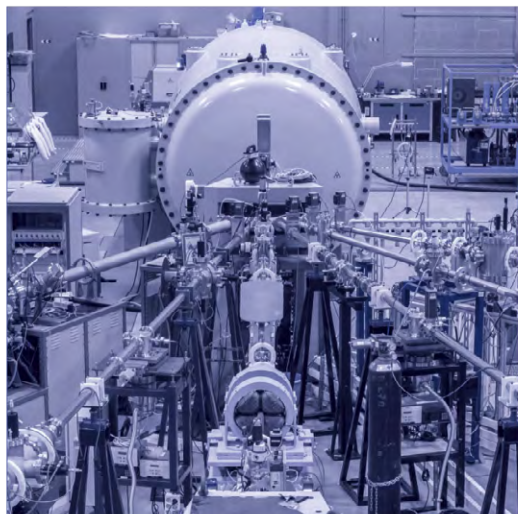
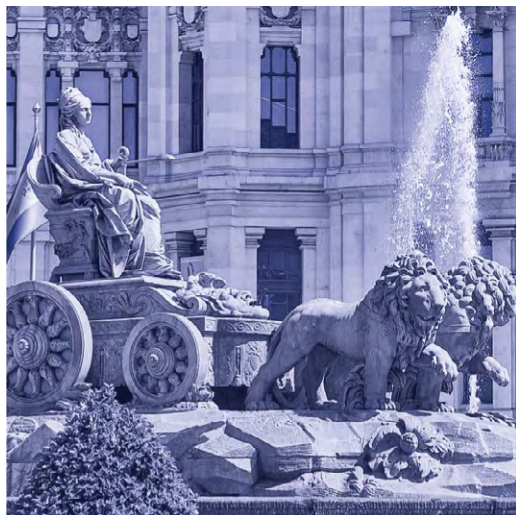


# ICNMTA 2024

**19<sup>th</sup> INTERNATIONAL CONFERENCE ON NUCLEAR  
MICROPROBE TECHNOLOGY AND APPLICATIONS**



**ABSTRACT  
BOOK**

**14<sup>th</sup>–19<sup>th</sup> July 2024 • Madrid**

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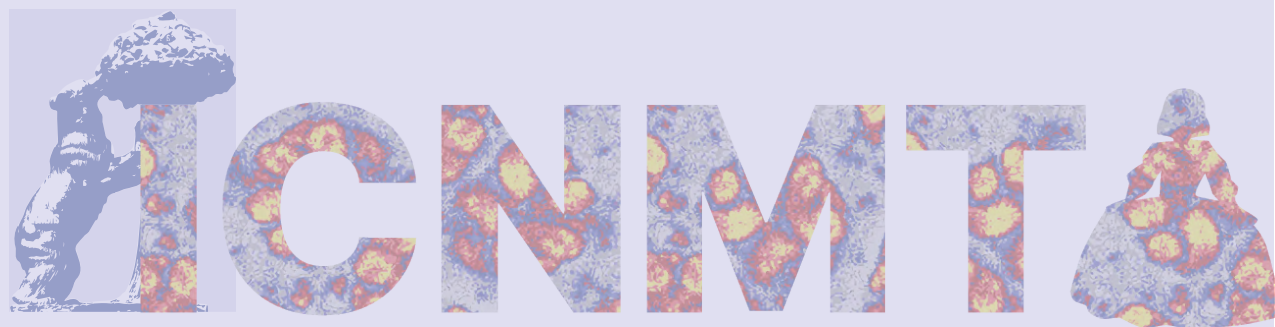
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PROGRAM

## SUNDAY 14<sup>th</sup> JULY

18:30 - 21:30 REGISTRATION & RECEPTION

## MONDAY 15<sup>th</sup> JULY

8:30 - 17:00 REGISTRATION & RECEPTION

9:00 - 9:15 **Welcome ceremony**

9:15 - 10:30 **Technologies. Micromodification**

Invited I | **Yanxin Dou**

Contributed I | **Kunpisit Kosumsupamala**

Contributed II | **Xingni Chai**

Contributed III | **Johnny Dias**

10:30 COFFEE BREAK

11:00 - 12:30 **Applications. Life Sciences & Radiation Oncology**

Invited II | **Franck Gobet**

Contributed IV | **Philippe Barberet**

Contributed V | **Rémy Lienard**

Contributed VI | **Theylor Klippel**

Contributed VII | **Teruaki Konishi**

12:30 CONFERENCE PHOTO AND LUNCH

14:00 - 15:30 **Applications. Life Sciences & Radiation Oncology**

Invited III | **Aliz Simon**

Contributed VIII | **Henrique Fonteles**

Contributed IX | **Diego Lecumberri**

Contributed X | **Matheus Ramos Caloni**

Contributed XI | **Thulaganyo Phillip Sehogela**

15:30 COFFEE BREAK

16:00 - 17:00 **Applications. Life Sciences & Radiation Oncology**

Invited IV | **Tamon Kusumoto**

Contributed XII | **Judith Reindl**

Contributed XIII | **François Vianna-Legros**

## TUESDAY 16<sup>th</sup> JULY

8:30 - 17:00 REGISTRATION

9:00 - 10:30 **Applications. Energy applications & Space, microelectronic and others**

Invited V | **Gyula Nagy**

Contributed XIV | **David Jamieson**

Contributed XV | **Željko Pastuović**

Contributed XVI | **Milko Jaksic**

Contributed XVII | **Sabrina Gouasmia**

10:30 COFFEE BREAK

11:00 **Technologies. Single ion technology & Quantum technology and nanodevices**  
- 12:30

Invited VI | **Steven Clowes**

Contributed XVIII | **Ettore Vittone**

Contributed XIX | **Andrew Bettiol**

Contributed XX | **Claire Léonhart**

Contributed XXI | **Elisa Redolfi**

12:30 LUNCH

14:00 **Technologies. Single ion technology & Quantum technology and nanodevices**  
- 14:30

Contributed XXII | **Elena Nieto Hernández**

Contributed XXIII | **Emilio Corte**

14:30 **Poster Session I**  
- 16:30

15:00 COFFEE BREAK  
- 15:30

17:30 **IAC Meeting**  
- 18:30

20:00 **IAC Dinner**  
- 22:30

## WEDNESDAY 17<sup>th</sup> JULY

8:30 - 14:00 REGISTRATION

9:00 **Applications. Environment, Cultural Heritage and Forensics**  
- 10:30

Invited VII | **Laura Guidorzi**

Contributed XXIV | **Iva Bogdanović-Radović**

Contributed XXV | **Noelia Maldonado Gavilán**

Contributed XXVI | **Shuichi Sada**

Contributed XXVII | **Thomas Calligaroa**

10:30 COFFEE BREAK

11:00 **Technologies. Detectors**  
- 12:15

Invited VIII | **Federico Picollo**

Contributed XXVIII | **Zhaohong Mi**

Contributed XXIX | **Carmen Torres Muñoz**

Contributed XXX | **Karla Ivanković Nizić**

12:15 **Sponsor talks**  
- 12:25

12:30 LUNCH

14:00 **Excursion to Toledo & Dinner at Hacienda El Cardenal**  
- 22:00

**THURSDAY 18<sup>th</sup> JULY**

8:30 - 17:00 REGISTRATION

9:00 **Technologies and Applications. Technique**  
- 10:30 **Advances and Emerging techniques**

Invited IX | **Catia Costa**

Contributed XXXI | **Gary Glass**

Contributed XXXII | **Anja Miokovic**

Contributed XXXIII | **Ce-Belle Chen**

Contributed XXXIV | **Melanie Bailey**

10:30 COFFEE BREAK

11:00 **Technologies and Applications. Technique**  
- 12:30 **Advances and Emerging techniques**

Invited X | **Zdravko Siketic**

Contributed XXXV | **Matjaz Kavcic**

Contributed XXXVI | **Victoria Corregidor**

Contributed XXXVII | **Yanxin Dou**

Contributed XXXVIII | **Matevž Skobe**

12:30 LUNCH

14:00 **Round Table:**  
- 15:00 **Data Acquisition**
**Round Table:**  
**High Precision**  
**Irradiation, Single**  
**Ion Detection**

15:00 COFFEE BREAK

15:30 **Poster Session II**  
- 17:00

**FRIDAY 19<sup>th</sup> JULY**

8:30 - 12:00 REGISTRATION

9:00 **Technologies. Instrumentation**  
- 10:30

Invited XI | **Geoffrey Grime**

Contributed XXXIX | **Robert Frost**

Contributed XL | **Bibhudutta Rout**

Contributed XLI | **Guanghua Du**

Contributed XLII | **Harry J. Whitlow**

10:30 COFFEE BREAK

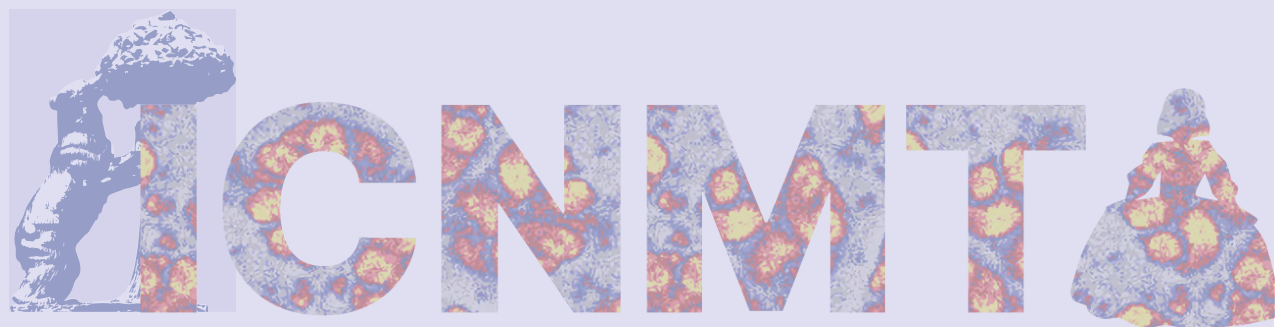
11:00 **Conference Summary & Highlights**  
- 11:30

11:30 **Best Posters Award Ceremony**  
- 11:45

11:45 **Concluding Remarks**  
- 12:00

12:30 LUNCH

14:00 **CMAM Visit**  
- 16:00



INVITED TALK

N° 11

# ELECTRIC FIELDS IN LIQUID WATER IRRADIATED WITH 3 MEV PROTONS AT ULTRAHIGH DOSE RATES

Gobet, F. (1); barberet, P. (2); Delville, M. (3); deves, G. (3); Guerin, T. (3); Liénard, R. (4); Tran, H. (3); Vecco-Garda, C. (3); Wurger, A. (4); Zein, S. (3); Seznec, H. (3)

(1) University of Bordeaux; France; (2) university of bordeaux; France; (3) CNRS; France; (4) university of Bordeaux; France

Recent advances in microbeam irradiation [1,2] as well as in electron or x-ray microscopy [3,4] of liquid or biological samples have revived studies of the physical, chemical, and biological effects resulting from the interaction between ionizing radiation and matter at ultra-high dose rates (10 kGy s<sup>-1</sup> – 100 MGy s<sup>-1</sup>). In the context of high-resolution imaging with this extreme regime of irradiation, the perturbation of the liquid sample outside the applied microbeam, through the generation of induced fields, remains an open question.

By coupling microbeam irradiation and fluorescence microscopy, we study the effects of irradiating liquid water with 3 MeV protons at high doses by observing the motion of charged polystyrene beads outside the proton beam [5]. By single-particle tracking, we measure a radial velocity of the order of microns per second. Combining electrokinetic theory with simulations of the beam-generated reaction products and their outward diffusion, we find that the bead motion is due to electrophoresis in the electric field induced by the mobility contrast of cations and anions produced in the radiolysis of water. This work sheds light on the perturbation of biological systems by high-dose radiations and paves the way for the manipulation of colloid or macromolecular dispersions by radiation-induced diffusiophoresis.

[1] V. Ialyshev et al., ACS Omega 7, 28182 (2022).

[2] L. Eling, et al., Radiother. Oncol. 139, 56 (2019).

[3] H. Wu, H. Friedrich et al., Adv. Mater. 32, 2001582 (2020).

[4] C. Y. Hémonnot and S. Koster, ACS Nano 11, 8542 (2017).

[5] F. Gobet et al., Phys.Rev.Lett. 131, 178001 (2023).

N° 76

# THE OXFORD TRIPLET REVISITED. A LONG VIEW OF THE DEVELOPMENT OF PROBE FORMING SYSTEMS FOR MEV IONS

Grime, G. (1); Palitsin, V. (1); Watt, F. (2)

(1) University of Surrey Ion Beam Centre; United Kingdom; (2) Oxford Microbeams Ltd.; United Kingdom

It is over forty years since the first MeV ion microbeam with 1  $\mu\text{m}$  beam diameter was established at the University of Oxford [1]. This instrument employed a coupled triplet configuration of magnetic quadrupoles which was selected following a detailed survey of the beam optics of quadrupole multiplets [2]. This has since become known as the 'Oxford Triplet'.

In the 1980s, the belt driven Van der Graaff accelerators commonly used provided beams with a large energy spread and often low brightness, which meant that the lens aberrations were dominated by 'chromatic' effects. This was in some ways fortunate, since calculating chromatic aberration was simple. Calculating aberrations with orders higher than first order in entrance angles relied on algebraic solutions to the off-axis motion of ions in multipole fields and was a severe computational challenge using computers readily accessible at the time. Raytracing software was under development [2], but tracing enough rays to optimise a system or obtain spot profiles required many hours of CPU time on a mainframe.

In the intervening four decades since the 1980s, accelerator energy spread has been virtually eliminated and beam brightness has improved, which means that the dominant focusing aberrations are now the third order 'spherical' aberrations. In parallel, computer performance has improved exponentially to the point where a laptop can model the optics of a real quadrupole lens in a few minutes [3]. Thus it becomes possible to explore new systems in a short time.

This talk discusses how the approach to quadrupole probe lens design has changed since the 1970s and describes enhancements to the Oxford Triplet which significantly improve the focusing and scanning performance. Along the way we derive a simple expression highlighting how all the components of a microbeam system (accelerator, lens, detectors) conspire to restrict the achievable spatial resolution.

## References

- <sup>1</sup> F. Watt, G. W. Grime, G. D. Blower and J. Takacs, "A Coupled Triplet Configuration of the Oxford Microprobe," in IEEE Transactions on Nuclear Science, vol. 28, no. 2, pp. 1413-1416, April 1981,
- <sup>2</sup> G.W. Grime, F. Watt Beam Optics of Quadrupole Probe-Forming Systems, Adam Hilger Ltd., Bristol (1983)
- <sup>3</sup> G.W. Grime, WinTRAX: A raytracing software package for the design of multipole focusing systems, Nucl. Instr. and Meths. B: 306 (2013) 76-80,



N° 83

# ION-BEAM DRIVEN HIGH RESOLUTION MATERIALS SCIENCE RESEARCH COORDINATED BY THE IAEA PHYSICS SECTION

Simon, A. (1)

(1) International Atomic Energy Agency, Austria

Ions from accelerators have a prominent role in testing and developing novel materials and devices due to their capability in introducing controlled damage based upon the possibility to define with high accuracy the ion fluence, determine the damage profile and localize the damaged region. Both immediate effects of radiation induced damage on material and device electrical properties and also the longer term accumulation of damage which can limit the useful lifetime can be characterised. Direct experimental access to study the dynamics of radiation induced defects, from femto-seconds to seconds, makes it possible to design materials with tailored responses to radiation, from radiation hardness to the engineering of desired defects [1].

The IAEA supported a coordinated a project to develop materials for quantum technologies (F11020). The CRP included the development of new experimental techniques and the refinement of theoretical models, with an aim to understand radiation effects and ion interaction processes.

This paper will present some of the scientific results of the IAEA supported activities and projects in the above areas and an outlook for planned projects will be also given.

A new coordinated research project on Sub-cellular Imaging and Irradiation using Accelerator-based Techniques (F11024) started in 2023. The main objective of the project is to develop novel accelerator-based techniques for sub-cellular imaging and biological cell irradiation techniques in order to advance knowledge and capabilities in understanding how biological cells respond to radiation towards more efficient and tailored particle therapy.

This talk will give an overview on our strategies how to foster accelerator science and technology in the above fields.

[1] INTERNATIONAL ATOMIC ENERGY AGENCY, Guidelines for the Determination of Standardized Semiconductor Radiation Hardness Parameters, Technical Reports Series No. 490, IAEA, Vienna (2023)

[https://www-pub.iaea.org/MTCD/Publications/PDF/PUB\\_DOC\\_490\\_web.pdf](https://www-pub.iaea.org/MTCD/Publications/PDF/PUB_DOC_490_web.pdf)

N° 91

# QUANTIFICATION OF COMPOSITION GRADIENTS IN METAL-ORGANIC FRAMEWORKS

Nagy, G. (1); Gschwind, W. (1); Ott, S. (1); Primetzhofer, D. (1)

(1) Uppsala University; Sweden

Rutherford-backscattering spectrometry (RBS) is a versatile analytical method that can provide depth-resolved information on the elemental composition of a sample. A decreasing concentration of an element with probed depth shows up as a decaying signal intensity towards lower energies of ions backscattered from this particular element. This information, however, cannot be directly transformed to an absolute concentration gradient value.

In the present work we measured the composition of metal-organic framework (MOF) single crystals by  $\mu$ -RBS. MOFs feature extremely large surface-to-volume ratio, making them highly attractive in different technological applications. Active sites are often introduced post-synthetically, resulting in various spatial/depth distributions. We have already shown that the scanning nuclear microprobe is a unique tool for the sensitive, non-destructive depth profiling of MOF single crystals in a qualitative manner [1].

In this presentation we propose a method for the quantification of the composition depth gradients. Zr-based UIO-67 MOFs were synthesized on Si substrates and metalated post-synthesis by  $\text{NiCl}_2$ . After metalation, the crystal composition was measured by  $\mu$ -RBS at the scanning nuclear microprobe in Uppsala [2].

The recorded  $\mu$ -RBS spectra were processed using the SIMNRA software [3]. The elements of interest featured a gradient according to a hypothetical gradient function. The shape of this gradient function is indicative of the governing process of the post-synthetic element incorporation, such as molecular diffusion, while the function parameter value describes the process quantitatively, such as it can directly provide the diffusion coefficient of a given system. The deviation between the measured and simulated spectrum was minimized by a Nelder-Mead-based minimizer algorithm. The simulated spectrum, calculated by SIMNRA, is a function of the surface concentration as well as the gradient function form and parameter(s) of the gradient element(s), therefore, the minimization returns the depth-resolved target composition which best fits the measurement.

## References

- <sup>1</sup> B.D. McCarthy, et al, JACS 143, 44 (2021) 18626-18634.
- <sup>2</sup> G. Nagy, H.J. Whitlow, D. Primetzhofer, NIM B 533 (2022) 66-69.
- <sup>3</sup> M. Mayer, SIMNRA User's Guide, Report IPP 9/113, Max-Planck-Institut für Plasmaphysik, Garching, Germany, 1997.

N° 125

## MEV SIMS MOLECULAR IMAGING USING A CONTINUOUS COLLIMATED ION BEAM

Siketic, Z. (1); Barac, M. (2); Brajković, M. (1); Bogdanović-Radović, I. (1); Krmpotić, M. (1); Popović Hadžija, M. (1)

(1) Ruđer Bošković Institute, Bijenička c. 54, Zagreb, Croatia; (2) Ruđer Bošković Institute, Bijenička c. 54, Zagreb, Croatia. Jožef Stefan International Postgraduate School, Jamova c. 39, Ljubljana, Slovenia

Time-of-flight Secondary Ion Mass Spectrometry utilizing MeV heavy ions (MeV SIMS) has emerged as a cutting-edge technique for the identification and imaging of molecular content within organic samples. Its ability to analyze high-mass compounds with exceptional sensitivity, combined with its surface sensitivity, makes MeV SIMS a versatile tool for the analysis of various organic materials, including fingerprints, paints, inks, tissues, etc. As MeV SIMS employs MeV ions, commonly used in Ion Beam Analysis (IBA), it complements IBA by providing additional chemical information about the analyzed samples.

In this study, we introduce a novel system for molecular imaging of organic materials, incorporating a continuous analytical beam and a start trigger mechanism based on the detection of secondary electrons. The sample is imaged using a collimated primary ion beam, allowing for scanning of the target with a lateral resolution of approximately 20  $\mu\text{m}$ . The START signal for TOF measurement is generated by secondary electrons emitted from a thin carbon foil ( $\sim 5\text{ nm}$ ) placed over the beam collimator, and the mass of the analyzed molecules is determined using a reflectron-type TOF analyzer.

This configuration enables the use of a heavy primary ion beam with maximum electronic stopping power, resulting in the highest secondary molecular yield, and enables the analysis of samples of varying thickness. By collecting electrons from the thin foil rather than from the sample surface, uniform detection efficiency of secondary electrons is achieved across different material types. Additionally, the ability to scan samples using a piezo stage allows for the imaging of samples with surface areas of a few square centimeters.

The imaging capabilities of the presented MeV SIMS setup will be demonstrated on crossing ink lines deposited on the paper, on thin sections of mouse brain tissue, and analysis of fingerprints on thick Si wafers. These results underscore the potential application of the presented technique for analytical purposes in biology and forensic science.

N° 126

## FOCUSSED BEAM SINGLE ION IMPLANTER FOR QUANTUM TECHNOLOGY

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Deterministic implantation of single ions is currently of high interest for quantum technology applications. We have recently commissioned two instruments to carry out deterministic implantation across a range of ion species. These systems located in the UK National Ion Beam Centre (UKNIBC) are bespoke focussed ion beam systems designed specifically for the task of deterministic implantation and comprise one, which has a liquid metal ion source, and the second, a duoplasmatron source. Both systems use a pulsed, low current beam of ions and contain Wein filter mass/charge filtering; neutral filtering and secondary electron detection to determine when an implantation event has occurred. They operate an EBL-like strategy for precise location of the ion pulse at the desired locations and with resolutions on the same scale as the ion straggle.

While we have now run across the two systems a range of sources such as In, Bi, Si, C, N, O, Ge, Er, Sn and Cr. The task ahead is to establish stable and reliable sources for additional species (for example rare earths) of particular interest to the quantum research community and we have an ongoing sources development program to expand the range of available species. Here we also present single ion implant detection results from the single ion implanter where we study detection efficiencies for different ion species/substrate combinations.

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N° 128

## ION MICROPROBES FOR PROVENANCE STUDIES OF CULTURAL HERITAGE MATERIALS

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Ion Beam Analysis (IBA) provides different insights on samples, simultaneously examining chemical composition, structural and luminescent properties, even on very small features when a microbeam is employed.

Over the years, the Solid State Physics group at the University of Torino and the Torino section of the National Institute for Nuclear Physics (INFN) established several collaborations to perform IBA on different materials of interest in the Cultural Heritage field.

Since 2008,  $\mu$ -PIXE and  $\mu$ -IBIL have been performed on lapis lazuli samples with the aim of searching markers in reference geological rocks to build an analytical protocol that can be later applied to ancient artefacts for the provenance determination of the raw material used in antiquity. Measurements were performed in vacuum on rocks samples and ancient small working flakes at INFN-LNL (Legnaro, Padua) microbeam line, as well as at international facilities like the Ruđer Bošković Institute (Zagreb, Croatia) and the Surrey Ion Beam Centre (Guilford, UK) via CERIC and RADIATE transnational access programs, respectively. For the ultimate purpose, measurements on archaeological or artistic lapis lazuli objects were performed in air without sample preparation, initially at INFN-LABEC (Florence) and in the last ten years at the NewAGLAE facility (C2RMF, Paris), thanks to CHARISMA, IPERION CH and IPERION HS European programs. Up to now the protocol can discriminate between five provenances and it has been successfully applied to precious artworks from museum collections.

