18, 2015		
	Session 13: Plenary session 3 (Orhideja Hall)	
	Chair: Milko Jakšić	
8:30 – 9:15	PL-5 Peter Bauer, Johannes Kepler Univ	versity, Linz, Austria,
	"Quantitative Low Energy Ion Scattering - accomplishments and challenges"	
9:15 - 10:00	PL-6 Lyudmila Goncharova, University	of Western Ontario, London, Canada,
	"High-resolution ion depth profiling: be	eyond high-k materials"
10:00 - 10:30	Coffee break (Mimoza I and III Halls)	
	Session 14: Microprobe and	Session 15: MEIS, LEIS
	Nanoprobe Analysis	(Mimoza II Hall)
	(Orhideja Hall)	Chair: Lyudmila Goncharova
	Chair: Lucile Beck	
10:30 - 11:00	I-8 Andrew Bettiol, NUS, Singapore,	I-9 Denis Jalabert – CEA/INAC,
	"Super-resolution imaging with	Grenoble, France,
	focused MeV ions: Fluorescence and	"MEIS analysis at the nanoscale,
	Structural imaging"	advantages/disadvantages versus X-
		Rays and TEM"
11:00 - 11:20	O-38 Pablo Jobim, Fedral University	O-41 Sahar Mirzaei, ANU, Australia,
	of Rio Grande do Sul, Brazil,	"Structural properties of SiGe
	Elemental signature of memory recall	nanoparticles in LPCVD Si <sub>3</sub> N <sub>4</sub> "
11:20 - 11:40	O-39 Claire Pacheco, Centre de	O-42 Victor Alarcon-Diez, Sorbonne
	Recherche et de Restauration des	Universités, UPMC, CNRS, Paris,
	Musees de France, France,	France,
	"Towards RBS/EBS mapping at the	"IBA analysis of Iron-doped Bi <sub>2</sub> Se <sub>3</sub>
	AGLAE facility: a new implement to	topological insulator"
	apprehend Cultural Heritage	
	materials"	
11:40 - 12:00	O-40 Iva Božičević Mihalić, RBI,	O-43 Thomas Grehl, ION-TOF GmbH,
	Croatia,	Münster, Germany,
	"Capabilities of miniature high	"TRBS modelling of LEIS spectra -
	resolution X-ray spectrometer at the	fundamentals and applications"
	ion microprobe"	
12:00 - 13:30	Lunch (Restaurant Camellia, Grand Hot	el 4 opatijska cvijeta)
	Session 16: New detectors	Session 17: Molecular and chemical
	(Orhideja Hall)	imaging (Mimoza II Hall)
40.00 44.00	Chair: Gyorgy Vizkelethy	Chair: Andrew Bettiol
13:30 - 14:00	I-10 Timo Sajavaara, University of	I-11 Katarina Vogel-Mikus, University
	Jyvaskyla, Finland,	of Ljubljana, Slovenia,
	"PIXE using large area transition-	"Complementing X-ray and MS based
	edge sensor array"	techniques in studies of trace
		elements in biological systems"
14:00 - 14:20	U-44 Tomihiro Kamiya , Japan Atomic	U-47 Zdravko Siketić, Ruđer Bošković
	Energy Agency , Japan,	Institute, Zagreb, Croatia, –
	"Study of diamond membrane	"Submicron molecular imaging of
	detector aiming at highly-efficient	cells by combined use of MeV SIMS
	and position-sensitive particle	and STIM"
	detection for ion beam applications"	

		Session 18: Ion induced luminiscence
		(Mimoza II Hall)
		Chair: Andrew Bettiol
14:20 - 14:40	O-45 Daniel Spemann, University of	O-48 Zhaohong Mi, CIBA, NUS,
	Leipzig, Germany,	Singapore,
	"Concept of single ion detector for	"Lanthanide-doped nanocrystals as
	deterministic ion implantation at the	bio-probes for ion beam induced
	nanoscale"	fluorescence imaging"
14:40 - 15:00	O-46 Jan Pallon, University of Lund,	O-49 Diana Bachiller-Perea,
	Sweden,	Université Paris-Sud, CNRS/IN2P3,
	"Technology steps to fabricate a thin	France,
	Silicon Carbide membrane based	"Ionoluminescence as a sensor of the
	particle detector"	defects creation and damage
		kinetics: application to fused silica"
15:00 - 15:30	Coffee break (Mimoza I and III Halls)	
	Session 19: Materials science	Session 20: Energy Applications
	(Orhideja Hall)	(Mimoza II Hall)
	Chair: Pedro L. Grande	Chair: Tonči Tadić
15:30 - 15:50	O-50 Craig M. Comrie, University of	O-55 Elisabetta Carella, CIEMAT –
	Cape Town, South Africa,	UNED, Spain,
	"RBS-Channelling analysis into the	"Deuterium behavior in ceramics for
	effect of thermal annealing on GeSn	Fusion Breeder Blanket after γ-
	strained layers"	irradiation: a comparison between
		different characterization
		techniques"
15:50 – 16:10	O-51 D.Nd. Faye, IPFN, University of	O-56 Yongqiang Wang, Los Alamos
	Lisbon, Portugal,	National Laboratory, USA,
	"Mechanisms of damage formation	"Deuterium Retention Studies in Ion
	in AlGaN alloys implanted with Ar	Beam Damaged W"
	and Eu ions"	
16:10 - 16:30	O-52 Noriaki Toyoda, Unversity of	O-57 Eduardo Alves, IST,
	Hyogo, Japan,	Universidade de Lisboa, Portugal,
	"Isolated defects created by gas	"Total IBA a quantitative tool to
	cluster ion impact and their use for	study plasma wall interactions"
	templates of carbon nanotube	
46.20 46.50	growth"	
16:30 - 16:50	U-53 Sergio Costa Miranda, KU	0-9 LUCIIE BECK, JANNUS, France,
	Leuven, Beigium,	Hydrogen benavior in materials
	Rutherford backscattering study of	after single and triple-beam
	Ni(Pt/Pd)Ge formation on Ge (100)	and uRomon"
16.50 17.10	O E4 Agonor Hontz, Instituto do	and $\mu$ Random $\Omega$
10.50 - 17.10	Efficient Universidade Endered de Ric	O-55 Summarampillar Mevutilasan,
	Granda da Sul Brazil	Record Institute Optor Foundation
	Giallue uu Sul, Bidzil,	"In Situ NDA DDS VDS and UDS
	core shall CdSe/7pS by thermal	Studios of Hydrogon Diffusion in TiO
	trootmont"	Single Crystale"
	treatment	Single Crystals

19:00 - 22:00	Conference banquet – Hotel Royal Ballroom	
	Julian Demarche, University of Surrey, Guilford, UK - "Log-periodicity and the	
	epistemic dynamics of Ion Beam Analysis research"	
	Best Poster Awards	
	Best Manuscript Awards	
	Music: Dino Antonić & 4 Shades, guest: Marijana Varašanec	

Friday, June 19, 2015		
	Session 21: Facilities and Instrumentation (Orhideja Hall)	
	Chair: Hidde Brongersma	
8:30 - 8:50	O-60 Nicolae C. Podaru, HVE, The Netherlands,	
	"Requirements on ion source performance for ion beam analysis"	
8:50 - 9:10	O-61 Andreas Markwitz, GNS Science, New Zealand,	
	"Ion beam analysis of advanced coating products and air particulate matter"	
9:10 - 9:30	O-62 Nemitala Added, University of Sao Paulo, Brazil,	
	"External milibeam imaging applications at LAMFI-USP"	
9:30 – 9:50	O-63 Chaohui Lan, Institute of Fluid Physics, China Academy of Engineering	
	Physics, China,	
	"Ion Component Diagnosis of Metal Hydride Cathode Vacuum Arc Ion Source	
	by Using an in-Cavity Minitype Magnetic Mass Spectrometer"	
9:50 - 10:20	Coffee break (Mimoza I and III Halls)	
	Session 22: Analysis of light elements by IBA (Orhideja Hall)	
	Chair: Michael Kokkoris	
10:20 - 10:40	O-64 William A. Lanford, University at Albany, USA,	
	"Nuclear Reaction Analysis for H, Li, Be, B, C, N, O and F with an RBS	
	Completeness Check"	
10:40 - 11:00	O-65 Masahiro Saito, Toray Research Center, Shiga, Japan,	
	"TOF-ERDA analysis on the effect of water for SEI formation in Li-ion	
	batteries"	
11:00 - 11:20	O-66 Jacek Jagielski, Institute for Electronic Materials Technology, National	
	Centre for Nuclear Research, Poland,	
	"Hydrogen release from irradiated elastomers measured by Nuclear Reaction	
	Analysis"	
11:20 - 11:40	O-67 Manfredo H. Tabacniks, Institute of Physics, University of Sao Paulo,	
	Brazil,	
	"Boron quantification limits using Ion Beam Analysis techniques"	
11:40 - 12.00	Closing remarks	

# ABSTRACTS PLENARY TALKS

# PL-1: The Role and Application of Ion Beam Analysis for Studies of Plasma-Facing Components in Controlled Fusion Devices

<u>M. Rubel (1)</u>\*, E. Alves (2), S. Brezinsek (3), J.P. Coad (4), M. Mayer (5), P. Petersson (1), A. Widdowson (4) and JET Contributors\*\*

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First wall materials in controlled fusion devices undergo serious modification by several physical and chemical processes arising from plasma–wall interactions. This includes material erosion, transport of eroded species in the plasma and re-deposition leading to the formation of mixedmaterial layers. Detailed information is required for the assessment of: (i) material lifetime; (ii) accumulation of hydrogen isotopes in wall materials, i.e. fuel inventory; (iii) dust formation. These issues are crucial for the economy and safety of reactor operation. As a consequence, these are the driving forces for detailed study of plasma-facing materials (PFM) and components (PFC).

A large variety of materials research methods are used to determine the morphology and properties of wall components and probes retrieved from fusion devices after entire experimental campaigns (plasma operation of up to  $10^5$  s) or short-term tests in order to perform relevant experiments and to obtain broad characterization of materials. Ion beam analysis techniques play a particularly prominent role here because of their isotope selectivity in the low-Z range (1-10), high sensitivity and possible combination of several methods in a single run.

The aim of the talk is to provide an overview of experimental procedures and results obtained in the examination of materials from JET (the largest tokamak), TEXTOR and ASDEX Upgrade. The presentation is structured along the points: "what" is to be analysed and "why", and then "how" the analyses are carried out. The role of <sup>3</sup>He-based NRA, RBS, PIXE (standard and microsize beam) and HIERDA in fuel retention and material migration studies is presented. The use of tracer techniques with rare isotopes (e.g. <sup>13</sup>C, <sup>15</sup>N, <sup>18</sup>O) or marker layers on wall diagnostic components is described. Development of equipment to enhance research capabilities and issues in handling of contaminated materials are addressed.

#### PL-2: 50 years of ion channeling in materials science – where do we go next?

#### A. Vantomme

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When nuclear physicists started abandoning their small accelerators in the sixties, research activities in ion beam – solid interactions were booming. Among others, it was discovered that energetic charged particles can be steered through a single crystal over a long distance, without undergoing any large-angle scattering. This effect, well-known as (ion) channeling, was computationally predicted by Robinson and Oen, experimentally confirmed by 3 groups independently and to a large extent theoretically explained by Lindhard - in merely a couple of years (1962-1965)! Since then, numerous variations of the channeling effect have been used and even proven crucial in a wide scala of materials science investigations. Any material property which is related to (a deviation of) its crystallinity can be investigated with the same depth, elemental, isotopic... resolution as the ion beam analysis technique it is based on, e.g. Rutherford backscattering spectrometry, particle-induced X-ray emission, nuclear reaction analysis... In this talk, we will dwell on a number of the paramount advances, which brought along the strength of the ion channeling technique, even beyond its standard use. These include examples where ion beam analysis has been driven to its extremes, where the experiment was performed in exotic conditions, or where non-conventional schemes or approaches were used – including progress in the experimental set-up and in simulations of the channeling effect. Moreover, we will reflect on the future of ion channeling during the decades to come. In particular: what will the role of channeling be in a research era which largely focuses on structures and properties at the nanometer-scale? It is anticipated that channeling will remain competitive with and complementary to other characterization techniques providing (local) structural information, including synchrotron-based approaches and state-ofthe-art electron microscopy.

#### PL-3: Progress in Accelerator Mass Spectrometry

#### H.-A. Synal

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The technical evolution of Accelerator Mass Spectrometry (AMS) instrumentation over the last ten years is summarized. A key characteristic of AMS is destruction of molecular interferences and subsequent analyses of atomic ions. It makes the extreme abundance sensitivity possible (in specific cases, below isotopic ratios of 10-16). This can be reached with instruments having quite modes mass resolving power (M/AM less than 300). Today, 1+ charge state is used, molecular interferences are destroyed in multiple collisions with stripper gas atoms or molecules, and a high yield atomic ions is reached at energies of a few hundred keV. Thus, AMS instruments develop towards lab size or tabletop devices. The use of He as stripper gas has further improved performance with respect to overall detection efficiency and reproducibility of measurement conditions. In parallel, implementation of permanent magnets into dedicated radiocarbon AMS system is progressed. This reduces complexity of the instruments and significantly reduces operation and installation costs. For radiocarbon, He stripping has potential to further down size instruments and reduce the ion energy below 50 keV. I will summarize the latest achievements. But, low energy AMS is not limited to radiocarbon only and there is a great potential for <sup>10</sup>Be, <sup>26</sup>Al, <sup>129</sup>I and actinides measurements at compact AMS systems. These developments have launched the wide spread use of AMS in various research fields and has resulted in a boom of new AMS facilities. The related impact to the wide variety of applications of AMS in modern research is not covered.

#### PL-4: Secondary Ion Mass Spectrometric Analysis of Soft Materials Current Challenges and Future Applications

#### <u>J. Matsuo (1,2)</u>

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(2) SENTAN, Japan Science and Technology Agency (JST), Chiyoda, 102-0075 Tokyo, Japan.

Secondary particle emission under ion irradiation provides unique opportunities for material analysis. Secondary ion mass spectrometry (SIMS) is now widely used in elemental analysis of semiconductors and metals. Chemical analysis of soft materials such as polymers, organic semiconductors and biological materials, is now in strong demand because of the increasing importance of these materials in emerging technologies. Newly developed ion beams, such as cluster and swift heavy ions, are used for organic- and bio-SIMS [1, 2]. These beams have higher secondary molecular ion emission yields than conventional monomer ion beams in the keV energy in range, because of their dense excitation during ion impact. In the last decade, molecular imaging and molecular depth profiling techniques have been developed for organic materials, and beams of small metal cluster ions or large Ar clusters ions with energy in the keV range are now widely used in commercial SIMS instruments.

We have demonstrated the technique of molecular imaging with swift heavy ion beams (MeV-SIMS) in biological material analysis. In this method, a high-energy ion accelerator is combined with an orthogonal acceleration time-of-flight mass spectrometer (oa-ToF-MS) that uses quasi-continuous beams, enabling high mass (>10,000) and high lateral resolution (~1  $\mu$ m) with a finely focused beam. Furthermore, swift heavy ion beams (>MeV) have a high transmission capability in matter, which allows their use for analysis of volatile samples such as liquids, solid–liquid interfaces and wet biological samples under ambient pressure (100,000 Pa). This technique creates new possibilities of ion beam analysis.

Current challenges and future applications of the SIMS technique with these new ion beams will be discussed.

[1] J. Matsuo, S. Torii, K. Yamauchi, K. Wakamoto, M. Kusakari, S. Nakagawa, M. Fujii, T. Aoki and T. Seki, Applied Physics Express 7, 56602 (2014)

[2] J. Matsuo, S. Ninomiya, H. Yamada, K. Ichiki, Y. Wakamatsu, M. Hada, T. Seki, T. Aoki, Surf. Interface Anal., **42**, 1612 (2010)

#### PL-5: Quantitative Low Energy Ion Scattering - accomplishments and challenges

#### P. Bauer

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Some fifty years ago the well-established ion beam techniques were applied at keV energies using noble gas ions and enhanced surface sensitivity was observed [1,2,3]. Very soon, Low Energy Ion Scattering (LEIS) developed to a widely used tool for analysis of structure and composition of solid surfaces [4,5] and references therein. Two features contribute to the great success of LEIS: first, its superb surface sensitivity and second, the fact that for most applications the yield of ions back scattered from one atomic species is independent of the other atoms present in the surface (absence of matrix effects), e.g. [6,7] and others.

Quantitative surface composition analysis is based on accurate knowledge of the differential scattering cross section  $d\sigma/d\Omega$  and the fraction of ions amongst the backscattered particles. Due to the low ion energies involved, a realistic model is required for the influence of electronic screening on nuclear scattering. For this purpose, the universal potential has been shown to be a good choice as long as not too low ion energies are employed [8]. To apprehend why in general LEIS is not sensitive to band structure effects requires understanding of the prevailing charge exchange processes – mainly Auger neutralization and reionization in a close collision [5]. Recently, it has been demonstrated that due to distinct neutralization efficiencies of different allotropic forms of carbon [9] the concentration of organic carbon on graphene can be quantified when an optimized set-up is used [10].

Very recently, interesting LEIS applications to ultrathin subsurface layers were reported. To gain quantitative information in this case one has to successfully handle additional processes: electronic stopping and multiple scattering related processes such as depth-dependent angular spread and increase in path length, loss of the unique relationship between final energy and scattering depth for a specific collision partner. It will be pointed out how to gain quantitative information on electronic stopping and how the influence of stopping and multiple scattering can be handled by use of Monte-Carlo simulations [11].

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[8] D. Primetzhofer et al., Nucl. Instr.Meth. 269, 1292 – 1295 (2011).

[9] S.N. Mikhailov et al., Nucl. Instr. Meth. B93, 210 – 214 (1994).

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#### PL-6: High-resolution ion depth profiling: beyond high-k materials

#### L. Goncharova

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Progressive reduction of dimensions and introduction of novel materials in sub-32 semiconductor technology nodes demands continuous improvement in high-resolution ion depth profiling (HRDP). One of the challenges here is that electron transport properties of these diverse materials are closely linked to the basic interactions at the interface. We will demonstrate novel application of HRDP methods, including medium energy ion scattering (MEIS), low energy ion scattering (LEIS), nuclear reaction profiling (NRP) and secondary ion mass spectrometry (SIMS) to high-k/metal gate, high-mobility (III-V) channel materials and ultra-shallow junctions. We will present the application of MEIS in combination with O-18 isotope labeling to follow oxygen migration in high-k/metal gate ultra-thin stacks. Recently it was established that deposition of an oxygen-gettering overlayers such as Ti on top of the highk metal oxide can result in reduction and even possibly elimination of the interfacial layer. Both oxygen exchange and interfacial silicon oxides growth rates were examined as a function of time, temperature, chemistry and crystallinity of dielectric layer. We find that incorporation oxygen is suppressed with addition of silica and nitrogen. Whereas presence of interstitials (O<sup>2-</sup>, OH<sup>-</sup>) is responsible for the fast O incorporation at the interface and interfacial oxide growth. We will also discuss application of complementary low-energy ion scattering and time-of-flight SIMS surface analysis to determine (a) high-k thin film growth mode for different deposition techniques; (b) the feasibility of backside SIMS on III-V high-mobility channel stacks to obtain high depth resolution at the source/drain /III-V interface.

# ABSTRACTS INVITED TALKS

#### I-1: SigmaCalc recent development and present status of the evaluated cross-sections for IBA

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The evaluation of the cross-sections for any particular reaction consists in the elaboration of the most accurate possible cross-sections through incorporation of the all relevant experimental data in the framework of nuclear physics theory. The SigmaCalc Web site was developed as an interface to the evaluated cross-sections in order to provide IBA practitioners with an access to the evaluated data. Initially it was hosted by Surrey University, UK and then by the IAEA while this was technically possible. After a devastating hacker attack in 2012 the SigmaCalc site was shut down along with other nuclear data services located at the IAEA server and after a long period of outage it was moved first back to Surrey University and then to the server of the Institute for Nuclear Power Engineering in Obninsk, Russia, where it is currently hosted (http://sigmacalc.iate.obninsk.ru). In the new 2.0 version of SigmaCalc the results of the evaluations performed during the SigmaCalc outage as well as the evaluations recently made including the cross-sections for PIGE were added. Many corrections were made in the previously evaluated cross-sections following a feedback from the SigmaCalc users and the results of benchmarks, i.e. integral experiments which consisted of measurements of chargedparticle spectra from well characterized uniform thick targets followed by standard simulations using microscopic cross-sections. The idea of benchmarking is to verify the validity of the data for the purpose they are intended for through their use in a typical application. A database of the available benchmarks was incorporated into SigmaCalc and a feature was developed to compare spectra simulated using the evaluated cross-sections against benchmarks. In order to provide users with possibility to compare evaluated differential cross-sections with the available results of the cross-section measurements the presentation of the calculated crosssections was modified to combine them with experimental data taken on the fly from IBANDL. In the case of the data for PIGE there is also possibility to compare the evaluated data both with experimental cross-sections and with thick target yields, the evaluated cross-sections being converted into the thick target yield on the fly. Though a majority of most wanted crosssections have already been evaluated there are still numerous gaps. Also the extension of the evaluated cross-sections up on energy is vital in some cases. The work on the cross-section evaluation and SigmaCalc site development is in progress with the ultimate goal of making SigmaCalc a versatile tool which will meet all needs of the IBA community in the cross-section data.

#### I-2: Study on dependence of structural evolution on Se sensitivity of semiconductors by c-RBS

#### D. Kabiraj (1), S. Hooda (1), S. Ojha (1), S. Mishra (2), A. Roy (2), D. Kanjilal (1)

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Studies of structural modification in GaAs, InP, Ge single crystal wafer irradiated by swift heavy ions (SHI) and pre-damaged Ge single crystal wafer on further irradiation with SHI are reported in detail. The irradiation by 100 MeV Ag was performed at room temperature with ion fluence ranging from 10<sup>12</sup> to 10<sup>14</sup> ions/cm<sup>2</sup> at beam current of 1-2 pnA. The damage evolution of irradiated samples was studied by Rutherford back scattering channeling (c-RBS) technique. In all the materials electronic energy loss (Se) of 100 MeV Ag is approximately same, ranging between 16-14 keV/nm and nuclear energy loss (S<sub>n</sub>) is approximately 0.1% of S<sub>e</sub>. We observe negligible damage formation in single crystal GaAs [1] and Ge whereas InP undergo heavy damage at higher fluences as detected by c-RBS measurement. Three sets of Ge samples with sub-threshold, at threshold and above threshold fluences of amorphization, as estimated by c-RBS were prepared by 100 keV Ar irradiation. After SHI irradiation of these samples, it is observed that first set of samples (sub-threshold amorphization) has undergone substantial recrystallization whereas in the second set of samples recrystallization is restricted to the region close to amorphous-to-crystalline boundary. Apart from results of c-RBS, Raman spectroscopy, high resolution transmission electron microscopy (HRTEM) and associated Fast Fourier transform (FFT) pattern studies confirm recrystallization. In the case of heavily damaged samples no change in amorphous phase is recorded. However, the instead swelling of the sample is observed after Ag irradiation. The relative swelling is found to increase steadily with increasing ion fluence up to  $3 \times 10^{13}$  ions cm<sup>-2</sup> and then to saturate at a maximum value of 20% at highest fluence of  $1 \times 10^{14}$  ions cm<sup>-2</sup>. The results are explained on the basis of thermal spike model.

[1] Shramana Mishra, Sudipta Bhaumik, Jaya Kumar Panda, Sunil Ojha, Achintya Dhar, D. Kabiraj, Anushree Roy. Nucl. Instr. Meth. Phys. Res. B 316 (2013) 192.

#### I-3: Transmission secondary ion mass spectrometry using 5 MeV C<sub>60</sub><sup>+</sup> ions

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There has been an increasing demand to extend accessible mass range in secondary ion mass spectrometry (SIMS) particularly for biological and biomedical molecular imaging. During the past two decades, various kinds of large clusters, such as  $C_{60}$  ions, argon gas cluster ions, water cluster ions, and metal cluster ions have been used as primary ions. It was shown that these cluster ions enhance emission of intact large molecular ions compared to monatomic ion bombardment. In SIMS, secondary ions emitted from a sample in the backward direction with respect to the direction of a primary ion are generally measured. If a specimen of a self-supporting thin film is used, the secondary ions emitted in the forward direction upon transmission of the primary ions can also be measured. So far, there have been only few studies about the transmission SIMS. Boussofiane-Baudin et al found small enhancement of secondary ion yield in the forward direction compared to the backward direction [1]. The origin of the enhancement was suggested to be the larger stopping power at the exit surface due to higher charge states achieved during the passage.

In this presentation, we demonstrate that large enhancement of the secondary ion yield of intact biomolecules can be achieved by combining the cluster ions (5 MeV  $C_{60}^+$ ) with the transmission SIMS [2]. We measured secondary ions emitted in the forward direction from phenylalanine amino acid films deposited on self-supporting amorphous Si<sub>3</sub>N<sub>4</sub> films. We found significant enhancement of the intact phenylalanine ion yield and large suppression of fragment ions compared to the backward direction. The origin of the observed enhancement and suppression will be discussed in the conference.

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[2] K. Nakajima, K. Nagano, M. Suzuki, K. Narumi, Y. Saitoh, K. Hirata, and K. Kimura, Appl. Phys. Lett. 104 (2014) 114103.
### I-4: Ion beam analysis of light elements in cultural heritage materials

### T. Calligaro (1,2)

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IBA application to Art and Archeology is a vibrant and innovating field. While PIXE remains the predominant technique owing to an easy implementation in air, alternate ones such as BS, ERDA and NRA exhibit attractive features, notably an unrivaled ability to non-destructively quantify light elements. Elements from hydrogen to fluorine are often present in cultural heritage materials where they play a key role; their direct determination is thus highly relevant. While external beam implementation of NRA, EBS and ERDA is challenging, progress made over years permits to carry them out at ambient pressure with a quality reaching that obtained in vacuum. After a quick review of the IBA techniques suitable for the measurement of light elements in heritage materials, this contribution focus on specific developments and results obtained with a small accelerator (less than 2 MV) and classical ion beams (excluding deuterons and heavy ions), in the present case with the AGLAE facility of the C2RMF.

Hydrogen is the first and most important light element. It is implied in hydration and weathering processes affecting, for instance, the surface of ancient glass. Concentration in hydrogen can be determined using ERDA with He beams to provide insights on the preservation state of historical objects and hydrogen depth profile can be used as an authentication criterion to detect fakes. Boron, a fluxing agent used in modern glassmaking was found to be also an important tracer in Renaissance glazed ceramic; its measurement by PIGE allowed discriminating between historical ceramic productions. Identification of organic matter usually requires sampling for identification by GC-MS or IRTF, but quantification of organic compounds is particularly difficult. EBS permits to determine non-destructively the absolute carbon content which is very useful, for example, in the estimation of the binder/pigment ratio in paintworks. The dating of ancient bones by C-14 AMS requires the presence of sufficient preserved quantities of ancient collagen. Quantitative concentrations of nitrogen can be obtained using the reaction <sup>14</sup>N( $\alpha$ ,p $\gamma$ )<sup>17</sup>O to quantify the presence of this amino acid, providing a quick and non-destructive test before undertaking the dating of prehistoric bones.

### I-5: Materials analysis using channeling and ionoluminescence in a helium ion microscope

### G. Hlawacek

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Helium ion Microscopy [1] is a versatile microscopy technique that provides high resolution imaging and nano-machining in combination with a high surface sensitivity and large depth of focus. It utilizes a narrow beam of  $He^+$  ions to achieve a lateral resolution of less than 0.5 nm. Backscattered Helium ions (BSHe) and secondary electrons (SE) can be used to obtain an image of the specimen.

When using crystalline samples channeling of the particles can occur. This effect can be exploited in several ways in the HIM. First of all it is possible to map out the different channeling directions and intensities and thus obtain information on the crystal structure of the sample. A simple geometrical model is introduced that can predict the channeling directions and relative intensities observed in the HIM [2]. By exploiting channeling and making use of the dechanneling contrast thin surface layers can be made visible in SE as well BSHe images [3]. We used this to observe composition and structural changes in a 2 ML thin silver layer on Pt(111). Work function differences as small as 40 meV between Ag and Pt rich areas on the surface reveal the position of mono—atomic surface steps. A regular arrangement of areas with reduces the channeling probability reveals the surface reconstruction of the top 2—3 ML which has a periodicity of only 5.8 nm.

lonoluminescence on the other hand allows to obtain information on defects in the bulk of the material. I will show results obtained for a variety of materials including semiconductors [4], rare earth containing perovskites and ionic crystals. The types of defects were identified and the influence of the scanning conditions on the IL signal has been investigated [5]. We used IL to map out the interaction volume of the beam in NaCl, and demonstrate the possibility of subsurface patterning. In our setup using a 35 keV He<sup>+</sup> beam and NaCl only 3vac/nm<sup>2</sup> are needed to obtain a detectable IL signal [6].

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#### I-6: State of the art of IBA analysis of high time resolution aerosol samples

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Most environmental studies on particulate matter (PM) are based on 24-h data. However many PM emissions as well as atmospheric transport and dilution processes may change within a few hours. As a consequence, the measurement of the PM composition with higher time resolution gives a better insight on aerosol emission, transport and dilution processes, as well as a better quantification of the human exposure. Furthermore, receptor models, which are used for the aerosol source apportionment, need series of samples containing material from the same set of sources in different proportions: increasing the time resolution provides samples that have greater between-sample variability and this strongly enhance the capabilities of these techniques. PM samplings with 1 h time resolution can be performed by the Streaker sampler (PIXE Int. Corp.). With this device the aerosol fine (<2.5  $\mu$ m) and coarse (2.5–10  $\mu$ m) fractions are simultaneously collected on two rotating substrata: this produces a circular continuous deposition of PM ("streak") on both stages. The analysis of these aerosol streaks with a properly collimated ion beam, which scans the deposit in steps corresponding to 1 h of aerosol sampling, provides the aerosol composition with hourly time resolution. It is worth noting that IBA techniques are unrivalled in the analysis of this kind of samples, which cannot be analysed chemical methods. At the 3 MV Tandetron accelerator of the LABEC laboratory aerosol Streaker samples have been analysed by Particle Induced X-ray Emission (PIXE) since a long time, and the external beam set-up dedicated to these analysis has been continuously improved to increase the measurement sensitivity and throughput (hourly time resolution studies produce a high number of very low mass aerosol samples: 168 for stage for weak). The adopted strategy is the use of a quite high proton current (100-500 nA) and a two-detector system, optimized for low-Z and medium-high-Z elements, to balance for the different cross sections. Si(Li) detectors have been replaced by SDDs, as they provide better resolution with modest cooling and can cope with higher counting rates, and, recently the SDD used for medium-high-Z elements has been duplicated to double the statistics. The optimised setup, together with a convenient choice of beam energy and suitable collecting substrata, allows obtaining good statistics in less then 1 min per 1-hour sample. Particle Induced Gamma-ray Emission (PIGE) is also routinely used, simultaneously with PIXE, to correct the underestimation of PIXE in quantifying the concentration of the lightest detectable elements, like Na or Al, due to X-ray absorption inside aerosol particles, while the use of elastic scattering for H, C, O and N detection in these samples is still under development. A detailed description of the experimental set-up and of the adopted measurement conditions will be given, together with some examples of application in recent PM field campaigns.

### I-7: Modern data analysis techniques for ion beam analysis

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Many experimental methods in ion beam analysis have reached a very high standard with respect to physics understanding and hardware capabilities and often further progress is expected to be only incremental. An area which still holds the promise for significant progress is, however, the optimized collection and improved analysis of the gathered data. Recent developments in the handling of ill-posed and/or undetermined problems based on sparse regularization or fast inference methods still wait to be explored within the ion-beam community. Several examples, mostly centered around NRA-measurements will demonstrate the potential of the new approaches to ion-beam analysis.

#### I-8: Super-resolution imaging with focused MeV ions: Fluorescence and Structural imaging

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Imaging fluorescence generated by MeV ions in biological systems such as cells and tissue sections requires a high resolution beam (< 100 nm), a sensitive detection system and a fluorescent probe that has a high quantum efficiency and low bleaching rate. For cutting edge applications in bioimaging, the fluorescence imaging technique needs to break the optical diffraction limit allowing for sub-cellular structure to be visualized, leading to a better understanding of cellular function. In a nuclear microprobe this resolution requirement can be readily achieved utilizing low beam current techniques such as Scanning Transmission Ion Microscopy (STIM). In recent times, we have been able to extend this capability to fluorescence imaging through the development of a new high efficiency fluorescence detection system. This paper discusses the current state-of-the-art in super-resolution fluorescence microscopy using focused MeV ion beams at the Centre for Ion Beam Application (CIBA), NUS Singapore.

#### I-9: MEIS analysis at the nanoscale, advantages/disadvantages versus X-Rays and TEM

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Numerous analyzing methods have been successfully and widely employed in material sciences in order to obtain chemical, elemental, morphological and structural information in bulk materials as well as in layers of various thicknesses. The emergence, since the middle of the 80s, of nanoscience poses new questions for analysis techniques, including IBA, in terms of spatial resolution and sensitivity. The challenge posed by the analysis of nanostructures is different in each case. Rather than attempting to describe all the possible situations, I will focus on the structural study of single crystal nanostructures.

GaN quantum dots deposited on (and embedded in) AlN give an interesting example of structural analysis at the nanoscale. Indeed, the strain state of these quantum dots embedded in a matrix is an important parameter which governs to some extent their optical properties. The most usual wurtzite crystallographic phase of these two materials exhibits both piezoelectric and spontaneous polarization. Because of the elevated value of the piezoelectric constants, a huge internal electric field is currently observed in nitride heterostructures. As a consequence of the resulting quantum confined Stark effect, a strong red shift is induced, leading to luminescent emission at energies smaller than the GaN gap value for dots higher than 2.5 nm.

Amongst the different available techniques to measure strain, the Geometrical Phase Analysis (GPA) of HRTEM images, has the advantage to provide strain map of the nanoobject with, in principle, an atomic resolution [1]. However, large strain fluctuations are observed in the strain map along the c axis and, in addition, the expected vertical strain gradient is not visible on these images. The comparison with the electron diffraction techniques, namely CBED and NBED, will also be discussed.

An alternative possibility is the use of x-ray diffraction, in grazing incidence to enhance the contribution of the dots (and AIN capping) with respect to that of the substrate. More precisely, by varying the scattering power of a selected element, for instance Ga, it is possible to localize specifically GaN regions in reciprocal space. This is the purpose of Multiwavelength Anomalous Diffraction (MAD) measurements, which consists in recording the scattered intensity as a function of the energy across the absorption edge of an element, namely Ga in the present case. The position of the Ga signal maximum along the [10-10] direction is directly related to the average in-plane strain state and structure in the QDs [2]. The diffraction peak corresponding to the GaN QDs is clearly due to the presence of an in-plane strain gradient within the dots but also to the finite lateral size of the dots (about 40 nm). Therefore, the extraction of a strain profile is not straightforward and model dependent.

By contrast, Medium Energy Ion Scattering (MEIS) can measure the deformation profile of quantum dots with a depth resolution in the monolayer range [3]. Indeed, ion blocking reveals the angular position of a nucleus with respect to the scattering center that can be directly related to the out-of-plane to in-plane lattice parameters ratio. Of course, the ion beam spot size is much larger than the dots and the MEIS measurement averages the strain profile over

about  $6 \times 10^8$  dots. From this point of view, MEIS is similar to X-rays techniques but the contrary to X-rays, the depth strain profile along depth within the dots can be accurately measure and its dependence clearly appears comparing the profile of uncapped dots in figure 1(a) to the one of capped dots in figure 1(c). Another example of a MEIS structural analysis will be given by the measurement of in-plane and out-of-plane mosaïcity of a set of GaN nanowires and the comparison with XRD results.



FIG. 1. (left) Scheme of the scattering geometry in the (11-20) plane for MEIS experiments. (a) Depth profile of the ratio c/a in one plane of uncapped GaN QDs. (c) Depth profile of the ratio c/a in one plane of GaN QDs capped with 20 MLs of AlN. Experiment performed at the Korea Research Institute of Standards and Science, Daejeon (South Korea) [3].

[1] E. Sarigiannidou et al., Appl. Phys. Lett. 87 (2005) 203112

[2] J. Coraux et al., Phys. Rev. B 74 (2006) 195302

[3] D. Jalabert et al., Phys. Rev. B 72 (2005) 115301

#### I-10: PIXE using large area transition-edge sensor array

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In the field of PIXE the division between the use of the two detector types has for a long time been clear: most commonly Si or Ge based energy dispersive (ED) detectors are used when a broad energy range and large solid angle are needed, and wavelength dispersive (WD) detectors are used to obtain ultimate energy resolution. The limitations of the WD detectors are the narrow energy range and small solid angle.

Today energy dispersive transition-edge sensors (TES) have matured to the state that they are used in number of applications, thanks to their superior energy resolution and sensitivity. Here we present the Jyväskylä TES-PIXE measurement setup, in which TES detector arrays are used to detect X-rays in proton and heavy ion PIXE. The energy resolution of a TES detector, when used in PIXE, is over an order of magnitude better compared to silicon drift detectors (SDD) and comparable to that of WD detectors. This makes it possible to recognize spectral lines in materials analysis that have previously been impossible to resolve over large energy range (1.0–15 keV), and even obtain chemical information from the analyzed sample. Our 160 sensors with total active area of 15.6 mm<sup>2</sup> are cooled to the operation temperature of about 65 mK.

In this paper the design, electronics and data acquisition of the Jyväskylä TES detector array are described. The merits of the TES detector array in the analysis of different materials research problems from thin films to cultural heritage objects will be presented. The benefits and shortcomings of the TES detector in comparison to WD and silicon drift detectors will be



discussed.

Figure 1: The reference material NIST SRM1157 (Tool steel) measured with TES-detector in vacuum using 2 MeV protons and with SDD-detector in air using 3 MeV protons.

[1] M.R.J. Palosaari et al., Journal of Low Temperature Physics 176 (2014) 285.

### I-11: Complementing X-ray and MS based techniques in studies of trace elements in biological systems

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Trace elements are essential components of biological structures, but at the same time they can be toxic at concentrations beyond those necessary for their biological functions. In addition, the toxicity can be extended to other non-essential elements of very similar atomic characteristics that can mimic the properties of a trace element. Trace element malnutrition affects more than half of the world's population, while on the other hand industrialization, traffic and extensive use of fertilizers have resulted in exceedingly high concentrations of nonessential elements in food crops, posing risks to human health. In order to be able to develop and improve phyto-technologies that enable production of safe and quality food, knowledge on the basic mechanisms involved in trace and non-essential element uptake, transport, accumulation and ligand environment in plants is needed. Such studies are now-days supported by highly sophisticated techniques (micro-PIXE, SR-micro-XRF, LA-ICPMS, (micro)-XAS, TOF-SIMS and MeV-SIMS) enabling imaging of element distribution and speciation in plant tissues and cells with high spatial resolution and sensitivity, as well as imaging of organic molecules that play a role in metal detoxification and are altered due to metal toxicity. Selected case studies of metal distribution and speciation in selected model plant species, achieved by interdisciplinary work, will be presented.

### IAEA Panel on Ion Beam Techniques Roadmap

### Panel co-ordinator:

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Ion Beam Techniques (IBTs) encompass a suite of analytical and modification techniques in which energetic (0.1 to 100 MeV) beams of charged particles (Z=1, 2 and higher) are directed onto a material to be analysed or modified. In analysis, the composition and in some cases structure of the material are inferred from the nature of detected induced radiation: the type of particle, and its probability distributions, as a function of incident beam energy and direction, and detected particle energy and angle. IBTs have been used for over 50 years now, and much of the underlying physics, data, and machinery are considered to be mature.

Individual accelerators often find application across a broad range of disciplines and domains. The underlying technology associated with megavolt accelerators is thus common across many fields of application, so that developments and evolution driven by one particular application will feed through to other areas.

The aim of this Panel to discuss where further concerted research efforts are likely to lead to improved performance or new competitive IBTs, in view of the development of an Ion Beam Techniques Roadmap. The primary Goals of the Roadmap are:

- To keep Accelerator Based Ion Beam Techniques at the forefront of scientific endeavour
- To improve key aspects of performance of IBA
- To show significant impact of IBTs on societal problems
- To significantly increase human knowledge
- To ensure transmission of competencies across generations
- To promote internationally the adoption of Best Practice

The conference participants will have an opportunity to provide their feedback on the technological developments during the conference, and also in the form of a questionnaire. Please also visit the **IAEA Accelerator Knowledge Portal** for more information on the IBT Roadmap and provide your feedback even before/after the conference: <a href="http://nucleus.iaea.org/sites/accelerators/Pages/default.aspx">http://nucleus.iaea.org/sites/accelerators/Pages/default.aspx</a>

### ABSTRACTS CONTRIBUTED TALKS

### O-1: Mott vs Rutherford Scattering: Wave-Particle Duality of MeV Carbon, and Boron Ion Scattering from Thin Graphite Foil

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In Rutherford scattering experiments, we always treat ions as particle where classical mechanics such as conservation of energy and conservation of momentum during scattering holds. Mott Scattering is a special case in which ion projectile is identical to the target atom such as  $C^{12}$  scattered from  $C^{12}$ , and Quantum interference occurs due to the ambiguity of scattered particle and the recoiled particle. As a consequence, angular dependence of the particle yield is highly oscillatory, and this is recognized and documented before. It is interesting to note that the de Broglie wavelength of MeV particle is extremely small. For example, 4 MeV  $C^{12}$  has a wavelength of 8.3 fm, yet behaves like a wave while scattered from  $C^{12}$ .

In this talk, we will show experimental results of  $C^{12} - C^{12}$  scattering experiment vs  $B^{11}$  scattered from graphite. One is understood by QM, while the other is purely classical. This scattering experimental comparison demonstrates the wave-particle duality of ion beam. We will draw comparison of ion beam wave properties with Young's interference experiment on light beam going thought a double slits. In the talk we will also propose future experiments to explore more QM wave nature of ion beam.

## O-2: A Review on Benchmarking of Proton Elastic Scattering - Implementation in the cases of <sup>nat</sup>Si, <sup>19</sup>F, <sup>nat</sup>B and <sup>nat</sup>O

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The implementation of all Ion Beam Analysis (IBA) depth profiling techniques critically depends on the accuracy of the available differential cross sections for the reactions involved. Unfortunately, the existing experimentally determined differential cross-section data are in many cases quite scarce and/or discrepant, thus their reliability is highly questionable. On the other hand, the evaluated cross-sections, when available, are the most reliable ones to be used in analytical studies, since they involve a critical assessment of the experimental datasets, followed by a proper tuning of the corresponding nuclear model parameters. However, it is important to point out that most of the evaluated datasets are still not adequately validated. A carefully designed benchmarking experimental procedure (i.e. the validation of differential cross-section data via the acquisition of thick-target spectra followed by their simulation) is thus mandatory. Benchmarking can also provide the necessary feedback for the adjustment of the parameters of the nuclear model used in the evaluation process, and can help in assigning realistic uncertainties to the cross sections. Moreover, in the absence of evaluated cross sections, it can indicate recommended experimental datasets.

Recently, a dedicated effort was made to thoroughly document this procedure [1], followed by a technical meeting organized by IAEA. In the present review an attempt is made to present the recommended steps and to critically assess the problems of the benchmarking process in the following cases:

- (1) In <sup>nat</sup>Si(p,p<sub>0</sub>), for E<sub>p</sub>=1.5-3.5 MeV, where channeling perturbations in crystalline wafers, if not carefully treated, can seriously affect the accuracy of the measurements, while the size of the powder used in pressurized tablets can affect the shape of resonances in the experimental thick-target yield spectra,
- (2) in <sup>19</sup>F(p,p<sub>0</sub>) and <sup>nat</sup>B(p,p<sub>0</sub>), for E<sub>p</sub>=1.5-2.5 MeV, where, for the removal of the important underlying  $\alpha$ -particle background,  $\Delta E/E$  telescopes have been implemented, and
- (3) in  $^{nat}O(p,p_0)$ , for  $E_p=1.5-4$  MeV, where target related effects (e.g. roughness) need to be taken into account.

[1] V. Paneta, J. L. Colaux, A. F. Gurbich, C. Jeynes, M. Kokkoris, Nucl. Instr. and Meth. B328 (2014), p. 1-7.

# O-3: IAEA CRP on Development of a Reference Database for Particle-Induced Gamma-ray Emission (PIGE) spectroscopy

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Particle-Induced Gamma-ray Emission (PIGE) is a powerful analytical technique that exploits the interactions of rapid (~1-10 MeV) charged particles with nuclei located near a sample surface to determine the composition and structure of the surface regions of solids by measurement of characteristic prompt  $\gamma$  rays. The potential for depth profiling of this technique has long been

recognized, however, the implementation has been limited owing to insufficient knowledge of the physical data and lack of suitable user-friendly computer codes for the applications. Although a considerable body of published data exists in the nuclear physics literature for nuclear reaction cross sections with y rays in the exit channel, there was, until now, no up-todate, comprehensive compilation specifically dedicated to IBA applications. A number of PIGE cross-section data had already been uploaded to the Ion Beam Analysis Nuclear Data Library (IBANDL) (http://www-nds.iaea.org/ibandl) by members of the IBA community by 2011, however a preliminary survey of this body of unevaluated experimental data has revealed numerous discrepancies beyond the uncertainty limits reported by the authors. Using the resources and coordination provided by the IAEA, a concerted effort to improve the situation was made within the Coordinated Research Project on the Development of a Reference Database for PIGE spectroscopy, from 2011 to 2015. The aim of the CRP was to create a data library for Ion Beam Analysis that contains reliable and usable data on charged particle y-ray emission cross sections that would be made freely available to the user community. As the CRP has reached its completion, we shall present its main achievements, including the results of nuclear cross-section evaluations and the development of a computer code that will become available to the public allowing for the implementation of a standard-less PIGE technique.

### O-4: Drawing the geometry of 3d transition metal – boron pairs in silicon by means of electron emission channeling experiments

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The behavior of transition metals (TMs) in silicon is a subject that has been studied extensively during the last six decades. Their unintentional introduction during the Si production, crystal growth and device manufacturing have made them difficult contaminants to avoid. Once in silicon they easily form deep levels, either when in the isolated form, where they act as fast diffusers, or when forming precipitates. These effects usually reduce the efficiency of siliconbased devices, being dramatic, in particular, in photovoltaic applications. To mitigate these effects, the industry has relied on a class of procedures based on gettering techniques where TMs are immobilized away from the active region of devices, e.g. by using p-type layers rich in immobile boron dopants which easily pair with positively charged TM impurities through Coulomb interactions. Although some electrical properties of TM-boron pairs have been investigated experimentally, the information about their geometries is still scarce. In that respect, electron paramagnetic resonance [1] and Mossbauer spectroscopy [2] measurements have suggested a configuration where one of the most important TM in Si, Fe, is located on interstitial sites with tetrahedral symmetry next to substitutional boron acceptor dopants. Furthermore, density functional calculations have suggested the existence of a breathing mode relaxation around the boron atom within this arrangement [3]. A direct experimental confirmation of such a configuration is, however, still missing.

In this work we assess experimentally the lattice location of some TMs in silicon when pairing with boron acceptors. One effective way is to use the electron emission channeling technique where the location of radioactive isotopes is deduced by the channeling effects of the  $\beta^{-}$  particles emitted by the probe atoms. In the present investigation, we used the TM isotopes  ${}^{56}$ Mn,  ${}^{59}$ Fe,  ${}^{61}$ Co and  ${}^{65}$ Ni. We have observed that the lattice location of all TMs changes profoundly with doping. In particular, we have found that the majority of  ${}^{56}$ Mn,  ${}^{59}$ Fe and  ${}^{61}$ Co is located on sites near the tetrahedral interstitial site in *p*-type silicon, along the whole used annealing temperature range. On the contrary, in the case of  ${}^{65}$ Ni the interstitial fraction was much reduced. This confirms that the charge state of the TMs plays a major role in their immobilization, since Fe, Co and Mn are most positively charged in *p*-type silicon while Ni is assumed to be neutral. We have found that the detected displacement with respect to the ideal tetrahedral interstitial site is in accordance with the predicted displacements of transition metal-boron pairs. We discuss in detail the application of the emission channeling technique in this system in extracting information about the geometries of such pairs.

- [1] W. Gehlhoff *et al.*, Solid State Phen. 32 (1993) 219
- [2] H. P. Gunnlaugsson et al., Hyperfine Interact. 169 (2006) 1315
- [3] M. Sanati et al., Phys. Rev. B 76 (2007) 125204

# O-5: Atom location and ion damage studies of gold hyperdoped silicon via ion implantation followed by pulsed laser melting

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Previously, we have demonstrated that silicon hyperdoped by gold through ion implantation followed by pulsed laser melting (PLM) shows enhanced absorption in the infrared, making it a promising material for photodetector devices [1]. It has been hypothesized that this enhanced absorption relates to discrete sub-band gap levels introduced by substitutional gold in silicon, though this relationship is not yet quantified. We note also that high concentrations of gold may lead to cellular breakdown via solute partitioning during rapid solidification following PLM [2]. To optimise device performance, we employ Rutherford backscattering spectrometry combined with channeling (RBS/C) to determine the degree of substitutionality for supersaturated gold of different doses, implant conditions and laser powers. Angular scans from major axes were undertaken for precise atom location. Furthermore, we investigate the mechanism of cellular breakdown and its impact on optical absorption by combining RBS and transmission electron microscopy analysis. Finally, we explore the effect of excessive helium ion damage during the RBS-channeling analysis on the atom location of the metastable supersaturated gold in silicon.

[1] J. P. Mailoa et al., Nature Communications 5, 3011 (2014)

### [2] D. Recht et al., Journal of Applied Physics 114, 124903 (2013)

### O-6: Ion Channeling Revisited

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An MS Excel program has been written that calculates virtually all ion channeling parameters for any ion and energy on any cubic bcc, fcc or diamond lattice crystals for <uvw> axes and [hkl] planes up to (555). All of the tables and graphs in the three Ion Beam Analysis Handbooks that previously had to be manually looked up and read from were programed into Excel in handy lookup tables, or parameterized, for the case of the graphs, using rather simple yet accurate functions. The program offers an extremely convenient way to calculate axial and planar half-angles, minimum yields, and the effects on half-angles of amorphous overlayers. The program can automatically generate standard stereographic projections of axial and planar channeling with the half-angles graphically represented to scale. The program can also generate a new orthogonal projection of channeling that shows the channeling probability as a function of polar and azimuthal crystal orientation angles. This projection is used to theoretically predict accidental (i.e. unintentional) channeling probabilities for randomly oriented crystals or crystallites. This becomes an important issue when simulating the creation by energetic neutrons of point displacement damage and extended defects using beams of ions. The program is open source and can be downloaded from the website:

http://www.sandia.gov/pcnsc/departments/iba/ibatable.html

### O-7: HI-ERDA versus SIMS to study helium profiling in pure bcc metals

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Helium effects on microstructural and mechanical properties are key issues in fission or fusion materials research. Due to its extremely low solubility in most metallic alloys, helium strongly interacts with pre-existing and radiation-induced defects, leading to structures degradation. In order to extend the longevity of structural materials in the advanced reactors, it is necessary to comprehend the helium diffusion down to the atomic-scale.

Pure bcc metals (V, Fe, Ta, Nb and Mo) were implanted with <sup>4</sup>He<sup>+</sup> ions at energies ranging from

45 keV to 90 keV with a fluence equal to 5x10<sup>16</sup> ions/cm<sup>2</sup>. After implantation, He depth profiles were deduced either from high energy heavy ion-induced elastic recoil detection analysis (HI-ERDA) or from secondary ion mass spectrometry (SIMS) profiles. Both techniques give similar results: all samples exhibit a projected range of around 200 nm, in agreement with TRIM theoretical calculations. The latter results were then compared with the He bubble distribution obtained by Transmission Electron Microscopy (TEM). TEM micrographs reveal that the nanometric bubbles are homogeneously distributed in the microstructure.

The main aim of this study is to compare the experimental conditions and performances allowed by the use of HI-ERDA and SIMS in quantitative analysis of helium in pure bcc metals. Complementary post-implantation annealing treatments are carried out to characterize the <sup>4</sup>He-depth profile evolution.

### O-8: Ambient Analysis of Liquid Materials with Wet-SIMS

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A measurement system that operates at ambient conditions is required for the analysis of biological samples and solid-liquid interfaces, because the samples contain relatively large amounts of volatile materials, such as water, alcohols, and fatty acids. Particle induced X-ray emission (PIXE) has been used to analyze such samples in air, but this technique does not provide molecular information. Recently, an ambient secondary ion mass spectrometry (SIMS) system has been developed, but volatile liquid (wet) samples are difficult to measure using conventional SIMS, because samples must be dried and introduced into a high vacuum chamber. The mean free path of ions with energy in the keV range is very short in low vacuum and these ions cannot penetrate the surface. In contrast, wet samples can be measured using heavy ions in the MeV-energy range without dry sample preparation because of the high transmission capability of these ions at low vacuum pressure. MeV-energy ion beams also excite electrons near the surface and enhance the ionization of high-mass molecules and thus fragment-suppressed SIMS spectra of ionized molecules can be obtained. We have developed an ambient analysis system with secondary ion mass spectrometry for wet samples (Wet-SIMS) that operates from low vacuum to atmospheric pressure using MeV-energy heavy ion beams. The system is equipped with fine nozzles that avoid vacuum degradation at both the primary beam incidence side and the secondary ion measurement side, even when the target chamber is filled with gaseous He at atmospheric pressure. Water evaporation was suppressed in He at atmospheric pressure and a solution of benzoic acid could be measured using MeV-energy heavy ions without dry sample preparation.

### O-9: Hydrogen behavior in materials after single and triple-beam irradiations characterized by SIMS and $\mu$ Raman

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Implantation of hydrogen in materials is used for several applications: in electronics for doping or thin film production and for nuclear studies to simulate gas accumulation during reactor operation. In the last case, interaction of hydrogen with the host matrix can cause damage through embrittlement, hardening, and swelling. During and after implantation, hydrogen diffuses and can be trapped, resulting in possible changes to its chemical bonding or molecular structure. In order to characterize the behavior of hydrogen implanted in depth in materials, we have combined two techniques. SIMS (Secondary ion mass spectrometry) is used for profiling H from the surface to about 5  $\mu$ m in depth without any preparation of the sample.  $\mu$ Raman is applied on cross-sections of the samples. Mappings of Raman spectra as a function of the depth are obtained along the ion path evidencing possible matrix-H bonds or H<sub>2</sub> molecular structure. This methodology has been applied to different materials: Si, SiC, Fe and ODS steel (Fe-12%Cr-4%Y<sub>2</sub>O<sub>3</sub>). We have observed in silicon the formation of Si-H and hydrogen-vacancy bonds. In SiC, we have observed in addition C-H bonds. In iron and the ODS steel, H profiles have been measured by SIMS but no Raman bands have been obtained in case of single beam irradiation. These results show that hydrogen is present in atomic form. Conversely, when ODS steel is irradiated in triple beam condition (Fe<sup>8+</sup>, He<sup>+</sup>, H<sup>+</sup>), H<sub>2</sub> molecules are detected at a depth corresponding to the ion ranges, suggesting a synergistic effect between H and He or H and damages. Dual irradiations ( $Fe^{8+}$ ,  $H^+$ ) and ( $He^+$ ,  $H^+$ ) are in progress to discriminate these effects.

#### O-10: Glass deterioration mechanisms using Total-IBA of Rosslyn glass

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Using Surrey's in-air scanning focussed microbeam with a large-range motorised sample manipulator and a high count-rate spectrometry system for both particle and X-ray detectors, we have analysed deteriorated stained glass from the Rosslyn chapel (built in 1446). The glass panels were manufactured around 1875 with a "grisaille" technique: the image is defined by Pb-rich monochrome paints fired into the glass. Many panels have suffered extreme image loss due to an aggressive internal environment, and this work aims to uncover the lost imagery by revealing the original artists' paint strokes. Using Total-IBA (self-consistent ion beam analysis) methods we have demonstrated that the lost grisaille leaves an (invisible) surface

enrichment of grisaille components, and that the lost image can efficiently be recovered by PIXE (particle-induced X-ray emission) with high acuity, providing invaluable guidance for the restorers and conservators.



Heavily damaged glass panel from the Rosslyn Chapel: photograph (left, 20 cm across) and reconstructed PIXE image (right) combining the images from both the front (internal) surface, and the back (external) surface containing the halo painted in silver.

From the large data cubes collected (512×512 pixels, front and back surfaces) we will extract PIXE/EBS spectra of selected areas, infer the composition modifications as a function of depth from the surface, and interpret these profiles by modelling the grisaille technique used and the expected glass deterioration mechanisms informed by our knowledge of the environmental history of the panel. Currently there are only a few studies with reliable elemental depth profiles, using spot analyses (not mapping).

This project is partially funded by The Rosslyn Chapel Trust (Registered Charity number SC024324) Lord and Lady Rosslyn, Owners and Trustees, and WREN Heritage Fund (www.wren.org.uk)

### O-11: MeV SIMS - a tool to study modern paint materials and their stability

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Development of the modern industry in the 20<sup>th</sup> century caused production of a large variety of synthetic organic materials (binders and pigments) that have been used extensively by modern and contemporary artists. Degradation mechanisms and aging of those materials are typically studied with methods that can provide some information on the molecular structure like Gas Chromatography Mass Spectrometry (GC/MS), Pyrolysis-GC/MS, FTIR or Raman spectroscopy, Atomic Force Microscopy (AFM), and Nuclear Magnetic Resonance (NMR). In the present work for the first time, potential of the Secondary Ion Mass Spectrometry (SIMS) technique with MeV heavy ion excitation was explored for identification of synthetic organic materials used for paints materials. Self-made paint mock-up samples were prepared by mixing different pigment powders with alkyd and acrylic binders on glass slides as well as some commercially available paint formulas. Additionally, the mock-ups were analysed using MeV SIMS setup at the heavy ion microprobe in Zagreb: unaged, aged for two (UV1) and four months (UV2). As a primary ion beam focused 5 MeV Si<sup>4+</sup> was used. We will demonstrate that different synthetic

organic pigments and binders can be easily identified by their molecular masses with MeV SIMS. Also, measurements have shown that it was possible to distinguishing among different blue phthalocyanines (PB 15:x) as well as between different binders (alkyd and/or acrylic) in the prepared mock-ups. At present, the potential of chemical imaging of aged paint surfaces compared to unaged ones is tested and first results will be reported.

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## O-12: Development of the time-resolved ion beam luminescence technique and its application to the provenance studies of lapis lazuli

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A setup for time-resolved ion beam induced luminescence (TRIBIL) has been developed at the pulsed beam facility of the 3 MV Tandetron accelerator of the LABEC laboratory. Measurements are performed exploiting the pulsed beamline DEFEL [1], consisting of two electrostatic deflectors operating together to produce a pulsed beam from a continuous one. By controlling the beam current it is possible to obtain a variable and finely controlled number of particles per bunch. The new TRIBIL setup, here described, is based on a photomultiplier tube (PMT) detecting in vacuum the light emitted by the target. Optical filters in front of the PMT window allows for wavelength discrimination. The signal from the PMT is sent to the VME sampling ADC (CAEN/V1724). The digitizer (250 Ms/s sampling rate) was programmed to start acquisition in coincidence with the signal from the DEFEL system, corresponding to the bunch creation. The acquisition time window can be adjusted in the  $\mu$ s-ms range; a user-definable trigger delay can be used to acquire longer signals. With luminescent targets, for each trigger signal we obtain a waveform whose initial part is acquired before the ions hit the target (baseline determination), while the rest of the signal correspond to the light burst induced by the beam bombardment and to its decay after the excitation is turned off. Pushing forward a study started in 2007 [2, 3], TRIBIL has been applied to the provenance study of lapis lazuli, a semi-precious stone admired for its blue color. Lapis lazuli has been used since the early civilizations as an ornamental stone and it can be found only in a few places (Afghanistan, Siberia, Chile). As shown in previous studies [3], the IonoLuminescence (IL) technique can help to discriminate materials of different origins, being the emitted light dependent on the nature and traces of the minerals composing the stone. We have thus proposed the use of TRIBIL to contribute to finding new markers of lapis lazuli provenance, since it can separate overlapping features not distinguishable through steady-state luminescence. Lapis lazuli of known certified provenances were studied. Since IBIL is dependent on dose and dose rate [4], we used these parameters to characterize the components of the luminescence decay. Preliminary results show differences in the TRIBIL signals collected from samples of different provenances: it was possible to highlight that the Chilean lapis lazuli is characterized by decay times longer than those of all the other provenances. IBIL allied with lifetime measurements can thus constitute a strong and innovative method to discriminate among diverse provenances.

