## 16th International Conference on Nuclear Microprobe Technology and Applications

# **ABSTRACT BOOK**



University of Surrey, Guildford, UK 8-13 July 2018 The conference is organised in cooperation with the IAEA



The committees gratefully acknowledge the conference sponsors











TOKYO, JAPAN

Surrey Ion Beam Centre





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#### SECURITY

Delegates are asked not to leave their baggage unattended and to wear conference badges at all times. The University accepts no liability for loss or damage to property or vehicles and their contents, nor for injury to visitors to the campus except where required by law.

#### **CONFERENCE DESK**

The conference desk is based in the foyer area outside Austin Pearce rooms 3/4 and will be open daily from 0900 to 1700 hours except on the Wednesday afternoon when it will close at 1300 hours.

#### IN AN EMERGENCY OR IF MEDICAL ASSISTANCE IS REQUIRED

If the building alarms sound during the day members of the Surrey team will be available to escort you to a place of safety. If you feel unwell or require medical assistance during the day please make your way either to the conference desk or approach a member of the Surrey team. Members of the Surrey team are identifiable by pink conference badges.

If you need medical assistance when the conference has closed for the day please make contact with personnel in the Security office who are all first aid trained. In an emergency situation <u>and ONLY in</u> <u>an emergency situation</u>, i.e. the fire service, an ambulance or the police need to be called to the campus, should the Security Office be contacted on the University's dedicated emergency telephone number which is +44 1483 683333 from an external telephone or by dialling 3333 from any internal telephone.

#### SOCIAL EVENTS

#### Registration and Welcome Reception

Registration will commence at 1630 hours and a welcome reception will be held from 1800 hours onwards both on Sunday 8 July in Wates House.

#### Conference Outing

An outing to Osborne House on the Isle of Wight has been arranged for the afternoon of 11 July. Coaches will depart from the field opposite the PATS building (indicated on the campus map in your delegate pack). You will be escorted from the lecture theatre by members of the Surrey team to the pick-up point however if you are not attending the morning session please ensure you are at the pick-up point by 1225 hours at the very latest as the coaches will depart promptly at 1230 hours. A packed lunch will be available for delegates who are participating in the outing and this is to be collected when you alight the coach prior to departure.

#### **Conference Banquet**

The banquet has been arranged for the evening of 12 July at Brooklands Museum. Coaches will again depart from the field opposite the PATS building - please ensure you are at the pick-up point by 1800 hours at the very latest.

#### **MEALS AND REFRESHMENTS**

Breakfast and evening meals for delegates staying on-site will be served in Hillside Restaurant (those eligible are identified by their yellow name badges) with the costs included in their accommodation fees. For delegates staying off-site they may, if they wish, purchase an evening meal in the

restaurant for approximately £15.00. Tea, coffee and lunches will be available in Austin Pearce rooms 3/4 during the day at the times stated in the programme.

#### PRESENTATIONS

Presentations are to be uploaded prior to the relevant session commencing. Members of the Surrey team will be on hand to assist with this. If any further specialised equipment is required, please make your needs known to the conference desk at your earliest convenience.

#### **POSTER SESSION**

The two poster sessions will be held on Monday afternoon at 1630 hours and on Thursday afternoon at 1605 hours. Posters are to be mounted on Monday and removed on Friday. Velcro dots will be provided for poster mounting - you must not use pins on the boards. The posters boards will be numbered as listed in this booklet - please ensure that you place your poster on the appropriately numbered board.

#### SUBMISSION OF MANUSCRIPTS

Full papers will be reviewed for inclusion in the conference proceedings which will be published as a special edition of the Elsevier Journal Nuclear and Instruments in Methods in Physics Research Section B: Beam Interactions with Materials and Atoms.

The website for paper submission will have a hard deadline of 30 July. The normal refereeing standards of Nuclear Instruments and Methods will apply and only papers that are presented at the conference can be accepted for the proceedings. It is intended that the refereeing process will be completed by the end of November.

Authors can submit their papers on the Elsevier EES system by selecting: "VSI: ICNMTA 2018" as the article type. The EES system can be found at (https://ees.elsevier.com/nimb\_proceedings).

## PROGRAMME

#### Sunday 8 July 2018

- 16:30 Registration in Wates House
- 18:00 Registration and Welcome Reception in Wates House

#### Monday 9 July 2018

08:30 Registration (continued) in Austin Pearce Rooms 3/4 (Lecture Theatre Block) 08:45 Welcome *R P Webb, Conference Chair, University of Surrey* Session 1 **Biomedical Applications Chair: Teresa Pinheiro** 09:00 101 Targeted Substructure of Cells and Preclinical Studies for Proton Minibeam Radiation Therapy at the Ion Microprobe SNAKE G Dollinger, J Reindl, K Ilicic, S Girst, C Greubel, S Rudigkeit, M Sammer, B Schwarz, C Siebenwirth, D W M Walsh, A A Friedl, J J Wilkens, T E Schmid 09:40 001 Ultra-Thin Diamond Detectors for Cell Micro-Irradiation Experiments P Barberet, M Pomorski, G Muggiolu, E Torfeh, G Claverie, C Huss, S Saada, G Devès, M Simon, H Seznec 10:05 OO2 The Radiobiology Beam Line Facility at the Centre for Ion Beam Applications, National University of Singapore TYe, THQi, MZhaohong, C-BChen, VLYMan, SKChee, TOsipowicz, FWatt, A A Bettiol 10:30 Coffee **Biomedical Applications** Session 1 (continued) **Chair: Philippe Barberet** 10:55 102 Investigating the Enhanced Radiobiological Effectiveness of High-LET Particles Utilizing Ion Microprobe SNAKE and STED Microscopy J Reindl, K Ilicic, S Girst, C Greubel, M Sammer, B Schwarz, C Siebenwirth, D W M Walsh, A A Friedl, T E Schmid, G Dollinger 11:35 OO3 A Novel 3D Tissue Engineering Tool for Beam Therapy Screening of Pancreatic Cancer S Totti, R P Webb, A Nisbet, G Schettino, E Velliou 12:00 O04 Correlative Structure-Fluorescence Imaging of Single Cells with Fast Helium-Ion Nanoprobes Z Mi, C-B Chen, F Watt, A A Bettiol 12:25 Lunch and Posters Session 1 **Biomedical Applications** (continued) **Chair: Günther Dollinger** 14:00 O05 Diamond-Based Multi-Functional Biosensors Realized by Means of MeV Ion Beam Lithography F Picollo, A Battiato, V Bonino, E Carbone, V Carabelli, A Marcantoni, L Mino, A Pasquarelli, G Tomagra, M Truccato, E Vittone, P Olivero

14:25	O06	PIXE Imaging of Hyperaccumulator Plants Using the MAIA Detector Array J S Laird, C G Ryan, R Kirkham, A van der Ent, D N Jamieson
14:50	007	Applicability of Multi-Elemental Distribution Analysis in Plants P Pongrac, K Vogel-Mikuš, M Regvar, I Kreft, M Kelemen, P Vavpetič, P Pelicon
15:15	O08	Can we Use the Invasive Apple Snail <i>Pomacea Maculata</i> for Biomonitoring of Lead Contamination in Freshwater Wetlands? <i>H J Whitlow, S Banerjee, H Udegou, N Deoli, A de Vera, P L Klerks</i>
15:40	O09	On the Number of Metallic Atoms in a Cage Protein Molecule by Micro-PIXE P Pelicon, P Vavpetič, M Kelemen, B Jenčič, K Majsterkiewicz, A Biela, J Heddle
16:05	010	Modelling the Uptake of Suspended Materials and Salts in Nearshore Waters by Plastics Using IBA Depth Profiling Analytical Tools N Tuccori, T Pinheiro, L C Alves, T Peña, M J Botelho, J Raimundo, C Vale
16:30		Poster Session 1 (posters with red board numbers)

#### Tuesday 10 July 2018

#### Session 2 Facilities and Techniques Chair: Hiroyuki Nishikawa

- 09:00 IO3 Considerations About Projectile and Target X-Rays Induced During Heavy Ion Bombardment F Fernandes, L Amaral, J F Dias
- 09:40 O11 Performance of the 3.5 MV Singletron HVE Accelerator Developed for LUNA-MV A Sen, G Domínguez-Cañizares, N C Podaru, D J W Mous, M Junker, G Imbriani, V Rigato
- 10:05 O12 An Optimized 19F  $(p, \alpha \gamma)^{16}$ O Elemental Mapping Facility S K Vajandar, M Q Ren, D Zheng, C S Hsu, T Osipowicz
- 10:30 O13 In-Situ Irradiation and IBIC Probing Using Pulsed and Dual Ion Microbeams M Jakšić, A Crnjac, S Fazinić, W Kada, N Skukan, I Sudić
- 10:55 Coffee

#### Session 3 Quantum Devices Chair: Jan Meijer

- 11:20 IO4 An Introduction to Quantum Technology Devices with Donor Impurities in Silicon and the Role of Precision Ion Beams B N Murdin
- 11:45 O14 Nitrogen Implantation with a Scanning Electron Microscope S Becker, N Raatz, S Jankuhn, R John, J Meijer
- 12:10 O15 High-Sensitivity Deterministic Implantation Devices by Ion Beam Induced Charge: Towards 10 Quantum Bits in 5 Years S G Robson, A M Jakob, V Mourik, B C Johnson, V Schmitt, F Hudson, C Lew, J C McCallum, A Morello, D N Jamieson
- 12:35 Lunch and Posters

#### Session 3 Quantum Devices

#### (continued) Chair: David Jamieson

- 14:10 IO5 A Concept for Deterministic Ion Implantation by Image Charge Detection *P Räcke, D Spemann, J W Gerlach, B Rauschenbach, J Meijer*
- 14:50 O16 SIMPLE Single Ion Multispecies Position at Low Energy A Single Ion Implanter for Quantum Technologies N Cassidy, D Cox, R P Webb, B N Murdin, L D Antwis, P Blenkinsopp, I Brown, T Adams, R J Curry

- 15:15 O17 Statistics of Deterministic Single Ion Implantation *R P Webb, D Cox, L D Antwis, L dos Santos Rosa, B N Murdin, P Blenkinsopp, I Brown, T Adams, N Cassidy, R J Curry*
- 15:40 I06 Fabrication of Single Atom Devices by Direct Write Nanofabrication *E S Bielejec*
- 16:30 Conference Photograph, Laboratory Visit and Opening of SIMPLE

#### Wednesday 11 July 2018

## Session 4 Detectors Chair: Ettore Vittone

- 09:00 IO7 Extreme Radiation Hardness and Signal Recovery in Thin Diamond Detectors N Skukan, I Sudić, M Pomorski, W Kada, M Jakšić
- 09:40 O18 Comparative Study of the Changes Produced by the Implantation of Focused High Energy Ions in Diamond *M D Ynsa, A de Andrés, R J Jiménez-Riobóo, N Gordillo, M A Ramosa*
- 10:05 O19 IBIC Microscopy The Most Powerful Tool for Testing Micron-Sized Sensitive Volumes in Strip Detectors and Microdosimeters Used in Synchrotron Microbeam Radiation Therapy and Hadron Therapy *Z Pastuovic, T L Tran, J Davis, D Prokopovich, M Lerch, M Petasecca, R Siegele, J R Paino, A Dipuglia, B James, A Rosenfeld, D Cohen*
- 10:30 Coffee

#### Session 4 Detectors (continued) Chair: Maria Dolores Ynsa

- 10:55 O20 Determination of Radiation Hardness of Silicon Diodes *E Vittone, J Garcia Lopez, V Grilj, M Jakšić, M C Jimenez Ramos, A Lohstroh, Z Pastuovic, S Rath, R Siegele, S Skukan, G Vizkelethy, A Simon*
- 11:20 O21 Quantitative Determination of Fluorine Using a Double Sided Silicon Strip Detector (DSSSD) N De La Rosa, E J C Nilsson, M Elfman, P Kristiansson, J Pallon
- 11:45 O22 Position-Selective Introduction of Electrically Excitable Color Centers in SiC PN Junction Diode by Proton Beam Writing Y Yamazaki, Y Chiba, T Makino, S I Sato, N Yamada, T Satoh, Y Hijikata, K Kojima, S Y Lee, T Ohshima
- 12:25 Conference Outing to Isle of Wight

#### Thursday 12 July 2018

#### Session 5 Forensics and Cultural Heritage Chair: Milko Jakšić

- 09:00 IO8 From Potential to End-Users: Accelerator-Based Analytical Techniques for Forensic Science A Simon, N P Barradas
- 09:40 O23 MACHINA: Movable Accelerator for Cultural Heritage In-Situ Non-Destructive Analysis F Taccetti, L Giuntini, L Castelli, M Chiari, M Fedi, C Czelusniak, L Palla, P A Mandò, G Calzolai, S Mathot, G Anelli, M Vretenar, A Lombardi, E Montesinos
- 10:05 O24 DAPNe-IBA: A New Tool for Forensic and Biomedical Science? *M J Bailey, J de Jesus, H Lewis, C Costa, J Bunch, R P Webb*
- 10:30 Coffee

#### Session 6 Nano Structures and Devices Chair: Gyorgy Vizkelethy

- 10:55 O25 Refractive Index Change and Thermo-Optic Effect in Polydimethylsiloxane Nanocomposites with Oxide Nanoparticles Induced by Proton Beam Writing *Y Kaneko, H Hayashi, Y Ishii, W Kada, H Nishikawa*
- 11:20 O26 Micro Heavy Ion Beam Irradiation as the Tool to Create the On/Off Function of Glass *T Wang, L Chen, P Lv, B Zhang, H Peng, F Munnik, R Heller*
- 11:45 O27 Electronic Circuit Formation on Flexible Polymer Surface Processed by 1 MV Accelerated Hydrogen Molecular Ions *H Hayashi, W Furukawa, H Nishikawa*
- 12:10 O28 The Concept of Super-Resolution Ion Beam Imaging with High Energy Single Ions *G Du, J Guo, G Mao, W Liu, R Wu, Y Li, J Zhao, X Y Li, A Ponomarov*
- 12:35 Lunch and Posters

## Session 6Nano Structures and Devices(continued)Chair: Guanghua Du

- 14:00 O29 Nuclear Microprobe Characterisation of Defects in Zinc Recycled Cemented Carbides *C S Freemantle, M Topic, C A Pineda-Vargas*
- 14:25 O30 Microstructural and Elemental Characterisation of Cemented Tungsten Carbide Tool Grade Scrap Metal J Kurasha, N Sacks, M Topic, C Mtshali
- 14:50 O31 Optical Properties of an Ensemble of G-Centers in Silicon C Beaufils, W Redjem, E Rousseau, V Jacques, A Yu Kuznetsov, C Raynaud, C Voisin, A Benali, T Herzig, S Pezzagna, J Meijer, M Abbarchi, G Cassabois

- 15:15 O32 Optical Effects of Focused Proton Irradiation of Diamond A A Bettiol, S P Tugara, H Jin, E J Teo
- 15:40 O33 Measurement of D2 Amount in ICF Target by Proton-Deuterium Scattering Method *H Zhang, W Zhang, X Zhang, N Guo, Y Zhang, D Gao, Q Wang, X Ma, H Shen*
- 16:05 Poster Session 2 (posters with blue board numbers)
- 18:00 Conference Dinner at Brooklands Museum

#### Friday 13 July 2018

#### Session 7 MeV SIMS Chair: Hao Shen

- 09:00 I09 Adsorption of Liquids Measured with MeV-SIMS in Ambient *J Matsuo, T Seki, T Aoki*
- 09:40 O34 Chemical Imaging of the Healthy and Diabetic Mouse Liver Using MeV TOF-SIMS Z Siketić, I Bogdanović Radović, M Barac, M Popović Hadžija, M Hadžija
- 10:05 O35 Mass Spectrometry Ion Beam Analysis: A New Tool for Molecular and Elemental Speciation? *C Costa, J Bunch, R Goodwin, R P Webb, V Palitsin, J de Jesus, M J Bailey*
- 10:30 Coffee

#### Session 8 Beam Optics Chair: Istvan Rajta

- 10:55 O36 Characteristics of Focus Conditions for a Magnetic Quadrupole Triplet Lens W Sudprasert, R Meesat, H J Whitlow
- 11:20 O37 Development of a Separated Magnetic Quadrupole Doublet Focusing System at the University of North Texas *B Rout, T A Byers, J E Manuel, G A Glass*
- 11:45 O38 Effects of Stray Magnetic Field on the Performance of Focused Ion Microbeam *Y Dou, J Liu, D Pan, Z Sun, L Li*
- 12:10 O39 The Beam Optics of Spaced Triplet Configurations of Magnetic Quadrupoles for High Energy Ion Microbeam Applications *G W Grime, V Palitsin*
- 12:35 Close of Conference and Lunch

## POSTERS

- P01 Heritage Science Applications at the Nuclear Microprobe of MTA ATOMKI Studies on Early Medieval Belt Sets
   Á Csepregi, B Tobias, Z Kertész, A Angyal, E Papp, Z Szoboszlai, Z Török, Z Szikszai
- P02 The New Sumy Nuclear Microprobe with High Resolution Probe-Forming System A G Ponomarev, V A Rebrov, S V Kolinko, A S Lapin, V F Salivon, A A Ponomarov
- P03 Optimization of Probe-Forming System of High Energy Heavy Ion Microbeam in Lanzhou for Low Current Mode

A Ponomarov, G Du, J Guo, L Sheng, A G Ponomarev, W Liu, R Wu, Y Li

- P04 Stigmatic Imaging of Secondary Ions in MeV-SIMS by a Linear Time-of-Flight Mass Spectrometer and a TimePix Detector
   B Jenčič, L Šepec, P Vavpetič, M Kelemen, Z Rupnik, M Vencelj, K Vogel-Mikuš, N Ogrinc Potočnik, S Ellis, R M A Heeren, P Pelicon
- P05 An Integrated MIDAS and LabVIEW Based Data Acquisition, Scanning and Stage Control System for the Nuclear Microprobe at the Materials Research Department, iTHEMBA Laboratories *C Pieters*
- P06 Development of Nano Apertures for Ion Beam Collimation C Scheuner, N Raatz, P Räcke, S Jankuhn, S Pezzagna, C Trautmann, J Meijer
- P07 Analysis of SPICE Microbeam Profile Using Fluorescent Nuclear Track Detector (FNTD) D Ohsawa, Y Furusawa, A Kobayashi, M Oikawa, T Konishi
- P08 Trace Element Composition of In-Situ Isotopically Enriched Silicon S G Robson, D Holmes, J S Laird, A M Jakob, B C Johnson, J C McCallum, H Fiedler, P Gupta, A Markwitz, C G Ryan, D N Jamieson
- P09 Micropatterning of Polytetrafluoroethylene (PTFE) by Proton Beam Writing D de Vila Bauer, C Telles de Souza, L Amaral, J F Dias
- P10 Data Acquisition and Control System for an Evolving Nuclear Microprobe D Cosic, M Bogovac, M Jakšić
- P11 Rutherford Backscattering Spectroscopy/Channelling for Damage and Strain Measurements in Semiconductors E Schneider, J G England, I P Marko, S J Sweeney, M Sharpe, D Cox
- P12 Capabilities of an Independent Single Ion Hit Detection Systems Based on Emission of Secondary Particles During the Impact *G Provatas, M Brajković, M Jakšić*
- P13 Investigation of Effects of NS, High Intensity Ion Pulses on Defect Creation Using a Nuclear Microprobe and Deep Level Transient Spectroscopy (DLTS)
  G Vizkelethy, E S Bielejec, B A Aguirre, P A Seidl, T Schenkel

- P14 Comparative Study of the Effect of Swift Heavy Ion Irradiation at Room Temperature and 500°C on the Migration of Silver Implanted into Polycrystalline SiC
  H A A Abdelbagi, J B Malherbe, V A Skuratov, E G Njoroge, S V Motloung, M Mlambo, T T Hlatshwayo
- P15 Assembling Gold Nanoparticles by Dielectrophoresis with Pit Arrays on PMMA Fabricated by Proton Beam Writing *T Shibuya, S Uchida, Y Ishii, H Nishikawa*
- P16 First Resolution Test Results of the ATOMKI Nuclear Microprobe I Rajta, G U L Nagy, I Vajda, S Z Szilasi, G W Grime, F Watt
- P17 A New Q-Pole Configuration at the LIBAF Microbeam J Pallon, N De La Rosa, M Elfman, P Kristiansson, E J C Nilsson
- P18 Ion Implant Induced Disorders and Its Effect on the Structural and Optical Properties of CdS Nanowires J Kaur, R C Singh, H Kaur
- P19 Automated Alignment and Focusing System for Nuclear Microprobes S Qureshi, W Jiacheng, J A van Kan
- P20 Positioning Single Ions by Fluorescence Method to Achieve Higher Analysis Precision at LIHIM J Guo, G Mao, G Du
- P21 Measurement of Various Ions in Human Teeth Using in-Air Microbeam PIXE/PIGE K Naito, K Yagi, H Yamamoto, Y Iwami, Y Matsuda, K Okuyama, N Yamada, M Koka, T Sato, M Hayashi
- P22 Fluorine Distribution from Fluoride-Releasing Luting Materials to Human Dentin K Okuyama, Y Matsuda, H Yamamoto, Y Tamaki, T Saito, M Hayashi, Y Yoshida, H Sano, T Sato, M Koka
- P23 Spatial Characterization of Schottky Barriers Formed in β-Ga<sub>2</sub>O<sub>3</sub> Using a Proton Microprobe M Peres, L C Alves, E Alves, T S Monteiro, S Cardoso, A Kuramata, E G Víllora, K Shimamura, K Lorenz
- P24 Development of MeV TOF-SIMS Capillary Microprobe *M Barac, Z Siketić, I Bogdanović Radović*
- P25 Investigation of the Lateral Homogeneity of Plasma-Drive Deuterium Permeation Through Tungsten
   M Kelemen, S Markelj, P Vavpetič, S Kapser, M Balden, S Elgeti, P Pelicon
- P26 Micro-PIXE in Frozen Hydrated State
  P Vavpetič, M Kelemen, K Vogel-Mikuš, B Jenčič, P Pongrac, E Punzón-Quijorna, M Regvar,
  P Pelicon
- P27 A Simple Model of MeV-SIMS Profiling *R P Webb, V Palitsin, L Matjačić, A Abdul-Karim*

- P28 Improvements in Rez Ion Microprobe Parameters: Numerical Calculation O V Romanenko, A G Ponomarev, A Macková, V Havránek, A Ponomarov
- P29 Micro-PIXE Mapping of Several Key Mineral Nutrients Localised Within the Brain Tissue of Pearl Millet (*Pennisetum Glaucum*) Grains *R Ndimba, J Kruger, J Taylor, C Mtshali, C A Pineda-Vargas*
- P30 Tertiary Electrons in Single-Event Time-of-Flight Rutherford Backscattering Spectrometry S Abo, A Seidl, F Wakaya, M Takai
- P31 Comparative Study on Phase Proportions in Dental Amalgams Using Micro-PIXE and XRD Techniques W Sudprasert, R Meesat, K Kolanan, H Udeogu, A B de Vera, N Deoli, H J Whitlow
- P32 Antibacterial Effect of Fluoride-Containing ZnO/CuO Nanocomposite Y Matsuda, K Okuyama, H Yamamoto, M Fujita, S Abe, T Sato, N Yamada, M Koka, S K Sidhu S Takashi
- P33 Combined μ-PIXE/μ-EBS Analysis of Surface Stains in Gold Coins/Discs *J Cruz, V Corregidor, L C Alves*
- P34 Reconstruction of H Distribution in ICF Target Shell by PPS-T H Zhang, N Guo, T Yu, D Gao, Q Wang, X Ma, H Shen
- P35 Improvement of Analytical Measurements of Non-Trivial Biological Samples with Nuclear Microscopy T Pinheiro, P Teixeira, J Bento, R Godinho, F Marques, L C Alves, N Lourenço, H Pinheiro
- P36 Design of a Collimation System for the 300 MeV Proton Microbeam System in Harbin *Y Dou, J Liu, L Li*
- P37 The Influence of Thermal Annealing on Defects Induced in Xe Implanted n-Type 4H-Silicon Carbide E Omotoso, W E Meyer, E Igumbor, H T Danga, B A Taleatu, F D Auret
- P38 High Throughput Large Area Mapping of Geological Samples Using a MAIA Detector Array on the Nuclear Microprobe
  J S Laird, C G Ryan, R Kirkham, S Hu, D P Siddons, P A Dunn, A Kuczewski, D Parry, F Rudzik, R Szymanski, D N Jamieson
- P39 Multi-Energy IBA to Study Solar Energy Conversion Materials V Corregidor, A Barreiros, L C Alves
- P40 Evolution of Impurities in Metals Used in Cultural Heritage Objects V Corregidor, J Cruz, L C Alves
- P41 Depth Profiling of Organic Electronic Systems with 3D OrbiSIMS L Matjačić, R Havelund, I S Gilmore
- P42 Optimisation of Ambient Pressure MeV SIMS L Matjačić, V Palitsin, J Demarche, E Moura Stori Rosa, L dos Santos Rosa, R P Webb

P43 Coupling of AP MeV SIMS and HIPIXE for Simultaneous Molecular and Elemental Mapping in Air

L Matjačić, V Palitsin, G W Grime, A Abdul-Karim, R P Webb

- P44 The New Spaced Triplet Beamline at the Surrey Ion Beam Centre *V Palitsin, G W Grime*
- P45 Analysis of Forensic Traces Using Direct Analyte-Probed Nanoextraction Mass Spectrometry (DAPNe-MS) and Ion Beam Analysis (IBA)
  H-M Lewis, R P Webb, G Verbeck, J Bunch, J de Jesus, C D Costa, V Palitsin, P Pelicon, M J Bailey
- P46 Preliminary Study of Glass Production Remains from a Spanish Glass Furnace Dated to the 16th Century
  *I Coutinho, L C Alves, I C Campaña, T Medici*
- P47 The Response of Genetically Different Glioblastoma Cell Lines to Vertical and Horizontal Proton Irradiation N Mayhead, L Meira, R P Webb

## **SESSION 1**

## **BIOMEDICAL APPLICATIONS**

Chairs: Teresa Pinheiro Philippe Barberet Günther Dollinger

## 101 TARGETED SUBSTRUCTURE OF CELLS AND PRECLINICAL STUDIES FOR PROTON MINIBEAM RADIATION THERAPY AT THE ION MICROPROBE SNAKE

Günther Dollinger<sup>1</sup>, Judith Reindl<sup>1</sup>, Katarina Ilicic<sup>2</sup>, Stefanie Girst<sup>1</sup>, Christoph Greubel<sup>1</sup>, Sarah Rudigkeit<sup>1</sup>, Matthias Sammer<sup>1</sup>, Benjamin Schwarz<sup>1</sup>, Christian Siebenwirth<sup>3</sup>, Dietrich W M Walsh<sup>1</sup>, Anna A Friedl<sup>4</sup>, Jan J Wilkens<sup>2</sup> and Thomas E Schmid<sup>2,5</sup>

<sup>1</sup>Universität der Bundeswehr München, Germany <sup>2</sup>Klinikum Rechts der Isar, TU Munich, Germany <sup>3</sup>RARAF Laboratory, Columbia University, New York, USA <sup>4</sup>Klinikum der Universität München, LMU, Germany <sup>5</sup>Helmholtz Zentrum München, Germany

SNAKE (<u>Superconducting Nanoscope for Applied nuclear (Kern-) physics Experiments</u>) is able to focus 10 MeV - 25 MeV protons and heavy ions (e.g. 55 MeV carbon ions) to sub-micrometer spot sizes. The high energies and the various kinds of ions are ideally suited to irradiate cells, tissue sheets and even live animals to address topics in radiation biology and medicine. The talk will summarize some of the achievements and new ideas for the use of SNAKE in radiation biology and medicine.

The live cell irradiation setup at SNAKE, equipped with an epifluorescence microscope, allows for irradiation of stained substructures in living cells with a defined number of ions. Furthermore it is possible to track and analyze the irradiation response of single cells. As a first example, nucleoli, the RNA synthesizing areas in the cell nuclei, were targeted by single carbon ions. The DNA density inside the nucleoli is only about 5% of that in the other parts of the cell nucleus. Although, based on density calculations, less DNA double strand breaks (DSB) are expected in the nucleoli a large number of irradiated nucleoli show yH2AX foci which are commonly used as DSB marker. We show that transcription has stopped locally in the irradiated nucleoli while it is not affected in other parts of the cell nucleus. Another example is the targeting of mitochondria, the power plants of the cell located in the cytoplasm. All mitochondria can be damaged to stop working after applying a certain number of carbon ions. Further analysis shows that radiation induced H<sub>2</sub>O<sub>2</sub>, a severe cytotoxin, is connected to mitochondrial depolarization and is limited to a small region around the damaged mitochondrion. A main obstacle of such targeted irradiation experiments is that only a few to several hundred cells can be irradiated. It hinders common clonogenic cell survival experiments, the gold standard in radiation biology. Therefore, in order to address the cellular reaction longer times after irradiation, a live cell system was installed where individual cells can be followed several days in order to obtain information on their viability, apoptotic or necrotic status and their potential for proliferation.

