

# **18<sup>th</sup> INTERNATIONAL CONFERENCE ON NUCLEAR MICROPROBE TECHNOLOGY AND APPLICATIONS** 11-16.09.2022

STRA

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Sund	ay, 11.9.2022	Moi	nday, 12.09.2022	Tue	sday, 13.09.2022	Wedr	nesday, 14.09.2022	Thursday, 15.09.2022		Friday, 16.09.2022	
		08.00-	Registration	08.00- 09.00	Registration	08.00-	Registration				
		09.00-	Welcome Address	09.00-	Session: Radiation	09.00-	Session: Multimodal	09.00-	Session:	09.00-	Session:
		09.15	Welcome Address	10.30	Biology	10.30	Imaging	10.30	Instrumentation III	10.30	Micromodifications
		09.15- 10.30	Session: Instrumentation I	09.00- 09.30	Invited: Tim Schneider (1111)	09.00- 09.30	Bogdanović Radović (1100)	09.00- 09.30	Invited: Shigeo Matsuyama (1096)	09.00- 09.30	Invited: Jeroen Van Kan (1113)*
		09.15- 09.45	Invited I: Dou Yanxin (1081)*	09.30- 09.45	Contributed I: Sarah Rudgikeit (1008)	09.30- 09.45	Contributed I: Melanie Bailey (1018)	09.30- 09.45	Contributed VII: Georgios Provatas (1087)	09.30- 09.45	Contributed I: Gyorgy Vizkelethy (1027)
		09.45- 10.00 10.00- 10.15	Contributed I: Vladimir Palitsin (1022)	09.45- 10.00	Contributed II: Pierre Beaudier (1031)	9.45- 10.00	Contributed II: Catia Costa (1001)	09.45- 10.00	Contributed VIII: Hongjin Mou (1011)*	09.45- 10.00	Contributed II: Hidetaka Hayashi (1034)*
			Contributed II: Gyula Nagy (1030)	10.00- 10.15	Contributed III: Teresa Pinheiro (1036)	10.00- 10.15	Contributed III: Ebrahim Golami Hatam (1026)*	10.00- 10.15	Contributed IX: Iva Božičević Mihalić (1089)	10.00- 10.15	Contributed IV: Guanghua Du (1092)*
		10.15- 10.30	Contributed III: Robert J. W. Frost (1010)	10.15- 10.30	Contributed IV: Ce- Belle Chen (1041)	10.15- 10.30	Contributed IV: Marjana Regvar (1082)	10.15- 10.30	Contributed X: Žiga Šmit (1045)	10.15- 10.30	
		10.30- 11.00	Coffee Break	10.30- 11.00	Coffee Break	10.30- 11.00	Coffee Break	10.30- 11.00	Coffee Break	10.30- 11.00	Coffee Break
		11.00- 12.30	Session: Environment and Cultural Heritage	11.00- 12.30	Session: MeV-SIMS	11.00- 12.45	Session: Detectors	11.00- 12.30	Session: Space	11.00- 11.30	Johnny F. Dias: Conference Summary and Highlights
		11.00- 11.30	Invited I: Lorenzo Giuntini (1067)*	11.00- 11.30	Invited: Zravko Siketić (1028)	11.00- 11.30	Invited: Marco Petasecca (1035)	11.00- 11.30	Invited I: Jafar Shojaii (1047)	11.30- 11.45	Best Posters Award Ceremony
		11.30- 11.45	Contributed I: Maria Dolores Ynsa (1019)	11.30- 11.45	Contributed I: Boštjan Jenčič (1102)	11.30- 11.45	Contributed I: Greta Andrini (1004)	11.30- 12.00	Invited II: Guanghua Du (1084)*	11.45- 12.00	Concluding Remarks
		11.45- 12.00- 13.30 13.30- 15.15	Contributed II: Marta Magalini (1056)	11.45- 12.00	Contribued II: Toshio Seki (1105)*	11.45- 12.00	Contributed II: Ettore Vittone (1032)	12.00- 12.15	Contributed I: Željko Pastuović (1038)		
			Conference Photo and Lunch	12.00- 13.30	Lunch	12.00- 12.15	Contributed III: Andreo Crnjac (1062)	12.15- 12.30	Contributed II: Stefania Peracchi (1075)*		
			Session: Life Sciences	13.30- 15.00	Session: Quantum Technologies	12.30- 13.30	Lunch	12.30- 13.30	Lunch		
		13.30- 14.00	Invited I: Geoffrey W. Grime (1020)	13.30- 14.00	Invited: Andrew A. Bettiol (1110)				Round Table I (Kolarjeva Lecture		
		14.00- 14.30	Invited II: Tilo Reinert (1106)	14.00- 14.15 Contributed I: P. Räcke (1063) <sup>1</sup> 14.15- 14.30 Contributed II Nicholas Collir (1072)	Contributed I: Paul Räcke (1063)*			13.30- 15.00	W. Grime: Data Acquisition	5	
		14.30- 14.45	Contributed I: Richard Ortega (1003)		Contributed II: Nicholas Collins (1072)				Round Table II (IPS		
		14.45- 15.00	Contribued II: Henrique Fonteles (1007)	ll: eles Harry 44)					Željko Pastuović (High Precision	9	
		15.00- 15.15	Contributed III: Harry J. Whitlow (1044)						Irradiation, Single lon Detection)		
		15.15- 15.45	Coffee Break	14.30		13.30-	Excursion: Postojna Cave and			14.00- 16.00	Lab Visit
		15.45-	Session:								
		16.45	Instrumentation II	14.30- 17.00	Poster Session I		Predjama Castle				
		16.15	Corregidor (1090)					15.00- 17.30	Poster Session II		
		16.15-	Contributed IV: Kristina Isaković					17.50			
		16.30	(1048)							L	
18.00	Registration	16.30- 16.45	Contributed V: Gary A. Glass (1098)*								
		16.45- 17.00	Contributed VI: Barney Doyle (1112)*								
21.00 and Reception		40.55							Conference		
		18.30- 19.30	Visit Ljubljana		Public Lecture: Geoffrey W			17.30-	7.30- Dinner: Grad		
		18	18.30- Grime (Town Hall,				Otočec				
		19.30-	0- Town Hall Reception		Ljubljana)						
				20.00-	IAC & PC meeting						

\*indicates on-line talk

## PROGRAMME

## 11 SEPTEMBER, SUNDAY

18.00-21.00 Registration and Reception – IJS Lecture hall, Jamova cesta 39

## SCIENTIFIC PROGRAMME

12 SEPTEMBER, MONDAY

IJS Lecture hall, Jamova cesta 39

08.00-09.00 Registration					
09.00-09.15 Welc	ome Address				
09.15-10.30 Sessi	on: Instrumentation I				
09.15-09.45	Invited I: Dou Yanxin (1081)				
09.45-10.00	Contributed I: Vladimir Palitsin (1022)				
10.00-10.15	Contributed II: Gyula Nagy (1030)				
10.15-10.30	Contributed III: Robert J. W. Frost (1010)				
10.30-11.00	Coffee Break				
11.00-12.30 Sessi	on: Environment and Cultural Heritage				
11.00-11.30	Invited I: Lorenzo Guintini (1067)				
11.30-11.45	Contributed I: Maria Dolores Ynsa (1019)				
11.45-12.00	Contributed II: Marta Magalini (1056)				
12.00-12.15	Conference Photo				
12.15-13.30	Lunch				
13.30-15.15 Sessi	on: Life Sciences				
13.30-14.00	Invited I: Geoffrey W. Grime (1020)				
14.00-14.30	Invited II: Tilo Reinert (1106)				
14.30-14.45	Contributed I: Richard Ortega (1003)				
14.45-15.00	Contributed II: Henrique Fonteles (1007)				
15.00-15.15	Contributed III: Harry J. Whitlow (1044)				
15.15-15.45	Coffee Break				
15.45-17.00 Instru	mentation II				
15.45-16.15	Invited II: Victoria Corregidor (1090)				
16.15-16.30	Contributed IV: Barney Doyle (1112)				
16.30-16.45	Contributed V: Gary A. Glass (1098)				
16.45-17.00	Contributed VI: Kristina Isaković (1048)				
18.00-19.00	Visit Ljubljana (in front of Ljubljana Turistic Information Centre,				
	Adamič-Lundrovo nabrežje 2, Ljubljana)				
19.00-20.00	Reception in a City Hall (Mestni trg 1, Ljubljana)				

# 13 SEPTEMBER, TUESDAY

### IJS Lecture hall, Jamova cesta 39

08.00-09.00 Regist	tration
09.00-10.30 Sessio	on: Radiation Biology
09.00-09.30	Invited I: Tim Schneider (1111)
09.30-09.45	Contributed I: Sarah Rudgikeit (1008)
09.45-10.00	Contributed II: Pierre Beaudier (1031)
10.00-10.15	Contributed III: Teresa Pinheiro (1036)
10.15-10.30	Contributed IV: Ce-Belle Chen (1041)
10.30-11.00	Coffee Break
11.00-12.00 Sessio	on: MeV-SIMS
11.00-11.30	Invited I: Zdravko Siketi <b>ć</b> (1028)
11.30-11.45	Contributed I: Boštjan Jenčič (1102)
11.45-12.00	Contributed II: Toshio Seki (1105)
12.00-13.30	Lunch
13.30-14.30 Sessio	on: Quantum Technologies
13.30-14.00	Invited I: Andrew A.Bettiol (1110)
14.00-14.15	Contributed I: Paul Räcke (1063)
14.15-14:30	Contributed II: Nicholas Collins (1072)
14.30-17.00	Posters I
18.30	Public Lecture: Geoffrey W. Grime (Ljubljana Town Hall)
20.00	IAC & PC meeting (Skuhna, Trubarjeva 56, Ljubljana)

## 14 SEPTEMBER, WEDNESDAY

IJS Lecture hall, Jamova cesta 39

08.00-09.00	Regist	ration
09.00-10.30	Sessio	on: Multimodal Imaging
09.00-	09.30	Invited I: Iva Bogdanovi <b>č</b> Radovi <b>č</b> (1100)
09.30-	09.45	Contributed I: Melanie Bailey (1018)
09.45-	10.00	Contributed II: Catia Costa (1001)
10.00-	10.15	Contributed III: Ebrahim Golami Hatam (1026)
10.15-	10.30	Contributed IV: Marjana Regvar (1082)
10.30-	11.00	Coffee Break
11.00-12.30	Sessio	on: Detectors
11.00-	11.30	Invited I: Marco Petasecca (1035)
11.30-	11.45	Contributed I: Greta Andrini (1004)
11.45-	12.00	Contributed II: Ettore Vittone (1032)
12.00-	12.15	Contributed IV: Andreo Crnjac (1062)
12.30-	13.30	Lunch
13.30-		Excursion to Postojna Cave and Predjama Castle

## 15 SEPTEMBER, THURSDAY

IJS Lecture hall, Jamova cesta 39

09.00-10.30 Sessio	on: Instrumentation III
09.00-09.30	Invited I: Shigeo Matsuyama (1096)
09.30-09.45	Contributed VII: Georgios Provatas (1087)
09.45-10.00	Contributed VIII: Hongjin Mou (1011)
10.00-10.15	Contributed IX: Iva Božičević Mihalić (1089)
10.15-10.30	Contributed X: Žiga Šmit (1045)
10.30-11.00	Coffee Break
11.00-12.30 Sessio	on: Space
11.00-11.30	Invited I: Jafar Shojaii (1047)
11.30-12.00	Invited II: Guanghua Du (1084)
12.00-12.15	Contributed I: Željko Pastuović (1038)
12.15-12.30	Contributed II: Stefania Peracchi (1075)
12.30-13.30	Lunch
13.30-15.00	Round Table I (Kolarjeva Lecture Room): Geoffrey W. Grime: Data Acquisition
	Round Table II (IPS Lecture Room): Željko Pastuović: High Precision Irradiation, Single Ion Detection
15.00-17.30	Posters II
17.30-	Conference Gala Dinner (Grad Otočec, Grajska cesta 2, Otočec)

## 16 SEPTEMBER, FRIDAY

### IJS Lecture hall, Jamova cesta 39

09.00-10.30	Sessio	n: Micromodiffications
09.00-	09.30	Invited: Jeroen Van Kan (1113)
09.30-	09.45	Contributed I: Gyorgy Vizkelethy (1027)
09.45-	10.00	Contributed II: Hidetaka Hayashi (1034)
10.00-	10.15	Contributed III: Anusmita Chakravorty (1068)
10.15-	10.30	Contributed IV: Guangbo Mao (1092)
10.30-	11.00	Coffee Break
11.00-	11.30	Invited: Johnny F. Dias: Conference Summary and Highlights
11.30-	11.45	Award Ceremony
11.45-	12.00	Concluding remarks
14.00-	16.00	Lab Visit

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### MONDAY, 12 SEPTEMBER

### **SESSION 1**

### **INSTRUMENTATION I**

Chairs: Andrew A. Bettiol and Klemen Bučar

### Invited talk

### 1081

# The Singapore quadruplet: a focusing lens configuration for high resolution nuclear microscopy

Yanxin Dou<sup>1, 2</sup>, Thomas Osipowicz<sup>1</sup>, Jeroen Anton van Kan<sup>1</sup>

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Beam optics of a focusing lens configuration plays a key role in determining the performance of a nuclear microscope system. However, only a few focusing lens configurations were shown to be capable to reach resolutions smaller than 200 nm [1-3]. Here, we propose a quadrupole lens configuration (namely the Singapore quadruplet) with high resolutions. Results of numerical studies of this beam optics designs are presented, using the FANM software [4], and a comparison of the Singapore quadruplet and other typical configurations is shown in this work. Preliminary experimental results of the Singapore quadruplet configuration are discussed, with a resolution of 80×130 nm<sup>2</sup>.

- [1] Watt F, Van Kan J A, Rajta I, et al. The National University of Singapore high energy ion nano-probe facility: Performance tests[J]. Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms, 2003, 210: 14-20.
- [2] Jamieson D N. New generation nuclear microprobe systems. Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms, 2001, 181(1-4): 1-11.
- [3] Spemann D, Reinert T, Vogt J, et al. Materials analysis and modification at LIPSION–Present state and future developments[J]. Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms, 2011, 269(20): 2175-2179.
- [4] Dou Y, van Kan J A. FANM: A software for focus and aberrations of nuclear microprobe[J]. Ultramicroscopy, 2021, 220: 113163.

### **INSTRUMENTATION I**

#### Contributed talk I

### 1022

# Recent developments on the micro-PIXE beamline at the University of Surrey Ion Beam Centre

Vladimir Palitsin<sup>1</sup>, Catia Costa<sup>1</sup>, Pierre Couture<sup>1</sup>, Roger Webb<sup>1</sup>, Geoffrey Grime<sup>1</sup>

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The microbeam line at the University of Surrey Ion Beam Centre has been a key resource supporting the analytical activities of the Centre for two decades. The beamline has recently been upgraded to provide significant enhancements in performance and ease of use. These include the use of a spaced quadrupole triplet configuration using object and collimator apertures mounted on motorised ladders and a high stability piezo-electric sample stage with rapid-access zero loading force sample holders.

The tantalum object apertures are fabricated using a combination of wire erosion and FIB polishing to provide a high-quality low scattering finish. The sample stage incorporates a vertical axis of rotation which will allow us to explore new characterisation methods such as topographic imaging using stereoscopic pairs, 3-D imaging using IBA-tomography and depth profiling using multiple-angle PIXE.

This presentation describes the new features. Illustrative results obtained using the new beamline will be presented.

## **INSTRUMENTATION I**

### Contributed talk II

### 1030

### Present status of the Uppsala scanning nuclear microprobe

### Gyula Nagy<sup>1</sup>, Harry J. Whitlow<sup>2</sup>, Daniel Primetzhofer<sup>1,2</sup>

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In this presentation we give a status report about the Uppsala scanning nuclear microprobe, as well as an overview of some recent applications. As a prominent example, we demonstrated the capability of Elastic Backscattering Spectrometry ( $\mu$ -EBS) to non-destructively depth-profile the elemental composition of intact MOF (Metal-Organic Framework) single crystals [1].

Besides the applications, we also give an overview about the status of the microprobe facility from a technical point of view. The Uppsala scanning nuclear microprobe, named SLIM-UP (Scanning Light Ion Microprobe in Uppsala), was originally installed in The Svedberg Laboratory, Uppsala University, during 1989/90 [2]. The main components of the facility, i.e. the object forming apertures, collimator apertures, magnetic deflection unit, probe forming quadrupole lenses and magnet driving power supplies were the first ones that were sold by Oxford Microbeams Ltd. Since then, the microprobe has undergone several minor and major modifications. The present configuration is a rebuild of the SLIM-UP, now attached to the 5 MV tandem pelletron accelerator (NEC) of the Tandem Laboratory, at the Ångström Laboratory, Uppsala University. We present a detailed description of the components, the most recent technical developments, as well as the most recent performance tests.



Figure 1: The Uppsala scanning nuclear microprobe.

[1] B. D. McCarthy et al., J. Am. Chem. Soc. 143 (2021) 18626-18634. doi: https://doi.org/10.1021/jacs.1c08550 [2] T. Sunde, J. Nyström, U. Lindh, NIM B 54 (1991) 80-83. doi: https://doi.org/10.1016/0168-583X(91)95495-Y

### 1010

# A study of luminescent materials for application in the beam imaging system at the ESS

C. Thomas<sup>1</sup>, <u>R.J.W. Frost<sup>2</sup></u>, R. Johansson<sup>1</sup>, M. Hartl<sup>1</sup>, K. Michel<sup>1</sup>, H. Kocevar<sup>1</sup>, M. Elfman<sup>2</sup>, S. Joshi<sup>3</sup>, S. Björklund<sup>3</sup>

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- <sup>2</sup> Department of Physics, Lund University, Lund, Sweden
- <sup>3</sup> Department of Engineering Science, University West, Trollhätten, Sweden

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Scintillating screens are frequently used in the diagnostic systems of particle accelerators [1]. The European Spallation Source [2], will incorporate a linear accelerator which will accelerate protons up to 2 GeV with and average power of 5 MW. The beam imaging system is therefore critical for the safe operation of the accelerator, ensuring the beam delivered to the target is always within its nominal envelope. As part of the development of the beam imaging system [3] luminescent screens have been fabricated from  $Al_2O_3$ :Cr and YAG:Ce; these materials being common to the diagnostic systems of other high-energy accelerators [4]. In order to evaluate the lifetime of the luminescent screens, and thus qualify them for production, the screens will be irradiated at the Nuclear Applications Laboratory, Lund University. Irradiation will be performed using a 3 MV Pelletron accelerator, with a proton beam of 2.5 MeV energy and a current of 1  $\mu$ A. The objective of the irradiation is to measure the lifetime of the luminescence as function of the proton dose measured with the total charge received by the samples.

- [1] B. Walasek-Höhne and G. Kube, Scintillating screen applications in beam diagnostics, Proceedings of DIPAC2011, Hamburg, Germany, 2011, pp. 533–557.
- [2] Source European Spallation Source ERIC, Box 176, SE-221 00, Lund, Sweden. https://europeanspallationsource.se.
- [3] M.G. Ibison, C.P. Welsch, E. Adli, H. Gjersdal, G. Christoforo, T. Shea, C. Thomas and D. Naeem, Development of a beam imaging system for the European spallation source tuning dump, Nuclear Instruments and Methods in Physics Research Section A 950 (2020), 162790. doi:10.1016/j.nima.2019.162790.
- [4] K. Renuka, W. Ensinger, C. Andre, F. Becker, P. Forck, R. Haseitl, A. Reiter and B. Walasek-Höhne, Transverse beam profile monitoring using scintillation screens for high energy ion beams, Proceedings of BIW2012, Newport News, VA USA, 2012, pp. 183–185.

### MONDAY, 12 SEPTEMBER

### **SESSION 2**

### ENVIRONMENT AND CULTURAL HERITAGE Chairs: Gastón García López and Žiga Šmit

#### Invited talk

### 1067

# Status of the MACHINA project, the Movable Accelerator for Cultural Heritage In-situ Non-destructive Analysis

Francesco Taccetti <sup>1,2</sup>, <u>Lorenzo Giuntini<sup>1,2</sup></u>, Lisa Castelli<sup>1</sup>, Giulia Calzolai<sup>1</sup>, Massimo Chiari<sup>1</sup>, Caroline Czelusniak<sup>1</sup>, Mariaelena Fedi<sup>1</sup>, Mirko Massi<sup>1</sup>, Pier Andrea Mandò<sup>1,2</sup>, e Mathot<sup>3</sup>, Giovanni Anelli<sup>3</sup>, Alexej Grudiev<sup>3</sup>, Alessandra Lombardi<sup>3</sup>, Eric Montesinos<sup>3</sup>, Maurizio Vretenar<sup>3</sup>

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As it is well known, over the years, we have seen an ever-growing request need for in-situ compositional analyses in the field of Cultural Heritage. Ion Beam Analyses constitute a set of powerful techniques able to provide rich insight into samples. They are most often based on the use of MeV ion beams produced by a particle accelerator. This has limited the number of applications of IBA techniques to Cultural Heritage (CH) problems because, in the vast majority of the cases, artworks are hard to move to accelerator laboratories and no transportable accelerator has been developed so far.

To overcome this limitation, the Italian National Institute for Nuclear Physics (INFN) and the European Organization for Nuclear Research (CERN) have jointly started the MACHINA (Movable Accelerator for Cultural Heritage In-situ Non-destructive Analyses) project for the development of a transportable accelerator system.

The MACHINA project, started at the beginning of 2018 is now in its final stage and first test applications are scheduled for the end of this year, notwithstanding the COVID-19 pandemic and all the connected problems.

After a brief overview of the MACHINA project, we will discuss its evolution from the beginning to present, pointing out problems and results. Expected future activities will close the presentation.

### ENVIRONMENT AND CULTURAL HERITAGE

#### **Contributed I**

### 1019

# Micro-PIXE analysis of metal archaeological pieces of Torre Monreal necropolis

Silvia Viñals<sup>1</sup>, <u>Maria Dolores Ynsa<sup>1</sup></u>, Andres Redondo<sup>1</sup>, Rita Pestana<sup>2</sup>, Berta Balduz<sup>3</sup>, Victoria Corregidor<sup>4</sup>, Joaquin Barrio<sup>5</sup>

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- <sup>2</sup> Universidade de Lisboa Departamento de Física Lisboa-Portugal
- <sup>3</sup> Gobierno de Navarra Institución Príncipe de Viana Pamplona-Spain
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In 2019, the Torre Monreal necropolis was discovered in the urban centre of Tudela, in the north of Spain (Navarra). This Islamic necropolis has a great amount of graves with rich funeral's dowrys, therefore it is of a high archaeological and historical interest. In this work, in order to determine the elemental concentration, different objects, mainly metallic pieces, such as rings, earrings, necklaces, belt buckles, etc. have been analysed by Particle Induced X-ray Emission (PIXE) using the external microbeam line at CMAM (Centro de Microanálisis de Materiales in Madrid). Most of the metallic objects with a gilded look exhibit Au concentrations higher than 50 %, Ag and Hg levels close to 20 %, and low or trace concentrations of other elements such as Cu or Fe. The glass settings of the rings were also analysed. The elemental contains of these pieces have been compared with the other Islamic piece concentrations corresponding to the same historic period, XIII – XIV century, such as those from Calatrava la Vieja collection.

### ENVIRONMENT AND CULTURAL HERITAGE

### **Contributed II**

### 1056

### Improving the protocol for provenance determination of lapis lazuli with new analyses on samples from Myanmar

Laura Guidorzi<sup>1</sup>, <u>Marta Magalini<sup>2</sup></u>, Alessandro Re<sup>2</sup>, Alessandro Lo Giudice<sup>2</sup>, Fulvio Fantino<sup>3</sup>, Alessandro Borghi<sup>4</sup>, Gloria Vaggelli<sup>5</sup>, Matteo Campostrini<sup>6</sup>, Valentino Rigato<sup>6</sup>, Quentin Lemasson<sup>7</sup>, Laurent Pichon<sup>7</sup>, Claire Pacheco<sup>7</sup>, Brice Moignard<sup>7</sup>

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Although the historical source for lapis lazuli used in antiquity is commonly known to be the Badakhshan Province (Afghanistan), other hypotheses have been suggested and their confirmation on a scientific basis could be of uttermost importance for the knowledge of ancient trade routes. In the last years, our group have proposed and successfully applied to lapis lazuli objects a protocol to identify the provenance of the raw material by means of a micro-analytical non-invasive methodology [1]. The analytical protocol is mainly based on a comparison of the physico-chemical properties of rocks of known provenance with archaeological finds and historical objects, obtained by means of lon Beam Analysis (IBA) techniques. In particular, micro-PIXE and micro-ionoluminescence (IL) were used to detect trace elements and characteristic luminescence of some rock-forming minerals in lapis lazuli, specifically diopside, wollastonite and pyrite. The micro-scale of the beam is needed for the micrometric size of the crystals under investigation. The protocol includes so far Afghan, Tajik, Siberian and Chilean provenances.

12 lapis lazuli rock samples were recently acquired in situ from Myanmar. Prepared as semi-thin sections (ca. 100  $\mu$ m), they have been primarily characterized via petrographic and SEM-EDX analyses [2]. The samples have been now investigated with IBA and measurements were carried out on 28 diopside and 7 pyrite crystals. The analyses have been performed both in vacuum at INFN-LNL (Legnaro, Padova, Italy) with 2 MeV protons and with extracted beam at NewAGLAE (Paris, France) using 3 MeV protons. Moreover, calcite and K-feldspar have been recently taken into consideration as new mineral phases to improve the discrimination among Afghan, Tajik and Myanmar provenances.

Micro-IBA investigations allowed to determine similarities and differences of Myanmar samples with respect to other provenances. An updated analytical protocol is proposed to include all five lapis lazuli origins.