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## O-13: SIMS on FIB instruments: a powerful tool for high-resolution high-sensitivity analytics at the nano scale

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FIB-based instruments become of increasing importance in materials and life sciences. They are an ideal tool for high resolution 2D and 3D imaging and for nanofabrication (nano-machining and deposition, etc.), yet their analytical capabilities are currently limited. By contrast, secondary ion mass spectrometry (SIMS) is an extremely powerful technique for surface analysis owing to its excellent sensitivity, good dynamic range, high mass resolution and its ability to differentiate between isotopes. Adding SIMS capability to FIB instruments offers not only the prospect of obtaining elemental information at much higher resolution than state-ofthe-art SIMS instruments, but allows for a direct correlation of SIMS images with high resolution secondary electron images. Past attempts of integrating mass spectrometers on FIB instruments were rather unsuccessful because of poor detection limits which were due to (i) low ionization yields of the sputtered matter, (ii) extraction optics with low secondary ion extraction efficiency, and (iii) mass spectrometers with a low duty cycles and/or transmission. In order to overcome these limitations, reactive gas flooding during FIB-SIMS has been investigated and compact high-performance magnetic sector mass spectrometers with dedicated high-efficiency extraction optics developed by our group. Secondary ion yields have been maximised by using O<sub>2</sub> flooding for positive secondary ions and Cs flooding for negative secondary during the SIMS analysis. Compared to Ga or noble gas primary ion species without flooding, the ionisation probabilities have been increased by up to 4 orders of magnitude, which leads to detection limits varying from  $10^{-3}$ - $10^{-7}$  for lateral resolutions between 10 nm and 100 nm. Images recorded with poorer lateral resolution but excellent detection limit (e.g. 1 ppm @ 50 nm) can be correlated with secondary electron images recorded at high lateral resolution using Ga, He, Ne or electron beams, overcoming the trade-off between detection limit and smallest feature size. The SIMS add-on has been optimised for FIB-SIMS applications. The extraction optics have maximum efficiency without distorting the primary ion beam and are coupled to a specially designed compact magnetic sector mass spectrometer with a high mass resolution (>2000), full mass range (H-U) and detection of several elements in parallel. The results are very encouraging and the prospects of correlating SIMS with high-resolution imaging and nanofabrication instruments are very interesting. High-resolution microscopy and high-sensitivity elemental mapping are combined on a single instrument, which represents a new level of correlative microscopy.

### O-14: Ion Beam Analysis in the Helium Ion Microscope

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Helium ion microscopes (HIM) have turned into a frequently used imaging device in several laboratories around the world. Beside a sub nano-meter resolution and its high field of depth the latest generation of HIM devices (Zeiss Orion NanoFab) offers the ability to make use of Neon ions enabling additional opportunities in terms of surface modifications on the nm scale [1].

While the image generation in a HIM is based on evaluating the amount of secondary electrons the information carried by the back-scattered He/Ne projectiles (BSP) is not taken into consideration at the moment. Thus the HIM offers excellent topographic imaging capabilities but chemical information (in terms of elemental composition) of the surface is not accessible. Nevertheless back-scattered particles carry that information and may be used to provide additional contrast mechanism(s). First attempts to measure BSP energy spectra were done by Sijbrandij *et al.* [2] and gave evidence for the general feasibility but also revealed that a quantitative chemical analysis of thin layers would require development of more sophisticated detection concepts than those used in their experiments (silicon surface barrier detector).

In the present contribution we show the development and the implementation of a Time-of-Flight back-scattering spectrometry (ToF-BS) technique within our HIM. Pulsing the primary ion beam by using the existing beam blanker with a customized pulsing electronics enables us to generate pulses as short as below 10 ns. BSP detection is done by means of a micro channel plate detector. Our measurements demonstrate that this technique is capable to achieve an energy resolution as good as 2 keV (for 30 keV He incident ions) by simultaneously keeping the spatial resolution in the order of a few 100 nm. We further show that with some slight modification the presented setup can be utilized to acquire ToF spectra of sputtered particles as well, thus enabling lateral resolved ToF-SIMs within the HIM.

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#### O-15: Applications and benefits of a Xe plasma FIB-SEM

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The combination of a focused ion beam (FIB) with a scanning electron microscope (SEM) in a FIB-SEM or DualBeam instrument provides a wide range of applications for material characterization. So-called FIB tomography [1,2] has enabled 3D imaging of structure, crystallography (via EBSD) and chemistry (by EDS) invaluable for a range of characterization applications. However the conventional gallium ion FIB also presents some limitations. First, there are many scenarios in materials science that require analysis across length scales up to many 100s of microns that lie beyond what a Ga ion FIB can typically access, relating to grain microtextures, grain neighborhoods, grain boundaries, inclusions and cracks. Second, gallium-based FIB at 30 keV induces a ~20 nm damage layer in crystalline samples that can hamper subsequent analysis.

In this paper we discuss the application of a DualBeam system equipped with a Xe Plasma beam FIB, the FEI Helios PFIB. This  $Xe^+$  beam provides higher currents and higher sputter yield (leading to a 20-50× throughput improvement) as well as significantly reduced material damage.

We demonstrate the performance of the PFIB through a large dimension (~100  $\mu$ m) 3D tomography study of steel samples. The results show that PFIB tomography produces low levels of surface damage as evidenced by good channeling contrast as well as clear Kikuchi patterns from the machined surface.

Other ongoing experimental studies across a wide range of materials from paint to ceramics suggest the Xe PFIB can routinely, quickly and with high material quality provide 3D serial section tomograms over dimensions of many hundreds of microns. This not only expands the accessible length scales for FIB-SEM material characterization, it also means an improvement of the results compared to Ga FIB thanks to a remarkable surface finish quality.

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### O-16: Application of micro-ion-beam analysis to investigate the distribution of Cs in silt particles for environmental remediation of Fukushima

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After the great east Japan earthquake on 11 March 2011, the big tsunami caused Fukushima Dai-ichi nuclear power plant accident. The northeastern region of Japan (mainly Fukushima prefecture) was contaminated with the radioisotopes of <sup>131</sup>I, <sup>132</sup>I, <sup>132</sup>Te, <sup>134</sup>Cs and <sup>137</sup>Cs. Among these radioisotopes, <sup>131</sup>I, <sup>132</sup>I and <sup>132</sup>Te disappeared soon due to their short half lives. The half life of <sup>134</sup>Cs, and of <sup>137</sup>Cs are 2 years and 30 years, respectively. The contaminated soil of 5 cm in thickness from the ground surface has been removed and its total volume was estimated about  $2 \times 10^7$  m<sup>3</sup>. It is a burden to Fukushima residents to reserve a very large place to secure such a huge quantity of contaminated soil. Therefore, it is needed to reduce the volume of contaminated soil. It was known that Radioactive cesium atoms have been adsorbed with the silt particles in the soil. We developed a methods by which the decontaminated soil volume / the contaminated soil volume was ~ 0.9, namely, the volume reduction rate was ~ 0.1 [1]. By classifying soil particles, we found that the specific activity of silt particle depended on diameter [2] and proposed that, in the case of the silt particles with the diameter of less than 10~30 mm, over the husks [3], respectively.

The use of PIXE-CT is proposed to investigate the distribution of Cs in the silt particle. In consideration of decrease of X-ray production cross sections and self-absortion of X-ray in the silt particle in the case of 3 MeV proton bombardment, PIXE-CT can be applied to the silt particles with the diameter of less than about 10 mm. We extracted silt particles from the muddy water obtained by washing the soil and then we sprayed the solution of cesium carbonate ( $Cs_2CO_3$ ). We used the micro-beam formation system of Tohoku university to PIXE-CT and obtained 2D-projection data of 0° ~ 348° at intervals of 18°. On the basis of the filtered backprojection method, we reconstructed cross-sectional images reflecting the distribution of Cs in the silt particle. This supported the previous result of the classification method of soil particles. To investigate the interior structure of the silt particles with the diameter of larger than 10 mm, we apply an X-ray CT using a micron X-ray source produced by micro-ion-beam bombardment [4]. The existence of Cs is expected to be emphasized in the difference image between a V-K-X-ray (4.94 keV) image and a Cr-K-X-ray (5.41 keV) image owing to the strong absorption of Cs-L<sub>3</sub>-absorption edge (5.01keV).

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### O-17: Application of IBA to taphonomy and paleontology

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Recent discoveries of fossil feathers preserved with some theropod skeletons provided new insights on the origin of the plumage in non-avian dinosaurs. In this study, we performed geochemical analysis on fossil feathers in order to characterize their composition and, therefore, their preservation state. The fossil soft tissues are preserved around the skeleton of a new specimen of the Jurassic paravian theropod Anchiornis huxleyi. Feather chemistry has been revealed by combining IBA techniques: Proton-Induced X-ray Emission (PIXE), Nuclear Reaction Analysis (NRA) and Rutherford Backscattering (RBS) analysis. By combining NRA, RBS and PIXE induced by deuterons, we demonstrate that it is possible to measure quantitatively light and heavy elements in the fossil feathers as well as in the sediments around the fossil. PIXE and RBS were acquired simultaneously with a proton beam of 3.0 MeV, allowing identifying the elemental composition of the plumage and the surrounding matrix. They both contain elements as C, O, Cu, Cr, Fe and Ca. The melanin pigment found in all extant feathers contains chelated Cu. The presence of such element in the spectrum of the fossil could indicate melanin remains preserved in the Anchiornis plumage. RBS permitted to observe enriched carbon and oxygen layers on the fossil plumage and thus, to differentiate the fossil tissues from the surrounding sediment. The combined analysis showed we can quickly determine the elemental enrichment and depletion, and the structure of the fossil feathers using techniques that are new in the field of paleontology.

### O-18: Evaluation and mapping of PM2.5 atmospheric aerosols in Arasia region using PIXE and gravimetric measurements

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Air pollution is a worldwide problem that can affect the environment and human health. However, in Arasia region (Arab countries in Asia) there is lack of enough data and study dealing with this issue. It is obvious from the few studies conducted in the Eastern Mediterranean region that Particulate Matter (PM) levels in air are much higher than in other regions, even when compared to the Western Mediterranean. Besides, atmospheric aerosols of PM10 and PM2.5 (aerosol particles with aerodynamic diameter less than 10 and 2.5 µm respectively) are trans-boundary and can effectively contribute to air pollution in a certain localized areas. Thus, the current work is a part of a study conducted among several Arab countries in west Asia, under an IAEA regional technical cooperation project for Arasia region. It consists of a simultaneous sampling of PM10 and PM2.5 using an ISAP®1050e sampler having a combined inlet. Few months ago, the first regional campaign, within the IAEA-RAS/0/072 project, was launched simultaneously in Lebanon, Irag, Jordan, Syria and United Arab emirates, using a unified sampling and analysis protocol. For the moment, it is based on 24-hour sampling, every six day and is focused on the sampling of PM2.5 from a single location in each country, supposed to represent the urban background of air quality. The different PM2.5 samples collected on Teflon filters were analyzed by PIXE technique at the external beam facility of LABEC laboratory of INFN in Florence (Italy) using a 3 MeV proton beam and the dedicated array of 3 Silicon Drift Detectors in order to determine their elemental composition. The following elements were detected simultaneously: Na, Mg, Al, Si, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn and Pb, beside some other occasional elements found in particular samples. Furthermore, a gravimetric measurement was done to determine the total aerosol mass. This project is unique as it addresses a gap in the available data and information about regional background pollutants. It aims to produce for the first time a data base of PM elemental concentrations in the region that will help in future studies on air quality and consequent pollution. In this work, it will be shown the first results of this opportune regional study.

### O-19: Increased Accuracy and Sensitivity in Hydrogen Analysis

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Proton-proton (pp) scattering has proven to be the most sensitive ion beam method for hydrogen depth profiling [1]. For microscopic imaging in 3 dimensions it is in fact the only usable method because of its low radiation damage potential and its compatibility of using a large solid angle detector arrangement at a proton microprobe [2].

The high sensitivity is achieved by a narrow coincidence filter set on the detection of both protons from a pp-scattering event. In order to reduce background from accidental coincidences additional constraints are required, e.g. filtering the 90° scattering angle of both particles to each other, that is well defined due to equal masses of the scattered particles, using an angular resolving strip detector. The efficiency in the detection of pp-events, however, is reduced for tight filters due to multiple (small angle) scattering of the scattered protons for long path length in particular when analyzing hydrogen in samples containing high Z elements.

Hence, the detection efficiency is a function of depth and the filter tightness. Additionally, detector effects due to its granularity and calibration uncertainties have to be carefully controlled.

Accuracy in quantification strongly depends on knowing all of these effects. Thus, we introduce CORTEO [3] Monte Carlo simulations to calibrate the efficiency in dependence of matrix composition, sample thickness, depth of origin of a pp-scattering event and detector granularity. The agreement of simulations with measured depth profiles is demonstrated using multilayered sandwich targets.

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- [3] F. Schiettekatte, Nucl. Instr. and Meth. B266 (2008), pp. 1880–1885

### O-20: Developments and applications of nuclear microprobes in China

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An aperture-collimated nuclear microprobe was set up in the early 1980's at Fudan University. Since then, techniques and applications of nuclear microprobes in China have been developed and progressed for more than thirty years. There are seven accelerator-based nuclear microprobe facilities available in China now. They are able to provide micro beams with ion energies from 1 MeV to 1 GeV. These microprobes have been utilized to obtain spatial information or radiation effect in solids and biological samples. In air and in vacuum microprobes were constructed. Techniques of ion beam analysis methods, single ion hit were also developed for these applications. This paper describes their technological performance. It also presents the latest progress of applications on ICF (Inertial confinement fusion) target, CdZnTe detector, information safety and so on.

### O-21: Advanced pulse processing for Time-of-Flight ERD

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Time-of-flight ERD instrumentation is often based on traditional peak-sensing ADCs and time to amplitude (TAC) converters. An advanced timestamping data acquisition setup for these ADCs allows the monitoring of beam-induced damage and setting the coincidence criteria post-acquisition [1]. The use of gas ionization chambers (GIC) as an energy detector in ToF-ERD has become popular recently. To make the most of the detector, it makes sense to capture not just the pulse height from the anode of the GIC, but full traces of anode, Frisch grid and cathode pulses. The timing and shapes of these pulses can be used to achieve position sensitivity [2] and

even particle species discrimination. A complete data acquisition and pulse processing setup for ToF-ERD in Jyväskylä [3] consists of a fast amplifier, an analog discriminator and a CAEN N6751 digitizer for the time-of-flight and three Amptek CoolFET preamplifiers and a CAEN N6724 digitizer for the GIC. A desktop computer running Linux is connected to the digitizers over a CONET optical link. The computer runs in-house developed acquisition software with a user-friendly graphical user interface built using Qt 5. The software can process the raw events to list data during the acquisition with customizable software algorithms. In this presentation the used algorithms and software design are described. The time-of-flight digitization is shown to have a <200 ps resolution, which is comparable to a well-tuned CFD+TDC setup and improved hydrogen detection efficiency. By capturing full traces of all signals various effects affecting the detection efficiency, resolution and background can be studied in detail. The digitization of GIC signals is shown to reduce unwanted background by making possible tighter coincidence criteria and rejection of certain events by pulse shape.

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 J. Julin, M. Laitinen, T. Sajavaara, Nucl. Instrum. Meth. B 332 (2014) 271
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### O-22: Performance Tests of a Scalable Multi-Detector Digital Spectrometer with Eight Channels

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Digital pulse processing in ion beam analysis is still not very common. Commercially available systems are not yet versatile enough to suite the needs for flexible and multi-modal ion beam analytical setups. Therefore, lab-specific solutions are under development with more or less success. If ion beam scanning is required, solutions become even more complicated since synchronization between the data streams from the detector channels and the scan control is necessary. We present the design and the results of performance tests of our scalable multi-detector digital spectrometer. It is currently configured with two digitizers (ADQ412), each with four-channel, 12 bit, and 1 GHz sampling frequency. The scan control module is a two-channel 16-bit, 50-MHz sampling frequency digital to analog converter. The digitizer and scan modules are synchronized via the chassis backplane PCI bus. Digital pulse processing of the detector signals is realized onboard the digitizer cards using a field programmable gate array. The system is designed to handle data rates of up to 1 MHz per channel.

### O-23: Recent progress on the new MeV SIMS setup at ETH Zurich

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An MeV SIMS set-up with a capillary microbeam is being installed at the Laboratory of Ion Beam Physics at ETH Zurich. By using a microcapillary collimator, primary beam spots of a few microns in diameter can be formed almost independent of mass and energy of the projectiles. The high energy beam transport is exclusively achieved by electrostatic ion optical elements. This allows to use high energy heavy ions as well as molecular ions (e.g. C<sub>60</sub>) in low charge states. The SIMS chamber is equipped with a piezo raster stage for imaging. Five samples at a time can be introduced and stored in the UHV chamber via a vacuum load lock. Secondary ions are detected by a linear Time-of-Flight (ToF) spectrometer with a 0.5 m flight path and a microchannel plate detector. ToF start signals can be obtained from incident MeV particles or by beam pulsing. For thin samples, a gas ionization chamber allows to detect transmitted ions to perform STIM measurements. Additional imaging and analysis of the sample will be possible with a secondary electron device and a silicon drift detector which can be mounted to produce PIXE maps. Details on the set-up and first results will be presented.

### O-24: Structure in the velocity dependence of heavy-ion energy-loss straggling

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Recent theoretical and experimental studies [1,2,3,4] indicate that the energy dependence of heavy-ion straggling has a multiple-peak structure. In the present work we study the systematics of this structure as a function of the atomic numbers  $Z_1$  and  $Z_2$  of ion and target. It is well known that linear straggling, bunching and packing produce separate, although not necessarily resolvable peaks in the energy dependence of straggling. More surprising is the observation [3] that charge-exchange straggling alone can produce a multiple-peak structure, although only two peaks have hitherto been unambiguously identified in calculations. Experimental studies of straggling are typically affected by insufficient knowledge of foil uniformity. To circumvent this source of error we focus on gas targets. Until now we have employed Si and Kr ions and He, N, Ne, Ar and Kr as target gases in the energy range of 0.5 - 12

MeV/u. Most measurements were performed at the K130 cyclotron at the Jyväskylä accelerator laboratory; for Si independent measurements were made at 0.5 - 2 MeV/u using the 6 MV EN tandem accelerator at ETH Zurich. While our measurements [2,4] confirm the existence of more than one peak, we have not yet identified an ion-target combination where at least two resolvable peaks have a predicted position within the energy range of any singe accelerator used. We report new calculations with the aim to confirm trends identified earlier for Kr and Si ions [3]:

1. For charge-exchange straggling, peak height and speed increase with increasing Z<sub>1</sub>,

2. With increasing Z<sub>2</sub>, peak velocity increases but peak height decreases with increasing Z<sub>2</sub>.

3. The significance of bunching relative to charge exchange increases with increasing  $Z_2$ .

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[4] C. Vockenhuber, M. Thöni, J. Jensen, K. Arstila, J. Julin, H. Kettunen, M. Laitinen, M. Rossi, T. Sajavaara, H. J. Whitlow, O. Osmani, A. Schinner and P. Sigmund (2014), presented at ICACS 26 in Debrecen (unpublished).

# O-25: Direct imaging of straggled ions for top-down fabrication of Si:P qubits using atom probe tomography

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Single <sup>31</sup>P ions can be deterministically implanted ("top-down") into isotopically pure <sup>28</sup>Si substrates to build devices in which information can be encoded in the quantum states of the <sup>31</sup>P donor electron spin or nuclear spin. In the case of the nuclear spin, coherent states can be sustained with remarkably long T<sub>2</sub> times in excess of 30 s. Exploiting these results to build a large-scale device requires careful architecture design consistent with the precision limitations of the deterministic implantation method. Donor atom positioning no deeper than 20 nm below the gate oxide and ion straggling constrains the implantation energy to below 14 keV and preferably below 10 keV. Models for the straggling process, for example the Stopping and Range of Ions in Matter (SRIM) and crystal-Transport of Ions In Matter (TRIM), allow for constraints imposed by various effects to be evaluated. However, we have employed Atom Probe Tomography (APT), to facilitate the measurement of low-fluence 14 keV <sup>31</sup>P ions implanted into Si with atomic scale accuracy. APT is based on the highly controlled fieldevaporation of individual ions from the surface of a very-sharp needle-shaped specimen. It provides a 3D reconstruction of the distribution of implanted ions, typically within a volume of 80 x 80 x 200 nm<sup>3</sup>, which can be imaged to a spatial precision better than 1 nm and a chemical sensitivity of less than 0.01 at.%. We observe that the experimental depth profile has a FWHM of 20 nm with a depth consistent with the SRIM simulations. Further work will refine these measurements and allow us to apply these experimental constraints to the design of a large scale device fabricated with the top-down deterministic ion implantation method.

# O-26: Stopping power study of H and He in Hydropaxyapatite films: A study with implications in ion-beam therapy

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Ion-beam therapy is a promising technique to treat deep-seated tumors. However for an accurate treatment planning, the energy deposition by the ions must be well known both in soft and hard tissues. Although the energy deposition in soft tissues is well determinate the same is not true concerning hard tissues (i.e., bones). In particular more knowledge is needed for the main constituent of the human bone, calcium hydroxyapatite (HAp) which constitutes 58% of its mass composition. Moreover the HAp is considered as a biomaterial because it forms a strong bond with the human bone. In the present work, the energy loss of H and He in those films was determined experimentally for the first time. The experiments were performed using the Rutherford backscattering technique in an energy range of 250-2000 keV for H and 300-5000 keV for He. The corresponding theoretical calculations derived from the dielectric formalism [1] and a proper description of the HAp electronic excitation spectrum shows a nice agreement with the experimental data. Even though the experimental results were obtained at rather low energies as compared with the ones used for ion-therapy, they validate the mean excitation energy I obtained theoretically. This parameter is the fundamental quantity to accurately asses the energy deposition and depth dose curves of ion beams at clinically relevant high energies. The effect of the mean excitation energy choice on the depth-dose profile is shown by the SEICS code [2], which is based on molecular dynamics and Monte Carlo techniques. This program shows that there is a significant difference between the present results and the theoretical ones obtained by using the Bragg rule. Finally in this presentation we show how the fundamental physics inspired studies, such as the present one, have implications in the field of ion-beam cancer therapy where an accurate knowledge of the energy by swift ions is required by a proper treatment planning.

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### O-27: Neutralization and wake effects on the Coulomb explosion depth profiling

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The Coulomb explosion of small cluster beams can be used to measure the dwell time of fragments traversing amorphous films [1]. Therefore, the thickness of thin films can be obtained with the so-called Coulomb depth profiling technique using relatively high cluster energies where the fragments are fully ionized after breakup. Here we demonstrate the applicability of Coulomb depth profiling technique at lower cluster energies where neutralization and wake effects come into play. To that end, we investigated 50-200 keV/u H<sub>2</sub><sup>+</sup> molecular ions impinging on a 10 nm TiO<sub>2</sub> film and measured the energy of the backscattered H<sup>+</sup> fragments with high-energy resolution. The effect of the neutralization of the H<sup>+</sup> fragments along the incoming trajectory before the backscattering collision is clearly observed at lower energies through the decrease of the energy broadening due to the Coulomb explosion. The reduced values of the Coulomb explosion combined with full Monte-Carlo simulations provide compatible results with those obtained at higher cluster energies where neutralization is less important. The results are corroborated by electron microscopy measurements.

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# O-28: Energy loss straggling of Cu, Si, O and C heavy ions through Si<sub>3</sub>N<sub>4</sub> thin foils over a range of energies 0.1–0.6 MeV/n by Time-of-Flight spectrometry

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The energy-loss straggling of <sup>63</sup>Cu, <sup>28</sup>Si, <sup>16</sup>O and <sup>12</sup>C partially stripped heavy ions crossing silicon nitride foils has been determined over a continuous range of energies 0.1–0.6 MeV/n, by using a method based on the Heavy Ion – Elastic Recoil Detection Analysis (HI-ERDA) technique using a Time of Flight (ToF) spectrometer. The obtained energy loss straggling values corrected for non-statistical straggling and the thickness variation using the Besenbacher's method have been analyzed and compared with the corresponding computed values including different contributions: electronic, nuclear and electronic binding straggling. For computed electronic straggling we have used alternatively the widely used formulations such as, Bohr, Bethe-

Livingston and Yang formulas. The aim of such comparison is to check the reliability and accuracy of the existing energy loss straggling formulations, in the light of the present experimental results. The experimental results of energy loss straggling of all ions are found to be greater than those predicted by the theories. These differences can be attributed to the charge exchange straggling. This effect has to be taken into account in order to explain the obtained results.

### O-29: MultiSIMNRA: a computational tool for self-consistent ion beam analysis using SIMNRA

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Computational tools always played an important role in the data analysis of IBA techniques during its historical development [1]. Many advances occurred in the comprehension of physical processes and measurement system effects, always followed by advances in computational modeling [2]. A recent and well succeeded case is SIMNRA [3], which is a software widely adopted by the IBA community. Its strength lies on trusted modeling of the physical processes involved in the scattering calculation and measurements system effects. It was recently reported that upgrades can be expected for its new version concerning the skewness of energy spread distributions, improved handling of reaction cross-sections with structure, generalized layer roughness, and sample porosity [4]. Another important software is DataFurnance, that uses advanced algorithms to minimize an objective function to fit energy spectra in scattering analysis [5]. The DataFurnance software is able to handle simultaneously and self-consistently different IBA spectra obtained by multiple techniques applied in the same sample [6,7]. This approach ensures the reliable and unequivocal modeling of the sample [7], but equally important, the self-consistent analysis inherits the accuracy of the most accurate component of the analysis [8,9]. SIMNRA still remains the most cited software according to reference [2].

Taking advantage of the recognized reliability and quality of the simulations provided by SIMNRA, we developed a multi-process program for a self-consistent analysis based on SIMNRA calculations. MultiSIMNRA also uses computational algorithms to minimize an objective function running multiple instances of SIMNRA. With four different optimization algorithms available, the code can handle sample and setup parameters (including correlations and constraints), to find the set of parameters that best fits simultaneously all experimental data. MultiSIMNRA has been tested in several situations, ranging from the analysis of simple multi-element thin films, up to very complicated multi-layered samples, that included 35 independent spectra being fitted simultaneously. The extreme case of 120 spectra generated by simulations were fitted as test, showing that MultiSIMNRA can handle a massive number of spectra simultaneously and self-consistently.

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## O-30: ERYAProfiling: a code for depth profiling of light elements in inhomogeneous samples by PIGE

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ERYA (Emitted Radiation Yield Analysis) code was developed in order to implement a PIGE standard free method for thick samples [1]. To achieve this, ERYA contains a database with several nuclear reaction excitation functions which are used to calculate the corresponding gamma-ray yields. However, ERYA code is only able to analyze samples with homogeneous concentrations across the depth. Thus emerged the need to develop a new code.

This work presents the ERYAProfling code, which was developed for quantitative analysis/depth profiling of light elements by a standard-free PIGE technique in samples with inhomogeneous concentrations across the depth.

One of the main characteristic of this code is the calculation of the beam's energy straggling in order to determine the beam's energy distribution across the sample. The beam's energy straggling is implemented based on theoretical models. For relatively large depth the beam's energy straggling is described by a Gaussian distribution. For relatively low depth the beam's energy straggling is described by Landau or Vavilov's distributions. For the calculation of the beam's energy distribution, the beam's natural resolution and the Doppler's thermal broadening are also accounted for.

To calculate the gamma-ray yield from each depth the code uses the corresponding excitation function (which is stored in a user-editable database) and the beam's energy distribution for that depth. The inclusion of the excitation function is a novelty feature of this code when comparing with the existing codes for PIGE depth profiling, which instead only use narrow resonances of the excitation function. This feature allows a simulation of a more realistic profile for the cases where more than one resonance significantly contributes to the gamma-ray yield.

Experimental results have been obtained from known samples and compared with ERYAProfling calculations, in order to validate its performance.

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### O-31: Spectrum simulation of rough and nanostructured targets from their 2D and 3D image by Monte Carlo methods

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Corteo is a program that implements Monte Carlo (MC) method to simulate ion beam analysis (IBA) spectra of several techniques by following the ions trajectory until a sufficiently large fraction of them reach the detector to generate a spectrum. Hence, it fully accounts for effects such as multiple scattering (MS). Here, a version of Corteo is presented where the target can be a 2D or 3D image. This image can be derived from micrographs where the different compounds are identified, therefore brining extra information into the solution of an IBA spectrum, and potentially significantly constraining the solution. The image intrinsically includes many details such as the actual surface or interfacial roughness, or actual nanostructures shape and distribution. This can for example lead to the unambiguous identification of structures stoichiometry in a layer, or at least to better constraints on their composition. Because MC computes in details the trajectory of the ions, it simulates accurately many of its aspects such as ions coming back into the target after leaving it (re-entry), as well as going through a variety of nanostructures shapes and orientations. We show how, for example, as the ions angle of incidence becomes shallower than the inclination distribution of a rough surface, this process tends to make the effective roughness smaller in a comparable 1D simulation (i.e. narrower thickness distribution in a comparable slab simulation). Also, in ordered nanostructures, target re-entry can lead to replications of a peak in a spectrum. Other improvements to Corteo include obtaining energy-depth maps that can be used to simulate the spectrum of IBA techniques based on photon emission such as PIXE and gamma-emitting NRA. Finally, we show how, in thin layers made of heavy elements on light substrates, the broad background due to wide-angle MS in RBS is mainly due to ions flying parallel to the surface inside the heavy layer and scattering a second time. This could be the basis of an analytical model to account for this particular case of double wide-angle scattering.

# O-32: VIBA-Lab 4.0: Computer program for Simulation and Quantitative analysis of PIXE, XRF and RBS Spectra

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VIBA-Lab computer program was originally developed as an interactive software package for simulation of Particle Induced X-Ray Emission (PIXE) and Backscattering Spectra (BS) for a wide range of experimental parameters and layered sample composition. The program is now upgraded with a number of new features. To enable program to run on all major operating

systems (Windows, Mac OS, Linux) new, JavaFX based Graphic User interface (GUI) is implemented. Physics routines, originally written in Fortran 77 and C++ are translated into Java programing language. In terms of functionality, several new features are added:

1. Besides simulating PIXE and BS spectra, user can now simulate X-Ray Fluorescence (XRF) spectra as well;

2. Spectrum fitting routine is added so user can perform quantitative analysis of samples excited with multiple, PIXE and XRF excitation sources;

3. Various auxiliary programs are available to calculate and graphically display functions like detector efficiency, mass attenuation coefficients, various theoretical approximations of ionization cross sections for K and L shells as well as fundamental atomic parameters such as Coster-Kronig transition probabilities, fluorescence yields and emission rates from the most commonly used references.

# O-33: Combination of RBS/C and Raman techniques for the investigation of the SNEEL phenomenon in ion-irradiated materials

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Very recently, a new phenomenon called SNEEL (Synergy between Nuclear and Electronic Energy Losses) was discovered to take place in some solids irradiated with a dual low- and highenergy ion beam [1,2]. This process induces a strong decrease of the damage induced by ballistic collisions generated by slow ions (S<sub>n</sub>) via self-healing mechanisms due to ionization from the electronic energy loss of swift ions (Se). This paper presents studies performed in order to make strides in the understanding of the SNEEL phenomenon occurring in SiC. The combination of RBS/C and Raman techniques were used to characterize the damage accumulated during the various irradiation steps. Results show that dual-beam irradiation of SiC induces a dramatic change in the final sample microstructure with a substantial decrease of radiation damage as compared to single-beam irradiation. Actually, a defective layer containing dislocations is formed upon dual-beam irradiation (S<sub>n</sub>&S<sub>e</sub>), whereas single low-energy irradiation (S<sub>n</sub> alone) or even sequential (S<sub>n</sub>+S<sub>e</sub>) irradiations lead to full amorphization. The results presented in this paper present a crucial interest for both fundamental studies and industrial applications. Concerning the operating cycle of future nuclear reactors, expected synergetic S<sub>n</sub>/S<sub>e</sub> effects may lead to a strong reduction of the damage production allowing the preservation of the physical integrity of materials submitted to severe irradiations.

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### O-34: Graphene to Graphane Transition Induced by Highly Charged Ion Irradiation

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Since its isolation in 2004 by Geim and Novoselov, graphene has attraced a lot of attention from researchers of various fields. Although its intrinsic physical properties are extraordinary when it comes to charge carrier mobility, thermal conductivity, mechanical strength, etc., some applications will require modifications of pristine graphene. One possibility consists of introducing defects into the graphene lattice to e.g. create nanopores in graphene for ultrafiltration filters. Furthermore, the presence of defects affects the adsorbtion of adsorbates on graphene and can be used to create graphene derivatives like fluorographene, graphene oxide or hydrogenated graphene (al so called graphane).

In this contribution it will be shown, that highly charged ions (HCI, ions with a relatively low kinetic energy and a high potential energy due to their charge state) provide a powerful tool to introduce defects into a graphene sheet [1,2]. These projectiles are interacting with matter primarily via electronic excitation exclusively in the first few nanometers at the surface. Using atomic force microscopy and Raman spectroscopy, defects due to the HCI irradiation can be observed in graphene. Interestingly, the defects are not of topographic nature but can only be observed in the friction image. Additional sum frequency generation measurements indicate that upon ion impact, graphene is locally hydrogenated. Finally in situ transport measurements of an irradiated graphene field effect device are presented which allows us to correlate the effect of the local hydrogenation with the charge carrier mobility and doping of the modified graphene device.

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## O-35: A new protocol to evaluate the charge collection efficiency degradation in semiconductor devices induced by MeV ion beam irradiation

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Focussed MeV ion beams are ideal tools to investigate the effects of radiation induced displacement damage on the electronic performances of semiconductor device. Their main advantage is that ions can be used as projectiles to induce damage in the bulk material and as ionization probes to study the effects of radiation-induced defects on the charge collection efficiency. Actually, as a damaging tool, beside the broad energy range and ion type, i.e. ion

range, they offer the advantage of a) a fine control of the ion beam current, b) an accurate determination of the ion fluence and c) area selective damage with high lateral resolution down to the micrometer scale. In parallel, the Ion Beam Induced Charge (IBIC) technique provides a powerful experimental method to probe the electronic properties of semiconductor devices. By using suitable computational tools to predict ionization and defect production profiles as well as a robust model for the induced charge mechanism, the degradation of charge collection efficiency (CCE) as function of radiation displacement damage can be properly interpreted and predicted. In order to exploit these advantages, an experimental protocol has been developed with the aim to establish a methodology for identifying key parameters that will subsequently allow the prediction of CCE degradation in the low damage (i.e. no-interaction between created defects) regime. Initially, the protocol was applied to Si pin diodes irradiated by focused MeV light ions. Several experiments were performed in different laboratories using various ionenergy combinations both to induce defects and to probe their effects on the electronic performances of the devices. The experimental data were interpreted by a comprehensive theoretical model, based on the theory of charge induction in semiconductor devices, using as input parameters, the ionization and vacancy profiles calculated by the SRIM and MARLOWE codes. The model is capable to properly simulate the degradation of the charge collection efficiency in the low damage regime and suitable to extract key parameters, such as the carrier capture cross section, which are in good agreement with data available in the literature or evaluated through other complementary techniques (i.e. DLTS). The potential of the experimental and theoretical methodology is finally discussed through several examples to compare the intrinsic radiation hardness of different materials and devices.

This work was carried out within the International Atomic Energy Agency (IAEA) coordinated research project (CRP No. F11016) on "Utilization of Ion Accelerators for Studying and Modelling Ion Induced Radiation Defects in Semiconductors and Insulators".

## O-36: A modified drift-diffusion model for evaluating the carrier lifetimes in radiationdamaged semiconductor detectors

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The transport properties of a series of n and p-type Si diodes have been studied by the Ion Beam Induced Charge (IBIC) technique using a 4 MeV proton microbeam. The samples were irradiated with 17 MeV protons and fluences ranging from  $1x10^{12}$  to  $1x10^{13}$  p/cm<sup>2</sup> in order to produce a uniform profile of defects with depth. The analysis of the charge collection efficiency (CCE) as a function of the reverse bias voltage has been carried out using a modified drift-diffusion (D-D) model which takes into account the possibility of carrier recombination not only in the neutral substrate, as the simple D-D model assumes, but also within the depletion region.

This new approach for calculating the CCE is fundamental when the drift length of carriers cannot be considered much greater that the thickness of the detector due to the ion induced damage. From our simulations, we have obtained the values of the carrier lifetimes for the pristine and irradiated diodes, which have allowed us to calculate the effective trapping cross sections using the one dimension Shockley-Read-Hall model. The results of our calculations have been compared to the data obtained using a recently developed Monte Carlo code for the simulation of IBIC analysis, based on the probabilistic interpretation of the excess carrier continuity equations.

## O-37: Determination of recombination radius in Si for Binary Collision Approximation (BCA) codes

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Displacement damage caused by ions or neutrons in microelectronic devices can have significant effect on the performance of these devices. Therefore, it is important to predict not only the displacement damage profile, but also its magnitude precisely. Analytical methods and BCA codes working with amorphous targets use the concept of displacement energy, the energy that a lattice atom has to receive to create a permanent replacement. It was found that this "displacement energy" is direction dependent; it can range from 12 to 32 eV in silicon. Obviously, this model fails in BCA codes that work with crystalline targets, such as Marlowe. Marlowe does not use displacement energy; instead, it uses lattice binding energy only and then pairs the interstitial atoms with vacancies. Then based on the configuration of the Frenkel pairs it classifies them as close, near, and distant pairs, and considers the distant pairs the permanent replacements. Unfortunately, this separation is an ad hoc assumption, and the results do not agree with Molecular Dynamics (MD) calculations. After irradiation, there is a prompt recombination of interstitials and vacancies if they are nearby, within a recombination radius. In order to implement this recombination radius in Marlowe, we used the comparison of MD and Marlowe calculation in a range of ion energies in single crystal silicon target. The calculations showed that a single recombination radius of ~ 7.4 Å in Marlowe for a range of ion energies gives an excellent agreement with MD.

#### O-38: Elemental signature of memory recall

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The aim of this work is to obtain elemental maps of rat brain tissues during different stages of

memory formation and recall. The experiment was performed considering four distinct situations related to different stages of Inhibitory Avoidance (IA) task according a protocol which we developed to study memory formation and maintenance in rats. Such situations correspond to: (1) control group - rats where we could found the baseline concentration of Cu, Fe, Zn, Ca, Cl, K, Mg, Na, P and S. Those Trace Elements (TE) are participating direct of memory formation and maintenance; (2) IA memory formation - rats passed through IA training session to established a new memory related to association between the electrical shock and the dangerous context in which the shock was applied; (3) IA weak memory recall - rats passed through IA training session to formed a memory and a day later, accessed the context in which the memory was formed with a single reactivation session of 15 seconds; (4) IA strong memory recall - the animals passed basically through the same experimental protocol as groups 2 and 3 with three long reactivation sessions (3 minutes each). All rats were sacrificed at the same time, under the same conditions. The tissues were immediately cryofixed in liquid propane cooled by liquid nitrogen and then freeze-dried. The samples were cross-sectioned with an ANCAP cryostat (ANCAP, São Paulo, Brazil). The cryostat chamber temperature was fixed at -15 °C and the sectioning thickness was 90 µm. Samples were mounted between two layers of Formvar film. Micro-PIXE analysis was performed using the nuclear microprobe at the Jožef Stefan Institute (Ljubljana, Slovenia). A proton beam with energy of 3 MeV and a diameter varying from 1  $\mu$ m to 1.5  $\mu$ m at ion currents ranging from 40 pA to 500 pA was used. Our results indicated that the TE concentration pattern doubles after the acquisition of new memory and its subsequently formation. The only exceptions were observed for Ca and Fe, whose baseline have the inverse ratio. All TE showed the same concentration pattern at re-exposure in the test session. A single brief reactivation causes a decrease of the TE concentration. Moreover, TE levels restored to the same level presented during memory formation when reactivation was long and happened three times. These results may suggests that: (a) TE indeed participates in memory formation and maintenance processes and it can be studied by PIXE; (b) TE related to memory formation also display a hole in the recall process and their concentrations have a positive direct relationship with the total or partial IA MT recall; (c) the phenomenon of reconsolidation can actually mean something but a simple second consolidation based on TE concentration pattern.

## O-39: Towards RBS/EBS mapping at the AGLAE facility: a new implement to apprehend Cultural Heritage materials

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The non-invasive study of Cultural Heritage objects by IBA gives precious information on their provenance, manufacturing process or conservation state, which are essential issues for AGLAE users (curators, conservators, archaeologists, geologists...) RBS/EBS is of utmost interest to

investigate layers at the surface such as restoration products, degraded or corroded surfaces, metallic leaf decorations, nanoparticles embedded in a glassy matrix...

In the frame of the New AGLAE project (grant ANR-10-EQPX-22), a multi-parameter acquisition system has been coupled to a vertical magnetic deflection of the beam and an XY stage allowing sample mapping over an area of interest of several centimeters with a pixel resolution of typically 20 to 40  $\mu$ m. EDS X-ray, gamma, and backscattered particle events are simultaneously recorded in list mode file used by our homemade software for rebuilding the matrix of any detector and/or re-bin the data with different pixel size off-line.

GUPIXWIN is conveniently used to extract quantitative data from the PIXE spectra recorded for each pixel of the maps. To obtain reliable depth profiling information we used the IBA DataFurnace, which is one of the "new generation" codes, able to handle RBS/EBS/PIXE data self-consistently.

In this work, we will show the methodology followed to establish a map of the thicknesses of the metallic leaf present in an ancient gilt-leather artifact. Used all over Europe between the 16<sup>th</sup> and the 18<sup>th</sup> century, these decorations were made by applying a silver leaf to leather, then covered with a yellow varnish giving a gold-like appearance. The composition and thickness of the silver leaf could be precious information in determining the provenance of the artwork as well as understanding the factors influencing the degradation of the silver leaf.

The first results obtained by RBS/EBS will be presented and the influence of various parameters from the set-up (determination of the charge at atmospheric pressure, solid angle, electronic gain...) and the target (surface and substrate roughness) on the thickness determination will be discussed.

## O-40: Capabilities of miniature high resolution X-ray spectrometer at the ion microprobe

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Particle induced X-ray emission (PIXE) technique is widely used as a routine tool for elemental concentration determination in samples irradiated with MeV energy protons. Although small variations of X-ray intensity ratios or energies due to chemical effects can be observed with energy dispersive detectors like Si(Li), Ge and/or SDD [1], they are usually ignored. Using wavelength dispersive spectrometers fine structure of X-ray spectra is revealed, clearly showing chemical effects and setting ground for chemical speciation applications. Recently we employed simple high energy resolution crystal X-ray spectrometer to study chemical effects on broad beam PIXE induced K $\beta$  X-ray spectra of 3d transition element compounds [2, 3, 4, 5]. Based on the experience gained with broad beams, we designed and constructed downsized wavelength dispersive X-ray (WDX) spectrometer for application on microscopic samples utilizing micrometer beam size available at our ion microprobe. Development of the spectrometer included several stages: optimization of spectrometer geometry with X-ray tracing program XTRACE [6, 7], design of dedicated vacuum chamber housing flat crystal, sample holder and charge coupled device (CCD) as X-ray detector and development of image processing procedure for transfer of X-ray images to energy dispersive spectra. In this work we will describe our

system and each construction step followed by discussion about its advantages and limitations. Effect of chemical environment on high resolution  $K_{\alpha}$  and  $K_{\beta}$  spectra will be shown for several silicon compounds and silicates irradiated with 2 MeV protons. Same compounds are used to study variations of intensity distributions in  $K_{\alpha}$  satellites induced with 20 MeV carbon ions. Spectrometer capability to distinguish sulphate from sulphide compounds based on chemical effects on  $K_{\beta}$  sulphur spectra will be presented. We will also demonstrate ability of spectrometer to separate K and M X-ray lines which are usually irresolvable with energy dispersive detectors.

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## O-41: Structural properties of SiGe nanoparticles in LPCVD $Si_3N_4$

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Semiconductor nanoparticles (NPs) are novel material systems that lie between the molecular and solid-state regimes with unique properties largely controlled by their size and shape. Due to their exceptional optical and electrical properties, semiconductor NPs have direct applications in optoelectronic and non-volatile memory devices. Of particular interest are Ge NPs embedded in a dielectric matrix due to their ability to both emit light [1] and store charge [2].

Previous studies have investigated the structural and vibrational properties of Ge NPs embedded in  $SiO_2$  [3]. Here, we discuss the structural properties of SiGe NPs synthesised by ion implantation in amorphous  $Si_3N_4$ .

Ge ions, were implanted into a 2  $\mu$ m amorphous Si<sub>3</sub>N<sub>4</sub> films were grown on Si (100) substrates. Implantations were performed at temperatures of 400 °C. The resulting peak Ge concentrations ranged from 0.3 to 12 at.%. In order to promote the growth of NPs, samples were annealed post-implantation at 700, 900 and 1100 °C for 1-10 hours under an N<sub>2</sub> ambient.

Multiple techniques were used to characterise the evolution of the structural properties of samples. The crystalline and amorphous components both as a function of concentrations, and post-implantation annealing were quantified by X-ray Absorption Spectroscopy. The formation of a Si-Ge bonding environments with different composition, for all examined concentrations and temperatures was readily evident. Complementary Raman spectroscopy measurements were also used to quantify the vibrational properties and composition of each sample and

confirmed the post-implantation annealing and atomic concentration concentration-dependent structure. Rutherford backscattering spectrometry and transmission electron microscopy measurements also show evidence of a phase transition of the host matrix, independent of atomic concentration, after post-implant annealing at 1100 °C. Crystallisation of the nitride matrix was found to facilitate the rapid diffusion of Ge atoms to the Si/Si<sub>3</sub>N<sub>4</sub> interface. Here, we discuss the role of implantation and post-implantation conditions on the growth of NPs in a Si<sub>3</sub>N<sub>4</sub> matrix and compare the differences to that previously observed for Ge in SiO<sub>2</sub>. We find that both implantation and chemical-induced defects appear to be responsible for the various structures that ensue with processing conditions. The complex mechanisms responsible for crystallisation of the matrix, including consideration of structure disorder, loss of N<sub>2</sub>, and non-stoichiometry will be discussed.

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## O-42: IBA analysis of Iron-doped Bi<sub>2</sub>Se<sub>3</sub> topological insulator

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In three-dimensional Topological Insulators (TIs), strong intrinsic spin-orbit coupling generates an insulator band inversion with spin-momentum locking leading to creation of protected Topological Surface States (TSS) due to time-reversal symmetry. These surface states are described by helical Dirac fermions with a topological order, meaning that opposing electron spin states generate opposing currents [1, 2]. The topological nature with its spin texture is extremely interesting from the perspective of using these materials in electronic technology: such as quantum computing and spintronic devices [3]. However for such applications it is necessary to be able to form a ferromagnetic TSS, therefore time-reversal symmetry has to be broken. This may be attempted through doping with a magnetic dopant.

In this work, we have studied growth by MBE on GaAs (111) substrates of epitaxial  $Bi_2Se_3$ , a topological insulator with a simple band structure, doped with Fe. Rutherford Backscattering Spectroscopy (RBS), ion channelling, and heavy ion Elastic Recoil Detection Analysis (ERDA) were applied. Nominal Fe/Bi atomic ratios were varied from 0 to 0.3, for films of 100 nm-thickness, either capped or uncapped with a thin Se or ZnSe layer to protect from oxidation.

In the undoped capped samples RBS shows stoichiometry very close to  $Bi_2Se_3$ , however iron doping introduces substantial variations of the Bi:Se ratio, and both lateral and depth composition inhomogeneity. Furthermore, in these doped samples significant light element contamination is deduced from the RBS observations, including capped samples. ERDA measurements currently underway will be presented to confirm this interpretation of the RBS spectra.

The RBS spectrum fitting was performed with the NDF DataFurnace, which allowed extraction of the layered composition, showing interface zones between the TI layers and both the capping layer and the substrate. RBS showed significant disagreement with the nominal Fe

doping and layer thicknesses. Synchrotron X-Ray Fluorescence (XRF) is therefore used as a complementary method to determine Fe areal density. The implications of these observations for the MBE growth processes of doped films, and the prospects for obtaining intrinsically ferromagnetic TSS are discussed.

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### O-43: TRBS modelling of LEIS spectra - fundamentals and applications

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Due to its monolayer sensitivity, straight-forward quantification and good detection limits, low energy ion scattering (LEIS) is routinely used in the characterization of ultra-thin films to address analytical questions like layer closure, surface composition, diffusion processes, or growth modes. For these applications, LEIS delivers essential information about the surface composition, especially in the early phases of film growth, before a full monolayer is formed. Nevertheless, the additional in-depth information contains valuable extra information about the thickness of the film and the thickness distribution or interface quality. In the past, this indepth information has mainly been exploited semi-empirically [1].

In order to get a more accurate description of the spectra and ultimately deduce a better analytical result for the samples, a physical modelling of the spectra is required. One possible approach is the application of TRBS [2] to simulate the scattering spectra. As this software was developed for much higher energies than those used in LEIS (1-10 keV), the application requires some modifications and corrections to the parameters used in the program.

Another complication arises from the fact that LEIS electrostatic energy analysers only detect scattered ions, not neutrals, while TRBS only handles the particle kinematics. Consequently, the ion fraction has to be handled independently. We found a way to measure the ion fraction and apply it to the simulated spectra from TRBS to fit the measured data. In addition to presenting the progress in our methodology [3] to model the in-depth signal of LEIS spectra in general, we will also show results from several applications. These applications include  $HfO_2/Al_2O_3$  films manufactured by atomic layer deposition (ALD) as well as other thin film system, e.g. Ru liners for advanced interconnects on Ta- and Mn-based barriers.

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## O-44: Study of diamond membrane detector aiming at highly-efficient and position-sensitive particle detection for ion beam applications

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Detection technology for high energy particles or ions is important not only in the field of highenergy physics or astrophysics but also for ion beam applications including micro-electronics, biology and also therapy. Highly precise beam control is crucial for such applications, because in interaction with material high energy heavy ions have a high linear energy transfer (LET). The irradiation effect by individual heavy ions with energy higher than MeV/u is investigated and utilized in biological applications or particle therapy. In these applications, it is also important to detect efficiently individual ions while irradiating sample in the atmosphere, with requirement to have high positional accuracy. To this demand, we proposed diamond membrane for a beam exit window as well as for a single ion detector, because of its excellent physical and electrical properties that were reached with improvement of its fabrication technology [1].

In this study, diamond membrane detectors were made to test the performance as particle detectors by measuring charge collection properties and the irradiation effect using various high energy ion beams. Microbeam systems at TIARA facility of Japan Atomic Energy Agency, Takasaki Japan and the ion accelerator facility of Ruđer Bošković Institute, Zagreb Croatia, have been used. In addition, alpha particle induced charge transient spectroscopy (APQTS) using alpha source and heavy ion induced charge transient spectroscopy (HIQTS) using the heavy ion microbeams were performed. These techniques were used to investigate the defect levels in diamond and to explore their influence to the performance of detection. This paper outlines the project which is being advanced in an international collaboration with Japanese and European scientists.

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### O-45: Concept of single ion detector for deterministic ion implantation at the nanoscale

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Nowadays, the ability to endow functionality to single atoms or molecules in materials becomes of increasing importance in nanotechnology. Atoms can be used as switches, single photon sources or quantum mechanical objects, e.g. for information processing and quantum computing. Deterministic ion implantation, i.e. the implantation of a counted number of ions, with a lateral accuracy of a few nanometers is one approach to achieve this goal. However, counting low-keV single ions with high detection efficiency is difficult - techniques used so far include secondary electron detection [1] or ion beam induced charge [2] caused by the ion impact. Ion traps have been used as single ion sources as well [3]. All these techniques have their individual limitations, e.g. the requirement for the implanted material to act as an efficient secondary electron emitter or particle detector or the restriction to specific atoms and substantial experimental effort in case of ion traps. Our planned approach to deterministic ion implantation uses a commercial Focused Ion Beam (FIB) system equipped with a dedicated single ion source. The latter one comprises an ion source, an E x B filter and a single ion detector which allows to detect the ion prior to injection into the FIB system, making the ion detection independent from the properties of the implanted material. We discuss the concept of a single ion detector which preserves the very low emittance of the ion source required for ion injection into the FIB.

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#### O-46: Technology steps to fabricate a thin Silicon Carbide membrane based particle detector

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Thin ion transmission detectors are of interest in several application areas that span over a wide range, for example as a part of a telescope detector for mass and energy identification but also as a pre-cell detector in a microbeam system for studies of biological effects from single ion hits on individual living cells. Such single ion thin detectors were previously fabricated by

etching deep cavities in silicon that leaves a membrane thickness down to 5  $\mu$ m [1]. The membranes are the active part of the detector where passing MeV ions deposit a small part of their kinetic energy. However, silicon detectors show noise and single protons are not easy to measure. In addition, beam damage may limit the detector lifetime. We propose a structure of graphene on silicon carbide (SiC) as a concept for a transmission proton detector with low noise. Generally, noise is related to the leakage current, which for 4H-SiC is several decades lower than for silicon due to the difference in bandgap (3.3 vs 1.1 eV). In our proposed structure, we will use graphene as a contact material that collects carriers generated by protons at the inner cavity of the membrane and graphene covers the inner cavity all the way to the backside of the SiC substrate. This can be used to evaluate the number of protons by a change in electrical conductivity of the graphene. A great experimental advantage in many scenarios is the physical strength of SiC which allows it to be used as a combined vacuum window and detector. The hardness of SiC is, however, a challenge in the fabrication process to create a thin membrane inside of a deep etched cavity. We have used ICP technique to etch circular cavities with depths down to 370  $\mu$ m in prototype samples. At the next step graphene is created by a special high temperature process converting the outermost layers of SiC into graphene (a layer of carbon atoms only one or a few atomic layers thick with extremely high electric conductivity). The smoothness of the etched membrane is critical for the graphene forming process, and in turn depends on the etching procedure, cavity depth and the quality of the SiC material. Evaluation of the membrane quality was done through surface profiler, SEM, AFM as well as by energy loss measurements using the focused micro-beam at Lund Ion Beam Analysis Facility. The graphene fabrication is challenged by that the graphene formation differs between the on-axis surfaces of substrate backside, the wall of cavity, and the bottom of cavity. This paper describes the technological steps in selection of SiC material, the etching process, graphene formation and evaluation of the prototype devices.

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## O-47: Submicron molecular imaging of cells by combined use of MeV SIMS and STIM

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In order to understand biochemistry processes governed in the cells, molecular composition at the submicron level has to be determined. For chemical imaging, different analytical imaging techniques can be used, and one among them is TOF-SIMS (Time-of-Flight Secondary Ion Mass Spectrometry) with MeV ions. Heavy ion beams in the MeV energy range can eject large molecular fragments (~1000 amu) with a yield that is several orders of magnitude larger than if keV ions are used for excitation. Thus, due to high sensitivity of MeV TOF-SIMS, measured biological samples and other materials remain intact.

For molecular imaging we installed in 2012 linear TOF SIMS spectrometer at the Heavy Ion Microbeam Facility in Zagreb [1]. TOF measurement is normally performed using START signal from the pulsed beam, and STOP from the MCP detector positioned at the end of the TOF

telescope. With this setup high sensitive molecular imaging with lateral resolution of  $\sim$ 100 nm can be achieved.

In the new setup, presented here, trigger for the START is replaced with the timing signal from the STIM (Scanning Transmission Ion Microscopy) detector placed behind the transparent target. This allows us to perform measurements with continuous beam in the low current mode (~1000 Hz), and significantly reduce beam dimension (below 1µm) with small opening of the object slits. Due to well defined submicron beam focus, molecular imaging of the single cell on a sub-cellular level is possible. Also, besides the molecular imaging, STIM image of the cell is also recorded providing additional information about distribution of cell density. Imaging of Na<sup>+</sup>, K<sup>+</sup>, and lipids inside single HeLa cells will be demonstrated (HeLa cell culture was grown on 100 nm thin Si<sub>3</sub>N<sub>4</sub> window).

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## O-48: Lanthanide-doped nanocrystals as bio-probes for ion beam induced fluorescence imaging

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Fluorescent probes play an important role in biological labeling and targeting for applications such as bioimaging, biodetection and therapeutics. We report a fluorescent material, lanthanide ions  $Yb^{3+}/Tm^{3+}$  co-doped NaYF<sub>4</sub> nanocrystal, which can generate intense emission under the excitation of mega-electron-volt (MeV) helium ions. A systematic spectroscopic study of NaYF<sub>4</sub>:Yb/Tm nanocrystals shows that both downconversion and energy transfer upconversion contribute to the fluorescence emission. More importantly, these lanthanide-doped nanocrystals exhibit enhanced resistance to iono-bleaching rather than those conventional fluorescent probes, such as quantum dots and organic dyes, allowing for long-term scanning for super-resolution imaging purpose. As a demonstration, sufficient brightness and inconspicuous iono-bleaching of the as-fabricated 100 nm nanoparticles under 1.6 MeV helium ions' excitation makes them singly resolved inside a whole Hela cell, which is significant for biological and biomedical analysis at a single whole cell level.

## O-49: Ionoluminescence as a sensor of the defects creation and damage kinetics: application to fused silica

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(3) Laboratorio Nacional de Fusión, EURATOM/CIEMAT, CIEMAT, Avda. Complutense 40, Madrid 28040 Ion Beam-Induced Luminescence (IBIL) is a very sensitive technique for the analysis of impurities and defect centers, such as those created by irradiation. In situ luminescence during ion beam irradiations can be used to investigate the microscopic processes accompanying the generation of damage and its kinetic evolution with the irradiation fluence. In this contribution we will talk about the advantages of the IBIL technique compared to other analysis techniques. We illustrate the power of ionoluminescence by showing some results obtained in amorphous silica which is a fundamental material in fusion technology [1] for optical viewports, plasma diagnostics, and safety and control systems. Degradation of optical and structural properties of silica due to irradiation is an important issue with a considerable number of features not yet sufficiently understood. We have compared the ionoluminescence in three different types of silica containing a different amount of OH impurities. For all samples, the IBIL spectrum shows two main peaks at 460 and 650 nm which have been associated with different defects in the material: Oxygen-Deficient Centers and Non-Bridging Oxygen Hole Centers, respectively. We have observed that, at the beginning of the irradiation, the red emission is much higher in the samples with high OH-content than in the samples with low OH-content, while the blue emission exhibits an opposite behavior. One of the advantages of the IL is that we can use different ions and energies, i.e. different stopping powers for the analysis. Therefore, we have compared the IBIL produced by ions with different stopping powers (dE/dx), and we observed that the kinetic evolution of the ionoluminescence varies in function of this parameter. For low dE/dx, the yield of both peaks increases monotonically with the dose. However, when we irradiate with high dE/dx, the yield of the two main emissions first increases with the fluence and then it reaches a maximum at a certain dose, where it starts to decrease. We have also studied the dependence of the dose at which this maximum is produced with the stopping power of the incident particle and we have compared our results to those obtained by other spectroscopy techniques used in silica by Awazu et al. [2], observing a good agreement between both results. Many other interesting possibilities can be studied with IBIL: application to other materials, low-temperature irradiations, irradiations at very low and very high energy to separate the electronic and nuclear regimes, irradiating with sequential and simultaneous beams, etc. We are currently investigating many of these possibilities. We conclude that the novel results obtained by comparing the IL behavior under light and heavy ions offer a useful tool to investigate structural damage in materials.

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## O-50: RBS-Channelling analysis into the effect of thermal annealing on GeSn strained layers

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Alloying Ge with Sn improves carrier mobility in the material and can also transform the material from an indirect bandgap into a direct bandgap semiconductor. Unfortunately  $\alpha$ -Sn is only stable at temperatures < 13°C and this, coupled with the large mismatch between Sn and Ge, results in an upper limit of Sn in Ge of around 1%. It is possible to produce alloys with the higher Sn concentrations but these alloys are metastable and can relax on thermal annealing. Rutherford Backscattering Spectrometry and channelling have been employed to investigate the effect of thermal annealing on epitaxial GeSn (6% Sn) strained layers grown on Ge-buffered Si(100) wafers, with channelling along the [110] axis being used to investigate the strain residing in the layers upon thermal annealing. Annealing at temperatures below 400°C for 20 minutes had no noticeable effect on the strain in the epitaxial layers. Once the temperature was raised above 400°C however, relaxation of the layer sets in and the GeSn layer has essentially completely relaxed following a 20 minute anneal at 650°C. The results are in good agreement with similar investigations conducted using X-ray diffraction.

The advantage of the RBS/Channelling approach however, is its ability to provide compositional information as a function of depth. One is therefore able to monitor the effect of the thermal anneal on the Ge and Sn distribution throughout the layer, and also to extract information about their lattice location. The results obtained show that that when the initial relaxation sets in both the Ge and the Sn are still situated in substitutional sites, and it is only around 600°C after substantial relaxation has taken place that the Sn finally breaks free from the lattice sites and diffuses to the surface of the sample. RBS-Channelling thus provides insight into the manner in which the relaxation process takes place and with this better understanding it might be possible to design more stable strained layers.

## O-51: Mechanisms of damage formation in AlGaN alloys implanted with Ar and Eu ions

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Group-III-nitrides, in particular  $Al_xGa_{1-x}N$  alloys, are important optoelectronic materials used for UV light emitting devices and high electron mobility transistors (HEMTs). Ion implantation is an important technique for selective area doping and implant isolation [1]. However, this technique produces damage which degrades the properties of materials. In III-nitride alloys, the formation of implantation damage is still largely unexplored and not yet fully understood.