Another topic investigated at SNAKE is the potential of proton minibeam radiation therapy, a kind of new tumor therapy option invented by us that allows to reduce side effects in the tumor while keeping tumor control as usual. We showed the potential of tissue sparing by producing 20 MeV proton micro and minibeams and irradiating mouse ears in a grid pattern. In future we plan to produce 100  $\mu$ m sized proton beams at 70 MeV by installing a post accelerator at the Munich tandem accelerator to further investigate the potential of proton minibeam therapy in preclinics. Here deep lying tumors in small animals shall be irradiated in order to further prove the substantial enhancement of proton minibeam therapy on the outcome of tumor therapy.

#### 001 ULTRA-THIN DIAMOND DETECTORS FOR CELL MICRO-IRRADIATION **EXPERIMENTS**

Philippe Barberet<sup>1,2</sup>, Michal Pomorski<sup>3</sup>, Giovanna Muggiolu<sup>1,2</sup>, Eva Torfeh<sup>1,2</sup>, Gérard Claverie<sup>1,2</sup>, Cédric Huss<sup>1,2</sup>, Samuel Saada<sup>3</sup>, Guillaume Devès<sup>1,2</sup>, Marina Simon<sup>1,2</sup> and Hervé Seznec<sup>1,2</sup>

<sup>1</sup>Université de Bordeaux, Centre d'Etudes Nucléaires Bordeaux Gradignan (CENBG), 33175 Gradignan, France <sup>2</sup>CNRS, UMR5797, Centre d'Etudes Nucléaires Bordeaux Gradignan (CENBG), 33175 Gradignan, France

<sup>3</sup>CEA-LIST, Diamond Sensors Laboratory, Gif-sur-Yvette, F-91191, France

Cell irradiation with a precise number of charged-particles requires the use of an efficient detection system. When irradiating with particles in the MeV energy range, the detector has to be positioned upstream the sample as the particle range in the biological sample is not enough to reach a detector Micrometer thick single-crystal diamond membranes have been positioned downstream. introduced recently by Grilj et al [1] and offer very unique features for cell micro-irradiation experiments.

We report the development of single crystal diamond membranes suitable for dose control in targeted cell irradiation experiments with a proton microbeam [2]. A specific design was achieved to deliver single protons with a hit detection efficiency approaching 100%. The membranes have thicknesses between 1.8 and 3  $\mu$ m and are used as vacuum windows on the microbeam line. The characteristics of these membranes as well as their use in radiobiology experiments will be presented.

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## O02 THE RADIOBIOLOGY BEAM LINE FACILITY AT THE CENTRE FOR ION BEAM APPLICATIONS, NATIONAL UNIVERSITY OF SINGAPORE

Tao Ye<sup>1</sup>, Tan Hong Qi<sup>1</sup>, Mi Zhaohong<sup>1</sup>, Ce-Belle Chen<sup>1</sup>, Vanessa Lam Yuk Man<sup>1</sup>, Soo Kee Chee<sup>2</sup>, Thomas Osipowicz<sup>1</sup>, Frank Watt<sup>1</sup> and Andrew A Bettiol<sup>1</sup>

<sup>1</sup>Centre for Ion Beam Applications, Department of Physics, National University of Singapore <sup>2</sup>National Cancer Centre Singapore (NCCS)

A new beam line facility dedicated to investigations into the radiobiology of biological single cells has been constructed at the Centre for Ion Beam Applications, National University of Singapore. This facility, which has a horizontal layout, has some novel features including post lens magnetic ion deflection and scanning, reproducible focusing using magnetic field sensors built into the magnetic quadrupole lenses, and provision for a diamond exit window and confocal fluorescence microscope for high performance online investigations.

The radiobiology facility uses a system of Oxford Microbeam compact magnetic quadrupole lenses enabling high demagnifications (up to  $123 \times 51$ ) at the cell plane, and a relatively short lens to cell distances (22.5 cm). Beam optics and SRIM calculations have indicated a theoretical design performance of up to 450 nm for the proton spot size at the cell surface. These calculations take into account the thickness of the exit window, the natural gap between the exit window and the cell holder, and the thickness of the mylar substrate on which the cells are plated

To measure the spatial resolution of the proton beam at the cell position, we positioned a gold calibration grid inside the external cell chamber at a position normally occupied by cells. Preliminary results were obtained by scanning a 2 MeV focused beam over a grid, and using a surface barrier detector to construct a STIM image of the grid we have measured an in-air spot size of sub-500 nm at the cell position. Although this performance measurement was limited by the relatively poor quality of the calibration grid edges, this result is consistent with the calculated beam spot size.

We shall discuss the design of the new radiobiology beam line, preliminary results of resolution tests, as well as presenting first investigations into the damage caused by focused proton beam irradiation of human liver cancer cells by detecting DNA damage markers gamma-H2AX and 53BP1.

## 102 INVESTIGATING THE ENHANCED RADIOBIOLOGICAL EFFECTIVENESS OF HIGH-LET PARTICLES UTILIZING ION MICROPROBE SNAKE AND STED MICROSCOPY

Judith Reindl<sup>1</sup>, Katarina Ilicic<sup>2</sup>, Stefanie Girst<sup>1</sup>, Christoph Greubel<sup>1</sup>, Matthias Sammer<sup>1</sup>, Benjamin Schwarz<sup>1</sup>, Christian Siebenwirth<sup>3</sup>, Dietrich W M Walsh<sup>1</sup>, Anna A Friedl<sup>4</sup>, Thomas E Schmid<sup>2,5</sup> and Günther Dollinger<sup>1</sup>

<sup>1</sup>Universität der Bundeswehr München, Germany <sup>2</sup>Klinikum Rechts der Isar, TU Munich, Germany <sup>3</sup>RARAF Laboratory, Columbia University, New York, USA <sup>4</sup>Klinikum der Universität München, LMU, Germany <sup>5</sup>Helmholtz Zentrum München, Germany

Radiotherapy is besides surgery, chemo and immunotherapy one of the four pillars of tumor therapy. Recent improvements make radiotherapy more effective and more tolerable for the patient regarding side effects. The major improvement arises from the use of particles rather than X-rays due to their unique depth dose distribution in tissue. Additionally high-LET (linear energy transfer) particles exhibit an enhanced radiobiological effectiveness, e.g. cell killing is significantly higher at the same dose. It is speculated that the underlying mechanism is the local DNA damage density, which is much higher for high-LET particles.

At the ion microprobe SNAKE at the 14 MV tandem accelerator in Garching near Munich, cells can be irradiated with a wide range of particles from low-LET 20 MeV protons (LET = 2.6 keV/ $\mu$ m) up to high-LET 33 MeV lithium (85 keV/ $\mu$ m) and 55 MeV carbon (360 keV/ $\mu$ m) ions. These ions can be applied either randomly over the cell sample or focused to a spot size of <1  $\mu$ m. This allows to simulate the irradiation of high-LET particles by focusing the corresponding amount of low-LET particles, in order to study the mechanistic effects of damage induction. Additionally, cells can be irradiated under a small angle using a broad beam at the same setup. This allows to investigate the nanoscopic structural differences in damage induction using a super resolution STED microscope, which is also available in our group.

The irradiation with randomly distributed protons has the same effectiveness as X-ray irradiation related to cell killing or genetic damage induction. In contrast, the effect can be significantly enhanced by focusing the same number of protons to 1  $\mu$ m sized spots. It is attributed to the interaction of double strand breaks (DSB) leading to enhanced damage structures. However, the effectiveness of carbon ions cannot be reached due to enhanced DSB production in the inner core of the high LET particles.

Despite the different damage distribution in the cell nucleus between high and low LET particles, super resolution microscopy interestingly showed no differences in nanostructural clustering of DNA double-strand break repair factors. Counting of DSB, visualized by 170 nm protein clusters is used to compare with the number of predicted DSB from simulations.

Not only the spatial focusing but also temporal focusing of low-LET particles has to be investigated and is going to be installed at SNAKE. It allows to test the time scale where interaction of DNA damage, in particular of DSB takes place. Furthermore, super-resolution analysis of directly labelled DSB has to be performed in order to count the real number of high-LET radiation induced damage. This will help to understand and to accurately predict the effects of high-LET particles to tissue and to further improve radiation therapy of tumors.

## 003 A NOVEL 3D TISSUE ENGINEERING TOOL FOR BEAM THERAPY SCREENING OF PANCREATIC CANCER

Stella Totti<sup>1</sup>, Roger Webb<sup>2</sup>, Andrew Nisbet<sup>3,4</sup>, Giuseppe Schettino<sup>5</sup> and Eirini Velliou<sup>1</sup>

 <sup>1</sup>Bioprocess and Biochemical Engineering Group (BioProChem), Department of Chemical and Process Engineering, University of Surrey, Guildford, UK
 <sup>2</sup>Surrey Ion Beam Centre, University of Surrey, Guildford, UK
 <sup>3</sup>Department of Physics, Faculty of Engineering and Physical Sciences, University of Surrey, Guildford, UK
 <sup>4</sup>Royal Surrey County Hospital, NHS Foundation Trust, Guildford, Surrey, UK

<sup>5</sup>National Physical Laboratory, Teddington, London, UK

Pancreatic ductal adenocarcinoma (PDAC) is an aggressive disease with the 5-year survival rate being a dismal 8% [1]. This resistance is partly attributed to the very complex and dense tumour microenvironment (TME) of pancreatic cancer, which consists of a cocktail of tumour cells, pancreatic stellate cells, epithelial cells, extracellular matrix proteins, exosomes and vasculature and contributes to a decreased treatment efficiency [2]. Traditionally used systems for irradiation screening of pancreatic cancer are (i) animal models and (ii) 2D cultures. Animal models are very informative however, they are complex to generate and use, highly expensive and not always reproducible. 2D in vitro systems are very easy to use, cheap and reproducible, however they lack structure and cannot provide realistic recapitulation of the TME, consequently being very limited regarding their translational potential. Recent progress in tissue engineering and the development of three-dimensional (3D) culture systems has enabled a more realistic recapitulation of a 3D tissue/ tumour, including the niches and structure of the TME, the extracellular matrix protein (ECM) composition and the drug and irradiation deposition, consequently increasing the accuracy of treatment screening [3]. We have developed a 3D biomimetic polymer based 3D model for in vitro studies of pancreatic cancer, which maintained phenotypic characteristics of the cancer cell growth for more than a month, which is the longest reported *in vitro* culturing period [4-6]. In this current work, we further extended the duration of the culture to 68 days (to enable long term post treatment monitoring of the culture) and perform X-ray beam therapy on the scaffolds. More specifically, we irradiated the 3D pancreatic cancer scaffolds with X-rays, with a range of 0 - 8 Gy doses (250 keV energy in a 20 × 20 field, Royal Surrey County Hospital) at day 28 and we monitored the post-irradiation cell viability and tumour shrinkage for three weeks. Additionally, as the nature of pancreatic tumours requires locality and accuracy, not to affect the surrounding healthy tissues and organs, proton irradiation could be an alternative to substitute the conventional X-rays. More specifically, several clinical trials show great potential of proton therapy for treatment of this malignancy [7]. Therefore, proton beam irradiation (8 Gy) with the 2 MeV vertical beam line of Ion Beam Centre in Surrey University was conducted on the 3D pancreatic scaffolds and quantitative assessment of cell viability was carried out with in situ cell viability assays. Our results showed significantly higher cancer cell death verified with the MTS in the 3D scaffolds when treated with protons, as compared to X-rays. Overall, our findings suggest that this 3D pancreatic cancer model is a novel, efficient irradiation screening tool for pancreatic cancer, as it enables screening of different irradiation methods, the possibility to perform fractionated radiation and long term post irradiation cell viability monitoring. Future work will focus on (i) the combinatory effect of irradiation and chemotherapy to further enhance treatment efficiency and (ii) the irradiation of the pancreatic cancer model with higher clinical proton beam energies.

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## O04 CORRELATIVE STRUCTURE-FLUORESCENCE IMAGING OF SINGLE CELLS WITH FAST HELIUM-ION NANOPROBES

Zhaohong Mi, Ce-Belle Chen, Frank Watt and Andrew A Bettiol

Centre for Ion Beam Applications, Department of Physics, National University of Singapore, 2 Science Drive 3, Singapore 117542

In biological and biomedical research, fluorescence microscopy allow researchers to identify cellular structures labelled with fluorescent probes. In order to locate those fluorescent probes within a cell, it is of interest to combine fluorescence microscopy with high resolution structural imaging. Recently, correlative light microscopy and electron microscopy (CLEM), in which super-resolution fluorescence techniques are combined with the TEM, has become an emerging technique for use in correlated fluorescence and structural bioimaging with super-resolution. However, a distinct drawback of CLEM is that the fluorescence image and the electron microscopic image are not obtained simultaneously, leading to possible mis-registration of fluorescent probes. Other alternatives involve simultaneous Cathodoluminescence-SEM/TEM imaging, but for high spatial resolutions, this combination is essentially limited to imaging surfaces, or analyzing ultra-thin slices.

In this talk, we will demonstrate correlative structure-fluorescence imaging of single whole cells by using combined Helium-ion Induced Fluorescence Microscopy (HeIFM) and Scanning Transmission Ion Microscopy (STIM). Early examples of non-induced fluorescence imaging of cells involved the use of protons as excitation sources and organic fluorophores (dyes, fluorescent proteins etc) as fluorescent probes. Attempts of extending the protons to heavier ions (e.g. helium ions) as excitation sources for ion-induced fluorescence imaging were not ideal because of rapid ionobleaching of organic fluorophores when irradiated with helium ions. However, when performing structural imaging of cells, Helium-ion-STIM has much better energy-loss constant than proton-STIM. As such, we recently investigated new fluorescent probes with considerable iono-resistance and optimized fluorescence yields for helium-ion excitation. These candidate fluorescent probes include lanthanide-doped upconversion nanocrystals and fluorescent nanodiamonds. They therefore enable the extension of proton induced fluorescence to helium-ion induced fluorescence (HeIF) with the imaging resolution of sub-30 nm. The correlative HeIFM-STIM technique is potentially superior to CLEM techniques in that it can more precisely locate the fluorescent probes within cells, and is also superior to correlated Cathodoluminescence-SEM/TEM in that it can image whole cells.

## 005 DIAMOND-BASED MULTI-FUNCTIONAL BIOSENSORS REALIZED BY MEANS OF MeV ION BEAM LITHOGRAPHY

F Picollo<sup>1,2</sup>, A Battiato<sup>2</sup>, V Bonino<sup>1,2</sup>, E Carbone<sup>3</sup>, V Carabelli<sup>3</sup>, A Marcantoni<sup>3</sup>, L Mino<sup>4</sup>, A Pasquarelli<sup>5</sup>, G Tomagra<sup>3</sup>, M Truccato<sup>1,2</sup>, E Vittone<sup>1,2</sup> and P Olivero<sup>1,2</sup>

<sup>1</sup>Physics Department, NIS Centre of Excellence University of Torino, via P Giuria 1, 10125 Torino, Italy

<sup>2</sup>National Institute of Nuclear Physics, sect Torino, via P Giuria 1, 10125 Torino, Italy <sup>3</sup>Department of Drug Science and Technology, NIS Centre of Excellence University of Torino, Corso Raffaello 30, 10125 Torino, Italy

<sup>4</sup>Department of Chemistry, NIS Centre of Excellence University of Torino, via P Giuria 5, 10125 Torino, Italy

<sup>5</sup>Institute of Electron Devices and Circuits, Ulm University, 89081 Ulm, Germany

Action potentials generation and synaptic quantal release of neurotransmitter molecules are key mechanisms of brain activity, which are at the basis of information transmission and signal communication in neuronal microcircuits. Their detection is therefore a prominent research field in neuroscience.

In the present work, we report about the employment of collimated MeV ion beams for the 3-dimensional selective modification of diamond. In fact, highly ion-damaged diamond layers can be fabricated below the crystal surface due to the peculiar damage profile of high-energy (MeV) ions and converted to graphite after high temperature thermal treatment.

The electrically conductive channels are the key structures of innovative diamond-based multifunctional sensors, which are employed to investigate cellular activity both as biomolecules secretion (quantal exocytic events) and as electrical signal generation (action potential firing).

In particular, we report about the study of several biological systems such as cells networks of hippocampal neurons, dopaminergic neurons, chromaffin cells, that are cultured for long period directly over the device's surface taking advantage of diamond biocompatibility, or tissue slices from adrenal gland and *substantia nigra* brain compart.

Moreover, these devices were also employed for novel radiobiological experiment devoted to the investigation of ionizing radiation on neuron-like cells: for the first time the variation of cellular activity (activation of exocytosis pathways) was observed in real-time during cell irradiation with X-ray nanobeam.

## 006 PIXE IMAGING OF HYPERACCUMULATOR PLANTS USING THE MAIA DETECTOR ARRAY

J S Laird<sup>1</sup>, C G Ryan<sup>2</sup>, R Kirkham<sup>2</sup>, A van der Ent<sup>3</sup> and D N Jamieson<sup>1</sup>

<sup>1</sup>School of Physics, University of Melbourne, Melbourne, VIC, Australia
 <sup>2</sup>CSIRO, Normanby Road, Clayton, VIC, Australia
 <sup>3</sup>Sustainable Minerals Institute, the University of Queensland, Brisbane, QLD, Australia

Particle Induced X-ray Emission (PIXE) mapping of plant material provides key insights into the deportment of elements in hyperaccumulator plant species [1,2]. However, radiation damage coupled with the inherent low bandwidth of single element detectors results in either small area analyses or poor pixel sensitivities over the larger areas required for functional studies in some plants. With the exploitation of the MAIA detector array [3] to PIXE analysis described elsewhere in these proceedings [4], the large solid-angle approaches ~1.3 sr resulting in potentially orders of magnitude higher event rates. With these rates shared over 384 detector elements, dead-times in each pixel and pile-up distortion of the spectra can still be kept low even at overall throughput rates approaching 5 - 10 M/s [5]. For plant studies, the higher acceptance throughput allows the use of thinner, less absorbing X-ray filters thereby affording combined light and heavy element analysis which is a key requisite for studies into hyperaccumulator plants and a key advantage of PIXE MAIA over competing methods. Another advantage of PIXE MAIA via its high solid angle is the ability to minimise the influence of specimen damage per pixel, which leads to scission, cross-linkage, elemental migration and image degradation in general.

In this paper we explore PIXE MAIA mapping of various hyperaccumulator species and outline key findings for each. Furthermore, complementary mapping using synchrotron-XRF on the X-ray Fluorescence Microscopy (XFM) beamline at the Australian Synchrotron also fitted with a MAIA detector array provides a unique data-set allowing a comparison of the relative advantages and disadvantages of PIXE versus S-XRF for plant tissue analyses. The results of these findings are discussed in detail.

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## 007 APPLICABILITY OF MULTI-ELEMENTAL DISTRIBUTION ANALYSIS IN PLANTS

Paula Pongrac<sup>1,2</sup>, Katarina Vogel-Mikuš<sup>1,2</sup>, Marjana Regvar<sup>2</sup>, Ivan Kreft<sup>3</sup>, Mitja Kelemen<sup>1</sup>, Primož Vavpetič<sup>1</sup> and Primož Pelicon<sup>1</sup>

<sup>1</sup>Jožef Stefan Institute, Jamova 39, 1000 Ljubljana, Slovenia <sup>2</sup>Biotehnical Faculty, University of Ljubljana, Jamnikarjeva 101, 1000 Ljubljana, Slovenia <sup>3</sup>Nutrition Institute, Tržaška 40, 1000 Ljubljana, Slovenia

Most essential elements in human diets are provided either directly or indirectly by edible plants, therefore variation in the element compositions of edible produce is of considerable importance for human nutrition [1]. Substantial progress has been made in increasing concentrations of elements often lacking in our diets in edible plant tissues to reduce malnutrition in a process called biofortification [2]. Besides increasing density of essential elements, we also need to limit accumulation of those potentially harmful (e.g. cadmium and lead) to edible plant tissues [3,4].

Our knowledge of the distribution of elements between plant organs has increased considerably over the last century. In addition, we are beginning to uncover the distribution of elements between specific cell-types within an organ. It is evident that the concentration of a particular element can differ by an order of magnitude between cell types within an organ. This could be associated with the site of delivery of these elements to the organ, sites of complexation or metabolism of the element, or cell-type-specific uptake, efflux or sequestration processes [5].

Since essential and non-essential elements are not evenly distributed within plant parts it is very important to reliably assess their distribution. Micro-PIXE is ideally suited to address this problem, as it can access majority of biologically relevant elements and it provides information on their distribution in a quantitative manner. Results obtained reveal two important key features. Firstly, micro-PIXE analysis identifies the tissues with the greatest concentrations of elements, which is very important for accurate prediction for element losses during food processing. Secondly, micro-PIXE enhances our understanding of the mechanisms of element transport to and deposition within the edible portions, which has crucial role in the efforts to increase element concentration in targeted tissue.

Comprehensive overview of numerous applications of micro-PIXE in different edible plants or their parts will be presented and discussed in connection with mineral nutrition in humans.

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# 008 CAN WE USE THE INVASIVE APPLE SNAIL *POMACEA MACULATA* FOR BIOMONITORING OF LEAD CONTAMINATION IN FRESHWATER WETLANDS?

Harry J Whitlow<sup>1,2</sup>, Sanjana Banerjee<sup>3</sup>, Henry Udegou<sup>1,2</sup>, Naresh Deoli<sup>1,2</sup>, Armin B de Vera<sup>1</sup> and Paul L Klerks<sup>3,4</sup>

<sup>1</sup>Louisiana Accelerator Center, University of Louisiana at Lafayette, Louisiana, LA 70504, USA

<sup>2</sup>Department of Physics, University of Louisiana at Lafayette, Louisiana, LA 70504, USA

<sup>3</sup>Department of Biology, University of Louisiana at Lafayette, Louisiana, LA 70504, USA

<sup>4</sup>The Ecology Center, University of Louisiana at Lafayette, Louisiana, LA 70504, USA

Lead contamination in a wetlands environment is problematic since these are important sources of food (wildfowl, fish and shellfish). The apple snail *Pomacea maculata* is an invasive species and becoming widespread in Louisiana coastal wetlands. Recently, Neutron Activation Analysis (NAA) has demonstrated the bioaccumulation and depuration of As, Hg and U in soft tissues of a related species [1]. Micro-PIXE has previously been used to characterize the chronology of bioaccumulation for heavy elements using hard tissues such as mollusc shells and fish otoliths. Here we test the hypothesis that the hard tissues (corneous operculum and shell) of apple snails may be used with micro-PIXE for studying Pb pollution in tropical and sub-tropical wetland environments.

Three groups of apple snails that were bred in the laboratory were first maintained in clean water for two weeks, then placed in reconstituted water or reconstituted water spiked with high or low concentrations of  $Pb(NO_3)_2$ . The snails were fed with identical food (lettuce from a local supermarket). After two weeks, the snails were sacrificed by freezing and the shell and operculum removed and air-dried. The cortex and nucleus of the operculum were then analysed by micro-PIXE using the MeV ion microprobe with 3 MeV protons. The X-ray spectra were analysed using the GeoPIXE software.

The results showed that the ratio of Pb concentration in the operculum nucleus to that of its cortex was systematically greater than unity by an amount many times larger than counting statistical uncertainties. This confirms that Pb is incorporated in the operculum since growth of this takes place from the nucleus outwards. Remarkably, the incorporation of essential elements (Ca, Cu, Mn, Zn etc) showed complex behaviours suggesting that Pb exposure influences the uptake and/or distribution of these other elements.

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#### O09 ON THE NUMBER OF METALLIC ATOMS IN A CAGE PROTEIN MOLECULE BY MICRO-PIXE

Primož Pelicon<sup>1</sup>, Primož Vavpetič<sup>1</sup>, Mitja Kelemen<sup>1,3</sup>, Boštjan Jenčič<sup>1</sup>, Karolina Majsterkiewicz<sup>2</sup>, Artur Biela<sup>2</sup> and Jonathan Heddle<sup>2</sup>

<sup>1</sup>Jožef Stefan Institute, Jamova 39, SI-1000 Ljubljana, Slovenia <sup>2</sup>Malopolska Centre of Biotechnology, Jagiellonian University, Gronostajowa Street 7A, 30-387 Krakow, Poland <sup>3</sup>Jožef Stefan International Postgraduate School, Jamova cesta 39, 1000 Ljubljana, Slovenia

Metals play an important role in protein biology, for example forming structurally relevant coordination complexes as well as active sites in enzymes. The identification and quantification of metals bound to proteins by micro-PIXE has been successfully applied over the last two decades. The high elemental sensitivity of micro-PIXE, combined with high lateral resolution and quantification, provides the information on the stoichiometric ratio between the identified metal atoms and the sulphur, present in the cysteine, a constituent amino acid of many proteins. The associated methodology represents a very interesting application of nuclear microprobe technology, as it is addressing very important aspect of protein research.

The methodology of the micro-PIXE protein analysis was adopted according to the work of Garman and Grimme [1]. Protein solutions were applied on the 100 nm thick pioloform substrate and dried. We applied a 3 MeV proton beam with a diameter of 1.0  $\mu$ m and standard micro-PIXE detector toolkit comprising of two X-ray detectors, EBS, on-off axis STIM and chopper for dose normalization [2]. The elemental maps indicate strong coffee ring effect [3], transporting the protein molecules at the very edge of the droplet area during the drying process, forming a necklace structure of protein aggregates. Quantification procedure by Gupixwin [4] considers the matrix finite thickness and the composition, and revealed the atomic ratio between the number of metalloid centers and sulphur in the protein.

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# O10 MODELLING THE UPTAKE OF SUSPENDED MATERIALS AND SALTS IN NEARSHORE WATERS BY PLASTICS USING IBA DEPTH PROFILING ANALYTICAL TOOLS

Nicolo Tuccori<sup>1,2</sup>, Teresa Pinheiro<sup>1,3</sup>, Luís C Alves<sup>1,4</sup>, Teresa Peña<sup>1,2</sup>, Maria João Botelho<sup>5,6</sup>, Joana Raimundo<sup>5,6</sup> and Carlos Vale<sup>6</sup>

<sup>1</sup>Departamento de Engenharia e Ciências Nucleares, Instituto Superior Técnico, Universidade de Lisboa, Portugal <sup>2</sup>Departamento de Física, Instituto Superior Técnico, Universidade de Lisboa,

<sup>2</sup>Departamento de Física, Instituto Superior Técnico, Universidade de Lisboa, Portugal

<sup>3</sup>Instituto de Bioengenharia e Biociências (IBB), Portugal

<sup>4</sup>Centro de Ciências e Tecnologias Nucleares (C2TN), Portugal

<sup>5</sup>IPMA, Instituto Português do Mar e da Atmosfera, Lisboa, Portugal

<sup>6</sup>CIIMAR, Centro Interdisciplinar de Investigação Marinha e Ambiental, Universidade do Porto, Matosinhos, Portugal

Increasing amounts of floating plastic debris in rivers, estuaries and sea is a preoccupying environmental and toxicological issue. The adherence of biota, suspended particles and soluble materials to plastics, and eventually their interaction with the polymer may favour structural changes and their degradation. Microbeam capabilities offer unique possibilities to characterize the materials deposited on the surface of daily use plastics, which are rejected to the environment, as well as penetration profiles of elements that are present in those materials. IBA analytical tools, which mainly cluster PIXE and RBS techniques, enable us to measure depth distribution of elements and reconstruct a 3-dimentional picture of the polymer matrix variations. Food packaging polymers, high-density polyethylene (HDPE) and polyethylene terephthalate (PET), exposed to turbid water of the Tagus estuary were inspected using nuclear microscopy. The deposition mosaic contained clastic, biota and saline components. The depth profile of the elements present was carried out inspecting transversal sections of plastic materials and by assessing the changes in matrix and

thickness of deposited materials. This procedure used dedicated software for analysis of IBA data obtained with nuclear microscopy techniques (DAN32 and MORIA) and general purpose program (IBA Data Furnace, NDF). A peak of 15 - 20 µm width defines the typical deposition profile of sediment materials on the plastic surface. On the other hand soluble elements, such as Cl showed a diffusion profile in the polymer (see Figure 1). To illustrate these features examples of typical and complex cases are given in what concerns analytical difficulties in simulating the interface and the changeable polymer matrix in deeper regions.



Figure 1. Profiles of C, O (polymer matrix), Cl and Fe in PET. An enrichment of O at the surface and a diffusion of Cl in the polymer matrix can be depicted.