Trace element concentrations can serve as strong provenance markers also for man-made materials like ceramics. Black-glaze ware and red figure pottery were produced in Southern Italian workshops during the 6th to the 4th century BCE;  $\mu$ -PIXE at INFN-LNL was employed to analyse the micrometric black vitrified layers on red-figured vases, aiming to differentiate between Calabrian and Sicilian workshops.

All the analytical approaches, tailored to the specific sample type, and the obtained results will be presented and discussed.

N° 129

## ENHANCING NUCLEAR MICROPROBES FOR ADVANCED MICROMODIFICATION

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Due to the principles of MeV ion-matter interaction, nuclear microprobes provide distinct advantages for advanced micromodification [1][2]. Nuclear microprobes fabricate structures with high aspect ratio, multiple depth, and smooth sidewalls. However, the resolution of nuclear microprobes limits the minimum achievable feature size. Here, we employ an enhanced nuclear microprobe to pattern hydrogen silsesquioxane (HSQ), achieving a feature size of approximately 11 nm. Our work demonstrates a direct patterning of perovskite material using nuclear microprobes.

Furthermore, this presentation will discuss sub-10 nm fabrication technologies using photons [3], electrons [4][5], atoms [6], and ions [7-10]. By comparing their advantages and disadvantages, we can identify a promising direction for micromodification using nuclear microprobes.

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N° 130

## ION BEAM MODIFICATION OF DIAMOND FOR CELLULAR SENSING

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In recent years, the Deep Ion Beam Lithography (DIBL) technique was optimized to microfabricate single-crystal diamond by means of MeV ion microbeams. This approach allows tuning the optical properties of the material, both concerning its refractive index and to the formation of luminescent centres. In addition, the structural properties of the material undergo significant modification upon MeV ion irradiation (surface swelling, stress-induced effects). Most importantly in the present context, by overcoming a critical fluence during diamond irradiation it was possible to create graphitic structures in single-crystal diamond. This approach takes advantage of the metastable nature of diamond, which can be converted into the stable allotropic form of carbon at room temperature and pressure conditions (i.e., graphite) by creating high defect concentration in the lattice [1].

These electrically conductive graphitic channels are the key structures of innovative diamond-based multi-functional sensors [2], which are employed to investigate cellular activity both as biomolecules secretion (quantal exocytic events) and as electrical signal generation (action potential firing).

In particular, the study of several biological systems such as cells networks of hippocampal neurons, dopaminergic neurons, chromaffin cells [3], that are cultured for long period directly over the device's surface taking advantage of diamond biocompatibility, or tissue slices from the adrenal gland and substantia nigra brain compartment [4] are reported.

Moreover, these devices were also employed for novel radiobiological experiment devoted to the investigation of ionizing radiation on neuron-like cells: for the first time, the variation of cellular activity (activation of exocytosis pathways) was observed in real-time during cell irradiation with X-ray beam [5-6].

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N° 131

# UNVEILING MECHANISMS OF FLASH RADIOTHERAPY BY RADIATION- CHEMISTRY AND RADIATION-BIOLOGY EXPERIMENTS

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(1) National Institutes for Quantum Science and Technology (QST), Japan

Radiotherapy performed at ultra-high dose rate (UHDR:  $> 40$  Gy/s) has been attracted a big attention due to its excellent advantage, which effectiveness of the therapy for tumors is maintained while damages to healthy tissues are reduced. The sparing effect on healthy tissues is observed in biological experiments using small animals. Furthermore, the inhibition of cancer metastasis was reported. However, the mechanism of FLASH radiotherapy is not clear. Therefore, we have addressed radiation-chemistry and radiation-biology experiments to elucidate the mechanism of UHDR effects. In the presentation, our recent progress will be presented, especially focusing on changes in yields of water radiolysis species (OH radicals, hydrogen peroxide and hydrated electrons) and DNA damages (double strand breaks, single strand breaks and base damage) in a wide dose rate range from 0.01 to 100 Gy/s. The detail is as follows;

1. OH radical measurements: By UHDR irradiation, dense track structure could be expected relative to that at conventional dose rate (CONV:  $< 0.1$  Gy/s). So, we evaluate changes in yields of OH radicals with dose rate using a chemical probe.
2. Hydrogen peroxide: When radical-radical reactions occur efficiently in UHDR region, compared to that in CONV dose rate region, due to the dense track structure, yields of hydrogen peroxide, which forms the reaction between OH radicals, could increase with increasing dose rate. Therefore, we follow changes in yields of hydrogen peroxide using Ghomley method.
3. Hydrated electrons: One of the hypotheses of the mechanism of UHDR effects is considered as a oxygen depletion. Hydrated electrons are known as a scavenger of oxygen, thereby they have strong relation to the reduction of oxygen concentration. Thus, we evaluate changes in yields of hydrated electrons using Saltzman method.
4. DNA strand breaks: To see a relation between changes in yields of water radiolysis products and the reaction and those of DNA strand breaks, we evaluated yields of DNA double strand breaks and DNA single strand breaks by agarose gel electrophoresis. Additionally, yields of base damages are derived by fpg enzyme treatments.

N° 132

## MULTIMODAL IMAGING USING ION BEAM ANALYSIS AND MASS SPECTROMETRY: WHERE ARE WE?

Catia Costa (1); Johanna von Gerichten (2); Vladimir Palitsin (1); Geoffrey W. Grime (1); Naoko Sano (3); Roger Webb (1); Melanie J. Bailey (1,2)

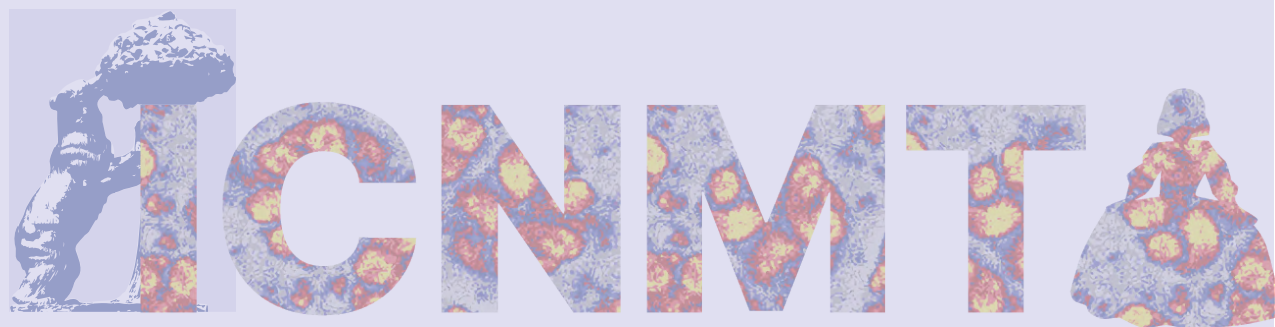
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Co-localising elemental and molecular markers can be of great importance in biology and medicine to help study and understand disease pathogenesis. For example, metal accumulation or depletion can impact the local molecular chemistry (e.g. metabolite content and/or protein expression), which in turn can promote or limit pathogen proliferation. To accurately correlate elemental and molecular markers, it is desirable to perform sequential elemental and molecular imaging on a single-tissue section. However, concerns regarding sample preparation requirements and technique compatibility can present some hurdles to the integration of the two modalities.

Here we highlight some of the challenges and successes associated with performing elemental mapping in sequence with mass spectrometry imaging. Specifically, we explore the feasibility of molecular mapping using the mass spectrometry imaging (MSI) techniques matrix-assisted laser desorption ionization (MALDI), desorption electrospray ionization (DESI) and secondary ion mass spectrometry (SIMS), in sequence with particle-induced X-ray emission (PIXE). Challenges for integration include substrate compatibility, as well as analyte delocalization and spectral changes by preceding analysis.

This work sets the stage for the first-of-its-kind **Multimodal Ion Beam Imaging Facility** that will combine conventional ion beam analysis (IBA) with SIMS, in the same analysis chamber, allowing for truly sequential molecular and elemental mapping on the same sample. This facility will consist of a commercially available J105 (Ionoptika) SIMS instrument placed at the end of an MeV beam line, and furnished with SDD and PIPS detectors. Not only will the system allow sequential IBA and keV-SIMS, but it will also offer the possibility for MeV-SIMS analysis using a mass spectrometer that offers high resolution, mass accuracy and MS/MS capabilities. The usefulness of the system will extend beyond biological applications to material sciences and clean energy research.





# POSTER COMMUNICATION

N° 5

# ONE-PARAMETRIC QUADRUPLLET WITH VARIABLE DEMAGNIFICATIONS

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The nuclear microprobe of the Institute of Applied Physics of the National Academy of Sciences of Ukraine in Sumy was commissioned into operation in 2008. The performance of the microprobe was published in [1,2]. The basic probe-forming system was chosen taking into account the parameters of compact 2MV Van de Graaff accelerator and is a separated orthomorphic quadruplet of magnetic quadrupole lenses. The basic system has low demagnifications  $Dx \times Dy = 23 \times 23$ , which is due to the relatively short length of the system  $\approx 4$  m and a large working distance of 24 cm. To improve the spatial resolution, theoretical and experimental studies have been carried out on a probe-forming system based on a two-parametric separated quadruplet with individual lens power supplies [3,4]. The demagnifications for the optimal two-parametric quadruplet are  $Dx \times Dy = 52 \times 96$ . During the operation of such a probe-forming system, its drawbacks disadvantages have been identified, related to the difficulty of adjusting the system, which takes a long time.

In this presentation, we consider a probe-forming system based on a one-parametric separated quadruplet of magnetic quadrupole lenses without changing geometry of the base system. The excitation layout for the quadrupoles Q2, Q3 and Q4 is a high excitation triplet C1D1C2, the first quadrupole lens Q1 is excited by a third independent power supply and can be either convergent or divergent. The value of the third power supply is a free parameter, that determines the ion-optical properties of the probe-forming system. The demagnifications are a function of the field values of the first quadrupole and may vary from  $104 \times (-52)$  to  $182 \times (-214)$ . One-parametric quadruplet significantly simplifies the adjustment process and is comparable in its focusing properties to a two-parametric system.

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N° 6

## PALLADIUM (PD) DECORATED ZINC OXIDE (ZNO) FOR GAS SENSING APPLICATION

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The detection of odors emitted by meat products when start to spoil remains a challenge as a result ZnO decorated with palladium nanoparticle are synthesis using hydrothermal method for the purpose to test their gas sensing properties. After the synthesis of these nanoparticles, various characterization techniques namely SEM with EDS, XPS, Raman, UV/vis and XRD for both physical and chemical properties investigation were employed. Different percentages of Palladium ranges between (0.2% to 0.7%) from PdCl<sub>2</sub> precursor with 99.9% purity was used during the synthesis. A gas sensor was fabricated through drop casting method on a gold grid. These gas sensor samples were exposed to both reducing and oxidizing gases. Sensing was performed at a temperature between 25°C to 225°C, the best result was obtained at 150°C using 0.2%Pd-ZnO sensor when sensing NO<sub>2</sub> gas. The sensitivity of 3.8 and response time and recovery time of both 3.5 minutes. The 0.5%Pd- ZnO sensor performs well at room temperature for CO<sub>2</sub>, SO<sub>2</sub>, and ethanol gases. Given the transformative potential demonstrated by ion microbeams in elucidating complex cellular responses and material interactions, the exploration of incorporating ion microbeams into our experimental framework holds immense promise for unlocking novel insights and advancing the frontiers of our research

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N° 7

## MICROFABRICATION OF STRUCTURES USING PROTON BEAM WRITING

Mongwaketsi, N. (1); Mtshali, C. (1); Khumalo, Z. (2); Kheswa, N. (1); Guga, A. (3); Barnard, H. (1)

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Research on PBW using nuclear microprobe beamline of the 3 MV Tandetron commenced in 2018 yielding promising results [1]. Subsequent efforts have been directed towards improving our PBW system, with ongoing exploration focused on enhancing beam focusing to achieve a smaller spot size. As part of the system optimization, OM150 triplet quadrupole lenses functionality and beam focusing capability were investigated. This investigation encompassed an analysis of beam optics, accounting for distances from the focal point of the analyzing magnet to the halo slit, as well as the initial and final positions of each of the three quadrupole lenses. Field strengths were measured consistently for each quadrupole to ascertain polarities. The findings revealed that the quadrupole

lenses exhibit a stronger focusing effect along the X-axis compared to the Y-axis. Subsequent to beam optics measurement and efforts to define the central position of the beam through adjustments of object and halo apertures, notable improvements were observed on the beam spot size. Patterns of structures fabricated on PMMA were generated using custom-developed software. In addition, PBW by raster scanning beam over PMMA was carried out to gain further insight into the shape and dimensions of the beam spot. The morphology of the fabricated structures was evaluated using atomic force and scanning transmission microscopy.

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N<sup>o</sup> 9

## DEPTH DISTRIBUTION OF HIGH DOSES OF BORON IMPLANTED IN IRON SURFACES

Gaal, Z. T. (1); Halasz, Z. (2); Haki, J. (2); Takats, V. (2); Fodor, T. (2); Molnar, S. (2); Csik, A. (2); Daroczi, L. (3); Benke, M. (4); Salyi, Z. (4); Nagy, G. (5); Oscarsson, J. (6); Primetzhofer, D. (6); Vad, K. (2); Rajta, I. (2); Benko, Z. (2)

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By using lead-free material in soldering technology, the degradation of industrial tools is accelerated. However, this can be circumvented with boriding, creating iron-boride surfaces on the steel tools. This protective layer increases resistance to wear and corrosion, resulting in more stable devices. With diffusion methods there are always two types of boride phases made, FeB and Fe<sub>2</sub>B. There is always stress on the interface of the two phases, making the structure mechanically unstable. Our aim with B<sup>+</sup> ion implantations is to avoid this weakness and create one type of iron-boride phase.

The ion implantation was performed by different doses (1e17-1e18 ions/cm<sup>2</sup>) of boron ions and with bombarding energies of 25, 50, 75 and 100 keV. The depth distribution of boron in high-dose implanted iron surfaces was investigated by Secondary Neutral Mass Spectrometry (SNMS) depth profile analyses, Scanning Electron Microscopy (SEM) measurements, and by Elastic Recoil Detection Analysis (ERDA). The purpose of these measurements was to get information about the boron distribution produced by high dose implantation. By our experimental analyses, we could show that the boron distribution is rather a Pearson-type than a Gaussian-type.

N° 10

## EVIDENCE FOR EFFICIENT RADICAL-RADICAL REACTIONS IN ULTRA-HIGH DOSE RATE REGION: CHANGES IN YIELDS OF OH RADICALS UNDER “MICRO” AND “MACRO” PROTON BEAMS

Kusumoto, T. (1); Okazaki, Y. (2); Mamiya, T. (1); Oikawa, M. (1); Kataoka, J. (2); Konishi, T. (1)

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Radiotherapy performed at ultra-high dose rate (UHDR: > 40 Gy/s) has been attracted a big attention due to its advantage, which effectiveness of the therapy for tumors is maintained while damages to healthy tissues are reduced. This therapy is so-called FLASH radiotherapy. To elucidate the mechanism from a point of view of radiation chemistry, we have tackled measurements of water radiolysis products, especially focusing on OH radicals that efficiently react with DNA and proteins.

Yields of OH radicals decrease with increasing dose rate by reactions between water radiolysis products (radical-radical reactions). This finding implies that dense track structure in high-dose rate region is formed, resulting in efficient reactions of water radiolysis products between neighboring tracks.

To demonstrate this hypothesis, OH radicals were measured using Amplifl Red solutions with 50  $\mu$ M, which is a scavenger of OH radicals. We shot 3.4 MeV protons with a constant beam intensity of  $5 \times 10^5$  protons/sec using a “micro” beam with  $2 \mu\text{m} \times 2 \mu\text{m}$  and a “macro” beam with  $100 \mu\text{m} \times 100 \mu\text{m}$ . After the irradiation, the fluorescence intensity of resorufin was measured using a fluorescent spectrophotometer (excitation: 352 nm, emission 382 nm).

When OH radicals react with benzene ring in Amplifl Red, a fluorescent product resorufin is formed. The intensity of resorufin increases monotonically with increasing absorbed dose (Figure 1). The fluorescence intensity with the “macro” beam rises  $1.5 \pm 0.2$  times rapidly than the “micro” beam. This finding demonstrates that radical-radical reactions occur more efficiently under dense track structure condition, which could be seen with increasing dose rate. Radical-radical reactions, resulting in the reduction of OH radicals that react with DNA and proteins, would be one of the mechanisms of ultra-high dose rate effects.

N° 12

## INSTALLATION AND PERFORMANCE OF A NUCLEAR MICROPROBE USING AN ELECTROSTATIC QUADRUPLLET

Naab, F. (1); Flick, A. (1); Niraula, P. (1); Jiao, Z. (1); Field, K. (1); Hirst, C. (1); Penders, A. (1); Was, G. S. (1)

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We will detail our advancements in microprobe ion beam analysis achieved at the Michigan Ion Beam Laboratory (MIBL). MIBL's integration of an upgraded beamline, accommodating a newly installed electrostatic quadrupole, has facilitated the precise manipulation of ion beams ranging from 5 to 100 microns. Our approach leverages the capabilities of the 1.7 MV tandem accelerator to conduct ion beam analysis on complex sample geometries and architectures due to the increased spatial resolution. Here, the steps taken to configure and validate the ion beam full width at half maximum, intensity, and spatial location will be detailed. The newly developed configuration is well aligned with the upcoming trends of performing multi-beam, gas-gradient ion irradiation experiments to expediate radiation tolerance evaluation of candidate materials for fission and fusion energy systems. A recent ion beam gas-gradient experiment – where a continuous gradient of helium and hydrogen ions implanted into a silicon substrate – with subsequent ion beam analysis using the new microprobe configuration will be highlighted. The findings from these coupled experiments provides invaluable insights into the properties of structural materials under irradiation, with broader implications for material science and nuclear engineering.

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Nº 13

## ION MICROPROBE ANALYSIS APPLIED TO THE STUDY OF WEAR PROCESSES IN TRIBOLOGICAL EXPERIMENTS WITH SOLID LUBRICANTS

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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. In a study, in which HZDR participates<sup>1,2</sup>, tribological experiments with counter bodies (CB) on a coating are used to study friction-induced surface changes like material loss of the coating, material transfer, or the formation of a tribolayer.

The current work presents Ion Beam Analysis (IBA) methods to deliver laterally and depth-resolved element analysis of the wear track on the coating and the contact area of the CBs. The coating is a tetrahedral amorphous carbon (ta-C) layer and the CBs are brass and steel. Both the IBA method and the ion beam have to be selected to maximise the information that can be obtained from the measurements. A combination of He and H ion microbeams and RBS and PIXE have been used for the analysis.

In this work it is shown that RBS is very useful because it is depth sensitive, which is needed to locate elements on the surface but that the interpretation of RBS maps is more difficult compared to PIXE maps and can even be misleading without taking the local spectrum into account. Also the type of ion beam can be optimised for the material under consideration. RBS with a 2 MeV He ion beam is useful to determine the transfer layer from the 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. Several other methodological considerations about the application of IBA methods to this project are discussed.

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Nº 14

## COMPOSITIONAL STUDY OF COINS FROM ERCAVICA ARCHAEOLOGICAL SITE IN SPAIN

Redondo-Cubero, A. (1); Rubio, R. (2); Isabel Ludeña, S. (2); Viñals, S. (1); Gordillo, N. (1)

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Coins are very valuable to establish historical, social, and economic context in many archaeological studies. In this work we analyze two sets of coins (both silver and bronze) found in the old Roman city of Ercavica, in Cuenca (Spain) [1]. The silver set is composed of denarius from pre-Roman epoch (ca. 160 BC) to the Middle Empire (ca. 220 AD). The bronze set is composed of ases from Ercavica and Segobriga. With the first set we study the compositional patterns of denarius across time, while the second serves to compare the trace elements in ases, with the final goal of identifying potential mines used in the past [2]. The coins were measured in the external microprobe at CMAM [3], using 2 MeV H beam, focused down to a 150 µm diameter. Two PIXE detectors for low and high energies were used, combined with an additional particle detector for RBS.

The analysis, carried out with GUPIX software [4], evidence high purity coins (more than 94% Ag) with Cu (5%) as the main alloying metal. Impurities found were Au (0.35%), Fe (0.18%), Pb (0.07%) and Bi (0.03%). This pattern has been confirmed in the other Iberian coins [5]. However, roman coins show a continuous decrease in Ag content with time, following the trend observed in [6]. Regarding bronze coins, the spectra show a rich variety of elements, with Cu as the main metal and Si as the main impurity. Among the impurities, the most relevant fact is the presence of a high amount of Pb in Ercavica coins (>15%) compared to Segobriga coins (<0.2%). Our studies demonstrate a continuous decrease in the silver content with time for the denarii. The preliminary data from bronze series suggest different geological origin or preparation method, but further coins will be analyzed to confirm this fact.

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N° 15

## CREATION OF NV CENTRES IN DIAMOND USING THE JSI NANOPROBE

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Nitrogen–vacancy (NV) centres are a type of point defect in the diamond lattice where a carbon atom is replaced by a nitrogen atom which is then coupled to a vacancy at one of the nearest neighbour sites. These pairs form quantum objects that exhibit specific optical properties (photon absorption and emission) which can be exploited in a variety of applications in the nano-technology related fields (e.g. nano-sensors).

On the other hand, by implanting single ions at highly localised sites close enough to each other ( $\leq 200$  nm), one can observe quantum entanglement between single NV centres which makes them a suitable model for exploring quantum phenomena and are showing potential as a candidate in developing quantum computing. For this to be achieved, one has to be able to deterministically implant ions while having a well focused and stable ion beam, which is the main challenge as the two criteria are often mutually exclusive.

By impinging N-15 into  $2 \times 2$  mm<sup>2</sup> electronic-grade diamond substrates at 3.8 MeV with a  $\sim 1 \times 1$   $\mu$ m<sup>2</sup> beam at our new nano-beamline we have implanted nitrogen atoms at predetermined sites and in chosen patterns at a range of surface densities ( $10$ – $10^3$  per  $\mu$ m<sup>2</sup>). After vacuum annealing at 1000 K for 12 h, NV centres have been created which we confirmed by investigating the samples under a fluorescence microscope. With further characterisation and optimisation of the beamline, especially in terms of beam spot size, we strive towards single ion regime in the near future.

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N° 17

## ARTIFICIAL INTELLIGENT FOCUSING OF A MICROBEAM SYSTEM BASED ON REINFORCEMENT LEARNING

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The ion microbeam facility is a highly effective tool for precise sample irradiation, ion beam micro-modification and ion beam analysis, and other applications at the micron and nanometer scale. However, achieving high-resolution beam spots requires meticulous adjustment of the microslit setting, beam transport and magnetic focusing field, which is even time-consuming for well-trained technicians. Nowadays, most beamline instruments and power supplies support remote control and automatic adjustment, which promote the application of artificial intelligence to microbeam formation. In this work, we simulated the 50MeV proton microbeam system with Oxford triplet lens configuration using a homemade ion optics package which can generate data about any number of particles passing through quadrupole magnets. Then, an agent interacted with the system and generated ten millions of pieces of data. After that, these data were used to train a DQN model, which is a neural network that recognizes the input state and gives the optimal action value for the state. Ultimately, we used the model to accomplish the intelligent focusing function for the Ion microbeam system. The comparative results show that the error between the results of the algorithm's intelligent focusing and the results calculated with the classical software is less than 1%.

N° 23

## CHARACTERIZATION OF ELEMENTAL COMPOSITION IN A 4TH CENTURY AD ANCIENT COIN USING MICRO-PIXE ANALYSIS WITH FOUR-CHANNEL SDDS

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The study of elemental composition in cultural heritage objects, such as ancient coins, provides valuable insights for archaeologists into the historical, geographical, and political contexts of these artifacts. Additionally, analyzing constituent elements and their changes over time is vital for implementing preservation measures. This article examines the composition and concentration of constituent elements in a Roman coin dating back to the 4th century AD.