In this study, the damage formation mechanisms and radiation resistance of  $Al_xGa_{1-x}N$  alloys is investigated combining Rutherford Backscattering Spectrometry/Channelling (RBS/C) and X-ray diffraction (XRD) in order to assess the damage profiles and the elastic response of the material to radiation.  $Al_xGa_{1-x}N$  alloys covering the entire compositional range ( $0 \le x \le 1$ ) were implanted at room temperature with 200 keV Ar ions, to avoid chemical effects, and fluences ranging from  $1 \times 10^{13}$  to  $2 \times 10^{16}$  Ar/cm<sup>2</sup>.

XRD 2 theta-omega curves show that implantation leads to the incorporation of large lattice strain in the implanted layer which increases with the fluence. Above a threshold fluence, an abrupt change of the elastic properties of the crystals is observed and strain saturates in the entire implanted region. This threshold fluence is reached earlier for GaN than for AlGaN alloys. Surprisingly, RBS/C reveals higher defect levels for high fluence implantations in samples with high AlN concentrations. These results contrast reports on rare earth implantation in AlGaN and ion implantation in AlGaAs alloys which reveal a strong increase of radiation resistance with increasing AlN content [2, 3]. The effect of AlN content on dynamic annealing processes and radiation resistance of AlGaN alloys as well as the differences between Ar and Eu implantation will be discussed.

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## O-52: Isolated defects created by gas cluster ion impact and their use for templates of carbon nanotube growth

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Carbon nanotubes (CNTs) are one of an allotrope of carbon with cylindrical structure, and they show excellent mechanical and electrical properties. However, these properties may be altered depending on the chirality and diameter of CNTs. When metal catalysts are used in CVD growth of CNTs, the size of metal catalysts are one of the significant parameters that defines the structure of CNTs. Therefore, it is important to control the size of metal catalysts.

In this study, isolated defects created by gas cluster ion impacts were investigated as a template of CNTs growth. Gas cluster ions are aggregates of atoms or molecules. Upon a gas cluster ion impact, dense energy is deposited on surface layer while energy/atom of gas cluster ion is low. Thus, depending on the energy/atom, gas cluster ion creates crater-like defects on a target surface.

Si and SiO<sub>2</sub> were irradiated with Ar gas cluster ion beams (GCIBs) with acceleration voltage of 20 kV, and fluence of  $4 \times 10^{11}$  ions/cm<sup>2</sup>. Then, cobalt dots on crater defects were formed by subsequent cobalt deposition and annealing. Subsequently, CNTs were grown with a thermal CVD. From SEM and AFM images, it was shown that CNTs were grown on SiO<sub>2</sub> or Si surface irradiated with Ar-GCIB. Raman spectroscopy also showed growth of single wall CNTs on Si and SiO<sub>2</sub> with Ar-GCIB irradiation. The diameters of CNTs were varied by acceleration voltage of Ar-GCIB. These results showed that isolated defects created by GCIB can be used as templates for cobalt dots formation, which results in the control of diameter of CNTs.

#### O-53: Rutherford backscattering study of Ni(Pt/Pd)Ge formation on Ge (100)

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Ge is a top candidate to replace Si in high performance metal-oxide-semiconductor (MOS) devices due to its high carrier mobility and compatibility with current silicon processing technology. However, a suitable contact material is needed for the successful realization of devices [1]. Based on the beneficial properties of NiSi contacts on Si, NiGe (which can be easily formed via solid-state reaction of a Ni thin film with the Ge substrate) has been identified as one of the most promising contact materials to Ge: it has a low resistivity, it can be formed at relatively low temperature and persists in a wide temperature range [2,3]. This thermal stability can even be further enhanced via co-depositing Pd or Pt with the Ni [4,5]. Moreover, for device integrity it is important to identify the dominant diffusing species (DDS) during the formation of the germanides [1,3]: If Ge is the DDS, overgrowth and bridging between gate and source regions in the device will occur [1]. Therefore, we investigated the phase formation, phase stabilization and diffusing species during solid-phase reaction of ternary Ni-Pd/Pt-Ge.

The diffusion and elemental redistribution during NiGe formation in Ni/Pd-Pt/Ge(100) were monitored via RBS analysis, using a variety of interlayer thicknesses (from 3 Å to 80 Å). Additionally, the use of a very thin extra interlayer of an inert element such as W [3] allows the identification of the DDS. Finally, the stability of the germanide phases is evaluated by sheet resistivity measurements. Despite the chemical similarity of Pd and Pt, a very different behavior is observed for the two types of interlayers: while Pd is mostly found near the surface after annealing at 300°C, the Pt interlayer has not moved at this temperature. After annealing at 600°C all elements have redistributed throughout the film. However, whereas most Pt is found near the surface, regardless of interlayer thickness, the Pd is primarily found near the surface for the thicker interlayer but diffuses towards the Ge-NiGe interface for thinner interlayers. The room temperature sheet resistivity decreases with increasing annealing temperature for all samples and, except for the thinnest Pt interlayer, increases with interlayer thickness. The measured values, in the order of  $10^{-5} \Omega/sq$ , are very similar for Pd or Pt interlayers of the same thickness. In this work, the dominant diffusing species during Ni-germanide formation is determined. The elemental diffusion during the NiGe formation is found to depend on the interlayer element, i.e. Pd or Pt. At 300°C Pt is an efficient diffusion barrier while Pd has already out-diffused. At 600°C Pt is found primarily near the surface for all interlayer thicknesses while the redistribution of Pd is found to depend on the thickness of this interlayer.

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#### O-54: Structural changes in quantum dots core-shell CdSe/ZnS by thermal treatment

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Core/shell semiconductor nanocrystals have been used for some time by the technological industry in a wide range of applications, such as LEDs and biosensors. However, the structural characterization of such materials, due to their small dimensions, represents a serious challenge, specially when they are modified via thermal annealing or ion irradiation. In the present work, we used the medium energy ion scattering (MEIS) analysis technique, combined with auxiliary techniques: transmission electronic microscopy (TEM), Rutherford backscattering spectrometry (RBS) and photoluminescence (PL), in order to characterize CdSe/ZnS commercially-available core-shell nanocrystals. Through the use of the auxiliary techniques, we were able to check the best set of annealing parameters to study the stability of the QDs and the mobility of the relevant atomic species: annealing at 400 celsius for 5 minutes. A much longer and/or warmer annealing tend to lead to an almost completely deterioration of the QD; on the other hand a much shorter and/or colder annealing tend to result in negligible changes in the nanocrystal structure. MEIS is an ion-beam technique, capable to obtain depth-profiling with sub-nanometric resolution, under the right conditions. This technique has been steadily optimized to the analysis of nanostructured materials, so it is an important tool in probing the structure of quantum-dots, among other materials. We used the MEIS simulation PowerMeis software to create hypothetical energy-loss distributions for a wide range of tentative QDs structures; these simulated distributions were then compared to the experimental results in order to identify the most-probable structure of all. This result was then used as feedback and the procedure was repeated until a satisfying agreement was found between simulation and experiment. We have found the core of the quantum-dots remains a stoichiometric CdSe crystal, as in the non-thermally treated samples; however the core diameter shrinks from ~5.2 nm down to approximately 4.2 nm, in agreement to TEM and PL results. We have found an excess of cadmium, located in the shell region of the QD, resulting in a CdSe/CdZnS structure. The shell symmetry seems to depart from a spherical shape, in agreement to what one would expect as the result of a possible "melting" behaviour.

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## O-55: Deuterium behavior in ceramics for Fusion Breeder Blanket after γ-irradiation: a comparison between different characterization techniques

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The ceramic breeders of an operating fusion blanket will be exposed to high levels of neutrons and gamma radiation. In ITER, it has been estimated a damage rate of about  $<10^{-7}$  dpa/s and  $<10^{3}$  Gy/s caused by the flux of high-energy neutron and ionizing radiation, respectively. The effects of ionizing radiation on insulators are quite marked due to the excitation of electrons from the valence to the conduction band, giving rise to charge transfer effects. On the other hand the comprehension of light ions behavior in solid breeder blanket (BB) candidates is of special interest for the optimization of the Tritium Blanket Module (TBM). In this contribution, the studies of processes taking place with temperature in irradiation damaged materials are analyzed with the support of several techniques. Several experiments are performed on breeder candidate ceramics selected by Japan (Li<sub>2</sub>TiO<sub>3</sub>) and EU (Li<sub>4</sub>SiO<sub>4</sub>) for ITER TBM-testing. The aim of this experimental work is the understanding of Deuterium diffusion behavior in these materials before and after their  $\gamma$ -irradiation by a  $^{6\bar{0}}$ Co source. Deuterium is implanted in selected disc-shaped ceramics with a 70 keV beam to a total fluence of 10<sup>17</sup> at/cm<sup>2</sup>, and the depth profile as a function of thermal annealing treatments of as-received and irradiated samples are characterized by Nuclear Reaction Analysis (NRA). For comparison purposes, a different set of pellet samples are exposed to a Deuterium atmosphere at room temperature inside of pressurized (1 bar) steel capsule during 25 days. In this case, the sorption behavior of previously dehydrated and irradiated samples is observed by Thermal Induced Desorption (TID) technique during heating. Whereas the barrier effect of the surface is more evident in NRA analysis, it is possible to observe more clearly the contribution of the ionizing radiation on D<sub>2</sub> outdiffusion by TID analysis.

Secondary Ion Mass Spectrometry (SIMS) technique is used for analyzing both D-implanted and exposed to a D atmosphere samples. A SIMS HIDEN Workstation with a 6kV oxygen ion gun as the primary ion source is used for the Deuterium profile determination. The elemental analysis shows different depth profiles of D-implanted and D-sorbed samples. As expected,  $\gamma$ -irradiations influence the in-depth location of D inside the ceramic matrix, due to radiation-induced further internal D trapping and higher desorption activation energies. Finally, in an attempt to establish the nature of the defects associated to ionizing radiation acting as D trapping centers, two techniques of Positron Annihilation Spectroscopy (PAS) are used for investigating defects in the bulk of the irradiated materials: positron annihilation lifetime spectroscopy (PALS) and coincidence Doppler broadening (CDB). The creation of F<sup>+</sup> defects due to  $\gamma$ -radiation is confirmed and better structural recovery in the case of a more pure material, after thermal treatments, is found. This comparative study is especially interesting for the understanding of the processes taking place inside the material and the role of the surface in the thermal desorption of Deuterium at relevant operation conditions for the BB system here analyzed.

### O-56: Deuterium Retention Studies in Ion Beam Damaged W

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The trapping and retention of tritium fuel within neutron damaged plasma facing components (PFCs) is of primary concern to next step fusion devices [1]. Tungsten is the principal plasma facing material candidate for first wall and divertor armor due to its high melting point, low H isotope retention, and excellent resistance to sputtering. As such, the use of heavy ion damage and deuterium as a proxy for neutrons and tritium in W allow for both more timely experiments and less specialization in radioactive materials. Most studies quantify damage with displacements per atom (dpa) when it is the concentration of trapping sites, i.e. traps per atom (tpa), that is a better metric for determining retention. In this work, we report our recent studies of deuterium retention in ITER-grade polycrystalline W before and after ion irradiation damage using  $D(^{3}He,p)\alpha$  nuclear reaction analysis (NRA) [2,3]. Multiple Cu ion beam energies (0.5, 2, and 5 MeV) were used to create a relatively uniform damage profile in W up to 1 micron depth at RT, with damage levels of 0.001, 0.01, and 0.1 dpa. To isolate the effects of annealing, some of the irradiations were performed at elevated temperatures (300, 600, and 750 °C). By exposing these samples to a low flux and low temperature D plasma, the near saturation of filled trapping sites effectively allows D to be used as a marker for traps in W via NRA. Results showed that increasing the level of damage decreases D retention in the damage region as well as reducing the amount of trapped D inventory beyond the damage region, while partial annealing during the irradiation before plasma exposure reduces D retention.

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## O-57: Total IBA a quantitative tool to study plasma wall interactions

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Ion beam techniques, by their nature, are valuable tools to study the effect of energetic particles on the surface region of materials making them very attractive to study plasma wall interactions in fusion devices. During operation the plasma suffers electric disruptions and

large amounts of energy and radiation loads are deposited on wall materials. Also neutrals and high energy particles are allowed to leave the closed plasma surfaces and hit the plasma wall chamber. The interactions cause severe changes on the reactor materials imposing dedicated studies to understand all these phenomena in order to improve and validate the theoretical models allowing a robust design of fusion devices.

In this contribution we study the plasma effect on selected tiles of the new ITER like wall (ILW) configuration of JET, combining different ion beam techniques and using the WiNDF code to analyse and interpret the data. The sensitivity, depth resolution and elemental analysis characteristics of the techniques are ideal to study the erosion, deposition, mixing and fuel retention on the near surface of the plasma wall materials. The results show areas of strong erosion in the centre of the tiles of the inner wall guard limiter (IWGL) whilst re-deposition occurs at the ends. The deposition zones retain large amounts of Deuterium with values reaching up to 2 orders of magnitude higher compared to the eroded area (from  $1x10^{17}$  at/cm<sup>2</sup> to  $5x10^{19}$  at/cm<sup>2</sup>). The tiles at the bottom edges of the divertor show a narrow and thick zone of deposition along the slope containing mostly beryllium.

[1] See the Appendix of F. Romanelli et al., Proceedings of the 25th IAEA Fusion Energy Conference 2014, Saint Petersburg, Russia

### O-58: Withdrawn

### O-59: In Situ NRA, RBS, XPS and UPS Studies of Hydrogen Diffusion in TiO<sub>2</sub> Single Crystals

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The intrinsic point defects associated with oxygen vacancies and  $Ti^{3+}$  ions play a crucial role in the usage of titanium dioxide ( $TiO_2$ ) in various technological applications including catalysis and photochemistry. It is well known that the interactions between H atoms and surface oxygen in  $TiO_2$  lead to the formation of  $Ti^{3+}$  ions at elevated temperatures. However the  $Ti^{3+}$  ion formation and accumulation as a function of elevated temperatures in UHV conditions during hydrogen diffusion in  $TiO_2$  is not well understood.

In this study, we have used ion implantation method to incorporate hydrogen in single crystal  $TiO_2$  (110) samples and investigated the behavior of point defects in both pure and hydrogen implanted  $TiO_2$  as a function elevated temperatures using Rutherford backscattering spectrometry (RBS), nuclear reaction analysis (NRA), x-ray photoelectron spectroscopy (XPS) and ultra violet photoemission spectroscopy (UPS).  $TiO_2$  single crystals were implanted with 40 keV hydrogen ions at room temperature with ion fluences of  $1x10^{15}$ ,  $1x10^{15}$  and  $1x10^{17}$  atoms/cm<sup>2</sup>. Samples were isochronally annealed in vacuum for 30 minutes at each temperature up to 1100K and hydrogen and  $Ti^{3+}$  defects were quantified. Hydrogen depth

profile measurements obtained from  $1 \times 10^{17}$  atoms/cm<sup>2</sup> implanted sample reveal that hydrogen diffused towards the surface at lower temperatures and it slowly diffuses out from the samples at higher temperatures. XPS and UPS measurements from the hydrogen implanted samples show significantly higher Ti<sup>3+</sup> defects in comparison to pure TiO<sub>2</sub> at these temperatures under UHV conditions. These defects reach a maximum around 880 K in which almost all hydrogen was removed from the sample. When the implanted sample further annealed to high temperatures, the amount of Ti<sup>3+</sup> in hydrogen implanted samples started to decrease and reaches the values from the pure TiO<sub>2</sub> samples around 1100K.

### O-60: Requirements on ion source performance for ion beam analysis

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Ion beam analysis techniques are performed routinely with MeV ions of H, D, He, C, N and O. In addition, ERD may require heavy ions as well. A variety of ion sources are used in differently configured ion beam injection systems, often feeding tandem particle accelerators. Most of the IBA techniques require modest ion beam currents (several hundreds of nA), easily provided by standard accelerator equipment. However, other applications including Nanoprobes, cultural heritage studies and microbiology might require submicron probe PIXE or external beam applications, which necessitate the highest possible ion beam brightness to reduce analysis time. To accommodate such requirements HVE has developed a new Tandetron injection system with an upgraded version of its 358 Duoplasmatron, that demonstrated increased hydrogen ion beam brightness from about 2 Am<sup>-2</sup>rad<sup>-2</sup>eV<sup>-1</sup> to approx. 6 Am<sup>-2</sup>rad<sup>-2</sup>eV<sup>-1</sup>. In addition, ion beam currents for H, D, He, C, N, O were increased and attention was paid to improve ion beam uptime, mean time between failures and serviceability. In this contribution we will discuss the requirements from ion source technology applied to IBA techniques, exemplify design details and quantify the performance on the new negative ion beam injection system based on the HVEE model 358 ion source.

#### O-61: Ion beam analysis of advanced coating products and air particulate matter

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GNS Science is a leading research organisation in New Zealand that is undertaking and developing ion beam science and technology for end-user needs. We present in this overview presentation ion beam science and research using technology development at GNS Science. We have developed ion beam based devices for industry trials, designed and built low energy ion implanters as well as ion beam analysis beam lines for materials research and air pollution

science. Our science focusses on the need of New Zealand's research community, regulatory authorities and small scale industries. We will present science in environment and materials research. In the first part of the presentation, we will present ion beam analysis results of our materials research. We will present ion beam analysis research using RBS, NRA, resonant NRA in diamond-like carbon coatings (DLC) produced with direct ion deposition, a technique that has proven to produce coatings with custom-designed thickness, adjustable sp<sup>2</sup>/sp<sup>3</sup> ratio, hardness and high elasticity. We have built three different systems for creating DLC coatings with high purity, fast growth rate and even the ability to coat inner walls of pipes. The latter uses a novel ion source design which uses non rotating parts to coat the inside of pipes in 360 degree geometry simultaneously. We will discuss the role of hydrogen in DLC coatings deposited at various ion energy as measured with the <sup>15</sup>N-method. In the last part of the ion beam analysis results section, we will reveal the stoichiometric changes on nanostructured multifunctional multiferroic thin films and present IBA results that have helped to determine the depth of heavy metal impurity in the polymer material used for microfluidic device. The presentation will be concluded by an outlook into the future: what are future niches for ion beam science and technology?

### O-62: External milibeam imaging applications at LAMFI-USP

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Proton micro and nanobeams for Ion Beam Analysis have become much of the attention, with astonishing results and over 400 publications since 1980. Millibeams however, though less complex to be implemented, have not earned equivalent recognition especially if considering its analytical potential. In a proton sub-millimeter beam, the beam is kept steady and the sample is rastered by means of a precise XYZ stage. Such external millibeam has just been installed at LAMFI-USP. A ~3MeV proton beam is extracted to air through a 7 micrometer thin Al window (the Al 845 and 1014 keV gamma rays are used for charge normalization) onto a target installed on a wide range high precision (60.0000 ± 0.0005 cm) XYZ computer controlled stage. A star-like detector array with 2 X-ray detectors, a particle detector, a light spectrometer, laser for alignment and a CAM for pictures allows simultaneous measurements of the irradiated spot for several interesting applications. The combination of a sub-millimeter beam with the large range XYZ robotic stage is being used to produce elemental maps of large areas in samples like paintings, ceramics, stones, fossils, and all sort of samples. The precise positioning feature is especially important in the selection of points in TID studies in electronic devices irradiation. Due to its particular characteristics, this is one the few devices of its kind in the world, in the sense of a multi-technique ion beam analysis of large areas. The continuous development of the external beam setup coupled to the robotic XYZ stage at LAMFI, is becoming a robust and reliable option for regular analysis of trace elements (Z > 5) competing in most cases with the traditional in-vacuum Ion Beam Analysis, with the additional advantage of simpler sample loading and absence of vacuum constrains of the sample. The authors like to express the enormous help and dedication of R.F. Assis, A.R. Leite, and M.R. Antonio in the development of LAMFI's external millibeam setup.

## O-63: Ion Component Diagnosis of Metal Hydride Cathode Vacuum Arc Ion Source by Using an in-Cavity Minitype Magnetic Mass Spectrometer

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Metal hydride cathode vacuum arc ion sources can produce microsecond to millisecond intense pulsed proton beams, which are applied in many areas, such as accelerator injection and ion implantation. Generally, the ion components in metal hydride cathode vacuum arc are complicated and consist of numerous metal and nonmetal ions with different charge states. In this paper, we firstly review the methods and devices for ion component diagnosis of vacuum arc ion source used by former researchers. Then the technical features of a home-made incavity minitype magnetic mass spectrometer mass are introduced. By using this mass spectrometer, the ion components and their charge state distributions at different discharge moments are presented. The evolutions of different ion components with time are also given.

## O-64: Nuclear Reaction Analysis for H, Li, Be, B, C, N, O and F with an RBS Completeness Check

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<sup>15</sup>N nuclear reaction analysis for hydrogen is combined with 1.2 MeV deuteron induced nuclear reactions which provide a simultaneous analysis for Li, Be, B, C, N, O and F. The energy dependence of these 1.2 MeV deuteron induced nuclear reactions has been measured and used to correct for the energy loss of the incident deuteron beam in the film being analyzed. After the nuclear reaction analysis is completed, a conventional 2 MeV He RBS measurement is made. All three measurements are needed for a self-consistent analysis: the complete film composition is needed both to interpret the <sup>15</sup>N nuclear reaction data and to apply the deuteron energy loss correction. The composition is determined by a self-consistent analysis of the light element nuclear reaction data (both <sup>15</sup>N and deuteron) combined with an RBS analysis of any heavy element (typically Si) present in the film. This composition is used to make RUMP simulation with no adjustable parameters of the complete RBS spectrum. Comparison of this simulated RBS spectrum with the measured spectrum provides a check that there are no major undetected components in the film, and, in some cases, a check that the film has uniform composition vs depth. This procedure has been used to analyze several hundreds of thin films

with a wide variety of compositions and has proven to be robust. All measurements are made using the same scattering chamber typically loaded with 14 samples with the bombarding beam changed from  $^{15}N^{++}$ , to 1.2 MeV deuterium, to 2 MeV  $^{4}He^{+}$ . While some laboratories are reluctant to run deuteron beams, our experience with 50 namp 2.4 MeV D<sup>2+</sup> beams, radiation is not a significant issue.

### O-65: TOF-ERDA analysis on the effect of water for SEI formation in Li-ion batteries

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In the field of Li-ion batteries, extensive researches have been carried out to understand the process of solid electrolyte interphase (SEI) formation on electrode surfaces. In terms of acquiring depth profile, time of flight elastic recoil detection analysis (TOF-ERDA) is a promising method for the measurements of light elements, including H and Li, with better depth resolution [1-3]. Here we demonstrate TOF-ERDA analysis, to evaluate the effect of water in a cell for SEI formation.

Electrodes were prepared by electrochemical cycling of a complete lithium-ion cell, whose positive / negative electrode consists of  $LiNiCoMnO_2$  / graphite, respectively. 1M  $LiPF_6$  dissolved in 1:1 ethylene carbonate / diethylcarbonate was used as electrolyte. We prepared two kinds of electrolytes, w/ and w/o water, where one contains 500 ppm moisture in the electrolyte, and the other is dewatered to below 30 ppm moisture content. All samples were handled in an argon filled glove.

Details of the TOF-ERDA experimental setup is presented in ref. [3]. In this work, we used two time detectors in order to improve the time resolution. The time detectors are positioned at a front and a rear of the flight pass, using carbon foils with a thickness of 3 mg/cm<sup>2</sup> (front) and 10 mg/cm<sup>2</sup> (rear), respectively.



Figure above shows the comparison of C, H, and Li depth profiles of the negative electrode in the cell, whose electrolyte was dewatered. In the case of the cell after 1 cycle, there is almost no additional layer except for the carbon. On the other hand, after 300 cycles, Li, H content increases at the surface region, which can be explained by the SEI formation. We applied TOF-

ERDA analysis to evaluate the SEI formation. The way to acquire the quantitative depth profile, and the comparison of w/ and w/o water will be presented.

[1] W. Wong, S. Hayakawa, K. Maeda, S. Fukuda, M. Yanokura, M. Aratani, K. Kimura, Y. Gohshi, I.Tanihata, Nucl. Instr. and Meth. B 124 (1997).

[2] W. Bohne, J. Rohrich, G. Roschert, Nucl. Instr. and Meth. B 139 (1998) 219.

[3] K. Yasuda, C. Batchuluun, R. Ishigami, S. Hibi, Nucl. Instr. and Meth. B 268 (2010) 2023.

## O-66: Hydrogen release from irradiated elastomers measured by Nuclear Reaction Analysis

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Advanced elastomers used in sealings of mobile connections must respond to numerous requirements: ability to work in wide temperature range, resistance to various fluids, ageing and oxidation. Several materials were developed to answer these needs. A common feature of these materials is a relatively high friction coefficient leading to increased wear rate and temperature rise in the contact area. Recently ion irradiation has been successively used to reduce friction and wear without affecting bulk properties of elastomers. Irradiation with ions leads to shrinking and smoothening of the surface layer, what suggests, that the effects obtained may be related to the hydrogen release from the surface layer. A detailed analysis of hydrogen concentration in irradiated samples is thus required to confirm or reject this assumption. Main experimental problem related to hydrogen measurements is a high susceptibility of organic materials to decompose under an ion beam. This excludes such methods as ERDA, as heavy ions lead to a very fast deterioration of the elastomers. The paper describes the experiments performed by using Nuclear Reaction Analysis (namely <sup>15</sup>N(1H,  $\alpha\gamma$ )<sup>12</sup>C reaction induced by <sup>15</sup>N ions at 6.385 MeV) to measure the amount of hydrogen atoms in several irradiated elastomers. Huge advantage of this reaction is a very low irradiation fluence needed to collect a decent spectrum. A massive loss of hydrogen has been successfully detected and the results compared to elastic and inelastic energy losses. The analysis points to the decisive role of ionization processes in hydrogen release. Beneficial effects of ion irradiation on functional properties of irradiated elastomers will also be discussed.

## O-67: Boron quantification limits using Ion Beam Analysis techniques

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Boron is an important element in silicon technology, as either a dopant or a contaminant. Whatever the problem, its precise quantification in very low levels is a need [1, 2]. Recently at USP, the requirement for quantification of Boron in metallurgical-grade silicon to produce solar-

grade silicon has motivated a revision of Ion Beam Methods best suited for boron quantification. In samples of this kind, boron is dissolved in a chunk of polycrystalline silicon, very different from the ideal boron doped Si planar thin film or wafer samples usually measured by IBA. We present experimental results comparing the quantification limits for B on Si analyzed by NRA, ERDA using a Si beam and PIGE. Thin natural B/Si films, produced by PVD were specially prepared for the experiment. For approximately the same analysis time, the results show that PIGE should be preferred for low-level boron quantification, taking advantage of its higher yield and the convenience of in air measurements, using an external beam setup. Although the quantification limits of ERDA and PIGE are similar, ERDA measurements need approximately 20 times higher dose to reach the same quantification limit of PIGE at comparable experimental conditions. However, the results are mostly indicative, since there is plenty of space to fine tune experimental conditions lowering B detection limits for a specific experimental setup.

[1] S. Abo, H. Horiuchi, et al., Surf. Interf. Anal. 44 (2012)

[2] P. Chau, N. Meshram, et al., Thin Solid Films 519 (2011).

# ABSTRACTS POSTER SESSION 1 Monday

## PA-1: <sup>14</sup>C environmental applications in Mexico.

### E. Chávez, C. Solís, M.E. Ortiz, A. Huerta, J.A. Flores

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LEMA is a new AMS facility in Mexico with the initial capacity to measure low concentrations of <sup>10</sup>Be, <sup>14</sup>C, <sup>26</sup>Al, <sup>129</sup>I and Pu isotopes. In this presentation, the main features of the facility are described along with results in radiocarbon analysis in aerosol samples from Mexican cities.

### PA-2: Studies of Be migration in the JET tokamak using AMS with 10Be marker and $\mu$ -NRA

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The JET tokamak is operated with beryllium limiter tiles in the main chamber and tungsten coated carbon fibre composite tiles and solid W tiles in the divertor. One important issue is how wall materials are migrating during plasma operation. To study beryllium redistribution in the main chamber and in the divertor, a <sup>10</sup>Be enriched limiter tile was installed prior to plasma operations in 2011-2012. Methods to take surface samples have been developed, an abrasive, metod for bulk Be tiles in the main chamber, which permits reuse of the tiles, and leaching with hot HCl to remove all Be deposited at W coated surfaces in the divertor. Quantitative analysis of the total amount of Be in cm<sup>2</sup> sized samples was made with inductively coupled plasma atomic emission spectroscopy (ICP-AES). The <sup>10</sup>Be/<sup>9</sup>Be ratio in the samples was measured with accelerator mass spectrometry (AMS). The microscopic distribution of deposited Be was studied with microbeam nuclear reaction analysis ( $\mu$ -NRA) using 3-4.8 MeV <sup>3</sup>He beam. The experimental setup and methods are described in detail, including sample preparation, measures to eliminate contributions in AMS from the <sup>10</sup>B isobar, quantification, background elimination, possible activation due to plasma generated neutrons, source dilution due to mixing with <sup>9</sup>Be and making sure that the sampling depths were adequate. Marker concentrations in the range 0.4-1.2% of the source <sup>10</sup>Be were found in the divertor deposits, with moderate poloidal variation. The Be deposited in the divertor is preferentially accumulated in pits and depressed regions at the surface. Different ways of averaging the areal density of Be at the surfaces are discussed and compared with ICP-AES. A reasonable agreement is found between the NRA and ICP-AES results, provided that the non-uniform deposition is taken into account.

## PA-3: The <sup>28</sup>Si(d, $\alpha$ )<sup>26</sup>Al cross section measured with the help of an accelerator mass spectrometry laboratory

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Recent experimental methods and techniques have opened new ways to explore nuclear reactions of significance for nucleosynthesis near the temperatures of stellar burning.

In this work we focus on the case of <sup>26</sup>Al. Regions of the universe were abundances of that isotope are seen become related to active heavy nucleosynthesis. Its presence on the solar system was unexpected until it was found in the Allende Meteorite. It is now understood that cosmic rays induce nuclear reactions on materials to produce it. On Earth, this process is well understood and it's the basis of environmental studies. So <sup>26</sup>Al is not just the product of some high metallicity star collapse.

Taking advantage of the recently upgraded laboratory facilities at the Instituto de Física, at UNAM in Mexico City, notably the new AMS laboratory (LEMA) we measured the cross section for the <sup>28</sup>Si(d, $\alpha$ )<sup>26</sup>Al nuclear reaction and report here our first results.

## PA-4: Performance report for the low energy compact radiocarbon accelerator mass spectrometer at Uppsala

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A range of ion beam analysis activities is ongoing at the Uppsala University, including accelerator mass spectrometry (AMS). Various isotopes are used for AMS but the isotope with the widest variety of applications is radiocarbon. Hitherto the 5 MV Pelletron tandem accelerator has been used for radiocarbon AMS, typically using 12 MeV  $^{14,13,12}C^{3+}$ .

Recently a new radiocarbon AMS system, The Green-MICADAS was installed at Uppsala, which is a joint collaboration between the Uppsala group and the ion physics group at the ETH Zurich. The system has a number of interesting characteristics, which will be described. The system operates at a terminal voltage of 170 kV and helium stripper gas, using singly charged carbon ions. The low- and high-energy mass-to-charge analysers in the system are stigmatic dipole permanent magnets (0.42 and 0.97 Tesla) requiring no electrical power or cooling water. The system measures both the  ${}^{14}C/{}^{12}C$  and the  ${}^{13}C/{}^{12}C$  ratio on-line. Performance of the system is presented for both standard mg samples as well as  $\mu$ g sized samples.

## PA-5: IBA investigations on 5th century A.D. loose garnets from Pietroasa, Apahida and Cluj-Someşeni treasures

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Scientific investigations on garnets from cloisonné polychrome jewels dated to the Late Antiquity and Early Medieval period can reveal essential information about gems provenance, commercial routes and access to precious mineral resources. A large number of cloisonné adornments dated to the 5<sup>th</sup>-6<sup>th</sup> centuries A. D. were discovered through archaeological research or by chance beyond the boundaries of the Roman Empire. Some of the most impressive gold objects adorned with garnets were found on nowadays Romania's territory. The most spectacular artefacts are the ones belonging to Pietroasa and Cluj-Someşeni treasures and to the inventories of the princely graves from Apahida. As a part of a more generous project aiming to reveal the chaîne operatoire employed in making these jewels, a set of 418 loose garnets belonging to the above-listed treasures were subjected to scientific investigations. At this stage of the research, only the gems dismounted by the accidental discoverers before the recovery of the gold artefacts from the authorities were analyzed. The structure and finishing of the garnets was studied by Optical Microscopy, and their chemical composition was non-destructively determined by micro-PIXE technique in external beam mode at AGLAE accelerator of C2RMF, Paris, France. The microscopy observations revealed a reduced number of mineral inclusions and different finishing manners. Some garnets show clear signs of recycling. PIXE concentrations indicated that a different sub-type of garnet from the pyralspite series is prevailing for each treasure. By comparing our results with previously published compositional data taken on garnets of known geological provenance, it was deduced that a large number of such gems from distinct sources were available to the craftsmen who made these Migration Period adornments. The variability in the chemical composition of the garnets analyzed in this study shows that Pietroasa, Cluj-Someşeni and Apahida II and III artefacts were produced using gemstones of distinctive provenances.

## PA-6: Compositional studies on bronze alloys of ancient Greek monetary arrowheads based on X-rays analyses

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An interdisciplinary program to study the alloys composition of Greek monetary and warfare Scythian design arrowheads found together in same deposits in Dobroudja (near Black Sea coast) was started using micro-PIXE and portable-XRF methods. Micro-PIXE experiments were performed using LNL-INFN AN2000 accelerator microprobe and XRF investigations using a X-MET 3000TX portable spectrometer in Bucharest. If XRF provided general information on elemental composition of alloys, micro-PIXE was used to study minor and trace elements as fingerprints for items provenance and - based on elemental maps - metallurgical aspects. Until now, we investigated discoveries in Istros (a famous Greek colony on Black Sea coast), Golovita and Cogealac (settlements near Istros) and Floriile (autochthonous fortified settlement). Besides the "classical" Copper-Tin-(Lead) bronze type, with various proportion of tin (to increase hardness) or lead (to facilitate the casting process), two unusual types of bronze used both for warfare (including pieces with cut pointed-end impossible to be used as weapon) and for monetary arrowheads were identified: Cu-Sn-(Pb)-Mn for Golovita, Cogealac and Floriile items and Cu-Sn-(Pb)-Sb for many Istros items. We also identified some monetary arrowheads with a mixed alloy containing both Mn and Sb, more probably from re-melting of warfare arrowheads. In-homogeneous aspects related to imperfect metallurgy and corrosion are also presented. A preliminary geological provenance of Cu-Mn and Cu-Sb (poly-metallic ores?) minerals could indicate Ukraine (very rich in manganese ores) or Caucasus for Cu-Mn and north-west of Hungary from Cu-Sb, all Scythian populated areas in VII-V Centuries BC. We propose the following time-evolution scenario: -Warfare Scythian design arrowheads used for trade between Greek colonies and "barbarian" neighbors (mainly Scythians); -Mechanically modified (without killing capability) arrowheads "safely" used as pre-coins; -Dedicated cast monetary signs (mainly leaf-shaped) issued by Greeks from re-melting of the real arrowheads. Interpretation of these results in relation with commercial and political aspects between Greeks and local (Scythian, Thracian) populations is presented.

## PA-7: Analysis of ancient alloys with luster: brass and treated base silver

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Non-destructive PIXE analysis using in-air proton beam was used for studies of earliest brass coins of the 1<sup>st</sup> c. BC minted by Greeks, Romans and Celts and for the studies of Roman coins of the late 3<sup>rd</sup> c. AD made of debased, yet surface enriched silver. Brass, an alloy of copper and zinc which can attain gold-like luster came into wider use after 100 BC, first for coins minted by several Greek cities in Asia Minor, and since mid 1<sup>st</sup> c. BC for parts of the Roman military equipment; Roman brass coinage was established decades later, in 23 BC. The analysis aimed to determine the level of zinc and important trace elements; among them, selenium was found as characteristic and confirmed connection of earliest brasses with the east. For silver coins, more and more debased metal was used until the late 3<sup>rd</sup> c. AD; however, to give the coins a bright silver luster, the coins blanks were surface enriched before striking. Applying the technique of concentration mapping, polished exposed parts and untouched depression regions of the coin revealed the enrichment and plating techniques. It was found out that the surface enrichment

in combination of tinning that simulated solid silver was replaced by mere silver plating around 270 AD.

## PA-8: Pigment identification in hand-painted issue of Sigmund Herberstein, 1560 AD

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Sigmund Herberstein, a leading diplomat of the Austrian emperor Ferdinand 2<sup>nd</sup>, described his diplomatic missions in a book that was printed in 1560 AD. The book is equipped with hand-colored graphic illustrations. Only a few such copies are preserved, one of them in Slovenia. As the coloring carries individual signature, we tried to determine the pigment types in the Slovenian book, which can serve for further comparison between different preserved copies. The measurements were made by PIXE, using in-air proton beam of 3 MeV nominal energy. The current was a few tenths of nA and measurement in particular points lasted up to 200 seconds. The results show some known mineral pigments, like cinnabar and minium for red and malachite or verdigris for green. As specific we found yellow made of gold dust and three types of blue: azurite, an organic one and a complex compound containing arsenic, cobalt iron and potassium. It seems this color is a based on iron-gall ink in combination with cobalt-bearing minerals. Iron-gall ink is also a component of black and brown color.

## PA-9: Ion beam modification in pigments: case of zinc white

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Paint materials are known to be sensitive to particle irradiation. During the analysis of paintings by ion beam techniques (IBA) a change in color may occur. The cause of this discoloration can be due to ion interaction with the two main components of the paint: the mineral pigment and the organic binder. In order to understand the modification, due to one (or both) of these components, we have irradiated pure white mineral pigments (lead white, basic lead sulphate, calcium sulphate, gypsum, calcite, zinc white, titanium oxide and lithopone). In this presentation, we focus on the zinc white pigment (ZnO) which was mainly used in the 19th century. In the first part of the study, ZnO was irradiated in conditions representative of IBA by using the external 3 MeV proton micro-beam of the AGLAE accelerator (Centre de recherche et de restauration des musées de France C2RMF, Paris, France). In the second part, complementary irradiations in vacuum with 2 MeV H<sup>+</sup> and 1.2 MeV Au<sup>+</sup> ions were conducted at

JANNUS-Saclay in order to observe damage effects in electronic and nuclear energy loss regimes. Modifications were characterized by ex situ and in situ  $\mu$ -Raman spectroscopy. 1.2 MeV Au ions induce a important damage characterized by a decrease of the 438 cm<sup>-1</sup> band assigned to the wurtzite structure and an increase of the broad band at 540-580 cm<sup>-1</sup> associated to disorder. Au ions irradiation also induces a strong yellow coloration of the pigment. Conversely, 2 MeV H<sup>+</sup> ions seem to have a very light effect on the structure and on the color, showing safe conditions for applying IBA to 19<sup>th</sup> century paintings.

## PA-10: Investigation of golden thread used in Persian precious carpets of Safavid period by nuclear microprobe

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To look at a Persian carpet is to gaze into a world of artistic magnificence nurtured for more than 2,500 years. The Iranians were among the first carpet weaver of the ancient civilizations. During the Safavid period (1499-1722 AD), the hand-woven carpets are known for their artistic beauty and quality produced in the royal carpet workshops. In these workshops, carpets are woven based on the patterns created by prominent painters. High quality raw materials sometimes decorated by golden threads were used to create unique precious carpets. In this research work, golden thread of precious Persian carpets belonging to Safavid period is studied using nuclear microprobe.

The samples of this work are collected from the carpet museum of Iran. Measurements are performed using the scanning microprobe system based on a quadrupole triplet (Oxford Microbeams). The probing beam focused down to about 10  $\mu$ m on the target in the reaction chamber is provided by the 3 MV single ended Van de Graaff accelerator of nuclear science and technology research institute in Tehran. The detection set-up is equipped to perform PIXE and BS analysis in list-mode acquisition. The thickness and elemental composition of the layered golden threads are characterized using micro-PIXE and micro-RBS. Based on these analyses, manufacturing process and quality of applied materials are inspected. Investigation of superficial layers of these golden threads could reveal the complex composition leading to their opaque and darkened color. Moreover, the practicalities and problems involved in the IBA analysis of this kind of material are discussed.

## PA-11: Provenance study of Persian Turquoise by IBA spectroscopy techniques

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Turquoise is one of the most priced and beautiful gemstones that has adorned the rulers of

Persia, Ancient Egypt, the Aztecs, and Mesopotamia. In many cultures turquoise has been considered a holy stone and a talisman, which brings good luck and health to the person carrying it. In Persia, turquoise was national stone for millenniums and its use in the civilizations of Shahr-e Sukhteh (3200-1800 BC) and Achaemenid dynasty (550–330 BC) is largely attested by the numerous artifacts decorated with this gemstone. The intense blue color of the gemstone was a symbol of heaven on earth, therefore they created turquoise mosaics, inlays and overlays and embellished the domes of the mosques, and other important buildings. The Persian style of turquoise was so popular and coveted that it was shipped all over the world.

Persian turquoise was considered as the best quality of turquoise for thousands of the years and it still sets the standard for quality. One of the most important deposits of this precious gemstone is in Neyshabur [1]. Regarding the cultural and archeological importance of the turquoise, its provenance study is a subject of major interest. However to our knowledge, the Persian turquoise has never been studied by IBA techniques.

The general formula of turquoise is:  $A_{0-1}B_6(PO_4)_{4-x}(PO_3OH)x(OH)_{84}H_2O$ , where x=0, Cu<sup>2+</sup>, Fe<sup>2+</sup>, Ca<sup>2+</sup>, and Zn<sup>2+</sup> can be the substitutes for the A-site, and Al<sup>3+</sup>, Fe<sup>3+</sup>, and Si<sup>3+</sup> could be found in B-site. In additional to its complex chemical formula, the bulk sample includes some rare earth elements that play a critical role in its provenance study, but attempts to develop trace element techniques for turquoise have been largely unsuccessful [2].

Combination of non-destructive techniques of PIXE, ERD and IBIL provides a means to obtain information regarding the chemical composition of gemstones. In this work, ERD has been employed to quantify hydrogen content in the turquoise samples, while PIXE as a multi-elemental analysis method has been applied to determine the elemental composition of the samples. In addition, detection of transition elements and rare earths has been enhanced using the fast and highly sensitive method of IBIL [3].

In this research work a collection of turquoise samples from different mines of Iran was studied in Van de Graaff lab of Tehran, using combination of PIXE, IBIL, and ERD methods. The results were so promising and signify their suitability for studying the turquoise samples. Moreover, the capability of these methods for the provenance study of turquoise artifacts has been confirmed.

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[2] S. Hull et al, J Archaeol Sci, Vol 35 (2008) 1355-1369.

[3] N. P. –O. Homman et al, Nucl Instrum Meth A, Vol 353 (1994) 512-519.

## PA-12: Identification of pottery production from the necropolis of Jiyeh archaeological site by PIXE

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The excavations of the ancient Porphyreon of the Jiyeh archaeological site, about 28km due south of Beirut, revealed the remains of some pottery production features beneath a level of

graves. The discovered material enabled the identification by the archaeologists of two different phases of pottery production, dated to the Late Hellenistic and Early Roman periods. Particle induced X-ray emission technique PIXE is used to determine the elemental composition of about 134 the studied excavated shards. The analysis protocol provided almost 20 elements in one spectrum, including majors, minors and traces. The elemental composition provided by PIXE and based on 12 most abundant elements, ranging from Mg to Zr, was used in a multivariate statistical program, were two well defined groups were identified.

## PA-13: Characterization of archeological pottery from Tyre historical site using PIXE technique and cluster analysis

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It is proposed to study the excavated ceramics from Tyre, the prestigious city of antiquity (locally named Sour and located at 85 km south of Beirut, Lebanon). The originality of Tyre in this context is its long permanence of prosperity as a great center of pottery production and maritime trade through the centuries without interruptions, which were experienced by neighboring cities and rivals. In this work, several series of excavated pottery are analyzed in order to characterize the Tyre production, based on the elemental composition, and thus to be distinguished from those of other neighboring workshops (Serapta, Sidon or Acre), possible sites of ceramic production at this period. Particle induced X-ray emission technique PIXE is used to determine the elemental composition of about 107 excavated shards. The elemental composition provided by PIXE and based on 12 most abundant elements, ranging from Mg to Zr, was used in a multivariate statistical program, were two well defined groups were identified.

## PA-14: Analysis of the Notger reliquary by PIXE, XRF and Raman spectroscopies

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Prince Bishop Notger reliquary (Curtius Museum, Liège) is a parchment manuscript of the 10<sup>th</sup> century with sumptuous binding composed of ivory, enamel and gilt brass from 10<sup>th</sup>, 13<sup>th</sup> and 16<sup>th</sup> century. Its binding consists of a central plate ivory (10<sup>th</sup> c.) representing Notger kneeling before a shrine-shaped chapel. The bishop holds a book with both hands. A complete analysis of its binding has been performed by PIXE, XRF and Raman analysis with particular attention on the enamels and the brass. Results will be presented and discussed in relation with similar

objects.

## PA-15: Study of Portuguese tiles used to decorate historic Brazilian buildings through non destructive analysis

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During the 19<sup>th</sup> century, several buildings in the north and northeast regions of Brazil were partially or entirely faced with tiles manufactured in Portugal [1]. In the southeast region this practice of use tiles to decorate the external face of buildings was not common however, two historical buildings in Itu (a city of São Paulo) presents this characteristic and were faced almost entirely with standard-pattern tiles [2] that exhibit a distinct design, usually geometric shapes or vegetable drawings, that repeats next to each other all over the wall [3]. With the goal of to identify the pigments and the ceramic matrix elements of some tiles from the historical building of the Museu Republicano, nine tiles were investigated with the non destructive analyses such as EDXRF, PIXE and IBIL. Particularly the PIXE technique was used to perform a large area mapping. The correlation of the concentrations with the positions can give more information of the overall distribution of the elements of the pigments in the decorative tiles. The motivation of the use of mapping for this kind of sample is related to the macro-scale of the object under study. This new facility for macro mapping analyses is now available at LAMFI laboratory in the external beam setup. This upgraded external beam system also permits simultaneous IBIL measurements. The results show that the studied tiles are composed of Si, Al and Ca oxides and by pigments with presence of specific elements such as Pb in the white pigment area, Mn in the black traces and Co in the blue areas. PIXE mapping also show that Sn is uniformly distributed in all area probably due to the composition of a glaze material that covers the decorative face of the tiles [4]. The Portable EDXRF system was used to confirm the systematic presence of these elements in all studied samples.

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#### PA-16: Thick multi-layers analysis with high energy PIXE

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High-energy PIXE is suitable to analyze thick target due to the larger range of the energetic ion. Among the applications of this method we find the characterization of ancient paintings [1] and metallic archaeological objects [2]. The latter was analyzed without removing the patina layer on the surface. Another advantage is the low energy loss per unit length of high energy incident ion allowing reduced radiation damage. To identify the pigment composition, sequencing and the thickness of paint-layers, K and various L X-rays for heavy element (like Pb) and K lines for lighter element (like Fe) were used. A comparison between the measured intensity ratio  $K_{\alpha}/K_{\beta}$ ,  $L_{\alpha}/L_{\beta}$  and  $L_{\alpha}/K_{\alpha}$ , influenced by absorption, and the intrinsic ratio, give information on the depth of the emitting layer. Several questions need to be treated such as how many layers can be analyzed and how to resolve the case where we have a repeated layer in depth (complex multilayer). In addition, the analysis of a thick multi-layer target is a good starting point to determine the concentration profile of an element in function of the depth. We have developed an experimental set-up for high energy PIXE at ARRONAX cyclotron (Nantes-France) with proton and alpha ions of 68 MeV [3]. It includes two X-ray detectors (SDD and LEGe) and one gamma ray detector (Ge) for PIGE. We made several experiments with multi-layer targets (simple and complex). For each multi-layer, we irradiated with multiple angles between the target and the beam. For layers with medium and heavy elements, we succeeded in locating the layers and in determining their thicknesses using the ratio of the K and L lines intensities. For layers with lightweight elements, in case their X-rays are absorbed completely, we also located the layers and determined their thicknesses by coupling the prompt gamma rays emitted from these lightweight elements in a one hand, with the ratio of the K and L lines intensities of medium and heavy elements from the other layers in another hand. In case of complex multi-layers, we used the variation in the ratio of the K and L lines intensities according to the irradiation angle and the intensity of gamma rays measured during and after irradiation. During this talk, a description of the experimental setup will be presented together with examples on simple and complex thick multi-layers targets solved with PIXE at high energy using informations given by the measured X and gamma rays.

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#### PA-17: PIXE analysis for characterization of flint archaeological artefacts

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Elemental composition is known to be a parameter for identification of archaeological objects. Nuclear techniques of analysis (NAA, PIXE, XRF etc.) are widely adopted for

characterization of archaeological artefacts. A paper on the flint artefacts identification is infrequently found. Inhomogeneity of flints complicates using of elemental composition for the characterization. But the inhomogeneity of elemental composition can be employed as an independent parameter for sample identification. Elemental analysis of the samples was performed using an electrostatic accelerator with proton energy of 1 MeV and beam diameter of 1 mm. The analysis involved 23 samples of flint excavated at the territory of National reserve "Khortytsa" (Ukraine). A copper jumper was used to avoid the sample charging. X-ray spectra were processed in GUPIXWIN software. Concentrations of Al, Si, K, Ca, Ti, Mn, Fe were measured. Data on elemental composition were analyzed by principal component analisys (Fig. 1). Some samples were analyzed at several points. Feature of this analysis is that the concentration of the flint samples were not averaged for several points. Instead, all the results of the analysis included as the separate data for the PCA. Si<sub>x</sub> clusters are observed.



Fig. 1. Plane of two first main components of the PCA analysis of silicon samples composition.

Results of the PCA analysis show that clusters don't demonstrate a provenance of flints but reflect difference of elemental composition for different parts of the artifact. Some samples are part of different clusters (with different frequency). Whatever the clusters nature is, we suggest that enter frequency may be an essential parameter at sample identification that describes inhomogeneity of elemental composition. Our further research is related to assessment of a suggestion that this new parameter is more stable as compared to averaged elemental composition of flint.

#### PA-18: The contribution of IBA to health and environmental studies

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The human health is related to a balanced diet, minimum exposure to contaminated environmental and a healthy lifestyle. These conditions include a broad range of factors like toxic metals and organic compounds, vitamins and nutrient-deficiency disorders. It is known that the human diet includes several organic and inorganic compounds that increase the risk of development of obesity, cardiovascular disease and cancer. Healthy food intake may provide essential nutrients to guarantee the chemical balance during regular organism functions and to avoid related diseases. In this context, the contribution of IBA has been notorious since it provides elemental composition of food, beverage and biological tissues from species exposure to contaminated areas. The aim of this work is to summarize the main contribution of IBA in Brazil to human health and environmental issues, and show recent results obtained from native fruits, which include elemental composition and physicochemical parameters analysis, animal tissue composition alterations by different diets or fruit juice intake. Fruits are an important source of nutrients related to prevention of aging diseases associated to oxidative stress, specially red, blue and black berries. However, regional and exotic fruits are sometimes used without any previous knowledge of inorganic and organic composition. Moreover, the benefits of those products to health should be studied before human consume. In this way, we determined by PIXE technique the composition of a native fruit (Bunchosia glandulifera (Jacq) Kunth Malphiguiaceae specie), popularly known as falso guarana, from the south Brazilian state and the benefits of acerola juice through elemental composition of kidney and liver of animals supplemented with it. Falso guarana is characterized by intense red pulp and seed and is used mainly to produce energetic juice, while acerola (Malpighia emarginata DC), originating from Central and South America, is considered an important source of vitamin C and dietary minerals. Fourteen elements were quantified for seed and pulp of falso guarana. P, Ca and Mn present higher concentration in seeds than in pulp. Anti-oxidants properties, as well as organic compounds, are being determined to correlate with the elemental composition. Concerning acerola, swiss male mice were fed with a hypercaloric diet (cafeteria diet) and, subsequently, were separated in different groups that received one of the following food supplements for one month: water; unripe acerola juice; ripe acerola juice; industrial acerola juice; vitamin C; or rutin. Mineral concentrations of the acerola juices, liver and kidney tissues were determined. Results suggest that the simultaneous intake of acerola juices, vitamin C or rutin in association

with a hypercaloric and hyperlipidic diet provides change in the mineral composition of organisms, which plays an important role in the antioxidant defenses of the body.

# PA-19: Simultaneous Quantification of Amoxicillin and Clavulanic Acid in different commercial drugs Using PIXE Technique

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Monitoring the quantity of an active ingredient (A.I.) in a commercial drug is a must for public health concerns. We have demonstrated, in previous studies [1,2], that Particle Induced X-Ray Emission (PIXE) is one of the most rapid and accurate choices for quantification of an A.I., in a solid drug, from the reactions induced on its specific heteroatom's using pellets made from original tablets. In this work, PIXE is used, for the first time, for simultaneous quantification of two A.Is., Amoxicillin and Clavulanic acid, in five different commercial drugs. Since the quality control process of drugs covers a large number of samples, the scope of this study was also too found the most rapid and low cost sample preparation needed to analyze these drugs with a good precision. The chosen drugs were analyzed in their tablets' "as received" form, in pellets made from the powder of the tablets and also in pellets made from the powder of the tablets after being heated at 70°C in order to remove the humidity present in one of the A.I.'s structure. The quantification validity related to the aspects of each sample preparation (homogeneity of the drug components and humidity) are presented and discussed.

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### PA-20: PIXE optimization for deciduous teeth analysis

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Measuring deciduous teeth by PIXE, though looking straightforward can be a hard task. PIXE

analysis employing 2.4 MeV proton beam is limited to the first 25  $\mu$ m, therefore restricted to the enamel, making the comparison with chemical methods of limited use. Tooth samples are small and irregular with an ill-defined geometry, thus spreading the data of a statistical analysis. Deciduous teeth are very clean samples. Their enamel is composed mostly of P and Ca, and about 40 ppm Fe, 4 ppm Cu, 500 ppm Zn and 100 ppm Sr. Other elements are bellow PIXE detection limits. Due to the intense signal of Ca and P, sum peaks must be carefully controlled not to interfere with the quantification of elements from Fe to Zn. The setup for PIXE analysis of deciduous teeth enamel needs to be carefully tuned and adapted to yield the lowest possible detection limits without increasing too much the acquisition time. PIXE at LAMFI was employed to analyze 80 deciduous teeth, half of them from preterm born children, searching for correlations with future defects and cavities. There are several causes for these defects, and it is known that low-weight infants, intubated or submitted to laryngoscopy are the most affected [1]. Since tooth enamel is considered a "biological recorder", environmental conditions can induce temporary or permanent changes [2]. The main goal of this work is to compare enamel trace elements of preterm and full term infants. PIXE data were complemented with XRF analysis showing close statistical agreement. Strontium concentrations have been identified a statistical significative parameter separating preterm from full term born children. (Financial support FAPESP 2012/03829-3)

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### PA-21: A study of trace elements in canine elbow dysplasia by PIGE and XRF

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The elbow dysplasia is characterized by an abnormal development of the elbow joint that occurs frequently in dogs aged between 4 and 6 months of medium and large size races [1]. It is a generic term for a set of conditions: osteochondritis dissecans of the humeral condyle, ununited anconeal process, joint incongruity of the elbow and fragmentation of the medial coronoid process. The last one is the most common injury of the elbow dysplasia [2] and can be caused by several factors, including dysfunction bone formation, genetic diseases, nutritional and mechanical effects [1]. In order to achieve a better knowledge of the pathogenesis of the disease were performed elementary studies in healthy and unhealthy coronoid processes, as well as in bone-cartilage interface by Proton Induced Gamma-ray Emission (PIGE) and X-Ray Fluorescence (XRF).

For PIGE analysis the experiment was performed at the Tandem accelerator facility of CTN/IST in Lisbon. The  $\mu$ -XRFS analysis was carried out using the Bruker's M4 Tornado spectrometer of LIBPhys at FCT/UNL.

PIGE allowed a qualitative and quantitative analysis of the coronoid light elements such as F,

Na, Mg, Al and P. The XRF was used for quantification and mapping of respective heavier elements such as P, Ca, Fe, Cu, Zn and Pb.

The role of Ca/P ratio and trace elements as well as cartilage organization at the bone-cartilage interface, implicated in fragmentation of the medial coronoid processes were interpreted.

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# PA-22: Measurement of the Ca/P ratio of healthy bone samples by micro-PIXE, PIGE and micro-XRF

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Bone diseases such as osteoporosis and Paget Disease of Bone have harmful effects in the patient quality of life. An earlier and precise diagnosis is important to improve treatment. The relative content of Ca and P is critical for sustaining mineral homeostasis and bone metabolism and has been investigated as a possible biomarker for the assessment of bone health [1].

Micro-X-Ray Fluorescence Spectroscopy ( $\mu$ -XRF) (Bruker's M4 Tornado) and Ion Beam analytical techniques, namely  $\mu$ -PIXE (Proton Induced X-ray Emission) [2] and PIGE (Proton Induced Gamma-ray Emission) are employed in this work both for measuring the Ca/P ratio and for clarifying how sample X-ray absorption and surface roughness can influence the obtained results, profiting from the different depth sensitivity range of each of the techniques.

The Ca/P ratio was measured in different points of each sample with  $\mu$ -XRF and  $\mu$ -PIXE and the results are compared, in order to understand how geometric effects and absorption inside samples can modify Ca/P ratio. To support our results the phosphorous concentration in the samples was measured by PIGE analysis, where absorption inside the sample is negligible.

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## PA-23: Imaging of biological tissue with MeV SIMS method: freeze-dried and frozen hydrated sample preparation

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To broaden our analytical capabilities with molecular imaging besides the standard elementar imaging with micro-PIXE, a linear Time-Of-Flight mass spectrometer for MeV Secondary Ion Mass Spectrometry (MeV SIMS) [1] was constructed and incrementally added to the existing detection setup of nuclear microprobe at Jožef Stefan Institute [2]. In the first part, we show the results on a series of standard reference materials deposited on silicon. We measured absolute molecular yields and damage cross-section, which ensure the execution of MeV SIMS in static regime, without significat alteration of the fragile biological samples. We explored the analytical capability of the technique for chemical mapping of plant tissue. A series of thin plant tissue slices were prepared by standard shock-freezing and freeze-drying protocol and deposited on the Si wafer. To improve the chemical preservation of the tissue, we innitiate the construction of the sample holders enabling the MeV SIMS analysis of the tissue in frozen hydrated state, previously implemented at JSI nuclear microprobe for micro-PIXE [3]. We show the molecular spectra and maps obtained, and report on the attempts to interprete the results by biochemical processes in plants.

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### PA-24: Withdrawn

## PA-25: Potential of the Bucharest 3 MV tandetron for IBA studies of deer antler mineralization

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Antlers are bony cranial appendages of the deer characterized by an annual cycle of loss and regrowth. They are the fastest growing bones in mammals, which make them a valuable model for studying mineralization of primary bone and the influence of hormonal, dietary and pollution factors. Antlers are typically formed in 120-150 days, but their cortical bone is formed beginning on 70<sup>th</sup> day of growth. A time-resolved view on mineralization status can be obtained by taking samples from the first, second and third antler beam and labeling with calcein, a fluorescent indicator of calcium. Elemental analysis by methods like PIXE, PIGE and RBS may help for a better understanding of mineralization dynamics. The present study reports a preliminary survey of the potential offered by IBA at the 3 MV tandetron in Bucharest for the investigation of cortical bone from deer antlers and femur. The antlers were labeled with calcein on 117<sup>th</sup> day of the growing period, in order to follow bone mineral deposition, and Ca was mapped by optical microscopy. The antler and femur samples were embedded in polymethylmethacrylate, cut as ~1 mm-thick cross-sections, and polished. IBA measurements were performed with 3 MeV protons. Reference materials included pelleted hydroxyapatite (bone ash), feldspar, glass and soil with certified compositions. PIXE analysis of P and Ca was done with 2 % uncertainties; for trace elements the uncertainties were of 10 - 50 %. In PIGE the uncertainties were of 10 % for Na, Al and P, 20 % for F, and 35 - 50 % for Mg. RBS provided information about Ca, C, and O contents. P, Ca and Ca/P ratio in femur were very close to the bone ash standard. Antler samples from the first (R1), second (R2) and third (R3) beam showed lower P and Ca than in femur, in the order R1  $\sim$  R2 > R3, evidencing a lower mineralization after R3 where the antlers break most frequently. There were appreciable differences between antlers from different animals. The same order ( $R1 \sim R2 > R3$ ) was found for Na, and in one antler for F. Compared to femur, S was higher in antlers, while Zn was lower in R3 beam where osteomalacia (softening caused by defective mineralization) was noted.