# **SESSION 2**

# FACILITIES AND TECHNIQUES

Chair: Hiroyuki Nishikawa

# 103 CONSIDERATIONS ABOUT PROJECTILE AND TARGET X-RAYS INDUCED DURING HEAVY ION BOMBARDMENT

F Fernandes<sup>1</sup>, L Amaral<sup>1,2</sup> and J F Dias<sup>1,2</sup>

<sup>1</sup>Ion Implantation Laboratory, Institute of Physics, Federal University of Rio Grande do Sul, Av Bento Gonçalves 9500, PO Box 15051, CEP 91501-970, Porto Alegre, RS, Brazil

<sup>2</sup>Graduate Program on Materials Science, Federal University of Rio Grande do Sul, Av Bento Gonçalves 9500, CEP 91540-000, Porto Alegre, RS, Brazil

In this work we present some results concerning the X-rays emitted by heavy ions during target bombardment. In this case, Cl<sup>4+</sup> and Cl<sup>5+</sup> ions with energies from 4 MeV to 10 MeV were employed to irradiate vitreous carbon planchets. Moreover, total X-ray production cross sections of Ti, Cr, Ni and Zn X-rays induced by chlorine ions were obtained as well for the same energy range. Only inner shell transitions were considered in the present work. The targets consisted of thin films deposited over vitreous carbon planchets. The results indicate that the projectile X-ray yields increase as a function of the bombarding energy for the present energy range. Effects due to projectile charge state appears to be of minor importance at these low ion velocities. It is shown that a simple exponential function can represent the continuum background of such complex spectra. The chlorine transition rates  $K\beta/K\alpha$  obtained from chlorine acting as a projectile interacting with a carbon target are about half the value when compared to the chlorine K $\beta$ /K $\alpha$  ratios obtained when a LiCl target is bombarded with  $C^+$  and  $C^{3+}$  ions with energies from 2 MeV to 6 MeV. As far as the total X-ray production cross sections induced by chlorine ions are concerned, perturbation theories like ECPSSR theory underestimates the experimental results for all targets. The disagreement varies from several orders of magnitude for inner shell transitions of Ti to a factor 5 for inner shell transitions of Zn. Although Ab Initio calculations improve the agreement between experiment and theory, it still fails to describe the experimental results at such low bombarding energies. The role of electron capture and possible mechanisms responsible for these effects are discussed.

# O11 PERFORMANCE OF THE 3.5 MV SINGLETRON HVE ACCELERATOR DEVELOPED FOR LUNA-MV

A Sen<sup>1</sup>, G Domínguez-Cañizares<sup>1</sup>, N C Podaru<sup>1</sup>, D J W Mous<sup>1</sup>, M Junker<sup>2</sup>, G Imbriani<sup>3</sup> and V Rigato<sup>4</sup>

<sup>1</sup>High Voltage Engineering Europa BV, PO Box 99, 3800 AB, Amersfoort, The Netherlands

<sup>2</sup>Laboratori Nazionali del Gran Sasso, INFN, Assergi, Italy

<sup>3</sup>Dipartimento di Fisica Università di Napoli Federico II and INFN, Napoli, Italy

<sup>4</sup>Laboratori Nazionali di Legnaro, INFN, Legnaro, Italy

HVE has designed and built a dedicated 3.5 MV Singletron accelerator to satisfy the stringent demands of the LUNA-MV project (Gran Sasso, Italy) for astronomy research, with high stability, low ripple and high beam currents for light ions. The system has incorporated a 10 GHz, all permanent magnet ECR ion source in the terminal of the single-ended DC electrostatic accelerator.

Early test results demonstrated intense ion beams of H<sup>+</sup> (~1 mA), <sup>4</sup>He<sup>+</sup> (~0.9 mA), <sup>12</sup>C<sup>+</sup> (150  $\mu$ A) and <sup>12</sup>C<sup>2+</sup> (100  $\mu$ A) by the ECR ion source and transported through the accelerator, in the Terminal Voltage range of 0.5 - 3.5 MV. Beam energy stability and ripple are in the order of 10<sup>-5</sup> and energy reproducibility is 10<sup>-4</sup>. These requirements on beam energy stability and reproducibility make the accelerator well suited for microprobe applications which require a terminal voltage stability of 10<sup>-4</sup> or less.

In this contribution, we will discuss the accelerator performance, specifically ion optical properties, beam energy stability and reproducibility.

# O12 AN OPTIMIZED <sup>19</sup>F $(p, \alpha \gamma)^{16}$ O ELEMENTAL MAPPING FACILITY

S K Vajandar<sup>1</sup>, M Q Ren<sup>1</sup>, D Zheng<sup>2</sup>, C S Hsu<sup>2</sup> and T Osipowicz<sup>1</sup>

<sup>1</sup>Department of Physics, National University of Singapore, Singapore 117551 <sup>2</sup>Faculty of Dentistry, National University of Singapore, Singapore 119083

We have implemented a new three-axis target-stage at the Centre for Ion Beam Applications (CIBA) nuclear microscopy end-station, in order to optimize the solid angles subtended by a large ( $5 \times 5$ ) NaI detector, a PIXE Si(Li) detector, and a PIPS detector. The replacement of the manual target positioning system with an in-vacuum piezo-driven actuator allows for a much closer geometry, leading to improved detection limits for Proton Induced Gamma Emission (PIGE) measurements as compared to our conventional set-up. Detection limits obtained from both setups will be discussed.

In a first application, the facility was used for the mapping of flourine distributions in untreated and laser-treated and human tooth samples. The reaction <sup>19</sup>F( $p,\alpha\gamma$ )<sup>16</sup>O has been used extensively for trace F detection because of its large cross section and because the  $\gamma$ -radiation produced (6 - 7 MeV) is in a typically near background-free spectral region. A focused 2 MeV proton beam was used and PIGE, PIXE, and RBS were carried out simultaneously. We will discuss the elemental correlations found and the effects of the laser treatment.

# O13 IN-SITU IRRADIATION AND IBIC PROBING USING PULSED AND DUAL ION MICROBEAMS

Milko Jakšić<sup>1</sup>, Andreo Crnjac<sup>1</sup>, Stjepko Fazinić<sup>1</sup>, Wataru Kada<sup>1,2</sup>, Natko Skukan<sup>1</sup> and Ivan Sudić<sup>1</sup>

<sup>1</sup>Laboratory for Ion Beam Interactions, Ruđer Bošković Institute, Bijenička cesta 54, 10000 Zagreb, Croatia
 <sup>2</sup>Gunma University, Kiryu, Japan

We present a new experimental setup for dual ion beam irradiation of materials using ion beams of the MeV energy range delivered by the two tandem accelerators. The scattering chamber of this dual microprobe accepts the ion beam from 1.0 MV Tandetron accelerator that is focused by a high excitation triplet, while the ion beam from 6.0 MV Tandem van de Graaff is collimated to a variable sized beam spots. System is dedicated for the performance of *in-situ* irradiation and probing experiments. The available imaging techniques for probing ion beam induced changes in materials include RBS and STIM channeling, IBIC and IL. The pulsed beam irradiation installed at the conventional RBI microprobe has been also employed for the same purpose. Controlling the sequences of sample irradiation and probing, could be also used to study the induced changes. The details of these new developments as well as the first applications will be presented.

# **SESSION 3**

# **QUANTUM DEVICES**

Chairs: Jan Meijer David Jamieson

# 104 AN INTRODUCTION TO QUANTUM TECHNOLOGY DEVICES WITH DONOR IMPURITIES IN SILICON AND THE ROLE OF PRECISION ION BEAMS

**B N Murdin** 

Advanced Technology Institute, University of Surrey, Guildford, GU2 7XH, UK

Shallow donor impurities in silicon, once frozen out at low temperature, share many properties in common with free hydrogen atoms, with a single electron orbiting a singly positively charged ion core. The electron's spin can be used as an information bit, e.g. down represents a 0 and up represents 1, and according to the laws of quantum mechanics, superpositions are possible where the spin is both up and down at the same time. Phosphorus donors in silicon have held the record for the longest electronic spin relaxation lifetimes in the solid state for over half a century, and the nuclear spin relaxation has recently been extended to hours by isotopic purification. Si:P is also very attractive because it can be placed with a precision approaching the bond length using an AFM/STM-based atomic-scale lithography technique. Bismuth in silicon is also useful for its very high nuclear spin, which interacts magnetically with the electron, but the atomic scale lithography is not possible for this species, and nanoscale implantation would help fill this gap.

In this talk I will give a short review quantum technology with impurities in silicon, including our own work on their electronic and dynamical properties, and describe what kinds of devices and new kinds of physics might be achievable with FIB-based single atom implantation.

# O14 NITROGEN IMPLANTATION WITH A SCANNING ELECTRON MICROSCOPE

Sascha Becker, Nicole Raatz, Steffen Jankuhn, Roger John and Jan Meijer

Felix Bloch Institute for Solid State Physics, Universität Leipzig, Linnéstrasse 5, Leipzig, Germany

Established techniques for ion implantation rely on technically advanced and costly machines like particle accelerators that only few research groups possess. We report about a new and surprisingly simple ion implantation method that is based upon a widespread laboratory instrument: the scanning electron microscope (SEM). We show, by the example of nitrogen implantation into diamond, that the SEM can be utilised to ionise atoms and molecules from the residual gas (by collisions with electrons of the beam) and subsequently accelerate and implant them into an insulating sample by the effect of a potential building up at the sample surface. The implanted nitrogen ions are subsequently converted (by thermal annealing) into nitrogen vacancy (NV) centres which are further measured by fluorescence confocal microscopy. To provide evidence that the observed centres are truly generated in the way we describe, we supplied a 98% isotopically enriched <sup>15</sup>N gas to the chamber, whose natural abundance is very low. Thus, we were able to verify that the investigated centres are actually created from the <sup>15</sup>N isotopes, by employing the method of optically detected magnetic resonance. Here, the nature of the nitrogen isotopes of the NV centres is revealed by the hyperfine coupling between the electron spin of the centre and the nuclear spin of the nucleus. Additionally, we show that this method is compatible with lithography techniques using e-beam resist, as demonstrated by the implantation of lines using PMMA.

# O15 HIGH-SENSITIVITY DETERMINISTIC IMPLANTATION DEVICES BY ION BEAM INDUCED CHARGE: TOWARDS 10 QUANTUM BITS IN 5 YEARS

S G Robson<sup>1,3</sup>, A M Jakob<sup>1,3</sup>, V Mourik<sup>2,3</sup>, B C Johnson<sup>1,3</sup>, V Schmitt<sup>2,3</sup>, F Hudson<sup>2,3</sup>, C Lew<sup>1</sup>, J C McCallum<sup>1,3</sup>, A Morello<sup>2,3</sup> and D N Jamieson<sup>1,3</sup>

<sup>1</sup>School of Physics, University of Melbourne, Melbourne, VIC 3010, Australia <sup>2</sup>Electrical Engineering and Telecommunications, UNSW, Sydney, NSW 2052, Australia

<sup>3</sup>ARC Centre of Excellence for Quantum Computation and Communication Technology (CQC2T)

We have fabricated nanoscale devices which exploit the quantum degrees of freedom of single <sup>31</sup>P donor atoms in <sup>28</sup>Si with nuclear spin coherence times above 30 s [1,2]. These devices bridge the foundations of modern information technology based on silicon into the future of ultra-scaled devices where quantum mechanics offers new functionalities for sensing, information storage, information processing and secure data transmission guaranteed by the laws of Physics. Driven by these results, the highly scalable flip-flop donor atom qubit architecture has been proposed [3] that features robust qubit entanglement over long distances, up to several hundreds of nanometres, which also loosens constraints on donor qubit placement precision. Implanted keV <sup>31</sup>P ions, positioned 20 nm below the surface, meet these constraints. To address the near-term challenge of building a large-scale 10 qubit device we have upgraded our deterministic ion implantation method [4] to operate at room temperature. A key feature of this upgrade is the use of on-chip ion impact detection electrodes, in a p-i-n configuration, with associated guard rings to reduce noise from stray charge that would otherwise degrade the signal to noise ratio. In addition, to allow for the parallel construction of a number of independent flip-flop devices, the detector electrodes are coupled to an array of independently targetable construction sites. A dedicated ultra-low noise, low capacitance, preamplifier circuit is used in conjunction with this architecture. Here we present images obtained with ion beam induced charge, employing 1 MeV He ions with the Melbourne nuclear microprobe system, to assess the internal electric field profile of the new devices and to show the effect of the guard ring structure to exclude stray noise signals from extraneous charge. These new devices form an essential component of our 10 qubits in 5 years strategy that takes advantage of the standard materials and tools of the information technology industry [5].

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# 105 A CONCEPT FOR DETERMINISTIC ION IMPLANTATION BY IMAGE CHARGE DETECTION

Paul Räcke<sup>1,3</sup>, Daniel Spemann<sup>2,3</sup>, Jürgen W Gerlach<sup>2,3</sup>, Bernd Rauschenbach<sup>1,2,3</sup> and Jan Meijer<sup>1,3</sup>

<sup>1</sup>Leipzig University, Felix Bloch Institute for Solid State Physics, Linnéstr 5, 04103 Leipzig, Germany <sup>2</sup>Leibniz Institute of Surface Engineering (IOM), Permoserstr 15, 04318 Leipzig, Germany

<sup>3</sup>Leibniz Joint Lab "Single Ion Implantation", Permoserstr 15, 04318 Leipzig, Germany

Ion implantation is one of the most powerful tools enabling modern solid state technology and research. For the functionalization of single defects for novel quantum technologies and for nanoscale semiconductor doping, a deterministic method for the placement of single ions will be the key concept. The approach in this work is based on pre-detecting ions on the way to the sample via their image charge signal induced in a dedicated detector arrangement. We present the new ion implantation set-up in Leipzig, which is based on a commercial FIB system equipped with an electron beam ion source with Wien filter to create and select a variety of ion species and charge states. Furthermore, initial image charge measurements demonstrate the path to achieving the sensitivity for single ion detection. To this end, bunches of a few hundred ions were generated and detected with detector prototypes using a frequency domain single pass detection scheme.

# O16 SIMPLE - SINGLE ION MULTISPECIES POSITIONING AT LOW ENERGY -A SINGLE ION IMPLANTER FOR QUANTUM TECHNOLOGIES

Nathan Cassidy<sup>1</sup>, David Cox<sup>1</sup>, Roger Webb<sup>1</sup>, Ben Murdin<sup>1</sup>, Luke Antwis<sup>1</sup>, Paul Blenkinsopp<sup>2</sup>, Ian Brown<sup>2</sup>, Tony Adams<sup>1</sup> and Richard Curry<sup>3</sup>

<sup>1</sup>Surrey Ion Beam Centre, University of Surrey, Guildford, GU2 7XH, UK <sup>2</sup>Ionoptika Ltd, B6 Millbrook Close, Chandler's Ford, Hampshire, SO53 4BZ, UK <sup>3</sup>The Photon Science Institute, University of Manchester, Oxford Road, Manchester, M13 9PL, UK

Quantum technologies and solid state quantum computation based on the intrinsic two level dynamics of electron spin in semiconductors has attracted widespread attention because of the strong microelectronic integration. Within the subsector of single dopant based qubits there are two main sets of dopant and substrate which look to be most promising; a single phosphorus impurity in a silicon substrate [1] and the nitrogen vacancy centre in diamond [2]. Single dopants have exhibited extremely long spin-coherence and spin-relaxation time [3] which contribute to the critical 'closed box' requirement of a quantum system, and coherent control of impurity wave functions of these single dopants has also been demonstrated [4].

A number of techniques have been utilised to construct solid state quantum devices including the use of scanning tunnelling microscopy and hydrogen resist lithography for single atom manipulation [5]. These have demonstrated the potential of such devices but are very limited in their flexibility and ability to scale-up. Hence there is a manufacturing bottleneck that is limiting further development in quantum technologies.

Current Ion Beam technology struggles to produce sub 20 nm spot sizes, and the Ion implantation even creates damage that must be annealed. There are however, significant advantages in terms of flexibility, with the ability to implant a range of ion species into a range of substrates and scale-up, with the proposed method being both quick and repeatable.

SIMPLE - Single Ion Multispecies Positioning at Low Energy - is an Ion Beam system with the capability to deterministically place ions into a substrate with sub-20 nm precision. The tool is being developed at Ionoptika, and will use current state of the art focused ion beam technology to act as an implantation and imaging tool, along with ultrahigh vacuum systems to create a clean environment for the construction of qubit systems. The key to deterministic implantation will be the >98% detection rate for the event of a single ion implantation through multiplied secondary electron detection. A liquid-metal ion source capable of delivering C, P, Si, S, Se, Bi and Er ions will be used and a separate gas source for N ions will be used. When detector efficiency is 100% with a dark count of 100/s and using a 10 fA beam current with a pulse length of 400 ns, Monte Carlo simulations of a 10 × 10 array demonstrate there is a 29% probability of implanting a perfect array of single dopants.

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#### O17 STATISTICS OF DETERMINISTIC SINGLE ION IMPLANTATION

Roger Webb<sup>1</sup>, Dave Cox<sup>1</sup>, Luke Antwis<sup>1</sup>, Lucio dos Santos Rosa<sup>1</sup>, Ben Murdin<sup>2</sup>, Paul Blenkinsopp<sup>3</sup>, Ian Brown<sup>3</sup>, Tony Adams<sup>3</sup>, Nathan Cassidy<sup>3</sup> and Richard Curry<sup>4</sup>

<sup>1</sup>Surrey Ion Beam Centre, University of Surrey, Guildford, GU2 7XH, UK <sup>2</sup>Department of Physics, University of Surrey, Guildford, GU2 7XH, UK <sup>3</sup>Ionoptika Ltd, B6 Millbrook Close, Chandler's Ford, Hampshire, SO53 4BZ, UK <sup>4</sup>The Photon Science Institute, School of Electrical and Electronic Engineering, University of Manchester, Manchester, M13 9PL, UK

Quantum technology devices based on solid-state quantum objects are not currently as advanced as those involving atoms trapped in vacuum because of the much stronger interaction with the However the advantages of solid state devices in that they provide strong environment. microelectronic integration (e.g. shallow donors in semiconductors like Si:P allowing electrical readout etc). A number of techniques have been demonstrated to construct solid state quantum devices including the use of AFM/SPM for single atom manipulation. These have demonstrated the potential of such devices but are very limited in their flexibility and ability to scale-up. Single ion implantation has less precision and creates damage that has to be annealed, but offers both flexibility and scale-up. A new single ion implanter designed by lonoptika aims to implant single ion species to within 20 nm of each other. With high quality focussed ion beams this part is relatively straight forward, the tricky part is placing precisely two atoms side-by-side. Three is too many and one is not enough. In an array of 10 × 10 (or more), gaining a high yield relies on a high success rate of detection of a single ion. Here we look at the underlying statistics for single ion doping and explore the requirements for the detection system such that implantation is achieved improved over just a reliance upon Poisson statistics. We also investigate the behaviour of the implanted ions. Even if two ions arrive at exactly the same place they will, of course, end up at different positions statistically. How good does the targeting need to be to achieve the optimum and what is the maximum energy that we can use for the primary beam to allow the two ions to still be with 20 nm of each other? Does the impact of one ion effect the position of the second ion?

# 106 FABRICATION OF SINGLE ATOM DEVICES BY DIRECT WRITE NANOFABRICATION

E S Bielejec

Sandia National Laboratories, Albuquerque, NM 87185, USA

We present on-going work to fabricate single atom devices via direct write nanofabrication using the Sandia National Laboratories nanoImplanter (A&D FIB100NI). This is a multi-species 10 - 100 kV focused ion beam system with a minimum spot size of 10 nm setup for both mass resolution using an ExB filter and single ion implantation using fast blanking and chopping (see Figure 1 for system overview and typical mass spectrum). We use a Raith lithography pattern generator for nanofabrication. The combination of high spatial resolution, variable energy and the ability to implant a range of elements from the periodic table makes this a versatile machine for a range of topics such as deterministic seeding of TaOx memristor devices [1], high resolution ion beam induced charge collection (IBIC) for probing the structure of defect cascades [2], deterministic single donor devices for quantum computing research [3], as well as, the formation of individual SiV centers in diamond [4,5] using in-situ detectors [6].



Figure 1. (a) SNL's nanoImplanter, a dual beam multi-species FIB system. (b) Mass spectrum for a AuSiSb source.

The idea for donor based quantum computing goes back to Kane [7]. We implement a fabrication pathway that combines focused ion implantation (FIB) with *in-situ* counted ion detection. We integrate avalanche photodiodes with quantum transport nanostructures and demonstrate low temperature transport in counted samples [3]. This FIB approach allows for a positioning accuracy of <35 nm, limited by the beam spot size. Figure 2 shows (a) the combined ion detector and nanostructure, (b) quantized ion detection, and (c) transport data showing a charge offset from a counted donor at low temperature.



Figure 2. (a) Optical image of the integrated in-situ single ion detector and SEM of the nanostructure for low temperature transport. (b) Histogram of the detector response showing quantized single ion detection for 200 keV Si ions with an average of 1.75 ions per pulse. (c) Low temperature transport data taken on a counted implant sample, the charge offset shown is likely due to an electron tunneling between the electrostatically formed SET and the donor.

Color centers in diamond have been used for a range of applications from metrology to single photon sources for secure quantum communication [8]. We demonstrate the ability to deterministically implant ions into photonic nanostructures with high spatial resolution [4,5]. Separately, we demonstrate the ability to detect single ion implants using an *in-situ* diamond detector with a SNR approaching 10 for detection of single 200 keV Si ions [6]. Figure 3 shows (a) an SEM image of a 2D photonic crystal in diamond, (b) IBIC map for in-situ diamond detectors fabricated at SNL.





A versatile multi-species FIB capability demonstrates utility for a range of applications including the direct write nanofabrication of single atom devices in both silicon and diamond substrates.

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# **SESSION 4**

# DETECTORS

Chairs: Ettore Vittone Maria Dolores Ynsa

# 107 EXTREME RADIATION HARDNESS AND SIGNAL RECOVERY IN THIN DIAMOND DETECTORS

N Skukan<sup>1,\*</sup>, I Sudić<sup>1</sup>, M Pomorski<sup>2</sup>, W Kada<sup>3</sup> and M Jakšić<sup>1</sup>

<sup>1</sup>Division of Experimental Physics, Ruđer Bošković Institute, 10000 Zagreb, Croatia <sup>2</sup>CEA-LIST, Diamond Sensors Laboratory, Gif-sur-Yvette, F-91191, France <sup>3</sup>Division of Electronics and Informatics, Faculty of Science and Technology, Gunma University, Kiryu, Gunma 376-8515, Japan \*Presently on long-term leave to the IAEA

Unique capability of single ion injection into the very well defined volume of the semiconductor device made microprobe technique IBIC very useful for the variety of studies of novel radiation detector structures. Development of radiation hard particle detectors has always been a challenge and of great interest for complex detectors in high-energy particle physics among the other fields. In that respect recent research of thin diamond detectors opened several new possibilities of their applications under the extreme conditions. We have investigated the signal recovery of heavily ion beam irradiated diamond membranes under the high electric field conditions. Small areas of the detector membrane were intentionally irradiated with 26 MeV oxygen ions to decrease signal amplitudes down to about 20% of the initial value. However, subsequent increase of the electric field to the values of ~100 V/ $\mu$ m almost completely recovers the signal amplitude. The interplay of signal recovery and the avalanche multiplication of the signal was therefore investigated. It was shown that moderate damage creation increases the impact of charge multiplication inside the diamond detector, so that signals are higher in damaged region comparing to the pristine detector parts. Results can lead to the development of true radiation hard particle detectors and zero dark count sensors based on avalanche multiplication.

In this presentation, several other examples of IBIC technique applications to problems associated with a development of detectors for high-energy physics community, through the Horizon 2020 project: Advanced European Infrastructures for Detectors at Accelerators, will be reviewed as well.

# O18 COMPARATIVE STUDY OF THE CHANGES PRODUCED BY THE IMPLANTATION OF FOCUSED HIGH-ENERGY IONS IN DIAMOND

M D Ynsa<sup>1,2</sup>, A de Andrés<sup>3</sup>, R J Jiménez-Riobóo<sup>3</sup>, N Gordillo<sup>1,2</sup> and M A Ramosa<sup>4</sup>

<sup>1</sup>Centro de Micro-Análisis de Materiales, Universidad Autónoma de Madrid, Madrid, Spain

<sup>2</sup>Departamento de Física Aplicada, Universidad Autónoma de Madrid, Madrid, Spain <sup>3</sup>Instituto de Ciencia de Materiales de Madrid, Consejo Superior de Investigaciones Científicas, Cantoblanco, Madrid, Spain

<sup>4</sup>Departamento de Física de la Materia Condensada, Condensed Matter Physics Center (IFIMAC) and Instituto Nicolás Cabrera, Universidad Autónoma de Madrid, Madrid, Spain

The implantation of focused high-energy ions is a promising tool in order to manufacture microdevices for technological, electronic, optical or even biological applications. Nevertheless, the quality of these micro-devices is conditioned not only by the implanted materials and the microstructure size, but also by the effects that the beam produces. Diamond is a material with remarkable properties. However, keV ion implantation can significantly modify its characteristics and limit its applicability. Although previous results suggest that MeV ion implantations cause less damage to the diamond lattice, many issues should be clarified yet. In this work, the changes produced in diamond by the implantation of different focused MeV ions such as boron, oxygen, silicon and protons are studied using Raman and photoluminescence analysis. The comparative study has allowed us to identify if the changes are due to the implantation itself or to the implanted ions. Particular attention has been devoted to the effects produced in the irradiation boundaries. Focalization capabilities of CMAM microbeam line for heavy ions are also evaluated.

# O19 IBIC MICROSCOPY - THE MOST POWERFUL TOOL FOR TESTING MICRON-SIZED SENSITIVE VOLUMES IN STRIP DETECTORS AND MICRODOSIMETERS USED IN SYNCHROTRON MICROBEAM RADIATION THERAPY AND HADRON THERAPY

Zeljko Pastuovic<sup>1</sup>, Thuy Linh Tran<sup>2</sup>, Jeremy Davis<sup>2</sup>, Dale Prokopovich<sup>1</sup>, Michael Lerch<sup>2</sup>, Marco Petasecca<sup>2</sup>, Rainer Siegele<sup>1</sup>, Jason R Paino<sup>2</sup>, Andrew Dipuglia<sup>2</sup>, Benjamin James<sup>2</sup>, Anatoly Rosenfeld<sup>2</sup> and David Cohen<sup>1</sup>

<sup>1</sup>Australian Nuclear Science and Technology Organisation, Locked Bag 2001, Kirrawee DC, NSW 2232, Australia

<sup>2</sup>Centre for Medical Radiation Physics, University of Wollongong, NSW 2522, Australia

Depending on the radiotherapy modality, i.e. synchrotron microbeam radiation therapy (MRT) or hadron therapy (HT), different detector architectures are required. For the synchrotron MRT, silicon strip detectors are ideally suited given their excellent spatial resolution allowing for accurate measurements of X-ray microbeam full width half maximum (FWHM) and peak to valley dose ratio (PVDR). For HT, devices based on the microdosimetric approach are more suitable. Microdosimetry is a methodology to measure the distribution of radiation dose delivered to absorbing medium (tissue itself or tissue equivalent) with the micrometre-scale spatial sensitivity. Today, most microdosimeters in a development or testing stage for applications in HT are based on the patterned array of micron-sized sensitive volumes achieved through the basic semiconductor hetero-junction configuration and manufactured by silicon semiconductor technology, although other materials like diamond are being used more often due to the higher radiation hardness and the better biological matching with the human tissue.

Ion Beam Induced Charge (IBIC) microscopy performed using highly tuned ion microbeams is the most powerful tool for the analysis of the charge carrier transport properties in semiconductor devices based on semiconductor hetero-junction or metal-on-semiconductor configurations.

Here we present overview of recent applications of the IBIC microscopy at the Centre for Accelerator Science of ANSTO in the field of microdosimetry in collaboration with the Centre of Medical Radiation Physics of the University of Wollongong. The reduced-rate H, He and C ion microbeams with energies in the MeV range and sub-micrometre spot-sizes have been used for the investigations of the charge collection efficiency (CCE) in order to measure the spatial distribution and uniformity of CCE in different polarization conditions. This information allows the determination of the charge carrier transport properties in a particular device and to quantify its ability to accurately determine the energy deposited by incident ionizing radiation - two fundamental requirements of any microdosimeter.

The results of the IBIC studies will be used for (i) optimization and further development of strip detectors and microdosimeters capable of measuring high radiation dose gradients with high spatial resolution sensitivity for the MRT and HT, (ii) quality assurance of microdosimetres, (iii) development of homogeneous and heterogeneous phantoms of increasing anatomical accuracy for radiation therapy in general, and (iv) development and verification of Monte Carlo based treatment planning systems.

#### O20 DETERMINATION OF RADIATION HARDNESS OF SILICON DIODES

E Vittone<sup>1</sup>, J Garcia Lopez<sup>2</sup>, V Grilj<sup>3</sup>, M Jakšić<sup>3</sup>, M C Jimenez Ramos<sup>4</sup>, A Lohstroh<sup>5</sup>, Z Pastuovic<sup>6</sup>, S Rath<sup>7</sup>, R Siegele<sup>6</sup>, S Skukan<sup>3</sup>, G Vizkelethy<sup>8</sup> and A Simon<sup>9</sup>

<sup>1</sup>Physics Department and INFN, Torino Unit, via P Giuria 1, 10125 Torino, Italy
<sup>2</sup>Departamento de Física Atómica, Molecular y Nuclear, Facultad de Física, Universidad de Sevilla, Spain
<sup>3</sup>Ruđer Bošković Institute, Zagreb, Croatia
<sup>4</sup>Centro Nacional de Aceleradores, University of Sevilla, Spain
<sup>5</sup>Department of Physics, University of Surrey, Guildford, Surrey, UK
<sup>6</sup>Centre for Accelerator Science, ANSTO, 1 New Illawarra Road, Lucas Heights, NSW 2234, Australia
<sup>7</sup>National Physical Laboratory, Council of Scientific and Industrial Research, Dr K S Krishnan Road, New Delhi, India
<sup>8</sup>Sandia National Laboratories, Radiation-Solid Interactions, PO Box 5800, MS 1056, Albuquerque, NM 87185, USA
<sup>9</sup>International Atomic Energy Agency, Vienna International Centre, PO Box 100, 1400 Vienna, Austria

In this contribution, we describe an experiment aimed to measure the physical observables, which can be used for the assessment of the radiation hardness of commercially available silicon photo diodes commonly used as nuclear detectors in particle accelerator laboratories. The experiment adopted the methodology developed during the International Atomic Energy Agency (IAEA) Coordinated Research Project (CRP No F11016) "Utilization of Ion Accelerators for Studying and Modelling Ion Induced Radiation Defects in Semiconductors and Insulators" [1].