Micro Particle-Induced X-ray Emission (micro-PIXE) analysis utilizing annular four-channel Silicon Drift Detectors (SDDs) proves to be an effective technique for studying coin surfaces, offering non-destructive analysis, large solid angle detection, limited penetration depth, and multi-element distribution detection capabilities. Investigations across different regions of the coin revealed major concentrations of (Cu) at  $37.41 \pm 2.15\%$ , (Pb) at  $32.18 \pm 1.40\%$ , and (Sn) at  $18.81 \pm 1.61\%$ . Notably, the detected elements (Al, Si, P, Cl, K, Ca, and Ti) exhibited significant difference levels likely attributed to the burial environment.

Furthermore, two-dimensional elemental distribution maps of major and minor elements were generated using the four-channel SDDs. Subsequent washing of the coin with acetic acid resulted in a notable decrease in Pb and Sn concentrations, indicating their presence primarily within the surface patina layer. Additionally, the detected elements were compared with X-ray fluorescence analysis.

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Nº 25

## DEVELOPMENT OF A PLASTIC SCINTILLATOR-BASED PROTON DETECTOR FOR TIMING APPLICATIONS IN CNA'S NUCLEAR MICROPROBE

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The demand for advanced detection systems for charged particles in laser-based accelerators, as well as the need for time of flight (ToF) systems and for precise timing measurements, has driven the promotion of detectors based on ultra-thin plastic scintillators [1,2]. This work focuses on the development and characterization of a new family of detectors designed for ToF and timing applications, particularly as external trigger, within the microprobe line of the National Accelerator Center (CNA, Seville) [3]. This innovative detection system is based on the use of an ultra-thin plastic scintillator that is coupled to a photomultiplier tube (PMT). The active volume consists of a sheet of organic scintillator of the type Eljen EJ-214 [4]. As a result of the ionizing radiation passing through the material, photons are generated with a decay time of approximately 2 ns. These photons are directed towards the PMT via two semicylinders of polymethylmethacrylate light guides, while aluminized mylar sheets shield the assembly from external light. A linear manipulator, equipped with the new diagnostic at one end, has been designed to be placed into the beam line and intercept the ion beam.

In this work we present the installation and the characterization of the temporal performance of the prototype including the influence of the ion impact location on detector's response, the counting efficiency for protons of several MeV and the time resolution. In addition, the composition, thickness and uniformity of the plastic scintillator and the aluminized mylar shield have been determined by Rutherford Backscattering Spectrometry. The timing capabilities of this new system will facilitate the investigation of temporal processes carried out in our microbeam chamber, such as transport properties in semiconductor detectors.

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Nº 28

# DEVELOPMENT AND APPLICATION OF PROTON BEAM CELL IRRADIATION FOLLOWED BY SINGLE CELL LIPIDOMICS

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Development of proton beam cell irradiation followed by single cell lipidomics to explore lipid droplet formation and bystander effects

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Lipids are an essential part of a cell's biomolecular pool and play important roles in a myriad of complex biological processes with biophysical, energy storage and signaling functions. Recent studies have indicated that lipid droplets, which are storage organelles within a cell, are involved in drug resistance and stress resilience. However, the understanding of lipid accumulation and their involvement in radiation response remains incomplete.

Our group have developed methodology to measure lipid profiles in living single cells [1]. The method uses newly commercialised technology from the Yokogawa Corporation, in which incubated, living

cells are visualised under confocal microscopy. Single cells are chosen for sampling at a particular point in time into capillaries and analysed using mass spectrometry lipidomics to obtain single cell lipid profiles. We have recently shown how single cell lipid profiles respond to drug treatment and hydrogen peroxide exposure [2].

We have recently adapted a live cell chamber from National University of Singapore to enable live cell imaging and sampling post proton beam irradiation. Here we present the adapted chamber, present the outputs of single cell lipidomics and compare lipid profiles of cells irradiated at 6 Gy under FLASH (40 Gy/s) and conventional (1 Gy/s) dose rates, as well as their bystanders.

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N° 29

## ADVANTAGES OF USING SEGMENTED ANNULAR SILICON DRIFT DETECTOR ON ION MICROPROBE

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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 efficient experiments. Using a high solid angle, the required measuring time is significantly reduced under beam conditions, further reducing the sample's damage. To approach the solid angle above 0.5 sr, a segmented annular detector can be employed and has been installed at the ion microprobe end station of the Jožef Stefan Institute accelerator laboratory.

The Rococo 2 (PNDetector, Germany) detector consists of an SDD chip with a central hole of 1.8 mm and four crescent-shaped segments, mounted on a 45° angled mechanic. Each segment is protected by a Be window and pre-amplifier which provides a readout for each segment individually, achieving the 130 eV energy resolution [1].

The key advantage of using such a detector is in summing all segment's spectra to increase the statistics of measured data. This can be done in GeoPIXE II software [2]. This approach is beneficial when only a small ion dose can be applied to the sample, for example for biological samples. An example of this approach to data analysis from biological samples will be presented and discussed.

The segments' arrangement also allows the 3D reconstruction of the surface topography [3], for which a generalized approach to data analysis and 3D surface reconstruction has also been developed in our lab and will be presented.

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Nº 31

## RADIATION HARDNESS STUDY OF NEW 3D-DETECTORS FOR MICRODOSIMETRY IN HADRONTHERAPY

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Proton therapy (PT) is one of the treatments used against cancer. In order to be able to describe biological damage at the cellular level it is necessary to have sensors with a small radiation sensitive volume to make the necessary microdosimetric measurements. In response to this question, the Instituto de Microelectrónica de Barcelona (IMB-CNM) has designed and manufactured a new cylinder-shaped silicon sensors with a size comparable to that of human cell nuclei.

The first microdosimetry measurements on a low energy proton beam with therapeutic-equivalent fluence rates [1] were performed on the external line of the cyclotron of the National Accelerator Center (CNA) and served to validate the use of these sensors in PT. However, knowing the radiation resistance of these detectors to estimate their time of use is fundamental for their possible clinical application.

In this work we will present the controlled damage study performed with 1.17 MeV protons in the nuclear microprobe of the Tandem accelerator of the CNA. By means of a model [2], based on the shape of the energy spectrum to evaluate the radial dependence of the Charge Collection Efficiency (CCE), the evolution of the CCE as a function of proton fluence has been studied for proton fluences up to 70 kGy.

The pristine sensor presents a nearly perfect CCE profile and this behaviour has been observed up to doses about two orders of magnitude higher than those used in a clinical proton therapy treatment (~ 50 Gy), indicating that the lifetime of these sensors is long enough for this application. At higher doses, the transport properties of the microdosimeters show a progressive degradation.

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N° 32

# IBIC ANALYSIS OF A LINEAR POSITION SENSITIVE DETECTOR: MODEL AND EXPERIMENT

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The ion beam-induced charge analysis of a commercial silicon photodiode configured as a Position Sensitive Detector is the subject of this report.

Although the PSD is designed for detecting the position of incident light and optimized for use in the UV region, in this contribution, we present evidence that it also performs well as a detector for ions, with energies in the MeV range.

The device consists of a uniform p-type layer formed on a high-resistivity n-type semiconductor substrate, a pair of electrodes on both ends of the resistive layer, and a common electrode located on the backside of the substrate.

The IBIC experiment was carried out at the Laboratory for Ion Beam Interaction (LIBI) of the Ruder Boskovic Institute in Zagreb (HR), using 1 and 2 MeV proton microbeams raster scanning the 2.5x0.6 mm<sup>2</sup> active area of the PSD.

Each of the three electrodes was connected to an independent standard NIM charge-sensitive electronic chain and the induced charge pulses associated with the position of individual ions were then digitized using a multi-channel analyzer interfaced with the SPECTOR software.

The longitudinal CCE profiles acquired from the top electrodes show linear behaviors with opposite slopes, whereas the profile relevant to the signals from the back electrode is almost constant.

A model based on the IBIC theory satisfactorily interprets these results. It offers an alternative viewpoint to the commonly adopted Lateral Effect Photodiode principle and paves the way for the development of new PSDs for the identification of the impact position of a MeV ion with a resolution at the micrometer scale.

N° 36

## UTILIZING CNN FOR ION INCIDENT ANGLE DISCRIMINATION THROUGH EMISSION PHOTON IMAGING

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The utilization of Single-Event Effect (SEE) mapping has significantly enhanced our understanding of the underlying mechanisms governing SEEs[1]. To conduct studies on SEE mapping for modern advanced microelectronics using the high Linear Energy Transfer (LET) ions, an SEE mapping technique that leverages emission photon imaging has been developed in National Laboratory of Heavy Ion Research Facility in Lanzhou (NIHIRFL), achieving sub-micron spatial resolution[2].

However, ion scattering introduces notable uncertainty, making it challenging to accurately determine the location of ion inducing SEE in electronic devices. Therefore, precise knowledge of the ion incident angle is crucial for improving the spatial resolution in SEE mapping endeavors. In this study, we constructed a three-layer Convolutional Neural Network (CNN) using TensorFlow and trained it over 10 epochs to calculate the incident angle of Argon ions in a YAG scintillator. The preliminary results indicate that the classification accuracy of ion angles reaches 98% across 15 different incident angle groups, demonstrating the high reliability and promising potential of this method. Even though, further application of this study to the SEE mapping technique is still pending.

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N° 37

## MICRO-PIXE (PARTICLE INDUCED X-RAY EMISSION) FOR IMPROVING MEDICAL DIAGNOSTICS IN CASE OF ENDOPROSTHESIS FAILURES

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The improvements in living conditions and healthcare systems have contributed to increased life expectancy in developed countries. Among medical interventions, hip replacements have emerged as a widely used solution, with approximately one million procedures performed worldwide each year. This number is expected to grow due to increased life expectancy and ageing population. However, despite technological progress, there has been a concerning surge in endoprosthesis failures, attributed to factors such as increased demands and quality issues. The complex physiological processes underlying endoprosthesis failure necessitate the use of complementary diagnostic tools. While conventional diagnostic techniques like X-ray scans and optical tissue microscopies can identify metal particles, they often fail to discern their specific metallic origin (Ti, V, Al, etc.) and concentration. To address this gap, the TissueMaps project was launched, with the support of the Marie Skłodowska-Curie Actions, recognizing the need for better diagnostic tools.

Micro-PIXE has proved to be a valuable tool, facilitating the mapping of metallic particles released from degraded endoprostheses, helping to identify which endoprosthesis component is causing the failure, and to what extent. The Micro- beam end station available at Jožef Stefan Institute's 2MV tandem accelerator is especially suited for this type of research.

With a high-brightness ( $14 \text{ A m}^{-2} \text{ rad}^{-2} \text{ eV}^{-1}$ ) focused proton beam, allows the reduction of the object-slit aperture and acceptance angle, resulting in a reduced beam size. In addition, the high elemental sensitivity, with detection limit down to  $0.1 \mu\text{g/g}$ , and lateral resolution down to  $600 \text{ nm}$ , makes it especially suited for these types of experiments.

Building on this application, our research has extended to collaborations with other Hospitals, and just recently, we have applied this approach to identify stem cells previously labelled with magnetic nanoparticles within a collaboration with University of Ljubljana.

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N° 41

## DESIGN OF A 2-STAGE ACCELERATION LENS FOR A 100 KEV SINGLE-ION IMPLANTATION SYSTEM

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An array of a pair of a nitrogen and a vacancy (NV Center), which placed at intervals of several tens of nanometer, in a diamond is expected to be applications of quantum devices. An ion implantation system manufacturing the array by the ion irradiation has not been developed in the world. In QST, a 100 keV single-ion implantation system is developed by the combination of a Paul trapped laser cooling device (PTLC)<sup>1)</sup> and a two-stage acceleration system (2stageAccLens)<sup>2)</sup>, namely "SIS", which were independently developed as elemental technologies, respectively.

The 2stageAccLens is required to be newly developed in the SIS. The SIS needs the 100% irradiation probability, a long working distance of over 80 mm, and depths of several tens of nanometer in a diamond



sample. The reasons are that single ions generated by the PLTC are an extremely few per a second and that the observation devices of the quantum effects are placed in the long working distance. The former and the later reasons lead to a collimator less lens and a lens with a low demagnification, respectively. Therefore, the new 2stageAccLens focusing ion beam width of less than 20 nm without a collimator at 100 keV ion beam is required to meet the aforementioned conditions.

In this study, a 2stageAccLens was newly designed on the basis of the previously developed 2stageAccLens at QST. Ion beam trajectories were numerically simulated using electrostatic lens parameters obtained by a calculation code based on FEM and using the assumption of the condition of ion beams generated by PLTC. As a result, the new 2stageAccLens was successfully designed for SIS.

## Acknowledgments

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N° 42

# STUDY ON THE BIOLOGICAL EFFECTS AND GENOMIC VARIATION OF RICE IRRADIATED BY HIGH-ENERGY ARGON ION MICROBEAMS

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Physical mutation breeding is one of the important methods to obtain new plant germplasm resources. Using high-energy microbeams can realize high linear energy transfer (LET), and low-dose irradiations, which can provide basic data for the study of biological effects of cosmic particle radiation on plants. In this study, the embryo and endosperm of rice seeds were irradiated with 0.1k, 1k, 10k, and 100k of argon ion particles provided by the microbeam terminal (TRO) of Heavy Ion Research Facility in Lanzhou (HIRFL). The germination rate, plant height, antioxidant enzymes in the first generation after seed treatment ( $M_1$ ), and the genomic variation in the  $M_2$  generation were investigated. This study found that different numbers of particles and different irradiation sites of seed have different effects on rice growth, physiological responses, and mutations. For the  $M_1$  generation, whether the site of irradiation is the embryo or endosperm of the seed, 1k of argon ion radiation increased

the root length of the seedling. The height of the plant at maturity was significantly reduced only when the embryo of seeds was irradiated with 100k argon ions. The endosperm of seeds irradiated with 0.1k, 1k, and 10k argon ions increased the superoxide dismutase (SOD) activity of the seedlings; 0.1k argon ion-irradiated embryos, as well as 10k and 100k argon ion-irradiated endosperms, increased the peroxidase (POD) activity of the seedlings; and 1k, 100k argon ion- irradiated embryos, as well as 10k irradiated endosperms, increased the catalase (CAT) activity of the seedlings. The results of whole genome sequencing showed that: the site of irradiation was either the embryo or endosperm of the seed, and argon ion beam radiation above 0.1k could lead to mutations in the genome of its  $M_2$  generation plants. These results

demonstrate that irradiation of rice seeds with high-energy argon ion microbeams can effectively induce mutations.

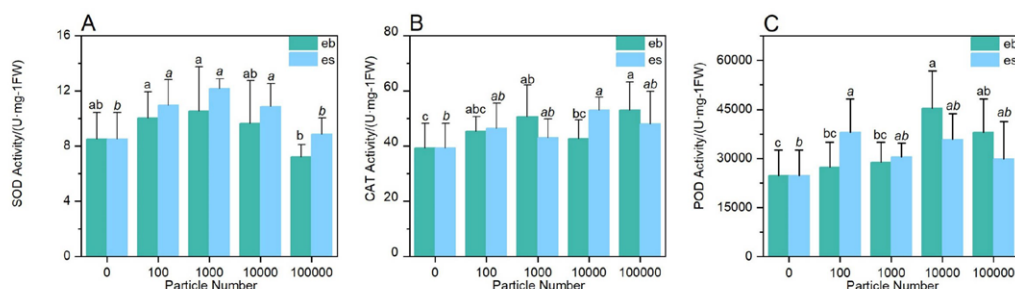


Figure 1. The effects of microbeam radiation on the activities of SOD, POD, and CAT in rice seedling.

Note: eb indicates that the irradiation site is the embryo, es indicates that the irradiation site is the endosperm; different letters indicate significant differences in the treatment of embryo-irradiation, and different italic letters indicate significant differences in the treatment of endosperm-irradiation ( $P < 0.05$ ).

N<sup>o</sup> 43

## DEVELOPMENT OF A COMPACT MICROBEAM SYSTEM

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High-energy microbeams are used in a wide range of fields. We have developed microbeam lines named MB-I and MB-II which could obtain beam spot sizes of less than 1  $\mu\text{m}$  and applied various fields [1]. Furthermore, to extend the microbeam application, we have developed MB-II AQUA, the simplified and including automatic focusing system, and has been used in a private company. These systems consist of quadrupole doublet lenses, object slits and divergence-defining slits. Higher demagnification factors of 40 and 10 for X- and Y-axes are achieved by extending object to lens distance of 6m. Therefore, the total length of the microbeam line is around 7 m, which causes a limitation for the installing to the existing accelerator laboratories.

In this study, we have developed a compact microbeam system with a total length of 5 m by modifying the MB-II. In this case, the demagnification factors are 25 and 6 for X- and Y-axes, respectively, and beam currents reduce to 1/3 to get similar beam sizes. Therefore, we decided to improve the demagnification factors by adding a quadrupole lens and used as a triplet mode. In the triplet configuration, the demagnification factors are 43 and 17 for X- and Y-axes, respectively. While the chromatic and spherical aberration factors are one of two order higher than that in the doublet configuration, the achievable beam spot size in the triplet mode was estimated ca. 0.5  $\mu\text{m}$  by the WinTrax code [2]. Beam spot size less than 1  $\mu\text{m}$  was obtained for X-axis, but could not be reduced to < 1  $\mu\text{m}$  for Y-axis, where chromatic and spherical aberration factors are large. The limitation might be affected by chromatic and spherical aberrations. We will survey the reason and try to achieve the design goal in the triplet mode.

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N° 45

## ANTARES MICROPROBE BEAMLINE FOR HIGH-PRECISION ION IRRADIATION OF MATERIALS AND DEVICES IN VACUUM AND IN AMBIENT FOR APPLICATIONS TO MICROELECTRONICS, PHOTOVOLTAICS, AND SPACE

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This work presents recent developments of ANSTO's ANTARES microprobe beamline for the high-precision irradiation of materials and devices for applications to microelectronics, photovoltaics, and space. The ANTARES microprobe ( $EM/q^2=120\text{MeVamu/e}^2$ ) [1] can produce focused beams of medium-heavy ions (1-3 MeV/u) and protons (1-12 MeV). To meet test requirements the new system offers: 1) the precision targeting in user preselected region of interest in a device; 2) three scanning modalities: i) rapid microbeam scanning, ii) slower, but larger in dimension, sample scanning and iii) hybrid-scanning combining the two [2]; 3) customized combination of the LET, energy, range, and flux; and 4) irradiation in vacuum or in ambient [3]. We show case studies for 1) the performance of d-CVD and SOI radiation detectors for microdosimetry [4], 2) the PEEK material radiation hardness [5], 3) the effectiveness of magnetic shielding against ions in LEO [6], 4) the radiation hardness of exposed solar cells [7], and 5) the SEU cross section validation in a reference SRAM chip [2].

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N° 46

## NEW DATA ACQUISITION SYSTEM FOR TOHOKU MICRO BEAM SYSTEM

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The Tohoku University has two microbeam systems, MB-I and MB-II, where various ion beam application experiments such as micro-PIXE analysis, STIM, and RBS analysis are performed [1],[2]. In these ion beam experiments, the signals from several detectors, such as the SiLi detector, germanium detector, and PIPS detector, were amplified and waveform-shaped before being digitally converted by an ADC and stored as list data. With the increasing accuracy of analysis in ion beam analysis, there is an increasing need for multimodal analysis, which combines different types of information from multiple detectors. For example, in the analysis of biological samples of non-uniform thickness, RBS, and STIM data can be measured in addition to the usual micro-PIXE data to simultaneously measure sample damage and thickness information and improve the accuracy of analysis. In such multimodal high-precision measurements, the number of detectors increases. The increase in the number of detectors also requires a data acquisition system that can withstand multi-channel and high rates. Therefore, a new high-speed, multi-channel data acquisition system incorporating a Digital Signal Processor (DSP) was developed. The use of a DSP allows the pre-amplification and waveform shaping circuits to be formed inside the DSP, making it possible to construct a compact, low-cost, multi-channel circuit. It can also store data for each signal waveform at a sampling rate of 100 MS/s.

Integral gating and timing analysis can then be performed offline on the accumulated waveforms. Furthermore, by configuring the DSP section to take data as digital inputs and outputs, the system can be easily integrated into accompanying systems such as the Compact-RIO beam scanner in our laboratory, making it suitable for actual analysis of plant samples and the like.

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N° 47

## CREATION OF NV CENTERS IN DIAMOND BY PROTON IMPLANTATION

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The nitrogen vacancy center in diamond is one of the most promising tools in quantum information theory and metrology. The reason for this is that this center has an exceptionally long spin lifetime and at the same time a high degree of optical stability. Because of these properties, numerous measurements that can be performed with the magnetic resonance method have also become available by optical techniques.

The nitrogen vacancy center in diamond can traditionally be produced by thermal annealing following electron or neutron irradiation. In our present collaboration, we have created these centers by ion irradiation. When using focused MeV ion beams the NV center positions can be deterministic. The possibility of localized exploitation offers promising properties of nitrogen vacancies, such as metrology at the microscopic level.

To manipulate the nitrogen vacancy centers, conductive structures placed close to the sample are used (bent copper wire, antenna, coplanar waveguide). At the same time, amorphous carbon structures with good conductivity can be created in the diamond lattice using high-energy ion beams.

The irradiated diamond sample was  $\langle 1,0,0 \rangle$  oriented, 3mm x 3mm x 0.3mm in size. Channeling effect was avoided by suitably rotating the sample under the beam axis. The deposited dose was in the range of  $1e14$ - $1e16$  ions/cm<sup>2</sup>. Lines of the full sample width (3mm) were drawn with a thickness of 20-50  $\mu$ m, gaps were 50-100  $\mu$ m in between the lines. The implantations were followed by thermal annealing, which created the NV centers in a well-defined localization. We have performed magnetic resonance and optical investigations of these NV centers, taking advantage of the manipulation possibilities provided by the conducting channels.

N° 48

# THE LINAEUS CODE SUITE FOR MAJOR ELEMENT ANALYSIS OF BIOLOGICAL TISSUE SECTIONS

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Biological life forms exhibit an astonishing degree of variation with the remarkable verity that they are mostly made up of C, H, N and O. Ion microprobes are very capable tools to image lesser and trace element distributions in biological tissue sections on a  $\mu\text{m}$  scale [1]. Knowledge of the matrix composition and mean thickness is needed to quantify these elements in the ex-vivo state. Simultaneous Elastic Backscattering Spectrometry (EBS) and Off-axis Scanning transmission Ion Microscopy (OA-STIM) has been used to quantify the major element composition concurrently with Particle Induced X-ray Emission (PIXE) quantification of lesser and trace elements [2].

A new Python code suite, LINear Analysis of Elements Using Spectroscopies (LINAEUS) has been developed to facilitate major element quantification from EBS and OA-STIM data. The code is based on linear fitting using the Dynamic Analysis method to determine the surface edge heights corresponding to C, N and O edges in the EBS spectra on a pixel by pixel basis. The OA-STIM signals for H cannot be linearised in this way [3]. Instead, a pseudo-linearised H surface-edge height is assigned for each pixel. (This converges to the mean value when averaged over many pixels.) Quantification is based on using a thin hydrocarbon reference standard and the DPASS stopping cross section data. Corrections for the film supporting the tissue sections and the effects of void areas in the tissue section are included.

The suite is implemented as companion code to GeoPIXE, which is used for PIXE analysis and to define the different tissue regions. The different codes in LINAEUS suite interact via a file structure that makes for ease of use by requiring only a single non-linear fit and set of calibration /reference data can be set-up for a measurement session comprising many measurements.