The main advantages of the 3 MV tandetron for antler studies are the following:

1. The visualization of sample's surface, focusing and precise positioning of the beam.

2. The X-ray detector placed inside the reaction chamber, which allows detection by PIXE of biologically relevant light elements.

3. The simultaneous detection of PIXE, PIGE and RBS spectra, which give complementary information (a "total IBA" approach).

4. The automated control of experimental parameters.

5. Depth profiling for studies of surface phenomena.

The advantage of bone samples for IBA is their physical-chemical stability. Their main disadvantages are the electroinsulating character, strong matrix effects (thick samples), and heterogeneous structure. Thus the 3 MV tandetron evidenced a high potential for studies of bone mineral.

### PA-26: A setup for oxygen isotope analysis in fish otoliths

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Otoliths are small, crystalline biogenic structures that mainly consist of aragonitic (CaCO3). It form part of the hearing and balance system of teleost fishes. The otoliths grow incrementally and the accretion is daily, so the age and growth rate of fish can be determined [1]. The chemical composition of the otolith reflects the environmental history of an individual fish. A set of trace elements and isotopic ratios can be used to track past behavior and changes in the water [2]. E.g. the strontium and calcium (matrix) -ratio reflects the salinity in the water, and can be used to study the migration pattern of an individual fish. During the last decade the Sr and Ca variations in several thousand otoliths has been analyzed at Lund ion beam analysis facility LIBAF [3]. The ratio between O<sup>18</sup> and O<sup>16</sup> is another useful tracer, as an environmental thermometer, i.e. it can be used to study the ambient temperature experienced by the individual fish [4]. To extend the analytical toolbox for otolith analysis at LIBAF, to also include oxygen isotopes, a new setup for photon tagged nuclear reaction analysis pNRA is under development [5, 6]. This new setup utilizes modern equipment such as a LaBr<sub>3</sub> scintillator detector and an annular double sided silicon strip detector DSSSD, both connected to a fast VME based data acquisition system. The setup has been tested using both protons and deuterons as a beam particle. In this work the results from these initial measurements as well as the results from oxygen isotope measurements with NRA and pNRA in otoliths will be presented.

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# PA-27: Assessment of fine ambient aerosol particles from a suburb area of Beirut using PIXE and gravimetric analysis

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In this work, it is investigated the elemental composition of fine and coarse air particulate matter PM2.5 and PM10-2.5 collected during 2014 in a suburb area of Beirut, using the ISAP®1050e sampler having a combined inlet. The collection of fine particles was carried out on thin Teflon filters while the coarse ones were collected by impaction using a greased ring Kapton foil. The characterization of the elemental content of the two fraction mode, fine and coarse particles, were analyzed using proton induced X-ray emission technique PIXE. It will be focused on the elemental composition of the fine fraction PM2.5, using PIXE in two runs. Proton beam of energy 1 MeV irradiated the samples in order to determine low Z elements, while 3 MeV protons, with 75  $\mu$ m Kapton filter used as X-ray absorber, were used to determine the high Z elements. This allows the determination of elemental concentration (in ng/cm<sup>2</sup>) of Na, Mg, Al, Si, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn and Pb. For each sample, there was a clear dependence of the elemental content, as well as the mass of total PM2.5, with traffic and meteorological conditions.

### PA-28: Characterization and source apportionment of fine particulate sources at Rijeka, Croatia from 2013 to 2015

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Daily aerosol samples with PM2.5 particulate matter have been collected in Rijeka, Croatia during period of 18 months (August 2013 to February 2015). Nearly 260 samples have been collected on Teflon filters and analyzed by Ion Beam Analysis (IBA) techniques PIXE, PIGE and PESA giving information for 22 elements and elements from H to Pb. Additionally, black carbon was determined with the Laser Integrated Plate Method.

Results were statistically evaluated using Positive Matrix Factorization (PMF). Major pollution sources: secondary sulphates, vehicles, ship emissions, heavy oil combustion, smoke, road dust, sea spray, and soil dust were identified together with their relative contributions to total PM2.5 pollution.

#### PA-29:Withdrawn

# PA-30: Mass spectrometry analysis of urban environmental particulate matter collected by mobile phones

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The increasing needs for controlling harmful influence of particulate matter (PM) air pollution require monitoring in the immediate vicinity of man. So far, stationary installations state for the typical equipment used to collect or control PM levels. These installations do not accurately characterize human 24-hour exposure. More reliable results could give mobile and personal devices, but such devices are usually inconvenient and impractical. In this paper, we apply and develop a technique recently presented by our group [1]. The technique relates to the collection of suspended dust particles from the air in the immediate vicinity of the person. Dust particles that get into the inner parts of mobile devices are extracted and analysed. In the present work the dust was collected from wasted mobile phones returned for the utilization by citizens of two cities in Poland: Gdansk and Krakow. The collected samples, in the number of over 200, have been studied by mass spectrometric techniques. Ion beam analysis was used in secondary ion mass spectrometry (SIMS) technique. Rastered Ar<sup>+</sup>, 5 keV ion beam sputtering was applied for samples placed on a substrate of high purity indium. Depth profile analyses were performed using oxygen-flooding technique. Bulk analysis of collected samples was done using spark-source mass spectrometry (SSMS) and inductively coupled plasma mass spectrometry (ICP-MS). The obtained results are compared with the results of PM collected with the use of standard urban environmental monitoring system. The comparison is done in the periods of use of the given mobile phones. Samples gained from mobile phones have shown some conformity to the PM collected conventionally, especially in their atomic composition. The material collected from mobile phones is additionally polluted with clothing fibber materials, sweat, epidermis, etc.

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#### PA-31: Characterization of explosive residues using MeV-SIMS

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Explosives are mostly organic substances, which rapidly release a large amount of energy during decomposition, which is dissipated as blast waves, propulsion of debris, or the emission of thermal and ionizing radiation. Millions of tons of explosives are used every year for obtaining minerals and metals from the ground; in most cases, explosive detonations are used for coal mining. Also, explosives have been used extensively in botmilitary and terrorist context. The misuse of explosives, which jeopardizes public safety, requires that appropriate detection and analysis measures are in place. These should be able to characterize the explosives and identify their source. While secondary ion mass spectrometry (SIMS) is a well-established surface characterization technique, recent years have seen the emergence of a new technique employing MeV heavy ions beams to produce secondary ions from an insulating sample surface: "MeV-SIMS". Unlike keV primary ions, MeV ions can be extracted through a thin  $Si_3N_4$ window and travel a few millimetres in air enabling imaging with a submicron resolution under ambient conditions, avoiding negative vacuum effects on sample, simplifying sample preparation and significantly decreasing the total analysis time. In addition to MeV-SIMS analysis which provides information on chemical composition of sample, PIXE analysis can be performed simultaneously and give information on trace elemental composition. The combination of MeV-SIMS and PIXE potentially gives a good quality image at high spatial resolution. Samples of post blast material containing RDX, HMTD and PETN have been analyzed using a 2MV Tandetron with ambient pressure MeV-SIMS. Valuable information on the sample collection and storage has already been obtained. In this work we are aiming to identify the molecular signature of the explosive residues and their fragments in the spectra. For the spectra interpretation principle component analysis is performed to help specify and group different types of explosives. Additionally, the samples are also analyzed with keV-SIMS to demonstrate the sensitivity of the MeV technique.

# PA-32: Micro-PIXE studies on Transylvanian native gold samples from Rosia Montana and Cavnic deposits

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Ten samples (hundreds of microns diameter) of native gold from Rosia Montana (Apuseni Mts) and from Cavnic (Baia-Mare district) were scanned by micro-PIXE to obtain information on

electrum structure - values of ratio Ag/(Au + Ag), on presence of Au and Ag minerals (Te and Sb compounds) - many as micro-inclusions and on other characteristic elements as Hg. Rosia Montana is one of the oldest and most interesting gold deposit with both veins and stockworks. Cavnic deposit is located in the eastern part of the Carpathian belt, one of Europe's major metallogenic provinces. The samples are from recovery obtained by inhabitants from landfills of former mining plants. Micro-PIXE analyses were performed at LNL AN2000 accelerator from Legnaro and at AGLAE Louvre Accelerator in Paris. On Rosia Montana samples we detected micrometric areas (5µm X 5µm) rich in Ag and Sb and without Au, Sb/Ag ratio being 1/5-6, indicating the presence of stephanite - Ag<sub>5</sub>SbS<sub>4</sub>. In one sample we detected the micro-presence of mercury, suggesting Hg could be used as a "fingerprint" for Rosia Montana gold deposit. On Cavnic samples, to investigate Au and Ag minerals, we analyzed areas of approx. 50 microns diameter. The study was focused on Sb and Te presence and on the variation of Ag/(Au+Ag) ratio which characterize electrum's metallogeny. The ratio varies from 0.221 to 0.395 - average value of 0.27, with big differences from point to point illustrating electrum's nonhomogeneities. One micro-area revealed an important presence of Te (16657 ppm), a significant presence of Sb (2861 ppm) and an increase of Ag content (Ag=32.75% versus Au =50.05%), indicating the presence of a Ag telluride containing also Sb. The high As content (6.64%) could indicate benleonardite - Ag<sub>8</sub>(Sb,As)Te<sub>2</sub>S<sub>3</sub> highlighted in Kremnica, Slovakia. A comparison with a micro-mineralogical study using a SEM (Scanning Electron Microscope) associated with EDX (Energy-dispersive X-ray spectroscopy) facility is presented. Some cassiterite (Sn oxide) micro-grains extracted from Valea Pianului alluvial gold samples were also analyzed and traces of Zr, Nb, Ta, W were identified.

#### PA-33: Optimization of the boron analysis system using a DSSSD at LIBAF

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Dating back more than 20 years, boron related analyses have been performed at LIBAF (Lund Ion Beam Analysis Facility), mainly with a focus on geological samples [1]. This rather extensive boron program has historically centered around the nuclear reaction  $^{11}B(p,2\alpha)\alpha$  [2], using a beam energy just below 700 keV as the reaction has a broad resonance here. In this work we present data from recent experiments, focused on the physics aspects of boron measurements/analysis rather than the geological applications. We have studied the nuclear reaction p+<sup>11</sup>B going to three "high energy" alpha particles over an energy interval of 500-850 keV, to study the energy dependence of the yield and related it to the main background components in geological samples. In addition, thanks to our upgraded experimental setup where a single detector has been replaced with a DSSSD (double-sided silicon strip detector) [3,4] – we have been able to study the angular distribution dependence of the reaction. Results from these experiments will be reported on followed by a discussion how the analysis could be optimized depending on aim, sample and background. Much of the geologically oriented activities at LIBAF during the past ten years have also included developments in the analysis of isotopic ratios in geological samples. We already have established techniques for measuring carbon, hydrogen and oxygen isotope ratios [5-7] by various nuclear techniques like scattering, NRA and pNRA, and more recently we have expanded the field to also include measurement of boron isotopic ratios  $({}^{10}B/{}^{11}B)$  utilizing a combination of NRA and pNRA techniques. Results from these experiments will also be reported on.

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### PA-34: Revealing the hidden earliest version of "The illustrious house of Ramires" by Eça de Queiroz using micro-XRF

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An original manuscript of Eça de Queiroz (1845-1900) "The illustrious house of Ramires", published in 1900, was analyzed using X-Ray Fluorescence. Several words and sentences were scratched as corrections for later versions. In two cases, however, almost half a page was covered with a new sheet of paper (glued) and rewritten. Taking advantage of the penetration power of the characteristic X-rays of the Chromium present in the ink, we were able to reconstruct the sentences that were edited by the author. In Fig.1 an example of the mapping obtained using Bruker M4 Tornado is presented. The images created combined with paleographic expertise will enable the discovery of the preliminary version of this classic of Portuguese literature.



Fig.1- Comparison of the mapping of the Cr (green) signal for both inks with the video image of the manuscript.

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# PA-35: Characterization of hydrogenated and deuterated thin SiC films codeposited by magnetron sputtering

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Silicon carbide is a material with many applications, including fusion reactor technology [1] and also fission power reactors. In the present work we will focus on investigations by Elastic Recoil Detection technique of the H/D content in silicon carbides thin layers deposited on silicon substrate.

Silicon carbide  $Si_xC_y$  thin films were codeposited from pure silicon (Si) and graphite (C) targets using a dual magnetron system configured in the so named confocal configuration (for increasing the uniformity of the deposited layers). The discharges are powered using two independent RF (13.56 MHz) power supplies. A number of 15 samples with stoichiometry in between y/x = 0.2 up to 1.25 were deposited on Si(100) substrates. The stoichiometry was adjusted by correspondingly adjusting the powers applied to C and Si targets in between 50-120W, using sole Ar as deposition gas. Deuterated / hydrogenated  $Si_xC_y$  (for  $y/x\approx1$ ) samples were obtained mixing H<sub>2</sub> or D<sub>2</sub> with Ar during codeposition process. Supplementary, samples of  $Si_xC_y$  (for y/x = 0.2; 0.63 and 1) were exposed for four hours to D<sub>2</sub> plasma generated in a RF glow discharge.

Material properties of all deposited samples were investigated by means of specific techniques. The surface morphology and topography were investigated by Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM). All deposited samples (deuterated/hydrogenated or not) present a smooth surface and their roughness present a slow dependence over the deposition parameters. The thickness of each sample was obtained, concomitant with their optical properties, from Elipsometry measurements Elemental composition of the SiC samples was investigated by RBS. The deposited deuterated/hydrogenated films were characterized by Elastic Recoil Detection technique using a <sup>4</sup>He beam. The experimental arrangement employed for the measurements was the following: the samples were mounted at 150 with respect to the <sup>4</sup>He beam and two Si detectors were placed at 300 (with a mylar foil to stop the elastically scattered <sup>4</sup>He ions) and at 1650. Spectra of recoiled H and D ions and of backscattered <sup>4</sup>He ions were measured simultaneously. Fourier Transform Infrared Spectroscopy (FTIR) investigations of the samples revealed the presence of D-C-D chemical bonds (respective H-C-H) in the samples codeposited using Ar/D<sub>2</sub> and Ar/H<sub>2</sub> mixtures, respectively. Still, exposure to D<sub>2</sub> plasma lead to incorporation of D<sub>2</sub> without formation of specific chemical bonds.

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# PA-36: Ion energy loss spectroscopy and neutron microradiography for analysis of structural parameters of biosensors with nuclear tracks

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Two novel techniques, developed in NPI ASCR Rez, i.e., microprobe ion energy loss spectroscopy and 3D neutron microradiography, were used for study of a new type of biosensors that is based on micropores formed by etching in thin ion-irradiated polymers. To possess a proper function, i.e., to sense targeted biomolecules (e.g., enzymes), the pores should acquire a certain shape - a conical form with a micrometre size opening on one site and a nanometre size opening on the opposite site. The process of etching is however delicate and can lead to pores that can differ even in the close neighbourhood, and thus affect the biosensor behaviour. It is therefore important to find, whether the etched pores are of a correct shape, either individually or in average. The applied methods proved to be a proper instrument for inspection of the structural parameters (shape and area distribution) of the pores. They also helped to control fabrication of a prototype of a biosensor for detection of the laccase enzyme.

### PA-37: Damage depth distribution in the halogen-irradiated PET and PEEK polymers

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Rutherford Backscattering (RBS), Neutron Depth Profiling (NDP), Atomic Force Microscopy (AFM), Electron Paramagnetic Resonance (EPR) and Raman spectroscopy were used for study of damage depth distributions in PEEK (poly-aryl-ether-ether ketone) and PET (polyethylene terephthalate), industrially important polymers, irradiated with the MeV halogen ions ( $F^+$ ,  $CI^+$ ,  $Br^+$ ,  $I^+$ ) up to the fluence  $10^{14}$  ions/cm<sup>2</sup>. The depth profiles of the damaged area were traced by the Li or Hg markers incorporated into the polymers by diffusion from the Hg vapours or LiJ water solution. As main results: (i) significant changes in morphologies, micromechanical properties and molecular transformations of the irradiated zones were found, different for different halogen ions; (ii) the distribution of the radiation damage was (for higher ion fluences) fractioned into 2 zones followed electronic and nuclear energy transfers, (iii) post-irradiation thermal annealing incited dramatic redistributions of the damaged sites, the process was enhanced at and above the glassy temperature.

### PA-38: Application of IBA methods for in situ study of deuterium retention and thermal desorption in mixed layers

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Components inside the tokamak fusion device are made of different materials. Beryllium (Be) and tungsten (W) will be used as plasma facing material in ITER for the first wall and divertor, respectively. Plasma-surface interactions during the fusion device operation lead to erosion and re-deposition of the material, forming mixed material deposits on various surfaces inside the vessel. Besides W and Be these mixed layers will contain intrinsic (C, O, etc.) and seeding impurities (N or noble gases).

We have studied deuterium (D) retention and thermal desorption from ITER relevant mixed layers with different compositions produced by thermionic vacuum arc technique [1]. In situ Rutherford Backscattering Spectroscopy (RBS), Nuclear Reaction Analysis (NRA) and mass spectroscopy were used. The experimental vacuum chamber is equipped with sample holder, capable of heating the sample up to 1200 K, hydrogen/deuterium atom beam source (HABS), quadrupole mass spectrometer and two PIPS detectors for RBS and NRA. The novelty of this work is that D loading of the samples, depth profile analysis and thermal desorption were all performed in situ in the same vacuum chamber under high vacuum conditions, without exposing samples to the air. Moreover, two complementary techniques, NRA and mass spectroscopy, were employed simultaneously for monitoring the D concentration in the sample and the desorbed hydrogen species during thermal desorption study. Heating rate for these studies was approximately 15 K/min. RBS was used to determine the initial layer composition and to monitor any change of layer structure during the heating.

Thermal desorption of deuterium was studied in mixed C:W and C:W:Al layers, where deuterium was already co-deposited in the layers. Deuterium depth profile was measured by NRA using <sup>3</sup>He ion beam at different ion beam energies before and after sample heating. During the heating the same ion beam at single energy (2.5 MeV) was used. The decrease of the D

signal was correlated with the increase of the masses 3 and 4 in the mass spectrum. Main release from the C:W mixed layer occurred at relatively high temperature, around 730°C. Some deuterium was found to remain in the layer after the heating of the sample.

Deuterium uptake and thermal desorption were studied in initially deuterium-free mixed W:Al, W:Al(N<sub>2</sub>) and W:Be(N<sub>2</sub>) layers (Al being used as a Be substitute [2]), studying the influence of nitrogen co-deposition in the layers. Samples were exposed to D atom beam at 100°C for 20 hours (fluence of  $3.24*10^{19}$  D/cm<sup>2</sup>) and D depth profile was measured by NRA using <sup>3</sup>He ion beam before and after sample heating. During the heating the remaining and desorbed deuterium was again monitored by NRA and mass spectroscopy, respectively. Significant increase of deuterium uptake was observed in the layers with co-deposited nitrogen. Main release of deuterium from the layers took place between 350°C and 600°C. After the heating no deuterium was found to remain in W:Al mixed layers and small amount of deuterium was still present in W:Be mixed layer. During the heating severe layer modifications were observed on W:Al samples using the RBS technique.

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# PA-39: The use of ERBS for thin film thickness measurements and the study of O diffusion in $TiO_2$ films

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Electron Rutherford Backscattering (ERBS) is a technique that depends on the recoil energy transferred from the scattering electron to a nucleus in a large-angle deflection. This energy transfer depends on the mass of the scattering atom. Analysing the energy of the scattered electrons reveals thus which atoms are present in the near surface layer [1, 2].

In simple cases, where there are 2-3 separate peaks due to different elements that do not overlap, one can simply fit the spectra with the corresponding number of Gaussians. In more complicated cases, where peaks overlap, a unique fit of the spectrum based on a larger number of Gaussians can not be obtained. The fit has then to be restrained by fixing the peak positions to the energy loss values of the different elements present. The added complexity usually requires the measurement of the sample under different geometries and/or incoming energies and the simultaneous analysis of all spectra. As this work shows, fitting of the data allows for very precise thickness and compositional determination. Such a case is the analysis of a Si<sub>3</sub>N<sub>4</sub> layer on TiO<sub>2</sub>. Here the peaks overlap strongly, but highly consistent estimates of the thickness of the Si<sub>3</sub>N<sub>4</sub> film are obtained for different measurement geometries.

This procedure was then applied to the study of oxygen selfdiffusion in TiO<sub>2</sub> through the use of isotopic markers. Conventional ion-beam techniques are capable of such measurement but

require particular care to ensure that radiation damage does not affect the measurement. Oxygen diffusion in  $TiO_2$  is of particular interest, as it is used for memristors the working of which depends on oxygen diffusion [3].

Here we use this electron-scattering technique to measure interdiffusion in  $Ti^{16}O_2 / Ti^{18}O_2$  and  $Ti^{18}O_2 / Ti^{16}O_2$  bilayers. High-resolution measurement of the energy of electrons backscattered from oxygen atoms makes it possible to distinguish between <sup>18</sup>O and <sup>16</sup>O isotopes at the surface and hence monitor the diffusion. The results obtained by ERBS will then be compared to the analysis using conventional IBA techniques such as RBS, MEIS and NRA.

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# PA-40: Rutherford Backscattering Spectrometry and X-Ray Fluorescence as probes to determine trace elements in hair samples

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Human hair consists basically of three layers: the cuticle (outer part), the cortex (middle part) and the medulla (inner part). Alpha-keratin is a spiral shape protein that supports the hair. It is immersed in the cortex and is composed mainly by a sulfur-containing amino acid, cysteine. Histological sections demonstrated high activity of heavy metals in the alpha-keratin. Although they constitute about 1% of the hair, the presence of certain metals indicate the absorption and exposure of the individual to that element. Thus is possible to diagnose, for example, nutritional deficiencies or high levels of elements that may cause damage to health. Faced with the analysis of other biological samples such as urine and blood, hair has the advantage of easy sampling without trauma to the individual, easy transport and storage, besides the chemical and structural stability of this sample. In the present work we compare the metal content in hair samples from two sets of people who live at the outskirts of the Rio de Janeiro city (Chácaras Arcampo - Duque de Caxias). While the first group drinks plain well water, the second group includes people who drink the water distributed by the Rio de Janeiro State Company, CEDAE. Two techniques have been used: X-Ray Fluorescence (XRF) and Rutherford Backscattering Spectrometry (RBS). Virgin hair samples of healthy people and non-smokers were collected, washed and stored according to the guidelines of the International Atomic Energy Agency (IAEA). The XRF measurements were made at the Federal University of Rio de Janeiro (X-Ray Fluorescence Spectrometer S8 TIGER – BRUKER), in the energy range of 0.5 to 56 keV, using the Best Detection Method. The RBS data were collected at the Federal University of Rio Grande do Sul (Tandetron - 3 MV HVEE ion accelerator using 2000 keV He<sup>+</sup> incident ions and detection at 165°). Although preliminary in essence, XRF measurements clearly evidence the presence of S, Ca, P, Zn, Fe, Mn, Cu, Cl, Al, and Ga in all samples. The presence of the nonessential element Pb was only observed in hair samples from people that ingested well water. Using the RBS technique, Si, Pb and Ti were also observed. In addition, this last technique allowed the observation of light elements such as C, N and O, which cannot be observed using the XRF technique. The non-essential element Pb was only observed in hair samples from the person who drinks well water, confirming the data obtained by XRF technique. The authors thank FAPERJ, CAPES and CNPq for financial assistance.

# PA-41: Use of Ion beam (RBS) and X-Ray techniques (XRF, NEXAFS) in the elemental characterization and chemical speciation of Amazonian plants

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Determination of the elemental composition and chemical speciation of macro and micronutrients brings an important light towards understanding the homeostatic mechanism of plants. In the present work, Rutherford Backscattering Spectrometry (RBS), X-Ray Fluorescence (XRF), and Near Edge X-Ray Absorption Fine Structure (NEXAFS) techniques are employed in the characterization and chemical speciation of trace elements present in samples derived from Amazonian plants.

While XRF and RBS give information on the elemental composition of natural samples, with a minimum effort on sample preparation, Near Edge X-Ray Absorption Fine Structure Spectroscopy (NEXAFS) is able to give answers concerning their speciation. RBS is a very sensitive technique that allows for the observation of both light elements such as O and large Z elements such as Fe. NEXAFS spectra, taken on specific core edges, may be related to the different functional groups to which each element is attached (chemical speciation).

We have obtained XRF and RBS spectra from samples derived from three Amazonian plants: Bixa orellana, Andira retusa and Duroia macrophylla. These plants are widely used among Amazonian inhabitants and are the focus of much bioprospection work. Bixa orellana, commonly known as urucum, is used to extract its natural colorific, bixina, which has also therapeutic applications. Andira retusa is used in helmint treatment and Duroia macrophylla produces a natural alkaloid with possible application in cancer treatment. XRF spectra were obtained both at the Brazilian Synchrotron Radiation Laboratory, LNLS, at 2500 eV and 4200 eV photon energies as well as at the Federal University of Rio de Janeiro. In the latter case, a Bruker (S8 Tiger 1 kW) X-Ray Spectrometer was used. The RBS data were collected at the Federal University of Rio Grande do Sul (Tandetron - 3 MV HVEE ion accelerator using 2000 keV He<sup>+</sup> incident ions and detection at 165°). NEXAFS spectra were measured both at the Sulfur and Calcium K-edges. We show in figure 1 a RBS spectrum of urucum seeds. A large amount of calcium and oxygen could be identified, along with smaller quantities of elements such as iron, nickel, zinc and others. Sulfur could also be identified using the XRF technique. Analysis of the chemical environment around some of these elements is currently under development, using the NEXAFS technique. As an example we claim, from the measured Ca 1s NEXAFS spectrum, shown in Figure 2, that this element is present in the seeds as calcium carbonate, CaCO<sub>3</sub>. This may be confirmed by comparing our data to an available spectroscopic analysis of calcium carbonate (Hayakawa et al, Analytical Sciences, 2008).



Fig 1: RBS spectrum of urucum seeds

Fig 2: Spectrum of urucum seeds taken at Ca K-edge

These and other results from the other plants will be presented and discussed in our presentation.

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### PA-42: First round robin exercise on sample handling and measurement protocol for MeV-SIMS

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Secondary Ion Mass Spectrometry induced by MeV heavy ions (MeV-SIMS) has emerged in recent years as a promising analytical technique for submicron mapping of molecular concentrations at the surface of organic and insulating samples. The numerous applications of this non-marking technique in art, archaeometry, forensics, or in the analysis of biological

samples, as well as the possibility to extract a focused beam in ambient pressure, motivate interest.

The promise of the technique was also recognised by the International Atomic Energy Agency (IAEA), which has recently initiated a Coordinated Research Project (CRP No. F11019) to support the development of molecular concentration mapping techniques using MeV focused ion beams. The aim is to provide robust theoretical and practical foundations and credibility to MeV-SIMS.

Within this CRP, a first round robin exercise has been carried out in order to outline best practice methods for sample handling and measurement protocol. Valuable insight in some fundamental parameters associated with mass spectrometry imaging is investigated. Samples of thin organic layers (leucine, angiotensin II, and ovalbumin) on silicon, PMMA, and PTFE filters have been provided to the round robin participants. Ions and energies have been chosen for a primary ion velocity of v ~1.0 cm/ns. Secondary ion yields (Y<sub>0</sub>) have been measured for the static limit at a fluence of  $\phi_{\text{static}}=10^{10}$  cm<sup>-2</sup> and their evolution recorded for fluences up to  $\phi_{\text{static}}=10^{13}$  cm<sup>-2</sup>. Damage cross-sections,  $\sigma$ , have also been measured and the measurement efficiencies (Y<sub>0</sub>/ $\sigma$ ) have been determined. An inter-laboratory comparison was performed through these. Insights in best practice for sample contamination and measurement protocol will be provided.

# PA-43: Comparison of PIXE and XRF analysis of airborne particulate matter samples collected on Teflon and quartz fibre filters

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In the framework of research projects for sampling and analysis of airborne particulate matter, Particle Induced X-ray Emission (PIXE) and Energy Dispersive X-ray Fluorescence (ED-XRF) techniques are routinely used in many laboratories in the world to determine the elemental concentration of the particulate matter samples. In this work an inter-laboratory comparison of the results obtained analyzing several samples (collected on Teflon and quartz fiber filters) with both techniques is presented: the samples were analyzed by PIXE (in Florence, at the 3 MV Tandetron accelerator of INFN-LABEC laboratory) and by XRF (in Elche, using the ARL Quant'X, Thermo Scientific Inc, USA, EDXRF spectrometer with specific conditions optimized for specific groups of elements). The results of the two sets of measurements are in good agreement for all the analyzed samples. A comparison of the Minimum Detection Limits for both techniques will be shown for all the measured elements and for the two different substrata.

Moreover, the comparison of PIXE and XRF results on Teflon and quartz fiber filters allows the quantification of possible self-absorption effects due to the penetration of the aerosol particles inside the quartz fiber-filters.

### PA-44: Production of thin targets by implantation for astrophysical purposes

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Due to the extremely small cross sections involved, the production of targets for astrophysical purposes is a real challenge [1]. Moreover, the use of thin transmission targets is sometimes relevant due to the measure of elastic scattering between identical particles, such as the  ${}^{16}\text{O}+{}^{16}\text{O}$  reaction [2,3]. The thickness of the target must be high to maximize the reaction products yields, but not so high as to allow a correct calculation of the effective beam energy and the charged products go through. Besides this, the target must withstand high beam current densities without noticeable deterioration, and contaminants must be minimal. The production of thin targets is performed with an innovative technique, in which a thin layer of  ${}^{12}\text{C}$  (with few nm) is produced over a glass sheet by evaporation and then implanted with the required isotope with 100% purity in most cases. The elements implanted through this process until now were the  ${}^{16}\text{O}$  and the  ${}^{19}\text{F}$ . The targets were them analyzed with a 2.5 Van de Graaff Accelerator and a 3.0 MV Tandem Accelerator, at the lon Beam Laboratory at CTN (Sacavém - Portugal). The targets were characterized using two different lon Beam Analytical Techniques: i) Nuclear reaction analysis (NRA); ii) Rutherford Backscattering Spectroscopy (RBS). The elemental concentrations and depth distributions were calculated using SIMNRA [4].

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### PA-45: Computer simulation of RBS spectra from rough samples

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Surface roughness of samples analyzed by RBS complicates the structure of the measured spectra and makes their interpretation more difficult and ambiguous. The roughness effects on RBS spectra have been addressed in several theoretical and experimental studies published from early 1980s but no systematic study of the adverse effects of different roughness kinds has been performed up to now. Fast code was written for simulation of RBS spectra from the rough samples. Sample surface or interface relief is described by a polyline and the simulated RBS spectrum is obtained as a sum of many particular spectra from randomly chosen particle

trajectories. The simulation of 1000 particular RBS spectra requires about 10 s on a standard PC computer. The code includes procedures generating different types of roughness. The effects of different types of surface roughness, random or periodic, on RBS spectra is demonstrated on the samples comprising either rough layer deposited on smooth substrate or smooth layer deposited on rough substrate. Significant differences between the RBS spectra from rough layers with thickness distributed according to Gaussian and Gamma distributions and from those with periodically and randomly distributed roughness are found. RBS spectra in various measuring geometries from the rough samples were simulated and rather dramatic spectra distortions were found for measurements in glancing angle measuring geometry. In some instances the simulations together with measured RBS spectra may provide useful, averaged information on the sample surface roughness complementary to that obtained by other techniques. Financial support of this work from the GACR project P108/12/G108 is gratefully acknowledged. The work was done at the CANAM (Center of Accelerators and Nuclear Analytical Methods LM2011019) infra-structure.

#### PA-46: Ion Beam Analysis of Laterally Inhomogeneous Materials

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Ion beam analysis (IBA) methods are often applied for the analysis of laterally inhomogeneous samples, such as rough or fuzzy surface layers, porous materials, or heterogeneous agglomerates like geological samples, paint layers, or collections of dust particles. Popular simulation codes for the evaluation of IBA spectra [1], however, assume a laterally homogeneous layered sample structure. This is a potentially serious problem for the quantitative IBA analysis of spatially inhomogeneous materials, because a priori there is no guarantee that one gets quantitatively correct results for laterally inhomogeneous materials if a homogeneous layered structure is assumed for the evaluation. The newly developed program STRUCTNRA allows the simulation of IBA spectra (MEIS, RBS with Rutherford or non-Rutherford cross-sections, ERDA, and NRA) from arbitrary 2-dimensional structures taking all correlations between incident and exit trajectories into account. It works on a grid with arbitrary grid size. Each grid cell can be either void, or it can be filled with an arbitrary material. Sample structures can be imported as scanning or transmission electron microscopy images of sample crosssections or as artificial sample drawings created by any graphics software. The well-known SIMNRA program [2] is used as simulation kernel. Stopping and straggling effects are calculated precisely, multiple and plural scattering can be taken into account approximately. Layers with a distribution of layer thicknesses are generally called rough. STRUCTNRA confirms the results derived from the simplified roughness model in [3] which is valid in the limit of small aspect ratios, i.e. for surface structures where the height is much smaller than the lateral extension: Deviations remain below about 5% if the lateral extension of structures is at least 3 times larger than the maximum distance between the entry and exit points of the incident and exit trajectories. For structures with larger aspect ratios scattering spectra get more and more distorted. Calculated spectra from fuzzy surfaces may have only little resemblance with spectra from a smooth or somewhat rough surface. For heterogeneous agglomerates the heights and shapes of IBA spectra depend on the size and spatial arrangement of the constituents. Only for very small constituents, i.e. if the energy loss in the constituents is small, the sample can be approximated as a layered structure with a depth profile. For larger constituents this approximation fails, and the spectra can deviate considerably from the spectrum of a layered structure. Quantitative analysis of heterogeneous agglomerates therefore generally requires taking the size distribution of the constituents into account; otherwise results can get incorrect.

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### PA-47: Limits of deuterium depth profiling in tungsten with the D(<sup>3</sup>He,p)<sup>4</sup>He nuclear reaction

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The D(<sup>3</sup>He,p)<sup>4</sup>He nuclear reaction is commonly used for deuterium detection as it is a very sensitive and non-destructive method. Absolute and differential cross sections are available from experiments as well as from calculations so that quantitative analysis is possible. However, the high energy of the protons, the inverse kinematics of this reaction, and its strong sensitivity to angular spread requires special experimental set-ups. While the protons are used to probe larger depths (up to 12 microns in tungsten for 6 MeV <sup>3</sup>He) the alphas are typically measured at glancing angle to achieve good depth resolution close to the surface (information depth < 300 nm in tungsten for 800 keV <sup>3</sup>He). To probe different depths the energy of the incident <sup>3</sup>He is varied. However, depth profiling with this reaction is not straight forward: First, it has a broad resonance region around 630 keV, second, it shows a tail at higher energies where the cross section has still 15% of its maximum value (roughly constant between 3 and 6 MeV). While the first limits the possible depth resolution the latter results in the fact that high energy <sup>3</sup>He always probes the whole information depth and not only the depth of interest. As a consequence one has to deal with an ill-posed inversion problem requiring the deconvolution of the measured data to derive the depth profile. In most publications only the resulting depth profiles are shown with no or little discussion of the assumptions used to derive them and without the confidence interval of the result. While most authors restrict themselves to modeling the integral proton counts as function of <sup>3</sup>He energy a tool exists that fits all measured proton and alpha spectra for all <sup>3</sup>He energies simultaneously. Even Bayesian experimental design was shown to be applicable to this case allowing to optimize the choices of measured <sup>3</sup>He energies "on the fly" during the measurement. Yet in every days practice the available beam time is typically spent to measure with prefixed energies which are selected based on the experimentalists sentience.

In this presentation the influence of the choice of initial energies as well as the choice of input parameters for the deconvolution such as the layer thickness or the energy calibration are presented for mock-up data as well as for real world examples. In addition it will be shown how a large D concentration close to the surface raises the detection limit and decreases its accuracy in depth. Analysis of deuterium plasma loaded tungsten specimen of different deuterium concentration profiles are chosen as examples. MeV tungsten self-damaging was used to modify the defect density and hence the deuterium retention. Isotope exchange with hydrogen

plasmas was used to selectively remove deuterium from the surface without influencing the deuterium content in depth. Several different approaches which are used in the community to deconvolute the measured spectra are applied for the very same data and the consequences are discussed.

# PA-48: Computer simulation program for medium energy ion scattering and Rutherford backscattering spectroscopy

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A new ion scattering computer simulation program for ion scattering and its graphical user interface (MEISwin) have been developed. The program has been used for analysis of both medium energy ion scattering (MEIS) and Rutherford backscattering spectroscopy (RBS) in Ritsumeikan University since 1998 and in Rutgers University since 2007. The main features of the program are as follows: (1) stopping power can be chosen from five datasets (Andersen and Ziegler (1977), Ziegler et al. (1985) [1], Ziegler and Biersack (1991), SRIM (2000), SRIM (2011)), (2) straggling can be chosen from two datasets (Bohr and Lindhard Sharff [2]), (3) spectral shape can be chosen from two datasets (Gaussian and exponential modified Gaussian [3]), (4) cross section can be chosen from two theories (Coulomb and screened cross section [4]), (5) resonant elastic cross section of  ${}^{16}O({}^{4}He, {}^{4}He){}^{16}O$  [5] can be used for simulation, (6) pileup simulation for RBS spectra can be supported, (7) both natural abundance and specific abundance of isotopes can be supported, (8) the charge fraction can be chosen from three patterns ( fixed ion fraction, energy-dependent fraction and ion fraction with charge-exchange parameters for medium-energy ion scattering [6-8]). The simulation is demonstrated, and the detail are discussed.

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# PA-49: A "third electron" model for simulation of electron yield for triggering in fine focused time-of-flight Rutherford backscattering spectrometry

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Single-event three-dimensional time-of-flight (TOF) Rutherford backscattering spectrometry (RBS) is an established tool for materials analysis with high space-resolution and detection efficiency. In case of very low ion beam currents the use of the beam chopping pulse is no longer acceptable as a start-signal for flight-time measurement. Therefore the use of secondary electron as start-signal has been proposed [1]. In this case a high electron yield (percentage of electrons which hit the electron detector) and a less fluctuation of the electron flight time are important to reduce the damege of the sample and to improve the depth resolution, respectively. A simulator has been developed, which computes flight trajectories of electrons and ions within the vacuum chamber [2]. The dependency of electron trajectories on the field distribution has been shown. Especially the sample bias as a tool to optimize electron yield and flight time has been demonstrated. Only secondary electrons emitted from the sample have been considered. However measurements have shown a maximum of electron yield at positive sample bias voltage, another maximum at negative sample bias voltage. The maximum at postive sample bias voltage only can be explained by the above described model. In contrast, at negative sample bias voltage the secondary electron is repelled from the sample and hits rather the focused ion beam (FIB) column than the electron detector (ED), thus bringing the electron yield to zero. These hits by secondary electrons occur at up to 200 eV, thus causing additional electron emission, which we call "third electrons" within this paper. The third electron model is implemented into the simulator as follows: the number of third electrons to be generated per impinging secondary electron is determined using a model from [3] third electrons are generated either as backscattered electron (energy depending on energy of impinging electron) or low energy electron (Gaussian distributed around 2 eV) Simulation-results show that the extended model also explains a second maximum of electron yield at large negative sample bias voltage.

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# PA-50: Erbium ion implantation into diamond – modelling and measurement of erbium positions in the crystal structure

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Nowadays various laser-active ion-doped materials are studied in the field of photonics and optoelectronics, mainly for their use in the optical communications systems and special laser applications. Diamond crystalline materials doped with various optically-active ions have gained attention recently. Diamond has outstanding physical and chemical properties – e.g. extremely high chemical and thermal resistance, highest hardness among materials and also a bio-compatibility because of the carbon nature of the crystal. Due to these properties in the recent years is diamond studied for the special high index contrast (HIC) waveguides as well as rare-earth doping.

In the contribution, we present a study of the erbium positions in the erbium ion-implanted diamond crystal structures. According to the crystal defect structures – and according to the current research knowledge – we have developed several erbium-doped diamond structure models and calculated geometry optimizations and overall energies of the structures. For the simulations of the structures we utilized DFT (Density Functional Theory) ab-initio methods. The DFT calculations were carried out with the aid of the CASTEP package to perform the structure optimizations, and full potential WIEN2k code for accurate calculations of total energies and crystal field parameters.

Erbium-doped diamond crystal samples were prepared by ion-implantion of  $Er^+$  ions using 190 keV implantation energy and various implantation fluences of  $1 \times 10^{14}$  ion/cm<sup>2</sup>,  $1 \times 10^{15}$  ion/cm<sup>2</sup> and  $5 \times 10^{15}$  ion/cm<sup>2</sup>. The erbium-concentration depth-profiles of erbium-implanted samples were studied by Rutherford Backscattering Spectrometry (RBS) and compared with the SRIM 2008 simulations. The structural properties of the as-implanted samples (such as relative number of disordered atoms in the implanted layers and positions of the implanted erbium ions) were characterized by RBS/channelling and fine-scans methods.

The theoretical results were compared with the experimental ones and the detailed study of the positions of erbium atoms in the diamond structure was created.

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#### PA-51: Mass-calibration of the energy axis in ToF-E ERDA and the analysis to ToF-M spectra

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Elastic recoil detection analysis (ERDA) is recognized for its potential to probe the light elements. Modern implementations of elastic recoil detection analysis make use of a multidispersive detector telescope, for example a time of flight – energy (ToF-E) detector telescope. This allows one to separate the mass and the energy of the recoil and scatter ions, and thus to determine the simultaneous and quantitative elemental depth profile of light and heavy mass elements in both light and heavy matrices. In this work, we report on the procedures that we have developed to calibrate the energy axis of ToF-E spectra. The convenience of the approach and the robustness of the procedures will be highlighted. The obtained calibration parameters allow one to transform the ToF-E spectra into calibrated ToF-M spectra, i.e. with the isotopic mass on the y-axis. From the ToF-M spectra on reference samples, we characterize the mass resolution of the set-up, and we use this information to extract (fit) for any spectrum the number of events for the various isotopes as a function of the time of flight. The latter results are conventionally used to determine the elemental depth profile. The decomposition of the contribution from various isotopes in ToF-M spectra will be illustrated with various examples relevant for the micro-electronics industry.

# PA-52: The influence of the beam charge state on the analytical calculation of RBS/ERD spectra

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Analytical codes dedicated to the analysis of Ion Beam Analysis data rely on the accuracy of both the calculations and of basic data such as scattering cross sections and stopping powers. So far, the effect of the beam charge state of the incoming beam has been disregard by general purpose analytical codes such as NDF. In fact, the codes implicitly assume that the beam is always in the equilibrium charge state, by using tabulated stopping power values e.g. from SRIM, which are in principle valid for the equilibrium charge state. The dependence of the stopping power with the charge state is ignored. This assumption is reasonable in most cases, but for high resolution studies the actual change of the charge state from the beam charge state towards equilibrium as it enters and traverses the sample must be taken into account, as it influences the shape of the observed data. In this work, we present an analytical calculation, implemented in NDF, that takes this effect into account. For elastic recoil detection analysis (ERDA), the changing charge state of the recoils can also be taken into account. We apply the calculation to the analysis of experimental high depth resolution ERDA data for various oxide

layers collected using a magnetic spectrometer.

#### PA-53: Analytical simulation of RBS spectra of nanowire samples

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Almost all, if not all, general purpose codes for analysis of Ion Beam Analysis data have been originally developed to handle laterally homogeneous samples only. This is the case of RUMP, NDF, SIMNRA, and even of the Monte Carlo code Corteo. Specific codes exist that can handle 3D samples, including simulation of the RBS spectra expected from user-input structures. General-purpose codes are, however, geared for the general case, and usually include only limited support for lateral inhomogeneity. This can be via a user-input distribution of the thickness of given layers, or via approximate models of surface and interface roughness. NDF and SIMNRA provide some support for the influence of inclusions and quantum dots on energy spectra. In this work, we show analytical simulations of samples that consist of a layer of parallel oriented nanowires on a substrate, using a model implemented in NDF. We apply the code to real samples, made of vertical ZnO nanowires on an Al<sub>2</sub>O<sub>3</sub> substrate. Two configurations of the nanowires were studied: 40 nm diameter, 4 micrometer height, 3% surface coverage; and 55 nm diameter, 1 micrometer height, 64% surface coverage. We discuss the accuracy and limits of applicability of the analysis.

### PA-54: Implementation of surface roughness algorithms in NDF

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The inclusion of the effects of roughness in analytical codes dedicated to the analysis of ion beam analysis data is a long standing problem. Many methods have been developed along the last decades to deal with specific types of roughness. The methods can be broadly classified in two groups. The first class of methods is based on an explicit summation over a given distribution of the thickness of one or more layers. The second class encompasses a wide variety of different approximations, usually developed specifically for a given type of roughness, and with varying ranges of applicability and accuracy. All those methods ignored the effect of secondary crossings due to surface asperities, that is, when the beam exits and re-enters the sample, which is particularly relevant in the presence of severe roughness, and can lead to strong changes in the observed data. In 2008, Molodtsov, Gurbich, and Jeynes (MGJ) presented an algorithm that effectively solved this case in an elegant and efficient way, by parameterising a large set of Monte Carlo calculations made for different rough surfaces and detection angles. We present in this work an implementation of the MGJ algorithm in the well-known code NDF. The details of the implementation, which is non-trivial for the general case where the beam has a non-normal direction of both incidence and detection, are given. The calculation is compatible with the other surface and interface roughness algorithms implemented in NDF, which include both analytical algorithms and generalised thickness distributions of any number of layers. The actual algorithm has been developed independently of any given code, which is possible because the calculation is made a posteriori, as extra convolutions on the individual elemental signals calculated without considering the effect of secondary crossings. The algorithm is distributed as open source, in order to facilitate the future inclusion in other codes as well.

### PA-55: Use of the DataFurnace code for high accuracy IBA

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The general-purpose code DataFurnace for unfolding elemental depth profiles from RBS spectra using the simulated annealing algorithm was first published in 1997 [1] and immediately used systematically in a wide variety of materials systems [2]. Major improvements continued: in algorithms for handling high resolution data, moderate roughness, elastic (non-Rutherford) backscattering (EBS) and – importantly – the introduction of an integrated module for PIXE [3]. The dramatic impact on the power of IBA made available by treating EBS and PIXE (and other IBA) data synergistically was reviewed in 2012 [4]. The IAEA also sponsored an Intercomparison of many IBA codes, including DataFurnace [5]. DataFurnace is continuing to be intensively developed, both the computation engine (NDF) and its GUI (WINDF). Severe roughness where the entrance or exit beam re-enters the sample through the asperities can now be treated, using Gurbich's algorithm [6]. At high resolution, resonant nuclear profiling (NRP) can detect the Lewis effect: except for the highest depth resolution, NDF can approximate this straggling with a gamma function [7]. We have long been able to impose chemical priors on the data: we have now implemented the imposition of error function depth profiles on molecular diffusion, together with the determination of robust parameter uncertainties by Bayesian methods [8]. Complementary data from SIMS can now be included in quantitative analyses [9]. Double scattering and other effects strongly affect forward recoil data and can now be taken into account [10]. The quality assurance programme for an implantation lab handles large quantities of RBS/EBS data routinely at very high accuracy [11]: this involves determining the electronic gain at 0.1% yielding RBS with 0.3% precision and 1% absolute accuracy, and beam energy determination at 0.06% [12]; these measurements are currently possible only using the sophisticated fitting and data handling facilities provided by DataFurnace.

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# PA-56: Structural characterization of ion-beam modified materials by GISAXS: models and software package GisaxStudio-iBeaM

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We present a new approach for the structural analysis of ion beam modified materials by GISAXS (grazing incidence small angle x-ray scattering). Passage of ion through material can induce its modification along ion trajectory, called ion track. These modifications often result in the formation of non-continuous nano-objects along ion track. We have developed several models describing different arrangement types of the formed nano-objects. They enable the determination of their structure and arrangement including mean shape, size, separation and number of objects along the track, as well as their statistical distributions. The efficiency of the method is demonstrated by the analysis of ion tracks produced in several systems.

The developed models are incorporated in a new program GisaxStudio-iBeaM for the analysis of GISAXS intensity distributions measured on ion-beam modified materials. GisaxStudio is a modular, multi-platform program for GISAXS analysis of various nanostructured materials. It is written in Java programming language, featuring a graphical user interface, built-in optimization algorithms and visualization. It stores all data in the relational database which facilitates data exchange and reproducibility. It is free for non-commercial use and can be downloaded from the http://homer.zpr.fer.hr/gisaxstudio

#### PA-57: Dechanneling function dependence on the proton-silicon interatomic potential

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This work reports on the dependence of the dechanneling function [1,2] on the proton-silicon interatomic potential, for proton energies between 1 and 10 MeV and for all the major widely used silicon crystal channels, namely, for <100>, <110> and <111>. The dechanneling function has been calculated taking into account the ZBL, Molière and the rainbow proton-silicon interatomic potentials [3]. The obtained dechanneling function is shown to be of sigmoidal type, characterized in all cases by two critical parameters, the dechanneling range and rate. Moreover, pronounced differences between the corresponding dechanneling functions have been observed, which can be exclusively attributed to the choice of the interatomic potential. Therefore, the experimental determination of the dechanneling range and rate from a carefully designed RBS/C channeling experiment, which would exploit the p+<sup>28</sup>Si elastic scattering resonances at 1.67 MeV and 2.09 MeV [1], could in principle be used for the investigation of the accuracy of the proton-silicon interatomic potential.

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### PA-58: Correlative microscopy based on SIMS for high-resolution high-sensitivity nanoanalytics

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The ever increasing complexity of devices and the continuously shrinking dimensions in materials research make characterisation tools and techniques face new challenges and future trends need to be anticipated. For investigations at nanoscale, nano-analytical techniques providing both excellent spatial resolution and high-sensitivity chemical information are of utmost importance in materials and life sciences. Electron microscopy, Helium ion microscopy (HIM) and scanning probe microscopy (SPM) are commonly used for high-resolution imaging. However, these techniques have all the same drawback of no or very limited chemical and

elemental information. In electron microscopy, elemental information can be obtained by using techniques like EELS or EDS, but the sensitivity is limited. Moreover they do not permit to distinguish between isotopes, which is a major limitation due to increasing isotopic labelling in life sciences, and have limited sensitivity in the low mass range. By contrast, secondary ion mass spectrometry (SIMS) is an extremely powerful technique for surface analysis owing in particular to its excellent sensitivity, high dynamic range, very high mass resolution and the ability to differentiate between isotopes. In order to get elemental information with a highest sensitivity and highest lateral resolution, we have investigated the feasibility of combining SIMS with transmission electron microscopy (TEM), HIM and SPM and developed the three corresponding prototype instruments. Good detection limits when probing small voxel sizes in ion imaging applications can only be obtained when ionization probabilities of the sputtered matter are maximised. Ga primary ion species (in case of the TEM - SIMS instrument) or He and Ne ions (HIM instrument) produce secondary ion yields which are much lower than those found in conventional SIMS. By using reactive gas flooding, i.e. O<sub>2</sub> flooding for positive secondary ions and Cs for negative secondary ions, secondary ions yields can be increased by up to 4 orders of magnitude, leading to detection limits in the range of  $10^{-3}$  to  $10^{-6}$  for a resolutions between 10 nm and 100 nm. First results are very encouraging and the prospects of performing SIMS in combination with TEM, HIM and SPM are very interesting. The combination of high-resolution microscopy and high-sensitivity chemical mapping on a single instrument represents a new level of correlative microscopy. In this paper we are going to give an overview of the instruments and their performance, and we are going to present the newest examples of applications.

# PA-59: X-ray production cross sections for K-, L- and M-shell by 11.2, 16.8 and 22.4 MeV nitrogen

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This paper considers the X-ray production cross section under excitation of K-, L- and M-shells of target atoms in the range of mass from Ti to Bi by <sup>14</sup>N ions at 0.8, 1.2 and 1.6 MeV/nucleon energies. The approach used is based on the calculation of X-ray production cross sections through the cross section of Rutherford backscattering, which can be calculated with high accuracy using the Rutherford formula. The experimentally obtained data are compared with the theoretical values calculated according to the ECPSSR and PWBA approaches by means of the ISICS code. The possible reasons for discrepancies between theoretical and experimental data are discussed. This work is a continuation of the activities started in 2014.

#### PA-60: Investigation on MeV C-ion-beam PIXE Cross Sections

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In exploration of a new ion beam analysis technique, i.e. MeV heavy ion beam particle-induced x-ray emission (PIXE) analysis of materials, we used our home-available 1.7-Mtandem accelerator and beam line to investigate MeV carbon (C)-ion-beam PIXE cross sections for setting up a database for further development and applications of the technique. Based on the capability of the beam line, C-ion beams at 0.8, 1 and 1.2 MeV were applied for PIXE analysis, which might be the first time for ion beam physicists to work in this energy range of heavy C-ion beams for PIXE. Samples of metals such as Al, Cu, Fe, Au, Zn, etc. and semiconductor Si were analyzed at a beam current about a few nA for 1,000 seconds per spectrum. The measured spectra were compared with spectra of standard proton PIXE of beam energy at 2 MeV as well as about 1 MeV. Results showed that the yields of 1-MeV C-ion PIXE were comparable with those of 2-MeV proton PIXE, while the former was dramatically higher than those of 1-MeV proton PIXE, which were actually negligible. This demonstrated the superior sensitivity and high cross sections of 1-MeV C-ion PIXE. The experimental results were tried to be interpreted by theoretical cross-section studies, however many confusions were involved. For the beam energy dependence of MeV C-ion PIXE, it was strangely found that, for example, the yield of 1.2-MeV PIXE was lower than that of 1-MeV PIXE, which could be hardly explained by theory. It would be said in a brief summary that a number of interesting results were found and a number of unknowns remained for further studies.

### PA-61: Proton elastic scattering cross-section on helium from 0.6 to 3.0 MeV at laboratory angles between 110 and 165 degrees

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The differential  ${}^{4}$ He(p,p) ${}^{4}$ He reaction cross-section was determined at laboratory angles between 110-165° for proton energies between 0.6 and 3.0 MeV using a solid thin target of porous silicon containing helium. The currently measured cross-section data are compared with the available literature values. The uncertainty in the present cross-section data is about 8.2%. The experimental cross sections have been analyzed within the R-matrix formalism, where its parameters (energy and width of the resonant states) have been adjusted in order to reproduce the experimental data.

# PA-62: Determination of ${}^{9}Be(p,p_{0}){}^{9}Be$ , ${}^{9}Be(p,d_{0}){}^{8}Be$ and ${}^{9}Be(p,\alpha_{0}){}^{6}Li$ cross sections at 150° in the energy range 0.5–2.35 MeV

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Beryllium and tungsten have been chosen as the new "plasma facing materials" for fusion research. The interaction between the plasma and the wall materials in a fusion reactor is the key factor in determining the useful life of the wall components. This interaction includes the erosion and redeposition of material in the plasma-facing wall, accompanied with the formation of alloys from the different materials present. These processes are critical for the retention of hydrogen isotopes by the wall materials. Determining the amount and the depth profile of Be and other elements is crucial for understanding those phenomena making the knowledge of its cross sections essential. In this work we determined the <sup>9</sup>Be(p,p<sub>0</sub>)<sup>9</sup>Be, <sup>9</sup>Be(p,d<sub>0</sub>)<sup>8</sup>Be and <sup>9</sup>Be(p,\alpha<sub>0</sub>)<sup>6</sup>Li cross sections in the energy range 0.5 - 2.35 MeV, at an angle of 150°. The data presented, besides being directly useful to experimentalists, can also be used to assist a full evaluation of Be migration patterns and surface analysis of the JET ITER-like wall and other fusion reactors containing Be in the first wall.

# PA-63: Measurement of proton induced thick target $\gamma$ -ray yields on B, N, Na, Al and Si from 2.5 to 4.1 MeV

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Thick target yields for proton induced  $\gamma$ -ray emission (PIGE) on low-Z nuclei, namely B, N, Na, Al and Si, were measured for proton energies from 2.5 to 4.1 MeV, at emission angles of 90°, 45° and 0°, at the 3 MV Tandetron laboratory of INFN-LABEC in Florence.

The studied reactions were:

•  ${}^{10}B(p,\alpha'\gamma)^7Be$  (Ey = 429 keV),  ${}^{10}B(p,p'\gamma)^{10}B$  (Ey = 718 keV) and  ${}^{11}B(p,p'\gamma)^{11}B$  (Ey = 2125 keV) for boron;

- ${}^{14}N(p,p'\gamma){}^{14}N$  (Ey = 2313 keV) for nitrogen;
- $^{23}$ Na(p,p' $\gamma$ ) $^{23}$ Na (E $\gamma$  = 441 and 1636 keV) and  $^{23}$ Na(p, $\alpha'\gamma$ ) $^{20}$ Ne (E $\gamma$  = 1634 keV) for sodium;

•  ${}^{27}Al(p,p'\gamma){}^{27}Al$  (Ey = 844 and 1014 keV) and  ${}^{27}Al(p,\alpha'\gamma){}^{24}Mg$  (Ey = 1369 keV) for aluminum;

•  ${}^{28}\text{Si}(p,p'\gamma){}^{28}\text{Si}(E\gamma = 1779 \text{ keV}) \text{ and } {}^{29}\text{Si}(p,p'\gamma){}^{29}\text{Si}(E\gamma = 1273 \text{ keV}) \text{ for silicon.}$
A pressed pellet of NaNO<sub>3</sub> and thick samples of BN,  $Si_3N_4$  and Al were used as targets; composition and stoichiometry of the thick targets were checked by simultaneous elastic backscattering spectroscopy (EBS) measurements with protons. The  $\gamma$ -rays were detected by three HPGe detectors with nominal relative efficiency of 50% for the detector placed at 90°, and 25% for both detectors placed at 45° and 0°. The overall uncertainty on the thick target PIGE yields was estimated to be between 8% and 20%, being 7.5% the systematic normalization uncertainty. Comparison with the few existing data sets on thick target PIGE yields in this proton energy range [1-3] will be shown.

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### PA-64: Extension of the ${}^{19}F(p,p_0)$ differential cross section evaluation up to $E_p=2.3$ MeV

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Elastic Backscattering Spectroscopy (EBS) is one of the most suitable IBA techniques for light element detection and depth profiling. The implementation of the technique depends mainly on the availability and accuracy of the existing corresponding differential cross-section data. A compilation of all the available experimental cross-section values is found in IBANDL, along with evaluated data provided by SigmaCalc. Concerning fluorine analytical studies, the corresponding experimental data are quite inadequate and discrepant, proving the significance of the evaluated values, which provide the most reliable data being produced by the incorporation of the available experimental cross sections within a unified theoretical approach. The present work contributes in this field by reproducing and extending the corresponding evaluation for  $^{19}F(p,p_0)$ , which ranges up to 1730 keV, to proton energies at 2250 keV, using the AZURE code. The performed R-matrix calculations involved the simultaneous analysis of several input datasets, as well as spectroscopic information concerning the formed compound nucleus <sup>20</sup>Ne, while valuable feedback information was provided by proton benchmarking spectra on  $ZnF_2$  taken at  $E_p$ =1730 and 2250 keV for the fine tuning of the parameters used. The problem of the  ${}^{19}F(p,p')$  and  ${}^{19}F(p,\alpha x)$  contributions in the obtained thick target yield spectra is also discussed.

## PA-65: Experimental determination of cross section data for the D(<sup>3</sup>He,p)<sup>4</sup>He nuclear reaction from 0.25 to 6 MeV

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The nuclear reaction  $D(^{3}He,p)^{4}He$  is commonly used to determine the depth profile of deuterium in solids or the total content of deuterium atoms in a sample. The cross section of this reaction has a broad resonance region around 630 keV and a non-resonant region where the cross section is not negligible. Precise differential cross section data are required for depth profiling of D, especially for depth profiling up to large depths with high incident energies.

Most of the existing data in literature are limited to a maximum energy of 2.5 MeV <sup>3</sup>He. Due to kinematic considerations we use an annular detector for proton detection under a reaction angle of 175°. To probe large depths energies of above 5 MeV are needed. In this work the cross section was determined in the energy range from 0.25 MeV to 5.6 MeV for laboratory angles of 135°, 144.5°, and 175°. A plasma deposited amorphous deuterated 50 nm thin hydrocarbon (a-C:D) film on flat Si plates was used as deuterium target. A thin Au film was evaporated on the a-C:D film and was used to determine the dose of <sup>3</sup>He ions via RBS. To minimize the influence of deuterium depletion due to ion-bombardment induced desorption different spots on the target were used for each energy. As the number of detected protons is proportional to the cross section, the relative shape of the cross section can be derived from the data were fitted in the energy range from 0.5 to 1.2 MeV to the total cross section of Möller and Besenbacher [1] assuming angular independence in the center-of-mass system. The uncertainty of the determined cross sections at 135°, 144.5° and 175° resulting from this procedure is between 5.2% and 6.6%.