This methodology is based on the selective irradiation of micrometer-sized regions with different fluences of MeV ions using a high rate ion microbeam and on the measurement of the charge collection efficiency (CCE) degradation by Ion Beam Induced Charge (IBIC) microscopy performed under different biasing conditions, using a range of different probing ions. The IBIC results are analyzed through a theoretical approach based on the Shockley-Read-Hall model for the free carrier recombination in presence of ion-induced deep traps. This interpretative model allows the evaluation of the material radiation hardness in terms of recombination parameters for both electrons and holes, which include the electron and hole capture cross sections of traps generated by ions and the yield of electrically active traps generated from vacancies created in the ion cascade - as predicted by Monte Carlo Simulations.

The device under study in this experiment was a Hamamatsu S1223 p-i-n photodiode. The pristine diode was initially characterized by (i) standard electronic characterization techniques to determine its doping, and (ii) the Angle-Resolved IBIC to evaluate its effective entrance window. Nine regions of (100 × 100)  $\mu$ m<sup>2</sup> were irradiated with 11.25 MeV He ions up to a maximum fluence of 3 × 10<sup>12</sup> ions/cm<sup>2</sup>. The CCE degradation at different applied bias voltages was measured by the IBIC technique using 2.3 MeV H, 11.25 MeV He, 1.4 MeV He as probing ions.

Here presented model proved to be effective for fitting the experimental data. The fitting parameters correspond to the recombination coefficients, which are the key parameters for the characterization of the effects of radiation damage in semiconductors.

 E Vittone, Z Pastuovic, M B H Breese, J Garcia Lopez, M Jakšić, J Raisanen, R Siegele, A Simon, G Vizkelethy, "Charge collection efficiency degradation induced by MeV ions in semiconductor devices: Model and experiment", Nucl Instrum Meths Phys Res B, Vol 372 (2016) 128-142.

# O21 QUANTITATIVE DETERMINATION OF FLUORINE USING A DOUBLE SIDED SILICON STRIP DETECTOR (DSSSD)

Nathaly De La Rosa, E J Charlotta Nilsson, Mikael Elfman, Per Kristiansson and Jan Pallon

Division of Nuclear Physics, Department of Physics, Lund University, Box 118, SE-221 00 Lund, Sweden

Fluorine quantification is of great relevance in material science [1,2], biology [3], environmental science [4], and geology [5], among others fields. One way of quantifying fluorine is through Nuclear Reaction Analysis (NRA). In this work, fluorine presence in standard reference material and geological samples was associated with the detection of  $\alpha$ -particles emitted in the <sup>19</sup>F(p, $\alpha$ )<sup>16</sup>O reaction. To optimize the technique, the reaction was studied with protons at different energies at the Lund Ion Beam Analysis Facility (LIBAF). Reaction yields and detection limits were investigated using proton energies from 600 keV to 2000 keV. A double-sided silicon strip detector (DSSSD) [6] was employed to detect the  $\alpha$ -particles at a solid angle of 2 sr. The DSSSD consists of 2048 segments, which enables the use of high analytical currents, as the total count rate is divided between many detector segments. The use of the DSSSD has been demonstrated appropriate for light element analysis, with detection limits reported below or around one wt-ppm for hydrogen [7], boron [8] and lithium [9] in previous works. In the case of fluorine, the work focused on evaluation both for optimization of fluorine measurements but also on finding the most suitable conditions for simultaneous analysis of boron, lithium and fluorine.

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- [7] L Ros et al, "Deuterium/hydrogen microscopy in astrogeological material using a elastic recoil approach", Nucl Instrum Meths Phys Res B, Vol 348 (2015) 273-277.
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# O22 POSITION-SELECTIVE INTRODUCTION OF ELECTRICALLY EXCITABLE COLOR CENTERS IN SIC PN JUNCTION DIODE BY PROTON BEAM WRITING

Y Yamazaki<sup>1</sup>, Y Chiba<sup>1,2</sup>, T Makino<sup>1</sup>, S I Sato<sup>1</sup>, N Yamada<sup>1</sup>, T Satoh<sup>1</sup>, Y Hijikata<sup>2</sup>, K Kojima<sup>3</sup>, S Y Lee<sup>4</sup> and T Ohshima<sup>1</sup>

 <sup>1</sup>National Institute for Radiation Science and Technology (QST), Takasaki, Gunma 370-1207, Japan
 <sup>2</sup>Saitama University, Saitama 338-0825, Japan
 <sup>3</sup>National Institute of Advanced Industrial Science and Technology, Tsukuba, Ibaraki 305-8568, Japan
 <sup>4</sup>Center for Quantum Information, Korea Institute of Science and Technology, Seoul, 02792, Republic of Korea

Recently, silicon carbide (SiC) has attracted much attention because its specific color centers such as silicon vacancy ( $V_{si}$ ) [1], carbon antisite-vacancy pair [2] and SiO<sub>2</sub>/SiC interface defects [3] have been proved to act as single photon sources (SPSs). These color centers are expected to be one of the most promising candidates for building blocks to manipulate electron spins toward realization of quantum computer, communication and sensors. As for quantum sensor applications, high sensitive thermometry and vector magnetometry have been demonstrated using  $V_{si}$  defects [4,5].

SiC has superior physical properties suitable for high-power and high temperature-resistant devices. In addition, there are commercially-available large size substrates and sophisticated device fabrication processes (growth, doping etc). For these reasons, SiC devices have been intensively developed for decades. Combining these devices and SPS based quantum sensors are sure to valueadded products. To realize such products, it is important to establish defect creation techniques with both high position and density controllability. Proton beam writing (PBW) is an effective technique fitting these requirements [6].

In this study, we created  $V_{si}$  into in-plane SiC pn junction diode by using focused proton beam as small as 1  $\mu$ m of diameter. Characterizations suggested that PBW successfully introduced electrically excitable  $V_{si}$  into the device without remarkable degradation of diode performance. Detailed results will be presented in the conference.

Acknowledgement: This study was partially supported by JSPS KAKENHI grant number 17H01056.

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# **SESSION 5**

# FORENSICS AND CULTURAL HERITAGE

Chair: Milko Jakšić

# 108 FROM POTENTIAL TO END-USERS: ACCELERATOR-BASED ANALYTICAL TECHNIQUES FOR FORENSIC SCIENCE

Aliz Simon and Nuno P Barradas

International Atomic Energy Agency, Division of Physical and Chemical Sciences, Vienna International Centre, PO Box 100, A-1400 Vienna, Austria

The IAEA Physics Section is fostering a new application field of particle accelerators, namely forensic science. A new coordinated research project (CRP F11021) will further develop and utilize the unique capabilities of accelerator-based analytical techniques towards recognized needs of forensic sciences that could not be efficiently addressed by other methods [1]. Three key areas have been selected for forensics applications covering both criminal investigations and law enforcement with a special emphasis on (a) food security and provenance, (b) forgery of cultural heritage objects, and (c) glass analysis.

This talk will present the key features of the new IAEA CRP project and will give a glimpse into some strategic aspects that the accelerator facilities need to consider and clarify during the pathway to adoption for new forensic methods. End-user requirements, standardization, data interpretation, chain of custody, and nature of the relationship between IBA scientists and forensic stakeholders are some of the topics which were discussed in relevant IAEA meetings, e.g. organized together with the Ion Beam Centre of the University of Surrey, England [2] with participants from both communities.

The recent developments of the IAEA Accelerator Knowledge Portal [3] with a special emphasis on Case Studies to promote Ion Beam Analysis will be also presented.

- [1] https://www.iaea.org/newscenter/news/new-research-project-to-focus-on-use-of-nucleartechniques-in-forensic-science.
- [2] http://www-naweb.iaea.org/napc/physics/meetings/TM52459.html.
- [3] IAEA Accelerator Knowledge Portal: https://nucleus.iaea.org/sites/accelerators/.

#### O23 MACHINA: MOVABLE ACCELERATOR FOR CULTURAL HERITAGE IN-SITU NON-DESTRUCTIVE ANALYSIS

F Taccetti<sup>1</sup>, L Giuntini<sup>1,2</sup>, L Castelli<sup>1</sup>, M Chiari<sup>1</sup>, M Fedi<sup>1</sup>, C Czelusniak<sup>1</sup>, L Palla<sup>1</sup>, P A Mandò<sup>1,2</sup>, G Calzolai<sup>1</sup>, S Mathot<sup>3</sup>, G Anelli<sup>3</sup>, M Vretenar<sup>3</sup>, A Lombardi<sup>3</sup> and E Montesinos<sup>3</sup>

<sup>1</sup>Istituto Nazionale di Fisica Nucleare, Sezione di Firenze, 50019 Sesto Fiorentino, Italy

<sup>2</sup>Department of Physics and Astrophysics, University of Florence, 50019 Sesto Fiorentino, Italy

<sup>3</sup>CERN - European Organization for Nuclear Research, CH-1211 Geneva 23, Switzerland

In recent times, there has been a constantly increasing demand for *in-situ* compositional analyses in many fields and in particular in Cultural Heritage (CH), as demonstrated for example by the noticeable increment of studies employing mobile XRF scanners. However, XRF systems show several limitations that do not affect the IBA techniques. Quite recently, the restoration of the "Adorazione dei Magi" by Leonardo da Vinci was carried out at the Opificio delle Pietre Dure (OPD) in Florence. After the routine analyses, a big debate was still present about the possibility of removing some details, possibly due to a later intervention. The standard analyses, including XRF, were not sufficient to assert whether the above mentioned details were drawn by Leonardo or successively. The big dimensions and delicate structure of the painting made it unfeasible to move it to INFN-Labec and no IBA analyses were performed. In such a case, an accelerator-based analysis system would have provided an insight into the structure of the picture, not possible to obtain with the other techniques, that could allow giving an answer to the many questions asked by the restores and art historians.

Starting from these evidences, the European Organization for Nuclear Research (CERN) and the Italian National Institute for Nuclear Physics (INFN), whit a long-lasting and significant experience in the development, use and application of particle accelerators in diverse fields, have decided to jointly develop a transportable system for *in-situ* Ion Beam Analysis (IBA) of materials.

This achievement will be possible thanks to:

- more than 35 years of INFN-Labec experience in the field of the CH diagnostic;
- more than 25 years of RFQ design and construction at CERN;
- an innovative beam dynamics developed at CERN for a high frequency radiofrequency quadrupole cavity (HF-RFQ), minimizing beam loss at the high energy side;
- INFN-Labec technology for the control of external beams of very weak intensity.

The MACHINA Project, started at the beginning of 2018, spans over two years and is organized in three phases. The first one aims at the construction of a system composed of the proton source, the HF-RFQ and the RF power source (the Low Energy Beam Transfer and accelerator), able to produce 2 MeV proton beams in vacuum.

In the second phase, a fully-operational prototype will be developed, adding to the previous system the High Energy Beam Transfer and the detection set-up. MACHINA as a whole will be then tested at INFN-Labec for tuning and characterization of the system and first IBA measurements.

In the final part of the project, MACHINA will be moved to the OPD for the first artwork studies.

The OPD will be the seat of MACHINA, which will be however used for in-situ measurements in different sites when IBA measurements will be advisable in order to solve problems otherways difficult or impossible for other techniques.

#### O24 DAPNe-IBA: A NEW TOOL FOR FORENSIC AND BIOMEDICAL SCIENCE?

Melanie J Bailey<sup>1</sup>, Janella de Jesus<sup>1,2</sup>, Holly Lewis<sup>3</sup>, Catia Costa<sup>2</sup>, Josephine Bunch<sup>4</sup> and Roger P Webb<sup>2</sup>

<sup>1</sup>Department of Chemistry, University of Surrey, UK <sup>2</sup>Surrey Ion Beam Centre, University of Surrey, UK <sup>3</sup>Advanced Technology Institute, University of Surrey, UK <sup>4</sup>National Physical Laboratory, Teddington, UK

Due to MeV-SIMS and micro calorimeter X-ray detectors, our community have recently been presented with new opportunities for providing chemical speciation of samples. These are exciting development that significantly increase the analytical power of IBA. However, the current ability of IBA techniques to provide **trace** molecular information AND to accurately identify compounds is limited. Even for MeV-SIMS, the fragmentation of analytes precludes the detection of large molecules and the types of mass spectrometers employed limit the unambiguous identification of many molecular signals.

Direct Analyte Probed Nano Extraction [1] Mass Spectrometry (DAPNe-MS) is a method of selecting and removing a small quantity of material from a surface, using a capillary tip attached to a nanomanipulator. The extracted material is then introduced into a mass spectrometer via nanoelectrospray ionisation. The technique has achieved sensitivity at the attogram level and is particularly suited to picking up trace materials from surfaces. Unlike MeV-SIMS, there is minimal fragmentation, and this provides better selectivity. It can also be used with a high resolution mass spectrometer to improve confidence in peak assignment.

We have been awarded a grant from the UK Electronics and Physical Sciences Research Council (EPSRC) to install a system for Direct Analyte Probed Nano Extraction (DAPNe) to be used in conjunction with ion beam analysis. Melanie Bailey has also been awarded a prestigious five year Fellowship from the EPSRC, which will begin in October 2018 and gives opportunity to grow this field. The basic concept is to provide a trace element or secondary ion image using IBA techniques and then select points of interest to be studied in greater detail using DAPNe. In this presentation, we will describe the technical set up of the recently installed system. We will show for the first time the possibility of applying DAPNe - liquid chromatography mass spectrometry (LC-MS) for peak assignment. Using the examples of automotive paints and hair, we will show how DAPNe and IBA together can add significant value in forensic and biomedical science.

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# **SESSION 6**

# NANO STRUCTURES AND DEVICES

Chairs: Gyorgy Vizkelethy Guanghua Du

# O25 REFRACTIVE INDEX CHANGE AND THERMO-OPTIC EFFECT IN POLYDIMETHYLSILOXANE NANOCOMPOSITES WITH OXIDE NANOPARTICLES INDUCED BY PROTON BEAM WRITING

Yuto Kaneko<sup>1</sup>, Hidetaka Hayashi<sup>1</sup>, Yasuyuki Ishii<sup>2</sup>, Wataru Kada<sup>3</sup> and Hiroyuki Nishikawa<sup>1</sup>

<sup>1</sup>Department of Electrical Engineering, Shibaura Institute of Technology (SIT), Tokyo, Japan

<sup>2</sup>Institutes for Quantum and Radiological Science and Technology (QST), Takasaki, Gunma, Japan

<sup>3</sup>Graduate School of Science and Technology, Gunma University, Gunma, Japan

Proton beam writing (PBW) is a direct writing technique of waveguides in various polymeric materials such as polymethylmethacrylate (PMMA) and polydimethylsiloxane (PDMS). Optical switching has been demonstrated by a Mach-Zehnder interferometer embedded in these polymer optical waveguides using thermo-optic (TO) effects [1].

In this study, nanoparticles of titania, silica, and alumina in diameter of 10 to 21 nm were mixed with PDMS to obtain nanocomposites to modify the optical properties of PDMS. The refractive index of the PDMS composites increased by 0.001 to 0.008 after mixing of these particles with concentration of 1.0 wt%. Proton beam irradiation was performed using either a proton beam writer at SIT, or a microbeam line with a single-ended accelerator at QST Takasaki. By 1.0 MeV proton beam irradiation, we observed refractive index increase of 0.007 for the titania/PDMS composite with a fluence of 100 nC/mm<sup>2</sup>, which is high enough to write optical waveguides embedded in the nanocompotites.

The thermos-optic coefficient (dn/dT) of these nanocomposites were evaluated by spectroscopic ellipsometry in the temperature range of 25 - 65°C. The TO effect of the titania/PDMS composite is more significant than those of PDMS and the composites with silica and alumina nanoparticles. This shows that the titania/PDMS nanocomposite is suitable for optical switching devices utilizing the TO effect.

[1] W Kada, K Miura, H Kato, R Saruya, A Kubota, T Satoh, M Koka, Y Ishii, T Kamiya, H Nishikawa, O Hanaizumi, Nucl Instrum Meths Phys Res B, Vol 348 (2015) 218-222.
# O26 MICRO HEAVY ION BEAM IRRADIATION AS THE TOOL TO CREATE THE ON/OFF FUNCTION OF GLASS

Tieshan Wang<sup>1</sup>, Liang Chen<sup>1</sup>, Peng Lv<sup>1</sup>, Bingtao Zhang<sup>1</sup>, Haibo Peng<sup>1</sup>, Frans Munnik<sup>2</sup> and Rene Heller<sup>2</sup>

<sup>1</sup>School of Nuclear Science and Technology, Lanzhou University, Lanzhou, China <sup>2</sup>Ion Beam Center, Helmholtz-Zentrum Dresden Rossendorf, Dresden, Germany

Glass is a very important function material and widely used for many applications. Different from amorphous and crystalline material, glass has some free volume, so it is not only transparent, but also easy to be changed by some treatment, become a special function material. In this work, heavy ion beam irradiation is used to enhance the gas diffusion probability in glass and to find a way to charge into glass spheres without changing the mechanical properties. The radiation effect of glass under heavy ion irradiation has been studied by Ar, Kr and Xe etc ion irradiation, the micro- and macro-effects of irradiated glasses were characterized by Raman, Scanning Microscopy (SEM), Atomic Force Microscopy (AFM), Transition Electron Microscopy (TEM) and Nano-Indentation etc methods. Significant change of micro next work, accumulation of oxygen, descent of hardness etc were found in ion irradiated glass samples following the increase of fluence. The migration of elements was also observed by X-ray Photon-Spectrometer (XPS). In order to study the gas diffusion behaviors changed by ion irradiation, micro-glass spheres were used to test irradiation effect. The hollow glass spheres are in size of ~600 microns with ~3 microns wall. Light gas atom can be easily charged into spheres under certain temperature and pressure conditions. But heavy gas atoms cannot be charged into the spheres. Argon gas (Ar) was used as trace element for this study. Ar, Kr and Xe etc ions with certain energies were used to irradiate glass spheres. With the increase of irradiation does, Ar can be occasionally charged into some spheres, but the amount of Ar in spheres was still out of controlling, and the mechanical properties of spheres were also changed significantly. So that, micro heavy ion beam was proposed to irradiate small area and to create micro diffusion pass. Silicon ion (Si<sup>6+</sup>, 15 MeV) in size ~20 microns was used for irradiation. At a fluence over  $1 \times 10^{16}$  Si/cm<sup>2</sup>, ~40% of spheres were charged into Ar with a pressure between 0.001 - 3.10 Bar. The Ar pressure in spheres were measured by the X-ray Fluorescence (XRF) and Quadrupole Mass-Spectrometer (QMS). However the Ar pressure in spheres was quite different under the same charging conditions, and no Ar was found in 60% of spheres. It implies that ion beam can be used to change the diffusion coefficient of glass, but the irradiation also damages the mechanical properties. Using micro beam can create micro diffusion pass in glass spheres' wall, but the diffusion coefficient depends also on the thickness of wall, temperature and gas changing gas pressure etc parameters. To optimize the parameters need to do more experiments. Further study is undergoing. The results in detail will be present in the conference.

## O27 ELECTRONIC CIRCUIT FORMATION ON FLEXIBLE POLYMER SURFACE PROCESSED BY 1 MV ACCELERATED HYDROGEN MOLECULAR IONS

Hidetaka Hayashi<sup>1</sup>, Wataru Furukawa<sup>2</sup> and Hiroyuki Nishikawa<sup>1,2</sup>

<sup>1</sup>Research Organization for Advanced Engineering, Shibaura Institute of Technology, Japan

<sup>2</sup>Shibaura Institute of Technology, Japan

One of the remarkable characteristics of accelerated hydrogen molecular ions [1] and Proton Beam Writer (PBW) is their capability to modify engineering plastics such as polyester and polyimide of no photosensitivity. Utilizing this capability we can make various carvings, grooves, pits, thru-holes and beam like structures of micron meter sized on the surface of plastics. As these engraved hollow structures are useful not only to fill various functional materials in them but also to give mechanical freedom of movement, we can change the surface of flexible polymer to functional surfaces. The functionality of these hollow structures is decided by the shape and materials filled in them and are very useful to make not only electronic but fluidic and photonic devices. In this report we show circuit structure made of nanometer sized silver oxide paste (Figure 1). We used 1 MV accelerator to accelerate H3+, H2+ and H+ (proton) and 5% KOH development process to form multi-level hollow structure that is useful to make multi-layered circuit for industrial applications.



Figure 1. Resonator with comb capacitor made of conductive Ag paste.

[1] H Hayashi, S Hayakawa, H Nishikawa, "Microstructure processing on plastics by accelerated hydrogen molecular ions", Nucl Instrum Meths Phys Res B, Vol 404 (2017) 233-237.

#### O28 THE CONCEPT OF SUPER-RESOLUTION ION BEAM IMAGING WITH HIGH ENERGY SINGLE IONS

Guanghua Du, Jinlong Guo, Guangbo Mao, Wenjing Liu, Ruqun Wu, Yaning Li, Jing Zhao, XiaoYue Li and Artem Ponomarov

Institute of Modern Physics, Chinese Academy of Sciences, Lanzhou, China

The resolution of a microbeam is traditionally defined by the size of the beamspot, in which the beam position is assured by the distribution accuracy inside the microbeam beamspot. Recently, the invention of single molecule localization microscopy is followed by many new technologies in improve the resolution of light microscopy to beyond the Abbe limitation. High energy ion beam, and especially the heavy ion beams are of advantages in the high energy deposition and high stopping power in material, and the induced scintillation photons might be enough to determine the ion position. This presentation will report the concept of ion beam imaging beyond the resolution of microbeam beamspot, and show the preliminary single ion positioning investigation using the scintillation light from several kinds of heavy ions.

#### O29 NUCLEAR MICROPROBE CHARACTERISATION OF DEFECTS IN ZINC RECYCLED CEMENTED CARBIDES

C S Freemantle<sup>1</sup>, M Topic<sup>2</sup> and C A Pineda-Vargas<sup>2</sup>

<sup>1</sup>Pilot Tools (Pty) (Ltd), PO Box 27559, Benrose, 2011, South Africa <sup>2</sup>iTHEMBA Laboratories, National Research Foundation, PO Box 722, Somerset West 7129, South Africa

Cemented carbide (WC-Co) alloys consist of tungsten carbide grains bound together with a metal binder, e.g. Co. The WC phase provides hardness and strength, while the binder imparts toughness [1]. These 'hardmetals' are widely used in industry.

Recycling of cemented carbides is of increasing importance due to resource depletion, and the drive towards a 'greener' global economy. The zinc process is the dominant recycling method used, in which molten zinc alloys with the binder phase in a vacuum furnace, separating the WC grains from the binder. An embrittled aggregate material is left behind which is mechanically broken down into powder for re-use [2]. The zinc is distilled at the end of the process, and can also be further recycled.

A problem with recycling is that it can introduce impurities. This article presents a case study, performed at a cemented carbide manufacturing plant, in which unacceptable batch defects (surface pits on the alloy) were investigated, and contamination was suspected. Particle-induced X-ray emission (micro-PIXE) techniques were used, along with other complementary methods, to determine the source of the defects, and propose corrective action for the company.

Micro-PIXE is relatively new to metallurgical engineering and generally can detect trace elements (impurities) in metals and alloys at low concentrations [3]. In this study, the quantification of W, Co, Zn, Fe, Ca and other elements was performed, and their 2-D distributions were mapped out. The zinc from the recycling process was studied in the same way. The defects in the sintered components were found to be associated with Ca, introduced during recycling, and an explanation for this was provided to the manufacturer. This case study proves that micro-PIXE can serve as a powerful diagnostic method in materials science and engineering, as well as industry.

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## O30 MICROSTRUCTURAL AND ELEMENTAL CHARACTERISATION OF CEMENTED TUNGSTEN CARBIDE TOOL GRADE SCRAP METAL

J Kurasha<sup>1</sup>, N Sacks<sup>1</sup>, M Topic<sup>2</sup> and C Mtshali<sup>2</sup>

<sup>1</sup>School of Chemical and Metallurgical Engineering and DST-NRF Centre of Excellence in Strong Materials, University of the Witwatersrand, Private Bag 3, University of the Witwatersrand, P/B 3, Wits 2050, Johannesburg, South Africa <sup>2</sup>iTHEMBA Laboratories, National Research Foundation, PO Box 722, Somerset West 7129, South Africa

Cemented tungsten carbides (WC-Co), also known as hardmetals, are widely used as cutting tools in the manufacturing industry. In order to improve the machinability of steels, small additions of cubic carbides such as TiC, TaC and NbC are added to the WC-Co alloys [1], which are then known as 'tool grade' cemented carbides. Due to the increasing demand for hardmetal tools in the manufacturing sector and the strain being placed on natural tungsten resources, the recycling of tungsten carbide scrap metal has gained significant momentum. In this study, tool grade scrap metal cutting inserts were subjected to the zinc recycling process [2]. Tungsten carbide, cubic carbides (TiC, TaC, NbC, TiCN) and Co were recovered from the recycled scrap material, and were then re-used as raw material in the production of new tool grade alloys. In typical industrial applications, commercial products may be produced using powder blends comprising at most 50 - 70% recycled powders. Here the alloys were produced using 100% recycled powders in order to assess commercial production possibilities. The powders and alloys produced from the recycled materials were compared to powders and alloys produced from new materials in order to benchmark commercial The micro-Proton-Induced X-ray Emission technique identified the elemental viability. concentrations and provided elemental maps showing the distribution of the elements in both powders and alloys. The powders were manufactured into alloys using a liquid phase sintering process during which the Co binder becomes liquid and forms a matrix around the carbide particles. During the sintering process a generally cubic free layer (CFL) was formed at the surface of all the alloys, i.e. a region which comprised of Co and W and did not contain any Ti and only trace amounts of Ta and Nb. Elemental mapping revealed that the recycled alloys had a narrower CFL layer compared to the new alloys. In general the distribution of the elements was more homogenous in the recycled alloys than the new alloys, with the exception of Co where the concentration was higher in the CFL compared to the bulk material. Further evidence of the inhomogeneity of the new materials was found when elemental mapping revealed pools of Ti, Co, Fe and Cr in the new powders, which were not found in the recycled materials. S, Cr and Fe were the major impurities detected in all the powders and alloys, with Zn being detected as an additional major impurity in the recycled materials. Trace elements such as Ni, Mn and Ca were also detected as impurities.

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#### O31 OPTICAL PROPERTIES OF AN ENSEMBLE OF G-CENTERS IN SILICON

C Beaufils<sup>1</sup>, W Redjem<sup>1</sup>, E Rousseau<sup>1</sup>, V Jacques<sup>1</sup>, A Yu Kuznetsov<sup>2</sup>, C Raynaud<sup>3</sup>, C Voisin<sup>3</sup>, A Benali<sup>4</sup>, T Herzig<sup>5</sup>, S Pezzagna<sup>5</sup>, J Meijer<sup>5</sup>, M Abbarchi<sup>4</sup> and G Cassabois<sup>1</sup>

<sup>1</sup>Laboratoire Charles Coulomb, Université Montpellier, CNRS 34095 Montpellier, France

 <sup>2</sup>Department of Physics, University of Oslo, NO-0316 Oslo, Norway
<sup>3</sup>Laboratoire Pierre Aigrain, Ecole Normale Supérieure, Université Paris Diderot, UPMC, CNRS UMR8551, 24 rue Lhomond, 75005 Paris, France
<sup>4</sup>CNRS, Aix-Marseille Université, Centrale Marseille, IM2NP, UMR 7334, Campus de St Jérôme, 13397 Marseille, France
<sup>5</sup>Department of Nuclear Solid State Physics, Felix-Bloch Institute for Solid-State

Physics, Universität Leipzig, Linnéstrasse 5, 04103 Leipzig, Germany

The steady demand of better performing electronics was always satisfied by reducing structure sizes on microelectronic circuits. This downsizing process comes to its limit by the so called interconnect bottleneck problem. When the feature sizes of integrated circuits become too small, the signal delay is no longer dictated by the gate switching time but by the wiring delay. Due to tighter packaging of metal conducting paths, parasitic capacitance increases and therefore signal propagation delay gets worse. Since this physical limitation is inevitable, new designs of circuits and computers are needed.