- [1] Lo Giudice, A. et al. (2017) Protocol for lapis lazuli provenance determination: evidence for an Afghan origin of the stones used for ancient carved artefacts kept at the Egyptian Museum of Florence (Italy). Archaeological and Anthropological Sciences 9:637-651. doi: 10.1007/s12520-016-0430-0
- [2] Vaggelli G. et al. (2019) Improvements to the analytical protocol of lapis lazuli provenance: First study on Myanmar rock samples. European Physical Journal Plus 134:104. doi: 10.1140/epjp/i2019-12523-4

### MONDAY, 12 SEPTEMBER

### **SESSION 3**

### LIFE SCIENCES

Chairs: Katarina Vogel-Mikuš and Philipe Barberet

### Invited talk I

### 1020

# The identification and quantitation of metal atoms in proteins using microPIXE: a critical evaluation

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Around 30% of all known protein molecules contain a small number of metal atoms as part their structure which are often crucial to the biological function of the molecule. Identifying and quantifying these is not possible using commonly available analysis tools and there is a growing awareness that a majority of the metal atoms in the over 30,000 metalloprotein structures deposited in the Protein Data Bank [1] are misidentified, with major consequences for the interpretation of protein function and mechanism [2,3]. In the 1990s the authors developed a method for the identification and quantification of metals in proteins by combining micro-PIXE and RBS. This has been used to solve many problems in structural biology and has recently been extended to provide high throughput capability [3].

In this contribution we will present some recent results illustrating the power of this method to answer biological questions. We will also give an overview of the methodology and provide a critical evaluation of accuracy, precision and detection limit. Our evaluation will provide guidance on optimisation of sample preparation and analysis conditions for these measurements.

- [1] H.M. Berman, K. Henrick, H. Nakamura Announcing the worldwide Protein Data Bank Nature Structural Biology 10 (12) (2003) 980
- [2] E.F. Garman, G.W. Grime, Elemental analysis of proteins by microPIXE, Progress in Biophysics and Molecular Biology 89/2, (2005) 173
- [3] G.W. Grime, O.B. Zeldin, M.E. Snell, E.D. Lowe, J.F. Hunt, G.T. Montelione, L. Tong, E.H. Snell, E.F. Garman. Highthroughput PIXE as an essential quantitative assay for accurate metalloprotein structural analysis; development and application. J. Am. Chem. Soc., 142 (2020) 185

### Invited talk II

### 1106

### MicroPIXE and the Iron in the Brain

<u>Tilo Reinert<sup>1,2</sup></u>, Malte Brammerloh<sup>1</sup>, Carsten Jäger<sup>1</sup>, Jan Meijer<sup>3</sup>, Primož Vavpetič<sup>4</sup>, Primož Pelicon<sup>4</sup>, Nikolaus Weiskopf<sup>1</sup>, Markus Morawski<sup>2</sup>, Evgeniya Kirilina<sup>1</sup>

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Iron in the brain is still the iron in the fire for a large part of the neuroscience community. One knows for what it is good for: Cellular energy metabolism, axonal myelination, or neurotransmitter synthesis. On the other hand, iron has also its bad side: If the cellular physiological processes can no longer safely handle iron, it will produce oxidative stress and a continuous iron overload condition may lead to neurodegeneration. Therefore, the challenge is to develop methods to analyze and quantify the distribution of iron in the brain, from the sub-cellular to the regional scale and of course all in 3D and in vivo to understand, diagnose and fight neurodegenerative diseases. Here is where microPIXE and MRI (magnetic resonance imaging) come together. MicroPIXE serves as ground-truth method for MRI-based iron-sensitive imaging and iron quantification. However, due to low data acquisition rates, microPIXE has longtime suffered from low sample throughput, which in turn reduces the statistical power of the analytical results. Additionally, the in situ analysis of neurons and glia cells in brain sections is affected by the partial volume effect or overlapping cells.

Based on a study on the iron distribution in the human substantia nigra, this presentation will discuss the pitfalls and uncertainties of the quantification of brain iron by microPIXE, the advantage of large efficiency X-ray detection systems and demonstrate how microPIXE contributes to the development of a biophysical model that allows the assessment of the iron content and neuronal density in the substantia nigra from MRI relaxation rates.

#### Contributed talk I

### 1003

# How PIXE analysis of infant formulas contributed to the re-establishment of a WHO drinking water guideline for manganese

Erika J. Mitchell<sup>1</sup>, Seth H. Frisbie<sup>2</sup>, Stephane Roudeau<sup>3</sup>, Asuncion Carmona<sup>3</sup>, Richard Ortega<sup>3</sup>

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A large body of recent research clearly shows that excess manganese (Mn) exposure is associated with neurological disorders in young children. This includes impaired cognitive development, lower IQ or intelligence scores, increased risk of attention deficit hyperactivity disorder (ADHD), and other cognitive and behavior problems. In response, the World Health Organization (WHO) recently established a guideline for Mn in drinking water to protect infants from excess Mn in reconstituted infant formula [1, 2], based in part on our work [3-5].

We measured the concentrations of Mn in 44 infant formulas and young child nutritional beverages from the United States (US) and France markets using PIXE (Particle Induced X-ray Emission). Micro-PIXE analyses were performed at LP2iB using a 3 MeV proton beam to quantify Mn. Rutherford backscattering spectrometry (RBS) was carried out simultaneously to quantify the sample matrix. Our results suggest that these products contain up to 1,000 times the Mn concentration of breast milk and sometimes exceed internationally recognized tolerable intakes for Mn exposure. Moreover, an additional risk comes from the Mn in the drinking water that is used to reconstitute infant formula. It is very likely that many children from many countries have a great and avoidable risk of neurological impairment from ingesting these products.

The WHO re-established its discontinued guideline for Mn in drinking water in March 2022, with a provisional guideline value of 80  $\mu$ g/L for total Mn 'to be protective against neurological effets in the most sensitive subpopulation – bottle-fed infants – and consequently the general population' [1]. This new provisional guideline value is a very important first step to ensure public health, especially for infants, who are sensitive to neurodevelopmental harm from exposure to excess Mn. The next step is to directly regulate the Mn content of infant formulas which to date is not subject to recommended maximum values.

- [1] World Health Organization. Guidelines for drinking-water quality: Fourth edition incorporating the first and second addenda, WHO, 2022.
- [2] Manganese in drinking-water. Background document for development of WHO Guidelines for drinking-water quality. WHO/HEP/ECH/WSH/2021.5, 2021.
- [3] Frisbie S.H., Mitchell E.J., Roudeau S., Domart F., Carmona A., Ortega R. (2019) Manganese levels in infant formula and young child nutritional beverages in the United States and France: comparison to breast milk and regulations. PLoS ONE, 14(11): e0223636. doi: 10.1371/journal.pone.0223636
- [4] Mitchell E.J., Frisbie S.H., Roudeau S., Carmona A., Ortega R. (2020) Estimating daily intakes of manganese due to breast milk, infant formulas, or young child nutritional beverages in the United States and France: Comparison to sufficiency and toxicity thresholds. Journal of Trace Elements in Medicine and Biology, 62, 126607. doi: 10.1016/j.jtemb.2020.126607
- [5] Mitchell E.J., Frisbie S.H., Roudeau S., Carmona A., Ortega R. (2021) How much manganese is safe for infants? A review of the scientific basis of intake guidelines and regulations relevant to the manganese content of infant formulas. Journal of Trace Elements in Medicine and Biology, 65, 126710. doi: 10.1016/j.jtemb.2020.126710

#### Contributed talk II

### 1007

# Imaging and Analysis of Human Glioblastoma U87 Cells Treated with Cisplatin by $\mu$ -PIXE

<u>Henrique Fonteles<sup>1</sup></u>, Pedro Luis Grande<sup>1</sup>, Johnny Ferraz Dias<sup>1</sup>, Deiverti Bauer<sup>1</sup>, Julia Marcolion<sup>2</sup>, Karine Begnini<sup>2</sup>, Guido Lenz<sup>2</sup>

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The study of chemotherapy drug's properties plays a vital role in the development of new cancerrelated treatments and the improvement of those currently in use. Cisplatin (Pt(NH3)2Cl2) and other platinum-based drugs are widely used medications that treat several different cancers, including, but not limited to, ovarian, testicular, and neck, among others [1].

The present work investigated the interaction of cisplatin with human glioblastoma U87 cells. Cells were treated with cisplatin at a concentration of 10, 20, and 100  $\mu$ M for 24h. The analysis was done using the  $\mu$ -PIXE technique, with a 2.2 MeV proton beam of 1.2 x 1.2  $\mu$ m<sup>2</sup> spot size, which gives us elemental information about the sample and allows us to create 2D maps with micrometric spacial resolution. By measuring the chlorine and platinum signals, we were able to identify individual cells in the sample and detect which ones had a greater cisplatin uptake, respectively. The study of the drug's uptake is crucial for understanding the mechanisms of resistance that are involved in the cisplatin interaction with cancer cells. Moreover, we noticed that the 100  $\mu$ M sample had a high iron signal correlating with the platinum signal, which was not present at lower treatment concentrations. This result could be a piece of evidence for ferroptosis, i.e. a type of iron-dependant cell death completely independent from regular apoptosis. Recent studies have already shown that cisplatin can induce ferroptosis in high treatment concentrations [2].

All experiments were conducted at the Ion Implantation Laboratory with a 3MV Tandem accelerator. This work also highlighted the versatility of the  $\mu$ -PIXE technique and how it can be used to perform interdisciplinary analysis.

- Galanski M. (2006). Recent developments in the field of anticancer platinum complexes. Recent patents on anticancer drug discovery, 1(2), 285–295. doi: 10.2174/157489206777442287
- [2] Guo, J., Xu, B., Han, Q., Zhou, H., Xia, Y., Gong, C., Dai, X., Li, Z., & Wu, G. (2018). Ferroptosis: A Novel Anti-tumor Action for Cisplatin. Cancer research and treatment, 50(2), 445–460. doi: 10.4143/crt.2016.57

#### **Contributed talk III**

### 1044

# Measurement of Ca sequestration molarities in simian nasal mucosa with a MeV ion microprobe

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Ca2+ ions are important intracellular molecular signals in many biological processes, e.g. apoptosis, muscle fibre contraction, neurotransmitter release, cell proliferation, and immune responses. Ca2+ triggers these responses by interactions with calcium binding proteins, after its regulated entry to the cytoplasm. It is therefore of interest to be able to microscopically analyse the molarity of Ca and other heavy element concentrations in tissues as an indicator of normal and disease states. Particle Induced X-ray Emission (PIXE) measurements provide quantitative data in terms of the mass content of an element as a function of the dry mass of the sample. It is non-trivial to relate this to the physiological meaningful unit of the molarity of the element in question in living (wet) tissue. This is because of anatomical differences and different shrinkage during dehydration give rise to significant thickness variations over the sample area. Here we developed procedures to determine the molarity of heavy elements in wet tissue based on simultaneous off-axis Scanning Transmission Ion Microscopy (STIM), PIXE and Rutherford Backscattering Spectrometry (RBS) to measure the local target thickness and major element composition. Incorporating these parameters considerably reduced the spread of lesser life elements (S, P, Cl, K, Ca, Fe, etc.) [Whitlow 2021] between tissue regions in the samples. This new approach was applied to study mucosa tissues which play an important barrier role in the immune defence by secretion of mucous which performs lubricative, hydration, cleaning as well as acting as a selective permeable layer for absorption of gases and nutrients. Mucus is produced as granules within goblet cells with H+ and Ca2+ concentrations are signals that trigger the exocytotic release of mucus from granules. Sections of nasal mucosa tissue from a control and a subject infected with simian immunodeficiency virus (SIV) revealed localised sequestration of Ca in the epithelial tissues with concentration in the 10-30 mmol//L range in the later subject.

## MONDAY, 12 SEPTEMBER

### **SESSION 4**

### **INSTRUMENTATION II**

Chairs: Johnny F. Dias and István Rajta

### Invited talk II

### 1090

### Nuclear microprobe in materials for solar cells devices

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In spite of the large improvements on the fabrication of solar cell devices with very high energy conversion efficiencies, still a better understanding is needed on the relation between structure and device performance. This work is focused on the results obtained by a nuclear microprobe in combination with ion beam analysis (IBA) techniques on the characterization of different materials used as absorber layers in solar cells devices, such as CdTe, nanocrystalline porous TiO2 films as photoanode, organo-metal halide perovskites, and Cu(In,Ga)Se2.

Such different materials were characterized with the nuclear microprobe in Lisbon, adapting the experimental conditions (particles and energy) to the specific challenges of each material. The final aim was to help understanding the limitations of the devices under study and then propose changes in the manufacturing conditions for obtaining optimum performance.

Both, a 2 MeV H+ and He+ focused beams were used to characterize CdTe nano-structured layers on Bi2Te3 seeds. Results showed the importance of the seeds for the later successful growth of the nanowires [1].

In the case of dye synthesized solar cells, one of the challenges was to evaluate the total dye adsorbed on the TiO2 film and its distribution. The IBA techniques were essential to study the distribution of Ru organometallic dye in the photoanode of about 2  $\mu$ m thick [2].

IBA techniques, mainly PIXE and RBS, allowed establishing the dependence of band gap variation of the perovskite films with the changes in the perovskite chemical composition (FA0.83Cs0.17Pb(I(1-x)Brx)3) upon the crystallization process [3].

The nuclear microprobe and IBA techniques were also important tools to characterize the promising Cu(In,Ga)Se2 (CIGS) solar cells, by detecting in-depth inhomogeneities in final devices which act as regions for their performance limitations [4].

Characterization challenges continue as cell structures are becoming more complex, for example: reducing the thickness of the absorber layer, or adding very thin passivation layers between the back contact and the absorber layer. Possible solutions and limitations with be shown and discussed.

- [1] V. Corregidor, L.C. Alves, N. Franco, M.A. Barreiros, N.V. Sochinskii, E. Alves (2013) CdTe nano-structures for photovoltaic devices, Nucl. Instruments and Methods B, 306: 218-221,Doi: 10.1016/j.nimb.2012.11.051.
- [2] M.A. Barreiros, V. Corregidor, L.C. Alves, F. Guimarães, J. Mascarenhas, E. Torres, M.J. Brites (2015) Assessment of dye distribution in sensitized solar cells by microprobe techniques, Nuclear Instruments and Methods B,, 348: 255-259, doi: 10.1016/j.nimb.2014.11.041.
- [3] M.J. Brites, M. A. Barreiros, V. Corregidor, L. C. Alves, J. V. Pinto, M. J. Mendes, E. Fortunato, R. Martins, J. Mascarenhas (2019) Ultrafast Low-Temperature Crystallization of Solar Cell Graded Formamidinium-Cesium Mixed-Cation Lead Mixed-Halide Perovskites Using a Reproducible Microwave-Based Process, ACS Applied Energy Materials, 2: 1844-1853. Doi: 10.1021/acsaem.8b02005
- [4] V. Corregidor, M. A. Barreiros, P.M. P. Salomé, L. C. Alves, In-Depth Inhomogeneities in CIGS Solar Cells: Identifying Regions for Performance Limitations by PIXE and EBS (2021) The Journal of Physical Chemistry C, 125: 16155-16165.

### **INSTRUMENTATION II**

#### Contributed talk IV

### 1112

# Microbeam RBS of Ion Tracks Caused by Single Event Burnout of HV GaN Diodes

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In high-voltage power devices, wide bandgap (WBG) semiconductors such as gallium nitride (GaN) and silicon carbide (SiC) offer significantly improved electrical performance compared to silicon. While most commercial devices utilizing WBG semiconductors involve SiC, GaN has certain advantages over SiC, such as a substantively higher critical electric field (3.3 MV/cm compared to 2.4 MV/cm). In addition, there is evidence that GaN devices are more resilient to select radiation damage modes due to the higher critical electric field, stronger molecular bonds, and other intrinsic material properties and device design practices. Radiation resilience is attractive to engineers designing circuits that must operate reliably in radiation environments. In particular, Single Event Burnout (SEB), characterized by unrecoverable damage, is of concern for devices operating at high voltages. The authors of this work studied SEB in vertical GaN PN diodes fabricated at Sandia using 7.5 and 30 MeV C ions from the Tandem Pelletron in the Ion Beam Laboratory.

These diodes have a 1 micron Au anode followed by 0.4 microns of heavily doped p-type GaN followed by 10 microns of lightly doped n-type GaN and finally a heavily doped n-type substrate and bottom metallization. For an SEB to occur, 1) the ions need to penetrate all the way to the substrate, 2) the ions must provide sufficient ionization, and 3) the bias on the diode must be greater than a threshold value. In our case, SEB occured for the 30 MeV C ion, which ranged out in the substrate on a diode bias voltage of 857V, but the 7.5 MeV C ion, with insufficient range, could not induce an SEB event. On the diodes suffering SEB, a crater also formed around the region where the C strike occured, removing part of the Au anode.

An experiment that timed the SEB event determined that the very first ion that hits in the anode region of the diode produces the SEB. The authors investigated whether the location of the ion impact could be determined and whether it could be validated that it was indeed just a single ion. Using 3D RBS on the 3 MV Pelletron at the IBL, a 2.5 MeV proton beam focused to ~5 microns was scanned across the crater blemish made during the SEB event. Single tracks were revealed in side view as a slight increase in the RBS signal in a straight line, 5 microns wide going >10 microns deep into the GaN on several of the diodes with the largest SEB craters. The authors suggest this increase is due to the decomposition of the GaN along the track into Ga + N2. The N2 migrates away, and the resulting track is 100% Ga and therefore has ~2x the RBS signal as GaN. The radii of such tracks are much smaller than the 5 micron microbeam resolution and that is why there was only a slight increase in the RBS signal that was observed.

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## **INSTRUMENTATION II**

### Contributed talk V

### 1098

### Status of Ion Microprobe activities at the University of North Texas

Todd A. Byers, Cory Nook, Darshpreet Kaur Saini, Charles Bowen, Bibhudutta Rout\*, and <u>Gary A.</u> <u>Glass\*</u>

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We are currently operating a high throughput ion beam focusing system utilizing two pairs of magnetic quadrupole doublets lenses separated at a distance of 85 cm. The system is being built in a microprobe beamline with Louisiana magnetic separated doublet (LMD) lens associated with a 3MV single-ended (NEC, 9SH) accelerator. The focusing system has an orthomorphic demagnification of ~100×100 for a working distance of 18 cm, regularly achieving an optimal spatial resolution of 0.5 µm with a beam current of 50 pA.

We will be presenting experimental results demonstrating the capabilities of the system and its applications in biomedical and environmental samples.

### **INSTRUMENTATION II**

#### **Contributed talk VI**

### 1048

# Upgrade of the external beamline at the Microanalytical center of Jožef Stefan Institute

<u>Kristina Isaković<sup>1,2</sup></u>, Marko Petric<sup>2</sup>, Zdravko Rupnik<sup>2</sup>, Ava Rajh<sup>2</sup>, Žiga Šmit<sup>3</sup>, Primož Pelicon<sup>2</sup>, Mitja Kelemen<sup>1,2</sup>, Matej Vereš<sup>2</sup>, Paula Pongrac<sup>2,4</sup>, Primož Vavpetič<sup>2</sup>, Matjaž Kavčič<sup>2</sup>

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Recently, the existing external beamline at the Microanalytical center was upgraded to extend the capabilities of the set-up from point measurements with a broad (~ 1 mm<sup>2</sup>) proton beam [1] to a 2D lateral in-air PIXE mapping across a surface area of ~1x1 cm2 with sub 100  $\mu$ m lateral resolution. In order to focus the beam, a doublet of magnetic quadrupole lenses was installed at the beamline. Additionally, the exit nozzle was redesigned to house an ultra thin 200nm Si3N4 foil and minimize the sample-exit window working distance. The characterization of beam size is presented together with the first application on longitudinal section of a whole thick wheat grain [2].

Additional upgrade is the installation of a new parallel beam wavelength dispersive spectrometer. The spectrometer combines polycapillary x-ray lens for efficient x-ray collection and diffraction on a flat crystal analyzer to reach high energy resolution. The spectrometer is designed for tender x-ray energy range (2-6 keV) so it is enclosed within a He bag to eliminate in-air x-ray absorption. Here we report the results of first characterization measurements yielding basic properties of the new setup.

### TUESDAY, 13 SEPTEMBER

## **SESSION 1**

### **RADIATION BIOLOGY**

Chairs: Judith Reindl and François Vianna-Legros

### Invited talk

### 1111

# Treating tumours with small ion beams: mini- and microbeam radiation therapy

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Despite major advances over the last decades, the dose tolerance of normal tissue continues to be a central problem in radiation therapy, limiting for example the effective treatment of radioresistant tumours. An innovative new approach to overcome this problem is the irradiation with very narrow beams that are spaced apart to create a socalled spatial fractionation of the dose. This creates a pattern of alternating high dose and low dose regions which has been shown to drastically improve the tolerance of normal tissue.

Depending on the dimensions and arrangement of the beams, several techniques are distinguished; here, the focus will lie on minibeam radiation therapy (MBRT, beam sizes from 0.1 to 1 mm) and microbeam radiation therapy (MRT, beam sizes from 25 to 100  $\mu$ m). Both MBRT and MRT have already demonstrated their tissue sparing potential with experiments showing that doses of 100-1000 Gy can be tolerated when delivered in the right configuration. This makes it possible to safely escalate the dose in the tumour which in turn can offer a more effective treatment. In this context, preclinical MBRT and MRT studies found tumour control rates that are comparable or even superior to those achieved with conventional radiotherapy.

The goal of this presentation is to give a general overview of MBRT and MRT by introducing physical and radiobiological core concepts and by discussing the mechanisms underlying the normal tissue sparing and treatment efficacy observed for both techniques. Moreover, challenges concerning a clinical translation will be addressed and the latest experimental results and technical advances will be presented.

#### Contributed talk I

### 1008

### Long term imaging of cells after targeted irradiation of mitochondria

<u>Sarah Rudigkeit</u>, Dietrich W.M. Walsh, Nicole Matejka, Matthias Sammer, Benjamin Schwarz, Günther Dollinger, Judith Reindl

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Mitochondria are the "power plants" of the cell and cover 30 - 40 % of the cytoplasmic volume in HeLa cells. Therefore, they are the main organelles for the survival of the cell. It is known, that mitochondria depolarize after targeted irradiation of them, but it is still unclear how this influences the cell. So, the aim of this work is to investigate which effects have the depolarization of mitochondria after targeted irradiation on the cells behavior, especially the cell survival. We performed targeted irradiation with carbon ions at the ion microprobe SNAKE (Super conducting Nanoprobe for Applied Nuclear (Kern) physics Experiments) at the 14 MV tandem accelerator in Garching near Munich, with a beam spot size of ~1 µm. With this setup it is possible to target mitochondria, which were fluorescently stained, with a defined number of 55 MeV Carbon ions (LET =  $365 \text{ keV/}\mu\text{m}^2$  at the cells). In a first study we targeted the mitochondria of single cells with 5,120 Carbon ions (equivalent to a dose of about 100 Gy/µm<sup>2</sup> on the cytoplasm) [1] and observed mitochondrial depolarization. Now we were able to further follow the irradiated cells for 3.5 days to score single cell survival using a phase contrast microscope (20x objective, PH2) together with a special Tokai Hit stage top incubator. While the number of the irradiated cells stayed constant during the observation, the unirradiated control group showed an exponential growth. An additional particle track detector test revealed that 3 % parasitic ions hit the cells up to 500 µm away from the targets. This approximately yields a dose up to 4.5 Gy per nucleus. Subsequent improvements to beam preparation reduces the dose to a half and the impact on cell survival has to be tested in future experiments.

[1] D.W.M. Walsh et al. (2017) Live cell imaging of mitochondria following targeted irradiation in situ reveals rapid and highly localized loss of membrane potential, Sci Rep. 7, 46684, doi: <u>https://doi.org/10.1038/srep46684</u>

### Contributed talk II

### 1031

### Combining In Situ Detection and Quantification of Metal Oxide Nanoparticles using Nuclear Microprobe Analysis with Complex Biological Analysis Techniques to reveal nano-induced ion homeostasis alterations on Caenorhabditis elegans intestine

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Metal oxide nanoparticles (NPs) are used in a wide array of industrial applications including direct human uses such as solar creams, cosmetic products, plastics, etc. These uses lead to the circulation of important amounts of NPs, thus posing potential risks to the environment and to health when they cross the physiological barrier, are ingested or inhaled. As such, there is a need to quantify and characterize the fate of these NPs for in vivo biological organisms as well as the mechanisms involved in the internalization and toxicity processes. A particular focus will be made on the impact of titanium dioxide nanoparticles (TiO2 NPs) on cellular ion homeostasis as they represent one of the most widely used type of NPs.

Previously, we illustrated that  $\mu$ -PIXE (AIFIRA facility, LP2iB) can quantify NPs internalization1 and how it influences in vitro the endogenous cellular content variation of some specific ions such as Ca2+. It was determined that these variations can be correlated to an endoplasmic reticulum stress-dependent toxicity2,3.

Now, we present our last observations related to in situ and in vivo analyses conducted on Caenorhabditis elegans exposed to TiO2 NPs.  $\mu$ -PIXE was found to be suitable for both accurate description of chemical structure of multicellular systems and for the detection of NPs.  $\mu$ -PIXE revealed that the calcium and potassium homeostasis were deeply modified in the proximal intestinal cells despite an absence of NPs internalization. In addition to physico-chemical characterization using  $\mu$ -PIXE, we use flow cytometry (reporter assay) and third generation sequencing (direct-RNA Transcriptome analysis) to confront the results and understand how ingested but non-internalized NPs could create stress-dependent toxicity and lead to adverse effect on nematode development and homeostasis.