The experimental data of the differential cross section are compared to theoretical predictions given in the literature [2, 3]. For energies below 1 MeV the theoretical values deviate significantly from experimental values. For higher energies the theoretical predictions agree with the experimental data very well. The data for the 135° angle were also compared with experimental results from [4] and agree within their error bars.

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- [4] V. Kh. Alimov et al. Nucl. Instr. Meth. B234 (2005) 169

### PA-66: Measurement and evaluation of proton elastic scattering cross section on S from 3.4 to 4.6 MeV

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Differential cross sections for the elastic scattering of proton on natural sulphur have been determined for proton energies between 3.4 and 4.6 MeV, using a variable energy step from 1 to 20 keV , and for scattering angles of 170°, 160°, 150°, 140° and 120°. The measurements were carried out at the Tandem Accelerator Laboratory of the Institute of Nuclear and Particle Physics, National Centre of Scientific Research (N.C.S.R.) "Demokritos", using the installed 5.5 MV TN11 Tandem accelerator. A TiS<sub>2</sub> thin target (1 keV energy loss for 3 MeV protons) evaporated on a self-supporting C thin film and covered by a Ta thin layer for normalization purposes was used. Absolute cross sections are calculated with a method not dependent on the absolute values of collected beam charge and detector solid angle, normalizing to the Rutherford cross section of protons on Ta; the overall accuracy is estimated to be around 5% at all the beam energies and scattering angles. To validate the obtained results, several benchmarking measurements were performed, using a thick MoS<sub>2</sub> target. The experimental data are compared to data from the literature, when available, and similarities and discrepancies are presented and analyzed. The parametrization of the present experimental data in the framework of nuclear physics models allows to extend the evaluated elastic scattering cross section of proton on natural sulphur from the present value of 3.6 MeV up to 4.6 MeV.

# PA-67: Differential cross section measurement of ${}^{16}O(d,p_{0,1})$ reactions at energies and angels relevant to NRA

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Nuclear Reaction Analysis (NRA) and more specifically d-NRA is a suitable technique for <sup>16</sup>O detection on or near surface of a compound containing heavy elements. The main problem of d-NRA is the limited number of NRA differential cross section dataset and their discrepancy [1].

In the present work the <sup>16</sup>O(d,p<sub>0,1</sub>) reactions were measured for  $E_{d,lab} = 700-1800$  keV at scattering angles 90°, 135°, 150° and 165° suitable for NRA. The experiments were performed using the deuteron beam of 3 MeV Van de Graaff accelerator of Nuclear Science and Technology Research Institute (NSTRI). Deuteron beam of 1×1.5 mm<sup>2</sup> with energy resolution of 1 keV was irradiated on the target with the current of 15 ~ 20 nA. A thin layer of Ta<sub>2</sub>O<sub>5</sub> 93 µg/cm<sup>2</sup> obtained by anodizing thick tantalum substrate was employed for reaction cross section measurements [2].The incident beam was accurately measured by deuteron elastic scattering of Ta substrate at the scattering angle of 165°. The detection system consisted of five 300 micron thick surface barrier detectors covered with 30 µm-thick Kapton foil in order to suppress elastically scattered deuterons, except for the one used for beam monitoring.

Differential cross sections data were collected with an energy step of ~10 keV. However, detailed measurements were carried out with an energy step of ~2 keV around resonances. The overall error in the differential cross sections measurements varied around 5-10% in most cases. The results of this work was discussed and compared with the published data [3].

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# PA-68: Differential cross section measurement of <sup>28</sup>Si $(p,p_1\gamma)^{28}$ Si and <sup>29</sup>Si $(p,p_1\gamma)^{29}$ Si reactions in the proton energy range of 2-3 MeV

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Particle induced gamma-ray emission (PIGE) with proton beam is a powerful analytical technique which could be used to determine the concentration of light elements in a wide variety of samples. To apply PIGE method as an analytical technique, accurate cross section data are required.

Recently the International Atomic Energy Agency (IAEA) carried out a coordinated research program to develop a reference database for PIGE spectroscopy. Van de Graaff lab in Tehran, participated in this project by PIGE cross section measurement of a number of isotopes.

The purpose of this research work is to provide reliable differential cross section data for gamma-rays originated from the  ${}^{28}\text{Si}(p,p_1\gamma){}^{28}\text{Si}$  (E $\gamma$ =1779keV) and from the  ${}^{29}\text{Si}(p,p_1\gamma){}^{29}\text{Si}$  (E $\gamma$ =1273keV) nuclear reactions in the energy range of about 2-3 MeV.

The experimental work was carried out in the 45 degree beam-line of the Van de Graaff lab of Nuclear Science and Technology Research Institute in Tehran. The 3 MV single-ended Van de Graaff machine provides the intense incident beams required in these measurements. The thin Si target was prepared by evaporating SiO on a backing of self-support Ag film. This type of target enables accurate measurement of the incident beam by collecting the BS particles. The reaction products, gamma-rays and protons, were collected with a HPGe and ion implanted Si detectors placed at the scattering angles of 90 degree and 165 degree, respectively. This

experimental setup has the advantage of differential cross section measurement independent on the absolute number of incident protons. In this work, measured differential cross section of <sup>28</sup>Si  $(p,p_1\gamma)^{28}$ Si and <sup>29</sup>Si $(p,p_1\gamma)^{29}$ Si reactions were presented, discussed and compared with the published data [1,2].

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#### PA-69: Screened cross sections for heavy ion IBA calculated from ab initio pair potentials

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The cross section values used in the analysis of ion beam measurements determine the accuracy of the resulting absolute concentrations. Traditionally non-Rutherford cross sections have been considered for high-energy light ions such as protons above ~1 MeV/amu. At lowenergy, different screening corrections to Rutherford cross sections, such as Andersen screening [1], have been presented, which provides a good approximation of the cross section based on Lenz-Jensen screened potential, when screening effects are above 85%. In recent years low energy heavy-ion elastic recoil detection (ERD) measurements have become common due to the improving depth resolution and increasing recoiling yield as the ion beam energy decreases, and the progress in Monte Carlo (MC) simulation techniques in the analysis of multiple scattering effects. In low energy heavy ion scattering processes, screening effect in atomic pair-potentials become important and thus the resulting cross sections may differ from pure Coulomb scattering by several percent. The ZBL universal potential [2] is known to be reasonably accurate as it is based on a fit, with a few percent precision, over several pair potential computed by ab initio Hartree-Fock methods. However, current computers power allow direct calculation of HF pair-potentials for all possible ion-target (Z<sub>1</sub>, Z<sub>2</sub>) combinations, solving the cross section values from each of these potentials, and using directly these cross section for IBA spectrum simulations. In this work, we present the results of potential calculations based on HF calculations using Gamess code [3]. We tabulate the screened potentials for all  $Z_1$ - $Z_2$  combinations of stable elements and compare the results to the existing analytical screening models. We also present open source software to use these potentials to calculate the absolute values of cross sections for any Z<sub>1</sub>-Z<sub>2</sub>-energy combination. The calculated cross sections are then compared to Rutherford values and some of common corrections to Rutherford cross sections. Finally, we also present examples of MC simulations demonstrating the effect of using HF-based potentials compared to other cross section values in ERD measurements.

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(1993) 1347

### PA-70: Ionization cross sections for selected elements by 35-55 MeV <sup>79</sup>Br and <sup>127</sup>I ions

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The emission of x-rays as a result of the atomic excitation with protons and alpha-particles has been widely studied during past decades. When heavier ions are used as bombarding particles, the processes involved are more complex and their effects have not been fully described neither by theories nor experiments. However, there is an increasing interest in the application of heavy ions for analysis with particle induced x-ray emission (PIXE), as it can be used simultaneously with other heavy ion based techniques (ERDA, MeV-SIMS). In addition, the possibility of selective excitation and higher ionization cross sections with heavy ions could improve the sensitivity of PIXE. In this work, ionization cross sections and x-ray yields for thick targets of Ti, Cu, Ta and W by 35-55 MeV <sup>79</sup>Br and <sup>127</sup>I ions have been determined. Simultaneous ERDA measurements were used to determine the exact composition of the sample materials and to determine accurate irradiation fluences. For beam current monitoring and irradiation fluence measurement, a method based on the utilization of beam profilometer signals, was also used. Determined ionization cross sections are compared with the predictions of the ECPSSR theory.

### PA-71: PIGE related differential cross section measurements of the ${}^{25}Mg(p,p'\gamma){}^{25}Mg$ reaction

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NRA, and especially Particle-Induced Gamma ray Emission (PIGE) is a well known and widely used method, usually in conjunction with PIXE, due to its enhanced detection sensitivity for many nuclides as well as its high isotopic selectivity. While a lot of effort has recently been devoted to the precise determination of reliable cross sections for use with charged-particle NRA, there is a lack of data in literature regarding PIGE. The use of differential cross section data for PIGE analysis along with a suitable code, instead of thin or thick target yields, will greatly enhance the use of the technique as it will lead to samples quantification without utilizing reference materials. Towards this goal, in the present work we report on the differential cross section measurement of the <sup>25</sup>Mg(p,p' $\gamma$ )<sup>25</sup>Mg reaction, in the energy range between 2400 to 4500 keV and at two detection angles (55° and 90°). The obtained results are validated through a rigorous benchmarking procedure and compared with the existing ones

from literature.

### PA-72: Study of the ${}^{11}B(p, p'\gamma){}^{11}B$ reaction for PIGE applications

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Boron is a highly regarded technological element and has numerous applications in various fields. It is widely used in the semiconductor industry as a dopant for Si and Ge substrates and it is also an essential ingredient of hard coatings on the walls of thermonuclear plants. Thus, the accurate quantitative determination of boron depth profiles in heavy and light matrices or substrates is of great importance. One of the Ion Beam Analysis (IBA) methods, that can yield accurate results and has been used successfully in the past, is Nuclear Reaction Analysis (NRA) and especially Particle Induced Gamma ray Emission (PIGE).

In the present work, differential cross sections were measured at 6 angles (0°, 15°, 90°, 105°, 150° and 165°) and at proton energies from 2.5 to 5.0 MeV (20 keV step) for the <sup>11</sup>B(p,  $p'\gamma$ )<sup>11</sup>B reaction. The  $\gamma$ -ray emitted (2125 keV) was detected by three HPGe detectors placed on a motorized turntable. The results of the present work are compared with existing ones from literature and possible explanations for the observed differences are discussed.

# PA-73: Si, Fe and Cu X-ray production cross-sections measurements for alpha particles between 6 and 12 MeV

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A new transport beam line has been developed at our CGR-520 MeV cyclotron for ion beam techniques. This facility allows us to produce proton and alpha particle beams with energies up to 20 MeV. A vacuum chamber dedicated to X-ray production and Non-Rutherford cross-section measurements has been recently constructed [1]. Measurements of the X-ray production cross-sections in the 6–12 MeV energy range have started using alpha particle beams on light element targets [2]. These experiments contribute to the filling a serious lack of experimental values for alpha particles of this particular energy range in databases. We are reporting here X-ray cross section measurements for Silicium, Iron and Copper. Experimental data are in good agreement with theroretical ECPSSR values.

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# PA-74: Measurements of proton induced gamma-ray emission cross sections on Na and Al in the energy range from 1.90 to 3.05 MeV

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Particle induced gamma-ray emission (PIGE) has proven to be very powerful non-destructive method for determination of light element concentrations, which are usually underestimated by proton induced x-ray emission (PIXE) analysis, due to self-absorption of low energy x-rays inside the sample. However, application of PIGE technique for quantitative analysis requires either measurements of thick target yields for systems with similar matrices or systematic cross section data of the prompt  $\gamma$ -reactions, which are known to show strong energy dependence.

In this work we present differential cross sections for  $\gamma$ -ray emission from the reactions  ${}^{23}$ Na(p,p' $\gamma$ ) ${}^{23}$ Na (E $_{\gamma}$ =440 and 1636 keV),  ${}^{23}$ Na(p, $\alpha'\gamma$ ) ${}^{20}$ Ne (E $_{\gamma}$ =1634 keV),  ${}^{27}$ Al(p,p' $\gamma$ ) ${}^{27}$ Al (E $_{\gamma}$ =844 and 1014 keV) and  ${}^{27}$ Al(p, $\alpha'\gamma$ ) ${}^{24}$ Mg (E $_{\gamma}$ =1369 keV). Differential cross sections were measured for proton energies from 1.90 to 3.05 MeV with a 15 keV step and beam energy resolution of 0.06%. Thin reference standards, 53.7 µg/cm<sup>2</sup> of NaCl and 55 µg/cm<sup>2</sup> of Al deposited on thin Mylar foil with additionally evaporated 4 nm Au layer were used as targets. The  $\gamma$ -rays were detected by a 20% relative efficiency HPGe detector placed at an angle of 135° with respect to the beam direction. For the normalization purposes and to avoid absolute charge measurement, the backscattered protons were collected simultaneously using silicon surface barrier detector placed at the backscattering angle of 165°. Obtained cross sections were compared with the previously measured data available from the literature.

### PA-75: Gamma-producing nuclear reaction cross sections in Al and Mg

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During the last sixty years, analytical techniques based on accelerated ion beams have established a firm ground of reference techniques for a multitude of problems pertaining to various domains, ranging from material science and technology to geology, archaeometry, biomedical sciences, art and environment.

An effort has been made to measure and compile necessary parameters to provide these

techniques with quantitative potentialities. One example is PIXE – Proton Induced X-Ray Emission; a strong commitment to measure and systematise X-ray producing cross sections led to the development of software to calculate concentrations from X-ray yields, freeing the technique from the dependence on comparative standards. Now, PIXE stands as a recognized technique for bulk analysis of homogeneous samples for elements heavier than Magnesium.

Although complementary to PIXE in the sense that it covers light element analysis with the same kind of experimental facilities, PIGE – Proton Induced Gamma-Ray Emission has not followed the same steps. On one side, the negligible absorption of radiation by the sample allows for the use of standards in a straightforward reasonable approximation; on the other side, contrary to PIXE, nuclear reaction excitation functions are rarely smooth, displaying narrow and broad resonances, which hindered the motivation to develop software for concentration calculations based on the cross sections.

In the last few years, we have shown that, if cross sections are available in numerical values at energy steps close enough to define in detail the resonances, trivial integration leads to elemental concentrations. This methodology becomes, as for the case of PIXE, standard free. The code we have developed, the ERYA code, integrates the relevant nuclear reaction cross sections along the depth of the samples.

Regarding the relevant cross sections, the experimental results are scarce and results of different authors show large discrepancies. An effort has been made within an IAEA concerted project to compile, evaluate and measure relevant cross sections.

Regarding the importance of PIGE analysis of Mg and Al, we point out that due to sample self absorption and detector window absorption, PIXE analysis of Mg and Al may have large uncertainties, being PIGE analysis an alternative solution. The sensitivity and the multielemental character of PIGE bulk analysis generally increase with energy, fact that motivated the extension of our work to a higher energy range that the one usually used for PIXE.

In this work, we have measured cross sections of the reactions  ${}^{25}Mg(p,\alpha\gamma){}^{22}Na$  ( $\gamma_1 - 585$  keV) and  ${}^{27}Al(p,p'\gamma){}^{27}Al$  ( $\gamma_1 - 848$  keV and  $\gamma_2 - 1014$  keV) at the proton energy range 1.5 to 4.0 MeV.

### PA-76: Measurements of K-X Rays cross sections production for high energy PIXE

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ARRONAX, acronym for «Accelerator for Research in Radiochemistry and Oncology at Nantes Atlantique», is a high energy cyclotron. It is characterized by the acceleration of several types of particle beams: 68 MeV alpha, 15-35 MeV deuterons and 30-68 MeV protons. A platform was implemented on ARRONAX to perform non-destructive materials analysis with X and gamma rays emission (PIXE-PIGE). A proper selection of the projectile type and beam energy allow us to analyze heavy and light elements in thin and thick samples. It is well known that PIXE is used at

low energies (<5 MeV) and on thin samples (<100  $\mu$ m) in different fields of applications as described by Z. Smit [1]. PIXE provide accurate analysis because the experimental cross section of X-ray production is known at low energies [3, 2] for different elements and is in agreement with the theoretical model ECPSSR. To take advantage of high-energy beams for analyzing thick samples, we need to determine the cross section production of X-rays as few experimental measurements are done in this case [4]. Therefore at Cyclotron ARRONAX, we started to make measurements of K X-ray cross sections production for various elements to complement the databases at high energy. The first experiments have been conducted to measure these cross sections for titanium, copper, silver and gold with protons energy between 34 and 68 MeV. During this talk, I will start by a review on the theoretical model ECPSSR at high energy, and then I will describe the experience we performed (beam, detector, target ...) for the measurements of cross sections followed by a comparison between the ECPSSR model and experimental measurements.

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## PA-77: In-situ luminescence analysis and imaging of G2000 glass scintillator fabricated for real-time measurement of heavy ion microbeam

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A real-time position detection of heavy ions from AVF cyclotron had been developed using high-efficient scintillators, which is designed to replace the position detection scheme using plastic nuclear track detectors [1]. The system requires the scintillators to have a transparency for the luminescence caused by ion impact and yet the scintillator has strong radiation resistivity during intense radiation irradiations [2]. On the other hand, it was reported that a G2000 and G9 glasses has strong crystal structure and the possibility of the peak-wavelength control by changing species and concentration of dopant impurities [3]. In this study, the material G2000 and G9 glass scintillator with doped Tb was analyzed at different ion microbeam irradiation conditions. Ion Luminescence (IL) spectrum was obtained at 3-MeV proton irradiation [4] and then 2-dimensional images of 260-MeV-Ne microbeam from cyclotron at JAEA/Takasaki was visualized using electron multiplying (EM)-CCD camera [2]. IL spectra of the G2000 scintillator had four peaks which emerged at 489, 542, 586 and 623 nm. Each of peaks in spectrum was attributed to inter-level transitions between the electronic

energy levels of Tb<sup>3+</sup>. Then, decay of these luminescent peaks was evaluated by continuous monitoring of IL under proton microbeam irradiation. Higher resistivity for continuous focused microbeam irradiation was observed from the G9 in each peak of the IL compared to the G2000. On the other hand, luminescence intensity of G2000 at low flux irradiation was higher than of G2000. Also 2-dimensional IL imaging was obtained from the G2000 for beam irradiation. Minimum irradiation rate of the heavy ions were approximately 3~8 ions/s, which were confirmed by silicon charged particle detector in between the irradiation. These results indicate that sensitivity of G2000 glass scintillator is sufficient for real time position detection of swift heavy ions generated from AVF cyclotron.

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## PA-78: Comparative study of radiation-induced damage in magnesium aluminate spinel by means of IL, CL and RBS/C techniques

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The development of new nuclear fuel requires the solution of numerous problems, one of them being related to the quantitative analysis of radiation disorder in materials subjected to the low and high energy ions. The problem of information about damage build-up, intensively studied for single crystals by the Rutherford Backscattering/channeling (RBS/C) technique, poses many difficulties for polycrystalline materials. It is especially a serious practical problem, as most of the investigated materials are single crystals, while these are the polycrystals which find the practical application in construction of nuclear reactors. The present paper is devoted to the use of luminescence techniques as an experimental method of quantitative analysis of the radiation damage in polycrystalline materials. The studied case is one of the inert matrix fuel candidates, MgAl<sub>2</sub>O<sub>4</sub>. The use of luminescence techniques is an interesting option, as they may be applied to both, single and polycrystalline solids, are non-destructive, fast and can be easily implemented in-situ. A comparative study of damage accumulation in magnesium aluminate spinel (MgAl<sub>2</sub>O<sub>4</sub>) has been conducted using ionoluminescence (IL), cathodoluminescence (CL) and RBS/C techniques. MgAl<sub>2</sub>O<sub>4</sub> single crystal samples (commercially available) and polycrystalline samples obtained by the hot-pressing (Astro, Thermal Technology) of

magnesium aluminate nanopowder were irradiated with 320 keV Ar<sup>+</sup> ions at fluencies ranging from  $10^{12}$  to  $2 \times 10^{16}$  cm<sup>-2</sup> in order to create various levels of radiation damage. RBS/C measurements provided quantitative data about damage concentration in the samples. These values were then compared to the luminescence measurements. The results obtained by IL and RBS/C methods demonstrate a two-step character of damage build-up process. The CL data analysis points to the three-step damage accumulation mechanism involving the first defect transformation at fluencies of about  $10^{13}$  cm<sup>-2</sup> and second at about  $10^{15}$  cm<sup>-2</sup>. The rate of changes resulting from the formation of non-luminescent recombination centers is clearly non-linear, and cannot be described in terms of continuous accumulation of point defects. Both, IL and CL techniques, appear as new, complementary tools bringing new possibilities in the damage accumulation studies in single- and polycrystalline materials.

#### PA-79: Erbium ion implantation into various crystallographic cuts of ZnO single crystal

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The ion implantation technique is one of the ways to prepare laser active ion-doped optical layers. Erbium ions (Er<sup>3+</sup>) are well-known for their use in telecommunication systems due to the  $Er^{3+}$  intra-4f emission which corresponds to the  $4I_{13/2} \rightarrow 4I_{15/2}$  transition. In this contribution we report on the erbium ion implantation into different crystallographic cuts (<0001>, <11-20>, <10-10>) of zinc oxide (ZnO). ZnO wafers were implanted with  $Er^+$  ions using 190 keV energy with fluences of  $1.0 \times 10^{16}$  cm<sup>-2</sup> and  $5.0 \times 10^{16}$  cm<sup>-2</sup>. We focused on a study of compositional and structural changes occurring after the ion implantation process. Post-implantation annealing at 1000 °C in oxygen atmosphere was also done. Chemical compositions and erbium concentration depth profiles of the prepared samples were studied by Rutherford Backscattering spectrometry (RBS) and compared with the SRIM 2012 simulations. Structural properties of the prepared samples were characterized by RBS/channeling. Luminescence spectra of the doped samples were measured in the region of 1400-1600 nm. Using RBS/channeling method it was found that the largest amount of Er (about 83%) was located in the substitutional positions of Zn after the ion implantation process. The amount of Er in the substitutional positions decreased significantly after the post-implantation annealing. The penetration and diffusion behaviour of the erbium ions in ZnO crystal during ion-implantation and annealing processes differs greatly in comparison with the Er:LiNbO<sub>3</sub> and Er:Al<sub>2</sub>O<sub>3</sub> samples. All prepared samples exhibited luminescence emission around 1.5 µm. On the contrary to mentioned crystals The post-implantation annealing caused the decrease of the intensity of luminescence at 1530 nm in ZnO and it can be also observed second main luminescence band at around 1480 nm.In this work we benefited from our broad knowledge of erbium-doped crystals of LiNbO<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub> and therefore the summary of the results from the erbium doping into other photonic crystals such as LiNbO<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub> will be mentioned.

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#### PA-80: Ionoluminescence investigations on Eu, Mn activated BaMgAl<sub>10</sub>O<sub>17</sub> nanophosphors

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In the present work, we have investigated ionoluminescence (IL) properties of Eu and Mn (seperatly as well codoped) activated BaMgAl<sub>10</sub>O<sub>17</sub> (BAM) nanophosphors under 120 MeV Ag<sup>9+</sup> swift heavy ions irradiation. BAM is widely used in plasma display panels (PDPs) and lamps, due to its high luminescence efficiency and good chromaticity and also used in other displays and lighting devices, for example, white light-emitting diodes (WLED). Doped BAM nanophosphors were synthesized by solution combustion approach with varying Eu and Mn concentration i.e. 1, 2, 3, 4, 5 and 6 %. For codping, the concentration of Eu and Mn varies with respect to each other by selecting Eu concentration (2%) based on preliminary results. The structure of the prepared nanoparticles were investigated by X-ray diffraction and scanning electron microscopy. For the ionoluminescence study, pellets of the prepared materials were used. The prominent IL observed emission peaks are centered at 592, 612, 652, and 704 nm and are attributed to the luminescence centers activated by  $Eu^{3+}$  ions corresponding to  ${}^{5}D_{0} \rightarrow {}^{7}F_{i}$  (j=1, 2, 3 and 4, respectively) transitions. Appearance of the shoulders at 577 and 599, 619 and 696 nm indicates the splitting of transitions corresponding to j=1, 2 and 4, respectively. On the other hand, Mn doped nanophosphors show broadband IL peak centered at 521 nm, which is due to the transition of  $Mn^{2+}$  that originates from  ${}^{4}T_{1}$  ex-citation state to  ${}^{6}A_{1}$  ground state, responsible for green emission. Results shows that the IL intensity increases with increasing dopant concentration. The codping results in appearance of the Eu<sup>2+</sup> band, indicating reduction of Eu<sup>3+</sup> to  $Eu^{2+}$ , while a decrease in the  $Eu^{3+}$  band intenstity, as shown in figure. An increase in the  $Mn^{2+}$ IL intensity was observed upto equal percentage of Eu:Mn doping followed by a reduction for further doping. To support the results, Mn and Eu oxidation states were investigated by X-ray absorption near edge structure (XANES) on virgin samples. The detailed analysis indicates energy transfer in codoped system which is suitable for energy conversion applications.



## PA-81: Use of Ion Beam Channelling for Lattice Site Location on Al<sub>x</sub>Ga<sub>1-x</sub>N Implanted with Tb<sup>3+</sup> ions

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The lattice site occupied by the rare earth (RE) ions will, to a large extent, govern the luminescence properties of the optically doped semiconductors [1]. Therefore, the identification of particular lattice sites as luminescence centers plays an important role in optimizing RE doped AlGaN based optoelectronic devices. The Ion Beam Channelling technique presents unique capabilities regarding the lattice site location under ion implantation for relatively high fluences  $(10^{14} - 10^{15} \text{ ions/cm}^2)$ . Moreover, as compared to the other direct technique, Emission Channelling, it has the advantage of using the reference signal from the host atoms for direct comparison with the signal of the implanted ions. While the GaN semiconductor has been extensively studied, few information about the lattice location of RE ions in Al<sub>x</sub>Ga<sub>1-x</sub>N and the influence of the AlN content has been published [2,3].

This work presents a detailed study of RE lattice site location in Al<sub>x</sub>Ga<sub>1-x</sub>N (0≤x<1) samples grown by halide vapour phase epitaxy on (0001) sapphire substrates. The samples were implanted with a fluence of  $5\times10^{14}$ cm<sup>-2</sup> of terbium ions and an energy of 150 keV. Lattice implantation damage is reduced using channelled ion implantation performed normal to the sample surface, along the <0001> axis. Besides, rapid thermal annealing treatment at 1200 °C was performed to remove the damage and to activate the optical emission of Tb<sup>3+</sup> ions. The study of lattice site location is achieved through the measurements of detailed angular scans across the <0001>, <10-11> and <21-13> axial directions. The precise location of the implanted Tb<sup>3+</sup> ions is obtained by combining the information of these angular scans with simulations using the Monte Carlo code FLUX [4]. Thermal treatment reduces the fraction of Tb ions on substitutional Ga/Al sites. Considering a large fraction of Tb ions, occupying a site displaced by 0.2-0.3 Å along c-axis from the Ga/Al substitutional site, in addition to the Ga/Al substitutional fraction, gives a good agreement between the experimental results and the simulation. The fraction depends on the AlN content of Al<sub>x</sub>Ga<sub>1-x</sub>N samples. The relation between AlN content and lattice site location of RE ions on Al<sub>x</sub>Ga<sub>1-x</sub>N will be discussed.

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### PA-82: High temperature annealing studies of strontium ion implanted glassy carbon

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Glassy carbon samples were implanted with 200 keV strontium ions to a fluence of 1×10<sup>16</sup> ions/cm<sup>2</sup> at room temperature. Analysis with Raman spectroscopy showed that ion bombardment amorphizes the glassy carbon structure. Partial re-crystallization of the glassy carbon structure was achieved after the implanted sample was annealed at 900 °C for one hour. Annealing the strontium ion bombarded sample at 2000 °C for 5 hours resulted in complete recovery of the glassy carbon structure. This recovery was also accompanied with some graphitization of the glassy carbon substrate. Rutherford backscattering spectroscopy (RBS) showed that the implanted strontium diffused towards the surface of the glassy carbon after annealing the sample at 900 °C. This out diffusion was also accompanied by segregation of strontium on the surface of the glassy carbon. Comparison between the as-implanted and 900 °C depth profiles showed that less than 30 percent of the strontium was retained in the glassy carbon after heat treatment at 900 °C. The RBS depth profile after annealing at 2000 °C indicated that no strontium ions were retained after heat treatment at this temperature.

# PA-83: Formation of CdTe nanophase by swift heavy ion induced mixing of Te/Cd/Te trilayer films

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Irradiation by energetic ion beam at 100 MeV/amu (Swift Heavy Ion) is a versatile technique for material modification. In the present work swift heavy ion irradiation induced mixing effects followed by nanophase formation have been investigated in Te/Cd/Te trilayer films. The Te/Cd/Te trilayer films prepared by thermal evaporation method are irradiated by 100 MeV Ni ion beam at a fluence of  $1 \times 10^{12}$ ,  $5 \times 10^{12}$  and  $1 \times 10^{13}$  ions/cm<sup>2</sup>. The structural changes induced by SHI irradiation are studied by X-ray diffraction (XRD) technique. The XRD studies indicate that irradiation results in Te/Cd/Te layer mixing and CdTe phase formation. The observed layer mixing is attributed to the interfacial reaction taking place within the molten zone created in

the wake of energetic ions. The particle size estimated on the basis of XRD is found to be 22nm, 20 nm and 13 nm for the films irradiated at fluence of  $1 \times 10^{12}$ ,  $5 \times 10^{12}$  and  $1 \times 10^{13}$  ions/cm<sup>2</sup> respectively. The elemental composition before and after irradiation is studied by Energy Dispersive X-ray Analysis (EDAX). The surface morphology was studied by scanning electron microscopy (SEM). The optical characterization is carried out using UV-VIS spectroscopy. The band gap values of all the irradiated films are found to be more than the reported value of 1.5 eV. The increase in bandgap value is attributed to the nanosized grain formation after irradiation.

#### PA-84: Mechanism of damage buildup in RE-ion bombarded ZnO

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Although ion bombardment of compound semiconductors has been studied extensively over the last three decades, a lack of understanding remains on the fundamental mechanism of damage buildup and amorphisation of these materials. For single element semiconductors a continuous growth of damage clusters up to amorphisation has been observed. In the majority of compound crystals structural transformations at specific fluences occur, which can be visualized as steps in the defect accumulation curve. Ion channeling is principally used in such studies. However, because this technique can hardly distinguish between different types of defects the oversimplified model of randomly displaced atoms (RDA) is usually applied and disorder accumulation model has been fitted to such data. Important step forward has been made by the recent development of the Monte Carlo McChasy simulation code, which made it possible to distinguish between RDA and extended defects producing long range lattice distortions. Our previous work on GaN, oxide single crystals, etc. has shown that in compound crystals the growth of extended defects (dislocation, dislocation loops, etc.) upon ion bombardment and their role in defect transformations are by no means negligible.

Here we report the results on damage buildup in RE-ion implanted ZnO. Two step damage accumulation process has been revealed. HRXRD analysis has indicated that ion bombardment to low fluences leads to strain build up in the implanted region. Once the critical value of the stress has been attained plastic deformation due to the dislocation slip takes place leading to the formation of dislocations tangle. The transition to the stage III – amorphisation – has not been observed.

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#### PA-85: Interfacial reactions and surface analysis of W in 6H-SiC

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Tungsten thin film was deposited on bulk single crystalline 6H-SiC substrate and annealed in vacuum at temperatures ranging from 500 to 1000 °C for 1h. The resulting solid state reactions (phase composition) and surface morphology were investigated by Rutherford backscattering spectroscopy (RBS), grazing incidence X-ray diffraction (GIXRD), scanning electron microscopy (SEM) and atomic force microscopy (AFM). The RBS spectra were simulated using the RUMP software in order to obtain the deposited layer thickness, reaction zone compositions and reaction zone thickness. The as-deposited spectra fitted well with those annealed at 500 and 600 °C. This indicated that there was no reaction taking place at these two temperatures. At temperatures of 700 °C and above, W reacted with the SiC substrate and formed a mixed layer of carbide and silicides. XRD was used to identify the phases present and to confirm the RBS results. WC and WSi<sub>2</sub> were the initial phases formed at 700 °C. At 800 and 900 °C, additional carbide and silicide phases (that is  $W_2C$  and  $W_5Si_3$ ) were also present; while at 1000 °C, tungsten carbide with different compositions together with both the silicides were present. The SEM images of the as-deposited, 500 °C and 600 °C annealed samples showed uniform granular surface of W. The W layer became heterogeneous during annealing at higher temperatures as the W granules agglomerated into island clusters at temperatures of 800 °C and higher.

## PA-86: Enhancement of superconducting NiBi<sub>3</sub> phase by swift heavy ion induced ballistic mixing

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Swift heavy ions (SHI) induced ion beam mixing (IBM) have their own advantages like spatial selectivity, relatively low temperatures, and mixing of thermodynamically immiscible systems to harvest stable/meta-stable phases, which has been understood on the basis of thermal spike and coulomb explosion models depends on metallic or insulator systems. In this study, we have chosen Ni/Bi bi-layers system for its interesting magnetic and superconducting properties of NiBi<sub>3</sub> phase [1]. Thermally evaporated Ni/Bi bilayers were irradiated by 100 MeV Au ions in the fluence range of  $1 \times 10^{12} - 1 \times 10^{14}$  ions/cm<sup>2</sup> to enhance the NiBi<sub>3</sub> phase fraction through IBM. Here we emphasize the effect of ballistic mixing and temperature evolution via thermal spike model to explain the mechanism of improved fractional phases after irradiation. Though there is a large mismatch of thermal spike based temperature, a reasonable ballistic mixing has been observed from cross-sectional transmission electron microscopy (STEM-HAADF) with energy

dispersive X-ray spectroscopy (EDX) (shown in Fig.1), X-ray diffraction, and Rutherford backscattering study. These observations were also strongly supported by the thermal spike model [2], where the phenomenon was understood on the basis of insensitivity of Ni layer towards the energy that has been used in this report.



Figure 1: The (STEM-HAADF) XTEM image of Ni/Bi bilayer thin films and the corresponding line scan EDX spectra to understand the diffusion of Ni and Bi layers, before and after ion irradiation.

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### PA-87: Studies of SHI induced modification in Ni doped SnO<sub>2</sub> crystalline thin films

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Swift Heavy Ion induced modification in thin films of transition metal doped tin oxide  $(SnO_2)$  grown by RF Sputtering technique were done by irradiating Swift Heavy Ion (SHI) beams of 100 MeV Au with varying ion fluencies from  $5 \times 10^{11}$  ions/cm<sup>2</sup> to  $1 \times 10^{13}$  ions/cm<sup>2</sup> using 15 UD Pelletron Accelerator at Inter University Accelerator Center (IUAC), New Delhi, India. The optical band gap as observed from UV/Visible results for pristine and irradiated samples with 100 MeV Au ion fluencies of  $1 \times 10^{12}$ ,  $5 \times 10^{12}$  and  $1 \times 10^{13}$  ions/cm<sup>2</sup> are 5.24, 5.15, 4.13 and 3.76 eV respectively. This is attributed to scattering due to surface roughness, variation in particle size and modification in local electronic structure. Significant increase in Urbach's energy was observed at lower irradiation fluence of  $5 \times 10^{11}$  and  $1 \times 10^{12}$  ions/cm<sup>2</sup>. The electrical properties were studied by measuring variation of current (I) w. r. t. applied potential (V) at transport measurement laboratory of IUAC, New Delhi, India. All the samples have linear curves with decrease in slope as irradiation fluence increases. Significant drop in resistance due to increase

in irradiation fluence was observed. Surface morphology studies by Atomic Force Microscopy (AFM) technique shows grain size dependence on irradiation fluence. Growth kinetics was studied by power spectrum density (PSD) technique using AFM data. The calculated roughness exponent and rms roughness w. r. t. irradiation fluence varies similarly. Magnetic Force Microscopy (MFM) images show uniformly distributed magnetized nanoparticles over the entire surface after irradiation. Particle size and phase transformation was studied by glancing angle X-ray diffraction (GAXRD) technique. The particle size was calculated using Debye-Scherrer's formula. Up to the fluence of Au-1×10<sup>12</sup> ions/cm<sup>2</sup> the particle size is around ~7 nm. And further increase in irradiation fluence of Au ion beam the particle size increases to ~14 and ~30 nm at fluence of Au-5×10<sup>12</sup> and Au-1×10<sup>13</sup> ions/cm<sup>2</sup> respectively. Based on uniform deformation model (UDM), the strain in these thin films along (101) and (110) planes was calculated using GAXRD data. Resonance Rutherford Backscattering (R-RBS) results confirms the presence Ni in the samples and impurity free phase modification. Detailed results will be discussed during presentation.

### PA-88: Modification of Microstructure of 100 MeV O<sup>7+</sup> Ion Irradiated Polyamide Nylon-66 Polymer by Positron Annihilation Lifetime Spectroscopy

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Polymers have prospective applications in all fields of science and technology, especially in medical, optics, electronics, biotechnology, photonics and space research. Importance of ion beam and radiation modification of the polymers has increased in recent years, prompted by the ion and radiation induced improvements in the mechanical, chemical, optical and electrical properties of various polymeric substrates and thus modification of polymer properties under ionizing radiation is a subject of great importance due to the enhanced utility of the polymers in a hard radiation environment such as high-energy particle accelerators, sterilization irradiators, nuclear power plants and space-crafts etc. Polyamide Nylon-66 polymer films were irradiated by 100 MeV  $O^{7+}$  ions to the fluences ranging from  $1 \times 10^{11}$  to  $5 \times 10^{12}$  ions/cm<sup>2</sup>. The other set of PET films were exposed to gamma radiation emitted by <sup>60</sup>Co source at various doses ranging from 100 to 1250 kGy. Both ion irradiation as well as gamma exposure showed modifications in the microstructure of polyamide nylon-66 polymer samples primarily in terms of changes in the size of available free volume and fractional free volume but the effectiveness of gamma radiation exposure was more pronounced than that of ion irradiation. UV-Vis studies showed a shift in absorption edge towards the visible region and a decrease in band gap energy (Eg) from 3.7 eV to 3.07 eV for the irradiated samples. The corresponding number of carbon atoms per conjugation length increased from 86 to 125 for the irradiated samples. The XRD analysis shows a shift in the diffraction peak and an increase in the crystalline nature of the polymer at higher fluence as a result of decrease in the peak width of XRD patterns. The FTIR studies showed insignificant modifications in the chemical structure of polymer samples. The results will be discussed.

## PA-89: A comparative study of the effects of Swift Heavy Ion and Gamma radiation on physico-chemical properties of Kapton-H Polymer

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Aromatic polymer such as Kapton-H is an important class of polyimide. Due to its exceptional physical, optical and mechanical properties, it has a potential use in electrical, electronics, space and nuclear industries. The effect of oxygen ions ( $O^{7+}$ ) of energy 100 MeV with fluences in the range of  $1 \times 10^{11}$  ions/cm<sup>2</sup> to  $5 \times 10^{12}$  ions/cm<sup>2</sup> and Gamma radiation of doses 100, 250, 500, 750 and 1000 kGy on physico-chemical properties of Kapton-H polymer samples were studied. The pristine and irradiated samples were characterized by X- ray diffraction, UV- visible and FTIR for structural, optical and chemical studies respectively. A comparative study of physico-chemical properties were carried out from the obtained values of the pristine and irradiated samples of the polymer. The band gap ( $E_g$ ) values of the irradiated samples show variations for both cases which are attributed to the shift of the absorption edges of UV-visible spectrum. The intensity of the various absorption bands of the infrared spectra were found to change indicating the changes in the chemical properties of the exposed samples. The results will be discussed.

### PA-90: F Center Distribution in Sapphire Under Low-Dose Low-Energy Ar Irradiation

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Optical spectroscopy and Rutherford Backscattering Spectrometry Channeling (RBS-C) have been used to study F center distribution with dose in 170 keV Ar<sup>+</sup> irradiated single crystals of sapphire ( $\alpha$ - Al<sub>2</sub>O<sub>3</sub>) at room temperature for implantation doses between 10<sup>13</sup> to 5 × 10<sup>14</sup> Ar<sup>+</sup> cm<sup>-2</sup>. F center density (N<sub>F</sub>) has been found to display an initial rapid linear increase with Ar<sup>+</sup> dose and then saturate to a maximum value of 1.74 ×10<sup>15</sup> cm<sup>-2</sup>. Fitting experimental results with a Poisson relation suggest an estimated electron capture range of 4.24 Å around an Oxygen vacancy. A possible explanation of this behaviour is presented.

### PA-91: Swift heavy ion induced structural and magnetic properties modification in Fe-based amorphous alloys

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Amorphous alloy (also known as metallic glass) is a kind of new alloy material synthesized by using modern rapid solidification metallurgical technology, owning excellent mechanical, physical and chemical properties that general metal and glass have. The unique glassy structure makes metallic glass have some high performances such as high strength, corrosion resistance, great ductility, and having a wide supercooled liquid region, etc. which usually belong to the high-quality magnetic functional materials, and hence showing enormous potential for development and application in the fusion reactor [1-3]. In this work, SHI irradiation as a kind of special non-equilibrium and exogenous energy deposition process will be applied to the study on modification of the structural and magnetic properties of the amorphous alloys.

Amorphous FeSiNbZrB alloy ribbons were prepared by melt spinning, and then the amorphous ribbons were irradiated at RT with 2.01 GeV  $Kr^{26+}$  ions for fluence range from  $1 \times 10^{11}$  to  $1 \times 10^{14}$  ions/cm<sup>2</sup> on the materials research terminal of the HIRFL-SSC (IMP, Lanzhou). X-ray diffraction (XRD), transmission electron microscopy (TEM), vibrating sample magnetometer (VSM), superconducting quantum interference device (SQUID) and mössbauer spectra (MS) were used to measure the structural and magnetic properties of the pristine and irradiated samples.

It's obviously that before and after SHI irradiation, such amorphous FeSiNbZrB alloy systems do not have a long-range order in atomic arrangement and exhibit only a short-range order. Under SHI irradiation at RT, local crystallization phenomenon of amorphous FeSiNbZrB alloy ribbons has been confirmed and formation of finer  $\alpha$ -Fe(Si) phases precipitations with diameter of 1-2 nm has been observed. In addition, after irradiation, magnetic anisotropy considerably changes from its original in-plane direction. Possible mechanism of structural and magnetic properties modification after SHI irradiation is discussed briefly.

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[2] H.S. Barnard, Z.S. Hartwig, G.M. Olynyk, J.E. Payne, Fusion Engineering and Design 87 (2012) 248.

[3] J.R. Sun, Z.G. Wang, Y.Y. Wang, et.al., Nucl. Instr. Meth. B 307 (2013) 486.

### PA-92: Low energy and high fluence cesium and iodine ion implantation into 6H-SiC

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Near surface effects induced by low energy, high fluence ion implantation in 6H-SiC have been investigated. Ions of cesium and iodine (of energy 150 keV) were implanted into 6H-SiC wafers

at room temperature. The ion fluence in both cases was  $2 \times 10^{17}$  cm<sup>-2</sup>. Depth profiles of the asimplanted and annealed samples were determined by Rutherford Backscattering Spectrometry. The near surface morphology of the samples was analysed by high resolution Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM). A Focused Ion Beam (FIB) column was employed for in situ cross-sectional SEM analysis of the samples. For samples implanted with cesium, deformations in the form of blisters were observed in some areas for the as-implanted samples. Certain areas were found to have undergone severe surface exfoliation and to some extent near surface layer splitting. For samples implanted with iodine ions, images taken from the as-implanted samples revealed the development of cone-like structures (protrusions) on the surface, indicating some form of sputtering process occured during implantation. Surface roughness increased from about 1 nm (as-received wafer ) to about 5 nm after the room temperature ion implantation. No blisters were detected for the iodine implanted samples even after high temperature annealing cycles up to about 1500 °C for 30 hours. No evidence of surface exfoliation or layer splitting was found for iodine implanted samples even after long time high temperature annealing. This was attributed to the possible strong binding of iodine within the SiC matrix.

### PA-93: Response of MgO to ion irradiation in the nuclear energy-loss regime and effect of irradiation temperature

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Magnesium oxide (MgO) is considered as a candidate material for neutron reflector in sodium fast reactors, matrix of ceramic-ceramic composite (cercer) nuclear fuel for minor actinides transmutation or electrical insulator for diagnostics components in ITER. Therefore, a deep understanding of the behavior of this material under ion irradiation environment is required.

Very recently [1], we demonstrated, through the use of RBS/C, XRD and TEM experiments, that the damage build-up in MgO irradiated with 1.2 MeV  $Au^+$  ions at room temperature occurs in three steps, and transitions from one step to the next one reveal microstructural transformations. Each step is characterized by specific features such as damage, strain levels and nature of defects. The present study aims to investigate the effect of the irradiation temperature on the response of MgO.

For this purpose, additional 1.2 MeV Au<sup>+</sup> irradiations have been performed in a broad fluence range (from  $5x10^{12}$  to  $4x10^{14}$  cm<sup>-2</sup>) at 300°C, 500°C and 800°C. Results show that disorder, which accumulates from the surface up to a few hundreds nanometers, is still a multi-step process at high temperature, but occurrence of the microstructural transformations is modified. In addition, disorder and strain levels also change with temperature. These findings reveal a competition between defect creation and recovery processes. A comparison with cubic zirconia suggests, at first sight, that MgO is less radiation resistant. However, electronic excitation effects can lead to damage healing in MgO.

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### PA-94: Electrical characterization of deep level created by bombarding nitrogen-doped 4H-SiC with alpha-particle irradiation

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Deep-level transient spectroscopy (DLTS) was used to investigate the effect of alpha-particle irradiation on the electrical properties of N-doped 4H-SiC. The samples were bombarded with alpha-particles at room temperature (300 K) using an americium-241 ( $^{241}$ Am) radionuclide source. DLTS reveals the presence of four deep levels in the as-deposited samples,  $E_{0.09}$ ,  $E_{0.11}$ ,  $E_{0.16}$  and  $E_{0.65}$ . After irradiation with a fluence of 2.6 × 10<sup>10</sup> alpha-particles-cm<sup>2</sup>, DLTS measurements indicate the presence of one new deep level,  $E_{0.62}$  with signature of energy,  $E_{c}$ -0.62 below the conduction band with an apparent capture cross section of 1 × 10<sup>13</sup> cm<sup>2</sup>. Furthermore, a stepwise irradiation of the samples revealed the decrease of intensity of the  $E_{0.09}$ , and a corresponding increase in the intensity of  $E_{0.62}$  and  $E_{0.65}$  defects, while  $E_{0.09}$  and  $E_{0.11}$  remained unchanged. The capacitance-temperature, current-voltage and capacitance-voltage characteristics of our samples showed good correlation as the irradiation fluence is increased. The defect  $E_{0.62}$  has the attribute of  $Z_1/Z_2$ . The average free carrier removal rate has been estimated to be 6500 cm<sup>1</sup>.

### PA-95: In-situ ion beam analysis for swift heavy ion track characterization

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Nanoscale damage produced when a swift heavy ion (SHI) passes through a solid material (called an ion track) can be observed directly by means of Transmission Electron Microscopy and Atomic Force Microscopy. However, in most studies related to the SHI irradiation of materials, the goal is to find how size of the ion track depends on the stopping power of the SHI. To achieve this, there is also a number of other techniques at disposal that can be used to evaluate ion track sizes indirectly.

One of the most frequently used techniques for indirect measurement of ion tracks is RBS/channeling. By measuring amorphisation of the single crystal as a function of applied SHI fluence, and by taking into account ion track overlap, one can extract ion track radius [1,2]. However, studies of this kind require several samples to be irradiated and measured independently, thus consuming a lot of beamtime. Also, at low values of the stopping power, close to the ion track formation threshold, even more samples need to be irradiated and analyzed due to deviations from the simple track overlap model. To acquire such substantial amount of experimental data needed for reliable analysis [1], an in-situ RBS/channeling could

provide adequate solution. Recent commissioning of the dual ion beam chamber at the RBI, Zagreb opens up an opportunity to study kinetics of ion track formation using in-situ RBS/channeling. Ion track formation can be accomplished using SHI delivered by the 6 MV EN Tandem Van de Graaff accelerator, while simultaneously RBS/channeling measurement can be done using ion beams delivered from the 1 MV Tandetron.

Another approach for in-situ IBA is to use SHI beam for simultaneous material modification and analysis by Elastic Recoil Detection Analysis (ERDA). As shown recently [2], by monitoring elemental changes in the sample, and using ion track overlap model, ion track characterization can be accomplished. This opens up new research possibilities at medium sized accelerator facilities because stopping powers of SHI beams used for ERDA are often sufficient to produce ion tracks in many materials. Here we present two examples of using TOF-ERDA setup at RBI, Zagreb [3] for a) monitoring stoichiometry changes during ion track formation in GaN and b) hydrogen loss in irradiated amorphous  $Al_2O_3$  thin films.

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[2] C. Rotaru et al., Track formation in two amorphous insulators, vitreous silica and diamond like carbon: Experimental observations and description by the inelastic thermal spike model, Nucl. Instr. Meth. B 272 (2012) 9

[3] Z. Siketić et al., Time of flight elastic recoil detection analysis with a position sensitive detector, Rev. Sci. Instr. 81 (2010) 033305

### PA-96: Simulation of alpha decay of actinides in iron phosphate glasses by ion irradiation

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A surrogate approach of ion beam irradiation on iron phosphate glasses was employed to simulate radiation damage in nuclear waste glasses, induced by actinide decay. Iron phosphate glasses are being considered as an immobilisation matrix for high level nuclear waste, including minor actinides and plutonium residues, due to their high chemical durability and ability to incorporate diverse chemical compositions. Alpha-decay of the incorporated actinides results in the formation of energetic alpha-particles (4.5 to 5.5 MeV), energetic recoil nuclei (70 to 100 keV), and some gamma rays. The alpha-particles with energy ranging from 4.5 to 5.5 MeV will predominately deposit its energy by ionization processes and can lead to the permanent displacement of atoms due to the conversion of a localized electronic excitation into atomic motion. While recoil nuclei with an atomic weight of 235 to 244 and energy 70 to 100 keV will lose most of its energy in ballistic elastic collisions with the nuclei of atoms in the glass. Consequently, interaction of alpha-particles and energetic recoil nuclei will potentially affect the structural integrity of glass matrix. Such modifications may affect glass durability and, therefore, its long term performance as an immobilisation matrix. In this study, ion irradiation approach has been adopted to study effect of alpha decay of actinides in iron phosphate glasses. Two types of ions have been selected (i) 2 MeV He ions to study the effect of electronic losses induced by alpha particles on glass matrix and (ii) 750 KeV Bi ions for simulating ballistic damage due to recoil atoms. In this contribution, we will report structural modification and change in coordination number of network former by employing Reflectance Fourier-Transform Infrared (FT-IR), and Raman spectroscopies as a consequence of ion irradiation. In addition, the ion-glass atom interaction will be discussed to understand the observed structural modifications.

#### PA-97: Mechanism behind origin of swift heavy ion induced larger clusters than track radius

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In this research article, a deep focus on the electronic excitation induced ejection process in Au: SiO<sub>2</sub> nanocomposite (NC) has been devoted to establish a correlation between average size of Au nanoparticles (NPs) in the film and size of Au clusters collected on the catchers. For this investigation, Au-silica NC samples containing 10 at% Au were prepared by atom beam cosputtering on quartz substrates. Some samples were annealed at temperature of 850 °C in an inert atmosphere for an hour to obtain bigger size of NPs. Transmission electron microscopy (TEM) measurements reveal the Au NPs having average size of pristine and annealed films are ~ 7 nm and ~ 21 nm respectively. Now, experiment is divided into two parts: (i) Case A: average size of NPs ( $\sim$  7 nm) is less than track radius ( $\sim$  10 nm for 100 MeV Ag in SiO<sub>2</sub>) and (ii) Case B: average size of NPs (~ 21 nm) is greater than track radius. Ion beam irradiation with 100 MeV Ag ions has been performed on both types of samples at the fluence of 1×10<sup>13</sup> ions/cm<sup>2</sup>. During irradiation, TEM grid and Si catcher were placed in irradiation chamber to collect the sputtered species ejected from the samples. Rutherford backscattering spectrometry (RBS) measurements have been carried out on all samples to determine the Au contents. In case A, electronic sputtering rate at the fluence of 1×10<sup>13</sup> ions/cm<sup>2</sup> is found to be very high (1150 ions/cm<sup>2</sup>) and average size of Au clusters on the catcher TEM grid is nearly same as the size of NPs in the target. However for Case B, it is observed that electronic sputtering rate is  $\sim 17^{\text{th}}$ times less and average size of Au clusters on the catcher is nearly same as in case B but less than the size of Au NPs in the irradiated film. The RBS measurements on Si catcher show that the Au content is higher in Case A than in Case B. However, clusters bigger than the track radius have been found more in Case B. The possibilities behind the cluster emissions are explained on the basis of thermal spike and shock wave model. It is proposed that the size of emitted cluster from such NC films strongly depends on the size of the Au nanoparticles (NPs), and the results completely rule out sputtering in form of atoms only.

#### PA-98: Swift Heavy Ion induced interface mixing in Fe/Cr/Al magnetic multilayer

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 $[Fe/Cr/AI]_{\times 10}$  multilayer having different thicknesses were deposited using electron beam evaporation in an UHV chamber and were irradiation with 150 MeV Ag+ ions to study structural and magnetic properties at the interfaces. X-ray reflectivity (XRR), Magneto-optic Kerr effect (MOKE) and X-ray absorption spectroscopy (XAFS) studies has been done to reveal interface diffusivity, change in magnetic coercivity as well as local structure due to ion irradiation, respectively. It has been observed from XRR that the height of 1<sup>st</sup> order Bragg peak decease fast for lower thickness of  $[Fe/Cr/AI]_{\times 10}$  multilayer than the higher. However, determined diffusion length,  $L^2_{d}$ , as a function of ion fluence indicates that mixing is higher for lower multilayer thickness as compare to higher one. MOKE results show that the Fe/Cr/AI multilayers are soft magnetic in nature and have different in-plane anisotropy in the as-deposited multilayers. As a function of fluence, coercivity trend has been observed opposite for different thickness of Fe/Cr/AI multilayer can be understood in terms of the thermal spike model by taking into consideration the changes in electron mobility due to scattering from surface and interfaces.

# PA-99: Evolution of ZrC RBS-C spectra under MeV ions irradiation: experimental and modeling approaches

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Ceramics constitute a key class of materials in numerous fields such as information and communications technologies, environment, transportation and energy. Regarding the energy field, the use of ceramics, principally oxides and carbides, is widespread in the nuclear industry. For this application, materials evolve in a very specific, harsh environment and are inherently submitted to various sources of irradiation, e.g. by neutrons, fission products and alpha particles. Therefore, it is mandatory to determine the behavior of these materials under this particular environment. For this purpose, a lot of investigations are carried out where materials are exposed to external ion beams delivered by particle accelerators, allowing to avoid radioactive safety issues, to accelerate the ageing process and to control the irradiation parameters for conducting parametric studies. Then, complementary experimental techniques,

such as Rutherford Backscattering Spectroscopy in the channeling mode (RBS/C), X-ray diffraction (XRD), Raman spectroscopy, transmission electron microscopy (TEM), are used to get a good knowledge and understanding of the materials behavior. The response of zirconium carbide to  $Au^+$  1.2 MeV irradiation has been studied by RBS in channeling mode,X-ray diffraction and transmission electron microscopy. Below  $4x10^{14}$  cm<sup>-2</sup>, we observe a build-up of elastic strain with increasing fluences. At this fluence the strain is released, important dechanneling starts to appear and visible TEM damage is observed. With increasing fluence, this damage is found to spread in the material deeper than the area of direct damaging by the ion beam. These experimental observations are reproduced and explained by DFT informed Rate Equation Cluster Dynamics simulations. The simulations show that the discontinuous build-up of damage as well as the in depth damaging of the material are driven by the diffusion and clustering of interstitials. These defects continuously form larger and larger dislocation loops and diffuse deep in the material below the directly damaged area.

## PA-100: The influence of ion fluence on the micromachining of PET foils using proton beam writing

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In this work, we investigated the influence of ion fluence on the development of micropatterns produced by 2.2 MeV H<sup>+</sup> impinging on 12  $\mu$ m thick poly(ethylene terephthalate) (PET, Mylar<sup>®</sup>) foils. Several lines of 1 x 100 pixels were patterned on the PET foils varying the fluence (from 1 x  $10^{11}$  to 6 x  $10^{15}$  ions/cm<sup>2</sup>) and the etching time (from 1 to 60 minutes). The curve of the opening times of the structured lines versus fluence presented an "u-shape". For fluence between  $6x10^{14}$  and  $5x10^{15}$  H<sup>+</sup>/cm<sup>2</sup>, full opening of the lines is reached within 2 min of etching. For fluence, below  $1x10^{14}$  H<sup>+</sup>/cm<sup>2</sup>, the etching times necessary to completely open the structures increase steeply, reaching values close to 60 min the lines patterned are rough and irregular. For very large fluences, the opening times also increase, most probably due to crosslinking of the irradiated polymer. In this regime, reproducibility was poor, and opening times could vary substantially from line to line. While at low fluences the exposed polymer areas may remain roughly intact after several minutes of etching until they were detached from the foils as a whole block. The variability in the linking points between the exposed block and the rest of the polymer matrix may explain the spread in the opening times observed at high fluence.

#### PA-101: On the nature of sputtered particle from Lithium Fluoride thin films

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In this paper electronic sputtering [1-3] induced desorption from 100 nm LiF thin films deposited on fused silica substrates is reported. The sputtering was performed using 120 MeV Ag ions in equilibrium charge state of +25 to avoid the charge state effect. Quadrupole Mass Spectrometer (QMS) was used to find out the nature of sputtered particle, complemented by elastic recoil detection analysis (ERDA) technique for quantitative estimation of sputter yield [4]. The ERDA results showed that the sputtering yield, ~1.9×10<sup>5</sup> atoms/ion, is stoichiometric and a reduction in the yield was observed with ion fluence. QMS results show that when beam was not falling on the sample, the signals of different particles were nearly constant and when sputtering started, an abrupt increase in the signals of the particles followed by a reduction was observed. In case of neutral atoms collection, the main, well-distinguishable lithium <sup>6</sup>Li<sup>0</sup> and <sup>7</sup>Li<sup>0</sup>, <sup>19</sup>F<sup>0</sup>, <sup>26</sup>LiF<sup>0</sup> and <sup>27</sup>LiF<sup>0</sup> are clearly seen, while Li<sup>+</sup> and F<sup>-</sup> are seen for positive and negative modes, respectively. No preferential sputtering of F was observed showing the stoichiometric nature of sputtering. It is also observed that the counts of Li and F decreases with ion fluence and start to saturates at higher fluence of 2×10<sup>12</sup> ions/cm<sup>2</sup>, while the counts of LiF is almost constant up to 2×10<sup>12</sup> and start to decrease after this fluence. The counts of neutral LiF are order of magnitude lower than that are for Li and F. This indicates that the ejection of neutral particles is dominated by Li and F in lower fluence region (can be assumed up to a region of non-overlapping fluence  $\sim 2 \times 10^{12}$  ions/cm<sup>2</sup>) and there after desorption of LiF is dominated. The QMS observation are consistent with ERDA results which also shows stoichiometric sputtering from LiF. It is also worth to mention that no signature of cluster emission was found.

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# PA-102: Compositional, structural and optical changes of heavy ions irradiated polyimide for the purpose of the stopping power measurement

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The stopping power and energy straggling of  ${}^{12}C^{n+}$  and  ${}^{16}O^{n+}$  heavy ions in 9 µm thin polyimide (PI) foil with heavy ion initial energies between 13 – 16 MeV were measured by means of an indirect transmission method using a half-covered PIPS detector. Ions were scattered from thin gold layer, which was evaporated on carbon substrate, under the scattering angle 150°. The values of experimental stopping power were compared with simulation in program SRIM-2008, MSTAR codes and other experimental data taken from Paul's data base. The energy straggling data were compared with those calculated by using Bohr's, Bethe-Livingston and Yang models. The measured energy straggling value in PI foil was corrected for foil roughness and thickness inhomogeneity. Bethe–Livingston formulation has been modified to make it appropriate for thicker targets. The measured energy straggling is higher than Bohr's value. The predictions by Yang are in good agreement with our experiment. Influence of the charge-exchange straggling for C and O ions in PI was discussed.

### PA-103: Dedicated experiments for reliable measurements of energy-loss straggling

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While there is a plethora of experimental data on electronic energy loss of various ion-target combinations, that are often triggered by applications, the situation for energy-loss straggling is different. Not only is there less experimental data available, but also existing results are often plagued by systematic effects. In particular, target inhomogeneities, incident beam energy spread and experimental resolution effects can lead to erroneous results. This situation is far from satisfying for applications in need of relevant energy-loss straggling data, but it also makes direct comparisons with theoretical predictions difficult. Recently, substantial progress has been made to advance these calculations (see contribution by Sigmund et al. this conference). This allows reliable predictions to be made that are comparable to semi-empirical formulas (e.g. Yang-straggling).