One way to overcome this problem is the use of silicon photonics. Silicon has been the main material in semiconductor production for decades due to its huge amount of natural resources and many ideal properties like high purity, easy manufacturing, temperature resistance, etc. Unfortunately, due to its indirect band gap, natural silicon is highly improbable to emit photons. But because of the very promising applications, a lot of effort was made to make silicon glow. One way to achieve this is the creation of a defect in the silicon lattice called G-center. This defect consists of one interstitial and one substitutional carbon atom in the silicon lattice and it can be excited to emit photons with a zero phonon line at 1280 nm, matching the near infrared O-band of telecommunication. Therefore, it is a very promising candidate for the realisation of photonic based microchips and quantum computers.

Within our project we will develop an easy method to generate G-centers only by well-established methods from semiconductor production. We use a solid source ion implanter with a maximum acceleration voltage of 100 kV to implant carbon ions with different concentrations and energies into various silicon structures like photonic crystals and SOI layers, or in dependence of crystal orientation and resistivity. After an annealing step to incorporate the carbon atoms into the lattice, we use our Singletron high energy accelerator with focussing setup and microbeam scanning system for microprobe applications to irradiate the samples with protons and other elements of up to 3 MeV to create the interstitial carbon. Afterwards the samples are measured with confocal photoluminescence microscopy to investigate the optical properties of the created centers. The aim is to develop fully working LEDs, lasers, and single photon sources directly from silicon itself.

Until now we have been able to characterize the saturation power of an ensemble of G-centers. We investigated the recombination dynamics, leading to a new value for the lifetime of the state at low temperature. According to the theoretical modelling of the vibronic spectrum we estimated the spatial extension of the electronic wave function in a G-center. Moreover, the temperature dependence of the emission spectrum and recombination dynamics was recorded, leading to new insights of the characteristics of this fascinating defect.

#### O32 OPTICAL EFFECTS OF FOCUSED PROTON IRRADIATION OF DIAMOND

Andrew A Bettiol<sup>1</sup>, Shuvan Prashant Tugara<sup>1</sup>, Huining Jin<sup>1</sup> and Ee Jin Teo<sup>2</sup>

<sup>1</sup>Centre for Ion Beam Applications, Department of Physics, National University of Singapore

<sup>2</sup>Institute of Materials Research and Engineering (IMRE), Agency for Science, Technology and Research (A\*STAR)

Optical centers in diamond have been studied for decades and the origins of many of them have been documented in several reviews. In recent years, new applications have emerged for optical centers in diamond in areas such as photonics, quantum computing and biology. One of the few methods of spatially controlling the formation of optical centers in diamond is through ion implantation. For this reason, there has been a concerted effort in recent times to develop technology for deterministic ion implantation. The characterization methods of choice for studying optical centers are Raman and photoluminescence spectroscopy. In this study, we utilize high resolution Raman and photoluminescence hyper-spectral imaging to study the formation and annealing behavior of optical centers formed by focused mega electron volt (MeV) proton implantation. We demonstrate that spectroscopic imaging is crucial for understanding the formation and migration of defects such as NV centers and radiation induced defects. In addition, we show that the native defects in the diamond sample play an important role in determining the optical properties of the diamond post irradiation. A knowledge of these centres is important for optical device applications such as waveguiding.

## O33 MEASUREMENT OF D2 AMOUNT IN ICF TARGET BY PROTON-DEUTERIUM SCATTERING METHOD

Hailei Zhang<sup>1</sup>, Wei Zhang<sup>1</sup>, Xuemei Zhang<sup>1</sup>, Na Guo<sup>1</sup>, Yajing Zhang<sup>1</sup>, Dangzhong Gao<sup>2</sup>, Qi Wang<sup>2</sup>, Xiaojun Ma<sup>2</sup> and Hao Shen<sup>1</sup>

<sup>1</sup>Institute of Modern Physics, Applied Ion Beam Physics Laboratory, Fudan University, Shanghai 200433, China <sup>2</sup>Research Center of Laser Fusion, China Academy of Engineering Physics, Mianyang 621900, China

A proton-deuterium scattering (p-D scattering) coincident measurement system was established in Fudan University to measure  $D_2$  amount in Inertial Confinement Fusion (ICF) targets. ICF targets with diameter of a few hundred microns was scanned with proton microbeam. Recoiled D atoms and scattered proton atoms were detected by two surface barrier detectors which were placed symmetrically at 53° to the beam direction. A piece of deuterated polyethylene thin film (D film) was prepared as a standard sample. By comparing the coincidence energy spectra of ICF targets and D film, the  $D_2$  amount in the ICF target was calculated. The pathlength effect during the beam scanning was analyzed. The measurement results were compared with results from other methods and the uncertainty was estimated. The irradiated damage to the target during the measurement was also analyzed.

# **SESSION 7**

# **MeV SIMS**

Chair: Hao Shen

#### **I09 ADSORPTION OF LIQUIDS MEASURED WITH MeV-SIMS IN AMBIENT**

Jiro Matsuo<sup>1,2</sup>, Toshio Seki<sup>1,2</sup> and Takaaki Aoki<sup>1,2</sup>

<sup>1</sup>Quantum Science and Engineering Center, Kyoto University, Gokasho, Uji, Kyoto 611-0011, Japan <sup>2</sup>SENTAN, Japan Science and Technology Agency (JST), Chiyoda, Tokyo 102-0075, Japan

Secondary particle emission under ion impact provides a unique opportunity not only for insights into ion collisions, but also for material analysis. A secondary ion mass spectrometry technique with swift heavy ion beams has been developed. Secondary molecular ion emission was significantly enhanced with swift heavy ion beams. Those molecular ions provide unique information of "soft-materials" such as polymer, organic semiconductors and biological materials. This technique, now known as "MeV-SIMS", opens new possibilities for investigating chemical composition and structure, as well as for molecular image. We have demonstrated molecular imaging of a rat brain and of a single animal cell [1]. Furthermore, swift heavy ion beams have a high transmission capability in matter, which allows their use for the analysis of volatile samples such as liquids, solid-liquid interfaces and wet samples under atmospheric pressure.

Specially designed SIMS system has been constructed in Kyoto University for "Ambient SIMS" measurements [2]. As shown in Figure 1, 6 MeV Cu ion beam was introduced into a target chamber that was kept at ambient conditions (100,000 Pa) with varying humidity levels (0 - 100%). Secondary molecular ions emitted from liquid surface, were extracted through a small orifice (100  $\mu$ m diameter) into a differentially pumped chamber. SIMS spectra were measured with orthogonal acceleration time-of-flight (oa-TOF) mass spectrometer.

SIMS spectra of a Si wafer under wet and dry conditions in an ambient are shown in Figure 2. Secondary ions with m/z of 19 ( $H_3O^+$ ) and 32 ( $O_2^+$ ) were observed in dry condition. Those ions were ionized at gas phase. However, water clusters indicating a liquid water layer on the Si wafer were found in the wet condition. This SIMS spectrum is very similar to that from liquid droplet on Si wafer. Those results indicate that very thin liquid layer was formed on the Si wafer in high humidity condition.



Figure 1. Schematic diagram of the Ambient-SIMS system.



Figure 2. SIMS spectra under dry and wet.

Recent progress on this technique will be presented and discussed along with possible applications for liquid analysis.

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#### O34 CHEMICAL IMAGING OF THE HEALTHY AND DIABETIC MOUSE LIVER USING MeV TOF-SIMS

Zdravko Siketić, Iva Bogdanović Radović, Marko Barac, Marijana Popović Hadžija and Mirko Hadžija

Ruđer Bošković Institute, Bijenička cesta 54, 10000 Zagreb, Croatia

Time-of-flight Secondary ion mass spectrometry (TOF-SIMS) is very well-known surface analysis technique used for the mass identification and 2D chemical imaging of the organic and inorganic materials. It has been recently demonstrated that the use of MeV primary ion beams significantly enhances secondary molecular yield of the heavy molecules (m > 500 Da), if it is compared with the ordinary keV TOF-SIMS experiments. MeV ions interact with the surface layers of the sample mostly through the electronic stopping, causing desorption of intact molecular ions making determination of sample molecular composition much easier. Interaction of MeV incident ion and sample, mostly through the electronic stopping power, is also a reason why secondary molecular ion yield is very high, which is a crucial prerequisite to enable molecular imaging of sensitive biological samples at the micrometre level.

In this work, the application of the MeV TOF-SIMS technique will be demonstrated on cryo-sections of healthy and diabetic mice liver. For inducing diabetes NOD mice will be fed with high (58%) fat diet (HFD) and compare with mice on the diet with standard fat content (11.4%). Fasting glucose level will be measured weakly by glucometer in one blood drop taken from the tail. Glucose level more than 7 mmol/L will be taken as diabetes. MeV-SIMS imaging cryo-sections will be performed in order to analyse a possible link of increased blood glucose and lipid composition and concentration in the liver.

#### O35 MASS SPECTROMETRY - ION BEAM ANALYSIS: A NEW TOOL FOR MOLECULAR AND ELEMENTAL SPECIATION?

Catia Costa<sup>1</sup>, Josephine Bunch<sup>2</sup>, Richard Goodwin<sup>3</sup>, Roger P Webb<sup>1</sup>, Vladimir Palitsin<sup>1</sup>, Janella de Jesus<sup>1,4</sup> and Melanie J Bailey<sup>4</sup>

<sup>1</sup>Surrey Ion Beam Centre, University of Surrey, Guildford, UK
<sup>2</sup>National Physical Laboratory, Teddington, UK
<sup>3</sup>AstraZeneca, Cambridge, UK
<sup>4</sup>Department of Chemistry, University of Surrey, Guildford, UK

Elemental imaging is typically carried out independently from molecular imaging. This is because no single technique can spatially map trace elements, intact proteins and metabolites. This limits the probative value of many investigations, where co-locating elemental and molecular markers can be a key requirement. We are therefore exploring the use of spatially resolved mass spectrometry (MS) methods, and MeV ion beam analysis (IBA), which provides micron-scale elemental imaging in air. As a first step, we report on the interoperability of the techniques. In this presentation, we explore the potential for (a) radiation damage (caused by performing ion beam imaging first) on molecular signatures and (b) erosion of elemental signatures (caused performing molecular imaging first) in spiked tissue homogenates.

Homogenised liver tissue mimetics were prepared as reported previously [1] and spiked with erlotinib, olanzapine, moxifloxacin and terfenadine. Extraction of molecular species from the tissues was carried out using direct analyte probed nanoextraction (DAPNe) [2]. This comprises extraction of analytes from a selected area of sample using a nanospray emitter (1  $\mu$ m internal diameter) filled with 90:10 acetonitrile:water. The solvent is injected onto the sample surface and then re-aspirated back into capillary tip. Extracted analytes are then analysed using nanospray ionisation followed by mass spectrometry using a Thermo Q-Exactive Plus Orbitrap.

An optimised protocol for the extraction of all spiked drugs using DAPNe was determined. This provided for the successful extraction of erlotinib, olanzapine and terfenadine and moxifloxacin at all spiked concentrations.

In addition to the detection of the spiked drugs, DAPNe-MS analysis also allowed the extraction of nine phosphatidylcholine (PC) lipids, however exact structural information for each of the extracted lipids cannot be confirmed due to short detection time, hindering further MS/MS experiments. This shows the capacity for detection of tissue endogenous substances that can be used to monitor any damage/changes to the sample as a result of the elemental and molecular measurements. The effect of extraction on these samples was also observed using optical microscopy, where the areas sampled by the nanoprobe are visible.

Next steps will include further investigation into the effects of beam irradiation prior to imaging MS measurements using the target substances identified above, as well as statistical evaluation of the dataset. Concerning the IBA measurements, we will attempt to find a trade-off between high trace element sensitivity, together with reduced damage to organic molecules for sample preservation purposes.

The end goal will be to establish a sequence of analysis using IBA and imaging mass spectrometry that will yield the highest level of molecular and elemental information under ambient pressure.

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# **SESSION 8**

# **BEAM OPTICS**

Chair: Istvan Rajta

## O36 CHARACTERISTICS OF FOCUS CONDITIONS FOR A MAGNETIC QUADRUPOLE TRIPLET LENS

Wanwisa Sudprasert<sup>1</sup>, Ridthee Meesat<sup>1</sup> and Harry J Whitlow<sup>2,3</sup>

<sup>1</sup>Department of Applied Radiation and Isotopes, Faculty of Science, Kasetsart University, Bangkok 10900, Thailand

<sup>2</sup>Department of Chemistry, Faculty of Science, Kasetsart University, Bangkok 10900, Thailand

<sup>3</sup>Louisiana Accelerator Center and Department of Physics, University of Louisiana at Lafayette, Lafayette, LA 70504, USA

The conditions for focussing an ion beam using a magnetic quadrupole lens elements to a conjugate focus depends on the number of lens elements, their separation, geometry and poletip fields. In practice the number of degrees of freedom are limited and with the consequence that for a fixed geometry, a small number of different conditions for focus exist. For example, for the commonly used Converging-diverging-converging (CDC) Oxford triplet configuration with two current supplies, two combinations of excitation current for focus exist.

Different microprobe applications such as high resolution microscopy, PIXE and RBS imaging, microchanneling, high fluence irradiation and the production of large area uniform ion fluxes have different requirement on the characteristics of the focused beams. In order to investigate how these conditions can be met, we have employed the WINTRAX code to investigate the focus conditions of the imaging microbeam at the Louisiana Accelerator Center (a generic Oxford triplet CDC geometry) for the cases where (i) one lens power supply feeds the first two C-D lens element pair and the second power supply the final C element, (ii) one lens power supply feeds the first C element and the second power supply the final DC elements coupled in series, and (iii) the three elements were each fed by an individual power supply. The metrics of comparison were: the beam spot size, the degree of collimation, transmission factor, scan sensitivity and magnification. These were subsequently verified using the imaging microprobe at the Louisiana Accelerator Center with a 2 MeV proton beam.

#### O37 DEVELOPMENT OF A SEPARATED MAGNETIC QUADRUPOLE DOUBLET FOCUSING SYSTEM AT THE UNIVERSITY OF NORTH TEXAS

Bibhudutta Rout, Todd A Byers, Jack E Manuel and Gary A Glass

Ion Beam Modification and Analysis Laboratory, Department of Physics, University of North Texas, Denton, Texas 76203, USA

We are currently developing a high throughput ion beam focusing system utilizing two pairs of magnetic quadrupole doublets lenses separated at a distance. The system is being built in a microprobe beamline with an existing Louisiana magnetic doublet (LMD) lens associated with a 3 MV single ended (NEC, 9SH) accelerator. The existing doublet system has a demagnification of ~20 × 60 (in X and Y direction respectively) for a working distance of 18 cm. The new system is a further optimization of the focusing system with addition of another pair of LMD system separated by ~1.25 m distance. The new system is simulated to have an increased orthomorphic demagnification of 120 × 120, thereby increasing the beam throughput by an order of magnitude.

We will be demonstrating theoretical simulation of ultimate spatial resolution for different distance of separation of the doublets as well high excitation separated triplet mode. We will be presenting experimental results demonstrating the high demagnification and high current density of the new microprobe system being developed at the University of North Texas.

### O38 EFFECTS OF STRAY MAGNETIC FIELD ON THE PERFORMANCE OF FOCUSED ION MICROBEAM

Yanxin Dou<sup>1</sup>, Jianli Liu<sup>2</sup>, Donghua Pan<sup>2</sup>, Zhiyin Sun<sup>2</sup> and Liyi Li<sup>1</sup>

<sup>1</sup>School of Electrical Engineering and Automation, Harbin Institute of Technology, Harbin 150001, China

<sup>2</sup>Research Center of Basic Space Science, Harbin Institute of Technology, Harbin 150001, China

The ion microbeam employing MeV ions has many applications in the fields of elemental analysis, micro/nano processing, ion implantation, irradiation effects of spacecraft, biological irradiation effects, agricultural breeding and so on. The key aims are to reduce the spot size and to improve the stability of spot position. After more than 40 years of development, a variety of well-known factors degrade the performance of ion microbeam with stray inhomogenous time varying magnetic fields being one of the key limiting factors [1-5]. This paper presents a new study of this important problem with the aim of optimizing the approaches to stray field mitigation. The approach applied here was to employ the WinTRAX beam optical software package to model the microbeam spot size and position on the sample for three scenarios chosen to investigate the important effects. These are: (1) the uniform stray magnetic field model, (2) the non-uniform stray magnetic field model, and (3) the uniform stray magnetic field with the anti-divergence aperture model. In each scenario the variation of spot size is due to the effective change of aberrations in the microbeam caused by the stray magnetic field, which is related to the magnitude and location of stray magnetic field. The variation of spot size is due to the change of aberrations of an ion microbeam caused by the stray magnetic field, which is related to the magnitude and location of stray magnetic field. The antidivergence aperture can also influence the spot size under the condition of stray magnetic field. The variation of spot position is also related to the magnitude and location of stray magnetic field, but is not affected by the anti-divergence aperture. The change of spot position is due to the deflection of stray magnetic field on ions. These results have practical significances for how to shield the stray magnetic field to enhance the performance of an ion microbeam. And the method using the WinTRAX to simulate the stray magnetic field in an ion microbeam can estimate the influence once the stray magnetic field is measured in the ion microbeam laboratory.

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## O39 THE BEAM OPTICS OF SPACED TRIPLET CONFIGURATIONS OF MAGNETIC QUADRUPOLES FOR HIGH ENERGY ION MICROBEAM APPLICATIONS

Geoffrey W Grime and Vladimir Palitsin

Surrey Ion Beam Centre, University of Surrey, Guildford, GU2 7XH, UK

The "Oxford Triplet" configuration of quadrupole probe forming lens has been used successfully for many years in high energy ion microbeam systems due to its combination of relatively high demagnification and moderate aberrations.

Recently however, several groups have been experimenting with increasing the distance between the first two lenses to form a 'spaced triplet' configuration. This configuration offers higher demagnification and an overall improvement in optimum acceptance for a given spot size as well as an increase in the maximum ion rigidity capability. Less well known is that the sensitivity to power supply ripple, quadrupole alignment and beam energy fluctuations increases significantly and there is a reduction in the maximum available scanning area using pre-lens deflection.

Using the WinTRAX raytracing package [1] this paper explores the dependence of these performance parameters on the spacing between the first two lenses and concludes that useful improvement in microbeam performance can be obtained using the spaced triplet.

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## POSTERS

#### P01 HERITAGE SCIENCE APPLICATIONS AT THE NUCLEAR MICROPROBE OF MTA ATOMKI - STUDIES ON EARLY MEDIEVAL BELT SETS

Ákos Csepregi<sup>1</sup>, Bendeguz Tobias<sup>2</sup>, Zsófia Kertész<sup>1</sup>, Anikó Angyal<sup>1</sup>, Enikő Papp<sup>1</sup>, Zoltán Szoboszlai<sup>1</sup>, Zsófia Török<sup>1</sup> and Zita Szikszai<sup>1</sup>

<sup>1</sup>Institute for Nuclear Research, Hungarian Academy of Sciences, H-4001 Debrecen, PO Box 51, Hungary <sup>2</sup>Institute of Archaeology, University of Innsbruck, A-6020 Innsbruck, Langer Weg 11, Austria

Analysis of cultural and natural heritage objects has been performed at the nuclear microprobe of MTA ATOMKI since its installation but in the recent years our activity in this field has increased significantly thanks to the participation first in the CHARISMA (Cultural Heritage Advanced Research Infrastructures: Synergy for a Multidisciplinary Approach to Conservation/Restoration, 2009-2014) EU FP7 project and now in the IPERION CH (Integrated Platform for the European Research Infrastructure ON Culture Heritage, 2015-2019) EU H2020 project. Performing ion beam analysis of cultural and natural heritage objects is not just a service but genuine collaboration with archeologists, conservators, curators. As an example, we present a study made on Early Medieval belt sets, of the so-called Bieringen type (7th century AD).

Around 600 AD, a new status symbol of multipart belt sets originating from the Mediterranean area prevailed in Central Europe. In the 7th century AD, only 20% of those belt sets were cast using a copper alloy, while most of the others were made of iron. Among the copper-alloy belts, those of the Bieringen type are the most frequently appearing and the most widespread, from Switzerland to Hungary and from Central Germany to Southern Italy. This special belt is characterized by a single strap which, like a tail-like extension, hangs down from the rear portion of the belt. The enormous benefit of bronze fittings in comparison to iron ones is that bronze and especially leaded bronze is less susceptible to corrosion - whereas the production process from the raw material to the finished product is much more complex and requires high technological skills. Thus, the basic requirements to produce such high-quality and standardized belt fittings were choosing the appropriate raw materials and possessing a high level of technological competence. In the current study the composition of alloys was determined on trace element level. Although bronze objects are often measured on the polished surface, in this case we had drilled material from the inside of the objects which necessitated the use of a microprobe. Differences based on geographical regions, and indications for the possible raw materials are discussed in the presentation.

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#### P02 THE NEW SUMY NUCLEAR MICROPROBE WITH HIGH RESOLUTION PROBE-FORMING SYSTEM

A G Ponomarev<sup>1</sup>, V A Rebrov<sup>1</sup>, S V Kolinko<sup>1</sup>, A S Lapin<sup>1</sup>, V F Salivon<sup>1</sup> and A A Ponomarov<sup>2</sup>

<sup>1</sup>Institute of Applied Physics, National Academy of Sciences of Ukraine, 40030 Sumy, Ukraine

<sup>2</sup>Institute of Modern Physics, Chinese Academy of Sciences, Lanzhou, China

The Sumy nuclear microprobe [1] was started operation in 2008 for microanalysis application ( $\mu$ -PIXE,  $\mu$ -RBS,  $\mu$ -ERDA). It has resolution of about 2  $\mu$ m for 100 pA beam current and its probeforming system on the base of separated Russian quadruplet has demagnification of 23. A new microprobe development is motivated by increasing of microprobe techniques especially for a proton beam writing application in a fabrication of X-ray optic elements. These techniques demand a higher resolution and therefore a higher demagnification is needed. The new Sumy nuclear microprobe has the same beam line with the present one and uses the same object and aperture collimators. The new probe-forming system based on five magnetic quadrupole lenses of integrating design with four separated power supplies [2] is allocated after the target chamber of the present microprobe. The electrostatic scanning system of a dog-leg type is located before the triplet [3] of final focusing. The new target chamber of rectangular design has an optical microscope, secondary electron detector, target stage and possibility to equip additional devises. The present report describes the status of the new Sumy nuclear microprobe, its beam optics and design.

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#### P03 OPTIMIZATION OF PROBE-FORMING SYSTEM OF HIGH ENERGY HEAVY ION MICROBEAM IN LANZHOU FOR LOW CURRENT MODE

Artem Ponomarov<sup>1</sup>, Guanghua Du<sup>1</sup>, Jinlong Guo<sup>1</sup>, Lina Sheng<sup>1</sup>, Alexander Ponomarev<sup>2</sup>, Wenjing Liu<sup>1</sup>, Ruqun Wu<sup>1</sup> and Yaning Li<sup>1</sup>

<sup>1</sup>Institute of Modern Physics, Chinese Academy of Sciences, Lanzhou, China <sup>2</sup>Institute of Applied Physics, National Academy of Sciences of Ukraine, Sumy, Ukraine

High energy heavy ion (HEHI) beams of tens to hundred MeV/u are widely used for studies of a spatial radiation response or local radiation effect in materials and biological samples. Additional advantages are gained from a focusing of HEHI beam by a microbeam facility. Its spatial resolution depends on both beam and system parameters and processes of their interaction. Therefore, for a high microbeam resolution its probe-forming system should be optimized for specific applications considering the beam parameters.

Microbeam at the Institute of Modern Physics (IMP) in Lanzhou mostly utilizes <sup>12</sup>C<sup>+6</sup> and <sup>86</sup>Kr<sup>+26</sup> ions accelerated by two sector cyclotrons to the energy up to 80 MeV/u and 25 MeV/u respectively [1]. The original system was designed for a high current density irradiation. However, in contrast to conventional microprobes the beam from the cyclotron has relatively large energy spread and HEHIs substantially scatter at the edges of the microbeam slits. Therefore, increase in the beam current at the target results in significant increase in the spot size due to chromatic and spherical aberrations. Therefore, Lanzhou microbeam is successfully implemented mainly in a single-ion mode.

In order to apply the microbeam in low-current mode its modernization was carried out as presented in this work. The new object and angular slits, which provide an optimal beam collimation for a maximum system acceptance has been manufactured and installed. The final triplet of magnetic quadrupole lenses has been upgraded with the aim of precision independent alignment for each singlet. Multipole components of these lenses were measured using a method of Hall probe rotation [2]. Their values were taken into account during the optimization of the system with the new geometry by means of a beam optics simulation. Acceptance of the system which simultaneously considers chromatic and spherical aberrations was the main optimization criterion. C and Kr beams with their maximum energy and variable phase coordinates at the entrance to the object slit were taken during the simulation. Parameters of the probe-forming system corresponded to its highest acceptance for a given resolution and their limitations were obtained from the optimization and validated by different beam optics software.

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#### P04 STIGMATIC IMAGING OF SECONDARY IONS IN MeV-SIMS BY A LINEAR TIME-OF-FLIGHT MASS SPECTROMETER AND A TIMEPIX DETECTOR

Boštjan Jenčič<sup>1</sup>, Luka Šepec<sup>1</sup>, Primož Vavpetič<sup>1</sup>, Mitja Kelemen<sup>1</sup>, Zdravko Rupnik<sup>1</sup>, Matjaž Vencelj<sup>1</sup>, Katarina Vogel-Mikuš<sup>1,2</sup>, Nina Ogrinc Potočnik<sup>3</sup>, Shane Ellis<sup>3</sup>, Ron M A Heeren<sup>3</sup> and Primož Pelicon<sup>1</sup>

<sup>1</sup>Jožef Stefan Institute, Jamova 39, SI-1000 Ljubljana, Slovenia <sup>2</sup>University of Ljubljana, Biotechnical Faculty, Department of Biology, Večna pot 11, SI-1000 Ljubljana, Slovenia <sup>3</sup>M4I Institute, Maastricht University, 6229 ER Maastricht, The Netherlands

Secondary ion mass spectrometry (SIMS), based on primary ions within the MeV energy domain, also known as MeV-SIMS [1], is a subject of increasing scientific interest. The main drive for the interest in the development of MeV-SIMS is the ability to desorb high yields of large non-fragmented organic molecular ions from the sample surface. This makes MeV-SIMS particulary useful in imaging of biological tissues.

Imaging methods based on scanning a focused primary ion beam are associated with demanding focusing of the heavy energetic ions. As an alternative, stigmatic imaging mode has been studied here, applying point-to-point imaging characteristics of secondary ions in the linear Time-of-Flight mass spectrometer. Stigmatic imaging [2,3], process of the secondary ions, originating from the selected location at the sample surface, through the electrostatic lens and the drift space of a linear Time-of-Flight mass spectrometer, to the final impact on a fast position sensitive detector TimePix [4,5], is studied as a novel approach in mass spectrometry imaging.

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## P05 AN INTEGRATED MIDAS AND LABVIEW BASED DATA ACQUISITION, SCANNING AND STAGE CONTROL SYSTEM FOR THE NUCLEAR MICROPROBE AT THE MATERIALS RESEARCH DEPARTMENT, ITHEMBA LABORATORIES

**Caswell Pieters** 

Instrumentation and Information Technology Department, iTHEMBA Laboratories, PO Box 722, Somerset West 7129, South Africa; National Research Foundation, South Africa

Due to the obsolescence of VAX workstations running the XSYS general purpose data acquisition package for PIXE, PIGE, RBS and mapping, the decision was made to replace these and connected components with modern software frameworks and hardware. A system was designed to integrate a multi-channel software frontend and online analyzers based on the MIDAS data acquisition framework developed at PSI/Triumf, along with PCI, PXI and C series National Instruments modules for both computer and hardware joystick stage control, FPGA-generated time critical scanning, beam blanking, ADC gating, total X-ray counting and scan coordinate tracking. A graphical user interface developed in LabVIEW communicates via calls to an online database that contains all the information needed to run and control an experiment. This is done by remote procedure calls from a Windows library or by a dedicated http server that can be accessed in a web interface, with pages customizable through a JavaScript library. Data files are synced using ownCloud and are immediately available for offline analysis on connected client PC's, as well as for the transfer of GeoPIXE dmm files for real-time image projection using the Dynamic Analysis method [1]. A Stanford Research Systems low-noise current preamplifier is used along with a Quantum Detectors 1 MHz V2F module connected to a scaler for capturing scanning charge maps. List mode files are created with metadata stored in a JSON header, as well as ROOT-defined energy spectra and maps for online and offline The configuration and integration of these hardware and software components is viewing. discussed, along with preliminary results.

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#### P06 DEVELOPMENT OF NANO APERTURES FOR ION BEAM COLLIMATION

Clemens Scheuner<sup>1</sup>, Nicole Raatz<sup>1,2</sup>, Paul Räcke<sup>1,2</sup>, Steffen Jankuhn<sup>1</sup>, Sébastien Pezzagna<sup>1</sup>, Christina Trautmann<sup>3,4</sup> and Jan Meijer<sup>1,2</sup>

<sup>1</sup>Department of Nuclear Solid State Physics, Universität Leipzig, Germany <sup>2</sup>Leibniz Joint Lab Single Ion Implantation, Germany <sup>3</sup>GSI Helmholtzzentrum für Schwerionenforschung, Germany <sup>4</sup>Technische Universität Darmstadt, Germany

Small beam-defining apertures are very important for focusing systems in ion beam technology. Objects with diameters below a micrometre can be demagnified down to a few nanometres. The use of nanocollimation is a low cost alternative to expensive microprobe systems and applicable for low current applications like cell irradiation or single ion implantation.