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N° 51

## TISSUE-SPECIFIC ELEMENT PROFILES IN EDIBLE SEEDS

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Due to rapid population growth and increasing effects of global climate change, optimising nutritional yield of existing farmland is paramount. This global objective can be approached from various angles, one of which is through seeds, which are one of the pillars of day to day nutrition in cultures all over the world, and through biofortification to optimise the nutritional profile of a chosen seed. Seeds differ in their element profiles and their distribution can importantly influence the success of biofortification and influence choices in food processing to achieve the ideal dietary outcome. Seeds of six plants were selected based on their small size, taxonomic diversity, similar utility and nourishment potential for humans: flax (*Linum usitatissimum*), chia (*Salvia hispanica*), poppy (*Papaver somniferum*), caraway (*Carum carvi*), oilseed rape (*Brassica napus*), and quinoa (*Chenopodium quinoa*). Seed tissues were captured and identified on their transverse cross-sections using bright field and autofluorescence microscopy and concentrations of Ca, P, K, S, Fe, Mn, Zn and Cl in those tissues were determined using micro-PIXE at Jožef Stefan Institute. P/S, P/Zn and Fe/Mn ratios and a Mineral Safety Index were calculated for each seed and element profiles across the primary tissues of all seeds were compared. Finally, the element profiles of seeds were compared to their taxonomic relations and distances to determine possible evolutionary patterns while also comparing the same data to other known seeds and grains of global nutritional importance.

N° 56

## MIXED MEASUREMENT OF THE STOPPING FORCE OF $^{12}\text{C}$ , $^{28}\text{Si}$ AND $^{59}\text{Co}$ IONS THROUGH METAL FOILS BY TIME OF FLIGHT SPECTROMETRY: CHARGE EXCHANGE CONSIDERATIONS

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The stopping force of energetic ions in matter is of importance in many aspects of materials research and development using ion beams. Common applications include ion implantation, ion beam modification of materials and ion beam analysis. In many instances stopping force data is obtained from semi-empirical and theoretical formulations. While the accuracy of these data sources is now generally acceptable for light ions (H, He), more work still needs to be done to improve their predictive accuracy for heavier ions ( $Z > 6$ ). Heavy ion-matter interaction parameters that are not so well described by theory include charge exchange effects among others. In this presentation we describe a simple

experimental method that may be used to account for charge-exchange effects in the energy loss rate of carbon ions through  $^{96}\text{Mo}$  and  $^{195}\text{Pt}$  foils.

The measurement was carried out in a Time of Flight – Elastic Recoil Detection Analysis (ToF-ERDA). A 40 MeV  $^{197}\text{Au}^{9+}$  beam was used to recoil  $^{12}\text{C}$ ,  $^{28}\text{Si}$  and  $^{59}\text{Co}$  ions from a thick carbon, silicon oxide and cobalt substrate towards the stopper foil. The energy loss of the incident recoils through the stopper foil was calculated from the measured ToF across a fixed path length, with and without the stopper foil, and the stopping force then calculated using the measured foil thickness [1]. The thickness of the used target was measured using 3.6 MeV  $\text{Li}^{3+}$  Rutherford Backscattering Spectrometry (RBS). Given the simultaneity of the measurements for both hydrogen and carbon recoils, the observed relative deviations between experiment and theory in the stopping force of these ions can be attributed to charge exchange effects.

Nº 59

## UNVEILING NEW HORIZONS: PROTON THERAPY'S POTENTIAL IN TACKLING NEURODEGENERATIVE DISORDERS

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Ionizing radiation is widely employed for medical purposes, encompassing both diagnostic and therapeutic applications. Radiotherapy (RT) is routinely employed in cancer treatment, and has demonstrated efficacy in addressing extra-cranial amyloidosis. Current evidence suggests its potential as a promising treatment for amyloid-associated neurodegenerative disorders such as Alzheimer's, and Huntington's diseases. Furthermore, emerging modalities of RT could enhance biological effects while mitigating potential toxicity.

Proton therapy stands out as one of the most effective RT techniques, due to the considerable clinical advantages of protons over conventional RT particles (photons or electrons). These advantages include a favorable depth dose distribution, reduced lateral spread, and minimal scatter, facilitating a decrease in collateral damage. While it is currently undergoing testing in cancer settings, its application in the context of neurodegenerative disorders remains largely unexplored.

In our multidisciplinary research, RT is being investigated as a potential treatment for neurodegeneration, with the capability to disassemble amyloid structures through the disruption of chemical bonds or by triggering cellular degradation mechanisms. We aim to simulate different radiation modalities using Monte Carlo tools and experimentally validate their effects on those abnormal protein deposits. Preliminary gamma-irradiation experiments conducted on cell lines expressing neurodegenerative



disease-associated proteins, demonstrated a decrease in the expression and aggregation of the pathological proteins, which was proportional to the applied dose. Subsequently, we progressed to irradiating biological samples with photons and electrons using a clinical linear accelerator at a medical facility, and the results evidence the same pattern of expression and aggregation decrease.

In this talk, we will showcase the results of the irradiations already performed. Our ongoing research is dedicated to laying the groundwork for the expansion of proton therapy applications beyond cancer. This expansion aims to amplify the adaptability of emerging proton therapy facilities and potentially transform the course of development for presently incurable neurodegenerative disorders.

Nº 63

## LI MAPPING AT CMAM INTERNAL MICROBEAM LINE FOR THE DEVELOPMENT OF SOLID-STATE LI-ION BATTERIES

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Lithium-ion batteries (LIBs) stand as pivotal components in the arsenal of energy storage technologies, playing a crucial role in the transition to a greener economy and combating global warming. Understanding the intricate Li dynamics within LIB components is imperative for improving their efficiency and durability. In this study, we introduce a pioneering approach to LIB analysis, harnessing ion beam techniques with microscale lateral resolution. For the first time, the generation of Li elemental maps is explored within the CMAM internal microbeam line [1] using the  $7\text{Li}(p, \alpha)4\text{He}$  nuclear reaction. A 3 MeV proton beam with a current higher than 1 nA and a resolution of 2  $\mu\text{m}$  were employed. To explore the sensitivity of this nuclear reaction with a focused proton beam, a testing device composed of Li/LIPON thin films was utilized. Additionally, simultaneous analysis with PIXE technique was performed to determine possible trace elements. This innovative approach represents an expansion of our capabilities for studying fundamental processes occurring in LIBs within the context of the OPERA Horizon Project [3] and opens up possibilities for conducting further analysis in the CMAM internal microbeam line of NMC811 ( $\text{LiNi}_{0.8}\text{Mn}_{0.1}\text{Co}_{0.1}\text{O}_2$ ) and LCO/LFP ( $\text{LiCoO}_2/\text{FeLiO}_4\text{P}$ ) cathodes, whose Li-depth profile was previously analyzed at the CMAM standard line.

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Nº 64

## CANCER WEAPONS AGAINST NEURODEGENERATION

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Proton Radiation Therapy (PRT) is a powerful anticancer tool, being the hadrontherapy modality with the strongest growth over the last decades. PRT has the unique capability to confine high doses in small and deep space regions due to its inverse dose deposition profile. While the reduction of dose in healthy tissue is significant in PRT, its Relative Biological Effectiveness (RBE) is comparable to that of other Radiation Therapy (RT) modalities [1]. Innovative approaches have recently enhanced its RBE, adding boron in low concentrations to the body and activating it through proton-induced reactions [2]. Low-dose RT has a hormetic nature, and it produces a wide variety of non-toxic biological effects that have therapeutic potential beyond killing cancer cells. For example, low-dose RT has been successfully applied to treat peripheral protein aggregation diseases known as amyloidosis, which resemble widespread neurodegenerative disorders such as Alzheimer's (AD), Parkinson's (PD), or Huntington's disease (HD) [3]. Simulation and experimental preliminary results with conventional RT sources indicate that radiation could prevent the formation of amyloid structures. However, the therapeutic potential of both conventional and proton RT has been barely explored in this context. In this contribution, I will present our goal of using PRT techniques to combat neurodegenerative disorders. By directing our focus towards toxic amyloids, we plan to utilize boron- and fluorine- based compounds in conjunction with low-dose PRT [4-6]. Specifically, our strategy involves irradiating amyloid plaques with a targeted proton beam at CMAM, leveraging Bragg's peak region for optimal effectiveness.

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Nº 67

## FOCUSED PROTON BEAMS FOR RADIOBIOLOGY AND DOSIMETRY STUDIES OF SINGLE CELLS

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Proton therapy stands out as a promising alternative in cancer treatment due to its capacity to minimize the integral dose received by patients. Yet, there is a need to improve its efficacy, both in terms of dose delivery and cellular response.

In this study, the CTN/IST 2.5 MV Van de Graaff accelerator served as the proton source for irradiating live cells to elucidate cell response to radiation, primarily focusing on DNA damage. A breast cancer cell model was used in the study. A specialised irradiation stage was mounted on the nuclear microprobe ensuring precise cell positioning and measurement of experimental physical parameters. A focused proton beam raster scanned over a monolayer of breast cancer cells delineated the irradiation area.

Experimental procedures include measurement of irradiated area, proton flux, particle attenuation through various materials, dose-response curves and calculation of cell doses across varying linear energy transfer (LET) along the Bragg peak curve. These tasks are facilitated by employing Gafchromic films to establish a dose-response curve using  $^{60}\text{Co}$  gamma-rays as reference radiation for water-equivalent material and Monte Carlo (MC) modelling (specifically TOPAS n-Bio). Breast cancer cells response post-proton irradiation centred on early DNA damage assessment using  $\gamma\text{-H2AX}$ . Quantification involved analysing the increase in foci within irradiated nuclei compared to unirradiated counterparts. Finally, DNA damage data integration into MC simulations together with a realistic three-dimensional cell model aimed to obtain micro- and nanodosimetry models. This comprehensive approach seeks to correlate micro- and nanodosimetric distributions with observed bio-effects, thereby enhancing

our understanding of the fundamental processes governing cellular response under different irradiation conditions.

This work was developed as part of the International Atomic Energy Agency Coordinated Research Project F11024, 'Sub- cellular imaging and irradiation using accelerator-based techniques' (SCIMIRAC).

Nº 69

## MATCHING NUCLEAR MICROSCOPY IMAGING OF METAL-BASED COMPOUNDS IN SINGLE CELLS: PEARLS AND PITFALLS

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Imaging techniques, such as STIM, PIXE, and RBS, have revolutionized the visualization of metal-based compounds (e.g., metal complexes and nanoparticles) within cellular environments. These techniques offer unparalleled capabilities to investigate preferential localization and quantify uptake at the subcellular level. It is possible to look for correlations between major cell compartments, and the concentration of metals by matching the different sorts of images and post- processing of data, gaining insights into cellular uptake. However, the reliability of results depends on methodological considerations due to inherent challenges at every step. Key factors influencing outcomes include substrate choice, compound characteristics and stability in culture media, compound concentration and incubation time, washing procedure without disrupting cell adherence, sample fixation, and optimal ion beam conditions to minimize radiation-induced damage.

Extensive research has sharpened our understanding of ideal substrates, sample handling, and irradiation conditions. Silicon membranes, with their adherence properties and resistance to radiation, have emerged as an optimal substrate for nuclear microscopy analysis. Under routine irradiation conditions, cellular integrity can typically be maintained using a proton beam with dimensions of a few micrometres and a current not exceeding 200 pA. Despite advancements, potential artifacts stemming from compound stability in culture media, uptake kinetics and washing procedure persist. These artifacts, can skew uptake assessments and compromise data integrity. Careful consideration and mitigation of these factors are imperative for accurate cellular uptake evaluations.

We provide comprehensive insights into qualitative and quantitative studies utilizing nuclear microscopy techniques. Emphasizing significant findings, we underscore the importance of avoiding potential pitfalls to ensure robust and reliable data interpretation in single-cell analysis.

Nº 71

## SIDE-EFFECTS OF CELLULOSE BASED MATERIALS INDUCED BY ION BEAM MEASUREMENTS

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The use of Ion beam analysis (IBA) for cultural heritage characterization is an established method to determine the concentration and the distribution of elements. Usually considered as non destructive techniques, recent concerns have emerged regarding potential side-effects on the analyzed materials. These effects, even when not immediately visible, may become apparent over time, thereby posing a risk to the integrity of these artefacts.

To investigate the possible occurrence of side-effects on cellulose-based materials induced by a proton beam, several samples were irradiated in open air conditions applying different fluences (given as deposited charge per unit area) both within and beyond the usual analytical practice: 0.1, 1 and 10  $\mu\text{C}/\text{cm}^2$ .

To comprehensively assess any side-effects, a multi-analytical approach was followed, which included colorimetry, X-ray Diffraction (XRD), Attenuated Total Reflection Fourier Transform Infrared Spectroscopy (FTIR-ATR), Thermogravimetric Analysis (TGA), and Differential Scanning Calorimetry (DSC).

To cover part of the material variability within of the historic objects, several types of cellulose-based materials have been tested, including Whatman filter paper, used as reference sample.

Preliminary results revealed that, while visible colour changes were detected (also quantified through colorimetry), structural characterization techniques such as XRD did not indicate alterations. Comparative analysis across all employed techniques will be presented to establish the potential origins of the visible damage observed in some cases.

This investigation underscores the critical balance between the use of IBA techniques for CH artefacts characterization and the imperative preservation of these materials for future generations. By identifying and understanding the potential side-effects of such analytical techniques, we can therefore refine our methodologies to ensure such balance.

N° 73

# INVESTIGATION OF ION MICROPROBE CAPABILITIES FOR MEV IMPLANTATION IN SINGLE CRYSTAL CVD DIAMOND

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Due to growing interest in quantum computing and sensing, there is an increase in research activity regarding color centers in various materials such as synthetic diamond, silicon, and silicon carbide.<sup>1,2</sup> To fabricate these color centers, defects need to be introduced in the substrate so that they form various complexes with interstitial atoms or lattice vacancies. That is why a large effort in the research community is being made to improve ion implantation in very pure (defect concentration <5 ppb) diamonds to be able to deterministically create single color centers in a certain well-defined volume which can then be used for quantum computing. Another approach is to use CVD diamonds which have an abundance of non-intrinsic defects incorporated during growth.<sup>3</sup>

In this work we present the capabilities of the ion microprobe located at the Ruđer Bošković Institute accelerator facility for ion implantation of MeV ions in diamond. The ion microprobe is coupled to two electrostatic accelerators (1 MV Tandetron & 6 MV tandem Van de Graaff accelerator) offering a wide range of ion species and energies. The focused beam can be as small as 200 nm under favorable conditions (few fA current) and then raster scanned over an area as large as 1x1 mm<sup>2</sup>. To show these capabilities, N-rich (<200 ppm) sc-CVD diamonds made by E6 were implanted with carbon ions to explore the efficiency and quality of NV centers produced without post-irradiation annealing treatment. Another experiment we made was co-implantation of Ge and O ions in electronic grade diamond from E6 on room and elevated temperatures to explore the creation yield of GeV centers under different thermal conditions.

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N° 80

# FABRICATION OF PICOWELL ARRAYS WITH MICROBEAMS OF SWIFT HEAVY IONS IN A CYCLIC OLEFIN COPOLYMER

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A method for the fabrication of picowell arrays (microwell arrays of individual well volumes below 1 picoliter) by implantation with microwells of swift heavy ions in polymer substrate was devised and demonstrated. It was found that implantation with microbeams of swift heavy ions in a cyclic olefin copolymer produced considerable swelling in the implanted surface regions. Thus toroidal structures could be fabricated on the sample surface by implanting an annulus of adequate diameter and width. The target material was ZEONOR copolymer. Microbeams of 10.5 MeV  $N^{4+}$  and 10.5 MeV  $C^{4+}$  ions were used in the experiments. Microbeam sizes were between 2 and 3 micron. Beam current was in the 16–800 pA range, and fluence was between  $10^{12}$ – $10^{13}$  ion/cm<sup>2</sup>. Picowells of various internal/external diameters were written from 14/16 to 40/42 microns. The best results were obtained by a 10.5 MeV  $N^{4+}$  microbeam, in the form of a 9 x 9 picowell array. Diameters of the individual picowells were 20/22 microns, their depth was 4.8 micron, and they comprised a volume of 820 femtoliter [1]. The results of the experiment series showed that picowell depth was proportional to the implanted fluence in the fluence range used. Very similar results were obtained by carbon and nitrogen ions. The crucial fabrical parameter proved to be the current density or flux of the implanted ions. At the same energy and fluence (and similar beam size), much higher picowell depth were obtained at a current density of 90 A/m<sup>2</sup> (800 pA) than at 20 A/m<sup>2</sup> (125 pA).

The proposed method could be an alternative to the standard ones for fabricating picowell arrays, due to its simplicity, fast production time, and flexibility.

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N° 85

# DEVELOPMENT OF MICRO-OPTICS FOR SPECTROSCOPIC IMAGING OF ION BEAM-INDUCED LUMINESCENCE/FLUORESCENCE (IBIL/IBIF) ANALYSIS USING FOCUSED PROTON MICROBEAM

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Ion beam-induced luminescence (IBIL)/fluorescence (IBIF) analysis techniques have been developed as analytical methods that can be used simultaneously with particle-induced X-ray emission (PIXE) analysis, which is a method for the analysis of trace element compositional distributions. The information available from IBIL fluorescence is diverse and correlatable with the chemical morphology and biochemistry of the sample under consideration[1-3]. To reveal such correlations, two spectral mapping systems for IBIL [4,5] and IBIF [6] analysis were developed. Both systems use CCD-based spectrometers and photon-counting detectors. By improving the detection efficiency of the optics, further research and development can be achieved and combined to accomplish IBIL/IBIF analysis utilizing microprobe. The development of a dedicated microscopic optical system and the establishment of an experimental system to enable analysis with the desired ion microbeam were the objectives of this study.

In this study, a new IBIL/IBIF analyzing system was constructed using the Dynamitron accelerator microbeam line at Tohoku University. The newly designed optical system enables a fast detection capability of IBIF due to the fast response of the photon counter monochromator with a multi-pixel photon counter (MPPC) array and an improved signal-to-noise ratio (SNR) of the imaging capability due to the detection of fluorescence caused by the proton microprobe. The ability to perform spectral mapping using particulate phosphors was evaluated. These detailed results will be presented in the presentation.

## Acknowledgments

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Nº 86

## COLLECTING MICROPLASTICS BY DIELECTROPHORESIS WITH MICROSTRUCTURES FABRICATED USING PROTON BEAM WRITING

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Microplastics (MPs), typically smaller than 5 mm, have been reported to be significant concerns in the global marine environments. Specifically, MPs with sizes smaller than 300 micrometers, so-called super microplastics (SMPs), cannot be collected by conventional sampling techniques such as the Neuston Net in oceans. Therefore, the SMPs could negatively affect marine biological systems.

Dielectrophoresis (DEP) is an electrokinetic phenomenon acting on polarized particles such as the SMPs in an inhomogeneous electric field. Insulator-based DEP utilizes dielectric microstructures under alternating current electric fields, in which we can manipulate the SMPs using the DEP forces. We have previously reported the applications of proton beam writing (PBW) to fabricate the DEP micro filters equipped with microstructures in SU-8 [1] and PMMA [2]. The PBW is a direct-write technique suitable for prototyping microstructures with high-aspect-ratio and vertical features.

In this study, we applied the insulator-based DEP technique to collect polystyrene (PS) microparticles as a model of SMPs in environments. We will discuss the effects of the DEP parameters, such as applied electric field frequencies, on the collecting efficiency of different PS sizes.

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N° 87

## THE ACCURATE DOSE DELIVERY SYSTEM FOR PROTON THERAPY RESEARCH THROUGH PRECISE CONTROL OF RBS/STIM RATIO IN THE CENTRE FOR ION BEAM APPLICATIONS IN NUS, SINGAPORE

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The Centre for Ion Beam Applications (CIBA) is a multidisciplinary research centre based in the Dept of Physics, NUS, specialising in the development of nuclear particle technology and subsequent applications of MeV ions (proton, helium nuclei etc).

One of the unique capabilities developed by CIBA is the ability to focus and target protons down to 10nm spot sizes. This unique performance characteristic of our system has been applied in the following research program: 1) Investigation into the targeted damage response mechanisms of single cells, 2) Low dose radiation research on multicellular cultures, 3) Comparison between targeted low dose ion radiation and photon radiation.

In order to deliver accurate proton doses to various samples for the above mentioned research programs, proton dose calibration is the utmost important step. Two Ion Beam Analysis techniques namely Rutherford Backscattering spectrometry (RBS) and Scanning Transmission Ion Microscopy (STIM) have been used. Due to the rare events of back scattered particle when ions are interacting with matter, the current of incoming proton beam is controlled to be less than 0.01pA for precise calibration. Then RBS and STIM are carried out simultaneously for the dose calibration prior to the delivery of the required doses to the samples.

This work will focus on the calibration method we have been using to govern that the right proton doses are delivered to the right samples.

N° 90

## MEASUREMENTS OF MEV-SIMS DAMAGE CROSS SECTION AS A FUNCTION OF PRIMARY ION ENERGY ON BIOLOGICAL MOLECULES OF VARIOUS SIZES

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Focused fast heavy ions can be utilized for desorption and analysis of secondary ions, a method known as MeV-SIMS [1]. Due to the nature of the desorption process, MeV-SIMS can analyze heavy organic molecules with high efficiency, which requires low primary ion fluences on the sample.

Within the present work, we investigated the so-called damage cross section, i.e., area of significant chemical alteration on the surface of the sample, caused by an individual primary ion, as a function of primary ion energy. Precise monitoring of the primary ion current was achieved by utilization of low-current primary ion beam, so the measurements were only done with pulsed primary ions on a small area. Four chlorine primary ion beams with energies between 3 and 10 MeV were used during measurements, and their profiles were analyzed by a Channel Electron Multiplier (CEM) detector, positioned on the beam axis. Analyses were performed on organic molecules of various sizes, from glycine ( $m/z = 75.0$ ) to polyethylene glycol (PEG) 2000. Samples were bombarded with fluences up to  $10^{14}$  primary ions/cm<sup>2</sup>.

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N° 92

## MEASUREMENT AND 3-D RECONSTRUCTION OF DOPED W ELEMENT CONTENT IN HDC TARGET PELLETS BY ION BEAM ANALYSIS METHOD

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Inertial confinement fusion (ICF) is a promising way to achieve controlled thermonuclear fusion and provide clean energy. The content and distribution of the doped elements in the pellets have an important effect on the performance of the pellets, which plays a crucial role in the research of ICF serving as the thermonuclear fuel vessel. This article used Rutherford backscattering spectrometry (RBS) and particle induced X-ray emission (PIXE) ion beam analysis techniques to determine the content of doped W element in high density carbon (HDC) planar samples and pellet samples, and developed a three-dimensional reconstruction method RBS-T by combining RBS method with nuclear microprobe

scanning system. The experiments showed that the distribution of W element in the planar sample was significantly uneven, with the W element content in the high concentration area about three times that in the low concentration area, while the W element distribution in the pellet sample was relatively uniform, with a content of about 0.31 at.%. Based on the RBS-T method, we reconstructed the three-dimensional distribution of W element in the sample and visually displayed it in the form of images. Compared with PIXE-T method, RBS-T method contains the depth distribution information of elements, avoiding multiple rotation of sample. Furthermore, the RBS-T method does not require the ion beam to penetrate the sample completely, so it can be applied to samples with larger volumes or with the presence of supporting cores, providing another practical technology for three-dimensional reconstruction of elements.

N° 94

## ELEMENTAL DISTRIBUTION IN KIDNEY OF RATS EXPOSED TO RENAL TUBULAR INVASION HEAVY METALS BY MICRO-PIXE

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The kidney is an important organ for the uptake and excretion of heavy metals from the body, and the renal proximal tubules are responsible for reabsorption of substances from the urine. The proximal tubules are divided into three regions but the region-specific elemental distribution has not been clearly understood. In the present study, the precise distribution of endogenous elements in kidney was examined in mouse exposed to renal tubular invasion heavy metals, such as platinum or cadmium, by micro-PIXE (particle induced X-ray emission) analysis.