In order to provide reliable experimental data for comparison with theory, we have used different experimental setups to measure energy-loss straggling. The selection of beams (Si and Kr), energies and gas targets (He, N<sub>2</sub>, Ar, Ne, and Kr) was guided by dedicated theoretical calculations. To cover a wide energy range, experiments were performed with beam energies in the range 0.5-2 MeV/u at the 6 MV EN Tandem accelerator at ETH Zurich, Switzerland, and with 1-12 MeV/u at the K130 cyclotron of the Accelerator Laboratory at the University in Jyväskylä, Finland. Different measurement techniques were employed: at ETH Zurich we use an open external gas stripper tube as a windowless gas target followed by energy profile measurement based on a magnetic spectrometer; at JYFL we use TOF detectors to determine the energy profile after a gas cell having thin silicon nitride entrance and exit windows. The overlap of measurements at around 2 MeV/u allows one to check for any systematic effects that might bias the results at either location.

In this contribution, we describe the two experimental setups and present first results. Comparisons to theoretical predictions and interpretation are presented in the contribution by Sigmund et al.

# PA-104: Energy loss by few MeV protons in Aluminum and Molybdenum measured with high accuracy

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To comply with the international demand - especially with the increasing use of the selfconsistent Ion Beam Analysis techniques - to improve the database of stopping powers and to check the confidence of theoretical models, this study aims at an accurate and traceable experimental approach investigating with high precision the energy loss of protons in Aluminum and Molybdenum targets in the energy range of [800 - 3400] keV. The choice of these two elements has the following motivation. The former has been accurately measured several times by other authors [3, 4] and was used to validate the present procedure. The latter is a high-Z material, which is an harder case for theory, and has been measured once before [4]. Further, our goal was also to provide a critical assess of the sources of uncertainty in our measurements [1, 2], using a thorough protocol for calibrations (beam energy, detector electronics, geometry, etc), suitable techniques to certify the thickness and purity of the targets and a rigorous analysis of the budget of uncertainty. The final stopping power results were obtained with relative uncertainties less than 0.69 % for Aluminum and 0.85 % for Molybdenum. Furthermore, the present data were used to provide a stringent test of different theoretical and semi-empirical models: SRIM-2013, ICRU-49, PSTAR, CASP-5.2 and Geant4 [5]. The discrepancy to the Geant4 prediction using the newest available physics list is also presented [6]. A general disagreement between the Molybdenum data for stopping power and the ICRU-49, PSTAR, and Geant4 has been found and reported. In the case of Geant4, a comparison with the full energy loss distribution (i.e. including straggling) will also be discussed. Finally, these accurate results can be used as input for databases and codes for stopping power and straggling calculations [7].

## PA-105: Energy loss of slow <sup>20</sup>Ne ions in Ag and Pt in transmission and backscattering geometries

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The energy loss of heavy ions with energies in the range of a few keV per nucleon is an important quantity for many ion-beam techniques e.g, sputtering and ion implantation [1]. For sufficiently low energy, a significant contribution from elastic losses due to multiple scattering events with the target nuclei along the ion trajectory is expected. To be able to study for example details of the electronic stopping process proper separation of these nuclear losses is required [2]. In the present work, we are investigating the energy loss due to elastic and inelastic losses in backscattering and transmission geometries. TOF-MEIS experiments are performed to determine the electronic energy loss of <sup>20</sup>Ne ions in thin films of Pt and Ag in backscattering geometry. To properly account for the contribution from nuclear losses, Monte-Carlo simulations using TRBS (Trim for BackScattering) are conducted. Since earlier results for Ne in Au indicated low nuclear losses for the characteristic trajectories in a backscattering spectrum [2], we study also in detail the expected elastic energy losses in both backscattering and transmission geometry from computer simulations using TRIM. The energy loss in transmission geometries for <sup>20</sup>Ne ions in Ag and Pt foils is calculated for a number of different target thicknesses, and for different ion energies. A systematic comparison of trajectory length, the total energy loss as well as nuclear and electronic contributions is performed. The relative contribution of elastic scattering to the total energy loss in transmission and backscattering geometries is estimated.

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#### PA-106: Low-Energy Electronic Stopping Power Evaluated by Molecular Orbital Theory

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The electronic stopping power of slow atom or ion has been theoretically studied by two alternative approaches [1]. One is the Firsov model, based on the collision complex formed in a close encounter of the projectile and the target atom. The other approach is the theory based on the analogy to the electric resistance by the impurity scattering of the free electron gas (referred to as the electric resistance model).

Both approaches have a critical disadvantage. In fact, the Firsov model has not yet been theoretically justified, and, on the other hand, the electric resistance model can be applied only

for the uniform medium, even though it is theoretically justified.

As our interest was the position dependent stopping power [2], we discarded the electric resistance model. Thus, our talk focuses on the updated version of the Firsov model [3], in which the evaluation of stopping power is done by the molecular orbital calculations of the collision complex.

By this evaluation, the stopping power observed in the surface grazing scattering experiments [2], and the  $Z_1$  oscillation of the electronic stopping power [4] could be quantitatively analyzed and understood in terms of the molecular orbitals of the collision complex. Moreover, we discuss the physical implication behind the two alternative approaches, i.e., the Firsov and the electric resistance models.

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#### PA-107: Withdrawn

# PA-108: Stopping Cross Sections of Protons in Ti, $TiO_2$ and Si Using Medium Energy Ion Scattering

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Understanding the way in which ions loose energy as they travel through a target medium is essential to the techniques of ion beam analysis. Potential corrections of commonly used stopping powers, such as the ubiquitous Andersen Ziegler values, would lead to greater quantitative accuracy as an analytic technique. In this study the energy loss of protons was studied for thin films of Ti, Si and TiO<sub>2</sub> and the stopping cross sections were determined using the medium energy ion scattering. Ti was deposited via molecular beam epitaxy (MBE) onto Si(100) and annealed at 2000 °C in O<sub>2</sub> to form TiO<sub>2</sub> thin-films. Si films were deposited on carbon substrates by MBE, and were transferred to MEIS UHV system in a dry-nitrogen-filled bag. Thickness and composition of the film and the structure of the TiO<sub>2</sub>/Si interface were determined using a combination of Rutherford Backscattering Spectroscopy (RBS), X-ray photoelectron Spectroscopy (XPS), and Medium Energy Ion Scattering (MEIS). Directly from the MEIS spectra, the stopping cross sections of Ti, Si and TiO<sub>2</sub> were calculated at a range of energies (50-170 keV) by an iterative program [1]. A comparison is made with the commonly used Andersen Ziegler values.

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#### PA-109: Stress measurements by medium energy scattering : Ge implanted Si on insulator

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Modifications of semiconductor interface of the type crystal/amorphous as Si/  $SiO_2$  have been recently investigated because of technological interest in microelectronics, in particular for the development of optical and electronic devices, such as volatile memories (flash memory) and field effect transistors (FET technology) [1].

Specifically Silicon-Germanium On Insulator (SGOI) samples have been produced by Ge implanted  $SiO_2$  with a subsequent bonding as in samples of SOI (Silicon on Insulator). Recent studies have shown that SGOI heterostructures have an effective increase in the mobility of carriers when compared with pure SOI [2].

The Medium Energy Ion Scattering (MEIS) technique can be used to analyze crystalline samples, being capable to determine not only the elemental composition and concentration profiles but also the crystalline structure through the analysis of the blocking lines. In addition, it was shown recently that MEIS can also be used to characterize nanoparticles systems on the surface as well as in deeper layers.

In this work we demonstrate the use of the MEIS technique for getting the stress in SGOI structures through the construction of crystallographic maps (3D-MEIS) as in ref.[3]. These maps are obtained by measuring the intensity of the backscattered ions as a function of the variation of the azimuthal and the scattering (polar angle) angles. Regions of low ion scattering intensity regions are easily observed and correspond to blocking directions that can be easily identified when confronted with the stereographic projection projection of the analyzed crystalline material. The value of the stress can be determined by simulating the new blocking directions due to expansion of the lattice parameter along the sample normal

We conclude that the stress in the samples (SGOI) is around 3%, which represents a stoichiometry in the alloy Si<sub>0,6</sub>Ge<sub>0,4</sub> (see Figure 1). This result is within a physical limit to the stability of the lattice, whereas above 4% stress the network becomes unstable and impractical for the construction of devices. The efficiency of MEIS technique for characterizing crystalline materials is established, and it is possible to determine that the increase in electron mobility in these materials is due to the stress caused by germanium atoms when incorporated into the crystalline silicon network.



Figure 1: 3D MEIS cartography for germanium (SGOI) with the simulation of stereographic map for 3% stress in the lattice parameter

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### PA-110: Accurate Ion Beam Analysis at the Surrey Ion Beam Centre

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We have recently demonstrated that Rutherford Backscattering Spectrometry (RBS) can be used as a primary direct method capable of an absolute traceable accuracy of 1% for the measurement of quantity of material implanted into silicon [1], suitable for certifying SIMS standards. The global standard uncertainty ascribed to this technique is evaluated by a full uncertainty budget [2] which has been validated through the analysis (at a precision of 0.3%) of standard samples over the past three years.

The uncertainty budget of this method shows that one needs to carefully characterise the acquisition system for reaching a global accuracy of 1% by controlling the electronic gain of the pulse height spectrometry system [3]. The scattering angle uncertainty is also surprisingly large and we will present a beam handling procedure using the 6-axis goniometer [4] together with the beamline laser to reliably control this at 0.2 degrees.

For the measurement of low quantities of material the pileup correction typically dominates the uncertainty. To enhance our sensitivity we have therefore implemented full digital pulse processing, enabling much higher time resolution in the pileup rejection circuit,.

At present, our high accuracy RBS method relies on samples where the a-Si yield can be used as an intrinsic measurement standard for Q $\Omega$ . But with sufficient effort Q $\Omega$  can be measured directly at high accuracy. A retractable Faraday cup has been installed and we will present data to show that this can be used to reliably infer total collected charge on the sample.

Finally, we have recently implemented rough focussing ( $^{0.4}$  mm for 1.5 MeV  $^{4}$ He<sup>+</sup> beam) and scanning over 5 mm into the millibeam chamber with the 6-axis goniometer [4], allowing us to

scan the beam across both a piece of a-Si (to determine  $Q\Omega$ ) and the sample of interest. Then any general sample can be analysed with a known  $Q\Omega$ . In particular, our high accuracy RBS method can be extended to the important case of certifying the thickness of self-supported thin foils for calibrating XRF. The importance of properly handling the dead times in such measurements will be discussed.

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### PA-111: Swift Heavy Ion irradiation induced interface mixing of Co/Sb bilayer thin films

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In the present work, the synthesis of Co-Sb alloys using swift heavy ion beam induced mixing of bilayer Co/Sb system is investigated. Co-Sb alloys are known to be good thermoelectric material in mid temperature range (400-700K). The bilayers of Co (~50 nm)/Sb (~100 nm) thin films were prepared by successive thermal evaporation of Co and Sb respectively over quartz substrate. Mixing of bilayer were studied by (i) annealing in temperature range 200°C to 400°C in the presence of atmosphere of Ar+2%H<sub>2</sub> for 1 hour, and (ii) irradiating with 100 MeV Ag ions with 1×10<sup>14</sup> ions/cm<sup>2</sup> fluence and post irradiation annealing in the temperature range 200°C to 400°C in the atmosphere of  $Ar+2\%H_2$  for 1 hour. All the samples were characterized by Rutherford Backscattering Spectrometry (RBS) and High Resolution X-ray diffraction (HRXRD). In case of annealed films, mixing of bilayer Co/Sb thin film is observed at 400°C while for high energy post irradiated annealed samples mixing take place at 200°C and at 400°C complete mixing is observed forming CoSb<sub>2</sub> and CoSb<sub>3</sub> phases. Therefore, thermodynamical growth of Co-Sb alloys are modified in irradiated samples at much lower post irradiation annealing temperatures than that observed for just annealed samples. The mechanism governing the formation of Co-Sb alloy in post irradiated annealed samples may be attributed to 'radiation enhanced diffusion'. To study the effect of interface mixing on thermoelectric properties Co-Sb system resistivity and thermopower measurement were also done using four probe and bridge method, respectively. Thermoelectric Power of 100 MeV Ag ion irradiated samples annealed at 400°C, was found to be higher than pristine and just annealed samples.

### PA-112: Modelling the diffusion of iron in silicon dioxide thin films: influence of excess oxygen

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In micro- and nano- elecotronics device fabrication, and particularly 3D designs, the diffusion of metal into sublayers during annealing needs to be minimized as it is usually detrimental to the device characteristics. Diffusion also impacts the formation and growth of nanoprecipitate in solid matrices. In this presentation, the diffusion behavior of low energy, low fluence, ion implanted iron into a thermally grown silicon oxide layer on silicon is investigated. Ex-situ Rutherford backscattering spectrometry (RBS) and particle induced X-ray emission (PIXE) is used to monitor the diffusion profile and the remaining iron content near the surface. Different ion beam analysis and imaging techniques were used. Magnetization measurements were also undertaken to evidence the nanocrystalline ordering. While standard vacuum furnace annealing and electron beam annealing lead to fast diffusion of the implanted species towards the Si/SiO<sub>2</sub> interface, we show that furnace annealing in an oxygen rich atmosphere prevents the diffusion of iron that, in turn, limit the growth of the nanoparticles. The diffusion and particle growth is also greatly reduced when oxygen atoms are implanted in the  $SiO_2$  prior to Fe implantation effectively acting as a diffusion barrier. The excess oxygen is hypothesized to trap Fe atoms and reduce their mean free path during the diffusion. A simple Monte-Carlo simulation of the diffusion which considers the random walk of Fe atoms is presented and compared with the data obtained from ion beam analysis.

### PA-113: Characterization of 95 MeV O<sup>6+</sup> ions induced modifications in PVDF polymer

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PVDF is a specialty plastic material in the fluoropolymer family. It has an easier melt process because of its relatively low melting point, low density and low cost compared to the other fluoropolymers. The passage of heavy ions in polymeric material produces a lattice deformation. The deformations may be in the form of latent tracks or may vanish by self-annealing in time. The availability of heavy ion beam from accelerator has brought new impetus
to the field of ion beam modifications in the electrical, chemical and optical properties of polymeric materials have been observed as a result of irradiation with swift heavy ion (SHI). Modification depends on the ion, its energy and fluence and the material itself. In the present study, Optical, chemical, structural and electrical changes induced by 95 MeV O<sup>6+</sup> ions in poly (vinylidene difluoride), PVDF films are studied by UV-Visible (UV-Vis) Fourier Transform Infra Red spectroscopy, and X-Ray Diffraction. Dielectric constant, dielectric loss (tan $\delta$ ) and a.c. conductivity measurements are carried out with a variable frequency LCR meter (Hewlett Packard 4284 A) in the frequency range of  $(1-10)x10^5$  Hz at room temperature. Irradiation was carried out under high vacuum of the order of 4 x  $10^{-6}$  Torr with oxygen ions from Pelletron accelerator at Inter University Accelerator Centre (IUAC), New Delhi, India to the fluences of 10<sup>10</sup>,10<sup>11</sup>, 10<sup>12</sup>, and 10<sup>13</sup> ions/cm<sup>2</sup>. A slight shift in the optical absorption edge towards the red end of the spectrum was observed with the increase in ion fluence. The optical band gap (Eg), calculated from the absorption edge of the UV-Vis spectra of these films in 200-800 nm region varied from 3.66 eV to 2.63 eV for pristine and irradiated samples. The cluster size varied in a range of 88 to 170 carbon atoms per cluster. In FTIR spectra, appreciable modification in terms of breaking of the cleavage through the ion energy deposition breaks the carbon – hydrogen and carbon -fluorine chemical bonds, produces a high rate of molecular emission of hydrogen and hydrofluoric acid and transforms the residual material in a carbon amorphous film. XRD analyses show significant change in crystallinity with fluence.

### PA-114: 2D and 3D imaging of Biological Cells and Tissue using pseudo-continuous keV SIMS

### Alex Henderson

### University of Manchester, Manchester, UK

Cluster ion beams have revolutionised keV SIMS analysis in the biology and biochemistry sector. The use of C60 (fullerene) as a primary ion source showed that sputtering of the sample surface did not cause undue damage to subsurface material. This allowed for 3 dimensional analysis of delicate material to be performed. In recent years, the use of giant gas cluster (GCIB) sources as a sputter source, together with an alternate analysis source, in a dual-beam analysis mode, has allowed for the gentle removal of sample layers with an interleaved imaging capability. This leads to snapshots of high spatial resolution images with regions of unknown chemistry between. Recently, pulsed GCIB sources have been developed to allow for spectrometry with these clusters, typically Ar500-5000, with good spectral resolution and sensitivity. However, the use of a pulsed primary ion source, in a ToF mass analyser configuration, results in a low duty cycle, leading to long analysis times for non-snapshot-style experiments.

The Ionoptika J105 Chemical Imager instrument, developed as a collaboration between Ionoptika Ltd. and the University of Manchester, uses a pseudo-continuous primary ion beam and pulses (bunches) the secondary ion flux. This has the benefit of a high sample erosion rate with the parallel collection efficiency of the ToF analyser. The increased duty cycle of the J105, coupled with the gentle sputtering mechanism of C60 and GCIB primary ion sources, leads to enhanced usefulness for large, easily damaged samples, such as those found in biology. In this contribution we will show examples of the benefits of the instrument configuration to generate data from biological cells and tissue.

ABSTRACTS POSTER SESSION 2 Tuesday

### PB-1: Influence of radiation damage on the thermal properties of silicon carbide implanted with heavy noble gas ions

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Fuel elements of high-temperature nuclear reactors make use of TRISO fuel particles, which are encapsulated by CVD-layers of pyrolitic carbon and silicon carbide to limit fission product release into the primary cooling gas system. The silicon carbide coating is the main barrier for most of the important isotopes produced by nuclear fission. TRISO fuel particles retain effectively most of the important fission products up to temperatures of 1000 °C. Currently design studies are considered for the International Generation IV Reactor Program, operating at temperatures significantly above 1000 °C to enhance the efficiency, especially in view of process heat applications for hydrogen generation. However, little reliable information on transport properties of fission products in and thermal stability of silicon carbide above this temperature is available. This investigation is part of a systematic study on transport properties of silicon carbide for environmentally relevant isotopes and its thermal stability at temperatures up to 1400 °C and the influence of radiation damage on it. For this work krypton and xenon ions were implanted in poly and single crystalline silicon carbide samples at temperatures ranging from room temperature to 600 °C. The different implantation temperatures make it possible to compare transport processes and thermal etching in amorphous, poly and single crystalline silicon carbide. Diffusion coefficients are extracted from implantation profile broadening after isochronal and isothermal annealing studies, using RBS/channeling analysis. Surface deterioration is investigated by scanning electron microscopy, using an in-lens detection system for high-contrast imaging. The influence of radiation damage on diffusion and thermal etching is studied by comparing it with the evolution of defect profiles in 6H-SiC during annealing, employing a modified version of the computer code DICADA [1,2].

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### PB-2: Behavior of Li on graphene surfaces observed using high-resolution ERDA

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Lithium ion batteries are widely used as power sources for portable electronics, electric vehicles etc. Because intercalation of lithium into the electrodes is one of the key processes in the Li ion batteries, understanding of the intercalation mechanism is of prime importance to improve battery performance. To this end, precise analysis of Li at the electrode surfaces is

required. Elastic recoil detection analysis (ERDA) is one of the powerful techniques for depth profiling of light elements. Recently, we have developed a compact high-resolution ERDA system for Li depth profiling [1]. We employed several hundred keV He ions as primary ions and a magnetic spectrometer for precise energy analysis. In this presentation, we observe the behavior of Li deposited on the surface of few-layer graphene films using high-resolution ERDA. The graphene films were grown on copper substrates by microwave plasma CVD. A small amount of lithium ( $\sim 1 \times 10^{15}$  atoms/cm<sup>2</sup>) was deposited on the graphene surfaces using a Li dispenser (SAES getters) at room temperature. As a reference, similar amount of Li was also deposited on a copper substrate, where the deposited Li atoms stay on the surface. Figure 1 shows examples of the observed high-resolution ERDA spectra using 200 keV He<sup>+</sup> ions. The closed and open circles show the results for Li/graphene and Li/copper, respectively. The Li/copper sample shows a sharp peak at 152 keV, showing that all deposited Li atoms stay on the surface. On the contrary, the Li/graphene sample shows a broad peak, which was decomposed into contributions of individual layers. This indicates that only a part of the deposited Li atoms stay on the surface and the rest of the Li atoms are intercalated into the graphene sheet and distribute almost uniformly from the surface down to  $\sim$  2nm.



[1] M. Nikko, K. Nakajima and K. Kimura, Nucl. Instrum. Methods Phys. Res., Sect. B in press.

# PB-3: Relevance of ERDA and RBS for Tungsten Tracing in Wall Materials from Fusion Experiments

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Interactions between fusion plasmas and the surrounding wall constitute one of the limiting factors for the performance of future fusion reactors. During these interactions, wall materials are modified by heat loads and particle fluxes. In order to better understand the processes of erosion, material transport and deposition that follow, many wall components are removed after plasma exposure and the atomic composition in their surface layer is studied. Rutherford Backscattering (RBS) serves a basic technique to perform such analyses with an accuracy in the order of percents. Heavy ion elastic recoil detection analysis with time-of-flight measurement (ToF-HIERDA) allows identification and depth profiling of most species in the near surface region (up to 1 micrometer) in a single experiment. Furthermore, it doesn't suffer the same mixing of depth and mass information as does RBS. The aim of this contribution is to report on the scientific relevance of the above mentioned ion beam methods for a few applications related to plasma-material interaction studies. In particular, the quantification and depth profiling of tungsten (W) in materials tested for a reactor wall or irradiated with high-Z ion beams are in focus. The first application to be demonstrated is related to plasma exposed Eurofer steels. These materials are considered for future fusion devices, but plasma sputtering causes an enrichment of tungsten on the surface which needs to be characterized. Secondly, samples from dedicated tungsten transport experiments in the TEXTOR tokamak are considered and the possibility to analyze depth profiles and deposition patterns of that tungsten is shown. Finally, the surface layer composition of tungsten implanted molybdenum mirrors is investigated. All these examples point out the broad range of applicability of RBS and ERDA and the continued importance of these methods for fusion research.

### PB-4: The potential of ion beams for characterization of metal-organic frameworks

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Metal-organic frameworks (MOFs) feature unique capabilities in the field of catalysis due to their chemical versatility and extraordinary high surface area. The molecular nature of MOFs allows the incorporation of a molecular catalyst which results in a new material that can exhibit increased catalytic reactivity and stability. In this context, it is of great interest to characterize the distribution of catalysts within a MOF. The low density and the organic character of MOFs make them a challenging substrate for depth profiling, as it is sensitive to a number of commonly used composition depth profiling methods. In this contribution, we present an investigation of the potential of ion beam analysis tools for compositional analysis of MOFs. The system investigated consisted of UiO66 (UiO = University of Oslo) metal organic thin films on p-type Si wavers with a post-synthetically incorporated molecular proton reduction catalyst Fe<sub>2</sub>(dcbdt)(CO)<sub>6</sub> (dcbdt = 2,3-dithiolato-1,4-benzenedicarboxylic acid). We have characterized samples by Rutherford Backscattering spectrometry (RBS), Time-Of-Flight Elastic Recoil Detection analysis (TOF-ERDA) and by Time-Of-Flight medium Energy Ion Scattering (TOF-MEIS). The beam induced sample modification during the analysis has been characterized by Scanning Electron Microscopy (SEM). The obtained results indicate that ion beams can be used for such

kind of analysis, but beam damage has to be expected under certain conditions. We discuss the quality of the information obtained from the individual methods. We also discuss restrictions in sample structure and its impact on the analysis.

### PB-5: Withdrawn

### PB-6: Investigation of the helium diffusion in Ti<sub>3</sub>SiC<sub>2</sub> by nuclear reaction analysis

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(3) Institute of Nuclear Physics and Chemistry, Chinese Academy of Engineering Physics, Chengdu, 610003, China

(4) Institute of Plasma Physics, Chinese Academy of Sciences, P.O. Box 1126, Hefei 230031, China Ti<sub>3</sub>SiC<sub>2</sub> MAX phase has been showing potential as a promising candidate structural material for use in future fission and fusion programs (e.g. Gen IV and ITER). The helium diffusion constant in materials is necessary to investigate for predicting the long-term behavior of the material. The helium migration in  $Ti_3SiC_2$  has been studied using the resonant <sup>3</sup>He (d, p) <sup>4</sup>He nuclear reaction. 3-MeV <sup>3</sup>He ions are implanted in  $Ti_3SiC_2$  at the depth around 7.6  $\mu$ m. The excitation curves by measuring the 13-MeV proton yields as a function of the incident deuteron energy were used as data processing method. The characteristics of the <sup>3</sup>He profiles obtained by the analysis of nuclear reaction are compared with SRIM calculations. The samples were annealed at the temperatures from 400 °C to 1100 °C. Diffusion constant can be deduced from the depth profile standard deviations of the same sample at two consecutive annealing temperatures. The diffusion behavior of He in the Ti<sub>3</sub>SiC<sub>2</sub> strongly depends on the annealing temperature. At lower annealing temperature such as 400 °C, He diffusion was dominated by the dissociative mechanism, and most of dissociated He atoms are released or diffusion into depth. With increasing annealing temperature, some of defects including Ti and C antisite defects begin to annihilitate due to increased movibility of C and Ti vacancies. At the same time, He<sub>m</sub>V<sub>n</sub> clusters or bubbles grow up, and phase decomposition occurs at some certain temperatures (600 °C and 1100 °C) as well. So the annealing temperature is key factor to determine both the depth profile broadening and moving direction of the profile.

## PB-7: Structural changes and defects evolution in $Ti_3AlC_2$ induced by 500 keV He-ions irradiation

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The MAX phases, and more particularly  $Ti_3AlC_2$ , combine the properties of metals with those usually attributed to ceramics, such as excellent mechanical properties and high-temperature stability (>1000 °C). These properties, arising from their specific structure composed of ceramic octahedral (i.g.  $TiC_8$ ) and a single intercalated metallic layer (i.g. Al layer), make them ideal for applications under extreme radiation conditions. Therefore, it may be used as not only the first-wall candidate material in the fusion reactor (ITER) but also the structural material in the fission programmes (ADS and Gen IV).

The studied samples consisting of about 99.5%  $Ti_3AlC_2$  were obtained with a hot isostatic pressing (HIP) method. And irradiation experiments with He<sup>2+</sup> of 500 keV were performed at different temperatures and influences under 320 kV multi-discipline research platform in the Institute of Modern Physics, Chinese Academy of Sciences (IMP, CAS), Lanzhou. All the samples studied in this work were characterized by low-incidence X-ray diffraction and analyzed by Positron Annihilation Spectroscopy (PAS), respectively.

The observed diffraction patterns revealed apparently the formation of new phases such as TiCx and Ti<sub>3</sub>Al within the matrix caused by irradiation. Moreover, the change in peak broadening was also indicative of the presence of a disordered phase, but the amorphous phase didn't occur in all the irradiated samples even at the fluence of  $1.0 \times 10^{18}$  ions/cm<sup>2</sup>. Besides, an expansion of the hexagonal close-packed lattice along the c axis increases with the influence at the same temperature. In addition, for the sample irradiated by He<sup>2+</sup> with a fluence of  $1 \times 10^{18}$  ions/cm<sup>2</sup>, S parameter was higher than that for all the other samples including the virgin one, which clearly indicates that positrons detect the presence of vacancy-type defects generated during irradiation of He in Ti<sub>3</sub>AlC<sub>2</sub>. Meanwhile, the combination of He-Vacancy was related to the lower density of defects induced by irradiation. Moreover, the recovery phenomenon of defects was evidently at high temperatures.

### PB-8: Temperature effects on co-implanted nanostructured W coatings

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One of the challenges in the design of future nuclear power plants is to develop materials capable to resist the hostile conditions of a nuclear fusion reactor. Because of its properties tungsten (W) is considered to be one of the best candidate for plasma facing material [1,2]. Nevertheless, it is demonstrated that light species tend to nucleate in radiation induced vacancies/vacancy clusters forming overpressurized bubbles that could lead to surface blistering and exfoliation in coarse grained W [3,4]. One possible alternative to delay the appearance of these undesirable effects is the use of nanostructured materials [5-7].

In this work we study the influence of sample microstructure on the temperature dependence of the H in W. For this purpose homemade pure  $\alpha$  nanostructured W (NW) films deposited by magnetron sputtering [8] and commercial coarse grained W (CGW) samples were sequentially

implanted at the Helmholtz-Zentrum Dresden-Rossendorf (HZDR) with C and H at energies of 665 and 170 keV, respectively, both at a fluence of  $5 \times 10^{16}$  cm<sup>-2</sup> and at room temperature. After implantation some of the samples were annealed at 373 K, 473 K and 573 K for 30 minutes. Scanning electron microscopy images evidence that: (i) NW samples preserve their nanometric features, and (ii) no sign of blistering is observed in any of the NW and CGW samples. X-ray diffraction patterns show the absence of secondary phases upon implantation and annealing. The H depth profile of the pristine and annealed samples was characterized by resonant nuclear reaction analysis using the <sup>15</sup>N(p, $\alpha\gamma$ )<sup>12</sup>C nuclear reaction. RNRA data reveal that there is not significant H diffusion in NW at 373 K and 473 K whereas in CGW a certain mobility of H is observed at 373 K. The role of grain boundaries in radiation induced damage and in the H behavior is studied by Object Kinetic Monte Carlo (OKMC) by comparing the experimental and calculated results.

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# PB-9: Thermal migration of Deuterium implanted in graphite: influence of free surface proximity and structure

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This paper is a contribution to the study of the behavior of activation products produced in irradiated nuclear graphite, graphite being the moderator of the first French generation of CO2 cooled nuclear fission reactors. Among them, Tritium is a major contributor to the initial activity of this graphite. So, this paper is focused on the thermal release of Tritium, taking into account the role of the free surfaces (open pores and graphite surface). For that purpose, two kinds of graphite were compared. On one hand, Highly Oriented Pyrolitic Graphite (HOPG), a model well graphitized graphite, and on the other hand, SLA2, a porous nuclear graphite (around 25% porosity) made of well graphitized coke grains and of much less graphitized and porous binder matrix. Deuterium ion implantation at three different energies 70, 200 and 390 keV allows simulating the presence of tritium at three different depths, corresponding respectively to projected ranges  $R_p$  of 0.75, 1.7 and 3.2 µm. The D isotopic tracing is performed thanks to the  $D(^{3}He,p)^{4}He$  nuclear reaction, with a millimetric beam at the 4 MV Van de Graaff accelerator of

IPNL (Institut de Physique Nucléaire de Lyon, France). The graphite structure is studied by Raman microspectrometry. Thermal annealing is performed in the temperature range 200-1200°C up to 300h annealing time. As observed in a previous study, the results show that the D release occurs according to three kinetic regimes: a rapid permeation through open pores, a transient regime corresponding to detrapping and diffusion of D located at low energy sites correlated to the edges of crystallites and finally a saturation regime attributed to detrapping of interstitial D located at high energy sites inside the crystallites. Below 600°C, D release is negligible whatever the implantation depth and the graphite type. The present paper clearly puts forward that above 600°C, for a given temperature, the D release decreases at deeper implantation depths and strongly depends on the graphite structure. In HOPG where high energy sites are more abundant, the D release is less dependent on the surface proximity compared to SLA2. In SLA2, in which the low energy sites prevail, the D release curves are clearly shifted towards lower temperatures when D is located close to free surfaces, i.e for  $R_{\rm p}$ equal to 0.75 µm. Extrapolating our data to Tritium mobility in irradiated graphite, our results show that thermal selective extraction of T would be all the more so efficient as the graphite structure is more disordered, which means in the most irradiated and damaged graphite zones in the reactor.

### PB-10: Capabilities and limitations of Ion Beam Analysis techniques to study the Li distribution in Lithium-ion batteries

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The development of efficient storage technologies for electrical energy plays an important role mainly in the progress of portable consumer electronics and electric vehicles. Nowadays, Li-ion batteries are attractive candidates for these applications since they can provide high energy and high power densities. The performance of a Li-ion battery (LIB) is mainly governed by the Li<sup>+</sup> exchange mechanisms. Thus, for LIBs improvement it is essential to characterize the Lithium behavior. However, Li characterization is not a trivial task, in particular, if depth resolution is required. Most of the traditional used techniques for Li characterization give information about the atomic or in the macroscopic scale. Nevertheless the lithium behavior at the atomic scale is not mirrored at the macroscopic scale. Therefore, the Li characterization in the mesoscopic scale would be of help to understand and to connect the mechanism taking place in the two previous ones. We show the capabilities and limitations of ion beam analysis (IBA) techniques to study the distribution in the mesoscopic scale of lithium and other elements present in the

electrodes. The potential of (p, p) and (p,  $\gamma$ ) nuclear reactions for Li characterization in real batteries (based on LiFePO<sub>4</sub> and Li<sub>x</sub>Ni<sub>0.8</sub>Co<sub>0.15</sub>Al<sub>0.05</sub>O<sub>2</sub> (0.75  $\leq x \leq 1.0$ )) is illustrated. The advantages of the use of IBA in comparison to more traditional techniques for electrode characterization are discussed.

### PB-11: Detailed study of the tokamak plasma exposed carbon tails by IBA methods

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A set of metal covered carbon tails exposed to COMPASS tokamak plasma have been studied at INP Rez by various IBA analysis (PIXE, RBS, ERDA, width protons and helium beams). The aim of the study was to investigate the corrosion and deposition rates and depth profiles of hydrogen and deuterium in tails surface layer. The tails has been placed for long time (17 months) at the inner wall of the tokamak vacuum vessel. During that time approximately 4200 plasma shots have been realized within more or less standard condition. Additionally eight boronization procedures, using about 2 cm<sup>3</sup> of carboran (C<sub>2</sub>B<sub>10</sub>H<sub>12</sub>), has been carried out at that period, resulting in deposition of boron rich layer over the tails metallic surface. The differences in the build-up layer properties for the different tail coatings (W, Ni and P92 steel) were minor. The 2.6 MeV protons were used for a determination of the boron layer and metallic layer compositions and thicknesses, including the trace element content. The hydrogen and deuterium depth profiles were measured by ERDA using 2.6 MeV and 5 MeV alpha particles. Details about the experiment will be presented in our poster.

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PB-12: Withdrawn

### PB-13: Application of a co-sputtering simulation code (CO-SS) to study sputtering yield amplification

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Sputtering Yield Amplification is a phenomenon discovered by S. Berg et al in 1991. This phenomenon occurs during the deposition of thin films by DC co-sputtering from a target composed of two pieces of different materials. If the difference in the atomic weight of the elements of the two parts of the target is large, then the sputtering yield of the lightest element has been observed to be greater than the normal expected value. However, currently there is no adequate phenomenological and theoretical explanation of this phenomenon. The co-sputtering simulation code, known as CO-SS, is a freeware program which was developed by the authors to simulate the spatial variation of the deposition by DC magnetron sputtering and co-sputtering. Thin films of AlTi, WC were produced to study the sputtering amplification experimentally and by modelling using the CO-SS code. The spatial variation of the thickness and compositions of the films was determined by the <sup>4</sup>He RBS technique. The results showed the existence of the previously mentioned sputtering yield amplification.

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# PB-14: Ion beam analysis and co-sputtering simulation (CO-SS) of bi-metal films produced by magnetron co-sputtering

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Magnetron sputtering is widely used to produce thin films on different types of substrates. An important application of this method is to make multicomponent thin films where two or more elements are included in the cathode. The thickness and chemical composition of the films depend on the experimental parameters of the sputtering, the system geometry and the spatial distribution of the elements in the target. If the target is a fine mix of the components the target and the deposit have the same composition, however, a new target is needed to vary the

composition. If the target is made of two, or more, spatially separate pieces of the materials then the composition of the deposit depends on a combination of the relative areas and the sputtering yield of each material and the procedure is called co-sputtering. The CO-SS code models the angular distribution of particles ejected by sputtering from the target, where this is assumed to vary as  $\cos\beta$ , where  $\beta$  is the angle of ejection relative to the normal of the surface of the target. The code uses as input parameters the variables from the experimental setup; the minimum and maximum diameters of the erosion racetrack, the target-substrate distance, Dt-s, the relative areas of the two metals, R = Area of Ti / area of Al, and the sputtering yields,  $Y_{sp}(Ti/Al)$ , of the two materials. The spatial thickness and composition of thin films of Ti and Al produced by DC magnetron co-sputtering were modelled using the CO-SS code, as a function of R and Dt-s. The spatial variation of the thickness and the composition of the films were determined by a standard 2 MeV <sup>4</sup>He RBS technique. There was very good agreement between the experimental results and those predicted by the CO-SS code.

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### PB-15: Hydrogen ion-implantation induced low resistive layer in KNbO<sub>3</sub> bulk single crystal: Evaluation by elastic recoil detection analysis

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The ferroelectric field-effect transistor uses the surface conduction of insulating ferroelectric under the gate oxide. In the present study, we artificially form a surface conducting layer in KNbO<sub>3</sub> (perovskite structure, a=5.697, b=3.971, and c=5.722 Å at room temperature) by the hydrogen ion-implantation, as was formed in the hydrogen ion-implanted ZnO [1]. The energy band gap of KNbO<sub>3</sub> estimated by optical absorption is 3.4 eV. Hydrogen is implanted into KNbO<sub>3</sub> bulk single crystals using the energy (the peak ion fluence) of 500 keV ( $5.0 \times 10^{15}$  cm<sup>-2</sup>) according to the TRIM (Transport of Ions in Matter) simulation of the projected range on the ions [2]. The simulated implantation depth and the H concentration are 3650 nm and  $1.65 \times$ 10<sup>20</sup> cm<sup>-3</sup>, respectively. In resistivity measurements using Van der Pauw technique, electrodes are fabricated using titanium/gold. The sheet resistance decreases from ~  $10^8$   $\Omega/\Box$  for unimplanted samples to 2.33 × 10<sup>5</sup>  $\Omega/\Box$  for as-implanted samples, 2.29 × 10<sup>5</sup>  $\Omega/\Box$  for 100 °C annealed samples, and 4.25  $\times$  10<sup>5</sup>  $\Omega/\Box$  for 150 °C annealed samples. Hydrogen is evaluated by the elastic recoil detection analysis (ERDA) using a 1.5 MeV <sup>4</sup>He<sup>+</sup> beam. In the present ERDA experiments, hydrogen can be detected up to 100 nm in depth. In as-implanted samples, hydrogen is observed in a range from the surface to 100 nm, indicating that the backward diffusion is caused under the implantation process. Furthermore the backward diffusion of hydrogen is enhanced by 150 °C annealing for 1hr. The slight increase in sheet resistance in the 150 °C annealed samples would be related to the out diffusion of hydrogen. The low resistive layer induced in H ion-implanted KNbO<sub>3</sub> suggests the existence of a shallow energy level related to hydrogen interstitials.

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# PB-16: Corrosion and wear resistance in Simulated Body Fluid for TiAlPtN/TiAlN/TiAl multilayer films deposited over a CoCrMo substrate by means of Plasma Enhanced Magnetron Sputtering method

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The goal of the present work is to find out whether the addition of Pt in TiAIN/TiAI multilayer films of 10 cycles deposited over a CoCrMo substrate helps to improve the corrosion resistance in Simulated Body Fluid (SBF) and to discern if such improvement reduces the wear resistance of the films in the same fluid. To achieve the objective, 7 different samples were deposited using a Plasma Enhanced Magnetron Sputtering method with a TiAl cathode (50 % Ti and 50 % Al), a Pt cathode and a controlled atmosphere of Ar that could be altered to a mixture of  $Ar/N_2$ . The different samples were obtained by co-deposition with the two cathodes polarized, and changing the time in which the Pt cathode was expelling particles, therefore obtaining different layers with Pt with different depths and concentrations. IBA was used to measure the atomic samples profiles. Also the samples were characterized with tribological experiments, such as Vickers micro-hardness testing and reciprocating tests on SBF, and corrosion resistance test on SBF such as Potentio-dynamic tests (PD) an Open Circuit Potential tests (OCP) In the reciprocating test the wear track was recorded, along with the ousted volume and the changes in the OCP during the whole test. These results were compared with those obtained in the PD tests in which the corrosion resistance current and the corrosion potential were measured using a Tafel fitting. The multilayer film with the best results (lower corrosion resistance current, less pitting potentials and lower ousted volume) was observed to be that with 10 minutes of polarization for the Pt in a cycle of 30 minutes. A discussion of the changes in the wear mechanisms and a model that describes this behavior as a function of the total Pt layer thickness is presented.

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# PB-17: Boundary estimation of statistical uncertainties in Rutherford backscattering spectroscopy analysis of rare earth doped high silica glasses

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Rutherford backscattering spectrometry (RBS) is a well-known analytical method with high efficiency in terms of detection limit and elemental selectivity. Count-rate experiments like RBS are statistical processes and are affected by statistical variations of the measured yield. In general these statistical uncertainties are very predictable and can be estimated by a simple probabilistic theory. However the inverse problem of estimating a reliable uncertainty of elemental compositions based on statistical fluctuations of measured spectra is a more complicated task. In this work several methods and thoughts about the uncertainty of the compositions determined by RBS are presented. The methods are based on statistical probabilistic theory or simply on best practice knowledge. The results of the different methods are given for experimental data from RBS analysis of high silica glasses doped with the rare earths La and Yb. Additionally the obtained elemental compositions including their uncertainties are compared with those resulting from wavelength-dispersive electron probe microanalysis (WD-EPMA).

### PB-18: Investigation of spatial elemental distribution in optical fibers with NRA and RBS

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The use as optical fibers in telecommunication and fiber lasers is a major application of fused silica. Such wave guiding applications require well defined radial profiles of the optical refraction index, which are achieved by doping with specific impurities. As one of the most effective chemical dopants for fused silica, fluorine is successfully applied in the production of optical fibers. It lowers the optical refractive index and has the advantage that no disturbing optical absorption bands are generated and therefore the transmission is not influenced by absorption over a wide range of wavelengths. In general the production of optical fibers and fiber lasers requires physically extreme conditions such as very high temperatures (usually above 1000°C). Thus the distribution of the dopants may change during the manufacturing by processes as for instance thermal diffusion. A common technique for studying doped fused silica is energy and wavelength dispersive x-ray spectrometry (EDX/WDX). However, fluorine has a low EDX/WDX detection yield and the optical changes connected with a redistribution of the fluorine atoms are hard to measure in case of fibers with the necessary spatial resolution of few micrometers. A possible solution is the detection of fluorine by nuclear reaction analysis

(NRA) using high-energetic protons in combination with a micro beam. Therefore the distribution of fluorine and other dopants of interest were investigated in industrial-grade optical fibers and fiber-preforms with the nuclear microprobe LIPSION and macro ion beams, respectively, using NRA and Rutherford backscattering spectrometry (RBS). Spatial distributions of various elements near the detection limit were analysed. The application of analytical methods with high detection yields (RBS) and low detection yields (NRA) in a single spectrum induces statistical problems in the simultaneous evaluation of the experimental data. A Poisson probabilistic approach is used to overcome these statistical problems and to investigate the spatial distribution of all intended dopants. Thus it is possible to generate a direct image of the chemical composition of industrially manufactured optical fibers.

### PB-19: Rutherford backscattering/channeling study of the implanted iron in silicon

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A combination of Rutherford backscattering spectrometry with channeling technique (RBS/channeling) was used to study the radiation damage produced due to the ions implantation. Since this technique have a high sensitivity to any lattice perturbation, including the detection of point and extended defects, impurities and amorphous layers. The created defects under irradiation and subsequent processes influence the properties of the material. We studied the behavior of iron in silicon, since iron is unwanted impurity. These deep centers have a detrimental influence on device properties, but it can be employed to produce semiinsulating layers. In the present study, the 100 keV ions of Fe produced from the high-dose ion implanter were implanted with various doses. Crystals were tilted off any major direction during irradiation to avoid channeling effects. To conduct such measurements, the sample mounted on a goniometer so that the crystal axis direction aligned with the incident beam. The aligning procedure was carried out with measuring the channeling dips in the angular distribution. Rutherford backscattering spectroscopy in channeling condition (RBS/C) was performed using a 1 MeV H<sup>+</sup> ion beam delivered by the on electrostatic accelerator of the Institute of Applied Physics of NAS of Ukraine. The channeling experiment system coupling with computer-based automatic control has been set up. Standard surface barrier detectors have been used to record energy spectra of a silicon crystal obtained in random and aligned geometries. Our study confirmed that Fe in Si is able to occupy substitutional, displaced substitutional and interstitial positions. But the dominant lattice sites of Fe are substitutional. Ion implantation induced damage in the samples and extent of amorphization depends on ion energy, implantation dose and flux of irradiation. The high fluence leads to the formation of a continuous amorphous layer. Therefore, it is worthwhile to study the optimal condition of the defects creation and annihilation in the ion implantation. Channeling spectra are well reproduced in computer simulations, confirming our understanding of the propagation of these ions in crystals.

### PB-20: Formation of Nanostructures on the Surface of TiO<sub>2</sub> Films due to Swift Heavy Ion Irradiation

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Swift Heavy Ion (SHI) irradiation is an excellent technique for creation of surface nanostructures. The surface topography of thin films can be modified in a controlled way by choosing suitable ion species, ion beam and irradiation fluence. During irradiation the SHI deposits it's energy to the target mainly through electronic energy loss. Intense electronic excitation of a confined volume around the ion trajectory because of the electronic stopping of the SHI results in nano patterning of the film surface. In the present investigation, polycrystalline thin films of  $TiO_2$  are irradiated by 100 MeV Au ion beam at various fluence. The surface topography of the films before and after irradiation is investigated by Atomic Force Microscopy (AFM). The AFM results indicate that the grain fragmentation occurs due to ion impact resulting in formation of nanostructures on the surface of films. After irradiation the grains assemble in various configurations. The grain size is found to decrease with increasing fluence. GAXRD studies are carried out to study structural phase transition. The as deposited film exhibits rutile phase of TiO<sub>2</sub>. The irradiated films exhibit dominant rutile phase with little traces of anatase phase. GAXRD studies also indicate decrease in grain size after irradiation. The optical characterization is carried out using UV-Vis spectroscopy. It is found that the bandgap values estimated on the basis of UV-Vis spectroscopy increase with increasing fluence indicating nanostructure formation. The reduction in grain size after irradiation can be explained in terms of ion induced stress generation and change in surface energy due to energy deposited by ion beam.

### PB-21: Metal ion implantated GaN study for spintronics applications

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The present generation of semiconductor electronic and photonic devices is based on transporting a charge of electrons and holes. The field of semiconductor spintronics seeks to exploit the spin of charge carriers in a new generation of transistors and lasers, which are going to be employed in ultra-low power high-speed memory and photonic devices. The practical

developments of these novel spin-based devices depend on the availability of materials with magnetic ordering temperature above room temperature. In this contribution we present the growth and physical characterization of metal doped GaN epitaxial layers. In this work, GaN layers (0001) crystallographic orientation, grown by low-pressure metal-organic vapor phase epitaxy (MOVPE) on c-plane sapphire substrates, were implanted with 200 keV Co<sup>+</sup>, Fe<sup>+</sup> and Ni<sup>+</sup> ions in the range of fluencies  $1 \times 10^{15} - 1 \times 10^{16}$  cm<sup>-2</sup>. Structural investigations are needed to understand the influence of defect distribution on crystal-matrix recovery and metal dopant positioning. The composition of the ion-implanted layers and concentration profiles of the implanted atoms were studied by Rutherford Back-Scattering spectrometry (RBS) and compared to secondary ion mass spectroscopy (SIMS) analysis. The profiles were compared to SRIM 2013 simulations. The structural properties of the ion-implanted layers were characterized by RBS-channeling and Raman spectroscopy to get the comprehensive insight into the structural modification of implanted GaN and to study the subsequent annealing influence on the structural properties. Photoluminescence measurement at variable temperatures was provided to control the desired optical properties of the prepared structures. Changes in the surface morphology caused by the ion implantation were examined by AFM. The post-implantation annealing can induce the metal diffusion or precipitation and the reconstruction of the surface which will be followed by the above mentioned analytical methods and HRTEM analysis.

The research has been realised at the CANAM (Center of Accelerators and Nuclear Analytical Methods) infrastructure and has been supported by project No. P108/12/G108.

### PB-22: Copper micro-rods fabrication using focused ion beam lithography

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Proton beam writing technique was recently introduced at 3 MV Tandetron accelerator at Nuclear Physics Institute in Rez (Czech Republic). It has been used, to produce threedimensional (3D) micro-structures in poly(methylmethacrylate) by 2.0 MeV protons microbeam. Fine micro-channels (52 micron x 52 micron) have been realized to be used as targets to generate electrons and ions acceleration in non-equilibrium plasmas by high power laser irradiation. After chemical etching, the quality of the bottom and side walls of the produced structures in PMMA were analysed using Scanning Transmission Ion Microscopy (STIM) and Atomic Force Microscopy (AFM) methods. Combining p-beam writing and electro-deposition, shaped micro-pillars were fabricated. Further investigation was carried out by using micro-PIXE method, to monitor the quality of the metallic pillar electrodeposited, as reported.

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# PB-23: Fabrication and Analysis of Polymer Microstructures through Ion Microprobe Techniques

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In this work we explore the capabilities of energetic focused beams of light ions for the analysis of microstructures produced on commercial polyethylene terephthalate (PET) foils. To that end, single lines and multi-structure patterns were drawn on the foils using Proton Beam Writing (PBW) techniques followed by chemical etching. The characterization of the microstructures was carried out with on-axis Scanning Transmission Microscopy (STIM) employing H+, He2+ and Li3+ ions in the MeV range. Scanning Electron Microscopy (SEM) was employed as well. Different etching times provided the kinetics of the etching procedure. The results indicate that basic variables like the speed of the chemical etching provided by the STIM measurements is well described regardless the ion used in the analysis. On the other hand, STIM images obtained from different regions of the ion energy spectra reveal patterns and cavities not seen by SEM. Moreover, striking differences are observed when different ions are used for STIM analysis. The results suggest that heavier ions provide additional information when compared with usual STIM employing protons.

# PB-24: Proton irradiation induced defects in GaN: Rutherfold backscattering and thermally stimulated current studies

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Proton irradiation into GaN was carried out in comparison with the neutron irradiation induced defects in GaN [1]. Proton irradiation may exclude the influence of neutron transmuted impurities such as Ge and C. A 500 keV proton irradiation was performed with a fluence of  $1 \times 10^{15}$  cm<sup>-2</sup> at room temperature. The proton beam passes through the 3-µm GaN single crystal film. The carrier concentration decreased three orders of magnitude to  $10^{15}$  cm<sup>-3</sup> by the proton irradiation. Hydrogen in samples was evaluated by elastic recoil detection analysis (ERDA) using a 1.5 MeV <sup>4</sup>He<sup>+</sup> beam. ERDA used here can observe hydrogen from the surface to the depth at ~100 nm. In un-irradiated, as-irradiated, and 200 °C annealed samples, the observed hydrogen concentration did not change much after the proton irradiation and the subsequent annealing, the electrical properties of GaN film would be related to proton irradiation induced defects. To evaluate the defects, thermally stimulated current (TSC) measurements were performed. A broad spectrum was observed at around 110 K. This spectrum can be divided into three traps,

P1 (peak temperature 104 K, ionization energy 173 meV), P2 (141 K, 251 meV), and P3 (178 K, 330 meV). P1 trap is related to the nitrogen vacancy and/or the defect complex involving nitrogen vacancy observed in electron- [2] and neutron-irradiated GaN [1]. This trap acts as an electron trap. P2 level is close to those of the calculated neutral Ga vacancy as the acceptor [3]. P3 level is related to the defect complex involving Ga vacancy. The Ga displacement concentration in samples was evaluated by Rutherford Backscattering Spectrometry (RBS) using a 1.5 MeV <sup>4</sup>He<sup>+</sup> beam. The minimum yield ( $\chi$ min) is 2.00 % for un-implanted samples and 2.04 % for as-implanted samples. The Ga displacement concentration evaluated from these  $\chi$ min values is 1.75×10<sup>19</sup> cm<sup>-3</sup>. Therefore the increase of resistance and reduction of the electron concentration due to proton irradiation would be related to these observed defects.

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### PB-25: Ion beam analysis of CaF<sub>2</sub> thin films deposited onto self-supported Al foils

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Thin calcium fluoride coatings deposited onto thin, self supported aluminum backings have been prepared by thermal evaporation. The achieved  $CaF_2/Al$  target samples were specifically designed for energy loss and energy loss straggling measurements that require a precise knowledge of target parameters (thickness, stoichiometry, purity and homogeneity degrees,..). Then, the  $CaF_2/AI$  targets have been extensively investigated both for their stoichiometry and their impurity contents by three IBA techniques: Rutherford backscattering (RBS), elastic backscattering (EBS) and nuclear reaction analysis (NRA) using, respectively, 1500 keV alpha particle, 2800 keV proton and 900 keV deuteron beams. Recorded corresponding particle spectra were simulated using the SIMNRA program, taking the scattering cross section data inputs from the IBANDL Nuclear Data Library. We found that homogenously distributed carbon impurities were detected within both the CaF<sub>2</sub> coatings and Al backings. Thickness value of ~  $23.3 \times 10^{18}$  atoms/cm<sup>2</sup> was derived for the CaF<sub>2</sub> coating with atomic stochiometry consisting of 30.5% Ca, 61% F and 8.5% C. Concerning the Al backing, a thickness value of ~ 45×10<sup>16</sup> atoms /cm<sup>2</sup> was determined with ~3.5% of C element contaminant. Besides, a layer of about 34×10<sup>15</sup> atoms/cm<sup>2</sup> of native oxide (Al<sub>2</sub>O<sub>3</sub>) film, arising from air-oxidation of Al surfaces, was also deduced. This thickness value is in good agreement both with the theoretical predictions and experimental observations cited in the literature.

### PB-26: Chemical speciation of phosphorus, sulfur and chlorine by means of high-energy resolution PIXE measurements

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High-resolution x-ray emission spectroscopy (XES) is directly probing the charge density around x-ray emitting atom and can be used complementary to x-ray absorption technique (XAS) to study electronic structure of selected element. Since spectral features, e.g. energy position and spectral shape are independent on the type of excitation, such analysis is not restricted to synchrotron facilities and can be performed also with laboratory excitation sources. In this work, 2 MeV proton beam was used to induce Ka and KB emission spectra of several phosphorus, sulfur and chlorine containing compounds including various oxidation states, structures and ligand environments. The high-energy resolution spectrometer in Johansson geometry [1] optimized for tender x-ray energy range was used in the experiment. The energy resolution (~0.5 eV) below the natural linewidth of the measured K lines allows us to study the chemical environment of the element in the sample. The measured energy shifts of the most intense  $K_{\alpha}$  diagram line can be related to oxidation state of the studied element in the target. The characteristic, atomic-like  $K_{\alpha 1.2}$  spectral shape allow us a precise and robust analysis of oxidation state. Chemical sensitivity is increased further in K<sub>B</sub> emission spectra, which originate from valence-core transition and reflects directly the chemical environment of the elements. Quantum chemical calculations employing the StoBe-deMon molecular/cluster software package, [2] based on density functional theory (DFT) were performed to obtain electron densities around selected atom together with corresponding molecular orbitals and partial charges. Measured  $K_{\alpha}$  energy shifts are reproduced by the calculations, clear correlation between measured energy position and calculated valence shell electron population was obtained. Measured K<sub>B</sub> spectra are well reproduced by the DFT calculations, main spectral components are interpreted in terms of molecular orbitals. Finally, the influence of bonding, ligand type and symmetry on the measured spectra is analyzed.

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### PB-27: Advanced characterisation of the crystal evolution via channelling and MC simulations – Application to nuclear oxides bombarded with low-energy ions

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Quantifying the evolution of the radiation damage inflicted on bombarded materials as a function of key parameters, such as the nature of ions, energy, fluence, concentration of foreign incorporated atoms, temperature of the target, is of prime importance for both fundamental and technological applications (e.g. semi-conductors, nuclear materials). Although ion channelling performed on single crystals was extensively applied to monitor the nature of defects as well as their depth-distribution, most of the analyses implicitly assume the sole presence of randomly displaced atoms, a feature at clear variance with the formation of extended defects at large concentrations. In fact, the radiation-induced defects are very complex and consist of various types with their distinct scattering factors to ion channelling. Such a complex configuration may nevertheless be simulated by MC, assuming a simplified model of defect, where the two dominant types, corresponding to extreme conditions of pure obstruction-type (RDA – Randomly Displaced Atoms) and pure distortion-type (BC – Bent Channels) channelling, are considered. As a matter of every complex mixture of defects involving different types can be in principle reproduced by a distribution of defects of both types. Such a model was successfully applied to the description of ion-irradiated urania single crystals bombarded with various ions (corresponding to common fission products Xe, La, Ce, with very similar Z and A, either fully soluble or insoluble into the matrix) over a very large fluence range, i.e. from the formation of isolated defects up a complete overlapping of the various damage cascades (in the range  $1 \times 10^{14}$  to  $1 \times 10^{17}$  cm<sup>-2</sup>). The evolution of RDA with increasing ion fluence leads to two (one step, respectively) steps in the damage kinetics for crystals bombarded with insoluble atoms (soluble atoms, respectively), separated by constant plateaus. The difference between soluble versus insoluble atoms is mostly due to the size of the implanted species. Conversely, the accumulation of BC leads to the very similar and regular evolution, irrespective of the nature of ion. This smooth increase (without distinct step) reveals a dramatic and continuous structural modification that was totally hidden according to the sole evolution of the RDA contribution. These evolutions are compared to results coming from complementary techniques, x-ray diffraction and transmission electron microscopy.

T.H. Nguyen, F. Garrido, et al. NIMB 326, 264 (2014) T.H. Nguyen, F. Garrido et al., submitted to Phys. Rev. B

#### PB-28: Withdrawn

#### PB-29: Irradiation studies on W-based materials

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Tungsten is a promising candidate for fusion applications since it has high melting point, good thermal conductivity and low sputtering yield, which minimizes the plasma contamination. The major disadvantage of tungsten-grades for plasma facing and structural components in nuclear fusion reactors is the low fracture toughness associated with the high ductile-to-brittle transition temperature. An attractive way to solve this problem involves the development of W alloys with other refractory metals, which always present low neutron activation. Pure tantalum shows high toughness, low activation and high radiation resistance and, moreover, transmutes to W under high-energy neutron irradiation. This tends to retard the formation of the brittle sigma phase originating from transmutation of W to Os and Re [1]. In the present study tungsten-tantalum alloys (W-Ta) were produced by Ta implantation at room temperature with a constant fluence of  $2x10^{21}$  at/m<sup>2</sup> with an energy of 340 kV. In order to comprehend the defect dynamics in these materials, the W-Ta plates were implanted at room temperature with 10 KeV of He<sup>+</sup> with a constant fluence of  $5x10^{21}$  at/m<sup>2</sup> and 10 KeV of D<sup>+</sup> ion beams with fluencies  $10^{20}$ - $10^{21}$  at/m<sup>2</sup> range. The materials were studied by scanning electron microscopy (SEM) coupled with energy dispersive X-ray spectroscopy (EDS), X-ray diffraction (XRD), Rutherford backscattering spectrometry (RBS) and nuclear reaction analysis (NRA). The microstructure observations revealed that after Ta implantation the W plates evidenced cavities and a more severe effect was observed after D implantation. In addition blistering was observed in W-Ta plates implanted with He. The NRA results show that D retention in the W-Ta alloys is higher after sequential He and D implantation than for single D implantation. The diffractogram of W-Ta alloys implanted with He evidenced the presence of broadened W peaks which is believe to be associated with the high volume fraction of the bubbles that may cause internal stress field giving birth a considerable defects as self-interstitial-atoms and dislocations resulting in a distortion of the crystal lattice.

#### PB-30: Applications of the 5.5 MV Van de Graaff Accelerator at IF-UNAM for IBA techniques

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Over the last three decades the 5.5 MV Van de Graaff accelerator at IF-UNAM has been commissioned for materials analysis, establishing several collaborations with a variety of research groups. Three years ago, a major upgrade has been performed in the associated laboratory allowing the use of more beamlines and the inclusion of a variety of new research projects. Some examples of the applications and current research performed at the Van de Graaff accelerator will be presented, with particular emphasis on the use of Ion Beam Analysis (IBA) techniques for materials characterization, such as Nuclear Reaction Analysis (NRA), Rutherford Backscattering Spectrometry (RBS) and Elastic Recoil Detection Analysis (ERDA) as

well as Particle Induced X-Ray Emission (PIXE). As it is known, results from such techniques allow the quantification of elemental concentrations and profiles in diverse samples and, when combined, has proven to be a unique and powerful tool even for the measurement of light elements such H, Li, C, N, O.