We will give an overview of our work in investigating nanocollimators for ion beams in the lower keV range. It will be presented how to create nanocollimators in different materials with either focused-ion-beam (FIB) sputtering or irradiation with swift heavy ions and subsequent track etching.

Furthermore, the interaction between ions and pore walls have been studied leading to an unexpected surplus of transmitted ions due to small angle scattering [1]. To gain information about the properties of the transmitted ion beam measurements and simulations have been carried out.

Additionally, we found that the open pore area gets reduced due to the ion bombardment under certain circumstances. This effect was examined with keV ions and keV electrons for different materials resulting in the development of a model that describes this process. It has been found that this process is temperature dependent and that it can be reversed for low temperatures, enabling the creation of nanopores down to the size of a few nanometres. Such small apertures may be important for biological applications or be applicable for the use as point like ion beam defining collimators for FIB technology with different ion sources.



SEM micrographs showing nano apertures created by FIB in SiN (left) and track etching in Mica (right).

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## P07 ANALYSIS OF SPICE MICROBEAM PROFILE USING FLUORESCENT NUCLEAR TRACK DETECTOR (FNTD)

D Ohsawa<sup>1,2</sup>, Y Furusawa<sup>1,3</sup>, A Kobayashi<sup>1,4</sup>, M Oikawa<sup>4</sup> and T Konishi<sup>1,3</sup>

<sup>1</sup>SPICE-BIO Research Core, NIRS-International Open Laboratory, NIRS-QST, Chiba, Japan

<sup>2</sup>Graduate School of Medical and Pharmaceutical Sciences, Chiba University, Chiba, Japan

<sup>3</sup>Department of Basic Medical Sciences for Radiation Damages, NIRS-QST, Chiba, Japan

<sup>4</sup>Department of Accelerator Physics, NIRS-QST, Chiba, Japan

Single Particle Irradiation system to Cell (SPICE) facility at the National Institute of Radiological Sciences (NIRS) provides 3.4 MeV proton microbeam, where the beam is focused with a magnetic quadrupole triplet lens. Over the years, we have demonstrated radio-biological studies using SPICE, however, realized that biological effect can be affected by the highly localized dose distribution of the microbeam and results may differ from that of broadbeam irradiation. Previously, we have reported that the beam size of our proton microbeam was approximately 2  $\mu$ m; the proton traversal was identified as etch pits produced on plastic nuclear track detectors, CR-39. However, this widely used method cannot distinguish individual proton tracks of highly focused microbeam due to the overlapping of etch pits, which was major limitation for beam profile analysis as well as estimation of localized dose distribution in the cells. To overcome this limitation, we used fluorescent nuclear track detectors (FNTDs; Al<sub>2</sub>O<sub>3</sub>: C, Mg) to investigate the beam profile and its proton-number dependence (7 ~ 1,000 protons/position).

Detail analysis of depth profile of the FNTD fluorescent images with a confocal laser scanning microscope clearly demonstrated that the FNTD has better position resolution and detection sensitivity compared to CR-39. The overall beam size ( $\pm 2\sigma,\sigma$ : standard deviation) at the entrance surface of the FNTD was measured to be in the range of 2.0 (1.5) to 3.0 (2.2) µm in the X (Y) axis directions depending on the number of protons/position, suggesting that out-focused fraction of the incident protons contribute the broadening of the beam size. Localized dose distribution in cell nucleus was also numerically simulated using beam profile obtained by irradiating 100 ~ 500 protons/position on the FNTD; in this simulation, radial dose distribution of single proton track in water was calculated with existing amorphous track structure models. On the other hand, targeted nuclei of WI-38 human normal cells were irradiated by the same number of protons/position and subsequently fixed and immunofluorescent stained against  $\gamma$ -H2AX, which is an antibody to detect DNA double strand breaks (DSB) in cells and widely used as a maker for it. The size of localized DSB identified as fluorescent spots of  $\gamma$ -H2AX was observed to be rather larger than that of the dose distribution, indicating induction of DSB in the targeted cell nuclei was enhanced beyond the dose distribution.

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#### P08 TRACE ELEMENT COMPOSITION OF IN-SITU ISOTOPICALLY ENRICHED SILICON

S G Robson<sup>1,2</sup>, D Holmes<sup>1,2</sup>, J S Laird<sup>3</sup>, A M Jakob<sup>1,2</sup>, B C Johnson<sup>1,2</sup>, J C McCallum<sup>1,2</sup>, H Fiedler<sup>4</sup>, P Gupta<sup>4</sup>, A Markwitz<sup>4</sup>, C G Ryan<sup>3</sup> and D N Jamieson<sup>1,2</sup>

<sup>1</sup>School of Physics, University of Melbourne, Melbourne, VIC 3010, Australia <sup>2</sup>ARC Centre of Excellence for Quantum Computation and Communication Technology (CQC2T) <sup>3</sup>CSIRO Earth Sciences and Resource Engineering, Kensington, WA 6151, Australia

<sup>4</sup>GNS Science - Te Pu Ao, National Isotope Centre, PO Box 31-312, Lower Hutt, New Zealand

Isotopically enriched silicon epitaxial layers, in which the nuclear spin I =  $1/2^{29}$ Si isotope has been depleted below 100 ppm, on natural silicon (<sup>n</sup>Si) wafers have been used to build quantum computer devices in which <sup>31</sup>P donor atoms have nuclear spin quantum bit (qubit) coherence times above 30 s [1,2]. Depletion is necessary to avoid magnetic dipole coupling between the <sup>29</sup>Si and <sup>31</sup>P nuclear magnetic moments that induce decoherence and limit the lifetime of the desirable long-lived quantum coherent states needed for large scale quantum computing. However, wafer-scale depletion is not necessary because the dipole coupling has a range of ~20 nm and hence <sup>31</sup>P qubits surrounded by volume of depleted silicon may be adequate for a robust device. We have developed a method of localised <sup>29</sup>Si depletion by ion implantation that could be deployed in a standard semiconductor fabrication facility to perform in-situ depletion. A critical issue with this process is the possible presence of contaminants such as Fe. Secondary ion mass spectrometry indicates that our novel process works. However analysis of localised areas in which there are spatial variations of depletion on the micron scale, along with possible haloes of contaminants including neutrals, is more challenging. Here we use the CSIRO-MARC nuclear microprobe system fitted with the MAIA detector [3] to obtain trace element images with proton induced X-ray emission in a localised <sup>29</sup>Si and <sup>30</sup>Si depleted area within a high purity <sup>n</sup>Si wafer. The MAIA detector combines a large solidangle annular energy-dispersive 384 detector array, stage encoder and flux counter inputs and a dedicated FPGA-based real-time event processor with embedded spectral deconvolution providing trace element maps of high sensitivity. This work is the first application of the MAIA detector to a problem of quantum device engineering.

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### P09 MICROPATTERNING OF POLYTETRAFLUOROETHYLENE (PTFE) BY PROTON BEAM WRITING

Deiverti de Vila Bauer<sup>1</sup>, Cláudia Telles de Souza<sup>2</sup>, Lívio Amaral<sup>1</sup> and Johnny Ferraz Dias<sup>1</sup>

<sup>1</sup>Ion Implantation Laboratory, Institute of Physics, Universidade Federal do Rio Grande do Sul, Av Bento Gonçalves 9500, CP 15051, CEP 91501-970, Porto Alegre, RS, Brazil

<sup>2</sup>Laboratory of Environmental Analytical Chemistry and Chemical Oil, Department of Inorganic Chemistry, Institute of Chemistry, Universidade Federal do Rio Grande do Sul, Av Bento Gonçalves 9500, CEP 91501-970, Porto Alegre, RS, Brazil

Polytetrafluoroethylene (PTFE) is a fluorinated polymer widely used by the chemical industry. This polymer, known as teflon<sup>®</sup>, is remarkably resistant to chemical agents, exhibiting inert behavior and capable of withstanding high temperatures. PTFE is the source of several innovations in the electronics, photonics and biomedicine industries due to its hydrophobic properties and biocompatibility. However, the attempt to explore ways of manufacturing microstructures through a lithographic process still remains a challenge.

The patterning process of materials through proton microprobe, known as proton beam writing, is a technique that allows the development of 2D and 3D microstructures with high aspect ratio. In the last decade, this technique has been extensively explored and has proved to be an effective polymeric patterning process. In this process, polymeric materials are irradiated with a focused proton beam, weakening and damaging their chemical structure, which is subsequently removed by chemical etching. Due to the inert properties of teflon<sup>®</sup>, this process becomes a challenge to create microstructures through the technique. The present work deals with the patterning of PTFE through ion microprobe techniques.

The aim of this work is to study the behavior of teflon<sup>®</sup> when submitted to the patterning process and subsequent chemical etching. Preliminary results have shown the feasibility of carrying out the micropatterning in teflon<sup>®</sup> through the proton beam writing. The samples were characterized by scanning electron microscopy. The use of ultrasound as an alternative to chemical etching is discussed.

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#### P10 DATA ACQUISITION AND CONTROL SYSTEM FOR AN EVOLVING NUCLEAR MICROPROBE

Donny Cosic<sup>1</sup>, Mladen Bogovac<sup>2</sup> and Milko Jakšić<sup>1</sup>

<sup>1</sup>Laboratory for Ion Beam Interactions, Ruđer Bošković Institute, Bijenička cesta 54, 10000 Zagreb, Croatia <sup>2</sup>Nuclear Science and Instrumentation Laboratory, Division of Physical and Chemical Sciences, International Atomic Energy Agency, Friedenstrasse 1, 2444 Seibersdorf, Austria

The nuclear microprobe at the Ruđer Bošković Institute, Zagreb, is constantly evolving with new projects and experiments, which bring new requirements on the data acquisition and microbeam control systems. Since the early development of the SPECTOR data acquisition software [1] and its subsequent upgrades [2], significant changes in both hardware and software have been accomplished.

In this work we will present the most recent upgrades of the system. MeV SIMS experiments for example require the ability to bunch a continuous beam and steer it in the X-Y plane. Buncher provides a trigger for the TOF mass spectrometer in that case. For the channeling RBS and STIM experiments sample needs to be rotated over the two axes. Once it is positioned in channeling direction, patterned irradiation may be needed. Irradiation of specific areas of the samples can be also programmed for different cycles of beam-on and beam-off cycles.

Additionally, with the evolution of digital electronics, it is now possible and sometimes preferred to perform signal processing in the digital domain as oppose to analogue signal chains in NIM modules.

To meet this wide array of requirements, a data acquisition/control system based on a Xilinx Virtex 6 FPGA was developed which can evolve with the microprobe. The real time reprogrammable nature of the FPGA coupled with a modular design approach allow for the ADCs, processing algorithms and communication protocols to be interchanged and upgraded while keeping a constant user interface through the SPECTOR software package.

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#### P11 RUTHERFORD BACKSCATTERING SPECTROSCOPY/CHANNELLING FOR DAMAGE AND STRAIN MEASUREMENTS IN SEMICONDUCTORS

E Schneider<sup>1</sup>, J G England<sup>1</sup>, I P Marko<sup>2</sup>, S J Sweeney<sup>2</sup>, M Sharpe<sup>2</sup> and D Cox<sup>2</sup>

<sup>1</sup>Surrey Ion Beam Centre, University of Surrey, Guildford, UK <sup>2</sup>Advanced Technology Institute, University of Surrey, Guildford, UK

Ion channelling in Rutherford Backscattering Spectroscopy (RBS) is a well-established technique that has repeatedly been used for many decades to probe amorphisation, damage, strain and defects in crystalline materials. While new semiconductor materials are developed, it is important that we have the capabilities to fully understand their underlying structure and as devices become smaller, measurement techniques must have high depth resolution capability.

In an RBS measurement, directing ions along a channelling direction (an "aligned" orientation) in a perfect crystal results in a low yield of backscattered ions. However, when ions are directed through a channel of a damaged material, which therefore contains atoms out of alignment, the backscattered ion yield will be higher than that of an undamaged sample. By comparing aligned yields between virgin and damaged samples, the lattice damage of a semiconductor can be quantified and located by depth.

The position of dopant atoms in a material can also be characterised by comparing RBS signals from aligned and random (non-channelling) orientations; aligned spectra are sensitive to interstitial atoms, whereas random spectra are influenced by both interstitial and substitutional atoms. Dopant atoms may cause strain of the parent lattice which can be measured by scanning the incident ion beam over a range of angles; the range of angles over which the yield diminishes indicates the lattice constant of the material.

In this presentation we will illustrate the method of RBS channelling with several examples including measurements of Ne implant damage in Si and strain in InGaAsBi thin layers on InP and incorporation mechanism of large Bi atoms into InGaAs crystal lattice during epitaxial growth. The choice of primary beam energy and experimental geometry of the RBS measurements chosen to optimise the depth resolution for these shallow samples will be discussed. The RBS results will be compared with that of other techniques such as X-ray diffraction and electron backscatter diffraction.

#### P12 CAPABILITIES OF AN INDEPENDENT SINGLE ION HIT DETECTION SYSTEMS BASED ON EMISSION OF SECONDARY PARTICLES DURING THE IMPACT

Georgios Provatas, Marko Brajković and Milko Jakšić

Laboratory for Ion Beam Interactions, Ruđer Bošković Institute, Bijenička cesta 54, 10000 Zagreb, Croatia

The expanding variety of applications of single heavy ion irradiation that require high spatial resolution offered by a modern microprobe facilities, appoint the importance of a reliable ion hit detection system. The latter should provide a trigger signal to the control of the beam scanning module, irrespectively of the type and thickness of the material being irradiated, both in transmission and non-transmission samples. The most useful process that can be utilized for this purpose is the emission of secondary electrons and secondary ions that are being emitted from the sample surface following the ion impact.

The present work reports on the updates and the status of the developed single ion hit detection set-up, installed previously at the Zagreb ion microprobe. The set-up is based on the use of a channel electron multiplier (CEM) detector mounted on an interchangeable sample holder that is inserted into the chamber in a close geometry along with the sample to be irradiated. In our previous work [1], a 100% hit detection efficiency was achieved only for impacts of ions heavier than Si on metals. Therefore, further improvements of the set-up efficiency were needed. We have examined possibilities to increase the solid angle of the system, employment of alternative geometries and the use of different types and number of CEM detectors. The influence of the microbeam halo, which may also decrease ion positioning efficiency, has been also investigated. Examples of the production of ordered single ion tracks in different samples will be presented.

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## P13 INVESTIGATION OF EFFECTS OF NS, HIGH INTENSITY ION PULSES ON DEFECT CREATION USING A NUCLEAR MICROPROBE AND DEEP LEVEL TRANSIENT SPECTROSCOPY (DLTS)

G Vizkelethy<sup>1</sup>, E S Bielejec<sup>1</sup>, B A Aguirre<sup>1</sup>, P A Seidl<sup>2</sup> and T Schenkel<sup>2</sup>

<sup>1</sup>Sandia National Laboratories, Albuquerque, NM, USA <sup>2</sup>Lawrence Berkeley National Laboratory, Berkeley, CA, USA

Ion beam irradiation creates defects in semiconductors that affect the operation of microelectronic devices by reducing carrier lifetime. Generally, the number of defects is considered to be linearly proportional to the ion fluence. Svennson et al [1] showed that higher fluxes create relatively fewer vacancy-related defects in silicon than higher fluences. These observations were based on the DLTS measurements of silicon diodes. They attributed this behavior to the highly mobile silicon interstitial atoms. Short, high intensity pulses were used to do rapid thermal annealing in the past. Those experiment used channeling to determine the amount of damage. Channeling is sensitive to relatively high damage while some electronic properties of microelectronic devices are more sensitive, such as DLTS and Ion Beam Induced Current (IBIC).

We used the Lawrence Berkeley National Laboratory's NDCX-II accelerator and Sandia National Laboratories' (SNL) accelerator facilities to irradiate Si PIN, PN diodes and pnp bipolar junction transistors. The SNL accelerators could produce ion beam pulses from tens of ns to DC current at low flux. On the other hand, the NDCX-II produces ns pulses with fluxes up  $10^{19}$  ions/cm<sup>2</sup>/s [2]. The SNL nuclear microprobe was used to analyze the irradiated devices and determine their charge collection efficiency (CCE). In addition, DLTS measurements were performed to determine the types and number of defects. We found significant differences between the CCE, leakage current, and the number of defects of the devices irradiated with the same fluences but fluxes different by several orders of magnitude. A COMSOL [3] simulation of the irradiation indicated significant temperature increase in the devices irradiated at NDCX-II, which lasted around a  $\mu$ s. The exact interpretation of these effects in in progress.

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### P14 COMPARATIVE STUDY OF THE EFFECT OF SWIFT HEAVY ION IRRADIATION AT ROOM TEMPERATURE AND 500°C ON THE MIGRATION OF SILVER IMPLANTED INTO POLYCRYSTALLINE SIC

H A A Abdelbagi<sup>1,2</sup>, J B Malherbe<sup>1</sup>, V A Skuratov<sup>3</sup>, E G Njoroge<sup>1</sup>, S V Motloung<sup>4</sup>, M Mlambo<sup>1</sup> and T T Hlatshwayo<sup>1</sup>

<sup>1</sup>Physics Department, University of Pretoria, Pretoria 0002, South Africa
<sup>2</sup>Physics Department, Shendi University, Shendi, Sudan
<sup>3</sup>Joint Institute for Nuclear Research, Dubna, Russia
<sup>4</sup>Sefako Makgatho Health Science University, PO Box 94, Medunsa 0204, South Africa

Silver (Ag) ions of 360 keV were implanted into polycrystalline SiC to a fluence of  $2 \times 10^{16}$  cm<sup>-2</sup> at room temperature. Some of the as-implanted samples were irradiated with xenon (Xe) ions of 167 MeV to a fluence of  $5 \times 10^{13}$  cm<sup>-2</sup> at 500°C and  $8.3 \times 10^{14}$  cm<sup>-2</sup> at room temperature. Both the as-implanted and implanted then Xe irradiated samples were isochronally annealed in vacuum at temperatures ranging from 1100°C to 1500°C in steps of 100°C for five hours. The as-implanted, irradiated and annealed samples were characterized by Rutherford backscattering spectrometry (RBS), Raman spectroscopy and scanning electron microscopy (SEM). Implantation of Ag into SiC at room temperature resulted into the amorphization of the implanted SiC layer. Swift heavy ion (SHI) irradiation at room temperature (RT) and at 500°C caused some recrystallization of the amorphized layer. This recrystallization was more pronounced in the samples irradiated at 500°C. No diffusion of Silver was detected after SHI irradiation at both temperatures. Diffusion of the implanted Ag started at 1200°C in the un-irradiated samples. This diffusion was accompanied by loss of Ag at temperatures above 1400°C. No diffusion was observed in the samples irradiated at RT and 500°C up to 1500°C. Decomposition of implanted took place in the un-irradiated at 500°C.

#### P15 ASSEMBLING GOLD NANOPARTICLES BY DIELECTROPHORESIS WITH PIT ARRAYS ON PMMA FABRICATED BY PROTON BEAM WRITING

Taichi Shibuya<sup>1</sup>, Satoshi Uchida<sup>2</sup>, Yasuyuki Ishii<sup>3</sup> and Hiroyuki Nishikawa<sup>1</sup>

<sup>1</sup>Department of Electrical Engineering, Shibaura Institute of Technology, Tokyo, Japan

<sup>2</sup>Department of Electrical Engineering, Tokyo Metropolitan University, Tokyo, Japan <sup>3</sup>National Institutes for Quantum and Radiological Science and Technology (QST), Takasaki, Gunma, Japan

Assembled nanoparticles of metals and oxides have attracted much attention for sensing applications such as strain gauges and gas sensors [1]. Among various techniques for the assembly of nanoparticles, dielectrophoresis (DEP) was found to be a useful technique to manipulate and collect colloidal particles under application of unequal AC electric field.

We have successfully applied proton beam writing (PBW) to fabricate microstructures such as arrays of pillars and pits on dielectric materials, which were effective to modulate the electric field to apply DEP force on Ag nanowires and nanoparticles [2].

We performed PBW to fabricate pit arrays (5 × 5 to 10 × 10 arrays in 200  $\mu$ m area) in a PMMA layer on a conductive indium-tin-oxide (ITO)/PET film using a dedicated proton beam writer at SIT or a microbeam line with a single-ended accelerator at QST Takasaki. Colloidal gold particles of 250 nm in diameter were used for DEP experiments to fill the pit arrays with the nanoparticles. We investigated effects of the dimension of pit arrays and the frequencies of applied AC voltage on the fill factor of gold nanoparticles to each pit and the distribution over the whole arrays.

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- [2] R Kataoka, H Tokita, S Uchida, R Sano, H Nishikawa, J Phys: Conf Ser, Vol 646 (2015) 012005.
## P16 FIRST RESOLUTION TEST RESULTS OF THE ATOMKI NUCLEAR MICROPROBE

I Rajta<sup>1</sup>, G U L Nagy<sup>1</sup>, I Vajda<sup>1</sup>, S Z Szilasi<sup>1</sup>, G W Grime<sup>2</sup> and F Watt<sup>2</sup>

<sup>1</sup>MTA ATOMKI, Debrecen, Hungary <sup>2</sup>Oxford Microbeams Ltd, UK

In this work we report about the layout, assembly and the first resolution tests of the nuclear nanoprobe at MTA ATOMKI, Debrecen, Hungary. We also present WinTRAX calculations, including focusing using different working distances, and the planned final length (object distance), as well as the scanning simulations using "dogleg" scan, and experimental test results.

For the longer object distance it is important to know that the new Multicusp ionsource will provide more beam brightness than the present value. Therefore the brightness of the present configuration was measured at different proton energies and different slit settings. We can conclude that the longer object distance (resulting in much smaller solid angle) will be filled in with beam due to the higher brightness (according to HVEE specifications).

For the first resolution tests we used off axis and on axis STIM with a simple pin diode. Later we plan to install additional detectors, including one SDD detector for X-rays, and a lightguide for optical applications.

### P17 A NEW Q-POLE CONFIGURATION AT THE LIBAF MICROBEAM

Jan Pallon, Nathaly De La Rosa, Mikael Elfman, Per Kristiansson and E J Charlotta Nilsson

Division of Nuclear Physics, Department of Physics, Lund University, Box 118, SE-221 00 Lund, Sweden

The LIBAF microbeam was originally designed as a split quadruplet with a distance of several meters between two pairs of quadrupoles and a cross-over chamber for optical inspection of the intermediate focus [1,2]. The calculated demagnifications were 171 in both x and y which would result in a submicron beam focus. However, this was seldom achieved due to a number of limiting factors, e.g. the degree of alignment possible with four quadrupoles where the two pairs were separated by approximately 3 m. For several of the long-running application projects the main demand was a high beam current up to 10 nA rather than a micron sized beam and also a scanning size of 3 - 4 mm on the target using post-lens deflection. Due to this, the system was mostly run in a doublet configuration not taking advantage of all four lenses. Recently, a major reconstruction of the beam optical system has been performed. Several configurations were calculated using the TRANSPORT code [3], and selected configurations simulated in the TURTLE code [4]. All four magnets are placed close to the analytical chamber and arranged as a split quadruplet configuration with a cross-over in-between. In the new design, the post-lens scanning system has been moved outside the chamber which limits the demagnification due to the (long) working distance of 35 cm between target and focusing lens. Mechanically the four Oxford OM-52 lenses are placed on a common rail system with a single beam pipe passing through all four magnets and the beam scanning device. With this design, all magnets are free to be adjusted along the beam line if desired. Each lens has its own power supply and the respective focusing currents are computer controlled by software. In the final system, based on the simulations, the linear demagnification (defined as the square root of the areal demagnification) is expected to be 42 as compared to 23 for a Russian Quadruplet and 13 for a doublet having the same working distances.

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- [3] PSI Graphic Transport Framework by U Rohrer based on a CERN-SLAC-FERMILAB version by K L Brown et al [http://aea.web.psi.ch/Urs\_Rohrer/MyWeb/optics.htm]
- [4] PSI Graphic Turtle Framework by U Rohrer based on a CERN-SLAC-FERMILAB version by K L Brown et al [http://aea.web.psi.ch/Urs\_Rohrer/MyWeb/optics.htm]

# P18 ION IMPLANT INDUCED DISORDERS AND ITS EFFECT ON THE STRUCTURAL AND OPTICAL PROPERTIES OF CdS NANOWIRES

Jaskiran Kaur, R C Singh and Harmanmeet Kaur

Department of Physics, Guru Nanak Dev University, Amritsar, India

CdS being a member of II-VI group, having a wide optical band gap of 2.42 eV makes it suitable for use in the area of photovoltaic and laser devices. It is normally an n-type semiconductor and by doping it with transition metals one can affect its optical and electrical properties. Present study shows the effect of Fe implantation on CdS nanowires. Nanowires were synthesized using track etched filters via electro-deposition technique involving the movement of ions into the pores due to the potential applied with respect to the anode.Synthesised CdS nanowires were implanted with Fe ions at different fluences ranging from 10E16 to 10E18 using low energy ion beam facility available at IUAC New Delhi. Though radiation damage is often considered as an undesirable consequence but in this case presence of Fe can modify the optical and structural properties of CdS nanowires. Synthesized samples were studies using SEM, XRD, PL and Raman spectroscopy.

# P19 AUTOMATED ALIGNMENT AND FOCUSING SYSTEM FOR NUCLEAR MICROPROBES

Sarfraz Qureshi<sup>1</sup>, Wu Jiacheng<sup>2</sup> and Jeroen Anton van Kan<sup>1</sup>

<sup>1</sup>Centre for Ion Beam Applications, Department of Physics, National University of Singapore, Singapore 117542, Singapore <sup>2</sup>Engineering Science Program, National University of Singapore, Singapore 117575, Singapore

In the nuclear microprobe systems at CIBA tungsten carbide slits are used for defining beam centroid in objective and collimator apertures. The slits are attached to the end of a manually operated micrometre to define the aperture size and alignment. The current manually controlled beam focusing is slow and requires skilled operators. Therefore an automated system for proton beam collimation and focusing using precision slits, controlled using stepper motors is introduced. The beam defining micrometres are coupled to stepper motors using 3D printed gears positioned around the slit housing. An adaptive convergent algorithm is developed and implemented for fully automated slit control to facilitate manipulation of both object and collimator apertures. We achieved fast and reproducible slit positioning with the developed hardware and software. The gear coupling backlash is negligible when the motors are moved in the same direction. However, a reproducible backlash (~1  $\mu$ m) is observed when the motion directions are reversed which can be easily compensated via software control. Key slit automation functions implemented include collimator and object slit alignment using ion induced fluorescence and beam current as feedbacks respectively. Coupled with the autofocusing algorithm, we managed to focus a 2 MeV H<sub>2</sub><sup>+</sup> beam to a spot size of 23 × 32 nm<sup>2</sup> with a brightness of 42 A/(m<sup>2</sup>srV).

Lower ion count rate at small slit opening increases the acquisition time to image a resolution standard and estimate the beam spot size. Imaging based on proton induced secondary electrons emitted from a free standing resolution standard was implemented to further improve the beam focusing and accurately estimate the spot size. Using a 1  $\mu$ m thick Ni resolution standard, a 75% electron per proton collection efficiency for a beam of 1 MeV H<sub>2</sub><sup>+</sup> ions was reported earlier [1]. A free standing gold grid was fabricated using PBW in PMMA and a 180 nm Au layer was deposited using magnetron sputtering followed by PMMA lift-off and copper etching. A comparative analysis was performed for secondary electron emission yield with respect to nickel resolution standards. A 30% enhancement in secondary electron collection efficiency was achieved in comparison to the nickel resolution standard for a beam of 2 MeV protons. Initial experiments showed the stress between the supporting nickel grid and the free standing finer Au grid to be a limiting factor.

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# P20 POSITIONING SINGLE IONS BY FLUORESCENCE METHOD TO ACHIEVE HIGHER ANALYSIS PRECISION AT LIHIM

Jinlong Guo<sup>1</sup>, Guangbo Mao<sup>2</sup> and Guanghua Du<sup>1</sup>

<sup>1</sup>Institute of Modern Physics, Chinese Academy of Sciences, Lanzhou, China <sup>2</sup>University of Chinese Academy of Sciences, Beijing, China

Microbeam is a powerful tool to study radiation effects of cosmic heavy ions in integrated circuits (ICs) and living samples [1,2]. For this aim a high energy heavy ion focusing facility, Lanzhou Interdisciplinary Heavy Ion Microbeam (LIHIM), was constructed at the Institute of Modern Physics, Chinese Academy of Sciences. The candidate ions were provided by the cyclotron accelerators of the Heavy Ion Research Facility in Lanzhou (HIRFL), and the representative beam was 80.55 MeV/u <sup>12</sup>C<sup>6+</sup> that has been focused to 3  $\mu$ m × 5  $\mu$ m in air in 2013 [3]. LIHIM has unique advantages such as a long penetration depth, less lateral straggling and wide linear energy transfer (LET) range. However, the high energy ions of hundred MeV/u require a strict collimating and magnetic system to obtain a microbeam. The high energy heavy ion microbeams inescapably have a disadvantage of lower spatial resolution in comparison with MeV energy microprobes. Meanwhile, a decrease in the ICs' feature size and development of intracellular studies demand higher analysis accuracy for high energy microbeams.