Renal cryo-sections (thickness, 10  $\mu\text{m}$ ) were subjected for micro-PIXE analysis. Areas of concentrated phosphorus and potassium were observed in the outer stripes of the outer medulla, where the proximal tubules in the distal region were distributed, after cadmium exposure, but not platinum. The formation of phosphorus and potassium concentrations was similar to our previous study of uranium, a metal that invades the proximal tubules in the distal region. The dynamics of platinum and cadmium distribution was also examined by SR-XRF (X-ray fluorescence spectrometry using high energy synchrotron radiation) using serial sections of micro-PIXE specimens. The correspondence between phosphorus and potassium concentrations and the distribution of the heavy metals will also be discussed.

N° 95

## COMBINATION OF MICRO-PIXE AND SR-XRF FOR ELEMENTAL DISTRIBUTION OF CESIUM AND BIOLOGICAL ELEMENTS IN THE SMALL INTESTINE OF MOUSE EXPOSED TO CESIUM CHLORIDE.

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Micro-PIXE is good at detecting light elements and is suitable for elemental distribution analysis of biological tissues. SR-XRF, on the other hand, is superior in detecting heavy metals. The small intestine is an important organ responsible for the absorption of orally ingested substances. In this study, intestinal elemental distribution was examined in mouse exposed to cesium by combination of micro-PIXE (particle induced X-ray emission) analysis for endogenous phosphorus, sulfur, and potassium and SR-XRF (synchrotron radiation X-ray fluorescence) analysis using high-energy synchrotron radiation for cesium and endogenous trace elements, such as iron, zinc, and rubidium.

Cesium chloride was administered orally (50 mg/kg) to mice and the small intestine was collected. Cryo-sections (thickness, 10  $\mu\text{m}$ ) covering from the basement membrane to the parenchyma placed on polypropylene films were subjected for micro-PIXE and SR-XRF analysis. The serial sections were stained with Hematoxylin-Eosin to understand the intestinal structure.

Firstly, SR-XRF was performed at an irradiation energy of 37 keV, followed by micro-PIXE of the same specimen.

Cesium was observed to be more abundant in the villi than in the basal area, with a trend of decreasing from the center to the outside in the villi. Potassium, the same alkali metal, also showed similar distribution trends, with higher villi than basal. On the other hand, iron was distributed linearly along the center line of the villi. The combination of micro-PIXE and SR-XRF imaging was useful for analyzing the distribution of elements in biological samples because it can analyze the distribution of elements in the low- and high-energy regions.

N° 98

## CHARACTERIZATION OF ICF TARGET USING NUCLEAR MICROPROBE

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The Inertial Confinement Fusion (ICF) is investigating the conditions to achieve controlled thermonuclear fusion. The surface of the target is ionized by laser in a very short time, and plasma is generated at the periphery of the target. The plasma expands and scatters. When a huge centripetal pressure is generated, the fuel in the target undergoes a fusion reaction under extremely high temperature and density conditions. Thus, ICF target fabrication quality is as vital as driving laser to ensure that energy deposition from the lasers results in uniform compression. Therefore Targets must be characterized for size, concentricity, surface roughness and distribution of dopant concentration, layer thickness and fuel pressure as well. An ideal analysis method would be able to obtain such information simultaneously and non-destructively.

Nuclear Microprobe (NMP) could be the one. With the ability to focus MeV ion beams down to micron spot sizes in Fudan University, NMP with particle-induced X-ray Emission (PIXE), Rutherford backscattering (RBS), Scanning Transmission ion microscopy (STIM), Elastic Recoil Detection Analysis (ERDA) and P-P scattering enables us to map trace elements of target, and Hydrogen isotopes in the target. Meanwhile, the optimal algorithm for characterizing the three dimensional distribution of doped elements in the target are also discussed.

N° 99

## MICROANALYSIS OF THE EFFECT OF P-DIFFUSION GETTERING ON UNCONVENTIONAL SI SUBSTRATES: COMPOSITIONAL ANALYSIS OF IMPROVED OPTOELECTRONIC MATERIAL

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In this work, the distribution of impurities on unconventional silicon substrates is studied through microprobe techniques. Si purified for photovoltaic applications by alternative methods, like upgraded metallurgical grade (UMG) feedstock, is expected to host significant concentrations of undesired foreign species. In order to obtain carrier transport parameters compatible with solar cell manufacturing, a Phosphorus Diffusion Gettering (PDG) process has proven effective in improving the optoelectronic

properties of Si wafers through the driving of diluted impurities to a diffused layer on the surface of the sample, which is then etched away. However, the nature and spatial segregation of impurities in the diffused layer is not well understood and the use of  $\mu$ PIXE (Proton-Induced X-ray Emission) measurements may allow valuable insight.

$\mu$ PIXE and  $\mu$ RBS (Rutherford Back-Scattering) analysis were conducted at CMAM internal microbeam line. The presence of metallic (mainly Fe, Ti, Zn, Ca, K) and non-metallic (O, Cl) impurities is detected within the diffused layer in non-etched, gettered UMG-Si samples, as to raise the impurity concentration over the detection limit. Through spatial distribution mapping,  $\mu$ PIXE measurements indicate that impurities may appear either uniformly distributed or in the form of clusters in the diffusion layer. Results are consistent with those obtained by RBS using a defocused ion beam.

Optical conductivity measurements indicate that after the PDG, the effective carrier lifetimes improve up to two orders of magnitude, correlating with the extraction of impurities from the sample's bulk. Similarly, carrier mobility values in UMG-Si, probed through Time-Resolved Terahertz Spectroscopy (TRTS) show an almost three-fold improvement after the PDG process, from  $283 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$  up to  $726 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ , with values matching those obtained from high-quality FZ-Si reference samples, thereby demonstrating the effectiveness of the PDG process as a way to improve charge carrier transport through impurity removal.

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Nº 103

## CHARACTERISATION OF $\beta$ -GA<sub>2</sub>O<sub>3</sub> ION-BEAM-EXFOLIATED MEMBRANE DEVICES USING A PROTON $\mu$ -PROBE

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Profiting from its wide bandgap ( $\sim 4.8 \text{ eV}$  at room temperature) and high breakdown electric field ( $\sim 8 \text{ MV/cm}$ ),  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> is one of the most promising semiconductors for future applications such as high power or transparent electronics. Moreover, it is compatible with conventional melt growth methods, which allows the fabrication of large, high-quality single crystals at a low cost [1].

Several works have demonstrated the potential for the easy mechanical exfoliation of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> along its (100) plane to develop different types of devices based on thin flakes [2]. Notably, it has recently been observed that it is possible to produce  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> microtubes by ion implantation into (100)-oriented single-crystals under specific conditions due to the induced strain profile. Upon thermal annealing, the strains are relaxed, promoting the unrolling of the microtubes and leading to the formation of a nanomembrane with bulk-like crystalline quality. Therefore, this fabrication method offers an enhanced control of parameters such as the membrane thickness or their optical, magnetic or electrical properties, which can be reproducibly tailored to the envisaged application [3].

In this work, multiple metal-semiconductor-metal (MSM) in-plane structures were fabricated, on top of Si/SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> substrates, using  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> membranes obtained by the previously mentioned technique, making electrical contacts with Ti/Au deposited on both ends. These devices had their photoconductive response characterised by I-V sweeps and transient I-t, with above and below bandgap excitation, to learn about the nature of the contacts and to test these devices as solar-blind UV photodetectors. These results were complemented with IBA techniques, using a nuclear  $\mu$ -probe, namely PIXE, RBS/EBS and IBIC, to obtain the spatially resolved beam induced current as a function of the applied voltage so to assess the homogeneity of the membrane as well as the properties and nature of the metal-semiconductor junctions.

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Nº 105

## EXPLORING LATE MEDIEVAL GLASS HERITAGE: INSIGHTS FROM $\mu$ -PIXE ANALYSIS OF GLASS OBJECTS WITH BLUE APPLIED DECORATION FROM PORTUGUESE ARCHAEOLOGICAL EXCAVATIONS (14<sup>TH</sup>-16<sup>TH</sup> CENTURIES)

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Decoration made by applying threads on the body of a glass vessel is common to all European glassmaking between the 13th and 16th centuries. In Portuguese late medieval archaeological excavations, several colourless objects with blue thread application have been brought to light. Usually referred to cylindrical glasses and pedestal goblets, this decoration can be found in several features: one or more lines applied horizontally or spirally, frills forming festoons or blue lines combined with a relief decoration made using the mould-blowing or mezza stampura technique, typical of 16th century Venetian production. The presence of these objects reveals a connection with the upper



strata of society, as they were considered luxurious and suitable for identification as imported, raising questions about their provenance.

This study examines 21 fragments of glass objects adorned with blue decoration, originating from 5 Portuguese archaeological sites across Lisbon, Almada, and Setúbal, dating from the 14th to 16th centuries. Utilizing  $\mu$ -PIXE (particle- induced X-ray emission) analysis, the chemical composition of the glass is examined, revealing insights into the raw materials and colorants, notably cobalt, utilized in producing the distinctive blue threads. Stylistic examination and analytical techniques shed light on how the raw materials used in objects with this type of decoration differ between older and more recent artefacts, indicating changes in society's taste and a change in the raw materials used, hence the place of production of the objects. The chemical composition of the analysed fragments, which falls within the soda-silica-lime group typical of the Mediterranean region, offers clues as to their origin, leading to debate as to whether these objects are imported or locally produced. This interdisciplinary approach combining archaeological findings, stylistic analysis and chemical analysis provides an understanding of the cultural and technological dynamics that shaped glass production in medieval and Renaissance Europe.

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Nº 106

## MICRO-PIXE AND OA-STIM FOR THE EVALUATION OF TRANSITION METALS AND RARE-EARTH ELEMENTS IN CROPS – ASSESSING THE SPREAD OF ANTHROPOGENIC RADIONUCLIDES IN THE ENVIRONMENT

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The ESS (European Spallation Source) [1], is a neutron research facility under construction in Lund, Southern Sweden. The Facility will produce neutrons by spallation, using a powerful linear accelerator to deliver protons to a tungsten target. In addition to the desired neutron production, a long list of radionuclides will be created as by-products of the nuclear reaction inside the target. The Swedish Radiation Safety Authority has established a list of the most relevant radionuclides in term of contribution to effective dose to the ESS workers and the general public in case of release of irradiated target material after an accident at the ESS [2]. This list includes radionuclides that are not produced by the nuclear energy industry, in particular  $^{178m}\text{Hf}$ ,  $^{182}\text{Ta}$ ,  $^{187}\text{W}$ ,  $^{148}\text{Gd}$  and  $^{173}\text{Lu}$ . Ongoing research efforts aim to determine the best analytical methods to assess these exotic and often difficult to measure radionuclides in environmental samples [3]. The present work, presents the use of micro-PIXE and OA-STIM, to investigate the detectability of natural concentrations of Hf, Ta, W, Gd and Lu in crop samples taken from the farmland around the ESS site. This establishment for the baseline of the relevant

transition metals and rare- earth elements in the local environment, will support later efforts to assess the potential for migration of artificially introduced radioisotopes of these elements.

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Nº 108

## MANUFACTURING OF DIFFRACTIVE GRATINGS IN SINGLE-CRYSTAL DIAMOND USING DIFFERENT MICRO-FOCUSED HIGH-ENERGY HEAVY-ION IRRADIATION

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Manufacturing diamond diffractive optical gratings using micro-focused high energy heavy ions demonstrates an inspiring manufacturing endeavor, considering the exceptional diamond material properties, exceeding the existing technological limitations.

It is well-known that MeV heavy ion implantation, with the proper parameters, produces severe structural damage inside monocrystalline diamonds. After the implantation, the internal density decreases significantly, keeping its surface almost intact. Consequently, the buried damaged diamond volume increases and the increase protrudes towards the sample surface, giving rise to a topography that depends on the cumulative damage below.

At CMAM internal microbeam, using 9 MeV  $^{12}\text{C}^{3+}$ , 8 MeV  $^{15}\text{O}^{3+}$ , and 8 MeV  $^{28}\text{Si}^{4+}$  ions of about  $5 \times 2 \text{ mm}^2$  spot, by scanning the beam on the diamond surface with an inbuilt homemade software, a specific diamond grating can be realized. Consecutive lines with  $2.5 \times 10^{14}$  to  $1.3 \times 10^{15}$  fluences have been implanted to manufacture a saw-tooth profile. The patterns have been characterized by optical and atomic force microscopy, and the structures manufactured with each ion are compared. The results

certify that this manufacturing method is highly promising for fabricating diamond optical gratings and that the 3D topography is reproducible despite the variety of high-energy heavy ions used.

N° 109

## DEVELOPMENT OF THE SHORT MICROPROBE SETUP AT BINA

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One of the typical requirements imposed on the geometry of the ion microprobe systems is the long length between the object aperture and quadrupole multiplet focusing system. In the case of the 1.7 MV Pelletron accelerator facility of the Bar Ilan Institute of Nanotechnology and Advanced Materials (BINA), an available space allocated for the installation of the microprobe beam line, limited the distance between the object aperture and the first quadrupole to only 285 cm. The

quadrupole focusing lenses developed at the Ruđer Bošković Institute (RBI)<sup>1</sup> are connected into the Oxford triplet configuration with a working distance of 16 cm. The newly designed scattering chamber, equipped with XYZ Piezo nano- positioning sample stage, maintained its operability with almost all ion beam analysis techniques. Despite the relatively small demagnification ( $D_x=33$ ,  $D_y=12$ ), it has been noticed that the small microprobe length has advantage in reduced influence of the stray magnetic fields, when compared to the long microprobe systems. In this presentation we will show the main setup characteristics, its main advantages and results of the first performance tests.

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N° 110

## PROTON-INDUCED PHOTODYNAMIC THERAPY THROUGH PHOTSENSITISER ACTIVATION MEDIATED BY RARE-EARTH-DOPED NANOPARTICLES

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In proton and heavy-ion radiotherapy, it is known that protons have much lower relative biological effectiveness (RBE, ~1.1) compared to heavy ions. This fundamentally limits the efficacy of protons

in radiotherapy. To increase the RBE of protons without over-exposure to healthy tissues and organs, a strategy is to employ biocompatible nanoparticles as radiosensitizers to enhance the killing of tumour cells. In general, however, the radiosensitization effect of nanoparticles under proton irradiation is not significantly effective [1, 2]. Nuclear DNA is considered the main target of ionizing radiation. Our recent work finds that, under proton irradiation, the secondary electrons and free radicals derived from the nanoparticles have little chance of affecting the nuclear DNA. The reason is that the number of electrons and radicals that can reach the cell nucleus is negligibly low [3]. But this implies that it might be an effective way to enrich the generation of the secondary products derived from the nanoparticles, so that they may find more chances to enter the nucleus and damage the DNA.

In this talk, we will show our latest results by combining photosensitizers (e.g. Rose Bengal and ZnPc) with rare-earth-doped nanoparticles to form nanocomposites, and performing proton-induced photodynamic therapy (proton-PDT). Here, the rare- earth-doped nanoparticles, composed of high-Z elements, are the primary absorbers of proton energy. Instead of radiative recombination, the rare-earth-doped nanoparticle transfers the energy to the photosensitizers within the photosensitizer-nanoparticle nanocomposite. As a result, the photosensitizers are activated and interact with intracellular water to produce reactive oxide species (ROS). The ROS are the main sources of radiosensitization to enhance cell killing. By using these photosensitizer-nanoparticle nanocomposites and performing proton-PDT, we have experimentally achieved more than 50% enhancement of cell killing, which is significantly high compared to other radiosensitization strategies (<20%) [2, 4].

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N° 112

## BRIGHT NANODIAMONDS: THE INTERPLAY OF SURFACE TERMINATION AND MEV ION IRRADIATION IN NV CENTERS FORMATION

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Currently, nanodiamonds (ND) are showing promising perspectives in many research fields due to their inertness, fluorescence, biocompatibility, and the possibility to easily functionalize their surface termination. These appealing properties are finding ever-growing interest, especially in biomedicine, with potential applications such as drug delivery and optical biosensing.

The presented work focuses on enhancing the fluorescence properties of Nitrogen-Vacancy (NV) centers by combining MeV proton irradiation with controlled surface termination. For this purpose, ion implantation processes were conducted using a focused microbeam, enabling the modification of small regions within a single batch of nanodiamonds. This approach ensures improved homogeneity of the sample under analysis since experienced the same thermal treatment. A wide range of fluences ( $10^{13}$  -  $10^{18}$  cm<sup>-2</sup>) to create new NV centers was explored determining the conditions maximizing their creation and the emission yield. The collected data were described by a mathematical model predicting the efficiency of the NV center formation as well as the defect charge state as a function of the fluence. This model can be applied not only for the case study described in the present work (MeV proton in ND), but more in general for different ion implantation processes willing to create new NV centers in diamond (ND or bulk). Thermal oxidations at different temperatures and times were also carried out on ND to study the evolution of surface chemical groups, as well as their influence on optical properties. Besides the general NV centers fluorescence, also NV<sup>-</sup>/NV<sup>0</sup> ratio was also evaluated at the different processing steps (both in terms of oxidation and implantation treatments) and the results were interpreted based on surface moieties.

Finally, in-vitro tests of cellular uptake and viability were conducted with oxidized and proton-irradiated ND to evaluate their potential as fluorescent bio-markers and cellular sensing systems.

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Nº 116

## MICROPIXE CHARACTERIZATION OF OUT-OF-PLANE 2D MOLYBDENUM DISELENIDE NANOSHEETS GROWN ON ULTRAFast LASER-STRUCTURED SUBSTRATES

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In out-of-plane configuration, TMDs exhibit roughness and high specific surface area along with a strong bonding to the surface through boundary atoms in the 2D plane and at the substrate. This latter feature adds complexity to the properties since the monolayers/multilayers acquire an interface-

induced asymmetry. These, and other kinds of porous TMDs are key in applications calling for ion or gas exchange, as in many catalytic, energy storage or sensing applications [1, 2].

In this work, out-of-plane 2D molybdenum diselenide nanosheets grown on ultrafast laser-structured substrates were characterized by microPIXE at the Center for Microanalysis of Materials in Madrid. A notable higher concentration of Mo and Se was observed in the laser-irradiated areas, indicating better nucleation in this area. Furthermore, through SEM characterization and Raman microscopy, the growth of out-of-plane flakes is demonstrated. Largely preferential out-of-plane flakes are observed over the structured regions, showing to be a promising strategy for sensing applications.

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Nº 118

## TOWARDS TRUE SINGLE ION DETECTION FOR DETERMINISTIC ION MICROBEAM IMPLANTATION SCENARIOS

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The project to revitalise the ion microprobe beamline was recently launched at the Laboratory of Ion Beam Physics (LIP) at ETH Zurich. The beamline is attached to the 1.7 MV HVEE Tandetron electrostatic accelerator as well as sputtering and duoplasmatron ion sources. The aim is to develop a dedicated system for the implantation of (sub-)MeV energy ions into target materials with independent, true single-ion detection capabilities. An important application is the maskless fabrication of single photon emitters in diamond and other materials hosting color centre.

Current progress includes the upgrades of the ion sources for higher brightness, as well as installation of new object slits and careful alignment of all ion optics elements along the beamline. The end station is equipped with a standard focusing quadrupole triplet and a vacuum chamber from OM. A new chamber geometry will be presented to allow for a short working distance and integration of the detector systems. The key aspect of the project is the development and testing of two new single-ion capable detectors, that will be installed directly in front of the sample in the focal plane of the beam. They are based on: 1) ultra-thin (approx. 200 nm) free-standing SiC Schottky membrane and 2) a miniaturised gas ionisation chamber (GIC). Both solutions will offer detection of direct ionization of transmitted ions that deposit a small amount of energy in the active volume of the device. This also ensures minimal deviation of impact trajectory. The first results of ion detection and precise implantation placement will be presented. The SiC membrane solution is a result of collaboration [1] with specialised detector technology start-up, while the development of GIC will utilize unique technical know-how present within the LIP group [2].

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Nº 119

# INTEGRITY IN BULLFIGHTING: ANALYSIS OF THE HORNS OF FIGHTING BULLS BY USING THE MICRO-PIXE TECHNIQUE

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One of the main issues in bullfighting is verifying the integrity of the bulls' horns, compromised by the practice known as afeitado (filing), which reduces the horn's length and weakens the animal's defenses.

In this context, we have proposed to use the Particle-Induced X-ray Emission (PIXE) technique with the external micro-beam at CMAM, in order to verify locally the composition at different points on the horn. Subsequently, the study will continue with alternative techniques such as Energy-Dispersive X-ray Spectroscopy (EDX) and X-ray Fluorescence (XRF), suitable to be performed with portable instruments. Furthermore, the research is complemented by a thermographic analysis of the bulls' horns, showing whether the regulated proportion between the irrigated part and the total horn is met.

The main objective is to evaluate the results derived from various techniques, to verify if any manipulation of the bulls' horns has occurred. This phase of the investigation seeks to identify alterations in the horn's natural composition, with a specific lookout for metallic traces indicative of filing. For this purpose, samples taken from bull horns have been measured at the CMAM external micro-beam. Preliminary correlations between the PIXE spectra and the different types of samples, some of which had been manipulated in a controlled way, have been detected and will be presented.

In short, if these two studies are satisfactory, two new methods will have been provided to analyze possible manipulation of bulls' horns and they will be helpful to maintain the animal's integrity during the bullfighting.

N° 120

## SEQUENTIAL SECONDARY ION MASS SPECTROMETRY (SIMS) AND ION BEAM ANALYSIS (IBA) IMAGING OF BIOLOGICAL SAMPLES

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In biology and medicine, understanding the elemental and molecular landscape of biological samples can provide vital information about disease state and ultimately assist in the development/improvement of treatments. As it stands, no single technique can provide full elemental and molecular information from a tissue section. Some approaches have looked at performing the two measurements on sequential tissue sections, but this poses problem with co-registration as tissue are not homogeneous at depth – two sequential sections may look different. Many elemental and molecular analysis techniques are available, but here we report on the exploration of sequential ion beam analysis (IBA) and secondary ion mass spectrometry (SIMS).

The simultaneous employment of both EBS and PIXE (total-IBA) will elucidate the composition of the sample at depth which can in turn be used to correct SIMS mass spectral data to give artefact free images and depth profiles. Sequential IBA and SIMS imaging experiments can provide a new level of information, bridging the elemental and molecular knowledge that no technique can produce on its own, and permit the accurate co-localisation of elemental and molecular markers at the micron scale.

Here we explore the suitability of PET frame slides for SIMS imaging experiments, explore different beams for SIMS imaging (water clusters and bismuth) and apply the methodology to a real biological tissue sample.

Future work will also build on this research with the installation of a commercial SIMS equipment (Ionoptika's J105) at the end of a beam line at the Surrey Ion Beam Centre. This will allow sequential SIMS and ion beam analysis imaging experiments without the need to remove the sample from the chamber. It will also allow MeV-SIMS experiments to be performed using a commercial mass spectrometer, improving the mass accuracy and resolution of the technique.

N° 122

## APPLICATIONS WITH TWO SIMULTANEOUS MEV-ENERGY ION MICROBEAMS

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The Dual Microbeam end-station (DuMi), a unique setup where the MeV ion beams from the two tandem accelerators are simultaneously focused to the  $\mu\text{m}$  scale, has been established at the RBI accelerator facility [1]. 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 [2] is used for the high rigidity ions provided by the 6.0 MV 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 demagnifications and 120 nm spatial resolution.