- [1] Muggiolu G, Simon M, Lampe N, Devès G, Barberet P, Michelet C, Delville MH, Seznec H. In Situ Detection and Single Cell Quantification of Metal Oxide Nanoparticles Using Nuclear Microprobe Analysis. J Vis Exp. 2018 Feb 3;(132):55041. doi: 10.3791/55041. PMID: 29443063; PMCID: PMC5912329.
- [2] Simon M, Saez G, Muggiolu G, Lavenas M, Le Trequesser Q, Michelet C, Devès G, Barberet P, Chevet E, Dupuy D, Delville MH, Seznec H. In situ quantification of diverse titanium dioxide nanoparticles unveils selective endoplasmic reticulum stress-dependent toxicity. Nanotoxicology. 2017 Feb;11(1):134-145. doi: 10.1080/17435390.2017.1278803. PMID: 28044465.
- [3] Simon M, Barberet P, Delville MH, Moretto P, Seznec H. Titanium dioxide nanoparticles induced intracellular calcium homeostasis modification in primary human keratinocytes. Towards an in vitro explanation of titanium dioxide nanoparticles toxicity. Nanotoxicology. 2011 Jun;5(2):125-39. doi: 10.3109/17435390.2010.502979. Epub 2010 Jul 15. PMID: 21425910.

#### **Contributed talk III**

### 1036

# Increasing the efficacy of proton radiation in tumour cells by exploiting the <sup>11</sup>B(p, $\alpha$ ) $\alpha\alpha$ reaction with metallacarboranes

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In the fight against cancer, proton therapy can offer an attractive alternative to conventional photon-based radiotherapy. However, there is a need to improve the efficacy of protons by enhancing the relative biological effectiveness (RBE). Metallacarboranes, which are boron (B) rich compounds, are promising as radiosensitizers for proton therapy when using the well-known <sup>11</sup>B(p, $\alpha$ ) fusion reaction. This reaction has a main resonance at 675keV, a high cross section (~1barn), and the three  $\alpha$ -particles emitted at the point of reaction have a broad spectrum with a predominant energy of 4MeV [1]. The combined effect of proton energy close to the Bragg peak and  $\alpha$  particles range within typical cell dimensions is expected to escalate cell-killing upon compounds's accumulation in tumour cells.

The external beam facility at IST microprobe was used to irradiate cell monolayers, using a 2MeV proton beam with a flux of approximately  $10^8$  protons/cm<sup>-2</sup> s and an appropriate sequence of attenuators (Mylar foils, air and water with thicknesses determined/calculated using the SRIM code) in order to reach the resonance energy of the  ${}^{11}B(p,\alpha)\alpha\alpha$  fusion reaction at the cell layer. Human glioblastoma and breast cancer cells were used in the experiments as cell models. Cells were treated with the compounds for 24h, irradiated and the irradiation effects assessed. A successful validation step with a Monte Carlo (MC) model (MCNP6.1 code) was achieved by comparing several MC and experimental parameters, such as divergence of the beam, energy spectrum, the variation of the Bragg peak depending on the point of reaction and the variation of the maximum dose.

The MC simulations point out for a dose enhancement resulting from  $\alpha$ -particle emission, which is in part compatible with the increase of cell-killing effects obtained after irradiation. Work is in progress to extend the MC model to more detailed and accurate simulations that will give a full account of the dose variation in cells.

[1] Spraker MC, Ahmed MD, Blackston MA (2012) The 11B( $p,\alpha$ )8Be  $\rightarrow \alpha + \alpha$  and the 11B( $\alpha,\alpha$ )11B reactions at Energies Below 5.4 MeV. J Fusion Energ 31:357–367. Doi: 10.1007/s10894-011-9473-5

#### **Contributed talk IV**

### 1041

# Complementary radiobiology beam lines for high linear energy transfer (LET) proton beam cellular studies at the Centre for Ion Beam Applications (CIBA), National University of Singapore

<u>Ce-Belle Chen</u>, Zhaohong Mi, Chengyuan Yang, Saumitra Vajandar, Lakshmi Jayakumar, Tze Yen Chua, Minqin Ren, Ginhao Yuen, Frank Watt, Thomas Osipowicz, Andrew Bettiol

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Proton beam therapy (PBT) is an advanced radiation modality in cancer treatment with enhanced precision in tumour targeting that minimises damage to healthy tissue. Three new PBT centres in Singapore, at different stages of planning and completion, are to capitalise on these benefits in view of an ageing population with rising cancer projections. Globally, the cancer burden is projected at 28 million in 2040, almost 50 % increase from 2020 cancer cases. Recent innovations in cell biology potentially stimulate new discovery pathways in PBT research. At CIBA, we have constructed two complementary facilities dedicated to live cell radiobiology research: a high resolution focussed proton beam line and a large-scale cell irradiation end station utilising broad proton beams. The focussed proton beam line has a sub-500 nm in-cell spot size enabling precise and selective cell irradiation, with quantitative dosimetry, because protons can be counted individually. Simultaneously, live-cell fluorescence imaging and selective subcellular targeting functionality are available. The second system, designed for large scale cell irradiation, was constructed to deliver uniform proton doses to up to four million monolayer cells in a matter of minutes. With many standard cell biology techniques requiring hundreds of thousands to millions of cells, this large scale cell irradiation system expands the potential for multiple cell biology assays and downstream applications, including high-throughput screening platforms for drug discovery programs, next generation sequencing and proteomics. Our 3.5 MeV single ended accelerator delivers protons at energies not far below the Bragg peak, so that LET values above 10 keV/micron in cells are available. These two complementary systems enable a plethora of cell biology studies directly using clinicallyrelevant high LET protons, additionally precluding extraneous absorber or water phantom requirements.

### TUESDAY, 13 SEPTEMBER

### **SESSION 2**

### MeV-SIMS

Chairs: Melanie Bailey and Primož Vavpetič

#### Invited talk

### 1028

### Recent advances in MeV SIMS at the Ruder Bošković Institute

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MeV SIMS is a variation of the standard technique TOF SIMS, in which primary ions with energies of ~MeV/amu, accelerated by a particle accelerator, are used for ion desorption. The basis of the MeV SIMS technique lies in the fact that electronic stopping is much more pronounced in this energy range than nuclear stopping. The energy introduced into the electronic system by the MeV ion is transferred to the host atoms via electron-phonon interactions, forming a cylindrical high-temperature region along the ion path. Electron sputtering of the inorganic material, and the fragmentation of the organic material, take place in the high-density infratrack region of the ion track. On the other hand, intact molecular ions are ejected from the so-called lower energy density ultratrack region where the coupling between the excitation energy and the internal vibrational modes of the organic molecules is weak. In this way, a higher yield of molecular secondary ions with lower fragmentation is obtained than in the conventional SIMS, which is performed with keV ions. This makes a MeV SIMS ideal for chemical imaging of organic molecules with masses up to 2000 Da.

Over the past decade intensive development and application of the MeV SIMS has been done at the Ruder Bošković Institute (RBI). Recent results on the application of MeV SIMS in the analysis of forensic samples, such as the determination of the deposition order of crossing lines of different writing tools, will be presented. In addition to the standard MeV SIMS imaging setup at RBI, based on the focused ion beam, the newly developed MeV SIMS imaging system, based on the collimated beam will be shown. Thanks to the flexibility of target scanning system (instead of the ion beam scanning), objects with larger dimensions can be imaged. The performance of the new system will be demonstrated by imaging a thin section of the mouse brain and imaging a fingerprint with a lateral resolution of ~10 µm. Last but not least, we will show the idea of using primary ions with energies in the range of 100 keV - 5 MeV for SIMS, where both inorganic species and larger biomolecules can be desorbed from the sample simultaneously due to similar contributions of nuclear and electronic stopping power (possibility of hybrid sample analysis). The ability to image hybrid organic/inorganic samples will be demonstrated on a target with lateral distribution of Cr and leucine. In addition, the ability of LE MeV SIMS to perform depth profiling in a dual beam mode with an Ar gun was investigated. LE MeV SIMS depth profiling of a Cr- ITO bilayer sample in a dual beam mode will be shown.

### MeV-SIMS

#### Contributed talk I

### 1102

### **Detection of metastable ions at MeV-SIMS**

Boštjan Jenčič, Žiga Barba, Mirjana Vasić, Mitja Kelemen, Primož Vavpetič, Primož Pelicon

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Primary ions within the MeV range domain exhibit promising characteristics for sufficient detection of large biomolecules by means of sputtering via electronic excitations [1]. Within the work, we present the recent development of the corresponding MeV-SIMS Mass Spectrometry Imaging technique at Jožef Stefan Institute. Although MeV-SIMS yielded encouraging results in the past, the method still lacked several technological features and routine availability. Recently, a new bimodal mass spectrometer has been added to the experimental setup, allowing detection of secondary ions in linear, as well as reflectron modes. The reflectron part of the spectrometer is also equipped with an up to -10 kV post acceleration discrete dynode detector, which further increases detection efficiency of large biomolecules. Additionally, finer vacuum within the spectrometer allows higher transmission of secondary particles, thus revealing new information of technique's unique sensitivity and its physical limit.

By means of such spectrometer, we can detect and characterize metastable ions, i.e. ions, which fragment during their drift within the mass spectrometer. We measure the quantity of such fragmentation as a function of the primary ion energy, and asses fragmentation pathways of amino acid arginine.

[1] Y. Nakata, Y. Honda, S. Ninomiya, T. Seki, T. Aoki and J. Matsuo, "Yield enhancement of molecular ions with MeV ion-induced electronic excitation," *Applied Surface Science*, vol. 255, pp. 1591-1594, 2008.

### MeV-SIMS

### 1105

### Ambient MeV-SIMS Measurement of Negative Electrode Surface of Lithium Ion Buttery

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The Li-ion battery (LIB) has become a widely used product, particularly in small devices such as laptops and smartphones. However, the reactions taking place in rechargeable batteries are not completely understood. A solid electrolyte interphase (SEI) is formed mainly at the interface between the LIB negative electrode (anode) and the electrolyte during charging. SEI is a reductive decomposition product of electrolyte molecules (solvent, additive, and anion) and is a mixture of inorganic and organic substances. SEI has a significant effect on LIB performance and safety. To observe such an interface reaction in situ/during operation, a suitable technology for molecular analysis of SEI in the presence of liquid is required. Such an analytical system must be able to operate at ambient conditions because liquid materials in electrodes are mixtures of inorganic and organic substances, not only elemental analysis, but also chemical state analysis with a high interface sensitivity is required.

Secondary ion mass spectrometry (SIMS) is a method with high surface sensitivity, which allows both elemental and molecular analysis. MeV-SIMS uses heavy ion beams with energy in the MeV range, and samples can be measured under ambient conditions because of the high transmission capability of these ions at low vacuum pressure. The evaporation of liquid materials is suppressed in He at atmospheric pressure, and samples containing liquid can be measured using the MeV-SIMS technique without drying. In this paper, MeV-SIMS measurements of negative electrode LIB after charging / discharging were performed at atmospheric pressure.

The measured samples were negative electrode of LIB after 40 cycles of charging / discharging at 4.8 V. Both charge and discharge times are 30 minutes. The negative electrode was prepared by applying a solution of graphite and PVDF (PolyVinylidene DiFluoride) (graphite: PVDF = 95: 5) in NMP (N-methylpyrrolidone) on Cu. The electrolyte of LIB is EC containing LiPF<sub>6</sub> (1000mM). The SIMS spectrum was obtained at measurement time of 100 seconds. Additions of Li to EC cluster and additions of Li to compounds of EC with LiPF<sub>6</sub> ([3EC+LiPF<sub>6</sub>+Li]<sup>+</sup>, [4EC+LiPF<sub>6</sub>+Li]<sup>+</sup>, [4EC+LiPF<sub>6</sub>+Li]<sup>+</sup>, and [4EC+ 2LiPF<sub>6</sub>+LiF+Li]<sup>+</sup>) were detected with high sensitivity from EC containing LiPF<sub>6</sub>. It is clear from the nozzle-sample distance dependence of the peak intensity that these ions of Li additions are secondary ions from the sample surface. Additions of Li to compounds of EC with NMP and LiPF<sub>6</sub> ([2EC+NMP+Li]<sup>+</sup>, [2EC+NMP+LiPF<sub>6</sub>+Li]<sup>+</sup>, [3EC+NMP+LiPF<sub>6</sub>+LiF+Li]<sup>+</sup>, and [3EC+NMP+2LiPF<sub>6</sub>+LiF+Li]<sup>+</sup>) were also detected from the surface of the negative electrode. Since these ions contain NMP, which is a negative electrode material, it is considered that MeV-SIMS succeeded in measuring the negative electrode surface.

## QUANTUM TECHNOLOGIES

Chairs: Ettore Vittone and Jafar Shojaii

### Invited talk

### 1110

### Wide bandgap semiconductors for quantum applications

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Quantum photonics is an emerging field of photonics whereby light (single photons) is used for quantum computation, communication and sensing technologies. One of the key enablers for these new technologies are colour centres. Colour centres are isolated defects in crystals that can emit single photons and that have spin properties that can be exploited for quantum technologies. Two semiconductor materials that hold enormous potential for such technologies are Diamond and Boron Nitride (BN). Both of these materials are wide band gap semiconductors and have been demonstrated to possess the desirable properties for quantum technologies. These include colour centres with high brightness, low photo-bleaching and room-temperature optically accessible spins. Colour centres can sometime occur naturally in semiconductors and are introduced during the growth process. This is clearly not desirable as they will occur randomly throughout the material making it difficult to exploit their properties for quantum applications. Ion implantation is an important technology that can be used to generate colour centres with a high degree of spatial precision. In this presentation an overview of the generation of colour centres in diamond and hBN using a mega-electron volt nuclear microprobe will be discussed. Some examples of single photon emitters and magnetic sensing will be presented.

### QUANTUM TECHNOLOGIES

#### Contributed talk I

### 1063

# Deterministic single ion implantation and defect engineering studies using a focused beam of highly charged ions

<u>Paul Räcke<sup>1</sup></u>, Simon Robson<sup>2</sup>, Alexander M. Jakob<sup>2</sup>, David N. Jamieson<sup>2</sup>, Jan Meijer<sup>3</sup>, Daniel Spemann<sup>1</sup>

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Deterministic single ion implantation will be a key to enable novel solid state-based quantum technologies based on impurities or defect-centres in silicon, diamond or other materials. The single ion implanter used here was constructed by combining a commercially available focused ion beam (FIB) machine with an electron beam ion source (EBIS), which selectively produces ions of low and high charge states of various species, such as noble gases, hydrogen, nitrogen and soon phosphorous, with nanoscale focus (acceleration potential 6 to 15 kV).

A recent addition to the setup includes an ion beam induced charge (IBIC) system, which was developed for ultra-low noise keV single ion detection [1]. By scanning the focused low energy ion beam across specially configured single ion detectors and spatially mapping the IBIC response, unprecedented studies of the three-dimensional near-surface (<100 nm) electrical landscape were performed, showing uniform near 100% charge collection over a large area in the newest detector generation. This setup thus offers a fabrication platform for scalable qubit arrays using mask-free deterministic ion implantation [2].

In parallel, a single ion pre-detection scheme based on image charge detection is developed to be implemented in the future. A new detector prototype designed and constructed in Leipzig is cryogenically cooled and combines the signals of four detector segments, substantially increasing the signal-to-noise ratio. As a result, single ion bunches containing down to 80 elementary charges were detected. Remaining challenges and potential for further improvement of the sensitivity towards single ion detection are discussed [3].

While highly charged ions are necessary for the proposed single ion image charge detection scheme, choosing different charge states furthermore enables to conveniently vary the implantation energy from less than 10 up to several hundred keV, which is remarkable using such a compact setup. This is beneficial for systematic defect engineering studies in any material, as we exemplify by direct writing of vacancies in different depths for NV-centre creation in diamond [4].

<sup>[1]</sup> Jakob AM et al (2021) Deterministic Shallow Dopant Implantation in Silicon with Detection Confidence Upper-Bound to 99.85% by Ion–Solid Interactions, Adv. Mat. 34, 2103235, <u>https://doi.org/10.1002/adma.202103235</u>

<sup>[2]</sup> Robson SG et al (2022) Near-Surface Electrical Characterisation of Silicon Electronic Devices Using Focused keV lons. <u>https://doi.org/10.48550/arXiv.2201.11339</u>

<sup>[3]</sup> Räcke P, Meijer, J, Spemann, D (2022) Image charge detection of ion bunches using a segmented, cryogenic detector. J. Appl. Phys. 131, 204502, <u>https://doi.org/10.1063/5.0096094</u>

<sup>[4]</sup> Räcke P et al (2021) Vacancy diffusion and nitrogen-vacancy center formation near the diamond surface Appl. Phys. Lett. 118, 204003, <u>https://doi.org/10.1063/5.0046031</u>
#### Contributed talk II

#### 1072

#### **Overcoming Surface Recombination for Diamond Single Ion Detectors**

<u>Nicholas Collins<sup>1</sup></u>, Alexander Jakob<sup>1</sup>, Simon Robson<sup>1</sup>, Shao Qi Lim<sup>1</sup>, Boqing Liu<sup>2</sup>, Brett Johnson<sup>3</sup>, Jeffery MacCallum<sup>1</sup>, David Jamieson<sup>1</sup>

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Diamond colour centres have the potential to be integrated into a monolithic device that employs the colour centre to store and transmit quantum information as well as to act as quantum sensors [1]. Production of a large-scale device requires addressing the challenge of engineering ordered arrays of near-surface colour centres [2,3] in diamond substrates. Deterministic ion implantation employing the Ion Beam Induced Charge (IBIC) single ion detection method detects the charge of free electron-hole pairs induced by a single ion impact in the diamond substrate to register the implantation event. Here we address the challenge of adapting this method from its successful applications in silicon [4] to diamond. We have fabricated on-diamond electrodes that can be biased to produce strong internal fields and minimise leakage current required for high fidelity single ion signals for typically sub-20 keV ions needed for near-surface implants. A difficult challenge was to supress surface charge recombination at the diamond surface which would otherwise allow most of the induced charge to recombine. Our method, employing a thin conductive electrode addresses this challenge and gives close to 100% charge collection efficiency for sub-20 keV ions and high-fidelity deterministic implantation despite the lower ionisation rate in diamond with its much wider band-gap compared to silicon. We use IBIC spectroscopy to demonstrate the charge collection efficiency in our new efficient detector architectures which is a significant advance over previous generations of devices.

- [1] Doherty, Marcus W., et al. "The nitrogen-vacancy colour centre in diamond." Physics Reports 528.1 (2013): 1-45.
- [2] Nemoto, Kae, et al. "Photonic quantum networks formed from NV- centers." Scientific reports 6.1 (2016): 1-12.
- [3] Pezzagna, S., et al. "Creation efficiency of nitrogen-vacancy centres in diamond." New Journal of Physics 12.6 (2010): 065017.
- [4] Jakob, Alexander M., et al. "Deterministic Shallow Dopant Implantation in Silicon with Detection Confidence Upper-Bound to 99.85% by Ion–Solid Interactions." Advanced Materials 34.3 (2022): 2103235.

#### 1009

## ACCURACY OF 3D QUANTITATIVE IMAGING OF MICROSCOPIC SAMPLES EVALUATED BY GEANT4 SIMULATION

Zhuxin Li<sup>1</sup>, Claire Michelet<sup>1</sup>, Sébastien Incerti<sup>1</sup>, <u>Philippe Barberet</u><sup>1</sup>, Hao Shen<sup>2</sup>, Shimei Wang<sup>2</sup>, Daniel Beasley<sup>3</sup>, Hervé Seznec<sup>1</sup>

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Proton micro-tomography is a powerful tool for three-dimensional (3D) imaging of microscopic samples. Two techniques are implemented: i) STIM (Scanning Transmission Ion Microscopy) to obtain the 3D struture of the sample; ii) PIXE (Particle-Induced X-ray Emission) to obtain the distribution of chemical elements [1]. The obvious first benefit of 3D imaging is to locate chemical elements within the analysed volume, without cuting the sample. Beyond spatial distribution, a major issue is quantitative imaging, i.e. accurate calculation of mass density (in g/cm3) in the reconstructed images. For PIXE tomography, signal attenuation is a concern, because it leads to an underestimation of the element content. It is due to two physical phenomena: variations of X-ray production cross-sections and X-ray absorption within the material. A correction has been implemented in the tomographic reconstruction process in order to take into account these phenomena. In the present study, our purpose is to quantify in a direct way the accuracy of the obtained tomographic images. For this, we used as a benchmark the Geant4 toolkit (http://geant4.org), which is the most widely used open source code in the world for simulation of particle-matter interaction.

A Geant4 simulation code of PIXE and STIM tomography experiments has been developed, in such a way to minimize execution time and memory space. The simulated data sets are used to quantify the accuracy of the reconstructed tomographic images. Geant4 constitutes a useful benchmark as it brings important advantages: i) it is independent of the inversion method used for the reconstruction; ii) one or more physical processes (X-ray absorption, proton energy loss) can be numerically turned off, giving access to data that cannot be obtained in real experiments [2].

Several examples will be presented, such as Caenorhabditis elegans microorganism and microsphere targets used for inertial fusion experiments [3]. An accuracy  $\leq 4\%$  was obtained for the mass density of phosphorus, which was the most attenuated element, compared to a about 40% discrepancy without correction. A comparison between results obtained from different reconstruction codes (TomoRebuild, DISRA and JPIXET [4]) will be also presented.

- [1] Michelet C et al (2015) Development and applications of STIM- and PIXE-tomography: A review. Nucl. Instr. Meth. B 363:55-60. doi: 10.1016/j.nimb.2015.08.070
- [2] Michelet C et al (2022) A Geant4 simulation of X-ray emission for three-dimensional proton imaging of microscopic samples. Physica Medica 94:85-93. doi: 10.1016/j.ejmp.2021.12.002
- [3] Wang S. et al (2022) Application of silicon drift detector in PIXE tomography system of Fudan University. Nucl. Instr. Meth. B 512:108-113. doi: 10.1016/j.nimb.2021.12.010
- [4] Beasley DG et al (2013) Fast simulation of proton induced X-ray emission tomography using CUDA. Nucl. Instr. Meth. B 306:109-112. doi: 10.1016/j.nimb.2012.12.053

#### 1023

#### Cold plasma treatment of buckwheat grain decreases water contact angle and husk Ca and Mn concentrations

Pia Starič<sup>1,2</sup>, Primož Vavpetič<sup>1</sup>, Katarina Vogel-Mikuš<sup>1,2</sup>, Paula Pongrac<sup>1,2</sup>, Ita Junkar<sup>1</sup>

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Common buckwheat (*Fagopyrum esculentum*) is an increasingly popular crop whose grain is rich in high-quality proteins, fibres, lipids, essential minerals, vitamins and antioxidants. Because it is gluten-free it is an excellent grain alternative to cereal grain in diets of gluten-intolerant people and people with celiac disease [1], [2]. Furthermore, it is a crop that is relatively easy to grow as it requires no abundant fertilizers or pesticides for optimal growth and yield [3].

The use of cold or non-thermal plasma (CP) technology in agriculture is a promising environmentally-friendly way to decontaminate grain and to improve germination, yield, and/or stress resistance in crops. Thus, this novel approach could improve the production and storage of grain, such as buckwheat [4].

We investigated the impact of CP treatment on pericarp morphology of buckwheat grain with scanning electron microscopy and localization of elements in grain tissues (pericarp, aleurone, endosperm, and cotyledons) with micro-proton-induced X-ray emission spectroscopy. In addition, water contact angle was evaluated.

The CP treatment caused no visible changes on the buckwheat grain surface, but a decrease in water contact angle was detected in CP-treated grain. CP-treated grain germinated slower than untreated grain, without effects on the total number of germinated grains. The concentration of calcium (Ca) and manganese (Mn) in the outer layer of the grain (pericarp) was lower in CP-treated grain compared to untreated ones. A trend of lower concentration of potassium (K) in cotyledons and pericarp of CP-treated grain compared to untreated to untreated grain was observed, but it was not statistically significant presumably due to large variability in K concentrations present.

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- [1] A. Ivankov et al., "Changes in Agricultural Performance of Common Buckwheat Induced by Seed Treatment with Cold Plasma and Electromagnetic Field," Appl. Sci. 2021, Vol. 11, Page 4391, vol. 11, no. 10, p. 4391, May 2021
- [2] P. Pongrac, K. Vogel-Mikuš, M. Regvar, P. Vavpetič, P. Pelicon, and I. Kreft, "Improved lateral discrimination in screening the elemental composition of buckwheat grain by micro-PIXE," J. Agric. Food Chem., vol. 59, no. 4, pp. 1275–1280, Feb. 2011
- [3] Popović, V., Sikora, V., Berenji, J., Glamočlija, Đ., Marić, V. (2013a): Effect of agroecological factors on buckwheat yield in convential and organic cropping systems, Institute of PKB Agroeconomik, Belgrade, vol. 19(1-2), pp. 155-165
- [4] L. K. Randeniya and G. J. J. B. de Groot, "Non-Thermal Plasma Treatment of Agricultural Seeds for Stimulation of Germination, Removal of Surface Contamination and Other Benefits: A Review," Plasma Process. Polym., vol. 12, no. 7, pp. 608–623, Jul. 2015

#### 1046

## Effect of Xylella fastidiosa infection on tissue-specific localisation of elements in almond and grapevine leaves

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Xylella fastidiosa (Xf) is a slow growing, xylem limited, vector-transmitted and gram-negative bacterium first described by Wells et al. in 1987 [1]. The pathogen can infect more than 80 plant species and genera. Large economical damage is observed in the Americas from where the bacterium originates [2], namely in grapevine, citrus and almond [1,3]. Since its first detection in Europe, in southern Italy [4] it has destroyed millions of olives and negatively affected almonds in Spain. In terms of effects Xf-infection has on mineral nutrition of plants, higher concentration of Ca and Mg, but lower concentrations of K have been reported for Xf-infected grapevine leaves compared to healthy leaves [5]. There is no information on tissue-specific redistribution of elements due to Xf-infection, thus the aim of our study was to evaluate redistribution of elements in Xf-infected and healthy leaves of two almond cultivars (Glorieta and Masbovera), and in grapevine (Cabernet Sauvignon) grown on calcareous or red soil. Leaf cross sections were prepared as described previously [6] or necrotic spots on leaves (of unknown origin) were excised and tissue-specific element analysis was performed using micro-PIXE at Jožef Stefan Institute. Spectra were fitted, and distribution maps were generated in GeoPIXE software [7]. There were cultivar-dependent differences in concentration of S, CI, Ca, and K in specific tissues in Xf-infected and healthy almond leaves. Necrotic spots on the almond leaves were encircled with Si and Fe, centrally there was an enrichment of P and S and exclusion of CI and K. In grapevine, results depended on the soil and significant differences in P and S concentrations in different leaf tissues when comparing Xf-infected leaves with healthy leaves were observed. This is the first report on the use of micro-PIXE in Xfinfected crop leaves.