In this work we presented a fluorescence method for precise positioning of single ions to obtain higher analysis accuracy at LIHIM. The samples were covered with a thin layer of fluorescent material at the back side which was not directly facing the incident ions. A microscope combined with a camera (Hamamatsu Digital CMOS Camera C11440-22CU) was employed to snap the fluorescence excited by penetrated LIHIM single ions on the target. Afterward, the sample image and the single ion images taken by the Hamamatsu camera were merged. Due to the long penetrate depth and less lateral straggling of LIHIM ions, the hit locations on the sample can be figured out precisely in the merged photos. The preliminary result showed that this method could effectively enhance single ion positioning accuracy in LIHIM analysis.

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- [3] L N Sheng et al, "Focusing giga-electronvolt heavy ions to micrometers at the Institute of Modern Physics", Rev Sci Instrum, Vol 84(5) (2013).

# P21 MEASUREMENT OF VARIOUS IONS IN HUMAN TEETH USING IN-AIR MICROBEAM PIXE/PIGE

K Naito<sup>1</sup>, K Yagi<sup>1</sup>, H Yamamoto<sup>1</sup>, Y Iwami<sup>1</sup>, Y Matsuda<sup>2</sup>, K Okuyama<sup>3</sup>, N Yamada<sup>4</sup>, M Koka<sup>4</sup>, T Sato<sup>4</sup> and M Hayashi<sup>1</sup>

<sup>1</sup>Osaka University Graduate School of Dentistry, Department of Restorative Dentistry and Endodontology, Japan

<sup>2</sup>Health Sciences University of Hokkaido Graduate School of Dentistry, Division of Clinical Cariology and Endodontology, Japan

<sup>3</sup>Asahi University School of Dentistry, Department of Dental Materials Sciences, Japan

<sup>4</sup>National Institute for Quantum and Radiological Science and Technology (QST), Takasaki, Japan

Numbers of epidemiological studies demonstrated that fluorine is useful for preventing dental caries. Recently, various ions other than fluorine such as zinc and calcium have been investigated with an aim of developing bioactive materials, which releases those ions to prevent dental caries.

In this study, we investigated the penetration of those ions from bioactive materials into human dentin using in-air microbeam PIXE/PIGE system at TIARA (Takasaki ion accelerators for advanced radiation application, Takasaki, Japan). The effect of those ions on acid resistance of human dentin, which is one of the indicators of caries inhibition using micro-computed tomography ( $\mu$ CT).

A total of eight sound human third molars from 24 to 31 years old were used. The human teeth were sectioned into four blocks and their dentin in the buccolingual surfaces were exposed. The dentin surfaces were covered with following materials: (1) coating agent containing ZnF (ZnF group), (2) glass ionomer cement (Fuji 7, which is a dental restorative material made of calciumalumino-fluorosilicate glass and polyacrylic acid or glass polyalkenoate cement) (F7 group), and (3) F7Ca (the half rate of Sr containing in Fuji7 was replaced by Ca) (F7Ca group). Root dentin without materials served as a control (control group). The specimens were kept in saline 37°C for three months. Each block was cut parallel to the longitudinal axis and perpendicular to the exposed root dentin area to make a 500 µm section.

Then the present study, multi-elemental sequential analysis were performed by measuring concentrations of F, Ca, Zn and Sr using the in-air microbeam PIXE/PIGE in accordance with the previous report [1]. For the Sr measurement, the detector with absorber of a thickness of 500  $\mu$ m was newly installed to exclude low energy X-rays. While the same F standard samples were used as in the report [1], for Zn and Sr, each standard sample was prepared by mixing zinc oxide or strontium sulfate with hydroxyapatite. For evaluation of acid resistance of dentin, each specimen was immersed in 10 ml of demineralizing solution (pH4.5) for three days at 37°C. The mineral concentrations of all specimens was measured with  $\mu$ CT. The difference of mineral concentration before and after the demineralization was defined as mineral loss which was an index of acid resistance.

ZnF group showed significantly higher Zn concentration in dentin than other groups (One-way ANOVA and Tukey multiple comparison tests; p < 0.05). ZnF and the F7Ca groups had significantly less mineral loss than the control group. However, there was no significant difference in mineral loss

among the materials. There was a negative correlation between the concentration of zinc and the amount of mineral loss (Pearson's correlation coefficient analysis).

The PIXE/PIGE system enabled multi-ion sequential measurements of human dentin. It was conducted that multi-ion such as fluorine, zinc and calcium promoted caries inhibition. This study was supported by Grants-in-Aid for Scientific Research (17H04382, 17K11705) from the JSPS.

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# P22 FLUORINE DISTRIBUTION FROM FLUORIDE-RELEASING LUTING MATERIALS TO HUMAN DENTIN

Katsushi Okuyama<sup>1</sup>, Yasuhiro Matsuda<sup>2</sup>, Hiroko Yamamoto<sup>3</sup>, Yukimichi Tamaki<sup>1</sup>, Takashi Saito<sup>2</sup>, Mikako Hayashi<sup>3</sup>, Yasuhiro Yoshida<sup>4</sup>, Hidehiko Sano<sup>4</sup>, Takahiro Sato<sup>5</sup> and Masashi Koka<sup>6</sup>

<sup>1</sup>Asahi University School of Dentistry, Japan
 <sup>2</sup>School of Dentistry, Health Sciences University of Hokkaido, Japan
 <sup>3</sup>Graduate School of Dentistry, Osaka University, Japan
 <sup>4</sup>Faculty of Dental Medicine, Hokkaido University, Japan
 <sup>5</sup>Takasaki Advanced Radiation Research Institute, QST, Japan
 <sup>6</sup>Beam Operation Co Ltd, Japan

There are some kinds of dental luting materials (cements) in the market. Recently, some of them have a function of fluoride releasing from material because of preventing secondary caries after cementing with prosthetic appliances (crown or bridge). The purpose of this study is to evaluate distribution of fluorine from fluoride-containing luting materials to adjacent dentin by in-air micro PIXE/PIGE system at Takasaki Advanced Radiation Research Institute.

Extracted human teeth were used in this study. Each tooth crown was cut and grinded to expose a flat dentin surface. An acrylic resin plate (2 mm thickness) was cemented with four kinds of fluoride-containing luting materials (Fuji I: FO, Fuji Luting EX: FL, Clearfil SA Luting plus: SA, BeatiCem SA: BC) on the dentin surface. The specimens were sliced (about 500  $\mu$ m thickness) and carried out on pH cycling (pH:4.5 and 7.0; simulate oral condition) for 8w for preparing artificial carious dentin using an automatic pH-cycling system [1]. After pH cycling, calcium and fluorine distribution were analyzed by an in-air micro-PIXE/PIGE system with a 1.7 MeV 1H+ microbeam [2]. The outermost surface of the dentin was defined at the position containing 5% of the calcium concentration in intact dentin. For the comparison of fluorine uptake, the average fluorine concentration in each specimen was calculated at area of 100  $\mu$ m from the defined surface. The data were analyzed by Kruskal-Wallis test and Mann-Whitney's U test ( $\alpha = 0.05$ ).

FL indicated highest concentration of uptake fluoride among all used materials (p < 0.05). Other three materials indicated no significantly different concentration between each other (p > 0.05). High concentration of fluorine was exhibited FL, because this material released large amounts of fluoride to water compared with other materials [3]. Therefore, it is speculated that FL were more effective in reducing the dental caries than other materials. The amount of fluorine distribution on dentin might be depend on amount of fluoride release from each material.

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# P23 SPATIAL CHARACTERIZATION OF SCHOTTKY BARRIERS FORMED IN $\beta$ -Ga<sub>2</sub>O<sub>3</sub> USING A PROTON MICROPROBE

Marco Peres<sup>1,2</sup>, L C Alves<sup>3</sup>, E Alves<sup>1</sup>, T S Monteiro<sup>2</sup>, S Cardoso<sup>2</sup>, A Kuramata<sup>4</sup>, E G Víllora<sup>5</sup>, K Shimamura<sup>5</sup> and K Lorenz<sup>1,2</sup>

 <sup>1</sup>IPFN, Instituto Superior Técnico (IST), Campus Tecnológico e Nuclear, Estrada Nacional 10, P-2695-066 Bobadela LRS, Portugal
 <sup>2</sup>Instituto de Engenharia de Sistemas de Computadores-Microsystems and Nanotechnology (INESC-MN), IST, Lisboa, Portugal
 <sup>3</sup>C2TN, Instituto Superior Técnico (IST), Campus Tecnológico e Nuclear, Estrada Nacional 10, P-2695-066 Bobadela LRS, Portugal
 <sup>4</sup>Tamura Corporation, Sayama, Saitama 350-1328, Japan
 <sup>5</sup>National Institute for Materials Science, 1-1 Namiki, Tsukuba 305-0044, Japan

 $\beta$ -Ga<sub>2</sub>O<sub>3</sub> with a wide band gap of ~4.8 eV, a high breakdown voltage of 10 MV.cm<sup>-1</sup> and a high saturation electron velocity of 2 × 10<sup>7</sup> cm.s<sup>-1</sup> is one of the most promising semiconductors for future high power and transparent electronics and solar blind radiation detectors.  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> compared with other competitive wide band gap semiconductors (e.g. GaN and SiC) presents several advantages namely the possibility to tune its electrical conductivity and the possibility of being grown by standard melt grown methods.

In this work, several co-planar back-to-back Schottky diodes were fabricated on thick single crystals and on thin flakes produced by mechanical exfoliation. These devices present an asymmetric I-V curve due to the difference between the properties of the two Schottky barriers that compose these unipolar devices. The potential of these devices as UV detectors will be discussed based on stationary and transient photoconductivity measurements. Different current gains and different persistent photoconductivity will be discussed in function of the properties of the different Schottky barriers.

Using a proton microprobe these different devices were characterized simultaneously with conventional Ion Beam Analysis techniques, namely, Particle Induced X-ray Emission and Rutherford Backscattering Spectrometry complemented by ionoluminescence and electrical characterization (I-V sweepings and transient I-time). The current gain (ion beam induced current) and the persistent ionoconductivity was studied with spatial resolution as a function of the applied voltage and of the irradiated region (on the contacts or in the region between them). Maps of ion beam induced current in the different regions will be presented and discussed. In particular, it will be shown that the detection properties (namely the induced current and the persistent conductivity) are very heterogeneous. In this context, this work will emphasize the potential of using a nuclear microprobe together with optical and electrical measurements to characterize electric contacts in general and specifically in  $Ga_2O_3$ .

The radiation damage resistance of these devices will be discussed based on the study of the ionoluminescence decay during the irradiation as well as the changes of the electrical properties.

#### P24 DEVELOPMENT OF MeV TOF-SIMS CAPILLARY MICROPROBE

Marko Barac, Zdravko Siketić and Iva Bogdanović Radović

Ruđer Bošković Institute, Bijenička cesta 54, 10000 Zagreb, Croatia

MeV TOF-SIMS is a mass spectrometry method for identification and structural categorization of broad range of biomolecules by measuring time-of-flight of secondary ions sputtered from the sample surface. As an excitation, instead of primary ions with keV energies that are used in keV TOF SIMS setups, fast and heavy primary ions with MeV energies produced in accelerators are employed. Advantages include higher secondary ion yields and less fragmentations for molecules in the mass range 100 - 1000, mostly due to the electronic stopping interaction of the primary ions with the sample surface.

The new MeV TOF-SIMS setup is recently installed at the central beam line of the 6 MV Tandem Van de Graaff accelerator, located at the Ruđer Bošković Institute. Heavy ions such as Cl, Cu, Br, and I with energies of 30 MeV can be delivered enabling to explore full capability of the MeV TOF-SIMS technique. To bypass the limitations of commonly used quadrupole lenses for focusing heavy energetic primary ions, microscale lateral dimension of primary ion beam in the new system is achieved with borosilicate glass micro-capillary. Focusing properties of such micro-capillary are independent on the ion mass and energy. 2D imaging can be achieved by moving the sample placed on a 4D piezo-based scanning stage. Secondary ions are collected and analyzed employing the reflectron type TOF analyzer. A PiN diode, placed behind the transparent target, is used as a START signal for TOF measurements.

In this work, the new imaging MeV TOF-SIMS setup based on the micro-capillary will be presented. First results on the current and energy distribution measurements through the micro-capillary for several heavy ions Cl, Cu and I (with energies up to 30 MeV) will be shown.

# P25 INVESTIGATION OF THE LATERAL HOMOGENEITY OF PLASMA-DRIVEN DEUTERIUM PERMEATION THROUGH TUNGSTEN

Mitja Kelemen<sup>1,2</sup>, Sabina Markelj<sup>1</sup>, Primož Vavpetič<sup>1</sup>, Stefan Kapser<sup>3,4</sup>, Martin Balden<sup>3</sup>, Stefan Elgeti<sup>3</sup> and Primož Pelicon<sup>1</sup>

<sup>1</sup>Jožef Stefan Institute, Jamova 39, SI-1000 Ljubljana, Slovenia
 <sup>2</sup>Jozef Stefan International Postgraduate School, Jamova cesta 39, 1000 Ljubljana, Slovenia
 <sup>3</sup>Max-Planck-Institut für Plasmaphysik, Boltzmannstrasse 2, 85748 Garching, Germany
 <sup>4</sup>Physik-Department, Technische Universität München, James-Franck-Strasse 1, 85748 Garching, Germany

Tungsten is a promising candidate as plasma-facing material in future fusion reactors, where it will be positioned in the divertor region of tokamak-design reactors. In this region of the inner wall, the power and particle fluxes are the highest which leads to lattice damage and fuel retention in the wall material. Main fuel in future fusion reactors will be a mixture of deuterium (D) and tritium (T). As tritium is radioactive, its retention causes safety restrictions for the reactor operation. Lab studies are usually conducted with D and the results are then extrapolated to tritium.

One of the unsolved questions is the transport of fuel (T,D) into the bulk of tungsten material. Is there a preferred path of permeation of D atoms, i.e. along grain boundaries? To gain information about such a preferred path for permeation, a specially prepared 25  $\mu$ m thick tungsten foil was investigated by microbeam NRA (nuclear reaction analyses) after it had been exposed to D plasma with a flux of 6 × 10<sup>19</sup> D/(m<sup>2</sup> s) for 144 h at 300 K. The recrystallized tungsten foil had a getter layer of Zr in which permeated D was accumulated on the side which was not exposed to the plasma [1]. For the microbeam NRA, the nuclear reaction D(<sup>3</sup>He,p) $\alpha$  was used, which is frequently used to analyse the depth distribution of D in tungsten [2].

The microbeam NRA, which was required due to the size of the tungsten grains (which was of the order of 50  $\mu$ m), was performed in the microbeam experimental chamber attached to the 2 MV tandem accelerator at the Jožef Stefan Institute [3]. The incident energy of the <sup>3</sup>He<sup>+</sup> ions was 1.8 MeV. This energy was selected to gain a strong signal from the deuterium in the getter layer. Under these conditions, we were able to produce a focused ion beam with dimensions of 5 × 5  $\mu$ m<sup>2</sup> and an ion current of 100 pA on the target at the measurement position. The focused <sup>3</sup>He beam was scanned over an area of 120 × 120  $\mu$ m<sup>2</sup>. During the measurements, in total four detectors were used simultaneously: a thick-depleted implanted silicon detector for NRA, a RBS PIPS detector, a HPGe X-ray detector for detection of particle induced X-ray emission (3HIXE) and a detector that detects ions scattered from the beam chopper for beam dose normalization. The mapped signals from the RBS and X-ray detectors allow to correlate the measured region of the microbeam analysis with scanning electron microscopy (SEM) images of the sample surfaces. For correlation of these maps markers are used, which had been created with a focused gallium ion beam (FIB).

The results obtained with the microbeam analysis will be presented and discussed. Some clustering of D was observed and the correlation of the clustering with the locations of grain boundaries was analyzed.

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### P26 MICRO-PIXE IN FROZEN HYDRATED STATE

Primož Vavpetič<sup>1</sup>, Mitja Kelemen<sup>1,2</sup>, Katarina Vogel-Mikuš<sup>1,3</sup>, Boštjan Jenčič<sup>1</sup>, Paula Pongrac<sup>1</sup>, Esther Punzón-Quijorna<sup>1</sup>, Marjana Regvar<sup>3</sup> and Primož Pelicon<sup>1</sup>

<sup>1</sup>Jožef Stefan Institute, Jamova 39, 1000 Ljubljana, Slovenia <sup>2</sup>Jožef Stefan International Postgraduate School, Jamova 39, 1000 Ljubljana, Slovenia <sup>3</sup>Biotechnical Faculty, University of Ljubljana, Jamnikarjeva 101, SI-1000 Ljubljana, Slovenia

The continuing development of nuclear microprobe at Jožef Stefan Institute we reached a level that enables users from biomedical field to routinely analyze samples in frozen hydrated state [1] with sub-micron resolution [2]. Successful implementation of a cryostat to the micro-PIXE chamber and careful design of a new sample holder enables us to examine a broad range of samples of various thicknesses. This is especially suitable for the outer collaborators from biology and medicine, since sample lyophilization is no longer mandatory. In the biological tissue analysis, it is preferably avoided in the sample preparation of biological materials with high water content [3]. Biological tissue samples are cryotome-cut, deposited on the dedicated sample substrates positioned within the metal blocks and stored in liquid nitrogen. The samples are in this way preserved at low temperature for the analysis at the microprobe until the scheduled beam-time. The entire nuclear microprobe setup operates with a set of two X-ray detectors, EBS, on-off axis STIM [4] and RBS detector detecting scattered protons from the in-beam rotating chopper. Selected recent results obtained with submicrometer lateral resolution will be presented.

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### P27 A SIMPLE MODEL OF MeV-SIMS PROFILING

Roger Webb, Vladimir Palitsin, Lidija Matjačić and Aniza Abdul-Karim

Surrey Ion Beam Centre, University of Surrey, Guildford, GU2 7XH, UK

With the addition of MeV-SIMS, and hence some molecular speciation, to Total IBA it is now able to provide qualitative and often quantitative information on the chemical, molecular and elemental make up of a material making it an extremely powerful technique. A good model to describe the MeV-SIMS process and the development of the sputtered target is preventing a comprehensive understanding of the MeV-SIMS experiments. Here we present a description of a simple model of MeV (which is also applicable to cluster) SIMS. The model is based on the results of Molecular Dynamics simulations and previous work on the sputtering of molecular materials via keV clusters and the interaction of MeV ions with weakly bonded frozen inert gases. In particular it is shown that cluster and MeV SIMS are very similar and give rise to a simple model which can describe the basic processes in both experimental set-ups. A more detailed description of fragmentation is introduced in the model and parameters derived from experiments for PMMA and PHEMA are used to describe the erosion through a layered sample containing both PHEMA and PMMA. The results are compared favourably with experiment.

# P28 IMPROVEMENTS IN REZ ION MICROPROBE PARAMETERS: NUMERICAL CALCULATION

Oleksandr V Romanenko<sup>1</sup>, Alexander G Ponomarev<sup>2</sup>, Anna Macková<sup>1,3</sup>, Vladimír Havránek<sup>1</sup> and Artem Ponomarov<sup>4</sup>

<sup>1</sup>Nuclear Physics Institute of the Czech Academy of Sciences, 250 68 Řež, Czech Republic

<sup>2</sup>Institute of Applied Physics, National Academy of Sciences of Ukraine, 40030 Sumy, Ukraine

<sup>3</sup>Department of Physics, Faculty of Science, J E Purkinje University, Ceske Mladeze 8, 40096 Usti nad Labem, Czech Republic

<sup>4</sup>Institute of Modern Physics, Chinese Academy of Sciences, Lanzhou, China

Ion scanning microprobe was implemented at the Nuclear Physics Institute in Rez in 2009. Since then it has been involved in numerous experiments for investigation and modification of the materials where PIXE, PIGE, RBS and STIM are the main analytical methods. Rez microprobe is coupled with a tandetron accelerator, which provides ions from hydrogen to gold, and able to focus heavy ions with the maximum mass energy product at the level 11 MeV·amu/q<sup>2</sup>. Therefore, microprobe can be used for polymers irradiation by protons and helium as well as for irradiation of various types of glass and other materials with heavy ions. The number of applications on Rez microprobe is constantly increasing, that leads to necessity to raise the efficient of the microprobe operation. Increasing the beam current density allows to reduce the time per one experiment with keeping events statistic. The present work is an attempt to improve the parameters of existing ion scanning microprobe based on a compact Oxford triplet lens system. Since the microprobe is used to investigate both thin and thick targets, the position of the detectors that can restrict the microprobe construction were taken into account in the calculation. The collimated acceptance of the probe-forming system was selected as an objective function as it takes into account both chromatic and all spherical aberrations. Two methods for improving the microprobe parameters were considered. The first one is based on separation of the first lens in the system, whilst the second on decreasing the working distance. Both ways need changing in the microprobe construction, but the last one requires a major alteration in a target chamber. The benefits of each way were studied. Obtained parameters of the new probe-forming system have shown several times increasing the current density of given probe size for Rez microprobe in comparison with the present system.

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# P29 MICRO-PIXE MAPPING OF SEVERAL KEY MINERAL NUTRIENTS LOCALISED WITHIN THE BRAN TISSUE OF PEARL MILLET (*PENNISETUM GLAUCUM*) GRAINS

R Ndimba<sup>1</sup>, J Kruger<sup>2</sup>, J Taylor<sup>2</sup>, C Mtshali<sup>1</sup> and C A Pineda-Vargas<sup>1</sup>

<sup>1</sup>iTHEMBA Laboratories, National Research Foundation, PO Box 722, Somerset West 7129, South Africa

<sup>2</sup>Department of Food Science and Institute for Food, Nutrition and Wellbeing, University of Pretoria, South Africa

Pearl millet (*Pennisetum glaucum*) is an important food security crop that serves as a primary staple for about 90 million people across Africa and Asia. The bran portion or outer layers of the grain are particularly nutrient-dense, and are known to be highly concentrated in essential minerals. Typically, however, precise information depicting the distribution and concentration of important mineral nutrients within mature millet grains are rare. In this study, fully quantitative elemental maps focused on the bran tissues of pearl millet grains were generated using the micro-PIXE technique at the nuclear microprobe facility, situated at iTHEMBA Laboratories, Cape Town, South Africa. The micro-PIXE maps revealed unique details of the spatial distribution patterns and tissuespecific concentrations of important minerals (such as P, S, K, Ca, Mn, Fe and Zn), as found within four distinctive areas of the bran tissue, namely the pericarp, the aleurone, the subaleurone and the outer endosperm layers of the grain. Across all the major bran compartments, the macroelements, K, S, P were always found to be dominant, with K exhibiting the highest average concentration (5249.8 mg/kg DW) in the aleurone; whilst S was found to be most dominant in the subaleurone, with an average concentration of 3599.0 mg/kg DW. For P, a clear similarity in distribution pattern with K was evident, with P also detected at its highest concentration in the aleurone layer, at 3221.2 mg/kg DW. In distinction to the pattern observed for all other elements measured, the peak average concentration of Ca was not localised to the aleurone/subaleurone zone, but instead was detected at its highest level in the pericarp at a concentration of 713.0 mg/kg DW. Amongst the micronutrients detected, Mn exhibited the lowest concentration values, with below detection limit quantities in the outer endosperm (i.e. <2 mg/kg), but peak average values of 15.2 and 19.7 mg/kg in the pericarp and aleurone tissues respectively. Higher average concentrations of Fe, in comparison to Zn, was observed for each distinctive area of the bran tissue, except for the outer endosperm, where Zn averaged at 21.3 mg/kg, whereas Fe was more than four times less, at 4.9 mg/kg. These unique insights into the differential patterns of mineral accumulation in pearl millet grains could be of great benefit in biofortification and food processing/refinement programmes aimed at improving the nutritional value of this important staple food.

## P30 TERTIARY ELECTRONS IN SINGLE-EVENT TIME-OF-FLIGHT RUTHERFORD BACKSCATTERING SPECTROMETRY

Satoshi Abo<sup>1</sup>, Albert Seidl<sup>2</sup>, Fujio Wakaya<sup>1</sup> and Mikio Takai<sup>1</sup>

<sup>1</sup>Graduate School of Engineering Science, Osaka University, Japan <sup>2</sup>Department of Engineering Science and Industrial Design, Magdeburg-Stendal University of Applied Sciences, Germany

The non-destructive analysis technique for three dimensional nanostructures using single event time-of-flight Rutherford backscattering spectrometry (TOF-RBS) has been developed with in-plane and spatial resolutions of 42 nm and less than 10 nm, respectively [1,2]. The short-time and high-resolution analysis can be realized by the control of the secondary-electron trajectories with positive sample bias voltage [2]. In this study, the effect of negative sample bias voltage in single-event TOF-RBS was discussed.

The rise times of the Pt peak in the TOF-RBS spectra with positive and negative sample bias voltage were 17% shorter and 115% longer than that without sample bias voltage, respectively. This means that positive and negative sample bias voltage improves and deteriorates the time-resolutions in single-event TOF-RBS, respectively. These phenomena could be explained by the difference of the secondary electron trajectories [2].

The analysis times with both positive and negative sample bias voltage were 33% shorter than that without sample bias voltage. The analysis time was related to the number of the detected electrons as a start trigger at the electron detector (ED). The experimental results means that the number of the detected electrons was increased with both negative and positive sample bias voltage. However, the increase of the detected electrons with negative sample bias voltage could not be reproduced by the trajectory simulation for secondary electrons. Thus, the trajectories of the tertiary electrons from the focused ion beam (FIB) column generated by incidence of the secondary electrons were simulated. The ED detects not only the secondary but also tertiary electrons as a start trigger in single-event TOF-RBS. Therefore, the short analysis time was realized with negative sample bias voltage. However, the trajectories of the secondary and tertiary electrons were spread, resulting in the worse time resolution with negative sample bias voltage.

The tertiary electrons from the FIB column with negative sample bias voltage caused of the short analysis time and worse time resolution in single-event TOF-RBS. The positive sample bias voltage was effective for short analysis time and highly time resolution in single-event TOF-RBS.

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# P31 COMPARATIVE STUDY ON PHASE PROPORTIONS IN DENTAL AMALGAMS USING MICRO-PIXE AND XRD TECHNIQUES

Wanwisa Sudprasert<sup>1</sup>, Ridthee Meesat<sup>1</sup>, Kullita Kolanan<sup>1</sup>, Henry Udeogu<sup>3,4</sup>, Armin B de Vera<sup>3</sup>, Naresh Deoli<sup>3,4</sup> and Harry J Whitlow<sup>2,3,4</sup>

<sup>1</sup>Department of Applied Radiation and Isotopes, Faculty of Science, Kasetsart University, Bangkok 10900, Thailand

<sup>2</sup>Department of Chemistry, Faculty of Science, Kasetsart University, Bangkok 10900, Thailand

<sup>3</sup>Louisiana Accelerator Center, University of Louisiana at Lafayette, PO Box 70504, Lafayette, LA 70504, USA

<sup>4</sup>Department of Physics, University of Louisiana at Lafayette, PO Box 70504, Lafayette, LA 70504, USA

Micro-PIXE is a powerful tool for analysis of metallic elements in several different materials including biomedical materials due to its many advantages. Our previous studies investigated the mercury pathways from dental amalgams of seventeen extracted teeth by micro-PIXE. The results indicated no evidence of migration through the enamel and dentine. However, some evidence of tribocorrosion of the wear surface were observed, which might lead to mercury intake through the ingestion route. The variations in the metallic composition in an amalgam can influence its physical properties such as creep, hardness, morphology and corrosion resistance. It was reported that the relative proportion of phases can modify the corrosion resistance of dental amalgams. This study was carried out to analyze the phase proportions in selected dental amalgams using micro-PIXE and compare the results with XRD technique. The result obtained from both micro-PIXE and XRD measurements revealed that  $\gamma_1$ -(Ag<sub>2</sub>Hg<sub>3</sub>) was the major phase observed in all measured amalgams. These findings were consistent with the corrosion of the wear surface evidenced by elemental maps obtained by micro-PIXE technique.