In this contribution, the authors focus on examples of applications carried out at the DuMi with simultaneous use of two ion microbeams. The latter are based on a damaging / probing concept, where one microbeam is used to create changes in an irradiated sample, while the other one is used to in-situ detect these changes. Focus will be given to examples of dynamic annealing processes taking place on Si/SiC crystals. Experiments carried out with one damaging beam and an in-situ probing beam in high current (RBS channeling) and low current (IBIC) will be presented.

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Nº 124

## Recyclable Materials development@ Analytical Research Infrastructures” Combined access to multiple facilities through EU Project “ReMade@ARI

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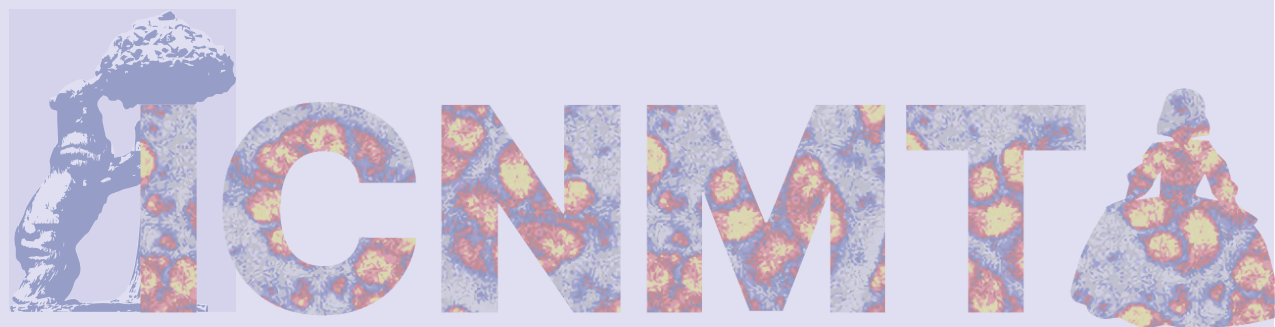
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A radical shift to the circular economy paradigm is urgently needed to cope with the challenge of finite resources diminishing at a frightening pace while the quantity of waste increases at an alarming rate. In the ReMade@ARI (REcyclable MAterials DEvelopment at Analytical Research Infrastructures) project, many of the major European analytical research infrastructures are joining forces to pioneer a support hub for materials research that will facilitate the transition to a circular economy.

ReMade@ARI started in September 2022 and will run for four years. It is funded by the EU Commission's Horizon Europe program with a budget of 13.8 million euros and co-funded by UK Research and Innovation (UKRI) and by the Swiss State Secretariat for Education, Research, and Innovation. The project commits to leverage the development of innovative, sustainable materials for key

components in most diverse sectors, such as electronics, batteries, vehicles, construction, packaging, plastics, textiles, and food at an unprecedented level.

ReMade@ARI provides a gateway and fully supported user access to more than 50 European analytical infrastructures for academic and industrial research addressing questions on circular economy. Proposals should include the use of at least two techniques available at the various facilities, financial support for travel and accommodation expenses is available. Users can submit proposals through two calls per year, supported by experienced experts in the various analytical techniques and by a network of highly motivated junior scientists. The proposals are evaluated by a newly constituted selection committee. The techniques offered include Nuclear Microprobe Ion Beam Analysis and Modification. Further information can be found on the ReMade@ARI website (<https://remade-project.eu/>).



# ORAL COMMUNICATION

N° 16

## MICRO-PIXE FOR IRON MAPPING IN FERROPTOSIS

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Ferroptosis, a non-apoptotic iron-dependent cell death, holds great potential in several biomedical applications, such as cancer therapy and neurological disease research. Particularly, the growing recognition of the role of ferroptosis in the treatment of glioblastomas is capturing the attention of the scientific community. It is seen as a promising approach to address one of the most prevalent malignant craniocerebral tumors<sup>1</sup>. However, sensitive and specific tools for ferroptosis imaging and quantification at the cellular level remain a challenge. The most used method for imaging involves the use of fluorescent markers, such as Bodipy 581/591 C11, which detects lipid peroxidation, one of the hallmarks of ferroptosis. This work explores the potential of micro-PIXE as a complementary technique for visualizing and quantifying ferroptosis in single cells. To evaluate morphological and elemental changes associated with ferroptosis, we treated the U87 glioblastoma cell line with several ferroptosis inducers, including RSL3 and cisplatin. We then measured the fixed cells on Mylar films in the microbeam line. Micro-PIXE successfully detected and localized iron accumulation within ferroptotic cells, in contrast to non- treated cells, demonstrating its potential for ferroptosis visualization. It was also possible to correlate the 2D micro-PIXE elemental maps with optical images to assess morphological changes due to ferroptosis inducers. This study demonstrates how micro-PIXE can be used as a powerful tool for ferroptosis analysis. Its high spatial resolution and elemental specificity offer unique advantages for studying ferroptosis in various biological contexts. Further research is needed to explore its applications in different cell lines and tissues.

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N° 18

## ENSEMBLES OF DONOR SPINS IN ISOTOPICALLY ENRICHED SILICON: NUCLEAR MICROPROBE ANALYSIS OF DEVICES FOR SPIN RESONANCE STUDIES

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Quantum information encoded on the nuclear or electron spins of donor atoms (qubits) in isotopically enriched <sup>28</sup>Si (nuclear spin I=0) have robust coherence times. Deterministically implanted donors <sup>31</sup>P (I=1/2) or <sup>123</sup>Sb (I=7/2) are suitable for a quantum computer device with high fidelity qubits<sup>3</sup>. We have

developed an enrichment process involving  $^{28}\text{Si}$  implantation into  $^{\text{nat}}\text{Si}$  that depletes the non-zero spin  $^{29}\text{Si}$  ( $I=1/2$ ) isotope to below 3 ppm resulting in a low-spin-background matrix<sup>2</sup>. To investigate the purity and structure of this matrix for background perturbations of qubits, we employ a novel Electrically Detected Magnetic Resonance (EDMR) device for measurements of ensembles of millions of implanted donor qubits. We probe the donor electron spins in a 20x20 micron square island of enriched  $^{28}\text{Si}$  in a  $^{\text{nat}}\text{Si}$  wafer. To investigate the properties of the novel EDMR device we employ nuclear microprobe mapping of the on-chip electrode structure surrounding the construction site used for spin read-out and the integrated radio frequency antenna used to induce spin flips of the donor electrons. The EDMR devices show resonances from the  $\text{P}_b$  centre from silicon surface defects and the two  $^{31}\text{P}$  donor electron hyperfine levels in devices with both P and  $\text{PF}_2$  implants<sup>1</sup>.

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N° 19

# HIGH RESOLUTION IMAGING OF DEFECTS IN DIAMOND GENERATED BY FOCUSED ION IMPLANTATION

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Solid-state defects in materials have been widely studied in search of defect centres with advantageous characteristics that can be integrated into solid-state quantum devices. Common techniques to analyse the formation of these defects created by ion implantation include the analysis of ion implantation range within the material with SRIM (Stopping and Range of Ions in Matter) software and PL (photoluminescence)/Raman line scan along varied z-axis on the implantation spot. However, the formation of defects along the ion implantation track cannot be fully understood with these techniques. A cross-section of the implantation track is useful to visually verify the spatial distribution of the defects created along the path. This can be exactly achieved by performing line implants through the edge of the material of interest and performing lateral imaging along the polished side of the material. In this study, the high resolution cross-sectional imaging technique has been employed to study light and heavy ion implanted diamond samples [1]. The cross-section of the ion implantation path is created on the lateral surface that can be easily accessed. Through state of the art confocal microscope imaging, a spatial distribution of the radiation-induced defects created can be visualized in high resolution. These images reveal such radiation-damage defects formed along electronic stopping and nuclear stopping regions and offer greater understanding of the formation dynamics of such defects within the material. In this study, various damage related colour centres formed over a range of implantation fluences and annealing conditions have been mapped.

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N<sup>o</sup> 20

## THE 10 NM NUCLEAR MICROPROBE AND ITS APPLICATIONS

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The spatial resolution plays a crucial role in determining the performance of nuclear microprobes. However, the formation of spatial resolutions below 10 nm remains a challenge in nuclear microprobes. Here, we discuss recently proposed novel technologies (near-axis scanning transmission ion microscopy and double-fragment scattering) utilizing molecular ions to address the resolution challenges. Our work demonstrates a H<sub>2</sub><sup>+</sup> molecular beam with 6.0×10 nm<sup>2</sup> lateral resolution and monolayer thickness resolution respectively [1]. Using the improved nuclear microprobe, preliminary application results are presented from several fields, including fundamental physics, two-dimensional (2D) materials, thin films, bioimaging and proton beam writing.

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N° 24

## ULTRA-THIN DIAMOND DETECTORS FOR ON-LINE MONITORING OF ION MICROBEAMS

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Studying the impact of ionizing radiation on living organisms is important both for radiation protection and radiation therapy applications. It requires to develop irradiation facilities that enable a precise control of the delivered dose and the localization of the irradiations. Charged-particle microbeams extracted in air are capable of irradiating living biological samples down to the micrometer scale. Unlike global irradiation, microbeam irradiation enables a specific cell or sub-cellular compartment to be targeted, with a small beam spot and a controlled number of particles. The ions available on the microbeam facilities at AIFIRA (LP2I Bordeaux, France) and MIRCOM (IRSN, Cadarache, France) are protons, alpha-particles or heavier ions like carbon with maximum energies ranging from 3 MeV to 8 MeV (carbon).

In order to deliver a precise and low number of ions per cell, it is necessary to develop dedicated detectors. Diamond detectors are suited to answer the problematic but face many technological challenges [1]. Indeed, as the ions stop in the biological samples, the detector must be placed as the extraction window and be very thin to limit the disturbance (in energy loss and deviation) of the beam. First prototypes have been previously tested successfully [2]. This work aims at developing thinner detectors (~1 µm) and improve their design.

Reactive Ion Etching (RIE) uses plasma bombarded onto the surface to etch diamond. Etch rate rise up to a few micrometers per hour. Starting from 50 µm bulk diamond, this technique has to be adapted for deep etching to achieve micrometer thickness. A good surface quality and thickness homogeneity of the etched diamond are necessary for efficient charge collection and accurate ion counting. Plasma composition and masking material are some of the parameters that can be optimized. We will present ongoing developments of etching techniques for the thinning of diamond.

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Nº 26

## MICRO-PIXE AS A SCREENING TECHNIQUE FOR EVALUATING GOLD NANOPARTICLE UPTAKE IN GLIOBLASTOMA CANCER CELLS

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Glioblastoma multiforme (GBM) is the most aggressive and prevalent type of glioma. Current standard GBM therapy combines surgical resection, radiotherapy, and temozolomide, although survival rates remain disheartening. Leveraging advances in nanoscience, nanoparticles (NPs) offer a novel approach to improve GBM treatment. Gold NPs have attracted significant attention in scientific research due to their optical, electronic, and physical properties. These nanoparticles can exhibit various shapes, while their sizes typically fall within the range of 1 to 100 nm. Various synthesis methods exist, employing different molecules as stabilizers and reducers. Diverse molecules used in synthesis impart distinct surface properties to the nanoparticles, which can influence their behavior in biological environments, particularly their potential for cellular internalization.

This study aims to determine which synthesis chemistry is most conducive to internalization by glioblastoma cancer cells. For this purpose, we synthesized four types of gold nanoparticles using different chemical agents as stabilizers: sodium citrate, a biopolymer, silsesquioxane (SSQ), and a thiol-containing molecule. Subsequently, we treated A172 glioblastoma cell lines with these nanoparticles for 24 hours. Following treatment, the cells were fixed using paraformaldehyde (PFA) into 8 µm mylar substrates, which were then subjected to micro-PIXE analysis. In biology, confirming the uptake of nanoparticles typically requires attaching fluorescent molecules to the nanoparticles to enable visualization under a microscope, which can be quite challenging and time-consuming. In this study, we employ micro-PIXE as an alternative technique to assess nanoparticle uptake, demonstrating its potential as a screening method.

We detected gold signals within the cells for all formulations except for the silsesquioxane-based gold NPs. Additionally, we correlated elemental maps obtained from micro-PIXE analysis with optical images to evaluate cell morphology. In conclusion, our study elucidates the differential cellular uptake of variously synthesized gold NPs by glioblastoma cells, providing valuable insights into their potential for targeted cancer therapy.



N° 27

# DEVELOPMENT OF A WORKFLOW FOR CORRELATIVE IMAGING OF ELEMENTAL AND CHEMICAL BIOMARKERS FROM A SINGLE TISSUE SECTION USING MICROBEAM PIXE AND RAMAN MICROSCOPY

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The correlation of elemental and molecular biomarkers at sub-cellular resolution is highly desirable in biomedical sciences and will provide new insight into the impact of metal-containing drugs, metal accumulation and nanoparticles on the local chemistry of the host organism. This is relevant to the treatment of a host of diseases such as tuberculosis, diabetes and cancer.

We have recently shown that PIXE elemental mapping can be carried out in sequence with the mass spectrometry imaging techniques desorption electrospray ionisation (DESI) [1,2] and direct analyte probe nano extraction (DAPNe) [3] to co-locate elemental and molecular markers. Whilst these techniques are very powerful in revealing the location of specific metabolites and lipids, the ability to perform correlative analysis is limited by the spatial resolution of these mass spectrometry imaging tools (at best 10 microns) which does not match the spatial resolution of the PIXE.

In contrast, Raman microscopy is a powerful and well-established method for bioimaging, capable of providing images of chemical functional groups (lipids, proteins) at sub-cellular resolution (~500nm). It therefore provides complementary information to PIXE imaging, at similar spatial scales. At high spatial resolution, features are not accurately reproduced in sequential tissue sections and image registration is difficult. It is therefore desirable to perform sequential PIXE and Raman analysis on the same sample. This is non-trivial, as each technique requires different procedures for sample handling and both are potentially destructive to the tissues.

We explore the integration of Raman microscopy, DESI and PIXE imaging to provide correlative images of metals and biomolecules in a single tissue section at sub-cellular resolution. We have developed a sampling handling procedure and analytical workflow that is compatible with all three techniques. As a proof of concept, we demonstrate sequential DESI- Raman-PIXE to correlate elemental and molecular markers in pig skin, showing complementary information.

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Nº 30

## MICROBEAM IRRADIATION REVEALS CYTOPLASMIC DAMAGE CONTRIBUTES TO CHROMOSOMAL ABERRATIONS

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Microbeam technology offers a valuable tool to investigate inter- and intracellular responses to radiation-induced damage. While direct nucleus irradiation is known to be the primary cause of various radiobiological effects, the cytoplasm also receives radiation and potentially influencing intracellular responses [1,2]. This study aims to determine whether cytoplasmic irradiation contributes to micro-nuclei induction when the cytoplasm and nucleus are sequentially irradiated.

Microbeam irradiations were performed using the SPICE-QST microbeam at the National Institutes for Quantum Science and Technology, delivering 3.4 MeV protons (LET in water: 11.7 keV/μm) with a 2 μm beam diameter [3]. Three targeted irradiation were employed: (N) nucleus-only, (C) cytoplasm-only, and (N+C) irradiating both the nucleus and cytoplasm. In (N+C), with 1000 protons delivered to the cytoplasm in the N+C group. Immediately post-irradiation, the cyto-block method was employed for micronucleus (MN) formation analysis. Cells were fixed 50 hours later, followed by staining the cytoplasm and nucleus with Cell Tracker Orange (10 μg/ml) and Hoechst33342 (1 μg/ml), respectively. Fluorescence images of over 1000 binucleated cells were captured using the SPICE offline microscopic system.

The MN induction rate per binucleated cell was compared between N and N+C (1000 protons) irradiation. N irradiation displayed a peak MN induction rate at around 100 protons, whereas N+C irradiation peaked at around 300 protons. The result implies that the cytoplasmic irradiation induced protective response against MN induction. Notably, at 1000 protons or more, N+C irradiation yielded an MN induction rate approximately twice that of N irradiation. This may be result of enhanced cellular progression triggered by cytoplasmic damage. These findings clearly demonstrate that not only DNA damage but also cytoplasmic damage contribute significantly to MN induction, suggesting a more complex interplay between cellular organelles during radiation exposure.

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N° **33**

## A MULTI-ELECTRODE TWO-DIMENSIONAL POSITION-SENSITIVE DIAMOND DETECTOR

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Deterministic ion placement is a crucial tool for the fabrication of devices based on single-defect engineering by ion implantation.

The target material can be exploited as a solid-state particle detector, thus enabling the detection of individual ion projectiles. In multi-electrode devices charge pulses at all the electrodes are induced concurrently by the motion of the excess charge carriers generated by a single ion.

This charge-sharing effect is such that the pulse amplitude at each sensitive electrode depends on the device geometry, its overall electrostatic configuration, and the charge transport properties of the detecting material.

Therefore, the cross-analysis of the charge pulses induced at each electrode offers implicit information on the position of the ion impact.

In this work, we investigate the 2-dimensional position sensitivity of a diamond detector fabricated by deep ion beam lithography. By exploiting the Ion Beam Induced Charge (IBIC) technique, the device was exposed to a 2 MeV Li<sup>+</sup> ion micro- beam to map the spatial dependence of the charge collection efficiency (CCE) on the nominal micro-beam scanning position. The experiment analysis revealed a 2-dimensional position sensitivity of the device with micrometric resolution at the center of the active region.

N° 34

# IN SITU STUDY OF RADIO-INDUCED DNA FRAGMENTATION IN AQUEOUS SOLUTION

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Understanding the physical phenomena behind DNA molecule irradiation by protons is key for proton therapy in cancer treatment [1] and enhancing cosmic radiation protection in manned space missions, given that high-energy protons constitute 90% of cosmic radiation [2]. Studying the dynamics of DNA fragmentation, event by event, is a fundamental physics challenge to understand the mechanisms of energy relaxation in the medium after irradiation [3].

Using an in situ approach, we study the dynamical and statistical properties of radio-induced fragmentation of T4 bacteriophage DNA (167kbp) in aqueous solution, marked with a YOYO-1 dye and observed via fluorescence microscopy. This irradiation is performed using the AIFIRA particle accelerator's microbeam line, delivering 3 MeV protons controllable in number, space, and time, ensuring uniform irradiation. We record hundreds of videos per proton fluence, ranging from 0 to 7000 protons/ $\mu\text{m}^2$ , providing significant statistics with over 15,000 individually tracked DNA molecules in 50-second videos.

A coupling between machine learning and a Python particle tracking algorithm allows us to have a temporal evolution of the spatial displacement of DNA molecules. This innovative approach enables us to determine DNA fragmentation probabilities under ionizing radiation and to study the Brownian motion of dissociating molecules. Our statistical analysis aims to generate benchmark data for this DNA, serving as a comparison point for simulations by the Monte-Carlo Geant4-DNA calculation code [4]. Simultaneously, the dynamic study of fragment dissociation offers essential insights into the hydrodynamic interaction characteristics of the fragments at a micrometric scale [5].

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Nº 38

## MEASUREMENT OF THE DEPLETION ZONE OF SiC DETECTORS USING THE IBIC TECHNIQUE

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For several years, SiC detectors have been considered among the most promising for use in extreme radiation and temperature environments, such as those in future nuclear fusion reactors or in high-energy physics experiments. For these applications, the replacement of the front electrode by a graphene layer could represent a significant improvement due to the thermal and electronic properties of this novel material. In this work, the response of a SiC detector fabricated at the Institute of Microelectronics of Barcelona has been characterized using the ion beam induced charge (IBIC) technique at the nuclear microbeam line of the National Accelerator Center (CNA, Seville).

The detector consists of a p-n junction with a nominal thickness of 50  $\mu\text{m}$  and its main feature lies in the removal of the front electrode, as a step prior to graphene deposition, except for an outer ring that surrounds it. Due to this special configuration, the values of the thickness of the depletion zone with the applied voltage, typically obtained by the C-V curve, may not be completely reliable and other complementary methods must be used for verification.

At CNA the size of the depletion zone versus voltage was studied by using a rotating sample holder that permits rotation in vacuum with an accuracy of 1°. This methodology uses a monoenergetic proton beam with a range larger than the depletion zone. Experiments performed as a function of detector rotation angle, compared with SRIM simulations of the energy deposited in the active zone, allow us to calibrate the MCA as well as to find the thickness of both the depletion zone and the surface passivation layer.

The values of the depletion zone at different voltages have been compared with those deduced from electrical measurements and those obtained from the Two Photon Absorption-Transient Current Technique (TPA-TCT) performed at IFIC.

N° 40

## STEREO MICRO-PARTICLE INDUCED X-RAY EMISSION (PIXE) ANALYSIS OF METALLIC ELEMENTS DEPOSITED ON ENGINEERED MICROPLASTICS SURFACES

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Microplastics, 3D objects that are irregular or spherical, are a concern with the increase of ocean plastic pollution. Natural microplastics can cumulate heavy metals that can harm the ocean environment and biota. Eventually, these particles may be ingested by humans through the food chain and cause health problems<sup>1-2</sup>. As a result, the composition of elements accumulated on/in microplastics has to be investigated to understand their threat. The 3D particles are not directly amenable to qualitative analysis by conventional 2D analysis with a microbeam. To provide the extra information, we have investigated stereographic imaging using a MeV ion microprobe to obtain 3D information in order to facilitate quantitative analysis.

In this work, we created microstructures using proton beam writing to mimic microplastics. These microstructures were subsequently coated with metals, representative of those that are deposited on microplastics. By applying multiple PIXE measurements from different angles (stereo PIXE), 3D elemental analysis could be accomplished. We present characteristics of the experimental set-up and preliminary results showing 3D image analysis of structures that mimic oceanic microplastic particles.

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N° 44

## SEU PRE-SCREENING OF SRAM DEVICES USING PROTON AND HEAVY ION MICROBEAMS

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This work presents a Level 3 pre-screening test results for Single Event Upsets (SEUs) in simple Commercial-Off-The-Shelf (COTS) SRAM devices. For SEU tests the die of decapsulated SRAM devices were precisely irradiated using proton (1-10 MeV/u) or medium-heavy ion (1-3 MeV/u) microbeams provided

by the ANTARES Heavy Ion Microprobe at ANSTO. The device is tested in operation using a modular PCB that is plugged into a microcontroller board that drives the device. The device is irradiated until the full memory bank is corrupted. The SEU cross section for a fully decapped 65nm SRAM 23LC512 irradiated by 9 MeV protons was  $2.34 \times 10^{-14} \text{ cm}^2/\text{bit}$ , while the SEU cross section for a thinned SRAM device with 200  $\mu\text{m}$  of emaining cap resin was  $2.73 \times 10^{-14} \text{ cm}^2/\text{bit}$ . This highlights the importance of the different LET profile in a device for decapped and thinned samples. For comparison to low direct ionizing protons ( $\text{LET} \approx 0.03 \text{ MeVcm}^2/\text{mg}$ ), we tested the device with microbeams of various light, medium, and heavy ions that have the LET value in the 1 – 30  $\text{MeVcm}^2/\text{mg}$  range and the range in silicon of at least 15  $\mu\text{m}$  to assess the suitability of the ANSTO setup for SEU pre-screening of COTS microelectronic devices.

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## DEVELOPMENT OF MICROFLUIDIC CHIPS TO IMMOBILIZE LIVING ORGANISMS FOR IRRADIATION WITH A PROTON MICROBEAM.

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Since their first use on cells, either individual cells or cell populations, ion microbeams have proven their interest in exploring the effects of ionizing radiation. With the growing interest to go towards irradiating multicellular organisms, the need to immobilize the samples to allow a fast, precise, and reproducible irradiation represents a non-negligible technical challenge. Although the use of anesthetics can be efficient to transiently stop the movements of some models, it can induce bias, and is not ideal to study phenomena such as motor function.

Microfluidics is a technology that has appeared in the 90's. It is quickly developing, with the use of new materials and fabrication techniques. With the development of bio-compatible materials, it can be applied to many biology domains, such as radiation biology. The versatility and precision of the fabrication techniques allows the design of microfluidics chips adapted to microbeam experiments, thus significantly improving the variety of biological samples that can be studied.