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[1] J.M. Wells, et al., Int. J. Syst. Bacteriol. 37 (1987) 136–143. [2] L. Nunney, et al., PLoS One. 9 (2014) e112463. [3] P. Baldi, N. La Porta, Front. Plant Sci. 8 (2017) 944 [4] M. Saponari, et al., J. Plant Pathol. 95 (2013) 668. [5] P.H. Goodwin, J.E. DeVay, C.P. Meredith, Physiol. Mol. Plant Pathol. 32 (1988) 17–32. [6] K. Vogel-Mikuš, P. Pongrac, P. Pelicon, Int. J. PIXE. 24 (2014) 217–233. [7] C.G. Ryan, Int. J. Imaging Syst. Technol. 11 (2000) 219–230.

#### 1064

#### Tissue-specific distribution of elements in date palm

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Date palm (Phoenix dactylifera L.) is an economically important perennial crop of (semi)arid areas. Although date palms are thought to be salt-tolerant, (extreme) salinity can result in economic losses [1]. In this study, seedlings of date palm cultivars Khalas and Sultana were watered with NaCl for eight weeks. At harvest, the root tip, root base, stem and bottom part of the leaf were excised, cross sections were prepared [2] and tissue-specific elemental analysis was performed using microparticle induced-X-ray emission (micro-PIXE) at Jožef Stefan Institute [3]. Spectra were fitted, and distribution maps were generated using GeoPIXE software [4]. Bulk concentrations of elements were determined in plant parts using inductively-coupled plasma mass spectrometry after acid digestion of dried samples. Sodium and CI concentrations were the largest in root tips, followed by the root base, stem and leaves. Tissue-specific allocation of Na could be determined for root tips only, where Na was accumulated in the cortex; elsewhere Na was distributed evenly. By contrast, Cl was found in the cell layer surrounding cortex and also in the vascular bundle in root tips, while in the root base,

stems and leaves, CI was accumulated in extraordinarily large cells and in tissue surrounding the vascular bundle (Fig. 1). Of the other elements detected with micro-PIXE, namely Si, P, S, K, Ca, Mn, Fe and Zn, the most remarkable distribution was observed for Si, which was found as Si phytoliths, which surround vascular bundles on adaxial and abaxial sides of leaves and flank Cl-rich cells (Fig. 1). These Si phytoliths have been described in detail previously [5], but their distribution has now been determined in all plant parts and at tissue level. Phosphorus was allocated to the vascular bundles (Fig. 1). Further work is underway to resolve expression of genes involved in response to NaCl treatments in these two date palm cultivars.

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- B. Du, et al., P, New Phytol. 229 (2021) 3318-3329. [1]
- K. Vogel-Mikuš, P. Pongrac, P. Pelicon, Int. J. PIXE. 24 (2014) 217-233. [2]
- [3] P. Pelicon, et al., Nucl. Instrum. Methods Phys. Res. B 332 (2014) 229-233.
- [4] C.G. Ryan, Int. J. Imaging Syst. Technol. 11 (2000) 219-230.
- [5] B. Bokor, et al., Front. Plant Sci. 10 (2019) 1-17.



Figure 1 Distribution of Si, Cl and P in palm leaf. Scale bar is in µm.

#### 1065

## Tissue-specific concentration of zinc in grain of two Slovenian proso millet (*Panicum miliaceum* L.) landraces

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Zinc (Zn) is one of the essential elements in humans, where it plays a crucial role in over 300 enzyme processes, in the transcription and expression of genes [1], and in protein complexes responsible for correcting DNA damage [2]. Tissue development, mineralization of bones, the workings of the immune system and thyroid function are only a few processes in animals that depend on sufficient levels of Zn. Therefore, its deficiency can lead to a plethora of health problems, ranging from low blood pressure and diarrhea to the more severe immune deficiency and stunted development [3]. While severe deficiency is rare, the WHO estimates that on a daily basis, around 2 billion people do not consume enough Zn. The estimate is higher in developing countries, where it rises to about 73% of the population [4]. One of the crucial and easiest ways to add Zn into diets is through the use of Zn-enriched crops. Proso millet (Panicum miliaceum L.) offers itself as a promising candidate, as it has many favoured nutritional (boasts high protein content [5] and is gluten-free) and agronomic traits (extremely low water requirements, which render it drought tolerant [6], and low nutrient requirements, which enable it to grow in poor soils with minimal agronomic input [7]). However, genomic resources and other relevant information about proso millet are still limited, and the variability in Zn-use efficiency has not yet been evaluated. We cultivated six Slovenian landraces of proso millet along with one Slovene autochthonous cultivar, Sonček [8] to determine the concentration of Zn in different tissues (roots, leaves and grain) at two developmental stages (flowering and at grain maturity). After evaluating the bulk Zn concentration using X-ray fluorescence, we selected the two extremes in grain Zn concentration for detailed grain tissue-specific analysis using micro-particle-induced X-ray emission at the Jožef Stefan Institute, Slovenia. Results confirm the bulk differences in Zn grain concentration and assign the differences to specific grain tissues.

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- [1] Tipton IH, Cook MJ (1963). Health Physics 9, 2:103–145.
- [2] Ho E, Ames BN (2002). PNAS 99, 26:16770-16775.
- [3] Deshpande J, Joshi M, Giri P (2013). International Journal of Medical Science and Public Health 2, 1:1-6.
- [4] Caulfield LE, Black RE, 2004. World Health Organization 287-279.
- [5] Saha D, Gowda MC, Arya L, Verma M, Bansal KC (2016). Crit Rev Plant Sci 35: 56-79.
- [6] Baltensperger DD (1996). Foxtail and proso millet. In: Janick J, editor. Progress in New Crops. Alexandria, VA, USA: ASHS. Press, pp. 182-190.
- [7] Sabir P, Ashraf M, Akram NA (2011). J Agron Crop Sci 197: 340-347.
- [8] Flajšman M, Štajner N, Kocjan Ačko D (2019). Turk J Botany 43:185–195.

#### 1085

#### Tissue-specific calcium and magnesium allocation explains differences in bulk concentration in leaves of one-year-old seedlings of two olive (*Olea europea* L.) cultivars

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Olive (Olea europea L.) leaves have recently been endorsed as a valuable by-product in cosmetic and pharmaceutical industries or in preparation of health-promoting beverages. Knowledge on the mineral composition of olive leaves is still scarce, while tissue-specific allocation of minerals within leaves has not been studied up to date. In this study, micro-particle induced X-ray emission (micro-PIXE) was used to identify and quantify minerals in different leaf tissues of two olive cultivars, Leccino and Istarska bjelica. In addition, leaf bulk mineral analyses were performed. One-year-old seedlings grown in pots were investigated to ensure the differences observed were cultivar-dependent only. Leaves of the Istarska bjelica cultivar had larger bulk concentrations of potassium (K), sodium (Na), molybdenum (Mo) and boron (B), but smaller concentrations of calcium (Ca) and magnesium (Mg) than leaves of the Leccino cultivar. Tissue-specific investigation revealed that larger concentration of Ca in epidermis and in leaf blade tissues (secondary veins, palisade and spongy mesophyll) contributed to the larger leaf bulk Ca concentration in the Leccino cultivar. For Mg, all leaf tissues, except the bundle sheath cells and consequently the main vascular bundle, contributed to the larger bulk Mg concentration in the Leccino cultivar compared to leaves of the Istarska bjelica cultivar. Potassium was not predominant in any of the leaf tissues examined. Results indicate that Ca and Mg sinks in specific leaf tissues are stronger in the Leccino than in the Istarska bjelica cultivar. Element profiles of three leaf tissues (i.e. clusters) were captured using multivariate statistics. The first cluster, rich in K, discerned the spongy mesophyll and the xylem from other tissues. In the second cluster, chlorine, K and Ca predominated and such profile was found in epidermis, bundle sheath cells and supportive tissue of the main vein. The third cluster, rich in Mq, phosphorus, sulphur and K was characteristic for palisade mesophyll. This report offers basic understanding of tissuespecific allocation of minerals in leaves of olive, economically the most important fruit tree in the Mediterranean. In addition, it could serve as a basis for detailed studies into the effects of (foliar) fertilisers in olive leaves and for elucidation of dynamics and differential extraction of minerals from olive leaves during beverage preparation.

#### 1094

## Uptake and tissue-specific localization of arsenic in roots and nodules in peanut

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Arsenic (As) is toxic to plants and animals. Flooding of agricultural land with As-rich groundwater increases the risk of As mobility in the food chain and has become a significant threat. One such situation is with peanut (Arachis hypogaea L.), an agriculturally valuable crop in Córdoba province in Argentina, where i) several cropping areas contain high concentration of As and ii) occasional flooding episodes increase As-containing groundwater level [1]. Both circumstances could lead to a reduction on peanut yield and increase the possibility of seed contamination with As. Arsenate (As(V); AsO43-), a form of As, is particularly problematic, because it is chemically similar to the plantavailable form of essential phosphorus (PO43-). Therefore, when arsenate reaches the rhizosphere, it is inadvertently taken up by the roots, transported within the plant and may enter edible seeds. Moreover, exposure to As and flooding can negatively affect the uptake of essential elements and could cause redistribution of these elements within the plant. The aim of this work was to study the uptake and distribution of As in different parts of peanut and to determine the tissue-specific localization of As in roots and nodules under non-flooded and flooded conditions. Nodule formation was induced by inoculation with the beneficial bacterium Bradyrhizobium sp. SEMIA6144 and plants were grown under controlled conditions for 45 days and exposed to the four treatments (Control, As, flooding and As&flooding). Using X-ray fluorescence total concentration of elements were determined. The highest concentration of As was detected in roots, followed by nodules, while the concentration of As detected in stems and leaves was significantly lower. Using quantitative microparticle-induced X-ray emission (Jožef Stefan Institute, Slovenia), allocation of As was determined at the tissue level in cross-sections of roots and nodules. Spectra were fitted, and quantitative distribution maps were generated using GeoPIXE software [2]. The detailed analysis of the localization and distribution of As in different parts and tissues of peanut plants will provide an insight into tissue-specific disturbances caused by As and flooding in this agriculturally important crop.

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<sup>[1]</sup> J.M. Peralta, C.N. Travaglia, M.C. Romero-Puertas, A. Furlan, S. Castro, E. Bianucci, Chemosphere. 259 (2020) 127410.

<sup>[2]</sup> C.G. Ryan, Int. J. Imaging Syst. Technol. 11 (2000) 219–230.

#### 1055

## Micro-PIXE analysis for the provenance investigation of ancient pottery from Locri Epizephiri (Italy)

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Black slip ware and red figure pottery were produced in Southern Italian workshops during the 6th to the 4th century BCE according to a Greek technological process [1]. The vases and fragments of red-figured pottery found at Locri Epizephiri (Southern Italy) has been under study for over 10 years at the University of Torino to investigate not only their production technology but also their provenance. The initial attribution to Attic or Southern Italian (mainly Locrian or Sicilian) workshops on grounds of stylistic criteria [2] was later supported by archaeometric studies by means of Scanning Electron Microscopy coupled with Energy Dispersive X-ray spectroscopy and Induced Couple Plasma Optical Emission Spectrometry [3]. These techniques revealed that the concentrations of major and minor elements in the clay body allow the discrimination between the Greek and the Italian origin. Now, the study aims at distinguishing the Calabrian from the Sicilian production by investigating the composition of the very thin vitrified gloss (some 20  $\mu$ m) on the pottery shards, chasing for possible provenance markers among the trace elements of the black coating. The quantitative analysis of this very small target was performed by exploiting the micro-PIXE technique, that guarantees high sensitivity and spatial resolution and a non-destructive approach.

The 16 pottery samples (6 Attic, 6 Locrian and 4 Sicilian) analysed in this work were small shards prepared as cross sections. Micro-PIXE measurements were performed in vacuum at INFN-LNL (Legnaro, Padova, Italy) with 2 MeV protons. For each sample, three to five 50  $\mu$ m lines were acquired along the coating layer to take into account its possible inhomogeneity. Two sets of ceramic standard samples that simulate different ancient production technologies, such as different firing conditions, were also employed for calibration.

The methodology of the PIXE investigation on red-figured pottery found at Locri Epizephiri and the preliminary results of the analysis will be presented. The trace elements found in the slip could be used to distinguish the Calabrian from the Sicilian production and to test the hypothesis that different workshops used raw materials from different sources to obtain the vitrified slip.

- [1] Mirti P. et al., Technology of production of red figure pottery from Attic and southern Italian workshops, Analytical and bioanalytical chemistry, 380 (2004) 712-718.
- [2] Elia D., Ph.D. thesis, University of Messina, Italy, 2001.
- [3] Mirti P. et al., The Provenance of Red Figure Vases From Locri Epizephiri (Southern Italy): New Evidence by Chemical Analysis, Archaeometry 46 (2004) 183-200

#### 1059

#### Mineral Glazed beads: similar composition but different colour

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The beads (five) under study have similar shapes and sizes and different colors, from pale orange to dark blue/black. They were found during a recent archaeological survey performed on an ancient vessel (Boa Vista 5), in Lisbon. They are representative of the beads found, chronologically attributed to the period between the last quarter of the 18th century and the beginning of the 19th century. The main objective of this work is the chemical and mineralogical characterization of the beads, by means of non-destructive techniques.

PIXE technique reveals the presence of Si as a major element and it was also possible to identify Fe, K, Ca, S, and Cl elements in all the beads, and no significant concentration differences were found in order to explain the different beads colors.

The ionluminescence signal recorded from the beads when irradiated with a 2 MeV proton beam using the external beam setup available in Lisbon shows a similar spectra shape for all of them, and it was not possible to identify the presence of impurities responsible for the different colors of the samples.

Complementary techniques, such as X-ray diffractrion (XRD), were used to understand the origin of the color of these discovered beads.

#### 1060

#### The use of IBA techniques to study iron gall inks. What more can we get?

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The use of IBA techniques, mainly PIXE and RBS, for the study of cultural heritage objects is increasing, mainly due to the efforts during the last decades in the diffusion of these techniques in international conferences, and the participation in several European programs with supported transnational access.

Among the cultural heritage objects, manuscripts written with iron gall inks have been investigated mainly with PIXE due to the high sensitivity to the elements present in the inks in trace concentrations [1].

On the other hand, the use of portable and/or handheld XRF analyzers to characterize these inks is also increasing, due to the relatively easy use and accessibility.

The advantages of PIXE, such as the high sensitivity and the possibility of element quantification, are clear and further enhanced when the possibility of obtaining 2D maps composition is added [2].

Furthermore, the depth penetration of the ink can be obtained if RBS spectra are also considered during the analysis combined with the use of protons or alpha particles beams to obtain better depth resolution for surface and sub-surface analysis.

In this work, results obtained from the characterization of a document from the 17th century, written with different iron gall inks will be presented. Different techniques were used, including IBA and XRF. The information obtained from them will be compared and discussed, providing an overview of what more can the IBA techniques give, considering for example the number of points/pages to be analysed in a short time.

[1] L. Giuntini, F. Lucarelli, P.A. Mandò, W. Hooper, P.H. Barker (1995) Galileo's writings: chronology by PIXE, Nucl Instruments Methods Phys Res Sect B Beam Interact with Mater Atom, 95:389-392. doi: 10.1016/0168-583X(94)00538-9.

[2] V. Corregidor, R. Viegas, L.M. Ferreira, L.C.Alves (2019). Study of Iron Gall Inks, Ingredients and Paper Composition Using Non-Destructive Techniques. Heritage 2:2691-2703. Doi: 10.3390/heritage2040166

#### 1070

#### Ion beam induced luminescence analysis of europium complexes in styrene-divinylbenzene co-polymer coated spherical silica by proton and argon ion beams irradiation

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In a nuclear fuel reprocessing, it is proposed that not only U and Pu but also minor actinides (MAs) such as Np, Am, and Cm are recovered for reduction in the volume and potential radiotoxicity of high level liquid wastes, and the study for recovery of MAs has been conducted in the worldwide. Among MAs, an extraction chromatography has been proposed to recover trivalent MAs, Am and Cm, in Japan. An extractant for trivalent MAs recovery is impregnated styrene-divinylbenzene co-polymer coated silica particles, and it is used as adsorbent. The adsorbent is packed into a column, and high level liquid waste is pumped through columns to recover MAs. It is important to understand the structure of complexes in the adsorbent to recover MAs efficiently. Ion beam induced luminescence (IBIL) analysis of complexes in the adsorbent is investigated as one of methods of evaluating the structure of complexes. In the experiments, various adsorbents extracting Eu were prepared with extractants for MAs recovery. Non-radioactive Eu was selected as simulated material of Am because Eu is easy to be detected the luminescence and the extraction behavior of Eu is similar to that of Am. The IBIL spectra of Eu complexes in the adsorbent were measured using two different ion beams, 3 MeV proton in the light-ion micro beam line connected to 3-MV single-ended accelerator and 107 MeV Ar8+ with the beam line for large-area uniform irradiation at the azimuthally varying field (AVF) cyclotron facility, in Takasaki Ion Accelerators for Advanced Radiation Application (TIARA), National Institutes for Quantum Science and Technology (QST). In this study, the structure of Eu complexes in various adsorbents was evaluated based on the IBIL spectra. It was confirmed that the spectral shape of Eu complexes in the adsorbents varied depending on the kinds of extractants. The detailed research results will be reported in our presentation.

#### 1074

## Mo(IV) and Pd(II) ion recovery mechamism onto baker's yeast from nitric acid medium

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Extraction chromatography is a promising technology for minor actinide (MA(III): Am and Cm) recovery from high level liquid waste generated in the reprocessing of spent nuclear fuel. Our group is proposing a two steps column operation process flow, and MA(III) recovery performance was demonstrated on simulated waste liquid containing Am. Although MA(III) was successfully recovered by the technology, accumulation of Mo(IV) and Pd(II) in the 2nd column was confirmed and improvement in the process is required for repeated use of the column. One of the promising improvements is selective adsorption of these metal ions from feed solution for the column. We have already found that addition of another column between two columns can achieve the improvement [1]. However, the improvement requires additional equipment and expensive reagents, and more simple and inexpensive process is desirable. Currently, we are focusing on baker's yeasts for their excellent metal ions adsorption characteristics [2], easy handling and low prices. In order to optimize adsorption performance and operation procedures as the reprocessing technology, adsorption mechanism has to be precisely investigated. In this study, adsorption performance of Mo(IV) and Pd(II) from nitric acid, functional groups on the surface of the baker's yeast, coordination structure around the ions, mapping of the ions on the yeasts are investigated by batch-wise adsorption experiments, FT-IR, EXAFS and micro-PIXE analyses, respectively.

Dry baker's yeast was put into 2 M nictric acid solution containing Mo(IV) and Pd(II), and the yeast was separated form the solution by centrifugation. Mo and Pd K edge EXAFS measurements on the yeast were carried out at BL11S2 beamline of AichiSR, Japan. The micro-PIXE analysis was carried out at a light-ion microbeam line connected to a 3-MV single-ended accelerator in TIARA of QST, Japan.

Mo(IV) and Pd(II) concentrations in the solution decreased after the batch-wise operation. Characteristic X-ray of Mo and Pd were observed in the PIXE spectra and two-dimensional PIXE images showed that Mo and Pd uniformly distributed on surface of the yeast. FT-IR showed various functional groups exist on surface of the yeast and those might contribute to the adsorption. Currently, quantitative EXAFS analysis based on the FT-IR analysis are underway.

<sup>[1]</sup> Abe R, Nagoshi K, Arai T, Watanabe S, Sano Y, Matsuura H, Takagi H, Shimizu N, Koka M, Sato T (2017) Micorscopic analyses of complexes formed in adsorbetn for Mo and Zr separation chromatography. Nucl Instruments Methods Phys Res Sect B Beam Interact with Mater Atoms 404:173-178. doi: 10.1016/j.nimb.2017.02.082

<sup>[2]</sup> Saito N, Fujimori R, Yoshimura T, Tanaka H, Kondoh A, Nomura T, Konichi Y (2020) Microbial recovery of palladium by baker's yeast through bioreductive deposition and biosorption. Hydrometallurgy 196:105413. doi: 10.1016/j.hydromet.2020.105413

#### 1077

## Evaluation of remaining spent extraction solvent in vermiculite after leaching test by PIXE analysis

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Spent PUREX solvent, which mainly consists of tributyl phosphate (TBP) and normal dodecane, is generated by solvent extraction of Plutonium and Uranium in the spent nuclear fuel reprocessing. The spent solvent should be appropriately treated for its safety disposal or storage. Although reprocessing plant has liquid treatment systems, some nuclear facilities such as laboratories do not have the treatment system.

One of the treatment options for storage of the spent solvent is adsorption of the liquid into vermiculite. The organic liquid is considered to be trapped between layers of the vermiculite. Our previous experiments on the adsorption of spent solvent into the vermiculite have shown that some parts of loaded solvent gradually leaked out from the vermiculite.

In order to investigate the adsorption mechanism and capacity, elution behavior of the loaded solvent into organic diluents such as hexane or acetone was evaluated. A part of the loaded solvent was easily leaked into the diluent, while some solvent remained inside the particle even after the leaching test. The different behavior might be attributed to different adsorption mechanism, and amount of the remaining solvent can be considered as adsorption capacity for safety storage of the solvent.

In this study, the adsorption capacity of the vermiculite was evaluated through amount of remaining solvent after washing with diluents. The amount of the remaining solvent was analyzed by Particle Induced X-ray Emission (PIXE) on P contained in TBP. PIXE experiments were carried out at a tandem accelerator of Tokyo City University, and 2 MeV H+ beam was irradiated on vermiculite powders in vacuumed atmosphere. Peak intensity of P-Kα line depended on the washing condition, and the behavior of the amount of change in adsorbed P atom qualitatively agreed with the results of the leaching test. PIXE analysis was able to evaluate the trace amount of remaining solvent in vermiculite, which could not be evaluated by measuring only the eluted solvent using NMR analysis. The cleanup method of the remaining solvent in vermiculite and adsorption behavior will be clarified by PIXE analysis.

#### 1079

#### Characterization of hourly-collected air-borne particulate matters by inair micro-PIXE analysis assisted by machine learning

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Transboundary propagation of air pollutions of Particle Matter (PM) is a global issue and often attracts interests among researchers in terms of long-term health care and risks of human beings. To estimate its origin, the elemental composition of PM is one of the most important key factors. However, it is sometimes hardly accomplished with general-purpose technology for hourly-collected PM samples because of its limited concentrations. On the other hand, our previous trials utilizing micro-PIXE analysis on several hourly-collected samples successfully revealed its elemental compositions. Since trend and changes in elemental compositions might play a key role, consecutive dataset of elemental composition should be obtained by micro-PIXE analysis for better understanding and characterization of PM collected in short term period from atmosphere.

In this study, we employed external (in-air) micro-PIXE system placed at QST Takasaki to analyze PM 2.5 and PM 2.5 - 10 particulate matters (PM) obtained through PM sampler of DKK FPM377B placed at Ohta (36.290° N, 139.381° E). Each spot correspond to each time-bin was separated from the tape and irradiated by 3 MeV H+ microprobe with typical scanning area of 400 × 400 µm2. Our house-made software allows us to analyze a set of PIXE spectra by utilizing library of Imfit, non-linear least square method, and elemental composition ratios were delivered from the amount of each element in the peak area divided by the L1 norm of each elemental amount, and dimensionality was reduced by principal component analysis. Through micro-PIXE analysis, data set was successfully and continuously obtained through 26 March 2020 00:00 -22:59, 27 March 2020 while the software code automatically identified several outlier data by classification throughout the campaign of the analysis. Results indicated data analysis successfully assisted micro-PIXE analysis to investigate consecutively obtained PM samples with better error correction functions.

#### 1099

## Trace-elemental analysis of herbal plant leaves and air particulates using Particle Induced X-ray Emission

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Tulsi (Ocimum Sanctum), Hibiscus (Hibiscus Rosa-Sinensis), and Neem (Azadirachta Indica) are known to be of great value in the field of medicine. Due to the many health benefits, these leaves offer, the elemental concentrations levels have been investigated in these types of leaves in past decades. Our study is focused not only on the quantitative estimation of the concentration of both major and minor elements but also on the individual elemental mapping and correlation between those elements at different spatial locations. Two samples of each type of leaf were taken and dried by two different methods to maintain their nutritional value to the highest extent. The solid sample of each leaf was used for micro-PIXE and the powdered sample for the broad beam -PIXE analysis. We obtained the elements in low concentrations and correlated some of the major elements for interesting areas. Our study also showed the distribution of elements in fresh and aged leaves of the same sample showing the increase in chlorine concentration in aged leaves thus turning them yellow. The relation between calcium and potassium yielded interesting arguments along with many trace elements playing a vital role in making these leaves fit for pharmaceutical use. Similarly, we have investigated the distributions of various elements in the air particulates collected from polluted urban areas.