# P32 ANTIBACTERIAL EFFECT OF FLUORIDE-CONTAINING ZnO/CuO NANOCOMPOSITE

Yasuhiro Matsuda<sup>1</sup>, Katsushi Okuyama<sup>2</sup>, Hiroko Yamamoto<sup>3</sup>, Mari Fujita<sup>4</sup>, Shigeaki Abe<sup>5</sup>, Takahiro Sato<sup>6</sup>, Naoto Yamada<sup>6</sup>, Masashi Koka<sup>7</sup>, Sharanbir K Sidhu<sup>8</sup> and Saito Takashi<sup>1</sup>

 <sup>1</sup>Division of Clinical Cariology and Endodontology, Department of Oral Rehabilitation, School of Dentistry, Health Sciences University of Hokkaido, Japan
 <sup>2</sup>Department of Dental Materials Science, Asahi University School of Dentistry, Japan
 <sup>3</sup>Department of Restorative Dentistry and Endodontology, Osaka University Graduate School of Dentistry, Japan
 <sup>4</sup>Department of Oral Microbiology, School of Dentistry, Health Sciences University of Hokkaido, Japan
 <sup>5</sup>Department of Biomaterials and Bioengineering, Hokkaido University Graduate School of Dental Medicine, Japan
 <sup>6</sup>Takasaki Advanced Radiation Research Institute, QST, Japan
 <sup>7</sup>Beam Operation Co Ltd
 <sup>8</sup>Restorative Dentistry, Institute of Dentistry, Queen Mary University, UK

Dental materials which are antimicrobial and acid-resistant can inhibit bacterial colonization and demineralization, and thereby prevent caries. Zinc and copper are well-known for their antibacterial effect; so is the nanostructured ZnO-CuO composite. Minerals such as fluorine and calcium can redemineralize teeth. Therefore, we developed novel fluoride-containing ZnO-CuO (ZCF) nanocomposites; to the best of our knowledge, this is the first nanocomposite of its kind. The fluoride concentration and antibacterial effect of the ZCF nanocomposites were then evaluated.

Nanocomposites comprising zinc and copper (ZC), and zinc, copper, and fluorine (ZCF), were prepared by a simple one-step homogeneous coprecipitation method at a low temperature (80°C), without using any organic solvent or surfactant.

The structure and composition of the ZC and ZCF nanocomposites were examined by scanning electron microscopy-energy-dispersive spectroscopy (SEM-EDS). Quantitative analysis of the percentage of weight concentration was performed using ZAF correction methods. The fluorine content in nanocomposites was evaluated using proton-induced gamma emission (PIGE) at the Takasaki Advanced Radiation Research Institute, in Japan. Using 96-well microtiter plates, we analyzed the antibiotic susceptibility of ZC, ZCF, and the control (PBS buffer) with S-mutans (ATCC, 25175).

The SEM image shows that the ZC and ZCF nanocomposites are composed of 3D flower-like microstructures with diameters of approximately 1  $\mu$ m. Environmental scanning electron microscopy and energy-dispersive X-ray analysis revealed that ZC contains 43.0% Cu, 54.3% Zn, and 2.2% F, whereas ZCF contains 47.2% Cu, 40.0% Zn, 5.9% Cl, and 7.0% F.

The ZCF nanocomposite contained 2553.6  $\pm$  199.2 ppm fluorine, whereas no fluoride was detected in ZC by PIGE. The control group shows a viability of 4  $\times$  10<sup>7</sup>  $\pm$  9  $\times$  10<sup>6</sup> CFU/mL, whereas that of ZC is only 12  $\pm$  8 CFU/mL, and ZCF shows no bacterial growth.

Thus, we developed novel fluoride-containing ZnO-CuO nanocomposites, which exhibited antibacterial effects and enabled remineralization, thereby demonstrating their potential as multifunctional dental materials.

# P33 COMBINED $\mu$ -PIXE/ $\mu$ -EBS ANALYSIS OF SURFACE STAINS IN GOLD COINS/DISCS

#### J Cruz<sup>1</sup>, V Corregidor<sup>2</sup> and L C Alves<sup>3</sup>

<sup>1</sup>Laboratório de Instrumentação, Engenharia Biomédica e Física da Radiação (LIBPhys-UNL), Departamento de Física, Faculdade de Ciências e Tecnologia da Universidade Nova de Lisboa, Monte da Caparica, 2892-516 Caparica, Portugal <sup>2</sup>IPFN, IST-UL, Campus Tecnológico e Nuclear, EN 10, 2695-066 Sacavém, Portugal <sup>3</sup>C2TN, IST-UL, Campus Tecnológico e Nuclear, EN 10, 2695-066 Sacavém, Portugal

In 2017, the Portuguese Mint House (INCM) commissioned the LATR (Laboratory of Accelerators and Radiation Technologies) at IST-UL, a study of surface stains that are appearing in 99.9% pure gold collector coins. This study aimed mainly to determine whether the contamination occurred during the blank production (external supplier) or during the minting process at the INCM facilities. The analysis were performed at the microprobe beam line of the 2.5 MV Van de Graaff Accelerator [1] using a 700 keV and 2 MeV proton beams (lower energies probe shallower in the sample but with better depth resolution). Beam scans over the surface of the samples ranges up to 2640 × 2640  $\mu$ m<sup>2</sup> when using a 2 MeV proton beam (beam dimensions are 3 × 4  $\mu$ m<sup>2</sup>).

Particle Induced X-ray Emission (PIXE) and Elastic Backscattering Spectrometry (EBS) spectra were obtained simultaneously and analyzed in a self-consistent manner using the Datafurnace code [2]. This approach allowed the determination of elemental depth profiles, as exemplified in Figure 1 for a silver splint carved on a gold disc. Conclusions are that the majority of the stains are due to contaminations prior to reaching the INCM facilities.



Figure 1. Ag splint carved on disc gold surface: Ag-La map pointing the analysed points and the measured thicknesses.

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#### P34 RECONSTRUCTION OF H DISTRIBUTION IN ICF TARGET SHELL BY PPS-T

Hailei Zhang<sup>1</sup>, Na Guo<sup>1</sup>, Tao Yu<sup>1</sup>, Dangzhong Gao<sup>2</sup>, Qi Wang<sup>2</sup>, Xiaojun Ma<sup>2</sup> and Hao Shen<sup>1</sup>

<sup>1</sup>Institute of Modern Physics, Applied Ion Beam Physics Laboratory, Fudan University, Shanghai 200433, China

<sup>2</sup>Research Center of Laser Fusion, Chinese Academy of Engineering Physics, Mianyang 621900, China

In Inertial Confinement Fusion (ICF) experiments, only the target with high sphericity and high uniformity of wall thickness could be used to avoid hydrodynamic instability during the implosion. Besides, the element distribution in the target shell should also be uniform. STIM-T and PIXE-T has been proven to be a good method to characterize the mass distribution and dopant element distribution in ICF target. While this work demonstrates the capability of tomography of light element like H or D in ICF target with proton-proton scattering coincidence measurement computed tomography (PPS-T). Glow discharge polymer (GDP) targets were used in this study, which contains H atoms in their shells. Proton microbeam was used to scan the targets and 30 projections over 180° were obtained in total. A reconstruction program run in MATLAB was developed based on Filtered Back Projection (FBP) algorithm. Geometrical misalignment and beam pathlength effect in the reconstruction was discussed.

# P35 IMPROVEMENT OF ANALYTICAL MEASUREMENTS OF NON-TRIVIAL BIOLOGICAL SAMPLES WITH NUCLEAR MICROSCOPY

Teresa Pinheiro<sup>1,2</sup>, Pedro Teixeira<sup>2,3</sup>, João Bento<sup>2,3</sup>, Rita Godinho<sup>1,4</sup>, Fernanda Marques<sup>1,5</sup>, Luís C Alves<sup>1,5</sup>, Nídia Lourenço<sup>2,3</sup> and Helena Pinheiro<sup>2,3</sup>

<sup>1</sup>Departamento de Engenharia e Ciências Nucleares, Instituto Superior Técnico, Universidade de Lisboa, Portugal
<sup>2</sup>Instituto de Bioengenharia e Biociências (IBB), Portugal
<sup>3</sup>Departamento de Bioengenharia, Instituto Superior Técnico, Universidade de Lisboa, Portugal
<sup>4</sup>IPMA, Instituto Português do Mar e da Atmosfera, Lisboa, Portugal
<sup>5</sup>Centro de Ciências e Tecnologias Nucleares (C2TN), Portugal
<sup>6</sup>CIIMAR, Centro Interdisciplinar de Investigação Marinha e Ambiental, Universidade do Porto, Matosinhos, Portugal

The task of sample preparation involves many challenges and demands for micro-analytical techniques. Appropriate preparative steps can be the determining factor between successful measurements and failures. Nuclear microscopy analysis offers a broad range of possibilities in terms of characterization of materials at the micrometric and sub-micrometric scale, such as, density measures, elemental distributions, and depth elemental profiles. These characteristics are very attractive to quantitatively inspect the ability of cells and/or tissues to perceive and respond to their microenvironment. As research problems become more multifaceted, encompassing various fields of science, the complexity of samples to analyse increases. The collection, processing, storage and preparation methods may need to be modified to comply with the sample specificities and the analytical requirements. Apart a short review of consolidated methodologies to analyse biological materials with nuclear microscopy, the development of methods tailored to analyse non-trivial samples will be discussed. This may include microbial biomass exploited for new biotechnological applications, cellular fingerprints to quantitate uptake of new metal drug compounds for the treatment of human inflictions, interaction with the environment of organisms managing the carbon cycle and global warming.

# P36 DESIGN OF A COLLIMATION SYSTEM FOR THE 300 MeV PROTON MICROBEAM SYSTEM IN HARBIN

Yanxin Dou<sup>1</sup>, Jianli Liu<sup>2</sup> and Liyi Li<sup>1</sup>

<sup>1</sup>School of Electrical Engineering and Automation, Harbin Institute of Technology, Harbin 150001, China

<sup>2</sup>Research Center of Basic Space Science, Harbin Institute of Technology, Harbin 150001, China

In Harbin, a 300 MeV proton microbeam system [1] is being developed [2,3] as a component of the Space Environment Simulation Research Infrastructure (SESRI) with many applications in space science studies including upset studies in microelectronic devices [4], radiation hardness of materials for satellites [5] and radiation affects in human tissues [6]. The beam for the microprobe system will be provided by a synchrotron accelerator and the aim of this paper is to address the design of the beam collimation system needed to shape the beam entering the probe forming lens system which is a critical issue for high energy microbeams. A new approach is presented that incorporates two thin collimator as divergence limiting collimator and object collimator and a thick collimator as antiscattering collimator to reduce the scattering of protons and other particles and to provide a well-collimated beam. The design of the new system is validated with reference to Turtle [7] and G4beamline [8] which shows that fewer than 2% of the beam in the focused microprobe consists of scattered protons. These will be useful in the application of single ion irradiation.

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# P37 THE INFLUENCE OF THERMAL ANNEALING ON DEFECTS INDUCED IN Xe IMPLANTED N-TYPE 4H-SILICON CARBIDE

E Omotoso<sup>1,2</sup>, W E Meyer<sup>1</sup>, E Igumbor<sup>1</sup>, H T Danga<sup>1</sup>, B A Taleatu<sup>2</sup> and F D Auret<sup>1</sup>

<sup>1</sup>Department of Physics, University of Pretoria, Private Bag X20, Hatfield 0028, South Africa

<sup>2</sup>Department of Physics, Obafemi Awolowo University, Ile-Ife 220005, Nigeria

In this study, 4H-silicon carbide samples were bombarded with 167 MeV Xe ions to a fluence of  $1 \times 10^8$  cm<sup>-2</sup> at 300 K prior to fabrication of Schottky contacts. The samples were also annealed at approximately 900°C before thermal fabrication of the contacts. When compared current-voltage results with the as-grown device, generation-recombination occurred in the implanted Schottky barrier diodes. The presence of four deep level defects (0.10, 0.12, 0.16 and 0.65 eV) were observed in as-grown devices when characterised by deep level transient spectroscopy. In addition, two deep level defects with activation energies of 0.40 and 0.69 eV below the conduction band minimum were induced as a result of implantation. These two induced-defects have similar signatures to other defects observed by MeV electron irradiation. It was observed that the two defects induced were annealed out at 400°C which indicated the instability of the defects after annealing the implanted sample.

# P38 HIGH THROUGHPUT LARGE AREA MAPPING OF GEOLOGICAL SAMPLES USING A MAIA DETECTOR ARRAY ON THE NUCLEAR MICROPROBE

J S Laird<sup>1</sup>, C G Ryan<sup>2</sup>, R Kirkham<sup>2</sup>, S Hu<sup>2</sup>, D P Siddons<sup>3</sup>, P A Dunn<sup>2</sup>, A Kuczewski<sup>3</sup>, D Parry<sup>2</sup>, F Rudzik<sup>1</sup>, R Szymanski<sup>1</sup> and D N Jamieson<sup>1</sup>

<sup>1</sup>School of Physics, University of Melbourne, Melbourne, VIC, Australia
<sup>2</sup>CSIRO, Normanby Road, Clayton, VIC, Australia
<sup>3</sup>Brookhaven National Laboratory, Upton, NY, USA

In this paper we outline the MAIA detector installation and commissioning on the high excitation quintuplet Nuclear Microprobe (NMP) at the University of Melbourne and demonstrate some of its key advantages on geological samples. With its 384 pad segmented design and a hole for the beam to pass through its centre, solid-angles approaching 1.3 sr [1,2] mean count rates are orders of magnitude higher than typical PIXE configurations. Dedicated on-board signal processing for the array combined with the sheer number of pixels translates into minimal dead-time losses and pileup distortion of the spectra even at event count rates approaching 10 M/s. Real-time processing and spectral de-convolution [3] produces PIXE elemental images up to 100 M pixels in size enabling the ability to cater for large area PIXE analysis, a feature sorely needed for many biological and geological samples. Coupled with the HYMOD DAQ36 system described previously [4], various stage and beam scanning modes allow one to cater for a variety of sample configuration and experimental needs. Here we demonstrate the system on thin sections made from geological samples extracted from active deep sea hydrothermal vents in the PACMANUS hydrothermal field in Papua New Guinea.

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# P39 MULTI-ENERGY IBA TO STUDY SOLAR ENERGY CONVERSION MATERIALS

V Corregidor<sup>1</sup>, A Barreiros<sup>2</sup> and L C Alves<sup>3</sup>

<sup>1</sup>IPFN, IST-UL, Campus Tecnológico e Nuclear, EN 10, 2695-066 Sacavém, Portugal <sup>2</sup>Laboratório Nacional de Energia e Geologia, LEN/UES, Estrada do Paço do Lumiar 22, 1649-038 Lisboa, Portugal <sup>3</sup>C2TN, IST-UL, Campus Tecnológico e Nuclear, EN 10, 2695-066 Sacavém, Portugal

There is a wide range of materials able to convert the solar energy reaching the Earth into the electrical energy needed by all of us. Among them, silicon is the dominant material with multiple technologies developed around it in order to: (i) increase the efficiency, and (ii) reduce the amount of material by using thinner wafers (usage for silicon cells has been reduced significantly during the last twelve years from around 16 g/Wp to less than 6 g/Wp).

CdTe is also another semiconductor widely used, especially for thin film solar cells. Thin film technology can also be found for CIGS materials (CuInGaSe) with the extra advantage of their application in flexible substrates.

All these materials are available in the photovoltaic market and at the same time a lot of research is on-going in order to increase the cell efficiency not only in lab conditions, but also under real conditions.

Besides these materials, a new one has recently appeared, namely the metal-halide perovskites, with very promising efficiency numbers and relatively easy and cost-efficient production. Perovskites materials are in the front row of research, and it is believed to be a good alternative in the future.

All these materials (Si, CdTe, CIGS and perovkites) have been analyzed by IBA techniques using a microbeam, in order to obtain (i) the elemental distribution (in depth and surface), and (ii) defects introduced during the growth, in order to explain and improve the efficiency numbers obtained by other techniques in lab.

Since different materials and distribution were studied, specific experimental conditions should be optimized: type of ion, beam energy, etc. Examples of the results obtained in a single region of interest when analyzed by WiNDF using the Total IBA approach will be presented.

# P40 EVOLUTION OF IMPURITIES IN METALS USED IN CULTURAL HERITAGE OBJECTS

V Corregidor<sup>1</sup>, J Cruz<sup>2</sup> and L C Alves<sup>3</sup>

<sup>1</sup>IPFN, IST-UL, Campus Tecnológico e Nuclear, EN 10, 2695-066 Sacavém, Portugal <sup>2</sup>Laboratório de Instrumentação, Engenharia Biomédica e Física da Radiação (LIBPhys-UNL), Departamento de Física, Faculdade de Ciências e Tecnologia da Universidade Nova de Lisboa, Monte da Caparica, 2892-516 Caparica, Portugal <sup>3</sup>C2TN, IST-UL, Campus Tecnológico e Nuclear, EN 10, 2695-066 Sacavém, Portugal

Most of the cultural heritage objects are made of metals or alloys such as gold, iron, silver, copper or brass. In general, knowing the composition of an object is extremely important for conservation purposes, dating or attribution processes, considering that along centuries the fabrication procedures and the elements used were modified.

The trace element concentration can, in some cases, indicate the provenance of the ore or relate it with other objects of the same type. Guerra showed [1,2] how it is possible (by using the proper proton energy beam) to determine the origin and provenance of gold, even to track the circulation of gold in the past.

In this work, several silver pieces were characterized by using the nuclear microprobe available at CTN/IST. The pieces, from different centuries, are all Portuguese but manufactured in different places and with different ores.

Results shown that impurity concentration changes not only along the centuries but, also, with the manufacturing place.

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# P41 DEPTH PROFILING OF ORGANIC ELECTRONIC SYSTEMS WITH 3D ORBISIMS

L Matjačić, R Havelund and L S Gilmore

National Physical Laboratory, NiCE-MSI, Teddington, Middlesex, UK

The 3D OrbiSIMS instrument [1] comprises an Orbitrap<sup>TM</sup> and a TOF analyser together with  $Bi_n^+$  and  $Ar_n^+$  ion beam sources. The combination of dual ion beam and dual mass analyser enables ten modes of operation for spectrometry, depth profiling, 2D and 3D imaging with different combinations of single or dual ion beam modality, and single or dual mass analysers. The Orbitrap<sup>TM</sup> mass analyser gives unprecedented capabilities for chemical imaging and depth profiling with mass resolution of >240,000 at m/z 200 and better than ppm mass accuracy while offering a MS/MS capability. The 3D OrbiSIMS instrument can achieve high spatial resolution 3D images with ~5 nm depth resolution and as such can be applied to many different research areas ranging from life sciences [2] to organic electronics [3] and beyond.

In this work, we will explore the use of 3D OrbiSIMS capabilities for the analysis of organic layers and investigate critical parameters related to the secondary ion generation, extraction and detectability. The effect of cluster size and energy on the molecular and fragment ion yields will be studied and the optimal ion beam conditions for the analysis of organic layers determined. Moreover, we will evaluate the influence of the target potential on the ion extraction and C-trap accumulation time on the detectability of secondary ions.

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#### P42 OPTIMISATION OF AMBIENT PRESSURE MeV SIMS

Lidija Matjačić<sup>1</sup>, Vladimir Palitsin<sup>2</sup>, Julien Demarche<sup>3</sup>, Elis Moura Stori Rosa<sup>2</sup>, Lucio dos Santos Rosa<sup>2</sup> and Roger Webb<sup>2</sup>

<sup>1</sup>National Physical Laboratory, NiCE-MSI, Teddington, Middlesex, UK <sup>2</sup>Surrey Ion Beam Centre, University of Surrey, Guildford, GU2 7XH, UK <sup>3</sup>International Atomic Energy Agency, Vienna International Centre, PO Box 100, Austria

Ambient Pressure MeV SIMS (AP MeV SIMS) is a special application of MeV SIMS technique for molecular detection and imaging under fully ambient conditions. There are several advantages of using an ambient over non-ambient technique such as minimising or completely avoiding sample preparation which implies both decreased cost and time consumption for the analysis and avoiding negative vacuum influence on samples. The emergence of ambient mass spectrometry techniques over the past decade has been enormous with a broad range of applications such as food quality, environmental analysis and life sciences [1-3]. The disadvantage of ambient pressure mass spectrometry techniques is the influence of ambient background which can suppress signal from the target. The competition between ambient and target peaks is very pronounced in AP MeV SIMS, as only a few sputtered species get ionised.

In the University of Surrey Ion Beam Centre, an extensive work on optimisation of AP MeV SIMS had been carried out. Optimisation parameters have been grouped into three different categories, depending on whether they are addressing optimisation of secondary ion yield of transmission and detection of secondary ions. A great volume of the optimisation process has tackled an issue of an immense background contribution by investigation of its identity and origin. Moreover, the atmosphere encompassing the sampling area was investigated and the effect of different angles and types of a sheath gas directing the sample was tested. In this work, we will give an overview of the optimisation process.

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# P43 COUPLING OF AP MeV SIMS AND HIPIXE FOR SIMULTANEOUS MOLECULAR AND ELEMENTAL MAPPING IN AIR

L Matjačić<sup>1</sup>, V Palitsin<sup>2</sup>, G W Grime<sup>2</sup>, A Abdul-Karim<sup>3</sup> and R P Webb<sup>1</sup>

<sup>1</sup>National Physical Laboratory, NiCE-MSI, Teddington, Middlesex, UK <sup>2</sup>Surrey Ion Beam Centre, University of Surrey, Guildford, GU2 7XH, UK <sup>3</sup>Cranfield University, Cranfield, MK43 0AL, UK

The use of MeV for secondary ion mass spectrometry (MeV SIMS) presents a major breakthrough for the IBA community, offering molecular characterisation to the wide range of elemental IBA applications [1-3]. Large surface molecules (up to hundreds of kDa) are desorbed under MeV heavy primary ions irradiation. Focusing these beams enables the molecular imaging at the submicron scale, in conjunction with the usual IBA techniques such as  $\mu$ -PIXE or  $\mu$ -RBS [4]. The simultaneous use of multiple IBA techniques has already been reported. RBS can be used to provide matrix and charge correction to improve the quantitative accuracy of PIXE analysis. More recently RBS, PIXE, PIGE (and possibly ERD) have been combined to create what has become known as the Total IBA method [5]. However, there are not that many cases where MeV SIMS has been combined with some other IBA techniques.

Here we present a study of the feasibility of coupling AP MeV SIMS and external HIPIXE. This is a novel combination of IBA techniques which enables information on molecular and elemental composition to be obtained simultaneously. The simultaneous use of AP MeV SIMS and HIPIXE faces the challenge of finding optimal experimental conditions such as the ion beam species, energy and current which would be most suitable for the sputtering of secondary ions and the excitation of X-rays. In this preliminary study, we will present molecular and elemental maps collected using 8 MeV O<sup>4+</sup> ion beam.

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# P44 THE NEW SPACED TRIPLET BEAMLINE AT THE SURREY ION BEAM CENTRE

Vladimir Palitsin and Geoffrey W Grime

Surrey Ion Beam Centre, University of Surrey, Guildford, Surrey, UK

A new microbeam line has recently been constructed at the University of Surrey Ion Beam Centre. This line uses a spaced triplet configuration of Oxford Microbeams quadrupole lenses in which the spacing of the first two lenses can be easily adjusted and novel beam defining apertures consisting of laser-machined apertures in tantalum foil mounted on motorised XY positioning stages. For the object aperture the holes are rectangular with a 4:1 aspect ratio (matching the demagnification ratio of the lens) while the collimator apertures are circular in order to reduce the influence of the spherical aberration cross-terms.

This paper describes the beamline and presents some initial experience of using the system.

# P45 ANALYSIS OF FORENSIC TRACES USING DIRECT ANALYTE-PROBED NANOEXTRACTION MASS SPECTROMETRY (DAPNE-MS) AND ION BEAM ANALYSIS (IBA)

Holly-May Lewis<sup>1</sup>, Roger Webb<sup>2</sup>, Guido Verbeck<sup>3</sup>, Josephine Bunch<sup>4</sup>, Janella de Jesus<sup>5</sup>, Catia Costa<sup>2</sup>, Vladimir Palitsin<sup>2</sup>, Primož Pelicon<sup>6</sup> and Melanie Bailey<sup>5</sup>

<sup>1</sup>Advanced Technology Institute, University of Surrey, Guildford, UK
 <sup>2</sup>Surrey Ion Beam Centre, University of Surrey, Guildford, UK
 <sup>3</sup>University of North Texas, USA
 <sup>4</sup>National Physical Laboratory, Teddington, UK
 <sup>5</sup>Department of Chemistry, University of Surrey, Guildford, UK
 <sup>6</sup>Jožef Stefan Institute, Ljubljana, Slovenia

Direct Analyte-Probed nanoextraction mass spectrometry (DAPNe-MS) is a spatially resolved technique which has the potential to analyse discrete areas of a sample to determine the molecular composition. DAPNe, which is coupled with nanospray ionization mass spectrometry (NSI-MS), uses a capillary tip filled with solvent which is placed in a nanomanipulator. An area of interest is found using a microscope and the capillary tip is positioned above an area of interest on a surface. The solvent is injected onto the sample, the analytes are dissolved and then the solvent is re-aspirated into the tip. The tip is then placed on a nanospray ionisation source and a mass spectrum is acquired. Due to the discrete extraction area, DAPNe causes minimal destruction to the sample. Although DAPNe-MS is a very sensitive technique, no elemental information is given. Like DAPNe, ion beam analysis is spatially resolved, and is non-destructive to the sample. The technique can successfully map the elemental composition of a sample however, gives no molecular information. The combination of these two techniques gives potential to simultaneously map the elemental and molecular composition of discrete areas of sample whilst causing minimal destruction.

In this project, DAPNe-MS and IBA were combined and applied to forensic trace evidence requiring spatially resolved analysis; in particular automotive paint and hair. IBA was used to determine the elemental composition of a sample's surface to find areas of interest. Once these areas have been found, DAPNe was used to provide molecular speciation of localised areas on the surface of the sample. With both techniques being sensitive to trace elements and molecules respectively, there is potential when analysing many other trace forensic samples. The project successfully distinguished between car paints from different manufacturers through both elemental and chemical differences in the paint layers. DAPNe-MS was also used to extract cocaine from the cortex of a drug user's hair that had previously been mapped using MeV SIMS.

# P46 PRELIMINARY STUDY OF GLASS PRODUCTION REMAINS FROM A SPANISH GLASS FURNACE DATED TO THE 16TH CENTURY

Inês Coutinho<sup>1,2</sup>, Luís C Alves<sup>3</sup>, Isabel Cambil Campaña<sup>4</sup> and Teresa Medici<sup>2</sup>

<sup>1</sup>Department of Conservation and Restoration, FCT NOVA Lisboa, Campus de Caparica, Caparica 2829-516, Portugal <sup>2</sup>Research Unit VICARTE, Vidro e Cerâmica para as Artes, FCT NOVA Lisboa, Campus de Caparica, Caparica 2829-516, Portugal <sup>3</sup>C2TN, Instituto Superior Técnico, Universidade de Lisboa, Bobadela LRS 2695-066, Portugal

<sup>4</sup>Restorer of Works of Art and Entrepreneur

A set of twelve glass samples and two glass/crucible remains from a 16th century glassmaking waste deposit excavated in Granada, Spain, was characterized by  $\mu$ -PIXE. This preliminary study constitutes the first analytical approach to glass remains from a Spanish production place dated to the early-modern period.

 $\mu$ -PIXE allowed for the quantification of major, minor and some trace elements of the glass fragments. It also allowed for mapping the elemental distribution of the remains that were identified as an interface of crucible/glass. This analysis constitutes an evaluation of the ionic exchange between glass and crucible.

The glass colours vary from the natural green and blue hues to completely colourless samples. The results show that from the twelve analysed samples of glass, eleven are of soda lime silicate composition, and only one proved to be of a potassium lime silicate composition. From this one can hypothesize that in this location a glass rich in sodium which followed the Mediterranean tradition was produced, and also a potassium-rich glass, which followed a central and north European glass production tradition was part of the production. Since this location was known as *la calle Horno del Vidrio* (Glass Furnace Street) it is possible that different structures were used to produce different glass types.

These results show that a deeper study is needed to characterize the glass production in Granada and comparisons with coeval glass found in the Portuguese territory will be made to identify possible connections between the two countries.

## P47 THE RESPONSE OF GENETICALLY DIFFERENT GLIOBLASTOMA CELL LINES TO VERTICAL AND HORIZONTAL PROTON IRRADIATION

Natalie Mayhead, Lisianne Meira and Roger Webb

Surrey Ion Beam Centre, University of Surrey, Guildford, UK

Glioblastoma is an aggressive brain cancer with a median survival rate of 14.6 months post diagnosis. Treatments for glioblastoma include surgery, radiotherapy, and chemotherapy with the alkylating agent temozolomide (TMZ). In 50% of patients, TMZ treatment is ineffective due to the reparative action of the protein O<sup>6</sup>-MeG DNA methyltransferase (MGMT). The base excision repair (BER) pathway repairs the most common lesions caused by TMZ.

This work reports the characterization of several glioblastoma cell lines in terms of their repair status and sensitivity to traditional therapy of X-ray irradiation and TMZ. We find that the expression of BER proteins differed between cell lines, with alkyladenine-DNA-glycosylase (AAG) showing the greatest variation in expression. Sensitivity to TMZ and X-rays was MGMT dependent. As such a comparison to proton therapy survival was explored.

Two different proton irradiation systems were analysed and developed within this work, leading to a high-throughput broadbeam irradiation system. These differences in methodologies lead to differences being detected in response to proton irradiation depending on the method used. This might in future, lead to further understanding of low-dose hypersensitivity.
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