Two types of microfluidic chips have been developed for proton microbeam irradiation on the MIRCOM facility, based in Cadarache (France). The first one is dedicated to the irradiation of the nematode *Caenorhabditis elegans*, a reference model in biology. It has been designed to immobilize adult nematodes, allowing head irradiation to study the effects of proton irradiation on the central nervous system. The second one is dedicated to the irradiation of sea bass (*Dicentrarchus labrax*) and zebrafish (*Danio rerio*) embryos. It has been designed to immobilize embryos, allowing their irradiation with protons to reproduce the radiative environment of an Earth-Moon travel, to study the feasibility of space aquaculture on the Moon, in the framework of the Lunar Hatch project.

In addition to the chips themselves, we will present the first validation studies of both designs, and the first results obtained during recent experimental campaigns.

N° 50

## LOW NOISE IBIC SYSTEM FOR THE INVESTIGATION OF WEAK RESPONSE DEVICES

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One of the great challenges in the application of IBIC technique is characterization of charge transport in systems with very weak response. Causing such low response is either low charge collection efficiency and/or small sensitive volume of the device which results in low number of charge carriers created within it. In either case, low noise signal processing electronics must be used. We have developed several systems based on charge sensitive preamplifiers (e.g. CUBE from XGLab and A250 from Amptek) positioned in close vicinity to a sample under investigation. Such setup is particularly useful for deterministic ion implantation in diamond, where noise levels need to be of the order of 1 keV. In these circumstances, low energy of impinging ions (tens of keV) and rather large amount of charge carriers being lost in the surface dead layer, impose quite strong requirements on the acceptable noise levels in the pulse processing electronics. In addition, as it will be shown, the arrangement of charge sensing electrodes becomes very important as well<sup>1</sup>. There are also other examples where IBIC signals, generated in very small, sub-micrometer volumes, are often buried in noise. One of these examples is the investigation of the core-shell p-n junction GaN microwires, where charge transport occurs in sub-micrometer thick layers<sup>2</sup>. To increase the signal to noise ratio, we have used low noise pulse processing electronics and sub-MeV energy Si ions for IBIC probing. Due to the trend of reducing sizes of radiation detectors and other types of sensors, IBIC technique can be considered as a technique of choice only if the appropriate upgrades of the measurement setup associated with the ion microprobe systems is performed.

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N° 53

## DEVELOPMENT OF A CONTEMPORARY ION BEAMLINE FOR NANOSCALE ION BEAM APPLICATIONS

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Institute, the microbeam ion beamline [2] has been used for many years for analytical methods such as PIXE, RBS, NRA, STIM, and MeV-SIMS with MeV ion beams. The current ion beamline setup combined with a quadrupole magnetic lens triplet, allows the focusing of an ion beam to a size of 700 nm. It is routinely utilized for analytical work in biology, medicine, and fusion.

Eager to achieve better focusing and to enable the focusing of swift ions in order to expand the range of applications with high-energy focused ions, we developed and installed a new ion beamline, "nanobeam". Built in 2022, nanobeam is the successor to microbeam, incorporating several contemporary technical improvements such as mu-metal shielding, an active vibration isolation system [3], and a quadrupole magnetic lens quadruplet able to focus ions with a magnetic rigidity of 40 MeV amu.

Since mid-2022, we have been testing the installed equipment, experimenting with various magnetic lens configurations, searching for optimal instrumentation settings, improving already developed detection systems, and adding new functionalities. The nanobeam currently enables PIXE, NRA, and RBS analyses of samples mounted on a 4-axes stage inside the vacuum chamber. With the current setup, we managed to bring the spot size of the 3 MeV proton beam down to 500 nanometres size. Currently, a beam blanker, a TOF mass spectrometer for MeV-SIMS [4], a STIM detector system beam chopper for current normalization, and an RBS detector station are under construction to complement the existing hardware.

Results of the performance tests, as well as selected results of the first applications of the novel nanobeam will be presented.

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N° 54

# ION BEAM ANALYSIS END-STATION WITH WAVELENGTH-DISPERSIVE X-RAY SPECTROMETER FOR ION MICROPROBE APPLICATIONS

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A novel modular end-station has been developed at the Ruđer Bošković Institute (RBI) for use at single-ion-beam microprobe. It supports analysis of micro-samples using simultaneously several ion beam analysis (IBA) techniques. These include wavelength-dispersive (WD) and energy-dispersive (ED) Particle Induced X-ray Emission (PIXE) spectrometry, Elastic Backscattering Spectrometry (EBS and RBS) and Nuclear Reaction Analysis (NRA). ED-PIXE and EBS/RBS/NRA measurements enable the creation of two-dimensional elemental maps. The in-house developed wavelength-dispersive X-ray (WDX) spectrometer, featuring a flat diffraction crystal and X-ray CCD camera, enables measurements of high-resolution spectra on selected sample micro-regions, making this system a novel complementary tool that significantly enhances the analytical capabilities of the RBI ion microprobe beamline. The key features of the WDX spectrometer are: I) the complementary analysis of complex ED X-ray spectra with many overlapping peaks measured simultaneously; and II) the chemical speciation studies.

In this presentation, the modular IBA end-station will be described in detail and its ability to perform simultaneous analytical measurements on selected heterogeneous micro-samples will be demonstrated.

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N° 55

# SEQUENTIAL MOLECULAR AND ELEMENTAL IMAGING WITH MEV SIMS AND HEAVY ION PIXE

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Secondary ion mass spectrometry with MeV ions (MeV SIMS), which is installed at the RBI heavy ion microprobe, has been used for the molecular imaging of various types of organic materials for ten years. The technique was successfully applied in the fields of biology, forensics and cultural heritage [1-3]. It is the first ion beam analysis (IBA) imaging method that can provide information on the lateral distribution of molecular species on the sample. MeV SIMS is a surface technique in which molecules are emitted from the uppermost layers and therefore is not ideal for elemental imaging. So far, elemental imaging by particle induced X-ray emission (PIXE) has usually been performed with 2-3 MeV protons, followed by MeV SIMS for molecular imaging with heavier ions such as O or Si. However, switching between PIXE with high current protons and MeV SIMS with low current heavy ions often requires beam optimization and sample shifting, resulting in a loss of lateral correlation between elemental and molecular composition. In many cases, especially in biomedicine, the exact match of elemental and molecular lateral position is of utmost importance for understanding the pathogenesis of diseases. Therefore, in the present work, heavy ion PIXE (HI-PIXE) and MeV-SIMS methods were performed sequentially with the same ion species by simply switching between the pulsed beam mode for heavy ion MeV-SIMS and the continuous beam mode for proton PIXE, while keeping the position fixed on the sample. Several heavy ions were tested to find the best possible conditions for the sequential application of both techniques in terms of intact secondary molecular ions and X-ray yields. The applicability of the sequential use of both techniques was demonstrated on different samples, biological, cultural heritage and forensic samples. Disadvantages and advantages of using heavy ions instead of protons for PIXE are discussed.

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N° 57

# MICRO-PIXE ANALYSIS OF INDIGENOUS SOUTH AFRICAN MEDICINAL PLANT LIPPIA SCABERRIMA SOND.

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The interest in the use of traditional medicinal plants for the treatment of various diseases has grown in the past years in developing countries due to the unavailability of modern medicine and modern techniques. Medicinal plants such as *Lippia scaberrima* Sond. and *Lippia javanica* (Burm.f.) Spreng, have been used for the past hundreds of years but were not investigated using modern techniques to determine their content, efficacy, and toxicity [1-4]. In this study, the elemental content of the indigenous South African medicinal plant *Lippia scaberrima* Sond. was investigated using the micro-PIXE technique. This plant is traditionally used for the treatment of diseases such as colds, fever and bronchitis [5], but its elemental content has never been investigated using nuclear techniques. PIXE measurements of the elemental content of plant seeds were obtained and were carried out using a 3 MeV collimated proton beam from the 6 MV Tandetron accelerator at NRF iThemba LABS, Cape Town, South Africa. The accumulated PIXE spectra were analyzed using GeoPIXE II software. The results showed the presence of Si, P, K, Cl, Mn, Se, Cu, Sr, Ca Fe at different concentrations ranging from 3.4 (0.8)  $\mu\text{g/g}$  up to 396 (70)  $\mu\text{g/g}$ . The heavy toxic metals such as Pb, As, Cd, and Hg were not detected. The results of this investigation are presented and discussed.

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Nº 58

## STUDY OF MICROSTRUCTURES PATTERNED ON PTFE USING STIM MULTI- STEREOSCOPIC IMAGES

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We investigated microstructures patterned on PTFE obtained through the Proton Beam Writing (PWB) technique. To this end, 2.2 MeV proton beams of about 2 square micrometers were employed for the irradiation of 25  $\mu\text{m}$  thick PTFE foils with a fluence of  $1 \times 10^{15}$  ions. $\text{cm}^{-2}$

The microstructures were developed with different liquid media including distilled water, ethyl alcohol, NaOH, and low-density oil under 40 kHz ultrasound waves. While the temperature of the media was kept constant over the treatment procedure, the time of etching was varied in order to check the impact of the etching time on the microstructures.

The post-etching structures were analyzed with on-axis STIM (Scanning Transmission Ion Microscopy), SEM (Scanning Electron Microscopy) and OM (Optical Microscopy). For the STIM measurements, the angle theta between the beam direction and the sample's normal varied between  $-20^\circ$  and  $+20^\circ$  in steps of  $1^\circ$ . The STIM images based on the proton energy loss spectra for each angle were generated by OMDAQ-3 software. Subsequently, the images were reprojected according to pixels combining different angles. Finally, the images are processed with the ImageJ® software in order to provide stereoscopic animated images of the structures.

Unlike other techniques, STIM multi-stereoscopic images proved to be effective for revealing buried microstructures in the polymer. In particular, the advantages and drawbacks of this technique when compared to SEM and OM are discussed.

Nº 60

## EVALUATING THE IMPACT OF SAMPLE PREPARATION ON ELEMENTAL ANALYSIS OF GLIOBLASTOMA CELLS WITH MICRO-PIXE

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Micro-PIXE is a powerful tool for elemental mapping of biological samples. Its application for cell screening has been explored for decades [1]. In principle, an appropriate sample preparation protocol must not interfere with the elemental composition of the species under study. Moreover, it should ideally preserve the original spatial position of the elements in the organic tissue. Consequently, establishing a proper preparation protocol can be challenging [2].

We report on the impact of sample preparation protocol on the micro-PIXE analysis of U87 glioblastoma cells. Particular attention is given to the rinsing procedure during the preparation of the cells for elemental analysis. To that end, unrinsed and rinsed cells with different rinsing protocols were analyzed with 2.2 MeV H<sup>+</sup>. An Oxford Microbeam system operating on triplet mode focused the beam to 2 x 2 μm<sup>2</sup>. X-rays were detected by a SDD detector placed at 45° with respect to the beam direction. Qualitative and quantitative analysis were performed with GUMAP [3] software. The 2D elemental maps were compared with the corresponding optical microscopy images.

Overall, substantial differences were observed in the elemental composition between unrinsed and rinsed cells due to impurities present in the culture medium. Such impurities negatively affect the determination of cell boundaries from the substrate. In addition, the presence of elements such as Na, Cl, K, P and S in the medium lead to a much higher concentration of those elements in unrinsed cells, resulting in a misleading characterization of endogenous elements. The results indicate that rinsing the cell culture with deionized water for 10 minutes lead to much better results.

Finally, the influence of different fixation protocols on the elemental quantification and distribution will be discussed.

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N° 61

## DEVELOPMENT OF A SEPARATED QUADRUPOLE DOUBLET MAGNETIC FOCUSING SYSTEM FOR HIGH THROUGHPUT MICROANALYSIS AT THE UNIVERSITY OF NORTH TEXAS

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We have developed a high throughput ion beam focusing system utilizing two pairs of magnetic quadrupole doublets lenses separated at a distance. The system is built in a microprobe beamline with an existing Louisiana magnetic doublet (LMD) [1] lens associated with a 3MV single-ended (NEC, 9SH) accelerator. The initial doublet system had a demagnification of  $\sim 20 \times 60$  (in X and Y directions respectively) for a working distance of 18 cm. The new system is a further optimization of the focusing system with the addition of another pair of LMD systems separated by  $\sim 1.00$  m distance. The new system has an increased orthomorphic demagnification of  $\sim 100 \times 100$ , thereby increasing the beam throughput by an order of magnitude. We have routinely imaged 2000 mesh Cu grids using each of the RBS, PIXE, STIM, and Secondary Backscattered Electron detectors using 2 MeV H<sup>+</sup> beams.

We will be presenting theoretical simulations and experimental results demonstrating the high demagnification and high current density of the new microprobe system at various excitation modes (doublet, triplet, and separated doublets). We will present some of the results of ion microprobe analysis of biomedical, environmental, and photovoltaic samples and devices.

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N° 65

## ION BEAM INDUCED CHARGE STUDIES OF SCCVD DIAMONDS AT RBI CRYOGENIC MICROPROBE

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Due to diamond's inherent properties, particle detectors using high quality scCVD diamond are often considered an ideal sensor for applications in harsh environments such as high radiation environments and low/high temperature environments. In order to study diamond response in such conditions, using  $\mu\text{m}$  size focused ion beams, a cryogenic modular end-station has been developed in the Laboratory for ion beam interactions of the Ruđer Bošković Institute (RBI) in Zagreb. This cryogenic setup allows

the samples to be cooled to temperatures of 48K while featuring a heater and a temperature sensor which enable stability for each measurement up to room temperature.

Previously, the setup was employed for the study of a thick ( $>300\text{ }\mu\text{m}$ ) scCVD diamond detector[1]. In the present study we aim to investigate the response of a thin,  $40\text{ }\mu\text{m}$  thick scCVD detector, constructed in the Laboratory for Ion Beam Interactions of RBI and the Diamond Sensors Laboratory of CEA-LIST, at low temperatures (from 48K) up to room temperature and, additionally, under extreme radiation conditions.

In order to study the behavior of charge carriers in low temperatures in pristine and damaged diamond, irradiations were conducted in transmission mode, and further probing was conducted by means of Ion Beam Induced Charge (IBIC) and Ion Transient Current Technique (TCT) with different ion beams.

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N° 66

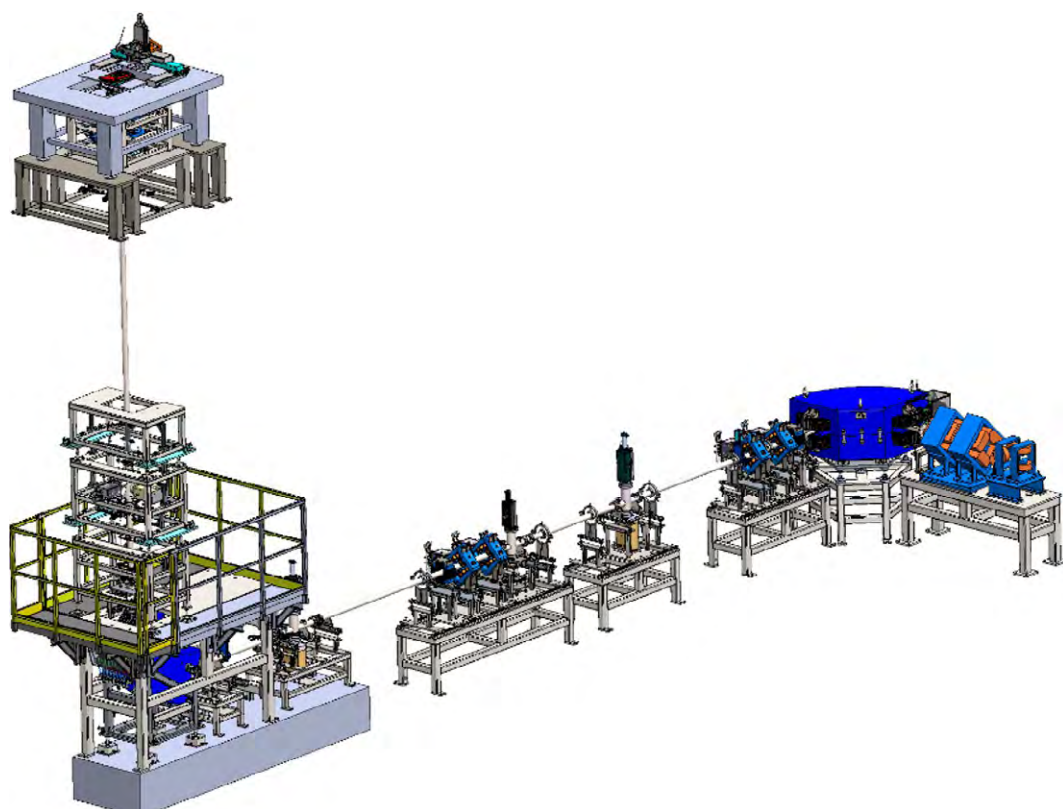
## HIGH ENERGY PROTON MICROBEAM FACILITY FOR RADIOBIOLOGY APPLICATION

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High energy proton dominates the space radiation in the solar system, and long-term exposure induces critical risk not only in astronauts but also to spacecraft electronics. Towards the risk assessment of manned space missions and future lunar bases, a high-energy proton microbeam facility for radiobiology research has been designed and installed at the National Space Science Center (NSSC) of the Chinese Academy of Sciences (CAS). The facility is based on a 50 MeV proton cyclotron which can provide beam current 1-10  $\mu\text{A}$  with energy spread of 1%, and the beam transport through an energy analysis system composed of the pre-microslit, vertical bending magnet and the object slit results in a beam acceptance of  $\sim 3000\text{ }\mu\text{m}^2\text{mrad}^2$  before entering the microbeam system. The microbeam is configured with triplet quadrupole focusing lenses and a post-lens scanning system, and is capable of delivering focused microbeam with 5 micron resolution to targets in air. The bended beamline and special radiation shielding container around the pre-slits are designed for the radiation safety and the reliability of beamline electronics against the secondary radiations. This state-of-the-art facility features an upward-vertical microbeam line, specifically designed for irradiating biological cells, tissues and small animals with online microscopy and single proton hit capability.





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## EXPLORING RADIATION EFFECTS IN BIOLOGICAL SPECIMENS: INSIGHTS FROM CHARGED-PARTICLE MICROBEAMS AND CAENORHABDITIS ELEGANS MODEL

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Charged-particle microbeams (CPMs) provide a unique opportunity to investigate the effects of ionizing radiation (IR) on biological specimens with a precise control on the number of delivered particles per cell. Initially used for targeting subcellular compartments in vitro, CPMs evolved to accommodate three-dimensional models, such as *Caenorhabditis elegans* (*C. elegans*).

In the last years, we conducted two projects based on the selective irradiation of single cells in living organisms with MeV protons.

The first approach involves in situ micro-irradiation of early-stage embryos expressing GFP-tagged proteins at specific cell division phases, observing the radiation-induced damage such as reduced cell mobility, incomplete cell division and chromatin bridges. Three-dimensional models of 2 cell-stage embryo were imported into the Geant4 Monte-Carlo simulation toolkit to investigate the energy deposit in various chromatin condensation states during the cell division phases [1].

The second approach focuses on well-characterized developmental systems in *C. elegans* : the vulva and gonad organogenesis. The consequences of the targeted irradiation of the progenitor gonad stem region were proven through an integration of methodologies. It encompasses micro-irradiation under reversible immobilization of worms, confocal imaging, cell sorting assays, and long-read sequencing analysis. We adapted high-throughput analysis protocols using cell-sorting assay (COPAS) and whole transcriptome analysis to the limited number of worms (> 300) imposed by selective irradiation [2].

In summary, the integration of CPMs with *C. elegans* as a model organism offers a robust framework for studying radiation effects across developmental stages.

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N° 70

## NEW DATA COLLECTION PARADIGMS FOR NUCLEAR MICROPROBES

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Collection, storage and presentation of data has been central in ion microprobe research since inception. The quintessential requirement is that collection of amplitude (energy) data from detectors is maintained in strict synchronism with fast scanning of the beam over a sample.

The ADC is the key interface component between detector(s) and the PC. The demise of NIM electronics means that new solutions need to be sought. Digital signal processors are to some extent able to fill the niche. However, the ~100 MS/s

ADCs and proprietary Field Programable Gate Array (FPGA) pipeline logic architecture<sup>1</sup>, which with data buffering makes pixel synchronisation non-trivial.

Low-cost Micro Controller Units (MCU) with clock speeds exceeding 200 MHz and a microprocessor with a rich set of analogue and digital peripherals. We report success and failures in development of MCU 1- and 2-channel 13-bit ADCs for use in ion beam analysis applications such as microprobes. Different ADC types have been tested including commutating integrators, Trapezoidal digital shaper, time-over-threshold Wilkinson and conventional peak-sensing conventional Wilkinson<sup>1</sup>. The best resolution, linearity and noise resistance using a STM32H7A3 MCU with 260 MHz counter-clock was obtained for a Wilkinson ADC. This was at the expense of a long deadtime, which could be mitigated by using a fast-trigger to flag piled-up pulses.

Synchronism between beam position and ADC-conversion(s) is confounded by of poor time determinacy of USB packet-data. A MCU can overcome this with a special type of anti-coincidence signal (pixel-inhibit and clock) from the control PC which avoids the challenging necessity of maintaining distributed ADC clocks in-phase for time-stamping and a need to unscramble the order of data.

The findings indicate that for microprobes as well as other IBA applications ADCs based on high performance MCUs can offer advantages of cost, flexibility and ease of interfacing.

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Nº 72

## ADVANCING NUCLEAR MICROPROBE ANALYSIS: FROM 2D ELEMENTAL MAPS TO 3D VISUALIZATIONS WITH MACHINE LEARNING

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A nuclear microprobe is an instrument able to focus and scan a beam of charged particles created by a MV electrostatic accelerator. Together with Ion Beam Analysis (IBA) techniques it allows immediate 2D elemental distribution maps providing also elemental depth profile along the ion path, with lateral precision down to the micrometric scale and depth resolution down to tens of nanometer. The goal of this work is to add an extra data dimension to achieve a 3D distribution mapping of elements. This additional dimension is derived from the spectra of Rutherford Backscattering Spectrometry (RBS).

Using OMDAQ software, each scanned area data is acquired as a 256x256 pixel matrix, each pixel containing all the IBA spectra recorded during the experiment. Machine learning algorithms,

as convolutional neural networks (CNN) are capable of processing this volume of RBS data in a reasonable time frame.

Through the classification capabilities of the previously trained CNN, it is possible to create a 3D graphical representation of the elemental distribution in a wide type of samples. This work will present different examples obtained in the Portuguese nuclear microprobe, including the distribution of gold contacts on p-n junction based on GaSb, with applications in thermophotovoltaic cells. This integration of machine learning with nuclear microprobe technology not only paves the way for advanced imaging capabilities but also opens up new horizons for research in complex material systems.

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## MICROBEAM ANALYSIS OF GAS DIFFUSION ELECTRODE FOR METAL-AIR BATTERY

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Metal-air batteries have attracted considerable attention for both mobile and stationary applications due to their exceptionally high theoretical energy density [1, 2] and storage capacity. Consequently, understanding the characteristics of these batteries in attempt to reduce effects of aging holds paramount is important in further developments of these batteries.

In this study, our primary focus was on characterizing, specifically through elemental analysis, the active layer (AL) of the Gas Diffusion Electrode in rechargeable alkaline zinc air batteries [3]. The measurements were carried out at the both microprobe systems of the Ruđer Bošković Institute using beams from either 1.0 MV Tandetron or 6.0 MV HVEC Tandem Van de Graaff accelerators. The first series of experiments were performed on Pristine and aged samples. The samples have been characterized by PIXE and RBS techniques in which 2D elemental maps were created. For each sample measurement, we conducted scans across different regions of interest, with varying scan sizes between 100 and 1000 micrometers.