#### 1109

## Microstructure Patterning for Dielectrophoresis-Based Collection of Microparticles for the Detection of Environmental Microplastics

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There have been growing concerns about environmental plastics spreading in our environments including soils, rivers, and oceans, which might affect the ecosystem and human health. To understand the distribution and the formation mechanisms of so-called "microplastics", we need to develop the detection techniques of microparticles ranging in size from micrometers to nanometers. We have been developing techniques to manipulate and collect the micro and nanoparticles such as microbes and metals utilizing dielectrophoresis (DEP). Our devices are typically equipped with microstructures of dielectrics to modulate electric field distribution that induces DEP force which can be switching repulsive and attractive depending on the frequency of applied AC voltage to manipulate micro/nanoparticles [1, 2]. The microstructures of dielectrics such as arrays of pillars and pits were fabricated by proton beam writing (PBW), which is a direct-write, versatile tool for micropatterning.

In this study, we present our study on the trapping effects of micro-sized particles by means of DEP using devices equipped with a single micro pit or arrays of DEP microstructures on 12-\_m-thick PMMA fabricated by PBW. As a model of microplastics, we used commercially available polystyrene (PS) particles with diameters of 1 or 5 m. We patterned the PMMA layer, spin-coated on an indium tin oxide (ITO) layer deposited on polyethylene terephthalate (PET) film. The PBW was performed using a PB writer with protons of beam energy of 1.0 MeV. After patterning the PMMA pit arrays by PBW, we fabricated a microfluidic channel with double-sided adhesive tapes of PET film with a thickness of 100 m to provide a channel with a dimension of 100 m in height, 5 mm in width, and 10 mm in length, where water suspension of the PS particles flowed by capillary force. We observed the trapping of the PS particles by DEP at arrays of various patterns, such as circles with diameters less than 50 m, where the particles were collected not only at the inside and circumference of the circular pits but also at the spaces in between of the pits. We will discuss the trapping mechanisms of the PS particles based on the frequency dependence of applied voltages, and electric field analysis will be made to improve the trapping characteristics for the development of devices to detect environmental microplastics.

 [1] Applications of microstructures fabricated by proton beam writing to electric-micro filters, Yusuke Furuta, Hiroyuki Nishikawa, Takahiro Satoh, Yasuyuki Ishii, Tomihiro Kamiya, Ryota Nakao, Satoshi Uchida, Nuclear Instruments and Methods in Physics Research B 267 (2009) 2285, doi:10.1016/j.nimb.2009.03.
 [2] Assembling gold nanoparticles by dielectrophoresis with pit arrays on PMMA fabricated by proton beam writing, Taichi Shibuya, Satoshi Uchida, Yasuyuki Ishii, Hiroyuki Nishikawa, Nuclear Inst. and Methods in Physics Research B 456 (2019) 60–63. <u>https://doi.org/10.1016/j.nimb.2019.06.050</u>

### MeV-SIMS

#### 1073

#### Chemical sensitivity of MeV-SIMS imaging mass spectrometry

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MeV-SIMS is a technique for chemical maping of biomolecules in organic tissues [1][2]. Within this study, various concentrations of organic molecules were measured with TOF-MS MeV-SIMS in order to assess the limits of its chemical sensitivity. The samples of selected purified molecular material were diluted in logarithmic steps. In parallel, the same sets of solutions were spiked with NaTFA (sodium trifluoroacetate) 0,5g/100ml. The solutions were deposited on a silicon wafer, dried, and inserted into evacuated experimental chamber. MeV-SIMS measurements were executed with primary ion beams of 35Cl at the energies/charge states of 5 MeV/5+, 7.2 MeV/6+ and 10 MeV/7+. Each sample was measured for 15 minutes on an area of 750 µm x 450 µm, which yields beam fluence of less than 109 ions/cm2. All samples exhibited drastic increase of secondary ion yield after the addition of sodium trifluoroacetate. The results of the non-spiked samples demonstrate excellent chemical sensitivity, which suggests high MeV-SIMS potential for molecular imaging of biological tissue. In addition, the latest results and the feasibility study of chemical sensitivity on the applications of the MeV TOF-SIMS setup, will be presented.

- [1] Y. Nakata, S. Ninomiya, and J. Matsuo, "Secondary ion emission from bio-molecular thin films under ion bombardment," Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms, vol. 256, no. 1, pp. 489–492, 2007, doi: <u>https://doi.org/10.1016/j.nimb.2006.12.073</u>.
- [2] L. Jeromel et al., "Molecular imaging of humain hair with MeV-SIMS: A case study of cocaine detection and distribution in the hair of a cocaine user," PLOS ONE, vol. 17, no. 3, pp. e0263338-, Mar. 2022, [Online]. Available: <u>https://doi.org/10.1371/journal.pone.0263338</u>

#### 1024

## Enhancing the irradiation capabilities of the AIFIRA cell micro-irradiation beamline

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The AIFIRA facility in Bordeaux is equipped with a microbeam line dedicated to targeted irradiation for radiobiology studies. This beam line is designed to deliver a precise number of ions at the micrometer scales and to observe the cellular response online using time-lapse fluoresence microscopy [1]. Irradiations with low numbers of protons or alpha particles are controlled using thin transmission detectors [2,3].

In recent years, radiobiology studies performed on this beam line have required to extend the available dose range and dose rates. Initially developped to perform experiments at the micrometer scale, we have extended the irradiation features to deliver homogeneous doses on cm<sup>2</sup> areas using the same end-station. In addition, biophysical studies are currently underway to measure DNA fragmentation and these experiments require to cover a wide range of beam currents, from fA to several hundreds pA. Beam currents above 1 pA can be efficiently measured using Faraday cups and ammeters. We have specifically developed a fast counting system, based on a SiPM (Silicon Photomultiplier) to monitor intensities between 0.1 and 1 pA (i.e. about 10<sup>6</sup> to 10<sup>7</sup> ions / second). Using SiPM technology presents the advantage to have a very compact detector that can be permanently inserted on the sample holder. Finally, a precise characterisation of the microbeam using fluorescent nuclear track detectors (FNTD) have been performed. All these methodological developments will be presented.

[1] S. Bourret et al., « Fluorescence time-lapse imaging of single cells targeted with a focused scanning charged-particle microbeam », Nuclear Instruments & Methods in Physics Research Section B-Beam Interactions with Materials and Atoms, vol. 325, p. 27-34, (2014), doi: 10.1016/j.nimb.2014.02.004.

[2] G. Muggiolu et al., « Single alpha-particle irradiation permits real-time visualization of RNF8 accumulation at DNA damaged sites », Scientific Reports, vol. 7, p. 41764, (2017), doi: 10.1038/srep41764

[3] P. Barberet et al., « Cell micro-irradiation with MeV protons counted by an ultra-thin diamond membrane », Applied Physics Letters, vol. 111, no 24, p. 243701, (2017), doi: 10.1063/1.5009713

#### 1058

#### Calibration of the Segmented Annular Silicon Drift Detector

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For particle induced X-ray emission (PIXE) measurements, the solid angle of the detector is one of the most important parameters for fast and effective experiments. By means of high solid angle, the required measuring time is significantly reduced under beam conditions which further reduces the damaging of the samples. To approach the solid angle of 1sr [1], we instead used a segmented annular detector at the micro-beam end station of the Jožef Stefan Institute accelerator laboratory.

The Rococo 2 segmented silicon drift detector consists of an SDD chip with a central hole of 1.8mm. Each segment is protected by a Be window that fits its crescent shape. Every segment has energy resolution of 130eV and is equipped with a pre-amplifier which provides a readout for each segment individually. In order to increase the statistics of the spectra, the GeoPIXE software allows us to fit spectra from all the 4 segments of the detector at once. To use this feature, a detailed description of the detector efficiency and geometry is needed.

The description of the detector was achieved by measuring thin metallic standards with emitted X-rays in the energy range of 1.7 keV to 27 keV. The measurements were performed in 1-1.5 kHz count rate regime and were recorded via the OMDAQ data acquisition system. The validation of the description was done on bulk samples. In the present work, we are going to analyze in detail the whole procedure of describing the detector as well as discuss the final results.

#### 1086

#### Characterization of fluorescent nuclear track detector based on phosphate glass with silver and europium activator by focused proton microbeam

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Particle Beam Writing (PBW) technique is a distinctive microfabrication technique with high selectivity of targets. Until now, PBW technique has been applied to microfabrication of polymer materials and defect engineering of wide bandgap semiconductor materials [1,2]. To obtain irradiation deliverables more rapidly, it is desired to accomplish precise beam monitoring techniques which allows us to evaluate the region of interest more rapidly during and more precisely after the irradiation. The Solid-State Nuclear Track Detector (SNTD) is commonly used for such usage to confirm particle irradiation. More recently, the Fluorescent Nuclear Track Detector (FNTD), which can be read-out irradiation position repeatedly, has been developed as an alternative to the SNTD. As a type of FNTD, we are investigating radio-photoluminescence (RPL) phosphate glasses (PG). In general, RPL dosimeters do not emit immediate luminescence, and in-situ evaluation using this material has not been realized. Also, it exhibits strong build-up effects that prevent its usage in real-time monitoring of high energy charged particle beams [3].

In this study, we aimed to engineer RPL phosphate glass as an FNTD and develop a device that enables convenient in-situ observation of PBW irradiation as well as precise post-irradiation pattern visualization through fluorescence. The co-doping of silver (Ag) and europium (Eu) as active centers in a base material composed of sodium metaphosphate (NaPO3) and aluminum metaphosphate (Al(PO3)3) was used to fabricate PGs with an integrating function of FNTD and scintillator. The fabricated PG:Eu,Ag were subjected to PBW microbeam irradiation at QST-Takasaki. After irradiation, the fluorescence of possible silver active centers was observed in the irradiated area using a simple modular microscope (Olympus IX71) equipped with an excitation light source wavelength of 365 - 375 nm.

Scintillation were observed during PBW pattering on PG:Eu,Ag by 3 MeV protons. Fluorescence disappeared from areas other than the irradiation position, indicating that only the sequential irradiation position could be confirmed through scintillation. After the irradiation, PG:Eu,Ag was observed under ultra violet light exposure. The irradiated pattern was clearly observed as the patterns of radio-photoluminescence with spatial resolution of approximately in 1µm. The results suggested that we have successfully demonstrated an integration of FNTD with scintillator in same substrate.

Acknowledgements: This work has been partially supported by MEXT/JSPS Grant-in-Aid for Scientific Research. JP21K12522.

<sup>[1]</sup> W. Kada, et al., Nucl. Instr. and Meth. Sec. B., 348 (2015), pp.218-222, doi: 10.1016/j.nimb.2014.12.041.

<sup>[2]</sup> H. Kraus et al., Nano Letters, 17(5), (2016), pp. 2865-2870. doi: 10.1021/acs.nanolett.6b05395

<sup>[3]</sup> T. Kurobori, et al., Jpn. J. Appl. Phys., 57(2018) 106402, doi: 10.7567/JJAP.57.106402.

#### 1107

## Characterisation of radiation detectors by IBIC: overview of experiments performed through transnational access at RBI

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Performance of over thirty experimental Sessions for external users of the RBI microprobe systems, with an aim to investigate charge transport properties of variety of detector structures, illustrates the strengths and the versatility of the ion microprobe technique IBIC. Experiments were funded in recent years through the transnational access (TA) scheme of the Horizon2020 projects AIDA2020 and RADIATE. Versatility of IBIC is demonstrated by the possibility to choose ions and their respective energies in a way to use appropriate ionization density and ion penetration depth for particular problem. Detectors based on different materials (Si, diamond, SiC, GaN, etc.) were probed to depths from as low as 100 nm (for 140 keV Cu ions in diamond) up to 0.5 mm (for 6 MeV protons in silicon). From very simple planar detector configurations, to complex multipixel detector arrays that may even include regions with charge multiplication, IBIC has shown to be the most suitable technique for resolving issues that limit targeted detector performances. Upgrades of the RBI microprobe systems that have been particularly useful include: possibility to conduct in-air IBIC measurements, as well as performance of time resolved (e.g. TCT and QTS) experiments. Characteristic examples of external users' experiments will be presented.

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### **Multimodal Imaging**

#### 1037

## Bioimaging of single cells with nuclear microscopy: far more than morphology

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Nuclear microscopy offers unique possibilities in single cell analysis. By combining common techniques such as, STIM, PIXE and EBS techniques together with ion beam analytical tools, such as OMDAQ2007 and WinDF, cells can be characterized beyond mass and elemental distributions. The depth structure of cells and the depth distribution of endogenous and exogenous elements can be obtained, providing detailed insights into the complex organisation of single cells.

The main goal is to establish biological signatures of single cells that will enable to identify cellular alterations upon exposure to metal complexes quantify the uptake and identify cellular targets.

To define biological patterns, ovarian cancer cell lines A2780, OVCAR3 (cisplatin sensitive) and OVCAR8 (cisplatin resistant) were used. A gold complex<sup>1</sup> which had shown promising biological properties was selected to examine its uptake and depth distribution in treated cells. Single cells were analysed in vacuum at the IST/CTN nuclear microprobe (Oxford Microbeams Ltd., UK) under routine conditions (2.0MeV proton beam of 100pA, and spatial resolution ~3×4µm<sup>2</sup>). For biological signatures, features such as, cell morphology, elemental compartmentalization and quantitation, and layer structure, were considered. Data was obtained by combining STIM, PIXE and EBS spectral information. Data acquisition, 2D mass and elemental maps, and elemental concentrations were carried out using OMDAQ 2007 software.<sup>2</sup> To define a high-resolution depth profile structure of the different cell lines, a WinNDF approach combining EBS and PIXE data for multi-layered structured samples was used.<sup>3</sup>

Results showed that structural details of A2780, OVCAR3 and OVCAR8 were similar, although minor morphological details associated with P and S compartmentalization were detected for OVCAR8. However, after incubation with the gold complex a differential elemental compartmentalization of P, S and K was observed for the three cell lines, which were not correlated with differences in cellular permeability for the gold complex. Altogether, the biological signatures, layer structure, and distribution pattern of gold in the three cell lines launched the basis of a cell inventory that will help identifying possible cellular targets for the studied gold complex. The approach is also very important to define strategies for the identification of modes of action of new metal-based drugs with therapeutic potential.

[2] http://www.microbeams.co.uk/index.html (accessed May 2022)

[3] Barradas NP, Jeynes C (2008) Advanced physics and algorithms in the IBA DataFurnace, NIMB 266:1875–1879.

<sup>[1]</sup> Costa JP, Sousa SA, Soeiro C, et al. (2021) Synthesis and Characterization of Camphorimine Au(I) Complexes with a Remarkably High Antibacterial Activity towards *B. contaminans* and *P. aeruginosa*. Antibiotics 10: 1272.

#### **Poster Session I**

### **Multimodal Imaging**

#### 1043

#### Proton irradiation induced changes in Polyvinyl Butyral (PVB) support films for analysis of biomedical samples in the MeV ion microprobe

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Support films are important for analysis in the MeV ion microprobe of biological cell suspensions and tissue sections. Si3N4 membranes have been used for cells e.g. [1]. Pioloform® which is a linear random terpolymer with polyvinyl butyral (PVB), polyvinyl alcohol (PVA) and polyacetate (PAC) repeating units (RU). Pioloform has been used by the CIBA group in Singapore and by some of us in Lafayette [2] for supporting tissue sections for analysis with a MeV ion microbeam. PVB has the advantage that is mechanically strong, can be easily mounted on a supporting ring to give a ~0.5 µm self-supporting films containing only C, H and O. PVB is expected to have a high resistance to degradation under irradiation because the repeating unit structure is largely made up of a heterocyclic ring which are generally act as sinks for excitation energy [3]. Recently, we have proposed to use PVB as an internal standard to quantify the major element content of dehydrated tissue sections. Hence, a quantitative understanding of the ion-irradiation degradation of PVB is important. Ion-irradiation induced degradation of PVB will also affect the energy loss contrast in onand off-axis Scanning Transmission Ion Microscopy (STIM). In this preliminary study, the samples were prepared by dipping a substrate and subsequently floating-off the Ploloform films on deionized water where they were picked up on supporting stainless steel rings. the loss of the constituent elements, H, C and O were measured using 2 MeV protons at the microprobe beamline at the Tandem accelerator Laboratory of the University of Uppsala. This was done by measurement of the H, C and O signals using off-axis Scanning Transmission Ion Microscopy (OA-STIM) at 30° and simultaneously the C and O signals by Rutherford Backscattering Spectrometry (RBS). The GeoPIXE code was used to produce 1D energy spectra. The results revealed a loss of material from the film with increasing fluence. The fluence dependence of the C and O contents measured with RBS and OA-STIM C and RBS were in agreement within the limits of statistical uncertainty. The H and C signal dependence on fluence could be fitted with the sum of an exponential decay and a constant residual content. O-loss exhibited a more complex behavior indicating O-loss through at least two processes. The O/C composition from OA-STIM and RBS, extrapolated to zero fluence corresponded to a composition with one H2O molecule absorbed per RU.

[1] Ren M., van Kan, J.A, Bettiol A.A., Lim D., Chan Y. G., Bay. B. H., Whitlow H.J., Osipowicz T., Watt F. (2007); Nanoimaging of single cells using STIM; Nuclear Instrum. Methods B 260; 124.

[2] Whitlow H.J., Deoli, N De Vera A., Morgan K., Villinger F.(2021); Heavy Elements Revealed in Jejunum of Simian Immunodeficiency Virus Infected Monkeys by Microparticle Induced X-Ray Emission; Phys Stat. Sol. A 218; ttps://doi.org/10.1002/pssa.202000107

[3] Chapiro A., (1988); Chemical midification of organic polymers; Nucl. Instrum. Methods B 32:111-114

## **Multimodal Imaging**

#### 1057

#### Artificial Neural Networks for high-resolution 3D imaging

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Rutherford Backscattering Spectrometry (RBS) spectra, when recorded using a nuclear microprobe, allows visualizing the elemental distribution in 2D maps, to identify the elemental matrix of an unknown sample and the depth profiling of those elements. Using OMDAQ software, each scanned area is acquired as a 256x256 pixel map, each pixel containing all the IBA spectra recorded during the experiment.

A step forward in 3D imaging would be to represent the elemental depth profiling obtained in each pixel of the map. This means to analyse more than 65 thousand RBS spectra, or more than 16 thousand spectra if the maps suffer a 2x2 pixel compression. In any case, the number of RBS spectra to be analyzed requires time and computing resources. To tackle this problem we are developing an artificial neural networks (ANNs) model, which once trained, can handle the analysis of large data sets instantaneously [1]. The potential of ANNs to automatically render depth profiles of several types of samples in a 3D environment will definitely extend the imaging capabilities of nuclear microprobes.

Herein we will report on an ANNs method to perform an automated analysis and classification of RBS spectra recorded during nuclear microscopy analysis of cells exposed to Cu and Au complexes or nanoparticles [2,3]. The 3D visualization of cellular distribution of Cu and Au, were previously obtained using the MORIA software by calculating the energy loss of the RBS spectra [2]. However, MORIA generates a solution for 3D visualization of heavy elements in light sample matrices. The development of an ANN algorithm trained to find a general solution for depth profiling using RBS data, has to be indifferent to sample elemental composition. In this context the results obtained with MORIA are very important in the validation of an ANN model. Challenges as the low statistics of the RBS spectra, the estimated time requirements for training the ANNs, or the visualization of the results will be presented and discussed.

- [1] J. Demeulemeester, D. Smeets, N.P. Barradas, A. Vieira, C.M. Comrie, K. Temst, A. Vantomme, (2010) Artificial neural networks for instantaneous analysis of real-time Rutherford backscattering spectra. Nucl Instruments Methods Phys Res Sect B Beam Interact with Mater Atom, 268: 1676-1681. Doi: 10.1016/j.nimb.2010.02.127
- M. Vasco, L.C. Alves, V. Corregidor, D. Correia, C. Godinho, I. Sá-Correia, I., A. Bettiol, F. Watt, T. Pinheiro, (2017)
  3D map distribution of metallic nanoparticles in whole cells using MeV ion microscopy. Journal of Microscopy, 267: 227-236. https://doi.org/10.1111/jmi.12561
- [3] Dominelli B, Jakob CHG, Oberkofler J, Fischer PJ, Esslinger E-M, Reich RM, Marques F, Pinheiro T, Correia JDG, Kühn FE. Mechanisms underlying the cytotoxic activity of syn/anti-isomers of dinuclear Au(I) NHC complexes. European Journal of Medicinal Chemistry 2020;203:112576. DOI:10.1016/j.ejmech.2020.112576

## **Multimodal Imaging**

#### 1083

## At the roots of the multiscale imaging of biological samples: charting the connectivity between kingdoms

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Plant structures are defined across differing scales. Subcellular organization of nanometer proportions predisposes structural composition at the micro, meso, and macro levels. However, plant tissues are not sterile. In the contrary, they are frequently colonized by organisms from different groups (viruses, bacteria, fungi).

Below the surface, roots of higher plants are colonized by fungi from differing taxonomic groups forming distinct structures called morphotypes. Using light microscopy, we discovered different fungal morphotypes on the willow's (*Salix caprea*) root system from a metal polluted area [1]. Beneficial fungal endophytes are of immense importance for successful phytoremediation [2]. However, not all fungi on plant roots are symbiotic and beneficial. To reveal the contribution of differing morphotypes to plant metabolism, we applied micro-particle-induced X-ray emission (micro-PIXE) for quantitative tissue-specific analysis of elements, thus allowing for discerning metal tolerance mechanisms in plants [3]. Expectedly, both fungal and plant partners had distinct elemental compositions. Even more, tissue-specific elements were found that enabled us to discern diverse plant tissues within a single root.

The structural composition of morphotypes is even more complex than the roots themselves. Scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (EDS) analysis was applied to allow for a more detailed insight into the morphotype's morphology and composition at the micro scale.

This experiment confirms that micro-PIXE is a suitable technique for charting plant-fungal interactions using quantitative tissue-specific elements analysis and that multiscale elemental analyses are required to improve our understanding of the mechanisms behind inter-kingdom interactions.

- [1] Regvar, M., Likar, M., Piltaver, A., Kugonič, N., & Smith, J. E. (2009). Fungal community structure under goat willows (Salix caprea L.) growing at metal polluted site: the potential of screening in a model phytostabilisation study. *Plant* and Soil, 330(1–2), 345–356. <u>https://doi.org/10.1007/s11104-009-0207-7</u>
- [2] Regvar, M., Vogel-Mikuš, K., Kugonič, N., Turk, B., & Batič, F. (2006). Vegetational and mycorrhizal successions at a metal polluted site: Indications for the direction of phytostabilisation? *Environmental Pollution (Barking, Essex : 1987), 144*(3), 976–984. https://doi.org/10.1016/j.envpol.2006.01.036
- [3] Vogel-Mikuš, K., Simčič, J., Pelicon, P., Budnar, M., Kump, P., Nečemer, M., Mesjasz-Przybyłowicz, J., Przybyłowicz, W. J., & Regvar, M. (2008). Comparison of essential and non-essential element distribution in leaves of the Cd/Zn hyperaccumulator Thlaspi praecox as revealed by micro-PIXE. *Plant, Cell and EnvironmentEnvironment*, 31(10), 1484–1496. <u>https://doi.org/10.1111/j.1365-3040.2008.01858.x</u>

#### **Poster Session I**

## **Multimodal Imaging**

#### 1103

## Co-location of lipids, drugs and metal biomarkers using spatially resolved lipidomics with elemental mapping

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Elemental imaging is widely used for imaging cells and tissues but rarely in combination with organic mass spectrometry, which can be used to profile lipids and measure drug concentrations. Here we demonstrate how elemental imaging using PIXE and a new method for spatially resolved lipidomics (DAPNe-LC-MS [1,2], based on capillary micro-sampling and liquid chromatography mass spectrometry) can be used in combination to probe the relationship between metals, drugs and lipids in discrete areas of tissues. This new method for spatial lipidomics, reported here for the first time has been applied to rabbit lung tissues containing a lesion (caseous granuloma) caused by tuberculosis infection. We demonstrate how elemental imaging with spatially resolved lipidomics, can be used to probe the association between ion accumulation and lipid profiles and verify local drug distribution.



- [1] de Jesus, J.; Bunch, J.; Verbeck, G.; Webb, R. P.; Costa, C.; Goodwin, R. J. A.; Bailey, M. J., Application of Various Normalization Methods for Microscale Analysis of Tissues Using Direct Analyte Probed Nanoextraction. *Analytical Chemistry* 2018, *90* (20), 12094-12100
- [2] Lewis, H.-M.; Webb, R. P.; Verbeck, G. F.; Bunch, J.; de Jesus, J.; Costa, C.; Palitsin, V.; Swales, J.; Goodwin, R. J. A.; Sears, P.; Bailey, M. J., Nanoextraction Coupled to Liquid Chromatography Mass Spectrometry Delivers Improved Spatially Resolved Analysis. *Analytical Chemistry* **2019**, *91* (24), 15411-15417

## **Multimodal Imaging**

#### 1108

## Towards high spectral AND spatial resolution in a single PIXE system: development of a microbeam line with a microcalorimeter detector

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The University of Surrey Ion Beam Centre has recently been awarded funding for a new microcalorimeter X-ray detector for particle induced X-ray emission (PIXE). These detectors can provide 1 eV energy resolution at 5 keV. Single pixel microcalorimeter detectors have been used in conjunction with PIXE before, but to our knowledge, this is the first system, and first multi-pixel detector to be installed on a nuclear microprobe. A resolution of ~1 eV is sufficient to enable the disambiguation of overlapping peaks and has been shown to reduce the minimum detectable limit by an order of magnitude. It is also sufficient to provide information on chemical shifts to provide information on the chemical state of atoms [1]. Our vision is to use this detector to catalyze new applications in several disciplines. For example in biosciences, the system could enable the binding state of bioaccumulated metals. In forensics and environmental science, the detector may be used to provide enhanced discrimination power for particulate materials and in earth sciences it could help to measure the phase partition of rare earth elements used as geothermometers. In this poster, we will report on progress towards the design and installation of a microcalorimeter detector on our microbeam line, for high energy resolution imaging.