### PB-31: Effect of annealing upon retention of He and H in irradiated SiC

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Fusion materials will suffer radiation damage effects due to energetic particles from the fusion fuel and its reaction products, including hydrogen and helium ions, and high energy neutrons. Silicon carbide is a material that offers excellent thermal properties, and less erosion than other carbon-based materials. The retention of hydrogen isotope fuel and helium ash from the fusion environment, as well as the interaction between radiation damage and these retained species, requires further investigation and the results will help to understand and predict failure mechanisms of components, the lifetimes for retention of acceptable limits of tritium, and the degradation of SiC thermal properties. SiC samples were irradiated with He ions of energy up to 30 keV and a fluence up to  $10^{16}/\text{cm}^2$ , to produce damage in the near-surface region. A duplicate set of He ion irradiated SiC samples, as well as undamaged SiC, were also irradiated with H<sup>2+</sup> ions of energy up to 20 keV and a similar fluence, to study the interaction of H species with SiC and radiation-damaged SiC. Modification to the surface following irradiation is observed via Raman spectroscopy, which exhibits development of damage states such as disordered carbon and Si-Si peaks. The retention of H and He were measured using elastic recoil detection analysis using 7.8 MeV  $C^{3+}$  ions. Changes to the surface chemistry were characterised using X-ray Photoelectron Spectroscopy and Near Edge X-ray Absorption Fine Structure spectroscopy. Samples were annealed in steps of 200 K, from 473 K to 1273 K. ERDA was performed after each anneal step. Only minor changes in the H and He profiles were observed up to 1073 K, however after the 1273 K anneal the H and He profiles changed considerably, with a marked difference between samples irradiated only with He and those irradiated with He and H.

#### PB-32: Withdrawn

### PB-33: Evaluation of highly Mg-ion-implanted GaN layers grown on free-standing GaN substrates

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A selective area doping technology is required for making high performance GaN devices. Usually, ion implantation is used as a method of the selective area doping, but formation of the p-type conductive layer by ion implantation has been difficult for GaN. In our previous work, we reported p-type conversion of Mg-ion implanted n<sup>-</sup>-GaN layers, which was realized by using epitaxial layers grown on a high quality free-standing GaN substrate.

In this conference, we will demonstrate evaluation of Mg ion implanted p-type layers using I-V and optical characteristics. Mg ions were implanted into the GaN layers at two different energies of 30 keV and 60 keV at a dose of  $3.5 \times 10^{13}$  cm<sup>-2</sup> and  $6.5 \times 10^{13}$  cm<sup>-2</sup>, respectively. After implanting Mg, samples were capped with SiN layer and annealed at 1230°C for 1min in N<sub>2</sub> gas These samples showed low-temperature Photo-luminescence(PL) spectra quite ambient. similar to those observed from Mg-doped MOVPE-grown p-type GaN, consisting of Mg related donor-acceptor pair (DAP) and acceptor bound exciton (ABE) emission. Diodes fabricated by the Mg-ion implantation showed clear rectifying I-V characteristics with turn-on voltage close to bandgap energy (Eg) of GaN and UV and blue-green electroluminescence was observed at forward biased conditions. These are definite evidences for the formation of the GaN p-n Detailed analysis was carried out about forward bias I-V characteristics, which junction. indicated two turn-on voltages close to band gap of GaN and to Pd Schottky barrier height energies to n-GaN. This indicates that Mg implanted layers consist of both p-type and n-type conductive layers. Room temperature PL spectrum mapping which was reflected from Mg implanted layer using scanning-PL-microscope also showed that implanted layers consisted of uniform p-type crystalline area and localized crystal defects having n-type conduction.

In summary, Mg-ion implanted layers in n<sup>-</sup>-GaN on a high quality free-standing GaN substrate show p-type conduction after high temperature annealing at 1230°C, but Implanted layer consisted of uniform p-type crystalline area and localized crystal defects having n-type conduction.

### PB-34: Diamond device with exceptionally long charge collection distance

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The advent of proposals for monolithic diamond-based devices that employ novel quantum technologies has helped drive the improvement of diamond synthesis techniques so that high quality "device-grade" diamond is now available on a routine basis. We have fabricated radiation dosimeter devices from this material and measured the charge collection efficiency using the technique of Ion Beam Induced Charge. By use of a specialized data acquisition system, we are able to map the trajectory of charge transported through the devices from signals induced in an array of surface electrodes. As a function of bias voltage we are sensitive to both positive and negative charge carriers and find that the charge collection efficiency for MeV ion impact approaches 100% even for impacts remote from the surface electrodes with a bias field of 0.03 V/ $\mu$ m. Devices fabricated from this material are also relatively immune from trapped charge induced polarization that can degrade the charge collection process. The charge collection efficiency maps of devices with limited-area surface electrodes as a function of bias show very little fall-off in efficiency away from the area covered by the electrode showing that the carrier mobility and lifetime are high enough to allow charge transport over long distances more characteristic of silicon that has been reported to date in diamond. Potentially, this material could form the substrate for an array of deterministically implanted reproducible colour centres in a large-scale device.

### PB-35: Medium-energy Cu and O ion co-implantation into different silicate glasses

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It has been shown that the oxidation state of Cu implanted into a glass depends on many factors. In this work we study how the oxidation state of Cu can be influenced by composition of silicate glasses and by subsequent implantation of oxygen ions.

Three types of silicate glasses having a different extent of cross-linking were implanted with  $Cu^+$  ions with an energy of 330 keV and a fluence  $1x10^{16}$  ions/cm<sup>2</sup>. Subsequently, the glasses were implanted with  $O^+$  ions into the same depth as the already implanted Cu. Concentration profiles of Cu in the glasses were studied by Rutherford Backscattering Spectroscopy. The as-implanted samples were also characterized by optical absorption and photoluminescence spectroscopy. The effect of annealing on distribution of Cu and absorption and emission spectra of the as-implanted glasses will be discussed as well. Therefore, the samples were annealed around transformation temperature of the glass (600 °C) in air for various times ranging from 1 to 5 hours.

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### PB-36: Silver ion implantation into silicate glasses containing Er – Yb and its influence on luminescence properties

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Silver nanoparticles in the glass matrix enhance emission efficiency of present rare earth ions. This effect is generally caused by surface plasmon resonance. This contribution is focused on the possibility to influence the luminescence properties of erbium and ytterbium doped glasses by presence of silver. Therefore, a set of silicate glasses having different ratio of erbium and ytterbium and different concentration of zinc oxide was fabricated. To achieve Ag-rich thin films on the substrate surfaces we used ion implantation technique at energy 1.2 MeV and 1.7 MeV with fluence  $1 \times 10^{16}$  cm<sup>-2</sup>. Post-implantation annealing was also applied. We measured waveguiding, spectroscopic and lasing properties as well as silver concentration depth profiles. Basic passive characteristics of the undoped glasses like absorption and luminescence were compared with absorption and luminescence of the glasses containing silver ions or silver nanoparticles. Results showed that silver nanoparticles positively influence luminescence intensity at 1550 nm by increasing this intensity almost 3 times. Applying post-implantation annealing did not show significant change in luminescence intensity for studied glasses.

The research has been realised at the CANAM (Center of Accelerators and Nuclear Analytical Methods) infrastructure and has been supported by project No. P108/12/G108

# PB-37: High energy resolution PIXE using protons and heavy ions in the analysis of thin film samples

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Energy dispersive transition edge sensor (TES) array is used at the University of Jyväskylä for high energy resolution PIXE measurements. The best achieved resolution for a single pixel so far is 3.06 eV at 5.9 keV energy [1]. Our TES-array consists of 160 pixels, the total active area is 15.6 mm<sup>2</sup> and it can be used for an energy range between 1 and 15 keV.

The use of Si beam has been earlier found to be an efficient way to differentiate SiO<sub>2</sub> from Si in

wavelength-dispersive detection system [2]. In this paper we will study the differences in satellite peak intensities using H, He, Li, C, O and Cl beams. Besides satellite peaks the other main focus of this work is to have quantitative elemental analysis. In this paper we will discuss the advantages and disadvantages of heavy ion beams in a point of view of TES detectors.

One of the benefits of using heavier ion beam is that the ions do not penetrate as deep in the sample as protons do and the measurement is more surface sensitive. On the other hand, heavier beams create more multiple ionizations which leads to more complicated satellite peak structure compared to the proton beams (see Fig. 1). This can be an advantage when studying chemical composition [2] but also a distraction for samples with complicated X-ray spectrum.



Figure 1: Evaporated Ti metal film measured with 2.0 MeV protons and 6.8 MeV <sup>12</sup>C beam.

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[2] Y. Mokuno, Y. Horino et al, Nucl. Instr. Meth. B: 136–138 368–372 (1998)

### PB-38: Formation of silver metal nanoparticles by ion implantation in silicate glasses

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It has been shown that glasses containing silver metal nanoparticles are perspective photonics materials for fabrication of all-optics components. Resulting optical properties of the nanocomposite glasses depend on the composition and structure of the glass, as well as on a type of the metal ion and involved experimental procedures. The main aim of this contribution is to study the influence of conditions of the ion implantation and the composition of the glass on formation of metal nanoparticles in such glasses.

Four various types of silicate glasses were implanted with  $Ag^+$  ions with different energy (330 keV, 1.2 MeV and 1.7 MeV), while the fluence was kept constant (1.10<sup>16</sup> ions/cm<sup>2</sup>). The asimplanted samples were annealed at 600 °C for various times. The samples were then characterized from the point of view of: nucleation of metal nanoparticles (linear optical absorption), migration of silver through the glass matrix during the implantation and postimplantation annealing (Rutherford Backscattering Spectroscopy), and the oxidation state of silver (photoluminescence).

The results showed that the ion implantation is suitable technique for preparation of metal nanoparticles mainly in silica glass. Silver nanoparticles were already created in this type of glass during the 330 keV ion implantation and the size of nanoparticles increased during the subsequent annealing. Keeping the same ion fluence, it is possible to prepare silver nanoparticles also in the glasses containing Na<sup>+</sup> ions but the energy of implanted silver had to be higher (up to 1.2 and 1.7 MeV).

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# PB-39: Optimization Analysis and Quantification of Boron and Indium in BGaInAs thin film by PIGE and RBS techniques

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Ion beam analysis techniques are used to characterize Boron (B) and Indium (In) in BGaInAs/GaAs thin film. The quantification of B in thin solid film by Particle Induced Gamma-Ray Emission (PIGE) is not an easy analytical task, which is due to the different parameters, such as its sensitivity for small quantities of boron in thin films and the absence of reference samples of similar structure. The scope of this work is the optimization of PIGE technique for Boron quantification in BGaInAs thin films by varying different entry parameters (e.g. energy and angle of incident ion, as well as the choice of boron characteristic Gamma-Ray). Furthermore, Rutherford Back Scattering Technique (RBS) is used to determine the Indium (In) concentration and the thickness of BGaInAs epilayers.

Two kinds of samples were prepared for this study. The first, a reference sample was used to optimize the Boron detection for PIGE technique. It consists of Boron Nitride (BN) thin film (200nm) on silicon substrate. The second kind of samples was BGaInAs thin films deposited on (001) GaAs substrate. The growth of BGaInAs epilayers was performed by atmospheric pressure Metal Organic Vapor Phase Epitaxy (MOVPE) in a T-shape horizontal reactor. Diborane (B<sub>2</sub>H<sub>6</sub>), triethylgallium (TEG), trimethylindium (TMI), and arsine (AsH<sub>3</sub>) were used as boron, gallium, indium, and arsenic sources, respectively. AsH<sub>3</sub> and TEG flow rates were kept constant, respectively, at  $2.7 \times 10^{-3}$  and  $4.2 \times 10^{-6}$  mol min<sup>-1</sup> as well as the TMI was also keep it constant during all the growth. Hydrogen was used as carrier gas. Moreover, the growth temperature was fixed at 580°C and the B2H6 flux was varied from 2 to 7.5 sccm Among three different prompt Gamma Rays resulting from proton-Boron nuclear reaction the on at 429 KeV, emitted

via the  ${}^{10}B(p, \alpha\gamma)^7Be$  reaction, was used for the analysis of B due to its high cross section and to it is specificity at any primary beam energy used in this work.

In order to improve the sensitivity for B detection and quantification determination, a systematic study was undertaken using proton ion beam at different energies (from 1 to 3 MeV) with different tilting angles of incidence (0°, 40°, 60°, and 80°) on the BN sample. Best conditions were found to be at 1.5050 MeV proton energy and 80° tilting angle which permit an accurate determination of B with high sensitivity within few tens of minutes as acquisition time. The limit of detection (LOD) of B in the present study was found to be less than 0.08 at%. Then these optimized conditions were applied for B detection inside BGaInAs thin films deposited on (001) GaAs substrate. A new formula for thin films instead of thick target was used in this study in order to deduce the B concentration by PIGE. It was found that the Boron concentration of different samples was varied from 0,8 to 2,5 at% when the  $B_2H_6$  flux was varied from 2 to 7.5 sccm respectively. Finally, The determination of Indium concentration as well as the thickness of BGaInAs epilayers was done by RBS using 2 MeV of He<sup>2+</sup> ion beam.

### PB-40: Determination using NRA of polymer concentration in partially polymerized films used in the development of biological or environmental sensors

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Polydiacetylenes (PDA) are a class of conjugated polymers currently actively studied for the development of biological or environmental sensors using Langmuir films or vesicles [1]. PDA sensors exploit a structural transition of the polymer chain, causing a color change, conventionally named the blue to red color transition, with the absorption maximum shifting from ~630 to ~540 nm.

Since polymerization of PDA films is usually not complete [2], the polymer content in the film is unknown, precluding development of quantitative titration. In this work we develop a method for accurate determination of polymer concentration, using only the absorption spectrum of the film. This requires knowledge of absolute absorptivities. These polymers are almost never soluble whatever the solvent, so polymer extraction and determination of the absorptivity of solution of known concentration [1] cannot be used.

We show here that  ${}^{12}C(d,p){}^{13}C$  NRA can be used to determine absolute absorptivities as follows. Films of thicknesses 30 to 250 nm were prepared by vacuum evaporation of the monomer and absolute C atomic areal densities were determined by NRA. Since NRA is insensitive to the C chemical form, beam induced degradation is only important if C atoms are expelled from the film; this was corrected for by fractionating the beam fluence and extrapolating to zero fluence. Films were then photopolymerized to polymer concentrations Xp from less than 10 % to over 50 % (we found that the reaction does not go further), polymerization being followed spectroscopically. The resulting polymer is in the blue form. After extracting residual monomer either by dissolution or by vacuum evaporation, the final polymer is in the red form. Xp was deduced from the remaining C atomic areal densities determined by NRA. One now has a series of polymer absorption spectra, blue and red, corresponding to known Xp, from which the absorptivities and molecular absorption cross sections may be determined. Specifically one may use the area under the complete spectrum, or the maximal absorbance. Corrections for small changes in spectral shape are also determined. The method is general and can be used for any film forming PDA.

Two PDAs were studied. The method was first validated using one of the very few soluble PDAs, 4BCMU, of known absorptivity [2]. It was then applied to 10-12,pentacosadiynoic acid which is by far the most often used PDA in sensor studies, yielding a set of parameters which relate optical absorption spectra to Xp. This method may readily be extended to any other PDA.

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[2] S. Spagnoli et al., Macromolecules 44 2613 (2011)

### PB-41: Preparation and structure properties of Ti/TiHx thin films

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Ti/TiH<sub>x</sub> thin films have been deposited on SiO<sub>2</sub> wafers by thermal evaporation in the 300 °C temperature. Some samples were deposited in high vacuum condition (Ti thin films) while others in a hydrogen gas atmosphere (TiH<sub>x</sub> thin films) with aim to study the effect of the H<sub>2</sub> gas on the structure and quality of the deposited layers. Development of microstructure and evolution of defect structure with increasing H concentration was investigated by X-ray diffraction (XRD), scanning electron microscope (SEM), Rutherford Backscattering Spectroscopy (RBS), Elastic Recoil Detection Analysis (ERDA), secondary ion mass spectrometry (SIMS) and slow positron implantation spectroscopy. RBS and ERDA can provide important information on the elemental composition and depth profile of thin layers. So we can obtain the components, atomic ratio and thickness of titanium hydride films. Besides, SIMS has been used to obtain the type and content of contaminant in the surface of titanium hydride films. In addition, slow positron implantation spectroscopy has been used to investigate the defect structure information of Ti/TiH<sub>x</sub> thin films. The experimental results show that the pure Ti thin films are found to contain hcp crystalline structure, and little concentration of vacancy defects. With the increase of hydrogen flow rate, the hydrogen partial pressure will improve the environment. Meanwhile, the diffraction peaks of titanium hydride become strong gradually in XRD patterns, the H/Ti atomic ratio of film is gradually increasing and the oxygen impurity in the surface of titanium hydride films is gradually deceasing. Besides, the S parameter of TiH<sub>x</sub> thin films is gradually increasing, which indicates the hydrogen will cause a lot of defects.

### PB-42: Interface reactions between Pd thin films and SiC by thermal annealing and SHI irradiation

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The solid-state reactions between Pd thin films and 6H-SiC substrates induced by thermal annealing, room temperature swift heavy ion irradiation (SHI) and high temperature SHI have been investigated using insitu and real-time Rutherford backscattering spectrometry (RBS), secondary electron microscopy (SEM) and X-ray diffraction (XRD). At room temperature, no silicides were detected to have formed in the Pd/SiC samples. Two reaction growth zones were observed in the samples annealed by insitu and real time RBS. The initial reaction growth region led to formation of Pd<sub>4</sub>Si as the initial phase to form a temperature of about 430 °C. Thereafter, the reaction zone did not change untill a temperature of 640 °C was attained where a mixture of Pd<sub>2</sub>Si and Pd<sub>4</sub>Si were observed to form in the reaction zone. Kinetic analysis of the initial reaction indicates very fast reaction rates of about  $8.99 \times 10^5$  (at. cm<sup>-2</sup>)<sup>1/2</sup> s<sup>-1</sup> and the the Pd silicide formed grew parabolic with time. Swift heavy ion irradiation (SHI) of Pd/SiC samples was performed by 167 MeV Xe<sup>26+</sup> ions at room temperature at fluences of 1.07×10<sup>14</sup> and 4×10<sup>14</sup> ions/cm<sup>2</sup> and at 500 °C (4×10<sup>14</sup> ions/cm<sup>2</sup>). The Pd/SiC interface was analysed by RBS and no SHI induced diffusion was observed for room temperature irradiations. The sample irradiated at 500 °C, SHI induced diffusion was observed to occur accompanied with the formation of  $Pd_4Si$ and Pd<sub>2</sub>Si phases.

### PB-43: Withdrawn

# PB-44: Phosphorus quantification by <sup>31</sup>P(alpha,p)<sup>34</sup>S nuclear reaction in phosphorus containing films

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Phosphorus has recently attracted attention in the silicon carbide (SiC) technology due to its promising effects improving the electrical quality of SiC devices. Incorporation of phosphorus in the SiO<sub>2</sub>/SiC interfacial region can reduce the density of interface states and increase the channel mobility of SiC devices. Besides the typical post-oxidation annealing in phosphorous ambient [1], it is necessary to investigate alternative routes to incorporate phosphorus in the SiO<sub>2</sub>/SiC interfacial region to expand the possibilities, as well as its quantification, since no reports considering this important aspect were found. The use of a nuclear reaction is an

interesting approach to detect and quantify phosphorus, due to its selective, simpler, and faster measurement compared to other techniques. The idea in this work was to develop a simple method to quantify phosphorus that could be used hereafter in the SiC technology, for instance.

Phosphorus detection and quantification were investigated, using the  ${}^{31}P(\alpha,p){}^{34}S$  nuclear reaction and Rutherford Backscattering Spectrometry, in sputter deposited silicon oxide films with phosphorus incorporated during film deposition and in carbon substrates implanted with 30 keV  ${}^{31}P$ . It was possible to determine the total amount of phosphorus using the resonance at 3640 keV of the  ${}^{31}P(\alpha,p){}^{34}S$  nuclear reaction [2] in samples with phosphorus present in up to 15 nm in depth. Nuclear reaction spectra were practically background-free and phosphorus amounts as low as  $1 \times 10^{15}$  P/cm<sup>2</sup> were detected. Besides, the results obtained by nuclear reaction were in good agreement with those obtained from RBS measurements. The advantage of using this nuclear reaction is that P quantification is not hampered by the presence of signals from other species present in the sample, such as Si, for instance. Higher  $\alpha$  energies inducing the nuclear reaction were used to quantify phosphorus in implanted samples, due to the phosphorus presence in deeper layers. Finally, the possible future applications of phosphorus deposition routes used in this work in the SiC technology will be also discussed.

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### PB-45: Withdrawn

# PB-46: Dependence of soft errors in silicon-on-insulator static random access memory on distribution of energy deposition by high-energy heavy-ion incidence

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Soft errors occur in an ultra large scale integration due to excess charge induced by a highenergy particle, which is originated from a cosmic ray. A silicon-on-insulator (SOI) device has advantages for the soft error over a conventional bulk device, because a SOI body is insulated from a silicon substrate with a buried oxide layer. In our previous studies, the soft error rates (SERs), the number of the soft errors with single ion incidence, in a SOI static random access memory (SRAM) with a technology node of 90 nm were found to depend on the incident energies [1] and the distance between the body electrode and the metal pad [2] evaluated by proton, helium, and oxygen ion probes. The SERs by beryllium, carbon, and oxygen ions were also found to be much higher than those by hydrogen and helium ions, even though SERs were believed to be constant when the amount of the generated charge in the SOI body was over the critical charge [3]. It was assumed that the difference of the SERs might be due to the difference in the distribution of the energy deposition by the ion incidence. In this study, to clarify the effect of the distribution of the energy deposition by the ion incidence to the soft errors, SOI SRAMs were irradiated with ions from the azimuthally varying field cyclotron accelerator in Takasaki ion accelerators for advanced radiation application, Japan Atomic Energy Agency. The incident particles were oxygen, carbon, nitrogen, neon, argon, krypton, and xenon ions with energies ranging from 56 to 454 MeV, which generated the excess charges ranging from 1.45 to 52.8 fC in the SOI body. The generated excess charges were almost the same as or much higher than the critical charge of 1.8 fC in the SOI SRAM cell. The SERs were evaluated with the distribution of the energy deposition in the SOI body, which calculated by the Katz theory [4]. The SERs linearly depended on the number of the incident ions. The SERs by the 454-MeV xenon ion were more than one. This means that a multi bit upset induces by the 454-MeV xenon ion. The SERs by the 150-MeV argon ion were higher than those by the 250-MeV argon ion. This means that the SERs does not depend on the excess charge in the silicon substrate because the higher energy ion generates higher excess charge in the substrate. The lateral distribution of the energy deposition by the 150-MeV argon ion incidence was larger than that by the 250-MeV argon ion incidence in the SOI body. These results indicate that the spread distribution of the energy deposition provides the soft errors not only in the SRAM cell with the high-energy heavy-ion incidence, but also in the surrounding SRAM cells.

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#### PB-47: Micro and Nano Mechanical Testing of Fe-ion Irradiated ODS steel

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Oxide dispersion strengthened (ODS) steel is a potential candidate for the nuclear fuel cladding material in the fast reactor application. The finely dispersed particles improve the tensile and creep rupture strength of material at the elevated temperature up to approximately 650 °C. Moreover, the interfaces between the particle and matrix do the roll as sinks for radiation defects. During irradiation, materials inevitably experience mechanical stress due to the bombardment by particles. With the oxide particle distribution, ODS steels usually show the better stress capacity than other candidates materials. The use of ion implantation for the simulation of radiation damage produced by neutrons within a reactor is common. However, the electronic stopping and relatively large cross-section of heavy ion interactions with the matrix limits the range of ions into a solid; the irradiation depth is limited to several micrometers, the mechanical testing method for such a thin area is also limited. Therefore, instrumented small-scale tensile testing approaches are needed. In this study, Fe self-ion irradiation is used as means of introducing irradiation damage in ODS steel. The ion accelerator named DuET (at Kyoto University, Japan) was used for irradiation with 6.4 MeV Fe<sup>3+</sup> ions at 300

<sup>o</sup>C. The maximum damage rates were estimated to be roughly 70 dpa. The irradiation-induced hardness change in the damaged layer was evaluated by using nano-indentation and the tensile properties were evaluated by using small-scale tensile testing device. Actuators and load measurement devices are integrated all on a single chip, the force could be measured during tensile testing of one-dimensional nanostructures.

### PB-48: PIXE Determination of the Stoichiometry of Ni-Pd and Au-Ag Nano-Particles Prepared by Laser Ablation in Liquid Solution

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Synthesized colloidal nanoparticles were prepared by laser ablation in liquids using YAG laser focused on bimetallic targets (Au<sub>75</sub>Ag<sub>25</sub>, Ni<sub>75</sub>Pd<sub>25</sub> and Ni<sub>95</sub>Pd<sub>5</sub>). The size and size distribution of the obtained particles in the solution was checked by TEM. The stoichiometric ratio of the particles was determined using ICP, EDX and PIXE, where the obtained results were comparable except for ICP. The PIXE determination of the stoichiometry of Au-Ag nanoparticles showed a composition similar to the target. However, for Ni-Pd, the composition approaches the nominal composition of the target at higher ablation times. PIXE succeed to demonstrate a linear trend of the Ni/Pd stochiometric ratio vs. time of ablation. The quantification of PIXE composition was done using GUPIX code, using thin film and multilayer approaches.

### PB-49: Investigating Pt-Pd bimetallic nanoparticles structure by MEIS

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Bimetallic nanoparticles present increasing interest in nanoscience due to their particular catalytic, electronic and optical properties compared to their bulk counter-part [1]. Pt-Pd nanoparticles are important catalysts for hydroisomerization, hydrocracking and hydrotreatment [2]. Three different types of PtPd nanoparticles have been synthesized in order to nominally obtain a Pt<sub>0.5</sub>Pd<sub>0.5</sub> alloy, a Pd@Pt (core@shell) and a Pt@Pd structures. Previous investigations were able to give insights into the PtPd nanoparticles structures by HRTEM, SR-XPS [3] or EXAFS [4]. However, a precise determination of their structure could not be achieved. Medium Energy Ion Scattering (MEIS) is an ion-beam technique suitable to explore the outermost layers of nanoparticles due to its high energy resolution. In order to determine the elements arrangements of Pt and Pd within the synthesized nanoparticle, we performed MEIS

simulations by using the PowerMEIS package [3]. The MEIS experiment was done at Laboratorio de Implantacao Ionica (UFRGS, Brazil) with a 200 keV He<sup>+</sup> ion beam. PowerMEIS is able to perform MEIS simulations considering any kind of geometry, size distribution and areal density of the nanoparticles [4]. In our simulations, we considered the following possible structures: a PtPd-alloy, Pd@Pt, Pt@Pd, Pd@PtPd and Pt@PtPd. By using the method [5], we were able to determine the arrangement of the chemical elements within the nanoparticle. The MEIS experiments were able to precisely determine the structure of the nanoparticles as a  $Pt_{5/8}Pd_{3/8}$  alloy, a  $Pd@Pt_{2/3}Pd_{1/3}$  or a  $Pt@Pt_{0.38}Pd_{0.62}$ .

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### PB-50: High Resolution Depth Profiling for the Study of Titanium Oxidation

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Ti has many important applications in both scientific research and industry. Our aim is understanding the mechanism of formation of a passivating Ti oxide via electrochemical oxidation, in the ultra-thin oxide limit. Ultra-thin Ti films were deposited by magnetron sputtering, onto a Si (001) substrate followed by in-situ exposure to isotopic, <sup>18</sup>O water to form a TiO<sub>2</sub> ultra-thin layer. Next, the TiO<sub>2</sub>/Ti/Si(001) samples were electrochemically oxidized in D<sub>2</sub><sup>16</sup>O water.Thicknesses and stoichiometry of the films including their surfaces and interfaces were determined by X-ray Photoelectron Spectroscopy (XPS), Rutherford Backscattering (RBS), and Medium Energy Ion Scattering (MEIS), Nuclear Reaction Profiling (NRA) and Elastic Recoil Detection (ERD). The relative concentration of O isotopes as a function of depth can be determined from the MEIS and NRA spectra. From such depth profiles, the principles governing oxidation can be inferred. The depth profiles suggest that after consecutive isotopic oxidations, <sup>18</sup>O remain in greater concentrations near the TiO<sub>2</sub>/Ti interface corresponding to new oxide growth at the liquid phase/TiO<sub>2</sub> interface. New oxide increases nearly linearly with increasing oxidation voltage. No deuterium incorporation was detected within the detection limits of ERD. Two possible mechanisms are possible with isotopic exchange with O as a mobile species or alternatively with Ti being a mobile species. Further details of the mechanism will be discussed.
### PB-51: Study of InAs quantum dots in silicon obtained through ion implantation

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Nanocrystals of III-V compounds embedded in a semiconductor matrix with higher band gap are of great interest for optoelectronic devices since the pronounced quantum confinement effects of such materials allow tuning their light emission by controlling the size of the nanocrystals. Molecular beam epitaxy (MBE) is the most used technique to produce such nanostructures. Another technique to form this kind of nanocrystals is the combination of ion implantation and thermal annealing.

In this work, we study the formation of InAs quantum dots by sequential implantations of As and In in silicon with subsequent Rapid Thermal Annealing (RTA) of the implanted samples. Three sets of samples were produced with fluencies of  $1 \times 10^{16}$  cm<sup>-2</sup>,  $2 \times 10^{16}$  cm<sup>-2</sup> and  $5 \times 10^{16}$  cm<sup>-2</sup>. The specimens were implanted at 500 °C with energies of 250 keV for As and 350 keV for In. The samples were annealed for 30 seconds with different maximum temperatures and durations in order to optimize the light emission from InAs nanocrystals embedded in Si. Besides photoluminescence (PL), the samples were characterized with complementary techniques like RBS-C, SEM and TEM, thus providing further structural information.

After RTA treatment, InAs precipitates are evidenced and a strong increase of photoluminescence yields were observed. Higher photoluminescence intensity was observed for the samples implanted at lower fluences.

#### PB-52: Characterisation of diamond-like carbon films implanted with magnetic ions

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Rutherford backscattering spectrometry was used to characterise diamond-like carbon (DLC) films produced by ion beam deposition in as-grown and implanted states. Magnetic ions of Ni and Co were implanted with 30 kV into the surface of mass selected ion beam deposited diamond-like carbon films. The first stage of the process was to form DLC films on Si. The process takes advantage of low sputtering yield to form films, which have decreasing amount of Si from the film/substrate interface to the surface. The films were grown with  $C_3H_6^+$  ions to 100 nm in thickness. The current density was chosen to 2  $\mu$ A to avoid heating effects. The films were grown in the low-energy GNS Science ion implanter. RBS was used to measure the

stoichiometry of the films. A typical stoichiometry of  $C_3H_1$  was measured across the film. Ultra smooth surface roughness of the films was measured with AFM. Raman spectroscopy revealed an I(D)/I(G) ratio of typically 0.6 indicating about 20% sp<sup>3</sup> content. The films were subsequently implanted with Ni and Co ions to study their magnetic properties. The films were implanted to varying concentration. Ferromagnetic order was observed at room temperature. The magnetic moment per implanted atom increases with decreasing temperature in the range from 5 – 300 K. Specific details will be presented. The paper shows the usefulness of RBS in developing diluted magnetic semiconductor material for use in spintronic devices.

### PB-53: Comprehensive Three Dimensional Ion Beam Analysis of Gold and Silica Nanoparticles Supported by Silicon Substrates

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There is increasing demand for comprehensive characterization tools to relate the detailed 3D structure of a given system to its behavior in a well-defined physical or chemical environment. Surface electric fields appear in many applications and play major role in the preparation, operation, and characterization of nanostructured devices. Electric field-induced effects are essential in practical issues, e.g., field enhanced electron emission, surface plasmon resonance, or the spatial reorganization of nanoparticles [1].

As powerful tools ion beams allow the buildup of a surface potential on the sample and its insitu monitoring [2]. Although planar layers have been analyzed in this manner less attention was paid to nanostructured materials. Previously we reported the detailed 3D analysis of nanoparticles via Rutherford Backscattering Spectrometry (RBS) [3, 4]. In the present work we combined 3D-RBS analysis with RBS-channeling (RBS/C) experiments for a monolayer of spherical particles deposited on single crystalline Si substrates. The sample structure (particle shape, size, surface coverage) was determined by 3D-RBS and scanning electron microscopy (SEM) imaging. Au, silica, and Au-silica core-shell particles in the size range 20-300 nm were characterized. Based on the evaluated sample geometry, comparison was made between the predictions of multiple scattering (MS) theory and the small angle scattering effect of nanoparticles determined from RBS/C angular scans. During the experiments the Si substrate was isolated from the metal sample holder to facilitate ion beam-induced sample charging. Also, the blocking of surface potential buildup was possible with a tunable electron source operated in the sample chamber. As typically the transversal kinetic ion energy required for dechanneling is ~100 eV, the surface potential of charged particles in the 10-100 V range may influence the ion dechanneling probability and the angular scan. The ion-beam induced surface potential developed for each element (Si, O, Au) in the sample was estimated from the shift of the leading edges in the RBS spectra recorded for 750 keV or 1.5 MeV He<sup>+</sup> ion energy. An excess potential was observed for the nanoparticles compared to the Si substrate thus suggesting the charging and/or polarization of the particles. This effect was considered to explain differences between measured and MS theory estimated minimum yields. For this purpose finite element calculations of the electric field distribution inside and outside the particles and in the substrate were performed. The collective effect of the planar set of charged particles on the buildup of a longitudinal and transversal electric field with respect to the He<sup>+</sup> ion trajectories was estimated.

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### PB-54: Ion Beam Analysis of Oxide Thin Films

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The understanding of the structural and chemical properties of oxides has become increasing interest due to their applications in several areas. In particular, high quality oxide thin films are grown on suitable substrates for fundamental science investigations to address the scientific questions in various applications in heterogeneous catalysis, magnetic thin films, high and lowdielectric materials, corrosion and integrated devices. We have grown various well-ordered oxide thin films and characterized using high energy ion scattering techniques including Rutherford backscattering spectrometry (RBS), nuclear reaction analysis (NRA), proton induced x-ray emission (PIXE) and elastic recoil detection analysis (ERDA). High energy ion beam analysis of oxide films along channeling and random geometries is very powerful in characterizing composition/stoichiometry, crystalline quality, buried interfaces and dopant/impurity locations and distributions. On the other hand, low energy helium ions can be effectively used in helium ion microscope (HIM) to image materials with a lateral resolution of approximately 0.35 nm. Although HIM imaging is similar to scanning electron microscopy (SEM) imaging, highly focused He ion beam provides high spatial resolution along with high depth of view in a significantly smaller interaction volume on a sample surface. We have utilized the combination of these ion beams to successfully understand various structural and chemical properties of oxide thin films and some of these results will be presented.

### PB-55: Electrical property of 4H-SIC Schottky diodes irradiated by electron

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The effects of electron radiation on the current-voltage (I-V) characteristics of commercial 4H-

SiC Schottky diodes were investigated. Electron beam with 3 MeV and 10 mA was used for irradiation up to 15 MeV ( $6.6 \times 10^{15}$  cm<sup>-2</sup> electrons). The results showed that post irradiation of forward bias (FB) current decreases, while reverse bias current (RB) increases. However, degradation of ideality factor n, saturation current I<sub>s</sub> and barrier height ( $\Phi_b$ ) was not signification decreased after irradiation. This suggested that electron irradiation was creating the defect density and due to that, the series resistance R<sub>s</sub> increased significantly. However, after irradiation with 15 MGy, FB I-V curve starts to saturate at 0.4 V. It can be concluded that radiation induced degradation of ideality factor n, saturation current I<sub>s</sub> and barrier height  $\Phi_b$  is insignificant while series resistance R<sub>s</sub> increases significantly by the irradiation dose. After subjection to 6.6 x 10<sup>15</sup> cm<sup>-2</sup> electrons irradiation dose, diodes lose their rectifying behavior.

### PB-56: Identifying the Dominant Interstitial Complex in GaAsN Alloys

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Highly mismatched alloys (HMAs) consist of highly immiscible solute atoms in a solvent. Due to the resonant interactions between the conduction and/or valence band of the solvent and energy levels of the solute, HMAs may provide an "intermediate band" for solar cells based upon multi-photon excitation [1,2]. In the case of GaAsN, the large composition-dependent bowing parameters are typically explained in the context of the band anti-crossing model, which considers interactions between the valence band edge states of the host and a localized state associated with the solute atom. Since these simple models consider a single local atomic environment, ignoring any fluctuations intrinsic to random alloys, such as clusters involving two or more atoms, they do not predict extraordinary physical phenomena, such as the nonmonotonic composition-dependent of the GaAsN effective masses [3] and persistent photoconductivity [4]. Several groups have suggested that N shares an arsenic site with either arsenic or another N atom, often termed (N-As)<sub>As</sub> or (N-N)<sub>As</sub> split interstitials [5-8]. However, the precise N atomic configuration, including the relative concentrations of  $(N-N)_{AS}$  or  $(N-AS)_{AS}$ . remains unknown. To distinguish (N-N)As and (N-As)As interstitials in GaAsN alloys, we compare nuclear reaction analysis (NRA) spectra with Monte Carlo (MC) simulations utilizing full numerical integration of ion trajectories. In both cases, incident particle paths along the [100], [110], and [111] directions are considered. For the MC simulation, we assume that (N-N)<sub>As</sub> is aligned along the [111] direction while (N-As)<sub>As</sub> is aligned along the [010] direction. Both the measured NRA and simulated channeling spectra exhibit the highest (lowest) yields in the [111] ([100]) directions, suggesting that dominant interstitial complex is likely (N-As)<sub>As</sub>.

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#### PB-57: Withdrawn

### PB-58: Study of Focused Ion-beam Induced Auger Electron Spectroscopy as a New Route for Three Dimensional Chemical Analysis

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In conventional Auger electron spectroscopy, a beam of electrons is used to create inner-shell atomic vacancies in the target material. Characteristic Auger electrons (or X-rays depending upon the fluorescence yield of the material) are then emitted as a result of the de-excitation of core vacancies [1]. Similarly the impact of energetic heavy ions onto the sample surface can cause generation of characteristic Auger electrons, among various other signals i.e. secondary electrons, X-rays, backscattered or sputtered ions etc. The mechanism for the generation of Auger electrons in the Ion-induced Auger Electron Spectroscopy (IAES) is however, different from the electron induced mechanism. As the incident ion collides with the target atoms, coreshell vacancies are generated as a result of promotion of inner shell electrons though crossing of molecular orbitals. Such excitation processes have been described by Barat and Lichten [2] for bi-particle gas phase collisions. In this paper the potential application of ion-induced Auger electron spectroscopy (IAES) for 3D chemical tomography using the focused ion beam is discussed. First we present our results of a successful integration of a Mass-filtered Focused Ion Beam (MS-FIB) from Orsay Physics with an X-ray photoelectron spectrometer (PHI Versaprobe) to explore the potential for high spatial and chemical resolution IAES. The MS-FIB is equipped with an Au-Si Liquid Metal Ion Source (LMIS) [3,4] and the generated electron spectra from different target materials are collected using the concentric hemispherical electron energy analyzer of the XPS system. The ion-induced generated electron spectra of some of the elements in the second (e.g. Na, Al, Si, P etc.) and third row (e.g. Ti, V, Cr, Mn, Fe, Co, Ni and Cu) of the periodic table are shown and the origin of different spectral features are discussed. An example of the IAE spectrum acquired using Si<sup>++</sup> and Au<sup>+</sup> incident ions is shown for Cobalt is shown in Figure 1. As a result of the energetic collisions between the incident and target atoms, in addition to plasmon excitations, Auger electrons from both colliding particles are generated. The efficiencies of Auger electron generation by ion impact from these elements, acquired under the same conditions, are also compared with each other and the origin of the differences in line shapes explained. Estimates of the absolute yields of Auger electrons were made using the acquired ion-induced electron emission spectra and, in particular, for clean Al and Cr

samples, where the values of ion-induced secondary electron emission were available in the literature. By taking into account the estimated values of Auger yield for Al and Cr (~9 and 6  $\pm 2\%$  respectively) and the effects of hardware parameters, in particular the solid angle of the detector and the transmission of the electron energy analyzer, values of ~50 and 100nm were estimated for the edge length of an information cube representing the minimum amount of material that has to be removed before a meaningful signal can be collected.

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Figure 1. Ion induced Auger electron spectrum of Co induced by  $Si^{++}$  (black) and  $Au^{+}$  (red) incident ions at 30kV. The inset shows an spectra acquired using high pass energy in differentiated mode

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## PB-59: Lattice location of nitrogen atoms into synthetic diamond single crystal by NRA/C technique

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Over the past decade, the use of nanoparticles as drug delivery agents has been the subject of intense research activities. Diamond nanoparticle (size<20 nm) containing nitrogen-vacancy (NV) color centers emerged as an attractive biolabel, owing to the perfectly stable photoluminescence of NV centers [1,2]. Nitrogen is naturally present at a concentration specified to be  $\approx$ 100-200 ppm (according to infrared absorption measurements) in high-

pressure high-temperature (HPHT) type 1b diamond. It is converted into NV center after the creation of vacancies by high-energy particles beam irradiation, followed by 800-900°C annealing in vacuum. The NV centers are only formed when vacancy is trapped in position adjacent to single substitutional nitrogen defect. The purpose of this work was to confirm the nitrogen atoms location at substitutional sites in type 1b diamond, and measure the nitrogen concentration with a Nuclear Reaction Analysis (NRA) approach. We analyzed synthetic diamond single crystals type 1b from "Element Six" company (thickness of 150 µm). The concentration of nitrogen was determined by NRA technique using <sup>14</sup>N(d,p<sub>0</sub>)<sup>15</sup>N nuclear reaction. Emitted protons have a sufficient energy (9.4 MeV) to be transmitted through the sample and detected at 0° by a detector located at a few millimeters from the backside of the sample. This measurement configuration associated to a large detection solid angle compensates the low cross section of  ${}^{14}N(d,p_0){}^{15}N$  and improves the sensitivity of this technique to about 5 at. ppm. In random position of the diamond single crystal, the nitrogen concentration is about 400-500 at. ppm. The results of the angular scans of <100>, <110> and <111> crystallographic axes performed by NRA technique in channeling mode show clearly that the nitrogen atoms are largely located in carbon substitutional site. Measurements by infrared transmission spectroscopy corroborate the results obtained by NRA/C.

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### PB-60: Study of In distribution on GaInSb:Al crystals by ion beam techniques

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III-V ternary alloys semiconductor materials, in particular Ga<sub>1-x</sub>In<sub>x</sub>Sb, are ideal candidates for devices substrates because of the possibility to define the lattice constant as a function of the third element, indium. Aluminum, an isoelectric dopant for Ga and In, increases the carrier mobility in GaSb crystals and it influences the concentration of native defects by passivating and/or compensating them. To understand the influence of Al on the distribution of indium in ternary alloys of Ga<sub>1-x</sub>In<sub>x</sub>Sb, pure and doped Ga<sub>0,8</sub>In<sub>0,2</sub>Sb crystals were obtained with approximately 10<sup>20</sup> atoms/cm<sup>3</sup> of Al using a vertical Bridgman system. Analysis by scanning electron microscopy (SEM), energy dispersive X-ray spectrometry (EDX), X-ray diffraction (XRD), particle induced X-ray emission (PIXE) and particle induced gamma ray emission (PIGE) were used for the structural and compositional characterization of the crystals.

The doped crystals show good structural homogeneity when compared with the undoped alloy, and they were free from cracks and micro cracks. All of the obtained crystals present precipitates, twins and grains with different concentrations of In.

The small compositional variation observed in the crystals along the radial direction (measured by PIXE), may be related to the solid-liquid interface's quasi-equilibrium behavior. Regarding the growth direction, it was observed that the undoped crystals exhibit a higher segregation phenomenon of the third element than the doped crystals.

The obtained results indicate that aluminum influences the indium distribution in the crystals, thus ternary crystals with more homogeneous composition can be obtained and consequently electrical properties improved.

# PB-61: Influence of low-temperature ion implantation on the magnetic properties of Highly Oriented Pyrolytic Graphite

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In the past, intense research has been reported on the modification of magnetic properties in Highly Oriented Pyrolytic Graphite (HOPG) by high-energy ion irradiation [1,2].

We investigated the effect of high-energy ion implantation on the magnetic properties of graphite. Protons with the energy of 500 keV and <sup>12</sup>C ions with the energy of 6 MeV were implanted in HOPG pellets with 3 mm diameter and thickness of 50  $\mu$ m, which were kept at the temperature of -140° C during the ion implantation [3]. At these implantation energies, the depth range of both types of implanted ions amounts 4.3 micrometres. By the selection of the corresponding implantation doses, we approximately matched the depth concentration profiles of induced lattice defects for both types of the implantation. We additionally varied the implantation doses to get insight into the evolution of magnetic properties by increased implantation dose. After the implantation, the samples were handled in Argon atmosphere to prevent against oxidation and incorporation of moisture into graphite.

Electronic paramagnetic resonance (EPR) and SQUID measurements were used to evaluate the effect of ion implantation on the magnetic properties of implanted graphite. The results by EPR revealed the occurrence of paramagnetic centres. With the increase of proton implantation dose, the amount of induced paramagnetic centres increased. On the other hand, after the increase of the <sup>12</sup>C implantation dose, we observed a reduced number of paramagnetic centres. Under the applied implantation doses and the controlled experimental conditions, we did not observe any interaction of the induced paramagnetic centres and excluded a possibility of induced ferromagnetic properties in HOPG.

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### PB-62: Ion beam studies of dual implantation effects on Silicon surfaces

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Silicon is the dominant material in electronics and considered a major player on MEMS contactbased operation (Microelectromechanical systems) devices. However surface properties of silicon at microscale are far from ideal due to large adhesion effects.

In this work we use ion implantation to introduce simultaneously C, Fe and Ti to change the silicon surface properties. Several fluences were implanted at room temperature and the energies selected to obtain the perfect overlap of the implanted profiles. After implantation the samples were annealed at 800 °C and 1000 °C at low pressure (2 x 10 <sup>-6</sup> mbar) and studied by RBS/Channeling combined with scanning electron microscope (FEG-SEM) and grazing incidence x-ray diffraction (GIXRD). The results do not reveal the presence of silicide formation during the implantation even for the highest fluences implanted, 2 x  $10^{17}$  cm<sup>-2</sup>. After annealing a nanostructured surface starts to develop. RBS/Channelling results show a segregation of both Ti and Fe towards the surface for fluences up to 5 x 10 16 cm<sup>-2</sup> while for higher fluences the profiles remain relatively stable. The XRD indicate the presence of different silicide phases for these fluences after the annealings. The silicides are in the form o irregular shaped precipitates covering the surface and randomly oriented with respect to the <001> recrystalization silicon direction.

## PB-63: Lattice location of deep level impurities in hyperdoped Si by ion implantation and short-time annealing

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Impurities play an important role in determining the electrical, optical and structural properties of semiconductors. It has been proposed that deep level impurities, such as Titanium (Ti) or chalcogens in Si, can induce an impurity band inside the bandgap at high enough doping concentration. The insertion of an impurity band can enhance the absorption at a broader wavelength range and leads to applications in the so-called intermediate band solar cell. However, deep level impurities have relatively low solid solubility limit in Si. We prepared deep level impurities doped silicon to above the Mott insulator concentration by ion implantation followed by sub-second annealing. The degree of crystalline lattice recovery in implanted layers and the lattice location of impurities in Si were analyzed by Rutherford backscattering

spectrometry/Channeling. Our results show that S and Se atoms are occupying substitutional lattice sites in Si [1], while Ti impurities have no ordered lattice occupation [2].

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[2] F. Liu, et al., in preparation (2015).

# PB-64: Migration of ion-implanted Ar and Zn in silica determined by backscattering spectrometry and ellipsometry

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SiO<sub>2</sub> is an important insulating material with broad applications in the semiconductor and glass industries; where ion implantation is frequently used as one of the technological steps. In silica the diffusion coefficient of several elements is higher compared to other materials. It is well known that it is extremely high for example for He. Ion implantation modifies also the refractive indices of lots of materials, including silica, which is important for waveguide fabrication. Therefore, the thermal stability of the ion implanted silica layers is always an important issue during device fabrication.

In this work the effect of Ar and Zn ion implantation on silica layers was investigated by Rutherford Backscattering Spectrometry (RBS) and Spectroscopic Ellipsometry (SE). Silica layers produced by chemical vapour deposition technique on single crystal silicon wafers were implanted by Ar and Zn ions with a fluence of  $1-2\times10^{16}$  Ar/cm<sup>2</sup> and  $2.5\times10^{16}$  Zn/cm<sup>2</sup>, respectively. The refractive indices of the implanted silica layers before and after annealing at 300 and 600 °C were determined by SE. The migration of the implanted element was investigated by in-situ annealing up to 500 °C combined with RBS.

It was found that the implanted Ar escapes from the sample at 300  $^{\circ}$ C. Although the refractive indices of the Ar implanted silica layers were increased compared to the as-grown samples, but after the annealing this increase in the refractive indices is vanished. In case of the Zn implanted silica layer both the distribution of the Zn and the change in the refractive indices were found to be stable.

### PB-65: Radiation hardness and polarization studies on thin diamond membrane

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Diamond is perceived to be an extremely radiation hard material, and therefore a potential alternative to silicon in high-radiation environments. However, polarization of diamond detectors under irradiation by densely ionizing particles highly reduces the induced signal and presents one of the biggest drawbacks for practical application of diamond in nuclear and particle physics. This effect is closely correlated with deep electrically active traps that are introduced by high fluence of energetic particles or already exist in intrinsic diamond crystals. Permanent trapping of free charge carriers that were separated by the externally applied bias results in lowering of the effective electric field inside the device. A thin diamond membrane represents an ideal sample for investigating the effect of polarization. Because of its small thickness it allows for build-up of extremely high electric fields that can reveal the saturation level of the polarization. Such saturating behaviour could not be observed with thick detectors where signal amplitude rapidly decreases below the noise level. Diamond membrane of 6 µm thickness was irradiated by several fluences of fully penetrating proton microbeam and a detailed radiation hardness analysis was performed by the IBIC technique. Afterwards, polarization tests were done with different ion beams on virgin as well as on damaged areas. During these tests, the signal degradation was monitored in dependence of the totally deposited ionizing energy of short range impinging ions. The results will be presented together with a simple modelling of the polarization effect.

### PB-66: Oxidation of nickel surfaces by low energy ion bombardment

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Nickel oxide (NiO) is a good example of a p-type semiconducting oxide, with a wide range of possible applications, from electrochromic devices or chemical sensors to antiferromagnetic layers in spin valve structures. The quality of devices depends critically on the electrical properties of NiO films, thus it is important to fully understand the oxidation mechanisms of nickel that seems to be quite complex [1].

We have shown recently that thin NiO films can be formed by bombardment of Ni surfaces with low energy oxygen ions [2,3]. In the present study, we compare the formation of NiO films by the  $O_2^+$  ion bombardment in the energy range of 0.5 - 5 keV with the thermal oxidation (RT to 500 °C) in oxygen atmosphere within an analytical ultrahigh vacuum chamber. The NiO films were analysed by X-ray Photoelectron Spectroscopy (XPS) and Secondary Ion Mass Spectrometry (SIMS) in order to determine different oxidation stages, stoichiometry and thickness of oxide films. We show that low energy oxygen-ion bombardment produces compact, more ordered and less amorphous oxide structures than films formed by some electrochemical methods.

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### PB-67: Investigation of damage cross section and yield dependence on experimental parameters for MeV SIMS

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In 2012 a Time-of-Flight Secondary Ion Mass Spectrometer using MeV energy heavy ions (MeV ToF-SIMS) was built at the Ruđer Bošković Institute (RBI) in Zagreb. Secondary molecular ions are extracted from the sample after the impact of heavy MeV Ions (primary Ions) using an HV acceleration potential difference between the sample surface and a grounded extractor. Using this technique imaging of heavy molecules (up to 1000 Da) is possible due to the increased yield produced in SIMS with MeV primary Ions.

First experiments using this setup have hinted on a strong dependency of the secondary ion yield on experimental parameters such as primary ion type and energy as well as setup characteristics (sample orientation and distance from the extractor). In order to explore in detail these dependencies a stable geometry and reliable measurement of the ion beam current is necessary. This can be a challenging task in a MeV SIMS setup since low microbeam currents (often lower than 1 pA) are used. This is particularly the case for heavy ions as they produce a high number of secondary electrons. Measurement of the current directly from the sample itself or by using a Faraday cup behind the sample is not reliable under these circumstances, especially as the sample holder for the MeV ToF SIMS setup is on a HV potential (5 kV at the RBI setup).

In order to enable reliable measurement of ion beam currents down to a fA range, that is needed to investigate the dependence of MeV ToF-SIMS yields on different ion microbeam parameters, we have considered several approaches. One alternative is indirect determination using RBS which is only effective for beam currents that are relatively high for MeV ToF SIMS. Therefore a more reliable and reproducible method has been selected which involves direct current measurement using a PIN-diode that occasionally intercepts the ion beam. This method has been realized at the RBI microprobe beamline using a stepper motor driven PIN diode holder.

In this work we present the design of the constructed system, its performance in terms of reproducibility, yield measurements for different sample types and a first measurement of the damage cross section. The measurement of the damage cross section is a value independent of the setup used and may lead to a better understanding of the processes occurring during electronic stopping and MeV SIMS.

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### PB-68: Simplified annular gas ionization chamber for backscattering experiments

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A new compact and simple gas ionization chamber (GIC) with an annular geometry has been built and tested for the use as backscattering detector. The design corresponds to a significantly downsized and simplified version of the chamber concept presented in [1]. Basically it consists of a cylindrical housing with 40 mm outer diameter and a 1/16" stainless steel capillary (1 mm inner diameter) mounted on the central axis which is guiding the primary ion beam through the detector. An isolated copper tube is slipped over the capillary and serves as detector anode. No Frisch-grid is mounted, which makes the assembly of the detector rather simple. Eight silicon nitride membranes (1 x 1 mm<sup>2</sup>, 90 nm thickness) are glued on the front side of the detector at a radius of 7 mm around the beam axis. Isobutane is used as detector gas and the electronic noise level is minimized by an Amptek CoolFET<sup>®</sup> preamplifier.

Because of the missing Frisch-grid the influence on the detector output signal by positive charge carriers produced along the ion track has to be minimized. Therefore, the detector is operated at high gas pressure of several hundred mbar in order to keep the absolute spatial spread of ions stopped in the gas as small as possible. To compensate for the reduction in electron drift velocity at high pressure the bias voltage has to be raised as well (few hundred V) [2].

First RBS tests have been performed with a 2 MeV He beam and an  $AI_2O_3$  layer on aluminum. The distance between detector and sample is about 50 mm resulting in a backscattering angle of 8° and a covered solid angle of 3.2 msr. In this configuration a resolution of 17 keV and 19 keV (FWHM) was obtained at the O and Al edge, respectively. The resolution was thereby dominated by the contribution of the electronic noise, which amounted to 15 keV. Latest results and possible applications of this simple GIC concept for backscattering experiments are discussed.

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### PB-69: Low cost digitisation of data for Ion Beam Analysis (IBA)

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The availability of high-performance micro-controllers with universal serial bus (USB) interfaces and analogue and digital inputs and outputs offer a new low-cost approach to data acquisition

that can be used at extremely for many ion beam analysis applications. While these have been used for some time in portable survey instruments, where the data rates are often low, the requirements for ion beam analysis are quite stringent. In particular, the paralysed and nonparalysed dead-time as well as the differential and integral non-linearity can have a significant impact on the measured spectral data. In this work experience as well as practical circuits and software codes from development of two instruments with a component cost of less than 100 euros are presented. The first instrument is a two-channel NIM ADC event-mode interface (EMI). This reads out two legacy NIM 100 MHz Wilkinson analogue to digital converters (ADC) to a USB serial interface. The instrument is used for digitising signals from high-resolution magnetic spectrometer for IBA and feeding them to a host computer. The throughput for IBA applications of the EMI is limited by the ADC performance. The second instrument is a headless single-channel multichannel analyser (MCA) that uses the on-chip ADC together with a fast precision peak detector circuit for digitising the peak amplitude of Gaussian-like pulses from a spectroscopy amplifier. Using an Arduino Mega, count-rates of 300/s could be maintained with dead-time loss of <3.5%. This is acceptable for many IBA applications. This could be improved using an ARM-based chip (Due) with a faster ADC so it reaches/or exceeds the throughput performance of a standard Wilkinson ADC. The stochastic arrival time of the pulses in IBA measurements implied the same microcontroller code could be used in both cases. This consisted of two concurrently executing strings, one of which feeds the ADC conversion values to a buffer and is initiated by the ADC service interrupt. The other is a state-machine that polls the USB serial input for command bytes and writes out the buffer contents to the serial interface and also performs housekeeping tasks. The host processor code that was implemented in the proprietary LabVIEW environment was also as state-machine that performs the starting/stopping/zeroing, sorting, display and storage of data.

### PB-70: Digital Pulse Processing for Ion Beam Analysis at ETH Zurich

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For many IBA experiments purely analog data acquisition (DAQ) chains are in use. This traditional approach typically requires several signal shaping and data handling modules, which are designed to the particular application specifications. The final step of such an acquisition chain is the digitization of the processed signals and forwarding to a connected data storage PC.

Recent developments in the section of digital pulse processing and the usage of modern field programmable gate arrays (FPGAs) open the field of single-board multi-purpose solutions at moderate cost. A state-of-the-art signal digitizer can directly process detector output signals or pre-amplified signals and extract the desired information on-board. In opposite to analog approaches, the incoming signals are digitized at the input stage and the following signal treatment is handled by digital filters. The usage of FPGAs allows fast switching of configurations and therefore the use of one digitizer system for several experiments.

At ETH Zurich two different commercial CAEN digitizer models (DT5724 and DT5780) are used for digital acquisition with custom made readout software. Both models are equipped with similar hardware in the signal processing part, which allows the usage of common DAQ software. Performance studies with RBS measurements will be shown for a typical analog DAQ chain compared to the two digitizer models. Furthermore, first results of the usage for Time-of-Flight (ToF) measurements and the continuative developments towards a DAQ system for the new MeV SIMS setup at ETH Zurich [1] will be presented.

[1] Presentation: M. Schulte-Borchers et al., "Recent progress on the new MeV SIMS setup at ETH Zurich"

### PB-71: Depth resolution of TOF-ERDA measurement using He and Cu beams

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A Time-of-flight Elastic Recoil Detection Analysis (TOF-ERDA) is effective for light element analysis and depth profiling of thin films thanks to the availability of analysis techniques with high depth resolution. We have developed a TOF-ERDA measurement system mainly for the purpose of thin film analysis [1]. This paper reports results of depth resolution measurements for a light element achieved by our TOF-ERDA system.

Details of the TOF-ERDA system were presented in [1]. Carbon thin layers (about 50nm thickness) coated on Si wafers were used as samples for measurements of depth resolution. He beams with energies of 2.0, 3.5, 5.7 MeV and Cu beams with energies of 5.0, 12.0, 20.0 MeV were utilized as incident beams. The incident/exit angles with respect to the sample surface were 20°/20°, 30°/10°, 35°/5° for the measurements with He beams and 20°/20°, 35°/5° for the measurements with Cu beams, respectively.

Energy broadenings at the surface was obtained from TOF spectra of recoil carbon. Depth resolutions were evaluated from the energy broadenings. Results are shown in Fig. 1. It is shown that the depth resolution becomes higher with decreasing the incident energy and with decreasing the exit angle for both beams. Depth resolution of  $1.3 \pm 0.1$  nm was achieved in the case of the He beam of 2.0-MeV incident energy, incident/exit angles of  $35^{\circ}/5^{\circ}$ .





[1] Keisuke Yasuda, Yoshinori Nakata, Ryoya Ishigami and Shogo Hibi, IBA2015 abstract.

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#### PB-72: In situ hydrogen isotope detection by ion beam methods ERDA and NRA

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There are not many methods available for detection of hydrogen isotopes in materials, without destroying the material or outgasing the hydrogen from it. Elastic Recoil Detection Analysis (ERDA) and Nuclear Reaction Analysis (NRA) are two of the few methods that are able to detect hydrogen and deuterium without having major impact on the sample properties or hydrogen content. In the case of ERDA one can detect both H and D at the same time down to few hundred nm from the surface, depending on the substrate material, being less for heavier substrates. On the other hand the NRA method utilizing the D(3He,p) $\alpha$  nuclear reaction is sensitive only for deuterium but the analyzing depth is bigger, down to few µm, and the sensitivity is better. The choice of the method depends on the processes that one wants to When studying hydrogen isotope interaction with materials such as adsorption, study. absorption, bulk diffusion, desorption or isotope exchange the optimal way is by analyzing the hydrogen isotope content in the sample in situ and in real time during specific experiments including exposure to hydrogen isotopes and varying sample temperature. Hydrogen is a light material and it easily diffuses through and out of the material during the time between exposure and analysis or interacts with adsorbing species such as water or hydrocarbons. Therefore it is best to avoid sample exposure to air between experiment and measurement. For detailed studies of hydrogen interaction with materials we have developed a system enabling us to analyze in situ hydrogen and deuterium concentration in the material by ion beam methods during the experiment. In the dedicated chamber at 2 MV tandetron accelerator the samples can be exposed to D or H neutral atom beam at different sample temperatures and during the exposure the H and D concentration is measured. Moreover the hydrogen isotope thermal desorption or adsorption can be followed during the sample heating or cooling or at constant temperature from room temperature to 900 K, employing computer controlled heater. We will present the principles of using the two IBA methods for the detection of hydrogen isotope concentration in the material in situ and in real time and the processes that can be studied at hydrogen interaction with materials. Examples of the studied processes will be presented together with the constraints for each method. In the case of ERDA we studied processes on the surface of tungsten [1] and amorphous hydrogenated carbon [2]. We have studied thermal adsorption of deuterium and isotope exchange on tungsten. Deuterium retention and diffusion deeper in the material was studied by NRA. Here the studies were preformed on W material irradiated by high energy W ions, simulating the neutron damage in fusion reactor [3] and on mixed layers (W-C, W-Al, W-Be) [4]. Moreover, we have studied also isotope exchange of hydrogen isotopes in the bulk of damaged tungsten material and thermal desorption of deuterium. The influence of the probing beam on the studied processes will be discussed.