 Chemical State Analysis Employing Sub-Natural Linewidth Resolution PIXE Measurements of Kα lines, M. Kavi, et al., X-Ray Spectrom. 34, 310, (2005)

## Through different eyes... Forty years of nuclear microscopy at the micrometre scale

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2020 marked the fortieth anniversary of the first published images using ion beam analysis (IBA) at a spatial resolution of 1 micrometre. IBA exploits the reaction products emitted when samples are exposed to a beam of megaelectron volt ions from a small nuclear accelerator. These include light and X-rays from atomic electrons, particles and gamma rays from the nuclei and forward or backward scattered beam particles. These can provide elemental concentrations with high sensitivity, specificity and accuracy. In many cases, depth profiles, structural and chemical information can also be measured. In the late 1970s, our group in Oxford developed the techniques for focusing high energy ion beams to micrometre size diameters and so produced the first imaging nuclear microprobe. Now the technique is available in laboratories across the world and is applied in fields ranging from archaeology to zoology. In this talk, I will outline the background and history of the nuclear microprobe and describe two recent projects carried out at the University of Surrey which highlight the wide range of applications: the reconstruction of the lost image on a weathered stained-glass window and identifying the metal atoms in protein molecules.

WEDNESDAY, 14 SEPTEMBER

## **SESSION 1**

MULTIMODAL IMAGING

Chairs: Johanna von Gerichten and Teresa Pinheiro

#### Invited talk

#### 1100

## IBA Imaging Techniques - exploring their potential in forensics as independent tools and within a multimodal approach

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In recent years, the Laboratory for Ion Beam Interactions has participated in several international projects (COST Action, IAEA CRP) aimed at exploring the potential of Ion Beam Analysis (IBA) imaging techniques and multimodal imaging in solving some difficult problems in forensic science. Here we will present the results obtained so far in the field of analysis of questioned documents. It will be demonstrated how elemental and molecular imaging using proton-induced X-ray emission and secondary ion mass spectrometry with MeV ions (MeV SIMS) can detect the correct order of deposition of different types of inks and toners [1]. MeV SIMS is a relatively new analytical technique in the ion beam community and is at the same time completely unknown to forensic community. Technique detects molecules from uppermost layers of the sample making it suitable for determining the order of deposition. Also, in cooperation with forensic experts from the Ivan Vučetić Center for Forensic Science, several very difficult cases were studied using standard non-destructive forensic techniques (optical techniques, Raman) and MeV SIMS. The studied cases are divided into those in which optical techniques can distinguish the inks used from those that are not completely different from them. In the cases of indistinguishable inks, the mass spectra of the inks used were also found to be very similar, indicating an almost identical chemical composition of the inks used. However, these very small differences in mass spectra in combination with advanced and objective analytical models were sufficient to resolve the correct order of deposition at the intersections in most of the cases studied [2].

- [1] Moore KL, Barac M, Brajković M, Bailey MJ, Siketić Z., Bogdanović Radović I., Determination of Deposition Order of Toners, Inkjet Inks, and Blue Ballpoint Pen Combining MeV-Secondary Ion Mass Spectrometry and Particle Induced X-ray Emission. // Analytical chemistry, 91 (2019), 20; 12997-13005
- [2] Barac M., Filko A., Siketić Z., Brajković M., Ledić A., Radović, Bogdanović Radović I., Comparison of optical techniques and MeV SIMS in determining deposition order between optically distinguishable and indistinguishable inks from different writing tools // Forensic Science International, 331 (2022), 111136-111146

#### 1018

## Sequential molecular and elemental imaging on a single tissue section using DESI and PIXE

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Elemental and molecular imaging play a crucial role in understanding disease pathogenesis but are rarely used in combination. However co-localising elements and metabolites is important to biologists understanding the impact of metallic impurities or bioaccumulated metals on the host. To accurately correlate elemental and molecular markers, it is desirable to perform sequential elemental and molecular imaging on a single-tissue section. However, very little is known about the impact of performing these measurements in sequence.

In this presentation, we highlight some of the challenges and successes associated with performing elemental mapping in sequence with mass spectrometry imaging. Specifically, the feasibility of molecular mapping using the mass spectrometry imaging (MSI) techniques matrix-assisted laser desorption ionization (MALDI) and desorption electrospray ionization (DESI) in sequence with the elemental mapping technique particle-induced X-ray emission (PIXE) is explored. Challenges for integration include substrate compatibility, as well as delocalization and spectral changes. We demonstrate that, further to our published work [1], delocalisation of trace elements can be mitigated through adaption of the DESI method. This allows sequential DESI-PIXE imaging without any compromises to either the DESI or PIXE spectra. This approach will enable research into the impact of metal accumulation to the host metabolism in tissues and should be of broad interest in the biosciences.

#### Contributed talk II

#### 1001

## Exploring new methods to monitor proton beam damage for sequential elemental and molecular imaging on a single tissue section

<u>C. Costa<sup>1</sup></u>, J de Jesus<sup>2,3</sup>, C. Nikula<sup>3</sup>, G. Grime<sup>1</sup>, V. Palitsin<sup>1</sup>, R. Webb<sup>1</sup>, R. J. A. Goodwin<sup>4,5</sup>, J. Bunch<sup>3</sup>, M. Bailey<sup>1,2</sup>

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Monitoring the effects of proton beam damage in biological materials is of interest to enable the integration of proton microprobe elemental mapping with molecular imaging modalities and further our knowledge of chemical changes caused to tissues during proton beam cancer therapy. In a recent publication, de Jesus *et al.* [1] reports on performing sequential mass spectrometry imaging (MSI) and ion beam analysis (IBA) on the same tissue sample. Data indicated that irradiating the sample with a proton beam caused measurable changes in the lipid profiles detected between irradiated and non-irradiated regions. Here we describe a novel approach to characterise proton beam damage to lipids in biological tissues, based on mass spectrometry imaging, specifically desorption electrospray ionisation (DESI). The developed methodology is applied to characterise changes to lipid profiles irradiated under different conditions: ambient pressure, high vacuum, beam scan speed and pattern and application of a matrix to the sample. This work shows that performing proton beam irradiation at ambient pressure, as well as the application of an organic matrix prior to irradiation can reduce changes in lipid profiles in tissues.



High fluence Medium fluence Low fluence

**Figure 1:** Extracted ion map for m/z 848.557 (tentatively assigned to PC 38:4) from a liver tissue homogenate sample, obtained using DESI following irradiation with a 2.5 MeV proton beam under vacuum, irradiated at low, medium and high fluences.

<sup>[1]</sup> de Jesus JM, Costa C, Burton A, Palitsin V, Webb R, Taylor A, Nikula C, Dexter A, Kaya F, Chambers M, Dartois V, Goodwin RJA, Bunch J, Bailey MJ (2021) Correlative Imaging of Trace Elements and Intact Molecular Species in a Single-Tissue Sample at the 50 µm Scale. Analytical Chemistry 93 (40):13450-13458. doi: https://doi.org/10.1021/acs.analchem.1c01927

#### **Contributed talk III**

#### 1026

## Application of segmented annular Silicon Drift Detector for 3-D surface topography reconstruction by micro-PIXE

<u>Ebrahim Gholami Hatam<sup>1</sup></u>, Primož Pelicon<sup>2</sup>, Esther Punzon<sup>2</sup>, Mitja Kelemen<sup>2,3</sup>, Primož Vavpetič<sup>2</sup> and Paula Pongrac<sup>2,4</sup>

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Having access to the third dimension of elemental distribution either in the form of surface topography or in the form of height profile is valuable when evaluating the experimental data in ion beam community. Authors, previously, had introduced an innovative approach to reconstruct the surface topography using stereovision of the 2D elemental X-ray map induced by focused MeV proton beams [1-5]. The proposed method using two X-ray spectrometer was restricted to drive the inclination angles just in the lateral direction of the obtained maps. Here, we benefit from the four segment Silicon Drift Detector (SDD), with an annular symmetric geometry mounted at the Jožef Stefan Institute (JSI) microprobe, to reconstruct the roughness height profile both in the lateral and longitudinal direction of the obtained 2D X-ray elemental maps.

We proposed that stereo-PIXE method can be extended to the four-segment detector by considering a set of stereovision maps acquired by individual segment. In a similar manner as in [1], the X-ray intensity disparity from the multi view of the elemental distribution, applying an ideal flat reference model of the sample, was used to infer the normal surface angle and in consequence the gradient of the topography along the planes that intersect the two opposing segments. Consequently, we executed the integration of the height gradient in the two orthogonal directions along the sample surface in order to reconstruct the 3-D sample surface tomography profile. The method is demonstrated on the bulk Ti sample with engraved grid structure.

Acknowledgment: This work was supported by the TNA No. 21002430-ST within the EU H2020 project No. 824096 RADIATE.

- [1] Gholami Hatam E, Pelicon P, Lamehi-Rachti M, et al (2012) Surface topography reconstruction by stereo-PIXE. J Anal At Spectrom 27:834-40. doi: 10.1039/C2JA10373G
- [2] Gholami Hatam E, (2012) Surface topography of 1€ coin measured by stereo-PIXE. Nucl Instruments Methods Phys Res Sect B Beam Interact with Mater Atoms 306:90-93. doi: 10.1016/j.nimb.2012.12.024
- [3] Gholami Hatam E, (2015) Reconstruction of relief by means of stereo-PIXE set-up for curved target. Nucl Instruments Methods Phys Res Sect B Beam Interact with Mater Atoms 348:48-52 doi: 10.1016/j.nimb.2015.01.047
- [4] Gholami Hatam E, (2017) Void and cavity determination in micro-PIXE analysis of composed material using binocular detectors: A computational study. Nucl Instruments Methods Phys Res Sect B Beam Interact with Mater Atoms 404:189-92 doi: 10.1016/j.nimb.2017.01.066
- [5] Gholami Hatam E, (2019) The application of stereo scanning transmission ion microscopy (stereo-STIM) imaging in biological specimen. Nucl Instruments Methods Phys Res Sect B Beam Interact with Mater Atoms 450: 127-30 doi: 10.1016/j.nimb.2018.11.014

#### **Contributed talk IV**

#### 1082

# Tartary buckwheat grain as a model for spatially-resolved element and molecular distribution in plants: solving structurally-related biochemistry using multimodal imaging

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Elements and metabolites in plant tissues are strongly affected by the environment in which they are produced. Homeostatic controls, therefore, exist to neutralize their potential adverse effects that strictly depend on the properties of the structures of target cells and tissues. Micro-particle-induced X-ray emission (micro-PIXE) enables quantitative tissue-specific spatial analysis of nutritionally important and potentially harmful elements in freeze-dried or frozen-hydrated samples with an imaging resolution below 1  $\mu$ m. Secondary Ion Mass Spectrometry (SIMS) imaging method, referred to as MeV-SIMS [1], is a complementary technique used for molecular imaging of biological tissues where large non-fragmented bio-molecules are to be analyzed. As such, both methods have an excellent potential to help resolve fundamental questions in food quality and safety.

To demonstrate the applicability of these two complementary techniques, we selected Tartary buckwheat (*Fagopyrum tataricum* Gaertn.) grain as a suitable model due to its high tissue diversity resembled in distinct elemental spatial resolution demonstrated previously using micro-PIXE [2] and its large concentrations of antioxidants rutin and quercetin [3]. Both analyses can be performed subsequently at the accelerator facility of Jožef Stefan Institute, Slovenia. Targeted and non-targeted approaches were used. However, the identification of metabolites and their biological interpretations remains challenging. Therefore, it is of utmost importance that these structures remain intact and discernable during sample preparation and imaging. After micro-PIXE, the morphology of the sample was captured using scanning electron microscopy followed by laser-ablation-inductively-coupled plasma MS to determine localisation of Al and Si with better sensitivity. Furthermore, prior to the MeV-SIMS, bright light and UV-excitation microscopies were used to capture morphology of the sample. This example demonstrates Tartary buckwheat grain is a well-suited model for testing several complementary techniques on the same heterogeneous, plant specimen of intermediate thickness.

<sup>[1]</sup> Nakata Y, Honda Y, Ninomiya S, Seki T, Aoki T, Matsuo J. Yield enhancement of molecular ions with MeV ioninduced electronic excitation. Appl Surf Sci. 2008;255:1591–4.

<sup>[2]</sup> Pongrac P, Vogel-Mikuš K, Jeromel L, Vavpetič P, Pelicon P, Kaulich B, et al. Spatially resolved distributions of the mineral elements in the grain of tartary buckwheat (Fagopyrum tataricum). Food Res Int. 2013;54:125–31.

<sup>[3]</sup> Fabjan N, Rode J, Košir IJ, Wang Z, Zhang Z, Kreft I. Tartary buckwheat (Fagopyrum tataricum Gaertn.) as a source of dietary rutin and quercitrin. J Agric Food Chem. 2003;51:6452–5. doi:10.1021/jf034543e.

### WEDNESDAY, 14 SEPTEMBER

## **SESSION 2**

### DETECTORS

Chairs: Željko Pastuović and Shigeo Matsuyama

#### Invited talk

#### 1035

## Development and characterisation of 3D MEMS technologies for isotropically responsive silicon radiation dosimeters.

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In recent years, a great deal of research has gone into develop radiation detectors with an isotropic response to radiation fields. Applications such as dosimetry for radiotherapy, space medicine, radioprotection impose scenarios so complex that the current detector technology and instrumentation does not satisfy the required performance. This work presents an overview of our approach, methods and achievements in addressing this technology gap using 3D MEMS technologies. 3D MEMS, successfully adopted in the past for applications such as microdosimetry, can be used to fabricate a detector with an electric field uniformily distributed across the entire sensitive volume, without any passive layer. Such detector would have a response that is invariant in respect to the direction of the radiation. This can be achieved by the use of active edges which consist in the creation of p-n junctions at every edge of the substrate. Two innovative technologies are proposed: a perforated edge detector from SINTEF (Norway) which requires no handling wafer, and the edgeless sensors by VTT (Finland), fabricated with a laterally ion implantation technique. The IBIC microscopy proved again to be an indispensable tool for prototyping semiconductor detectors with complex configurations on the micrometre scale. The perforated edge devices show a basic p+/n/n+ structure. The n-type substrate is 300 um thick with an area of 5x5 mm<sup>2</sup>. The edges are perforated by deep reactive ion etching in various distribution and geometries (N,R,O,V) [1]. The edgeless sensors from VTT are manufactured using a handling wafer and lateral implantation of Boron and Phosphorous ions. The substrate is 10 kOhm-cm p-type (samples PP/NP) or n-type (PN/NN), 1.5x1.5mm<sup>2</sup> and thicknesses of 100 and 500um [2]. The sensors have been investigated by IBIC using a high-resolution scanning 5.5 MeV He<sup>2+</sup> ion microbeam before and after irradiation with a Co-60 gamma source in order to evaluate the effect of accumulated dose on the charge collection efficiency. The effectiveness of the active edge technologies to create a uniform electric field distribution has been quantitatively assessed as a function of the reverse bias applied, the type of substrate and geometries of the trenches. Synopsys TCAD simula-tions was used for inter-pretation of the IBIC results and they will be presented in full at the conference.

- O. Koybasi, et al., Nuclear Instruments and Methods in Physics Research Section A, vol. 953, p. N.PAG, 2020.
- [2] M. Petasecca, et al., Medical physics (Lancaster), vol. 42, no. 8, pp. 4708–4718, 2015.



trenches at the edge of the detector from SINTEF. b)TOP: Median maps of the charge collection efficiency obtained for a PP and NP edgeless combinations; BOTTOM: TCAD derived electric field distribution of the NP edgeless diode (top) and PP edgeless diode (bottom). The simulation shows the weak electric field of the PP structure which justify the lack of CCE observed at the corners of the PP diode in the median maps.

### DETECTORS

#### 1004

## A new concept for an ion beam spot size monitor to evaluate the resolution of low energy micro and nano-beams

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MeV ion beams are a primary tool in the modification, functionalization and analysis of solid state materials. While the scientific community has recently started to employ the target samples themselves as resources for single ion detection with position sensitivity [1], their exploitation as diagnostic tools for the assessment of ion beam parameters is still under investigation. In the proposed case of study, a custom Si photodiode micromachined via FIB milling is exploited as an integrated beam diagnostic tool for the real-time assessment of the beam spot size of the probe beam. In particular, a dedicated Ion Beam Induced Charge experiment was performed to extract spatial information on the size of a 2MeV Li+ ion micro-beam taking advantage of the spatial correlation between the induced charge pulses amplitude and the micro-structures through Charge Collection Efficiency (CCE) measurements. In contrast to the main techniques commonly adopted by the scientific community, such as STIM (Scanning Transmission Ion Microscopy), RBS (Rutherford back-scattering) and PIXE (Particle Induced X-ray Emission), which rely on the imaging of patterned standards to access beam resolution, the proposed approach allows the qualification of the ion beam by the CCE mapping of the very same target of the ion beam analysis, avoiding possible limitations in the accurancy of the beam size estimation due to the need of a separate detection setup [2]. The suitability of the micro-structures to locally modify the functional properties of the device under test, was preliminary monitored by photocurrent confocal microscopy and the comparison with STIM results confirmed the effectiveness of the method, actually exhibiting a better resolution estimate. Additionally, numerical simulations based on the Shockley-Ramo-Gunn model were carried out for data analysis, validating the interpretation of the experimental results as originating from the effects of the charge implanted during the FIB micromachining on the the measured charge induction.

To conclude, emerging fields such us deterministic implantation, ion lithography and micro radiobiology, where the accurate control on the beam size and its resolution plays a crucial role, could benefit from the proposed technique, since it could offer a reliable method to routinely monitor the resolution of ion microbeams processes and experiments.
## DETECTORS

#### Contributed talk II

## 1032

## Assessment of the radiation hardness of 4H-SiC Schottky diodes by IBIC

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We report findings of the charge collection efficiency (CCE) degradation in a 4H-SiC Schottky barrier diode, which was exposed to selective area (patterned) heavy (carbon) ion irradiation with fluences ranging from 2 to 1000 ions/ $\mu$ m<sup>2</sup>.

The Schottky barrier diode was formed by a Ni/Au metal electrode deposited on a n-type 4H-SiC epitaxial layer grown (50  $\mu$ m thick) on a n+ substrate. The ohmic contact was a Ti/Pt/Au electrode deposited on the substrate side (C-face).

The cleavage of the diode allowed the exposure of the diode's lateral cross section to ion microbeam irradiation for the lateral IBIC microscopy.

I BIC microscopy was carried out at the Laboratory for Ion Beam Interaction (LIBI) of the Ruder Boskovic Institute in Zagreb (HR), following the guidelines in ref. [1]; we used 4 MeV protons and 20 MeV C ions as probing and damaging ions, respectively.

The widening of the depletion layer as function of the applied bias voltage is in a good agreement with the results extracted from capacitance-voltage characteristics, and the hole diffusion length of (~ 5  $\mu$ m) in the pristine diode.

The damage induced by the C ions in six different rectangular regions (about 150x70  $\mu m^2$ ) was evidenced by the CCE mapping resulting from lateral IBIC configuration.

The CCE profiles of the damaged regions show a progressive shriking of the depletion layer as the damaging fluence increases, which indicate a remarkable perturbation of the local electrostatics landscape, to be attributed to the creation of donor-like defect structures, and/or space charge generation induced by the carbon implantation.



Figure 1: Frontal (top) and lateral (bottom) IBIC map of the damaged region.

[1] The IAEA guidelines for the determination of standardized semiconductor radiation hardness parameters, is the outcome of the Coordinated Research Project F11016 of the International Atomic Agency, Vienna and is to be published in Technical Report Series n. 490.

## DETECTORS

## 1062

# Evaluation of charge transport and defect dynamics in semiconductors using ion microprobe – new insights and results

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The examples presented here will demonstrate the unique capability of the ion microprobe to extract information, at both spatial and temporal scales, about the charge transport properties and defect dynamics in semiconductors. This is in particular relevant for characterization of the operation of new generations of solid-state radiation detectors.

The first example is related to the operation of the novel silicon LGAD (low gain avalanche diode) detectors with superior timing resolution due to introduction of the charge multiplication. MeV protons of different energies were used to inject a high density of charge carriers in the device at different distances from the multiplication zones. The induced signal is measured as a function of electric field to quantify the gain factor, which is significantly affected by the charge cloud density, and which is reduced due to the diffusion of charge carriers. The mechanism of the observed gain suppression was fully explained by simulations of charge transport obtained using the TCAD code, which includes both drift and diffusion effects.

The second example is the study of trapping/detrapping phenomena in diamond, another example of the novel radiation detectors. High-purity single-crystal CVD diamond samples metallized with tungsten electrodes have been investigated at elevated temperatures. Focused MeV ions were used, firstly to induce the radiation damage, and secondly for the probing of charge transport properties at elevated temperatures of up to 450 °C. Thermally induced detrapping effect was measured at certain temperatures, by observing a long component ( $\sim$ µs) in the time structure of the induced charge signal. The energy position (0.53 eV) and other properties of the responsible deep trap were determined. In addition, space-charge-limited regime of charge transport has been studied by monitoring transient current signals in the pristine diamond. Shape analysis of signals collected at different temperatures was used to conclude that volume-polarization due to hole trapping is greatly mitigated at low elevated temperatures. This was attributed to the activation of shallow insitu defect(s) uniformly distributed in diamond. The importance of these results will be discussed in the framework of diamond detectors employment in high-temperature and high-radiation conditions.

Finally, the last example is related to the study of the so-called dynamic annealing effect, previously observed in Si and SiC irradiated with high-flux pulsed ion beams [1], used to investigate the mechanisms of defect stabilization in millisecond time scales. We report on the ongoing work to spatially map and quantify damage production in pulsed-beam irradiated diamond, using scanning ion microbeam RBS-channeling.

<sup>[1]</sup> Wallace J B, Charnvanichborikarn S, Bayu Aji L B, Myers M T, Shao L and Kucheyev S O (2015) Radiation defect dynamics in Si at room temperature studied by pulsed ion beams, Journal of Applied Physics, 118

THURSDAY, 15 SEPTEMBER

INSTRUMENTATION III

Chairs: Gyorgy Vizkelethy and Gyula Nagy

#### Invited talk III

## 1096

## Present Status of Tohoku Microbeam System

#### Shigeo Matsuyama, Misako Miwa, Sho Toyma

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We have developed two microbeam lines named MB-I and MB-II which could obtain beam spot sizes of less than 1 x 1 µm<sup>2</sup> and applied various fields. The microbeam line consists of quadrupole lenses (triplet or Doblet) and the slit-system of micro slits (MS) and divergence-defining slits (DS). These components are mounted on a heavy rigid support with vibration isolation. MS are composed of two adjustable wedge-shaped slits which are made from 5 mm diameter tungsten cylinders. The spacing is adjustable from 0 to 250 m corresponding to a longitudinal motion of 0-25 mm. The quadrupole lens is manufactured by TOKIN. The bore radius is 5 mm and dimensions of the yoke are 60 mm long × 220 mm outside diameter. The one-piece magnet poles and yoke was cut as one body from a single iron piece using a numerically controlled machine to reduce sextupole field contamination. Tolerance of the pole dimensions is better than 2 m. The coils consist of 22 turns of 2 mm thick by 22 mm wide copper plates which are soldered together after assembly. The high field gradient of 85 T/m is obtained at a current of only 40 A and the low current density in the coil effectively reduces temperature rise of the quadrupole lens assembly. The quadrupole lenses are set on stages which allow precise adjustment of translation, tilt and rotation. These components are controlled by LabVIEW based software via programable logic controller, PLC (FA-M3 Yokogawa Electric). The beam scanner is located downstream of the guadrupole lenses and controlled via compact RIO system (cRIO, National Instruments). The cRIO is composed of ADC, DAC, and input/output modules. with high-speed data processing and collection abilities. The cRIO system is capable of controlling a beam scan via a DAC. Data acquired by the X-ray, charged particle, and SE detectors are also integrated into the system. External ADCs are used for pulse signals from the Xray or charged particle detectors and is collected through an input/output module (NI9403), and recorded as a list together with an internally processed dataset of the scan position. An automatic beam-focusing system to reduce the experimental configuration time.

The system is routinely used in Tohoku University. The systems are shipped to a private company and a Laboratory.

#### **Contributed Talk VII**

## 1087

## The dual ion beam microprobe, setup performance and applications

Georgios Provatas, Iva Bogdanović Mihalić, Domagoj Cosic, Zdravko Siketić and Milko Jakšić

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The Laboratory for Ion Beam Interactions of the Ruđer Bošković Institute (RBI) hosts two tandem accelerators attached to nine beam lines, including the two microbeam end stations. Within the last years, the group has been developing the new dual microbeam (DuMi) experimental end station, aiming to offer the possibility of simultaneously focused ion beams from both accelerators. The setup uses in-house designed magnetic quadrupole triplet lens for focusing the ion beams provided by the 1.0 MV Tandetron accelerator, while the commercial electrostatic microprobe based on the "Russian" quadruplet configuration [1] is used for the high rigidity ions provided by the Tandem Van de Graaff accelerator. In addition, aiming to develop a versatile setup, the cylindrical DuMi chamber provides the possibility of several different working distances, including the short one (7 cm) with high demagnification and submicrometer spatial resolution. Also, the chamber enables the application of most of the IBA techniques, i.e., PIXE, RBS, NRA, PIGE, STIM and IBIC.

In the present contribution, the recent upgrades of the setup will be presented, while its current capabilities in terms of spatial resolution obtained for different ions and focus distances will be demonstrated. Moreover, the authors will focus on the latest applications within the group's activities (RADIATE, CERIC-ERIC, EuroFusion) carried out at the DuMi chamber. The latter include the application of IBA techniques in cultural heritage artifacts, fusion materials analyses, studies of biological samples, investigation of <sup>3</sup>He induced nuclear reactions, as well as semiconductor detectors studies by means of the IBIC technique. Lastly, the recent applications and the future perspectives of the simultaneous dual ion microbeam use will be discussed.

[1] N.C. Podaru, F.L.van de Hoef, A.Gottdang, D.J.W.Mous, Design and performance of the HVE electrostatic nuclear microprobe, Nucl. Instr. and Meth. B, 306 (2013) 25-28

## **INSTRUMENTATION III**

#### **Contributed Talk VIII**

## 1011

# The beam optics of 50MeV proton microbeam based on cyclotron accelerator

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High-energy proton have important applications in space science, cancer treatment, biological cells, and other fields. However, the design of a high-energy proton microbeam needs to take into account the limitations of the beam quality of the cyclotron capable of delivering high-energy proton and the environmental irradiation shielding generated by high-energy proton. Here we present the beam optic design of a cyclotron-based 50MeV high-energy proton microbeam with micron probe resolution, using the Russian quadruplet configuration with a low aberration coefficient to focus the ion beam with large emittance, large momentum dispersion stream from the cyclotron, in addition, the Russian quadruplet configuration can be converted to the oxford triplet configuration to achieve better imaging resolution in single ion mode. The effect of intrinsic aberrations, lens aberrations, and lens fringe field on the beam probe of the microbeam is also considered.

## **INSTRUMENTATION III**

#### **Contributed Talk IX**

## 1089

# Study of chemical effects on HR PIXE spectra of low Z elements with focused ion beams

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A decade ago, the first prototype of downsized flat crystal wavelength dispersive X-ray (WDX) spectrometer was designed and constructed for application on microscopic samples on our ion microprobe. The system was used to study high resolution Si K $\alpha$  and K $\beta$  spectra from silicon compounds, including several silicate minerals. In addition, we measured Mg and Al K $\alpha$  high-resolution spectra excited by 3, 4 and 5 MeV He ions of importance to study the influence of multiple ionization satellites on the analysis of spectra from the APXS (Alpha particle X-Ray Spectrometer) installed on Mars Exploration Rovers.

In the last few years upgraded spectrometer was designed, built and installed at the microprobe. The new system is equipped with the improved motorised sample and the diffraction crystal positioning stages. Additional ports on the chamber were added to enable integration of standard PIXE and RBS/NRA detectors. New CCD camera with better efficiency for detection of X-rays above 3 keV was installed. In combination with new diffraction crystals, capabilities for X-ray detection were extended to both low and high X-ray energy regions.

With the upgraded spectrometer we continued to study high resolution PIXE spectra of low Z elements (AI, Mg, P, S, CI, K and Ca) and their compounds with proton and helium beam excitations. In this presentation the upgraded spectrometer will be described and recent results on its use to study chemical effects on low Z elements and their compounds will be elaborated.

## **INSTRUMENTATION III**

#### **Contributed Talk X**

## 1045

## Xantho - a simple basic program for fitting X-ray spectra

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During past decades, we have developed several specific codes for evaluation of elemental concentrations, like those for metal alloys, usewear layers on flint tools, differential PIXE and combined PIXE-PIGE analysis of glass. All these codes relied on X-ray intensities, evaluated by generally accessible programs, notably AXIL. In order to have better control on the evaluated intensities we have decided to develop a new code. Its advantage aims to provide minimal typing of commands during the analysis of a series of spectra. The other advantage is using the code with new computer operating systems, which made many older programs not working.

The main decision concerned the choice of background subtraction. In practice, two approaches proved successful for removing the background of the type that is not calculated by a physical model: smoothing with a filter and top-hat transformation. We have decided for the filter smoothing, mainly because of good experience with the AXIL code. The approach we developed is based on a moving-window filter with specific weights (a variant of the Kolmogorov-Zurbenko filter), which is applied on the convex parts of the spectra. The latter are detected by a top-hat transformation, which in effect gives the second derivative of the spectrum. The X-ray peaks are assumed to be Gaussian, only below the silicon absorption edge, the peak shape was conveniently approximated by a sum of two Gaussians at a fixed ratio. K-alpha and K-beta lines can be fitted either individually or as an aggregate. Corrections to their ratio includes absorption in the added absorbers and in the target, as well the counting efficiency of the detector. For L-lines, the aggregates belonging to individual subshells are fitted independently. Individual lines at selected energies can also be defined. Non-linear fitting determines two parameters for energy calibration and two for energy resolution. A parabolic expansion of  $\chi 2$  is applied. The spectral region can be divided in separate parts, where the fitting procedure is performed in sequence.

The program was first written in Free Pascal and is run in DOS. For visual inspection of the fitting results, a separate program was written in Lazarus environment. However, the DOS version is only used for development, for on-line fitting the program can be run and commanded through a Lazarus interface. The program further allows automatic analysis of a series of spectra that are generated in mapping experiments.

## THURSDAY, 15 SEPTEMBER

## **SESSION 2**

## SPACE

Chairs: Milko Jakšić and Frans Munnik

#### Invited talk I

## 1047

## A Rad-Hard SRAM Memory Designed in 65~nm CMOS Technology

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Commercial ICs are usually not designed neither tested for applications in harsh environments, such as in space and high energy physics experiments. Applications in high radiation environments require ICs to be developed by modifying the fabrication process (RHBP: Radiation Hardening By Process), or by adopting design techniques (RHBD: Radiation Hardening By Design) and tested using radiation sources that simulate radiation fields in space or in facilities.

In this work, we present a 32 kbit SRAM Application-Specific Integrated Circuit (ASIC) specifically designed to be radiation hard using RHBD techniques and fabricated using a conventional 65 nm Complementary Metal-Oxide Semiconductor (CMOS) technology. We discuss its design and architecture, fault injection simulation technique for single event upsets (SEU), and test results for SEUs and total ionising dose effects following selective irradiations of a sensitive part of the chip (700  $\mu$ m x 700  $\mu$ m) by 5 MeV proton (LET=13.7 keV/ $\mu$ m=0.059 MeV\*cm2/mg) and 24 MeV (LET=836 keV/ $\mu$ m=3.59 MeV\*cm2/mg)) C4+ ion microbeams at the ANSTO Centre for Accelerator Science. The chip showed no SEU sensitivity for irradiation with proton microbeam of 16 pA up to 5 kGy total

accumulated dose. On the contrary, the chip showed SEU sensitivity during irradiations with the carbon ion microbeam with particle current in the 1 pA/ion -10 pA/ion range. The SEU number grows approximately linearly with increasing accumulated dose up to 5 kGy, and the SEU number is higher for the higher microbeam current. The chip's failure with many non-random errors in memory cells occurs for total accumulated doses above 500 kGy. The graph below shows the SEU number increasing with the delivered dose, during irradiation with 24 MeV carbon ions microbeam.



## SPACE

## 1084

# Single Ion Localization Microscopy for Imaging of Irradiation Effect in Microelectronics

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Cosmic rays in space environment contain high flux heavy ions and protons with energy of tens MeV up to tens GeV. Their ionizing interaction with microelectronics in spacecraft induces single event errors and malfunctions, which is one critical and major cause of in-orbit mission failures. The resolution of the present microbeam facilities for single event effect imaging using high energy heavy ion cannot meet the demand of radiation hardening technique and single event effect study with modern microelectronics with nanoscale fabrication technology. This work targets the development of a new concept of imaging technique for irradiation effect using high energy heavy ions. Based on the high energy heavy ion microbeam facility at HIRFL, single ion scintillation localization microscopy method was developed which allows imaging the single event upset and ion beam induced charge collection with submicron resolution using a slightly focused beam. Such a single ion localization microscopy brings the ion beam imaging technique towards a higher ion energy regime.

## SPACE

#### **Contributed Talk I**

## 1038

## New external ion microbeam irradiation facility of ANSTO

<u>Zeljko Pastuovic<sup>1</sup></u>, Stefania Peracchi<sup>2</sup>, Ryan Drury<sup>2</sup>, Nikolas Paneras<sup>2</sup>, David Button<sup>2</sup>, Michael Mann<sup>2</sup>, Justin Davies<sup>3</sup>, Chris Hall<sup>4</sup>, Ceri Brenner<sup>2</sup>, David Cohen<sup>2</sup>

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This paper presents a new accelerator capability for space research at the Centre for Accelerator Science of the Australia's Nuclear Science and Technology Organisation (ANSTO). The External Ion MicroBeam Irradiation Facility (EBIF) is dedicated to 1) the radiation testing of electronic components for space qualification to support an accelerated space industry in Australia and 2) the radiobiological studies on living cells for applications towards space travel and radiation therapy (reported in another paper). The EBIF is an upgrade of the existing ANTARES heavy ion microprobe beamline with the Oxford Microbeams triplet lens system (EM/Q2=120 MeV amu/e2) attached to the 10 MV FN Van de Graaff accelerator [1]. A chamber for the external beam inclosure with a safety drawer housing a micromanipulator for a target positioning was designed and built in-house, to satisfy the radiation and biological safety and regulatory requirements. It is capable of handling larger size (100 mm by 100 mm) electronic boards hosting test chips with an accuracy of 0.01 mm in 3D and a complex interrogation system otherwise unsuitable for operation in vacuum during irradiation. The microbeam is extracted from vacuum into the ambient through a 1 µm thin and 6 mm by 6 mm large SiN window for irradiations at atmospheric pressure and room temperature. A current measurement in the pA range which corresponds to dose rate measurement in Gy/s range is performed using a transition Faraday cup system in vacuum. A particle detector in STIM geometry is used for a dose rate measurement in mGy/s range for irradiation of cells in the single ion regime (1 kHz to 20 kHz). Total Ionizing Dose (TID) and Single Event Upset (SEU) studies of electronic components and chips can be performed by irradiations with protons and a selection of light and heavy ions having the energy up to 100 MeV and the LET in the 0.02 to 46 MeV\*cm2/mg (or 5.0 to 1.1\*104 keV/µm) range, suitable for the simulation of space radiation environments under laboratory conditions (Fig. 1). We present the experimental testing protocol and first results of the SEU and TID radiation tests of SRAM and logic gate ICs exposed to 9 MeV H ions and 36 MeV C ions microbeams in the external chamber with the in-house designed and built electronic readout system and control, interrogation and logging software. Lethal irradiation doses in the order of a few kGy can be delivered

(protons) minutes in or seconds (carbon), whereas irradiations with gamma rays require hours within at the ANSTO cobalt-60 gamma facility. irradiation The delivered dose is calculated by GEANT4 software, where we modelled the energy deposited in the chip placed in the external chamber.



- H - He - Li - Be - C - O - F - Si - Cl - Ti - Fe - Cu - Ge - As - Br - Ag - Te - I - Er - Au Figure 1. LET and Range of ions achievable with the ANTARES accelerator.

## SPACE

## 1075

# GEANT4 model to calculate the radiation dose absorbed by a custom target on the ANTARES EBIF beamline

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We present a GEANT4 model developed specifically for the calculation of deposited energy and radiation dose during irradiation of a custom target on the ANTARES External Beam Irradiation Facility (EBIF) beamline of CAS. For a high accuracy dose calculation we have taken into acount 1) a realistic propagation of the ion beam through all impinging materials defined by composition and dimensions, 2) a beam cross-section, and 3) all types of ion and secondary particle interactions with matter including tracing of all impacted particles and radiation. Each interaction of ion projectile with target is simulated by a single track with an accuracy of 10 nm. The energy deposited by both primary ion projectiles and secondaries particles is stored along the whole geometry. The model is highly flexible to easily adapt to any experimental condition: for example, by changing the beam crosssection and the target area, we can select the best suitable flux and beam current to achieve a specific dose rate at a particular depth in the target of interest. LET and dose rate profile in all materials through which the ion beam passes is calculated from deposited energy along the track. We demonstrate the model in a real case of 9 MeV proton micro-beam extracted from vacuum through a 1 µm thin Si3N4 window for irradiation of a SRAM chip positioned 10 mm behind the window in air. The chip consists of a 270 µm thick thinned down plastic encapsulation, a 10 µm thick SiO2 and 300 µm Silicon active layer. The calculated LET and dose rate profile allowed us to select a beam current of 5 pA to raster scan on a 2.3x2.3 mm2 chip area in order to deliver the total dose of 6.7 kGy (with a dose rate of 3.8 Gy/s) at 10 µm depth in the SiO2, and a total dose of 14.0 kGy (with a dose rate of 8.0 Gy/s) at 300 µm depth in the Silicon active layer.



S. Agostinelli, et al "Geant4 - a simulation toolkit," Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment, vol. 506, no. 3, pp. 250-303, 2003

## **Radiation Biology**

## 1016

# Characterization of ion microbeams (H<sup>+</sup>, He<sup>2+</sup>, C<sup>3+</sup>, O<sup>3+</sup>) and a new detection system on the MIRCOM facility

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lons microbeam are important in several fields, in particular in the improvement, identification and prevention of side effects resulting from the use of ionizing radiation, especially for therapeutic purposes in radiotherapy and more particularly in hadrontherapy. To study this field, the Institute for Radiological protection and Nuclear Safety (IRSN), in Cadarache (France), is equipped with the MIRCOM facility, whose main element is an ion microbeam dedicated to targeted irradiation of living biological samples with a given number of ions. The facility was commissioned at the end of 2018 [1]. It is based on a 2 MV Tandetron<sup>TM</sup> electrostatic accelerator manufactured by HVEE [2]. Before reaching the biological samples, the ion beam is focused in the microbeam line, reaching a diameter of less than 1  $\mu$ m in vacuum, and then extracted in air by a silicon nitride extraction window (Si<sub>3</sub>N<sub>4</sub>). The first effects of the irradiations can be visualized, in real time, using an epi-fluorescence microscope placed in front of the microbeam extraction. This microbeam can deliver four types of ions: Proton (H<sup>+</sup>), Helium (He<sup>2+</sup>), Carbon (C<sup>3+</sup>) and Oxygen (O<sup>3+</sup>).

A first study was performed to determine the beam spot size for all the ions available on MIRCOM on CR39 track detector and on cells. With CR39, the beam spot size can be evaluated by measuring the impact diameter of the ions after a chemical etching. With cells, the use of time-lapse imaging of fluorescent protein recruitment is a fast and reliable method to directly visualize the early consequences of the microbeam irradiation [3]. A beam spot size of 2.2 ± 0.3 µm for H<sup>+</sup>, 2.6 ± 0.2 µm for He<sup>2+</sup>, 3.4 ± 0.6 µm for C<sup>3+</sup> and 4.6 ± 0.7 µm for O<sup>3+</sup> was measured.

A second study focused on the ability to irradiate a target in a biological sample with single ions. To achieve this, a very efficient detection system needs to be used, to count the ions with a 100% accuracy. Given the energies of the ions used on MIRCOM, that prevent them to pass trough the biological sample to be counted after, the detection needs to be performed before the sample. When the ions pass through the extraction window, secondary electron are emitted. These electrons can be collected and converted to a counting signal by a Channeltron [3]. To optimize the detection efficiency, the production yield of secondary electrons can be improved by using deposits of caesium iodine and gold, directly on the vacuum side of the extraction window.

We performed simultaneous measurements with the Channeltron and a reference detector, with different CsI deposit thicknesses (25, 50, 100 and 200  $\mu$ g/cm<sup>2</sup>). We found a detection efficiency of 96,5% and 100,5%, respectively with 100  $\mu$ g/cm<sup>2</sup> and 200  $\mu$ g/cm<sup>2</sup> thick CsI deposits for He<sup>2+</sup>, 98,6% for C<sup>3+</sup> without CsI and 99,9% for O<sup>3+</sup> without CsI. Further measurements must be performed for He<sup>2+</sup> with intermediate CsI deposit thicknesses to have an optimal detection efficiency.

- [2] V. Gressier et al. New IRSN facilities for neutron production, NIM A, 505: 370-373 (2003)
- [3] S. Bourret et al. Fluorescence time-lapse imaging of single cells targeted with a focused scanning charged-particle microbeam, *NIM B* B 325 (2014)
- [4] M. Cholewa et al. Preparatory experiments for a single ion hit facility at GSI. NIM B. 210: 296-301 (2003)

<sup>[1]</sup> F. Vianna et al. Characterization of MIRCOM, IRSN's new ion microbeam dedicated to targeted irradiation of living biological samples, *NIM B*, 515: (2022)

## **Radiation Biology**

## 1039

# Cell radiation sensitivity – a step closer to understanding different effects of radiation on humans

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Radiation exposure is a major limiting factor for long duration manned space flights. Radiation protection standards assume that individuals are equally resistant to ionizing radiation. However, for over a century, there is evidence that humans do not respond equally to radiation. Studies of secondary effects post-radiotherapy have shown a great variability among individuals [1]. Also, large discrepancies among astronauts after the same flight were observed [2]. We have shown that the delay in the nucleoshuttling of the ATM protein may cause a lack of double strand break (DSB) recognition, incomplete DSB repair and radiosensitivity in fibroblast cell lines derived from patients suffering from genetic disease or post-radiotherapy [3-6]. Interestingly, the model of the ATM nucleoshuttling was shown to be relevant not only for low-dose and repeated exposures [7], but also for high-LET particles [8], which renders this model compatible with space radiation exposure scenarios. Lastly, this model could lead to a novel approach for radiation protection, consisting of radiosensitivity studies of fibroblast cell cultures in the 0.1 - 2.0 Gy radiation dose range using a focused raster-scanned external ion microbeam of a new dedicated facility for irradiation of thin biological samples in ambient at ANSTO Centre for Accelerator Science.

- [1] Berthel E et al., What Does the History of Research on the Repair of DNA Double-Strand Breaks Tell Us? A Comprehensive Review of Human Radiosensitivity. Int J Mol Sci. 2019;20(21):5339. Published 2019 Oct 26. doi:10.3390/ijms20215339
- [2] Testard I et al., International journal of radiation biology 70 (4):403-411
- [3] Granzotto A et al., International journal of radiation oncology, biology, physics 94 (3):450-460. doi:10.1016/j.ijrobp.2015.11.013
- [4] Belkacemi Y et al., International journal of radiation oncology, biology, physics 96 (3):629-636. doi:10.1016/j.ijrobp.2016.05.027
- [5] Pereira S, International journal of radiation oncology, biology, physics 100:353-360
- [6] Vogin G et al., International journal of radiation oncology, biology, physics 101 (3):690-693. doi:10.1016/j.ijrobp.2018.03.047
- [7] Devic C et al., International Journal of Low Radiation in press
- [8] Maalouf M et al., International journal of radiation oncology, biology, physics. doi:10.1016/j.ijrobp.2018.10.011
- [9] Ferlazzo ML et al. ,Proc Natl Acad Sci U S A. 2017 Aug 15;114(33):E6733.

## **Radiation Biology**

## 1095

## Recent upgrade of the radiobiology beamline at the Center for Ion Beam Applications for single proton irradiation at live cells

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The radiobiology beamline at the Center for Ion Beam Applications is dedicated to the study of live cells reacting to proton irradiation at a controlled dose. Previously the dose of protons was measured from an average count rate using a Si-surface barrier detector before irradiation. However, the accuracy of this method is subject to beam fluctuation, particularly at a low dose. To achieve precise control of dose at a single proton level, we upgrade the beamline using several techniques. First, we have developed both diamond-based and scintillator-based transmission detectors for counting protons during irradiation. The detectors are placed in the ion path before the cells. For a 2 MeV proton, the energy loss of the detectors is only ~330 KeV and ~160 KeV for the diamond and the scintillator, respectively, which is relatively lower compared with ion energy deposited in cells. In addition, the beam blanking system is upgraded with a fast high-voltage MOSFET switch to achieve a high-speed cut-off of the beam when a certain dose has been reached. The upgraded system allows on and off a 2 MeV proton beam at 16 nanoseconds. Last, we have also upgraded the optical imaging system of the beamline with an EMCCD camera, which enables us to capture fluorescence induced by single protons. These upgrades are of great potential to study the radiobiology of cells under single ion irradiation.

## 1002

# The potentialities of ultrasound as an alternative to chemical etching for proton beam writing micropatterning

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We explore the potentialities of using ultrasound waves as a post-irradiation treatment after proton beam writing (PBW) patterning of PTFE. To that end, 2-mm-thick foils of PTFE were irradiated with 2.2 MeV protons with an average current of 50 pA. Straight line structures were patterned on the polymer with a 3.5 x 3.5µm2 focused proton beam. Post-irradiation polymers were placed in a heat bath at 60 °C and immersed either in distilled water or in a 6 M solution of NaOH under the action of 40 kHz ultrasound waves for developing the patterned structures. The results indicate that distilled water submitted to ultrasound waves is very efficient for removing rough structures created by the proton irradiation and thus providing a good aspect ratio to the PBW microstructures. On the other hand, the use of 6 M NaOH instead of water did not improve the quality of the structures patterned with the proton beam. The results are discussed in terms of the parameters that characterize the interaction of ultrasound waves with the liquid media and the polymer.

- [1] D. de Vila Bauer, R.Debastiani, C. Telles de Souza, L. Amaral, J. FerrazDias, J. Appl. Polym. Sci.2022, e52407.https://doi.org/10.1002/app.52407
- [2] C. T. de Souza, E. M. Stori, L. A. Boufleur, R. M. Papaléo, J. F.Dias, Appl. Phys. A: Mater. Sci. Process. 2016, 122, 122.
- [3] E. M. Stori, C. T. De Souza, J. F. Dias, J. Appl. Polym. Sci.2016,133,1

## 1006

# Proton beam writing of X-ray diffraction gratings using a quadruplet of magnetic quadrupole lenses with individual power supplies

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To fabricate X-ray diffraction gratings using proton beam writing, it is most efficient to focus the proton beam into a thin line. For such focusing, we used the configuration of the probe-forming system in the form of a separated triplet of magnetic quadrupole lenses. In this case, refocusing was carried out in order to maintain a demagnification close to unity in one of the directions. In the experiment, the disadvantage of such a system appeared due to large positioning aberrations, which did not allow achieving the calculated parameters for the line thickness. For this reason, another variant of the probe-forming system was found based on a quadruplet of magnetic quadrupoles with four independent power supplies, the first two lenses having very low excitation, which reduces positioning aberrations. Such a system in the stigmatic focusing mode has demagnifications  $D_x \times D_y = (-71) \times 1.3$  and low chromatic and spherical aberrations.

## 1021

# Fabrication of periodic microstructures on diamon surface with focused swift heavy ions

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The development of novel, three-dimensional micro-fabrication techniques on diamond would open up new possibilities towards the realisation of diamond-based devices for e.g. x-ray optics applications. However, to date the available technology is unable to overcome the difficulties associated to the high mechanical strength and chemical stability.

In this work, topographic periodic microstructures have been produced on the diamond surface by the effect of implantation of two highly-focused ion beams, carbon and silicon, with energies of several MeV. It is well-known that MeV heavy ion implantation with the key experimental parameters, produces a serious structural damage inside monocrystalline diamond. Once implantation has been carried out, the internal density decreases significantly keeping its surface almost intact. Consequently, the buried damaged diamond volume increases and the increase is passed to the sample surface giving rise to a topography, which depends on the cumulative damage bellow. Consecutive lines with increasing fluences have been implanted in order to manufacture a saw-tooth profile. The required fluences have been calculated using the nuclear stopping power to produce a determined maximum height and blaze angle of the structure. The patterns have been characterized by optical microscopy and atomic force microscopy and the manufactured structures for each ion have been compared. Our results certify this manufacture method is highly promising for the reliable fabrication of optical and biomedical diamond-based devices.

## 1042

## Design of a ccollimetor-less two-stage acceleration lens for a single-ionimplantation system

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This report shows the design of a focusing lens for a single-ion implantation system (SII). The SII has been developed for the implantation of single-nitrogen-ion with the accuracy of nanometers in a diamond sample to create a nitrogen vacancy color-center (NVC) that functions as a quantum bit. The quantum entanglement using the array of NVCs is expected to lead us to develop a quantum computer. In the SII, single-nitrogen-ion needs to be implanted within 20 nm accuracy in a diamond sample with 100% probability, namely deterministic implantation. We propose a single-ion-implantation system combining Paul trap-type laser cooling device (PTLC) [1] and a focusing lens as the SII. PTLC individually generates single-nitrogen-ions with very low emittance. On the other hand, the focusing lens requires the high demagnification to meet the implantation accuracy of 20 nm. In addition, passing all ions through the lens is also required for the deterministic implantation as well as the shortening of the experimental time of the evaluation of the accuracy.

In this study, a collimator-less electrostatic focusing lens with high demagnification to pass all ions through the lens was designed for the SII. In general, a collimator is installed in front of a focusing lens to restrict a divergence angle of an ion beam injected into the lens. However, the collimator used in the SII to reduce spherical and chromatic aberrations, decimates some ions. Since the decimation leads to non-deterministic implantation, the collimator cannot be used in the SII. On the other hand, a two-stage acceleration lens with high demagnification, which is a kind of electrostatic focusing lens, was developed at QST to form ion nanobeam [2]. The lens is relatively bright as compared with other typical lenses because a divergence angle of outgoing beam can be controlled by the acceleration. In this study, a collimator-less two-stage acceleration lens to be installed for SII was designed on the basis of numerical calculations.

This study is supported by JSPS KAKENHI Grant Numbers JP20H00145, JP20H02673, JP19K12635, and Moonshot R&D Grant Number JPMJMS2062.

<sup>[1]</sup> Kenji Izawa, Kiyokazu Ito, Hiroyuki Higaki, and Hiromi Okamoto, Controlled Extraction of Ultracold Ions from a Linear Paul Trap for Nanobeam Production (2010), 10.1143/JPSJ.79.124502

<sup>[2]</sup> Y. Ishii and T. Kojima (2018) Reduction of the divergence angle of an incident beam to enhance the demagnification factor of a two-stage acceleration lens in a gas ion nanobeam system of several tens of keV, <u>https://doi.org/10.1016/j.nimb.2018.01.013</u>

## 1053

# Ion beam induced current and ion beam induced charge analysis in GaN core-shell p-n junction microwires

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Gallium nitride (GaN) nano- and microwires have gained increasing interest due to their unique geometry and flexibility, and their superior crystalline quality in comparison to their thin-film counterparts. GaN is also known for its high radiation hardness, owed to the large displacement energy of the atoms in its crystal lattice and efficient dynamic annealing properties, making it an interesting material for electronic space applications.