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### PB-73: A novel method to survey parameters of an ion beam and its interaction with a target

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Beam profile and composition of the pulsed ion beam from a vacuum arc source with deuterated electrodes are useful information for designing a high intensity deuterium-tritium neutron generator. Traditional methods are hard to get the information at the same time. A novel off-line diagnosis method is presented which can get the profile of the beam with high resolution as well as species of the ions in the beam. The method is using a silicon target with high purity to interact with the ion beam, and then use secondary ion mass spectrometry (SIMS) to analysis the silicon target interaction zone to get the beam information. More information on beam-target interaction could be got simultaneously. Proof-of-principle simulation and experiment works have demonstrated this method is practical.

### PB-74: The Novel Technique of Coincident Rutherford Backscattering Spectrometry

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Coincident Rutherford Backscattering Spectrometry (CRBS) is a novel combination of traditional Rutherford backscattering spectrometry (RBS), time-of-flight (TOF) coincidence, and positionimaging techniques. This combination aims at investigation of charge-state distributions of both backscattered and recoil ions in violent ion-atom collisions. A special apparatus was designed, fabricated, and installed at the end station of the RBS-beamline in the University of Jordan Van de Graaff Accelerator (JUVAC) [1]. CRBS measurements for He<sup>+</sup>–Ar collisions [2] and O<sup>+</sup>-Kr collisions were performed. From the recoil ion-projectile ion coincidence measurements of the charge state distributions, it was observed that backscattered projectile ions of the same charge state correlate with different recoil ion charge states and vice versa, indicating that any particular charge state may result from different reaction channels. The novel CRBS technique provides much more detailed information than conventional RBS. Additionally, CRBS measurements may shed some light on the microscopic phenomena which dictate specific excitation processes and may lead to a better understanding of the macroscopic phenomena of energy loss and charge state evolution of ions traversing gas targets as a prelude to simulate solid ones. It is worth mentioning that, to the best of our knowledge, our new approach of combining conventional RBS with TOF in coincidence measurements has not been attempted previously.

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# PB-75: An on-line fluence detector for precise irradiation measurements with pulsed ion beams

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One of the main applications of thermoluminescent (TL) materials such as, for example, quartz and feldspars, has been in the area of dating [1]. The dating protocols are based on the analysis of the TL response of these materials to precisely known radiation doses.

At LABEC, the 3 MV Tandem accelerator laboratory in Florence, an experimental apparatus has been developed for monitoring the ion fluence during irradiation measurements with protons, alpha-particles and heavy ion beams (Li, C, O etc.) of different energy and with fluences in the range  $10^{5} - 10^{11}$  cm<sup>-2</sup>.

Irradiations are carried out using a beam line equipped with an electrostatic deflector (DEFEL in the following: see ref. [2,3]). Beam pulsing is achieved by quickly sweeping the beam transversely with respect to the beam direction through a pair of adjustable slits. The bunch frequency can be set from single pulse up to 10 kHz and the average number of particles per bunch can be varied from <1 to many hundreds and more. If the beam current intensity remains constant the distribution of the number of particles inside the bunches follows a Poisson statistics with a well defined mean value.

Anyway, especially during long irradiations, the current intensity may not stay constant throughout the measurement. Therefore, in order to precisely control the ion fluence during the irradiation measurement a thin Al foil positioned along the beam path is crossed by the ions before reaching the target. A MicroChannel Plate (MCP) multiplier detects the electrons emitted from the foil. The response of the foil-MCP system is calibrated by means of a silicon detector positioned downstream the foil that, because of its good energy resolution, makes it possible to evaluate the particle multiplicity of each bunch and so the average multiplicity. The samples are housed inside a vacuum chamber at the end of DEFEL beam line and can be positioned in front of the beam by means of remotely controlled x-y linear displacements. After calibration the target is positioned in place of the silicon detector, it is irradiated with the desired number of bunches, later on the delivered dose is calculated.

Preliminary measurements performed with 7.5 MeV Li<sup>2+</sup> and 10 MeV O<sup>3+</sup> ions have shown that, after calibration, the foil-MCP system measures the particle fluence with a precision better than

1% also in case of large beam current fluctuations.

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### PB-76: Development of an angular sensitive time of flight detector for heavy ion ERDA

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Elastic recoil detection analysis (ERDA) is performed at the Munich Tandem Accelerator utilizing a Q3D magnetic spectrograph in order to obtain high resolution depth profiles for a single element in a single run. For simultaneously monitoring multiple elements of the sample a TOF-E setup has been installed.

The depth resolution in the TOF-E measurement however is affected through kinematics by a large solid angle of detection and by the enlarged beam spot due to the incident angles smaller than 5° with respect to the sample surface, required for optimal depth resolution for the Q3D measurements. In order to correct both kinematic contributions a true angular measurement of every recoil ion trajectory is required.

A new TOF detector concept has been developed which is based on secondary electron emission/detection from a carbon foil in a straight geometry towards a MCP stack for combined position and time detection.

First tests of the MCP module using alpha particles show an intrinsic time resolution of 100 ps FWHM and a principle intrinsic position resolution achieved by a 2D-Backgammon anode readout of 60  $\mu$ m FHWM. However the position resolution is highly affected by the momentum distribution of the secondary electrons ejected from the carbon foil. Therefore an electrostatic lens has also been developed to focus the electrons onto the MCP surface thus maintaining position information.

Simulations of the complete setup including the electron transport through the lens show a possible overall position resolution of 0.6 mm FWHM as well as a contribution of the lens to the time resolution of 80 ps FWHM. The experimental performance of the position resolving TOF detector is under study and will be shown.

#### PB-77: Depth profiles of D and T in Metal-hydride films up to large depth

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Metal hydrides are used in an increasing broad range areas and disciplines. A significant application is in the neutron generating devices in which the hydrides of transition or rare earth metals involving D and T are used as target materials bombarded by D ions. The exact determination of D and T concentration in the films with thickness up to several  $\mu$ m's is of importance. An analysis technique combining elastic recoil detection (ERD) and particles identifying system of  $\Delta E$ -E telescope detector may be used for simultaneous profiling of all hydrogen isotopes in the thick hydride films. However very high-energy heavy ions and extremely high standard  $\Delta E$ -E identifying system for very wide- energy range of recoil particles are needed to achieve large analysis depth up to above 5 µm. In this presentation, a method combining D(<sup>3</sup>He, p)<sup>4</sup>He nuclear reaction and proton backscattering (PBS) was adopted in order to detect D and T in Ti(D,T)/Mo film by adopting much low-energy ions compared to ERD method. The PBS is first used to measure the T and D concentration in the limiting depth range and depth distribution of Ti and Mo over whole film, and then D(<sup>3</sup>He, p)<sup>4</sup>He is used to determine D concentration over large depth and to deduce T. Increasing <sup>3</sup>He energy can increase detecting depth greatly and 3 MeV <sup>3</sup>He can achieve depth profiling up to 5  $\mu$ m. The depth resolution is a very important factor in depth profiling of hydrogen isotopes. Usually, the PBS analysis produces poor depth resolution, and a single incident energy for  $D(^{3}He, p)^{4}He$ analysis can achieves an acceptable depth resolution only in a limited depth range. By carefully varying incident energies, an optimum resolution of less than 0.5 µm for D and T distribution throughout the whole analyzed range can be obtained.

### PB-78: Development of a versatile user-friend IBA reaction chamber

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Most light elements appear as hydrides, borides, carbides, nitrides and oxides in the nearsurface region of engineered bulk materials and thin films. Composition and distribution of these light element compounds govern to a great extent, the bulk material's surface mechanical, optical, electrical and chemical properties. Reliable measurement of these light elements and their distribution, and knowledge on controlling and manipulating them are required to enhance the performance of materials and their functional devices. Nuclear Reaction Analysis (NRA) and Elastic Recoil Detection (ERD) Analysis are the two key acceleratorbased nuclear techniques for sensitive and selective tracing of light elements. To get the most of these analysis techniques, not only experimental nuclear cross-sections relevant to ion beam analysis and powerful IBA analysis software should be developed but also reliable experimental setups are required. To meet the latter requirement, recently a user-friend reaction chamber for IBA analysis is developed in Van de Graaff laboratory in Tehran with the following features and capabilities which greatly simplify the NRA and ERD analyzes.

- measurement of charged particles at fixed relevant IBA scattering angles of 0°, 30°, 60°, 90°, 135° and 165° symmetrically at both sides of the incident beam direction with the capability of altering detector position

- measurement of charged particles at any desired scattering angles

- measurement of gamma rays and X-rays at the scattering angles of 90° and 135°, respectively at both sides of the incident beam direction

- varying the polar angle ( $\theta$ ) of the sample wheel

-transverse adjustment of sample wheel to compensate the target thickness

- possibility of simultaneous accommodation of 12 samples on the sample wheel

In most commercially available IBA reaction chambers, 3-axis goniometer (x,y and z motions) are generally used for manipulation of the sample, while only a few samples could be loaded in the sample holder. However, in our developed heuristic sample manipulator, not only polar angle of the sample could be controlled, sample thickness could be readily compensated. Moreover, higher number of samples could be simultaneously loaded in the sample wheel. To confirm the performance and capability of the newly developed reaction chamber, relevant case study has been accomplished and presented.

# PB-79: Effect of Swift Heavy Ion Beam Irradiation on Europium Oxide Resistive switching Non volatile Memory

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Resistive switching memory effect observed in binary metal oxides is a fascinating phenomenon for the next generation non volatile memory. A simple Metal-Insulator-Metal (MIM) and Metal-Insulator-Semiconductor (MIS) device structure is the specialty of upcoming Resistive switching Random Access Memory (ReRAM). Present work reports fabrication of MIM structure comprising Al/Eu<sub>2</sub>O<sub>3</sub>/Al on a glass substrate. I-V characterization of this device has exhibited bipolar switching characteristics indicating a possibility of the use of  $Eu_2O_3$  as a novel material for ReRAM. It is essential to understand the effect of Ion beam irradiation on the resistive switching characteristics for the probable use of  $Eu_2O_3$  in space as a non volatile memory. Present work reports the effect of Ion beam irradiation and gamma irradiation on the  $Al/Eu_2O_3/Al$  device.

### PB-80: Continuous observation of IBIL spectra obtained from microscopic targets

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In this study, we demonstrate continuous spectroscopy of ion beam induced luminescence (IBIL) obtained from microscopic targets. IBIL is one of ion beam analysis (IBA) technique which has sensitivity to chemical composition of the target [1,2]. IBIL from inorganic and organic target were well obtained under proton microbeam irradiation condition [3]. However, effects

of irradiation damage caused by probe are not well evaluated, but it might severely influence the analytical result of IBIL. Qualitative characterization of IBIL spectrum is therefore required to evaluate the suitability of IBIL as the analytical tool of chemical composition.

Proton microbeam with a diameter of 1  $\mu$ m was employed for continuous measurement of IBIL from several microscopic targets. In addition to general inorganics microscopic targets, several organic materials i.e. nicotinamide adenine dinucleotide (NADH), tryptophan, and riboflavin, and a polycyclic aromatic hydrocarbon (PAH) were prepared as irradiation target. Measurement system of IBIL with microscopic optics was capable to obtain spectrum continuously during irradiation [4]. Decay of IL was recorded from various targets of IBIL. Structure of IBIL spectrum from PAH was differed from its original shape obtained at the beginning of the irradiation. The details of the changes appeared in both number of peaks and structure of IBIL spectrum will be discussed in the presentation.

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### PB-81: Total-IBA for serving Metrology

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Metrology is defined by the International Bureau of Weights and Measures as "the science of measurement, embracing both experimental and theoretical determinations at any level of uncertainty in any field of science and technology". Implementing a Metrological approach within measurement processes enhances the reliability of the results. These developments include (among other things): obtaining high quality standards (such as Certified Reference Materials); developing and validating analytical methods (such as RBS); assessing uncertainties robustly (using formal Uncertainty Budgets [1]) and establishing the traceability of the measurements (e.g: of stopping power to quantify RBS [2]).

To determine the elemental composition of thin films of the order of a micron or so thickness, these primary methods are usually used: INAA, ED-XRF, EPMA, ICP-MS, SIMS, XPS or IBA. Among these techniques, only IBA is capable of the high accuracy, non-destructive and model-free characterisation of samples inhomogeneous in depth.

We have demonstrated that RBS (Rutherford BS) can be used as a primary direct method capable of an absolute traceable accuracy of 1% (or better) for the measurement of quantity of material even where the measured element has an arbitrary depth profile [1]. The robustness and repeatability of RBS has been demonstrated through the analysis at a precision of 0.3% of standard samples over the past three years. The extension of this technique to the important cases of certifying SIMS standards or the thickness of self-supported thin foils for calibrating

XRF has also been proven.

One corollary of this work is a new recognition of the very high linearity of IBA spectrometry systems, which has permitted the energy of the 3038 keV  $^{16}O(a,a)^{16}O$  resonance to be determined with a significantly smaller uncertainty (of 1.3 keV, previously thought to be achievable only with gamma spectrometry [3]). The data tables from the TUNL group show an uncertainty for this resonance position of 2.3 keV [4].

PIXE (Particle-Induced X-ray Emission) is always simultaneously available with BS (as are also other IBA techniques), and PIXE is almost perfectly complementary to BS. Where BS is weak (sensitivity and mass resolution) PIXE is strong, and where PIXE is weak (absolute accuracy and depth resolution) BS is strong. Total-IBA [5] (the synergistic use of multiple IBA techniques) promises a range of new metrological applications, and we will discuss the uncertainty analysis in a number of interesting cases.

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### PB-82: Deuterium Microscopy using 17 MeV Deuteron-Deuteron Scattering

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Nearly background free elemental analysis is possible using the coincidence pattern of elastic scattering reactions. This has been demonstrated by proton-proton (pp) scattering for hydrogen analysis. Microscopy with sub-µm-resolution and sensitivity below 0.1 at-ppm for hydrogen analysis becomes possible at these energies due to the large elastic scattering cross section enhancement and lowest irradiation damage potential. Using 17 MeV deuterons as primary beam, we performed Deuterium microscopy by using the deuteron-deuteron (dd) scattering reaction. The high energies available at the microprobe SNAKE are required for this analysis of thin freestanding samples so that the scattered particles are transmitted to the 1 mm thick Si strip detector pairs covering about 2.5 sr solid angle of detection. The cross section for the elastic scattering reaction is about the same as for pp-scattering (~100 mb/sr). The main background due to nuclear reactions is outside the energy window so that ppm sensitivity is also available for Deuterium microscopy. dp-scattering events give an additional signal for Hydrogen atoms, so the H/D-ratio is monitored in parallel. This coincidence analysis becomes a valuable tool for studies of hydrogen incorporation or dynamic processes using Deuterium marking. The background from natural hydrocarbon or water contamination is eliminated. As a demonstration we present measurements on deuterated Poly-Propylen-sheets as well as first application to 3D deuterium microscopy on Tungsten foils.

### PB-83: Functionalized collectors for storing matter technique applied in secondary ion mass spectrometry

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We present results of the use of functionalized substrates that serve as collectors in 'storing matter', the quantitative technique of secondary ion mass spectrometry (SIMS). This technique allows separating the two base processes of secondary ion formation in SIMS. Namely, the process of ion sputtering is separated from the process of ionisation. The technique allows sputtering of the analysed sample and storing the sputtered material, with submonolayer coverage, onto a collector surface. Such deposits can be then analysed by SIMS and as a result, the so called 'matrix effects' are significantly reduced [1, 2]. We perform deposition of the sputtered material onto a variety of substrates, including functionalized substrates. The process of sputtering is carried within the same vacuum chamber where the SIMS analysis of the collected material is performed. For sputtering we use 5 keV Ar<sup>+</sup> beam of 1  $\mu$ A, and for SIMS analysis of the deposited material the  $Ar^+$  5 keV beam of 0.1  $\mu$ A. The presented results are obtained with the use of stationary and also with rotating collectors. Here we present a case study of ion sputtering of chromium. As collectors we use titanium, copper, nickel and coppernickel alloy. The electrolytic processes are used in order to obtain partial coverage of gold. We also tested, how several additions to electrolytic solvent as caesium chloride and several others, influence functionality of collectors. The obtained results show that the functionalised substrates used as collectors can increase adhesion of the sputtered material. Also ionization of the deposit is enhanced with respect to the pure material collectors. However, use of such multi-elemental substrates limits the range of possible elements for the analysis, because the background mass spectrum is significantly richer than the mass spectrum of pure elemental substrates.

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### PB-84: Optimum ion and energy combination for ambient pressure MeV-SIMS

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Secondary Ion Mass Spectrometry induced by heavy primary ions (MeV-SIMS) is a promising step towards molecular speciation in Ion Beam Analysis. Submicron molecular concentration mapping can be performed via focusing and scanning the beam, without marking the sample. A most promising possibility offered by swift heavy ions is the possibility to extract the beam in the ambient environment, while maintaining spatial resolution under 5  $\mu$ m. A strong interest arises in numerous applications such as archaeometry, forensics, biology, or biomedical sciences, where fragile, volatile or vacuum-alterable samples are to be characterised.

An ambient pressure MeV-SIMS set-up has been developed at the Surrey Ion Beam Centre. It consists of a quadrupole mass spectrometer coupled to an orthogonal time of flight mass spectrometer installed on an external micro-beam line. Secondary ions are collected thanks to a sniffing capillary inlet above the surface of the sample, which is placed a few millimetres behind a 200 nm  $Si_3N_4$  window. An atmospheric chamber can be used in order to control the gaseous environment surrounding the sample.

In this work, we will present the dependence in molecular yields (Y<sub>0</sub>), damage cross sections (s) and efficiencies (Y<sub>0</sub>/s) from thin organic layers on Si and caffeine standards, bombarded by a range of ions ( $^{16}O^{n+}$ ,  $^{35}Cl^{n+}$ ,  $^{44}Ti^{n+}$ ,  $^{63}Cu^{n+}$ ) with different energies up to 10 MeV. The results along with simulations are used to determine the optimum ion and energy combination to use for ambient pressure MeV-SIMS using a 2 MV tandem type accelerator.

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### PB-85: Possibility of light element ERDA using pulse shape identification technique

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Pulse shape identification technique as a tool for discrimination of different ions is known and established technique in the energy ranges of several MeV/amu and more [1]. It can effectively replace deltaE-E telescopes and other techniques by simplifying experimental setup and reducing the number of detectors on the expense of need for more sophisticated electronics. Elastic recoil detection analysis (ERDA) is well-established ion beam analysis technique utilized

in different ways, depending on the range of detected ion masses, energies and requirements on depth resolution. Pulse shape discrimination in the alpha-particle ERDA has been used in past together with the analog electronics chain [2]. Analog signal processing introduces significant limitations to the method as different characteristics of the signal shape can be utilized in the particle identification. With the development of high sampling rate analog-todigital converters and their decrease in price it is now feasible to collect the total current trace of the signal in one channel and its energy amplitude in another channel. The post analysis can then be optimized for the best mass resolution of the detector [3]. In this work we present examples of particle identification for light elements while application to ERDA and limitations will be discussed.

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# PB-86: New automated sample holder/scanner for PIXE-PIGE-IBIL fast 2D-macro-mappings of materials of geological, environmental and archeological interests

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For a wide range of materials and fields of investigations, quantitative macro mappings of large areas may be favored to multiple points selections, as long as the heterogeneity of the material is a priori known. In the framework of recent collaborations currently going on in our laboratory, a new automated sample holder scanner and a suited data acquisition system has therefore been developed and installed on the "Arkeo" external macro-beam line of the AVF-Cyclotron of the Institut de Physique Nucléaire Atomique et de Spectroscopie in Liege University. Routine 3 MeV proton beams were used in external beam mode under Helium flux to investigate potential benefits of automating sample scanning procedure and provide chemical 2D maps thus, for the analysis of various kinds of large samples. Both low energy and

high energy X-ray as well as Gamma rays were collected with the especially upgraded data acquisition system installed on the existing conventional PIXE-PIGE-IBIL detection set-up to test its performances and its suitability to address specific needs and questions raised in the fields of geology, archeology or in the frame of environmental studies. We first present results of an inter-laboratory and inter-techniques comparison initiated by Liege department of geology. As XRF Core Scanners are extensively used in geology for sediments investigations, the aim was also to compare PIXE-PIGE technique in scanning mode and XRF capabilities and to determine the best combination of techniques and sample preparation protocol to be applied for lake sediments core samples. The quantitative analysis of same sets of humid and dried sediments cores collected on Amik lake, location of Roman ancient city Antioch, in the south of Turkey were performed with all techniques and set-ups routinely in use in three different laboratories. Results and comparison of detection limits will be provided. Finally special features of a continuous scanning mode will be illustrated with recent applications for PIXE-PIGE analysis of environmental aerosol filters used in an air sampling campaign.

### PB-87: Detection limit of hydrogen in high resolution ERD

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Elastic Recoil Detection analysis (ERD) is a powerful ion beam analysis for quantitative and nonmatrix effect depth profiling of elements. In particular, ERD is the most widely used to detect hydrogen. Recently, we developed high-resolution ERD (HERD), which has better depth resolution [1]. HERD has advantage of detecting hydrogen with sub-nm depth resolution. On the other hand, there is no detailed report on detection limit of hydrogen with HERD, although it is important for measuring the materials having little hydrogen inside. Thus, we evaluated the detection limit by measuring hydrogenated amorphous carbon (a-C:H). HERD was performed at the accelerator facility in Univ. of Tsukuba. The a-C:H/Si samples were prepared by plasma chemical vapor deposition. To characterize the standard samples, we estimated the chemical composition of a-C:H/Si samples by conventional ERD and Rutherford Backscattering Spectroscopy (RBS). As probe ions in HERD, the 500 keV <sup>16</sup>O<sup>+</sup> ions were irradiated to the sample with angle of 75° from the surface normal. We measured the recoiled hydrogen having the angle of 30° respect to the incident beam direction.

Detection limit is defined as the density estimated when the net counts ( $N_{net}$ ) equals to statistical error of net counts ( $dN_{net}$ ).  $N_{net}$  is difference between measurement counts and background noise counts ( $N_{bg}$ ), and  $dN_{net}$  is described as ( $N_{net} + 2 N_{bg}$ )<sup>1/2</sup> by error propagation. There are two main background noises on HERD, one is the stray particles scattered from the inner wall of vacuum chamber, and the other is dark current generated on the Position Sensitive Detector. H. Hashimoto et. al. removed background noise of stray particles by mounting Mylar films in front of the detector in order to improve the detection limit on high resolution RBS [2]. This is because the almost stray particles are enough energy to be stopped on Mylar film in case of HRBS. Therefore the background noise contributed only dark current

described as  $N_{bg} = bT$  ( $N_{bg}$ , b and T are background noise counts, constant and measurement time respectively). Thus, we also put Mylar film (thickness; 0.5 µm) in front of the detector. From the HERD measurement on a-C:H, we found that the background noise of stray particles depend on density of hydrogen in sample in spite of mounting Mylar film. This means that stray recoiled hydrogen are dominant stray particles in case of HERD. Thus, we cannot define the  $N_{bg}$ is just proportional to measurement time. In order to estimate correctly detection limit on hydrogen by HERD,  $N_{bg}$  was re-defined as  $N_{bg} = bT + cDIT$  (c, D and I are constant, density of hydrogen in samples and beam current respectively). The first term is counts of dark current, and second term is counts of stray recoiled hydrogen. As a result, detection limit on hydrogen was estimated at 4.4 x 10<sup>19</sup> /cm<sup>3</sup> (measurement time; 300 sec) in HERD.

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## PB-88: A versatile beam line at Chiang Mai University for ion-beam analysis, lithography and implantation

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A versatile ion beam line based on a 1.7-MV Tandetron tandem accelerator has been established at Chiang Mai University, Thailand in a compact manner for multiple-purpose ion beam applications. The Tandetron accelerator employs two ion sources, a duoplasmatron ion source and a sputter ion source, capable of producing light ion species such as hydrogen and helium for normal ion beam analysis and lithography and heavy species such as carbon and oxygen for ion implantation and heavy-ion beam analysis. The ion beam analysis techniques currently available include Rutherford Backscattering Spectrometry (RBS), RBS/channeling, Elastic Backscattering (EBS), Particle Induced X-ray Emission (PIXE) and Ionoluminescence (IL) with assistance of commercial and in-house-developed softwares. Micro ion beams for MeV-ion beam lithography are developed utilizing inexpensive programmable aperture or capillary focusing techniques. Ion beam analysis experiments and applications have been vigorously performed, especially for novel materials analysis focused on archeological, gemological, biological and forensic materials besides other conventional materials. Ion beam lithography is applied for fabrication of microfluidic chip patterns. Heavy ion implantation of local gemstones and semiconductor materials is operated for enhancement of the gem quality and induction of nanostructure. The success in the beam line development and applications demonstrates

complex technology establishment possible in a developing country with our limited resources.

### PB-89: Development of TOF-ERDA measurement system for thin film analysis

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A Time-of-flight Elastic Recoil Detection Analysis (TOF-ERDA) is one of the promising methods for the simultaneous measurements of multi light elements with good depth resolution. Therefore the TOF-ERDA is especially effective for light element analysis of thin films. We have developed a TOF-ERDA measurement system for thin film analysis at the Wakasa Wan Energy Research Center, Japan. This paper describes details of the TOF-ERDA measurement system and results of performance tests

A schematic view of the measurement system is shown in Fig. 1. The system consists of two time detectors of the ion transmission type [1] and a silicon surface-barrier detector (SSB). The time detectors are used for the TOF measurement, whereas the SSB is for the energy measurement. The forward time detector is set in the target chamber, and the backward time detector and SSB are in a small vacuum chamber connected to the target chamber with vacuum pipe. Distance between two time detectors, which corresponds to the flight path, is 611 mm. Time resolution of the TOF measurement was 0.39 nsec (FWHM), which was obtained by the measurement of helium ions scattered by a thin gold layer.

A TOF-ERDA measurement of  $TiO_2$  thin film with a thickness of about 10 nm was performed for demonstration. A 5-MeV Cu beam was used as an incident beam. Depth profiles of titanium and oxygen were successfully obtained with this measurement. Depth resolution for the oxygen at the surface was evaluated to be 1.6±0.2 nm.



Fig. 1 Schematic view of the TOF-ERDA measurement system.

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### PB-90: Multiple Ion Beam Irradiation Facility at the University of Michigan

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For many years the main activities in the Michigan Ion Beam Laboratory (MIBL) in the University of Michigan were ion implantation, ion beam analysis and ion irradiation using a 400 kV ion implanter and 1.7 MV tandem accelerator. At the beginning of 2014 we started the reconfiguration of MIBL to install a new 3 MV tandem accelerator and reconfigure the laboratory to create a multiple ion beam irradiation facility in addition to the activities already conducted in MIBL. At the end of 2014 MIBL regained the full capability before the shutdown. Then the tasks were focused to put together the beamlines and target chamber associated with the multiple ion beam irradiation setup.

This presentation will show the new configuration of accelerators in the laboratory, the ion sources attached to each accelerator, the design of each experimental beamline, the progresses made in the construction of the multiple ion beam irradiation beamlines and chamber and future activities in the next few months.

### PB-91: Development and operation of a new Liquid Helium-free Superconducting ECR Ion Source for highly-charged ion matter interaction facility (HIMIRF)

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A new liquid helium-free superconducting electron cyclotron resonance ion source has been developed in 2004 and initially evaluated at the National Fusion Research Institute (NFRI) in Korea. The source, named SMASHI (Superconducting Multi-Application Source of Highly-charged Ions), will be dedicated for highly-charged ions matter interaction research facility (HIMIRF) where various ion interactions with gases and/or plasma, solid surface, and living cells are investigated. SMASHI features a flexible high magnetic field (>2 T) [1], two-frequency heating, plasma diagnostic ports for extraction region, remotely positional three-electrode extraction system (puller-Einzel lens), and capability to generate a wide range of ion elements from gas to metal [2]. In this presentation, we describe main features of SMASHI and its operation results. Beam charge state spectra and their maximum intensities are provided for helium, oxygen, argon, and carbon ion beams.

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#### PB-92: Withdrawn

#### PB-93: A New 6 MV accelerator system for IBA and AMS at ANSTO

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The Centre for Accelerator Science (CAS) facility at ANSTO has been expanded with a new 6 MV tandem accelerator system supplied by the NEC. The major components of the new 6 MV system (Fig.1a) are:

1) three NEC ion sources: the 136 MC-SNICS sputter source (S1), the Direct Extraction Duoplasmatron (S3) and the RF source with a Rb charge exchange (S4) for generation of negative ion beams with a total injection energy up to 90 keV;

2) two injection magnets at the low energy section: a  $\pm 30$ deg inflection magnet with ME/q<sup>2</sup>=15 amu-MeV for light ions from the S3 and S4 sources, and 90deg bending magnet with ME/q<sup>2</sup>=20 amu-MeV and isotope bouncing system for ions from the S1 source;

3) the newly designed 6.0MV 18SDH-2 Pelletron accelerator;

4) a high energy section with two 90deg analysing magnets with ME/q<sup>2</sup>=175 amu-MeV (AMS) and ME/q<sup>2</sup>=150 amu-MeV (IBA);

5) two separated high energy (HE) branches with three (3) AMS (A2, A3 and A5) and three (3) IBA beamlines (B5, B6, and B7) serviced by two switching magnets with  $ME/Z^2=175$  amu-MeV at ±28deg (AMS) and  $ME/Z^2=150$  amu-MeV (IBA) at ±45deg bending angles;

6) the AccelNET computer control software for the 6 MV accelerator system.

The AMS beamlines, end-stations and DAQ software are custom built by NEC for rare isotope spectrometry while the three IBA beamlines/end-stations are ANSTO designed and built system with specific applications in mind (Fig.1b; bottom up):

a) the dual beam surface engineering end-station combining ion beams from the 6 MV system and the 50 kV low energy ion implanter (GNS-ANSTO-NEC, B5);

b) the dual chamber beamline for channelling and NRA (ANSTO-NEC, B6);

c) the ion microprobe with  $ME/q^2=45$  amu-MeV (OMLtd-ANSTO, B7).

An overview of the 6 MV system details and its performance during a testing and commissioning phase is given with emphasis on the IBA end-stations and their applications for a material modification and characterisation.





Fig.1a Floor layout of the 6.0 MV system beamlines

Fig.1b 3D schematics of the IBA

# PB-94: Implementation of ToF-E elastic recoil detection analysis for micro-electronics applications

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Ion beam analysis techniques are versatile methods for probing the composition and thickness of thin films. More specifically, elastic recoil detection analysis is recognized for its potential to probe the light elements. Modern implementations of elastic recoil detection analysis make use of a multi-dispersive detector telescope, for example a time of flight – energy (ToF-E) detector telescope. This allows one to separate the mass and the energy of the recoil and scatter ions, and thus to determine the simultaneous and quantitative elemental depth profile of light and heavy mass elements in both light and heavy matrices.

In this work, we report on the ToF-E ERD set-up developed at imec and Jyväskylä to cope with the increasing demand for ToF-E ERD analysis for micro-electronics applications. We describe the beam line and chamber design. The set-up is optimized for maximum sample capacity and detection efficiency. Special attention is given to guarantee the robustness and reliability of the system.

The time of flight is measured with timing gates based on electrostatic mirrors, and is acquired in coincidence with the energy signal. The energy is measured with either a solid state Si detector, or with a gas ionization chamber detector. We describe the characteristics of the ToF-E spectrometer in terms of timing resolution, detection efficiency, and mass resolution. We describe the procedures to periodically measure the time-calibration, the scattering angle, and the detection efficiency. The performance of the ToF-E ERD implementation is illustrated with various examples for the characterization of thin films in the micro-electronics industry.

### PB-95: Design of a new high current metal ion source using a waveguide-coupled and permanent magnet-embedded Lisitano antenna

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To produce high current metal ion beam, a new type of high density 2.45 GHz electron cyclotron resonance (ECR) plasma source was designed. The plasma is generated by a waveguide-coupled Lisitano antenna, where permanent magnets are embedded [1]. The Lisitano antenna is directly coupled by a tapered WR340 waveguide and is capable of high power operation up to 6 kW. Permanent magnets are inserted into the up and down side of the antenna as to configure a toroidal shape. With this arrangement, the plasma becomes more uniform and dense; electrons are heated by ECR and additionally confined by the magnetic field configuration, since the drift motion of the ECR-heated electrons form a close loop along the inner wall of the antenna. This plasma source has many advantages for a metal ion source:

(1) surrounding the plasma, all components are metallic material,

- (2) low operating pressure enhances ion beam directionality,
- (3) target is placed on the side excitation at the up and down side of the antenna,
- (4) high power capability,
- (5) free control of antenna size,
- (6) high density,
- (7) easy power coupling.

We present the detailed design of the plasma source and show the results of electromagnetic field simulation and plasma generation.

[1] Soo Ouk Jang, Hyun Jong You, Young-Woo Kim, Yong Ho Jung, In Uk Hwang, Jae Yang Park, and Heon Lee, Plasma Chem Plasma Process (2014) 34:229-237.

## PB-96: Setup and First Results of the New External Micro-beam of the 5SDH Tandem Accelerator at LAEC

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Recently, a new external micro-beam was commissioned at the 1.7 MV tandem accelerator of the Lebanese Atomic Energy Commission. Despite the use of a RF ion source, it was possible to steer a measurable beam and extract it into air. The setup is performed using an assembly of object slits, collimating slits and two quadrupole magnets from "Oxford Microbeams". A

description of setup and its performance will be shown, as well as some preliminary results of case studies, such as the localization of nanoparticles in superconducting materials, characterization of single aerosol particle or metals retention in micro-porous polymers.

## PB-97: Time-of-Flight Elastic Recoil Detection Analysis with a new Bragg type gas ionisation detector

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Time-of-Flight Elastic Recoil Detection Analysis (TOF-ERDA) is an ion beam analysis (IBA) technique where heavy energetic incident ion is used to recoil ions from the sample. In a single measurement, quantitative information on composition and depth distribution of all elements/isotopes present in the near surface region of the sample (up to 500 nm in depth) can be determined with nm depth resolution. Ion mass identification is achieved by simultaneously measuring energy and time of flight. Mass resolution of the TOF-ERDA spectrometer depends on the time resolution of the timing gates in the TOF telescope as well as on the energy resolution of the used energy detector. Since time resolution of TOF-ERDA spectrometers is ~200 ps, main contribution to the overall mass resolution comes from the energy resolution of the silicon detector (SD) which is most often used for energy detection (M/ $\Delta$ M  $\approx$  28). Particle SD detectors are commercially available and very easy to handle, but their energy resolution is significantly worse for heavier ions than for protons, which limits the spectrometer mass resolution in the high mass region.

The appearance of commercially available  $Si_3N_4$  windows with thicknesses as low as 50 nm stimulated researchers to use them as entrance windows for gas ionization detectors (GID) instead of thicker Kapton<sup>TM</sup> or Mylar<sup>TM</sup> foils. Consequently, GID detectors become an interesting option for detecting energy of heavy ions in TOF ERDA spectrometers [1-3]. Implementation of GID in the TOF-ERDA drastically improves spectrometer mass resolution from M/ $\Delta M$  = 28, that was achieved with SD, to M/ $\Delta M$  = 40 [1-3]. Also, big advantage of GID detectors is that they are radiation hard, keeping the same energy resolution parameters under high implantation doses [4]. Contrary to that, SD detectors are very sensitive to the radiation damage, and they energy resolution degrades rapidly with the implanted dose [4].

Thus, in order to upgrade detection capabilities of our existing TOF ERDA spectrometer, we decided to develop and implement a GID detector for detecting energy of recoiled and scattered ions. Detector is designed in the Bragg geometry with a 3x3 array of 50 nm thick Si<sub>3</sub>N<sub>4</sub> membranes as an entrance window. 40 mbar isobutene gas was sufficient to stop 30 MeV primary iodine beam as well as all recoils inside the detector volume. Spectrometer performances were measured and significant improvement in the energy and mass resolution, comparing to the spectrometer with standard silicon particle detector for energy measurements is presented.

J. Julin, M. Laitinen, T. Sajvaara, Nucl. Instr. Meth. Phys. Res., B 332 (2014) 271
 C. Kottler, M. Döbeli, F. Glaus, M. Suter, Nucl. Instr. Meth. Phys. Res., B 248 (2006) 155
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[4] A. M. Müller, A. Cassimi, M. Döbeli, M. Mallepell, I. Monnet, M. J. Simon, M. Suter, H.-A. Synal, Nucl. Instr. Meth. Phys. Res., B 269 (2011) 3037

## PB-98: Application of the new ERDA-TOF beam line at CMAM for diffusion studies in thin films

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A new elastic recoil detection analysis with time-of-flight (ERDA-TOF) system has been recently developed at the 5 MeV tandem accelerator at CMAM as a necessity to obtain accurate depth profiles for light elements. In this paper we present for the first time the technical description of the beam line, placed at the exit of the 10° port of the switching magnet of the accelerator. The telescope is attached to the main ERDA experimental chamber with a scattering angle of 40° and consists of two TOF stations separated 420 mm. These stations were constructed following the finish design [1], based on the capture and acceleration of electrons in isochronous trajectories from both sides of the carbon foil. The energy of the recoils is measured in a Si solid state detector placed at the end of the telescope. We determined the time resolution to be 83 ps.

This set-up was used for the analysis of different test samples, but also for systematic studies in metastable  $Zn_3N_2$  thin films grown by reactive pulsed magnetron sputtering [2]. The aging of this material due to the in-diffusion of  $O_2$  was confirmed by ERDA-TOF measurements, which were used to determine the oxidation rate too. This natural aging process was contrasted with the effect of thermal treatments, demonstrating the similarity between them, and proving that the in-diffusion process can be controlled.

[1] M. Laitinen et al., Nucl. Instrum. Meth. B 337, 55 (2014)
[2] C. García Núñez et al. Thin Solid Films 522, 208 (2012)

# PB-99: A tailored 200 parameter VME based data acquisition system for IBA at the Lund Ion Beam Analysis Facility – hardware and software

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A new VME multi parameter (200 channels) data acquisition and control system has been developed and implemented at the Lund Ion Beam Analysis Facility (LIBAF). The system is based on the VX-511 Single Board Computer SBC, acting as master with arbiter functionality and

replaces the 20 years old CAMAC based system [1]. The system consists of standard VME modules like ADC, QDC, TDC, SCALER, IO- cards, high voltage and waveform units. The modules have been specially selected to support all of the present detector systems in the laboratory simultaneously. Typically, the detector systems consist of silicon strip detectors [2], X-ray detectors and PM-tubes. The data flow out from the VME bus (event builder), to the final storage place on a 16 terabyte network attached storage disk (NAS-disk) is described. The acquisition process is remotely controlled over one of the SBCs ethernet channels. The Kmax system [3] is used to implement a user friendly graphical user interface, with functionality written in Java. The environment is used to control the acquisition process as well as for advance online and offline data analysis. In this work the system implementation, layout and performance are presented. The user interface and possibilities for advanced off-line analysis will be described and illustrated.

[1] Nuclear Instruments and Methods in Physics Research B 130 (1997) 123-126

- [2] Nuclear Instruments and Methods in Physics Research B 267 (2009) 2065–2068
- [3] www.sparrowcorp.com

### PB-100: Active volume 0.02 cm<sup>3</sup> ion sorption pump

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The development of analytical techniques that require vacuum environment, such as mass spectrometry, sensors, micromechanical systems, is limited by the lack of technical solutions, capable of producing, maintaining and controlling vacuum at total volumes of 0.1 cm<sup>3</sup> and smaller. Recent construction reached an active volume parameter of 0.05 cm<sup>3</sup> [1]. This parameter describes the volume between the cathodes and within the anode ring. Here we present the design of the ion sorption pump, with an active volume lower than 0.02 cm<sup>3</sup>. The pump consists of two flat 50 microns thick titanium plate cathodes and 3 mm diameter tantalum anode cylinder. The pump is equipped also with a resistance vacuum gauge. We show current/voltage characteristics of the pump and the vacuum gauge. We show also registered pumping process, starting from atmospheric pressure. Ion pump was switched on after reaching a vacuum better than 10<sup>-2</sup> mbar. Fore vacuum was obtained using typical rotary vane pump. Also special procedure is used, that intentionally changes the composition of the pumped gas. Vacuum better than 10<sup>-5</sup> mbar is obtained within a half an hour using up to 1000V DC supply voltage. The pump housing, 3 mm inner diameter pipe, is made of soda glass. Electrical inlets are made of FeNi42Cu alloy, of expansion matched to the glass. Inside the volume of the pump there is a vacuum gauge resistance filament, of which one inlet is common to the cathodes of the pump. The pump head is positioned in 0.6T magnetic field due to the use neodymium magnet. We also discuss the working conditions of miniature vacuum systems, with particular attention to the phenomenon of gas desorption from the inner surface of the vacuum chamber. It has been shown that the relation between the initial amount of gas in the volume of the microsystem and the amount of gas adsorbed on its inner surface is very important. We calculated the estimated gas desorption rate for a few selected geometries of
the vacuum system. It has been shown that the planar structures, having high surface-to-volume ratio are not optimal concerning optimum conditions for obtaining a high vacuum.

Acknowledgements: Authors thank The National Centre for Research and Development, Poland, for project no PBS1/A9/9/2012 founded in years 2012 – 2015.

[1] T. P. Grzebyk, A. Górecka-Drzazga, J. A. Dziuban, A. Zawada, and P. Konarski, "Micropump for generation and control of vacuum inside miniature devices," Journal of microelectromechanical systems, vol. 23, no. 1, pp. 50–55, 2014.

# PB-101: New low cost <sup>3</sup>He recovery unit at IPNAS cyclotron irradiation facility – <sup>3</sup>He beams production dedicated to C coatings activation and wear measurements

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Isotopic pure <sup>3</sup>He shortage and high cost justifies the use of regain systems whenever one considers using even small amounts of such gas injected in the source of small accelerators. In the case of a cyclotron and its dimensions, it becomes even mandatory. Systems supplied by manufacturers are expensive and difficult to adapt to existing gas handling systems. Therefore adapted workshop-made solutions are known to have been in use at some laboratories. Mechanical design, electronic control system and modes of operation all taking into account the demands in terms of duration and occurrence of 3He among other particle beam produced on IPNAS AVF cylotron, are presented. Specific features and advantages of both operation modes and a dual trap configuration are further discussed together with performances achieved and first results obtained in terms of regain efficiency. The overall objective of upgrading IPNAS cyclotron gas handling system is to provide <sup>3</sup>He beam for the irradiation, activation, and study by means of IBA and spectrometry techniques of C-based coatings in the framework of the HAVCOAT project initiated recently in PMR UNamur and Region Wallonne. Basic principles of Thin Layer Activation technique used for wear measurements in tribology are rapidly presented and selected candidate nuclear reactions to be used to produce gamma emitting activation products of interest for such C coated materials are presented. Preliminary <sup>3</sup>He beam test results and ion beam analysis of samples prior and after irradiation for beam damage investigation will be finally provided.

## PB-102: Charge Collection Imaging of Single Collision Cascades Using Nanobeam IBIC

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A detailed physics understanding of how collision cascades produced by heavy ions affect the electrical performance of semiconductors remains elusive. This gap of understanding is disturbing because such clusters are known to be produced through atomic recoil processes by neutrons from fusion or fission reactors and high energy accelerators. We describe a project that nanoscopically images and examines the influence of these collision cascades, including defect-clustering, on carrier recombination at defect complexes produced by single heavy ions in semiconductors.

In the experiments to be described, the Sandia NanoImplanter (20 nm beams) was used to implant a single heavy ion to produce a collision cascade in a lateral pn diode. The location of this cluster was spatially controlled so that it was formed in either the depleted region near the junction or in the field free region away from the junction. Subsequently, the NanoImplanter performed scanned ion beam induced charge (IBIC) measurements in a small area around the heavy ion's impact site using a much lighter ion that produced minimal damage. Charge collection efficiency determined from these IBIC maps will be discussed from the perspective of minority carrier drift and diffusion, coupled with defect induced recombination.

### PB-103: Micro PIXE and IL studies on jadeite with the new nuclear microprobe at IF-UNAM

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A new ion microprobe beam line has been installed at the Physics Institute (IF) 3MV Tandem Pelletron accelerator type 9SDH-2, of the National Autonomous University of Mexico (UNAM). Since this is the first system of its kind in Mexico, we will be able to employ it in the study of a wide variety of Mexican materials, both natural and manmade. For now, the main focus of its application is in geology, material sciences, and in the study of art and archaeological materials, of which Mexico has plenty. The basic components of the beam line where acquired from Oxford Microbeams Ltd. These are the object and collimation slits, scanning system, quadrupole triplet lenses and the OMDAQ acquisition software. The fabrication of a cylindrical analysis chamber and the design of the rest of the line has been carried out at IF. To this moment we can perform analysis with the following techniques: particle induced X-ray emission (PIXE) using a two detector arrangement, Rutherford backscattering spectrometry (RBS) and ionoluminescence (IL). IL is a very useful technique, as the phenomenon is sensitive to the chemical state or the local crystal structure of the ion, information usually unavailable in IBA techniques. IL is similar to other luminescence phenomenon, only differing in its excitation source and sampling depth. An advantage of IL over the other luminescent techniques comes

from the ability to do simultaneous analysis with other IBA techniques, mainly with the elemental detection limits offered by PIXE. In the ion microprobe, this synergy becomes greater by the addition of PIXE's mapping ability and the almost punctual analysis, increasing the trace element detection limits in heterogeneous samples. In this work, we present a description of the main design features of the beam line, the difficulties of mounting this type of system in a high seismic zone, and some performance tests. We also present some of the first results obtained by the microprobe system in a collection of highly heterogeneous jadeite and other minerals, in which the advantages offered by the combination of micro PIXE and IL are clearly displayed.

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# PB-104: Method of an in-situ measurement of the lithium distribution in Li-ion batteries using micro-IBA technology

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In these days lithium (Li)-ion batteries (LIBs) have come to be widely used but room for further improvement of their performance and safety is still left much. One of the key technologies is visualizing a distribution of lithium in LIB because the performance of LIB can be evaluated most directly by the movement of Li ions in material, which are charge carrier of a cell. While particle induced X-ray emission (PIXE) method is one of typical ion beam analyzing (IBA) techniques for samples containing multi-element, particle induced gamma-ray emission (PIGE) method is rather used for lithium analysis detecting gamma-rays emitted by <sup>7</sup>Li(p,p' $\gamma$ ) inelastic scattering. Characteristic X-rays from lithium have too low energy to detect by a typical X-ray detector. In addition, proton microbeam scanning technology combining with those IBA techniques (micro-IBA) provides 2-D mapping of lithium atoms in material. We have shown micrometer-scaled distributions of lithium and other elements in electrode samples of LIBs using the micro-IBA system developed at TIARA facility of JAEA [1]. In this study, we prepared LIB samples for in-situ measurements of lithium distributions in charging and discharging conditions, and obtained time-depending distributions of lithium in the samples using the micro-IBA system. This paper reports the preparations of the Li-ion cells, methods of the in-situ measurements, and also the results of them.

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### PB-105: PIXE-CT Analysis of Adsorbent for Extraction Chromatography

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In this study, a PIXE (particle induced X-ray emission)-CT (computed tomography) [1] analyzer was developed in JAEA [2] and used for analysis of three-dimensional distribution of trace elements in tiny adsorbent particles for extraction chromatography, which is being developed by JAEA for recovery of minor actinides (MA) from spent fast reactor fuel [3]. Those MA adsorbents were prepared by impregnation of extractant onto porous SiO<sub>2</sub>-P particles (ca. 50 μm in diameter) coated with styrene-divinylbenzene polymer as a binder. Currently, insufficient recovery rates of MA in extraction chromatography are thought to be causally related to the internal structure of the MA adsorbent particles. Therefore, PIXE-CT analysis using 3-MeV proton microbeam with a diameter of 1 µm was applied to measurement of three-dimensional distribution of adsorbed trace elements in the MA adsorbent particles. The measurement area was  $100 \times 100 \ \mu\text{m}^2$  corresponding to  $128 \times 128$  pixels of projection images. The MA adsorbent particle target was placed on an automatic rotation stage, which rotated with steps of 9° in vacuum. Forty projections were finally measured per one adsorbent particle, and threedimensional image reconstruction was carried out by the modified ML-EM (maximum likelihood expectation maximization) method [4]. As a result, the three-dimensional distribution of remaining neodymium simulating MA in the MA adsorbent particle was successfully obtained, and it was revealed that neodymium remained both the surface and the core of the particle even after elution process. This fact will be key to improvement of the recovery rate of MA in the extraction chromatography process.

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# PB-106: Particle induced X-ray emission computed tomography (PIXE-CT) for inertial confinement fusion (ICF) target

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Inertial confinement fusion (ICF) target quality control is vital to ensure that the energy deposition from the lasers results in uniform compression and minimization of Rayleigh–Taylor instabilities. That quality includes the perfect shape as a sphere capsule and the symmetry of density and element distribution. Scanning transmission ion microscopy computed tomography (STIM-CT) and particle induced X-ray emission computed tomography (PIXE-CT) can reconstruct quantitative three-dimensional structure with mass density information and element distribution respectively. Furthermore, combination of two methods makes results more accurate because X-ray absorption by sample itself in PIXE-CT can be corrected with STIM-CT density information. We present a case of ICF target characterized by combination of STIM-CT and PIXE-CT in order to demonstrate potential impact of these two methods in assessing target fabrication processes. 2 MeV proton beam accelerated by tandem accelerator and focused by triplet quadrupole lens was used and ICF target was rotated 180° with a step of 18°. Though the on-axis STIM detector was utilized, STIM and PIXE were detected separately because STIM detector was moved away while collecting PIXE signals using higher beam intensity. STIM data was processed by TomoRebuild software package. PIXE data was processed by self-coded package to reconstruct 3D image using filtered back projection (FBP) after adjusting image misalignment.

## PB-107: In air ion beam analysis with high spatial resolution proton microbeam

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One of the possible ways to maintain the micrometer spatial resolution while performing ion beam analysis in the air is to increase the energy of ions. In the case of protons it is expected that for energies above 8 MeV the beam spot size degradation due to straggling in the exit foil and in the 0.5 mm of air will be below 1 micrometer. In order to explore capabilities and limitations of this approach, we have tested a range of beam energies (2 - 8 MeV) using in-air STIM setup. Measurements of the spatial resolution dependence on proton energy have been compared with modelling of proton multiple scattering using different approaches including the simulation by SRIM.

Among the important applications of external microbeams that could be applied using higher energy proton beam are IBIC studies of semiconductor detectors. This is especially applicable for tests of large and complex detection systems that cannot be placed into the vacuum chamber. Such detectors are used at different large-scale facilities for nuclear and high-energy physics. In many cases IBIC probing beam has to pass different layers of detector assembly before reaching the sensitive detector volume. Protons that are transmitted through the whole detector volume could be also very useful for radiation hardness tests [1]. In addition to presentation of the recent in-air IBIC experiments, we will also discuss possibilities of applications of higher energy protons to other IBA techniques (e.g. PIXE), in the circumstances when high spatial resolution is required.

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# PB-108: Comparison of Ambient Pressure Mass Spectrometry Techniques for Molecular Speciation and Imaging at Surfaces

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There has been a rapid increase in the number of ambient pressure techniques over the past 10 years and their use in a range of disciplines from proteomics to forensics. MeV-SIMS (secondary ion mass spectrometry) is a relatively new technique which is potentially capable of producing molecular images at ambient pressure. A round robin comparison between laboratories is currently investigating measurement efficiencies in vacuum and will be reported at this conference. Previous work has favourably compared MeV-SIMS with more conventional keV-SIMS in vacuum. Here we compare some of the more widely used ambient pressure mass spectrometry techniques with the newly emerging ambient pressure MeV-SIMS. In particular we investigate the sensitivity and repeatability of the techniques for various classes of analyte and comment on the complementarity of the various ionisation methods. In this study we compare MeV-SIMS, MALDI (matrix assisted laser desorption ionisation), DESI (desorption electro spray ionisation) and paper spray. Advice will be given on establishing a framework for quantitative measurements.

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### PB-109: Pulsed TOF-SIMS in a Helium Ion Microscope

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Helium ion microscopes (HIM) have become powerful imaging devices within the last decade. Their enormous lateral resolution of below 0.3 nm and the highest field of depth make them a unique tool in surface imaging [1]. So far the possibilities to identify target materials (elements) are rather limited or need complex detection setups. In the present contribution we will present a new and relatively easy to implement time of flight method for Ion Beam Analysis (IBA) in the HIM. We utilize pulsed time of flight spectrometry to obtain elemental information from the sample. We will show initial results demonstrating the flexibility and applicability of the method to image samples with target mass contrast and analyze the target compositions. Pulsing the primary helium or neon ion beam and measuring the time of flight of secondary particles from the sample allows to obtain the energy of the backscattered particles or the mass of the sputtered target ions. This has been achieved by chopping the primary ion beam down to pulse widths of 5.5 ns by use of the built in beam blanker and a customized plug-on beam blanking electronics. The secondary particles are detected by means of a multi channel plate mounted on a flange of the HIM. The focus of the contribution will be on the TOF-SIMS concept. SIMS complements the RBS in a way that lateral resolved TOF-SIMS would enable fast qualitative elemental identification and contrast where the TOF-RBS serves quantitative sample compositions free from standards. In addition we point out mayor challenges, downsides and physical limitation of IBA in the HIM.

[1] G. Hlawacek, V. Veligura, R. van Gastel, and B. Poelsema, J. Vac. Sci. Technol. B 32(2), 2014

## PB-110: The IAEA Accelerator Knowledge Portal

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The IAEA Physics Section is pursuing efforts on utilizing accelerators to support fundamental and applied research, characterize and qualify materials of nuclear interest and provide training of a highly educated nuclear workforce.

The IAEA Physics Section launched the Accelerator Knowledge Portal (AKP) in 2014 for the benefit of accelerator scientists, accelerator users and service providers worldwide. The knowledge portal offers not only a database of MV particle accelerators all over the world, but it has several networking and community features in an attempt to bring together the Accelerator Community, as well as provide information to accelerator users and policy makers, too.

There website consists of two main parts.

- The core part is the **Accelerator Database**: a publicly accessible and searchable repository providing detailed information about the world's low- and medium energy particle accelerators with ~t 190 facilities up to date. The content of the database is contributed by the research facilities in the Member States.
- Accelerator Collaboration Platform and Networking: for the particle accelerator community. By providing up-to-date information on relevant conferences, workshops

and schools; relevant papers and books; links to relevant software packages and database tools, etc., users shall be motivated to regularly return to the site, to contribute content themselves and to build a community around the Accelerator Database.

The AKP is a community driven website. The main aim of this talk is to introduce the new website to the Accelerator community and demonstrate the above features which are opened for the public and some of them exclusively for registered users.

For more information, registration and update your accelerator facility please visit the AKP: <u>http://nucleus.iaea.org/sites/accelerators/</u>.

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### PB-111: Activation Analysis with Charged Particles: Theories, Practice and Potential

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Activation analysis with charged particles (CPA) was one of the first analytical applications of Particle Accelerators. It is very sensitive and has certain advantages as compared to neutron activation analysis (NA). These are that with slow NA (the most commonly used technique) lighter elements, and certain other elements, such as Al, Si, Ti, Cd, Tl, Pb, Bi, etc., cannot be conveniently or at all determined. Similarly with PIXE, another ion beam analytical technique it is difficult to measure lighter elements and its sensitivity is mostly limited to ppm rather than ppb range which can conveniently be achieved by CPA. However, there are some difficulties with CPA too. These are heating of the target through the passage of charged particle beams, and that the matrices of the sample and the "Standard" have to be identical or at least similar, which is not always convenient. However, with Chaudhri's [1] method, the most accurate of CPA-theories, the CPA is reduced to the simplicity of NA even when matrices of "Standard" and sample are widely different. By making use of CPA, the effect of French Atomic Tests Series of 1974 in the Pacific on the east coast of Australia was studied. Furthermore, non-destructive depth profile of F in teeth was measured to a record depth of 15 microns. The sensitivity curves for detecting any element/isotope with Z=20 to Z=90 in any matrix, through 1, 2 and 3 particles emission reactions, activated with protons, deuterons and alphas of up to 35 MeV energy have been estimated and presented in graphical form. From these curves the sensitivity of detecting any element/isotope in the aforementioned range could be directly estimated in any given matrix. Furthermore, these curves would also help in selecting the most suitable nuclear reaction for the measurement of a particular element or isotope in a given matrix, and would also provide an indication of the unwanted and interfering activities being produced simultaneously.

[1] M.A.Chaudhri et al., J. Radioanal. Chem, 37(1977) 243.

# PB-112: Ion-Beam and ICP-MS-LA Techniques for the Sudy of Trace Elements in Biomedical Hard Tissues: Bones,Teeth and Kidney / Bladder stones

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As most of us would be aware that to determine trace element concentrations in hard tissues (bones, teeth and kidney/bladder stones) with normal chemical techniques, in not easy and contamination free We have, therefore, developed various nuclear and atomic activation techniques and applied these to determine the elemental composition of calcified tissues (teeth and bones). Fluorine was determined by prompt- gamma- activation- analysis through the reaction <sup>19</sup>F(p, $\alpha$ -gamma)<sup>16</sup>O. Carbon was measured by activation analysis with He-3 ions, and the technique of Proton-Induced X-ray Emission (PIXE) was applied to simultaneously determine Ca, P, and trace elements in well-documented teeth. Dental hard tissues: enamel, dentine, cementum, and their junctions, as well as different parts of the same tissue, were examined separately.

Furthermore, using a Proton Microprobe, we measured the surface distribution of F and other elements on and around carious lesions on the enamel. The depth profiles of F, and other elements, were also measured right up to the amelodentin junction. We have used the same technique to measure the F-contents of experimental animals- on fluoridated water supply and the controls. Some results on qualitative trace elements analysis on the distributions of various elements in kidney and bladder stones, using the powerful technique of Laser-Ablation Inductively-Coupled Plasma Mass Spectrometry (La-ICP-MS), are also presented.

The significance of these unique methods in the study of biomedical hard tissues would be discussed.

### PB-113: Early prediction of rice tolerance to salinity based on intake of essential elements

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Salt tolerance is one of major abiotic stresses threatening crop plants e.g. rice (Oryza sativa) production subsequently food security. The trait is complex and affected by numerous genetic and non-genetic factors. To date the most efficient selection tools are based on phenotyping in

hydroponics or in the field.

An investigation on the correlation between tolerance to salinity in sixty two rice genotypes and the intake of essential elements by the plants was performed by Particle Induced X-ray Emission (PIXE) and X-ray Fluorescence (XRF) analysis.

The multivariate statistical interpretation of the results proved that there are differences in the intake ratio of essential elements in the shoot (Na, Mg, P, S, Cl, K, Ca, Mn, Fe, Cu, Zn, Br, Rb ad Sr) across tolerant, medium tolerant and susceptible genotypes. A genotype differentiation was observed; indicating that the tolerant genotype has a larger intake of nutrients. A classification criteria based on Canonical Discriminant Analysis was proposed and verified. Therefore essential elements in shoot can effectively be used to screen for salt tolerance crop plant, particularly in rice.

The proposed methodology allows enhancing the efficiency of generating new lines by mutation breeding.

## PB-114: Analytical performance of XRF and PIXE for the analysis of rice shoots

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An investigation aiming at revealing differences in the intake of essential elements by seedlings of rice genotypes with different tolerance to salinity stress was conducted at IAEA Plant Breeding and Genetics Laboratory. Aim of the study was to assess the analytical performance of Particle Induced X-ray Emission (PIXE) and X-ray Fluorescence (XRF) techniques for the analysis of concentration of essential elements (Na, Mg, P, S, Cl, K, Ca, Mn, Fe, Cu, Zn, Br, Rb ad Sr) in plant samples. The quantitative analysis was made upon calibrations made using Certified Reference Materials (CRMs), and the performance of the two methods was assessed by analysing five CRMs covering the expected concentration ranges. The results are presented for comparison.

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