In this work, single GaN core-shell p-n junction microwires with a length of 25±5 µm and a diameter of 1-2 µm have been processed into radiation detectors and characterized using nuclear microprobe techniques. By taking advantage of the small lateral dimension of the focused ion beam and the ability to control the area of irradiation with precision, we can measure the ion beam induced current signal when irradiating the different regions of the detector. Typically using focused 2 MeV protons and beam currents in the range of 50 pA to 100 pA the CTN experimental setup allows to obtain PIXE and RBS maps and at the same time perform electrical measurements. Initial PIXE maps are used to precisely define the position of the small dimension sensors and then select the specific region we want to irradiate. This means that we can measure the electrical signals induced when irradiating the p-n junction and the n-GaN extremity of the wire independently and compare their contribution to the response of the detector. Besides this, it is also possible to analyze the evolution of the detector's reverse bias leakage current and ion beam induced current in function of the irradiation fluence to better define its radiation hardness. Results show that GaN radiation detectors withstand high beam fluences that would strongly degrade any Si-based device.

Further microwire electrical characterization can be accomplished when using fA beam currents as the ones usually applied in ion beam induced charge (IBIC) experiments. However, using proton beams on these thin GaN detectors would require a very high sensitivity IBIC system. Profiting from the capabilities of the tandem accelerator facility at RBI that also includes a nuclear microprobe beam line, the signal to noise ratio of the locally developed high sensitivity IBIC system can be further improved by irradiating the sensors using different ions (e.g. Cu, Si, C) and energies, then allowing to measure IBIC maps at different applied reverse bias and study the detection efficiency and charge collection efficiency of the sensors.

## 1061

## Depth distribution of Fe in Fe-MOF-74 thin films

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Fe-MOF thin films of metal-organic frameworks were synthesized by cathodic electrodeposition using new ligands with the aim of producing new electrocatalysts for energy-related reactions.

These films contain C, O, H and Fe as main constituents, and they were deposited on FTO-coated glass. Several Fe-MOF films, grown with different experimental conditions, were characterized using a 2 MeV proton beam, acquiring the PIXE and the RBS spectra simultaneously.

The 2D compositional maps show that the films partially cover the FTO surface, being the thickness and the Fe distribution dependent on the growth conditions.

In the PIXE spectra, the concentration of Fe is relatively straightforward to obtain using GUPIXWin software, however, there is an overlap in the low energy region regarding the Sn-L lines (from the FTO layer) and the Ca-K and K-K lines (from the glass substrate). Other challenges encountered during the analysis of the RBS spectra will be discussed.

## 1071

# Ion Microprobe analysis of wear processes in ta-C coatings and contact areas on the counter bodies.

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Solid lubricants, like ta-C (hydrogen-free tetrahedral amorphous carbon) coatings, are an active area of research to replace liquid lubricants. This substitution is important because of the negative environmental impact and high material consumption of liquid lubricants. The influence of soft metal counter bodies on the unlubricated friction behaviour of hydrogen-free carbon coatings has mostly been studied for doped a-C coatings so far. All these studies show that low friction and wear can only be achieved if a tribolayer is formed to protect the contact. Even with a formed tribolayer, its composition is crucial for the frictional behaviour, making the investigation of this composition essential. A study currently underway aims to identify friction-induced surface changes like material loss of the coating, material transfer between the counter body and the coating, or the formation of a tribolayer.

The current work presents Ion Beam Analysis methods to deliver laterally and depth-resolved element analysis of the wear track on the coating and the contact area of the counter bodies (CB). In a first test, a ta-C coating has been subjected to pin-on-disk tests with various metallic and ceramic CBs. He and H ion microbeams have been used to scan over the tracks and the contact areas of the CBs. Both RBS and PIXE have been used and first results are presented. It is shown that, in this case, RBS yields the more useful information. Both, a 2 MeV He ion beam and a 3 MeV H ion beam provide valuable results. The advantages of each type of ion beam depend on the sample and the information needed. As shown in this work, RBS with a 2 MeV He ion beam is useful to determine the transfer layer of a (soft) metal CB to the ta-C coating, whereas 3 MeV H RBS can be used to determine the presence of C and O on the CB because of the increased non-Rutherford cross-sections for these elements.

## 1076

# Calculation Study for the selective ion extraction from an ion source with Paul-trap-type laser cooling device

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The selective extraction of a single molecule nitrogen ion (N2+) with very low emittance from a linear-Paul-trap ion source (LPT-IS) for a single-ion implantation system (SII) has been studied by three-dimensional multiparticle simulations. The SII that is the combination of the LPT-IS and an electrostatic focusing lens1 in the QST Takasaki has been developed to create an array of nitrogen vacancy color centers (NVCs) at intervals of 20 nm or less in a diamond specimen. The NVC array has been proposed to realize the quantum entanglement used in solid-state quantum information devices. In the LPT-IS, several nitrogenions are cooled to a temperature close to absolute zero by the interraction of calcium ions (Ca+s) cooled by Doppler-laser-cooling technique2, namely "sympathetic cooling", so that an arrangement of N2+ and Ca+ is formed by sympathetic cooling. The distribution of N2+ extracted from LPT-IS is expected to have very low emittance. However, N2+s are extracted together with Ca+s from the LPT-IS. Therefore, only the extraction of a single N2+ is required without deteriorating the emittance at the SII.

In this study, the conditions to extract only a single N2+ from the arrangement of N2+ and Ca+ has been investigated by the simulation of multi-particle motion in the three-dimensional electric field. The result of the simulations shows that the single N2+ with low emittance can be extracted by increasing the voltage of the extraction side electrode at the optimal timing and magnitude while the arrangement of N2+ and Ca+ was passing through the electrode of the LPT-IS. The obtained emittance of the single N2+ is used in the design of electrostatic acceleration lenses.

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## 1017

## Status report on the Atomki nanoprobe setup

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Since the initial setup of our nanoprobe that we have reported in Guildford, we have extended the Tandetron Laboratory with three new ion sources (two multicusps for proton and helium beams, and a cesium sputter source for heavy ions), a 90-degree analyzing magnet and associated high-energy extension. This way the final layout was achieved that was originally planned.

The object distance of the nanoprobe is as long as 12 m, and the high brightness provided by the multicusp source allows sufficient beam current even with a very low beam divergence that is caused by the long object distance vs. limited collimator slits. The fact that the switching magnet is located after the object slits makes sure that the ions that are scattered on the slit edges thus lose energy are bent away from the target chamber, and do not cause beam halo.

In the initial setup we have reached 200 nm spot size for low current mode, and 600 nm for high curent mode. Based on preliminary ion optics calculations, we expect sub-100 nm spot size for the low current mode and sub-300 nm for the high current mode with the new laboratory layout.

However, during the re-installation process of the nanoprobe, we encountered several problems that lead to the degradation of the beam size. These problems involved possible mechanical vibrations from the rearranged mechanical workshop. The longer object distance made the beam path dependence on ion energy much larger thus we have decided to cover as much as possible of the beamline with mu-metal (Co-Netic, Magnetic Shielding Corp.). We measured that the vertical component of the Earth's magnetic field, which is about 435 mG (43.5 nT) in Debrecen, can be reduced to well below 10 mG (1 nT). Our calculations show that this will reduce the relocation of the collimator slits from about 2 mm in the horizontal direction to about only 100 µm when changing beam energy or using heavy ions, but it also helps to reduce the effects of possible stray magnetic fields. We also found instabilites in some of the resolution, which we could localize using the double-focusing property of the analyzing magnet.

In this presentation we show how we diagnosed the encountered problems and we also show the solutions of how we eliminated or reduced their effects.

I. Rajta, G.U.L. Nagy, I. Vajda, S.Z. Szilasi, G.W. Grime, F. Watt: First resolution test results of the Atomki nuclear nanoprobe. Nucl. Instr. Meth. B 449 (2019) 94-98. doi: 10.1016/j.nimb.2019.03.056

## 1029

## Stopping power measurement for protons with MeV energies in diamond

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In recent years, interest in high-purity diamond crystals has been growing due to their application in various fields ranging from high-energy physics (particle detectors) to radiation therapy (dosimeters) and quantum computing (NV centres). Precise control of the energy deposited in the material by MeV proton beams, resulting from accurate knowledge of the energy loss per unit path (stopping power), is a key parameter in all the aforementioned applications of diamond. The characterization of stopping power can be done by measuring the energy loss  $\Delta E$  of a monoenergetic beam passing through a thin target and knowing the thickness of the target  $\Delta x$ . With this information, it is possible to estimate the stopping power of the proton beam when the ionization profile is approximately flat along the trajectory of the incident ion:  $S(E) \approx \Delta E/\Delta x$ . To measure the stopping power, a single-crystal CVD diamond was thinned to a thickness of a few micrometres by Ar/O2 plasma etching [1] and prepared as a self-supporting membrane, which was then exposed to a proton beam with energies between 1.6 and 6 MeV in the microbeam vacuum chamber of the RBI accelerator facility. The thickness of the membrane was determined by two different methods: indirectly by measuring the absorption of X-rays in the membrane produced by particle induced Xray emission (PIXE), and directly from images of the membrane edge taken with a scanning electron microscope (SEM). Using these two methods, the thickness was estimated to be (3.5±0.2) µm. The energy loss was measured using scanning transmission ion microscopy (STIM) and the obtained results were compared with the values obtained from Monte Carlo simulations in the SRIM package [2] and with the experimental results of Fearick & Sellschop [3].

[1] Pomorski M, Caylar B, Bergonzo, P (2013) Super-thin single crystal diamond membrane radiation detectors. Appl Phys Lett 103: 112106. doi: <u>https://doi.org/10.1063/1.4821035</u>

[2] Ziegler JE, Ziegler MD, Biersack JP (2010) SRIM – The stopping and range of ions in matter. Nucl Instruments Methods Phys Res Sect B Beam Interact with Matter Atoms 268: 1818-1823. doi: https://doi.org/10.1016/j.nimb.2010.02.091

[3] Fearick RW, Sellschop JPF (1980) Energy loss of light ions in diamond. Nucl Instruments Methods 168: 51-55. doi: https://doi.org/10.1016/0029-554X(80)91230-6

## 1052

# Arduino controlled sample positioning stage and automatic data acquisition using OMDAQ3

Manuel Fortunato<sup>1</sup>, Norberto Catarino<sup>2</sup>, Luis Alves<sup>1,3</sup>, Victoria Corregidor<sup>1,3</sup>, Rui Coelho da Silva<sup>2,3</sup>

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In this work it is described a simple, effective and low-cost system for actuating a stepper-motor driven x-y stage based on an Arduino Nano board together with a stepper-motor power driver module (Geckodrive G540) with positioning control and automatic data acquisition under OMDAQ3 environment. The Arduino board is connected to a computer through an USB port making the device of universal use for any type of computer without the need of added internal cards. For robustness and ease of connection to the G540 a 3D printed compact box was produced to accommodate both the Arduino board and DB25 terminal. Most importantly the driving software operates under the OMDAQ3 environment, taking advantage of built-in capabilities but requiring the development (or alteration of available OMDAQ3 source codes) and compilation of specific DLL files. With code written in C++ the DLL files were created using easily accessible Embarcadero's C++ Builder Community Edition.

Apart from being a low cost system and having the perspective of a long-time compatibility with computer devices (USB specifications are expected to hold for some years) the most interesting characteristic is its integration with the OMDAQ software in charge of data acquisition in a large number of Nuclear Microprobes.

The mentioned system was successfully used in our external beam setup (1 x 1 mm2 exit nozzle window dimensions) for the irradiation of easel painting coupons containing lead white pigments. The experiment and further analysis required sample irradiated areas of 8x8 mm2 with accurate accumulated beam charge per unit area in order to determine exposure thresholds for minimizing or inhibiting beam irradiation alterations.

## 1078

# Development of High Voltage Stabilization System for Nanobeam Analisys

Misako Miwa, Shigeo Matsuyama, Sho Toyama, Yuta Sato, Yohei Kikuchi

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Tohoku University has constructed a microbeam line system using a doublet system and achieved a current of 300 pA with an object size of 40x10um2 which is equivalent to a beam size of 1umx1um[1]. For further high precision ion beam analysis, we are developing a nano ion beam system to converge the beam spot size to a nanometer. One of the technical challenges for nano ion beams is to reduce chromatic aberration and improve beam brightness. In the current microbeam line system at Tohoku University, an energy analysis system consisting of a slit and an electromagnet is placed upstream of the micro-slit that determines the object size and defines the energy width of the ion beam. Furthermore, a slit of the energy analysis system is used for slit feedback control[3]. However, in this system, beam loss occurs as the beam passes through the energy analysis system, causing a decrease in beam brightness. So a voltage control system that maintains the ion beam energy resolution in the 10-5 range without a slit feedback system was constructed as follows. First, a Capacitor Pick Off (CPO) plate was attached to the left and right sides of the terminal deck as well as to the center of the terminal deck, and a frequency analysis of voltage fluctuation was performed. The results showed that there were strong fluctuations around 2Hz and 5Hz, but this fluctuation was found to be due to the vibration of the generator on the terminal deck. To eliminate this effect, the voltage feedback system was changed to a feedback system using a High Voltage Divider (HVD), which is less sensitive to the vibration of the terminal deck. As a result, the ripple component of this frequency was greatly reduced. However, the HVD is less sensitive to the fluctuation component around 1 Hz, which is caused by the degradation of the vacuum tube that makes up the oscillation circuit part of the dynamitron accelerator, and the HVD is unable to suppress the fluctuation. So by adding the gain-adjusted CPO signal to the feedback signal, we were able to suppress fluctuations in this frequency range. Furthermore, by adding the CPO signal via the I/V conversion circuit which is equivalent to the differential signal of the CPO, feedback could be applied to fast fluctuations. As described above,  $\Delta E/E=6x10-5$  was achieved by voltage control using the HVD and high-sensitivity CPO signals, and the effect of chromatic aberration of the ion beam on the beam size was approximately 80 nm. The reduction of brightness in passing through the energy analysis system consisting of an analyzing magnet and a slit, which was used to guarantee the energy width, has been improved. It contributes greatly to the improvement of the brightness of the nano ion beam system and the microbeam.

## 1080

## Study of beam induced carbon deposition using a dual microbeam setup

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The deposition of carbon containing molecules onto samples is an effect which has long been known to occur in ion beam applications as well as in scanning electron microscopy. More recently it has become an issue with the use of ion irradiation to emulate neutron radiation damage in nuclear materials. A significant uptake of carbon in the irradiated region can change the material properties studied, and measures must be taken to reduce this phenomenon. The effect is also present to a lesser extent in other ion beam analysis (IBA) applications, where it can cause errors in the quantification of carbon and oxygen. In this work we examine the carbon deposition using the dual microbeam setup recently commissioned at RBI. This setup enables on-line monitoring of the deposition process. One microbeam is used to irradiate a small region of the sample with large current densities of heavy ions typical for the nuclear material irradiation experiments. The other microbeam is used to measure the carbon content using one of the available IBA techniques in the scattering chamber. Since this analysis is done simultaneously at both irradiated and unirradiated regions, errors induced by the measurement process are minimized.

## 1088

## Total-IBA data analysis with DT2 and NDF, a new interface

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In this work, the authors reanalyse data already published [1] that were obtained using the WinNDF code [2], considering the "Total-IBA" concept. The new data analysis is done using the DT2 code for PIXE spectra fitting and NDF code for RBS spectra fitting through the recent developed software interface, "Analytical Spectrometry Software Environment for Total-IBA" (AdF ASSET). The example chosen for this work consider different situations that can be found in the IBA data analysis, for example the use of different types of beams (particles and energy) to analyze the same samples. this case the active layer used for perovskites in solar cells. The new software offers versatility, a user friendly interface and, more important, it integrates independent software platforms to analyse dedicated spectra and considers additional effects such as the secondary X-ray fluorescence. As a result, the time-consuming for analysis is reduced providing similar results that previously obtained.



Fig.1: Visualising three PIXE spectra at the same time using the AdF\_ASSET interface.

- [1] M.A. Barreiros, L.C. Alves, M.J. Brites, V. Corregidor, (2017) Depth profile by Total IBA in perovskite active layers for solar cells, Nucl Instruments Methods Phys Res Sect B Beam Interact with Mater Atom, B, 404: 211-218 doi: 10.1016/j.nimb.2017.01.019
- [2] N.P. Barradas, C. Jeynes, R.P. Webb (1997) Simulated annealing analysis of Rutherford backscattering dataApplied Physics Letters, 71: 291-293. doi: 10.1063/1.119524

## 1093

## Nuclear microprobe application in electrochemistry

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The interface between the electrolyte solution and the solid widely exist in the natural environment, biological systems and industrial products. The solid-liquid interface between the 1 mol/L BaCl<sub>2</sub> solution and the Al electrode is analyzed in vacuum by micro-PIXE and RBS. The results show that an electric double layer structure of Ba<sup>2+</sup> ions is formed at the interface. The concentration of Ba<sup>2+</sup> ions decreases exponentially with depth. This conclusion verifies the theoretical model of the GCS electric double layer structure from the aspect of the distribution of ion. In addition, ion beam analysis can also analyze the changes of ions near the electrode with time. During the formation of the double layer, the compact layer is relatively stable, and ion attraction mainly occurs in the diffusion layer and deeper region. After applying different bias to the Al electrode, the depth distribution of the ions in electrolytic cell is also investigated. Such information is helpful for electrochemistry.

## 1097

## **Development of focal Guide System for Microbeam formation**

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We have developed an automatic beam focusing system to reduce to reduce the experimental configuration time. The system involves a two-stage process of contrast and phase-difference detection that includes an automated minimization algorithm for astigmatism. The contrast method focused a microprobe to approximately 1  $\mu$ m<sup>2</sup> within 30 minutes without the need for manual control when rotational misalignment was not present, if the initial beam size is ca. 10 x 10  $\mu$ m<sup>2</sup>. It is not difficult to reduce the beam size to 10 x 10  $\mu$ m<sup>2</sup> for the experienced user, but not easy for novice users or an untuned system. In this study, we developed focal guide system based on the thin lens approximation and the relation between focal length and magnetic field. Typical results for four quadrupole lens system are shown. The curves indicate relationships of the magnetic field strengths when one of the X- or Y axes is in focus. The focal point is the intersection of the focal lines. In this system four focal points are seen. The relation between magnetic field strength and focal depth is also shown. The present calculation is consistent with those obtained using the Transport code. The system is combined with the automatic beam focusing system.



## FRIDAY, 16 SEPTEMBER

## **SESSION 1**

MICROMODIFICATION

Chairs: Maria Dolores Ynsa and Luís Alves

#### Invited talk

## 1113

# 3D Lithography with fast protons: Proton beam writing a historic overview and outlook

#### Jeroen A. van Kan, Rudy Pang

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As protons are slowed down in materials they mainly interact with the substrate electrons, much like the incoming electrons in electron beam lithography (EBL). The big difference though is that as a proton is about 1800 times heavier than an electron, it will transfer typically a small amount of energy (~ 10s eV) to each electron it interacts with. This low energy transfer has a the direct consequence that a proton beam has a larger and straighter penetration in materials with more even energy deposition along its path compared to electron beams. Therefore using proton beams with energies ranging from keVs up to MeVs can provide unique ways to perform resist lithography and materials modification.

To exploit the unique interactions of fast protons with materials, researchers realized in the 1970s that masked exposure of resist materials can produce unique structures with high throughput and high resolution. Until the 1990s the technology to focus proton beams to submicron spot sizes was not very well developed. The introduction of improved focusing systems for light ion beams as well as the development of accelerator technology, spurred the development of proton beam writing (PBW) i.e. using focused proton beams in a direct write fashion for lithography. This led to the production of three-dimensional, high aspect ratio nanostructures with vertical, smooth sidewalls and low line-edge roughness. Typical applications can be found in the area of micro and nanofluidics, nanoimprint lithography, tissue engineering and optics.

The main limitation of wider implementation of focused proton beams in areas like PBW is the poor brightness of proton sources compared to electron beam sources. The introduction of a Nano Aperture Ion Sources (NAIS) is expected to support sub 10 nm proton beams with high currents, creating unique opportunities for PBW at significantly smaller footprint.

## MICROMODIFICATION

#### Contributed talk I

### 1027

# Ionization and displacement damage effects in high-voltage vertical GaN diodes

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Gallium-nitride (GaN) is the leading material to replace silicon and silicon-carbide in power electronics applications. Its large bandgap and large displacement threshold energy promises inherent radiation hardness against both ionization and displacement damage. In this talk, we examine the effect of displacement damage on the voltage required for impact ionization and the influence of ionizing radiation on the effective doping in the drift region.

We have investigated the effects of radiation on high-voltage (~1.8 kV) vertical GaN diodes manufactured at Sandia National Laboratories (SNL) using a nuclear microprobe and electron beam irradiation. Devices were irradiated with 1 MeV proton beam to create localized displacement damage. Ion Beam Induced Charge (IBIC) measurement was performed during and after the irradiations. We found that the onset of impact ionization was independent of the amount of damage, in this case ~ 500 V. The charge collection efficiency (CCE) below the impact ionization limit decreased with increasing damage as expected due to carrier capture and recombination at defects. Above the impact ionization limit the CCE increased with increasing displacement damage, which was attributed to trap assisted impact ionization enhancement. A simple model of IBIC was used to estimate the carrier capture rates by fitting the CCE dependence on the irradiation fluence below the impact ionization limit to the model.

Carbon is unintentionally incorporated in GaN diodes grown by metal-organic vapor epitaxy, and C increases the breakdown voltage by compensating n-type dopants. In most cases the concentration of C is not known, only the net doping. Theory predicts that during ionizing irradiation the negatively charged carbon captures holes, which leads to increased space charge and decreased breakdown voltage. We used a 70 keV electron beam to study this effect and to avoid complications due to displacement damage. Using capacitance vs. time measurements immediately after the irradiation at various temperatures, we determined the Si and C concentrations and the rate coefficient for hole capture by negatively charged carbon and the activation energy for hole emission by neutral carbon.

Sandia National Laboratories is a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525. This paper describes objective technical results and analysis. Any subjective views or opinions that might be expressed in the paper do not necessarily represent the views of the DOE or the U.S. government.

## MICROMODIFICATION

Contributed talk II

## 1034

## Application of focused hydrogen ion beam (FHIB) on MIcroelectroncs Fablication

#### Hidetaka Hayashi

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lonizing hydrogen molecule we can get proton and molecular type ions composed by plural protons such as H2+ (two protons) and H3+ (three protons). Accelerating these ions in a linear accelerator each ions get same kinetic energy. As the velocity is reduced by the number of protons in an ion. These ions are easily separated by mass separator. The velocity of proton defines the penetration depth into target material. This ion specific depth is very useful to form multi-level structure in material. The multi-level structure is extremely important in microelectronics. In microelectronics many miniature components are assembled in very limited space and even more the structural base is formed to get mechanical, electrical and optical functions. We will demonstrate important examples in microelectronic application. The first example is freestanding comb supported by frame structure. The comb is processed by H3+ beam and Frame is processed by H+ beam. Those two ions are accelerated mixed in 1MV linear accelerator and mass separated. The other example is component container formed on flexible base film. These two examples are universal structures in microelectronic and micro-mechatronic application.

## MICROMODIFICATION

#### Contributed talk III

## 1092

## Study on the Single Event Effect of PAVLOV Neuron Chip with Microbeam

<u>Guangbo Mao<sup>1,3</sup></u>, Jiale Quan<sup>2</sup>, Jinlong Guo<sup>1</sup>, Wenjing Liu<sup>1</sup>, Ruqun Wu<sup>1</sup>, Jing Zhao<sup>1</sup>, Cheng Shen<sup>1</sup>, Hongjin Mou<sup>1</sup>, Guanghua Du<sup>1</sup>

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#### Introduction

Artificial intelligence (AI) systems have very important application prospects in military and aerospace fields such as target recognition, on-orbit perception, automatic control and deep space exploration. Because the device architecture and operation mode of AI chips are different from traditional chips, it is necessary to study the influence of SEE, the most important irradiation effect in devices, on system reliability. In this work, a neuron chip, named PAVLOV, has been studied by simulation and microbeam experiments, trying to obtain the SEE soft error distribution and, moreover, to analyze the law of signal propagation.

#### **Description of the Work**

The PAVLOV neuron chip was designed and manufactured by the Institute of Microelectronics of the Chinese Academy of Sciences. First, the sensitive areas on the chip were obtained through simulations conducted by Cadence. Then, the laser microbeam and high energy heavy ion microbeam were used to irradiate the sensitive areas respectively. The chips were opened and thinned before the experiments to ensure that the laser photons and heavy ions could penetrate the device layer. The output signals were monitored to detect the SEE event, and at the same time the irradiation position was recorded by the scintillation imaging system. In the irradiation experiments, no SEE event was detected, indicating that the PAVLOV chip has excellent anti-irradiation performance. Some reasons have been taken out and discussed to explain the experiment results, and the unique operation mode of this neuron chip is the main factor.

#### Conclusion

The PAVLOV neuron chip has excellent anti-irradiation performance because the different device architecture and operation mode.

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Tohoku-University developed an automatic beam-focusing system to reduce the experimental configuration time for a submicrometer-scaled(\*) beam probe of MeV ions with various analytical applications.

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The system involves a two-stage process of contrast and phase-difference detection that includes an automated minimization algorithm for astigmatism.

The contrast method focused a proton microprobe to approximately  $1 \ \mu m^2$  within 30 min, without the need for manual control when rotational misalignment was not present, while the phase-difference method successfully reduced the rotational error of lenses.





(References: Nuclear Instruments and Methods in Physics Research B 496 (2021) 1-8)

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