Subsequently, we analyzed the RBS Spectra using SIMNRA software, while PIXE maps were further processed using PyMca software, a semi-quantitative examination of the 2D elemental maps to correlate the elements. In this presentation we will show results of the first and subsequent series of measurements and discuss important conclusions that microprobe analysis can provide in the characterization of these batteries.

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N° 77

## IN-AIR MICRO-PIXE ANALYSIS WITH A NEW HIGH ENERGY RESOLUTION X-RAY EMISSION SPECTROMETER

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An upgrade of the external beamline at the Microanalytical Center of the Jožef Stefan Institute (JSI) in Ljubljana has been recently completed. The upgraded external beam can now be used complementary to microprobe beamline to perform in-air micro-PIXE imaging over large sample surface area ( $\sim 1 \text{ cm}^2$ ) with  $\sim 50 \text{ }\mu\text{m}$  lateral resolution [1]. Within this upgrade, a new parallel-beam wavelength dispersive (PB-WDS) X-ray emission spectrometer has been constructed and installed to the beamline [2]. The spectrometer employs polycapillary X-ray optics to increase the solid angle of X-ray collection and convert divergent emission into a collimated X-ray beam, which is diffracted by a flat crystal analyzer yielding energy resolution  $dE/E$  of  $\sim 1\text{--}4 \times 10^{-3}$  and sensitivity at the level of few tens of ppm. The spectrometer is used complementary to the energy dispersive X-ray detectors used conventionally in micro-PIXE analysis. Its high energy resolution is used primarily to enhance the sensitivity and improve the contrast of the measured maps in case of close lying overlapping spectral components in the EDS recorded PIXE spectra, particularly within the tender X-ray range. Recently, we have demonstrated that the energy resolution of the new spectrometer is sufficient to provide even chemical state sensitivity and perform chemical speciation. In this contribution, a brief description of the instrument is given followed with the main results of characterization measurements yielding quantitative analysis of main operational parameters. Finally, the first examples of applications for high energy resolution in-air micro-PIXE mapping are presented, exploiting both spatial and energy resolution of the new set-up.

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N° 78

## TUDY OF THE DETERIORATION OF BIODEGRADABLE PLASTICS IN SEAWATER

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Among the most synthetic aliphatic-aromatic copolyesters with biodegradation properties, Poly Butylene Adipate-co- Terephthalate (PBAT) seems to be promising and popular by its potential development and applications [1] for CE incorporating them as filler in waste seashells during recycling, and food packaging [2, 3] or for palliating marine plastic pollution [4]. On the other hand, non-synthetic starch-based bioplastics extracted from plants such as Yucca are not lagging behind either in these application fields [4, 5]. In this work biodegradability in seawater of PBAT and commercial Yucca's starch-based bioplastic (YSB) was studied by different techniques such as  $\mu$ PIXE at the Internal Microbeamline from CMAM [6], after being subjected to different external factors in order to unveil heterogeneous elemental distributions of impurities in this kind of samples. Results obtained lead to a gradual degradation process potentially influenced by the polarity of the materials in which YSB showed accelerated degradation compared with PBAT. Although YBS structure is unknown, more hydrophilic surface is expected because of larger amount of OH- groups that characterize amylose and amylopectin of starches.

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## EFFICIENT GENERATION OF OPTICALLY ACTIVE DEFECTS IN DIAMOND THROUGH HOT MEV MICROBEAM IMPLANTATION

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The Nitrogen-Vacancy (NV) center in diamond is considered among the most promising platforms for the implementation of quantum technologies due to its optically addressable spin state at room temperature and its long coherence time<sup>1</sup>.

Quantum sensing, as one of the fields in which this center is being highly considered, requires a high sensitivity to external fields<sup>2,3</sup>. A promising strategy for the achievement of this sensitivity relies on the

exploitation of large ensembles of NV centers, whose fabrication by ion implantation is limited by the radiation damage induced in the diamond lattice.

In this work, we report on the Raman spectral and photoluminescence analysis of NV centers at ensemble level implanted upon 2 MeV nitrogen ions at several temperatures, including room temperature, 500°C and 750°C. We demonstrate an approach to increase the density of NV centers upon high fluence implantation, showing how with respect to room temperature, the high-temperature process increases the threshold in the ion fluence at which the diamond crystal is amorphized and is converted upon a thermal annealing process into a graphitic phase. Moreover, we describe the observation of an increment in the formation efficiency of the NV centers implanted at high temperature with respect to the traditional room temperature implanted and subsequent annealing ones, by performing several post-implantation annealing treatments to determine the difference between the in-situ annealing and room temperature implantation.

Additionally, to compare these results on the formation efficiency with other color centers in diamond, we carry out a similar investigation on MgV centers<sup>4</sup> created by ion implantation of 2 MeV Mg<sup>2+</sup> ions with in-situ annealing (50 – 700 °C). The yield is assessed by means of PL intensity measurements and resulted in an increase up to 14 % with respect to traditional room temperature ion implantation and subsequent thermal annealing.

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N° 84

## HOLLOW CYLINDRICAL MICROSTRUCTURE FABRICATED BY PROTON BEAM WRITING FOR PHOTONIC NANJET APPLICATIONS

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Photonic nanjets (PNJs), a sub-wavelength focusing non-evanescent light, have been reported as a promising optical phenomenon to overcome the limit of optical resolution<sup>1</sup>. After Chen et al. first introduced the PNJs by theoretical approach in 2004<sup>2</sup>, research studies on the PNJs formation have been theoretically realized across different microstructures<sup>3-5</sup>.

However, the experimental investigation is still challenging and depends on the fabrication techniques of microstructures. Proton beam writing (PBW) has been known as a high-aspect-ratio lithographic technique and provides smooth surface structures. Recently, we applied PBW to create microcylinders that successfully formed PNJs through the axial axis<sup>6</sup>. The design of the microstructure primarily governs the PNJ characteristics and is therefore needed to further investigate. In this work, the poly(methyl methacrylate) (PMMA) hollow cylindrical microstructures were produced by 1.0-MeV PBW on the silica substrates. After that, PNJs were observed by the confocal laser microscope<sup>7</sup> in an atmospheric environment. The PNJs were successfully formed by these double air-PMMA microcylinders and were characterized and compared with our previous work. The fabrication process and forming PNJs will be presented and discussed together with the application prospects.

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Nº 93

## AUTOMATED TARGETING OF SINGLE CELLS WITH FOCUSED PROTONS

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Proton beam therapy has been gaining popularity globally, owing to its superior precision in targeting tumors that mitigates damage to surrounding healthy tissue. In Singapore, three new proton beam treatment centres currently in operation are poised to cater to the escalating demands of an aging population with the anticipated surge in cancer incidences. In order to advance the current understanding in radiation research in the context of cellular responses, we have developed a method



to automatically target single cells for irradiation with focussed protons. The extended degree of automation we have implemented at the beam line enables the delivery of controlled numbers of protons to up to thousands of cells rapidly with improved targeted precision. Selective cell irradiation using this approach enables multiple possibilities in the study of the bystander effect for example, while simplifying the tracking and monitoring of irradiated cells and their non-targeted counterparts by eliminating the need for nuclear track detectors. This technique, which combines machine learning integration, in addition to instrumentation, software and procedural upgrades, has the adaptable potential for selective targeting of subcellular compartments, facilitating high resolution radiobiology investigations. This enhanced capability is instrumental in advancing our comprehension of organelle responses and their distinct contributions to the overall cellular reaction to radiation.

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## IN SITU ANALYSIS OF INFLAMMATION DETECTION WITH GOLD NANOSHELLS BY RUTHERFORD BACK SCATTERING AND PARTICLE INDUCED X-RAY EMISSION

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Optical coherence tomography (OCT) is a high-resolution optical imaging technology that is substituting other conventional cardiovascular diagnostic procedures (ultrasound, angiography). Today, OCT is used as a minimally invasive technique to detect and assess atherosclerotic plaques in vivo and to guide intervention procedures such as atherosclerotic surgery and stent placement [1]. Despite its high resolution, the lack of molecular-scale information available in OCT images makes the use of contrast agents a step-forward in the implementation of this technique. For this purpose innovative strategies like functionalized nanoparticles are being used to achieve molecular level contrast by targeting specific biomarkers. Proteins overexpressed by cells that are undergoing inflammatory processes are particularly interesting, as they are characteristic of the early stages of the atherosclerotic plaques development.

Gold nanoshells (GNSs) functionalized with inflammation-targeting molecules have already been used as OCT contrast agents, and their specific adhesion to inflamed endothelial human cells has been demonstrated in vitro [2]. However, the translation of these experiments to tissues is challenging, since the presence of gold on the tissue can go undetected if the OCT signal provided by the GNSs is similar to the one provided by tissues, making the optimization of the experimental conditions challenging. In this work we have addressed this problem by determining the gold presence on inflamed rat-aorta tissue after incubation with functionalized-GNSs using Rutherford back scattering (RBS) and Particle Induced X-ray Emission (PIXE) with a 2 MeV proton beam. The technique has demonstrated the presence of gold undetected by OCT, and thus can be used as a guide towards a better understanding of the

OCT detection of GNSs. The present study represents an important step forward for the use of contrast agents to target inflammation in the first stages of atherosclerosis by OCT.

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N° 97

## DEFECT ENGINEERING IN 2D MATERIALS USING FOCUSED MEV IONS

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Spin defects in 2-dimensional (2D) materials have emerged as important candidates for various quantum applications such as magnetic sensing and single photon emission. In materials such as hexagonal boron nitride (hBN), numerous emitters have been identified, many of which have stable bright emission at room temperature. The nature and origin of some of these emitters is still a subject up for discussion and many of the defects that are responsible for the observed emission are unknown. In this work we systematically generate spatial patterns of quantum emitters in various 2D materials using focused beams of light (protons and alpha particles) mega-electron volt (MeV) ions. The advantage of using MeV ions for these studies is that the beam spot resolution is maintained in these ultra-thin materials and the damage induced is predominately due to electronic energy loss which results in a well-defined point defect structure. We characterize their optical properties using techniques such as photoluminescence and Raman imaging, and study their quantum sensing properties and single photon emission using optically detected magnetic resonance (ODMR) and photon anti-bunching measurements. Transmission electron microscopy is used to directly image irradiated samples in order to generate statistical distributions of the atomic defects that are created. The combination of these characterization techniques help to identify candidate defects that are responsible for the observed optical properties.

N° 101

# HIGH-SENSITIVITY PEROVSKITE-BASED RADIATION DETECTORS FOR PROTON BEAMS

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Cancer radiotherapy with high-energy proton beams has expanded rapidly in recent decades. The rising demand for accurate dose control in radiobiological studies and proton therapies has led to extensive research into proton detectors [1]. Ideally, the detectors should be capable of quantifying the number of protons during irradiation in real-time, at the single proton level. A strategy is to fabricate high-performance thin-film detectors, which are transmissive to protons and allow simultaneous proton counting during radiotherapy. However, the detectable signal is fundamentally limited by the thickness of the detector. As a result, a proton-transmissive detector must be manufactured at an ultrathin thickness while retaining single-proton detection sensitivity. Existing particle detectors, such as ionization chambers, silicon-based detectors and single-crystal scintillators, are too bulky to transmit protons. Organic plastic scintillators suffer from low scintillation yields and low particle radiation tolerances due to their low electron density [2]. Organic thin-film devices based on proton-induced currents are limited by low charge collection efficiency, which hinders single-proton detection sensitivity [3].

In this talk, I will present our recent work on proton-transmissive thin scintillators made from all-inorganic perovskite nanocrystals  $\text{CsPbBr}_3$ , designed for real-time single proton counting [4]. These perovskite scintillators exhibit exceptional sensitivity, with a high light yield ( $\sim 10^5$  photons per MeV) when subjected to proton beams. This enhanced sensitivity is attributed to radiative emission from biexcitons generated through proton-induced upconversion and impact ionization. The combination of rapid response ( $\sim 336$  ps) and pronounced ionic-stability enables diverse applications, including single-proton tracing, patterned irradiation, and super-resolution proton imaging. These advancements have the potential to improve proton dosimetry in proton therapy and proton imaging in radiography. In addition, I will also show our latest results of single-crystal perovskite detectors for high-sensitivity proton detection.

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N° 102

# USE OF A 100 KEV PROTON MICROPROBE FOR THE DETERMINATION OF STANDARDIZED SEMICONDUCTOR RADIATION HARDNESS PARAMETERS.

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In March 2023, guidelines for the measurement and interpretation of the Charge Collection degradation in semiconductors caused by the displacement damage induced by ion irradiation were published on the IAEA technical report series [1]. The proposed methodology relies on the use of MeV ion microbeams both to induce defects in selected regions of a semiconductor diode and to probe the damage in the irradiated regions by the Ion Beam Induced Charge (IBIC) technique.

In this paper, we describe an experiment carried out at the Ion Implantation Laboratory of the Physics Department of the University of Torino, for the assessment of the radiation hardness of a silicon photodiode, adopting the same protocol but using 100 keV proton collimated beam as probing ions.

The device under study in this experiment was a commercial p-i-n photodiode; regions of (100 × 100) μm<sup>2</sup> were irradiated with different ions (He, Li, C, with energies ranging from 2 to 4 MeV) in the fluence range of (1-300)·10<sup>10</sup> ions/cm<sup>2</sup>.

IBIC maps at different bias voltages were then acquired by the raster movement of the diode with respect to the 100 keV proton beam collimated down to a 10 μm spot. These maps show the increase of the CCE contrast of irradiated/pristine regions as function of the damaging ion fluence. The CCE degradation was then analysed and interpreted through the model reported in [1] to evaluate the standardized parameters, which quantify the radiation hardness of the semiconductor material.

The use of 100 keV protons as probing beams was proven to be suitable to provide the measurement of the damage parameter relevant to a single charge carrier (electrons in this case), avoiding the cross-correlation between the minority and majority carriers already evidenced in the IAEA guidelines. In parallel, the experiment demonstrates that the IAEA guidelines can be adopted also using keV collimated protons, paving the way to the application of the protocol also to conventional ion implantation set-ups.

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# AN AUTO-FOCUSING SYSTEM FOR NUCLEAR MICRO-PROBES – BASED ON REAL- TIME IMAGE PROCESSING AND NUMERICAL OPTIMIZATION

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Manually controlled beam focusing for a typical nuclear-microprobe, such as the Oxford setup [1], is typically achieved by an operator adjusting the currents supplied to a series of quadrupole magnets. During adjustment, the operator must observe the resulting changes in size and shape of the beam spot via an optical microscope image of a fluorescent screen, on which the beam is incident. Through iterative adjustment, the operator reduces the size of the beam spot, whilst maintaining the lengths of the major- and minor-axis as equal as possible. This process requires a skilled operator to implement and, even then, can be slow. The current work demonstrates an automated solution to ion-beam focusing, based on real-time image processing of the microscope images. The image processing outputs provided feedback to a minimisation routine based on the downhill-simplex algorithm [2], which controls the currents supplied to the quadrupole focusing- magnets. This automated system is run from a professional level software package, implemented on the scanning light-ion microprobe [3], at the Tandem Laboratory [4]. The software also provides full camera control, manual-control of various parameters when needed, and a host of analytical outputs. The system consistently achieves the optimal beam-spot size in less than three minutes: further optimisation, by experienced human operators, is not achievable. The software is written in the Python programming language, is based almost entirely on open-source Python modules and is available on GitHub. This contribution will present an overview of the software and its functionality, along with experimental results benchmarking its capabilities.

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N° 113

## DEVELOPMENT OF A COMPACT MAGNETIC BACKSCATTERED ION BEAM DEFLECTOR SYSTEM FOR LIGHT ELEMENT MICROSCOPY

Bowen, C. (1); Byers, T. (2); Kaur Saini, D. (1); Sharma, M. (1); Parashar, M. (1); Rout, B. (1); Glass, G. (1)

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Improvements made to ultra-thin windows for X-ray detectors in recent years have allowed the detection of elements as light as lithium, however, in-vacuo microscopy application of proton-induced X-ray emission spectroscopy ( $\mu$ PIXE), has been limited to the quantification of elements due to the necessity preventing high-energy backscattered protons from damaging the detector. Methods used to eliminate, or significantly reduce, damage from these backscattered protons present significant limitations to the efficient detection of low-energy X-rays produced by low-mass constituents in the sample, thus precluding the trace quantification of those elements. A new technique developed at the UNT Ion Beam Laboratory allows the quantification of trace amounts of light elements in-vacuo.

Proton beams of 1 MeV were produced using a National Electrostatics Corporation 3 MV 9SH Pelletron® accelerator and focused to approximately  $2\ \mu\text{m} \times 2\ \mu\text{m}$  using a separated magnetic quadrupole focusing system. This probe was scanned over areas of approximately  $500 \times 500\ \mu\text{m}^2$  for selected samples mounted on a manually controlled 3-D stage secured inside a vacuum chamber maintained at a pressure of  $10^{-7}$  Torr. X-ray spectra were detected using an Amptek 70 mm<sup>2</sup> X-123 Fast Silicon Drift Detector (SDD) with a C2 entry window constructed of a 30 nm layer of Al on a 40 nm Si<sub>3</sub>N<sub>4</sub> supporting layer. The solid angle of acceptance for the detector was 3.4 msr. Elemental concentrations were obtained using GeoPIXE.

The key innovation of this system consists of a compact magnetic deflector that is installed inside the vacuum chamber between the the target and the entrance window of the X-ray detector without compromising detection efficiency or solid angle. Importantly, the in-vacuum capability allows the simultaneous application of complementary analysis techniques. We will present ion beam microscopy analysis of several light elements present in organic and inorganic samples.

N° 115

# NANOSCALE ION IMPLANTATION AND NANOPILLARS INTEGRATION OF GeV COLOR CENTERS INTO A DIAMOND MEMBRANE

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Diamond color centers are solid-state single-photon sources with optical properties suitable for quantum technologies applications. Color centers related to Group-IV impurities are experiencing an increasing relevance thanks to their narrow emission linewidth at room temperature, robust optical coherence and one of the shortest lifetime among the known solid-state color centers<sup>1, 2</sup>.

A key enabler for the technological uptake of these emitters consists in their deterministic fabrication and subsequent characterization. Ion implantation technique offers the highest spatial accuracy in the positioning control of the fabricated defects. Moreover, it is crucial to evaluate the conversion yield of the implanted ions to luminescent complexes within the diamond lattice to ensure the formation of single emitters at specific implantation sites.

We report on a systematic characterization of GeV single emitters fabricated upon nanoscale ion implantation and integrated in diamond nanopillars. A diamond membrane sample was irradiated in two different regions with Ge<sup>++</sup> ions respectively at 70 and 35 keV energies, employing a Focused Ion Beam (FIB) with ~25 nm spatial accuracy. The implantation was performed according to regular patterns with 3 μm spacing. Different ion fluences were considered, ranging from 50 to 150 ions per implanted spot. Then, the implanted regions were integrated into nanopillars to enhance the photoluminescence collection. A thermal annealing (1000 °C, 2 hours) was performed to achieve the conversion of the implanted species into optically active centers. The implanted spots were systematically characterized in photoluminescence confocal microscopy to estimate the formation efficiency of single emitters. The achieved results were compared with those discussed in literature<sup>3</sup>.

This study is helpful in defining a fabrication protocol relying on ion implantation and nanofabrication that guarantees the formation of stable single photon emitters, which represents the main challenge in the perspective of exploiting these systems for scalable quantum photonic devices.

## References

- <sup>1</sup> T. Iwasaki, et al. Sci Rep. 5 (2015), 12882
- <sup>2</sup> C. Bradac, et al. Nat Commun. 10 (2019), 5625
- <sup>3</sup> Y. Zhou, et al. New J. Phys. 20 (2018), 125004

N° 117

## GAS RELEASE FROM WHITE PAINT LAYERS UNDER PROTON BEAM: TOWARDS ON-LINE MONITORING OF SENSITIVE HERITAGE TARGETS DURING FOR PIXE ANALYSES

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The preservation of heritage objects during IBA is a major concern requiring both the understanding of the effects of the analytical beam and the determination of safe exposure limits. Lead white, the most common white pigment employed in historical painted works, is prone to physical and chemical modifications by ions. Our objective is to investigate these changes to alert at an early stage and to prevent undue modifications. The measurement of target outgassing might provide an on-line marker of the modifications, possibly more efficient than the measurements of faint changes in the bulk material. Mock-ups of lead white components (cerussite  $\text{PbCO}_3$  and hydrocerussite  $2(\text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2)$  mixed with linseed oil and egg yolk binders were exposed to fluences from 10 to  $40 \mu\text{C} \cdot \text{cm}^{-2}$  of 3 MeV protons, corresponding to routine values employed in PIXE analysis. A coupon taken from a lead-white bearing area in 18th c. original painting was irradiated in the same conditions. The gas release was measured using different techniques in two environmental conditions: in vacuum using a residual gas mass analyzer, and in a neutral atmosphere ( $\text{He}$  and  $\text{N}_2$  in an irradiation cell using FTIR and GEN-LIBS<sup>1</sup> techniques. Mass spectrometry allowed to identify the released gases, while FTIR and LIBS through the cell windows allowed to quantify their concentration ( $\text{CO}$ ,  $\text{CO}_2$  and H-bearing gases), by comparison with the injection of controlled microvolumes of reference gas. The results confirmed an important release (microliters) of hydrogen from the two binders and from hydrocerussite and showed the dissociation of both lead carbonates ( $\text{CO}$  and  $\text{CO}_2$ ). These results are foreseen to be exploited in the development of on-line monitors for the PIXXL beam line dedicated to the analysis of big heritage artifacts under construction at the AGLAE facility.

<sup>1</sup> Laser-induced breakdown spectroscopy of gas enhanced by solid initiator

### References

- 1 Measurement of hydrogen release of cultural heritage materials during ion beam analysis using Laser-induced breakdown spectroscopy of gas enhanced by solid initiator (GENS-LIBS), <https://doi.org/10.1063/5.0179543>
- 2 Practical advances towards safer analysis of heritage samples and objects, <https://doi.org/10.1016/j.trac.2023.117078>

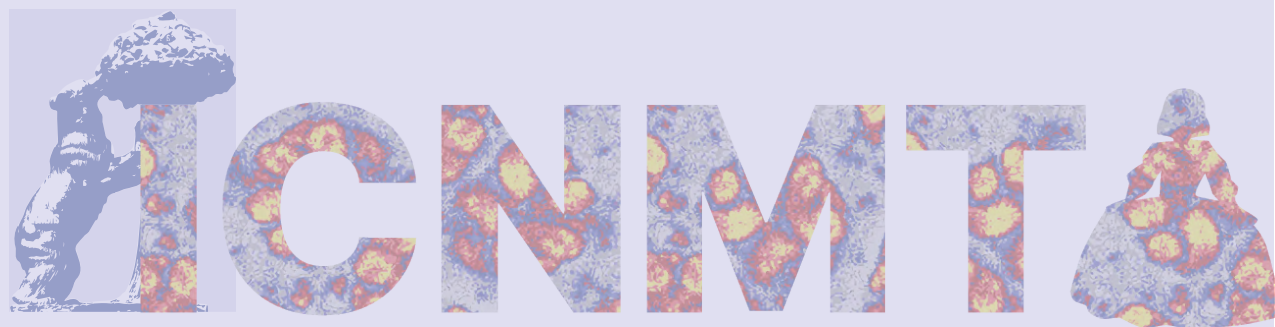


N° 121

## MINIBEAM BEAMLINE FOR PRE-CLINICAL EXPERIMENTS – MINIBEE

Reindl, J. (1); Kourkafas, G. (2); Bundesmann, J. (2); Denker, A. (2); Dittwald, A. (2); Dollinger, G. (3); Neubauer, J. (3); Rousseti, A. (3)

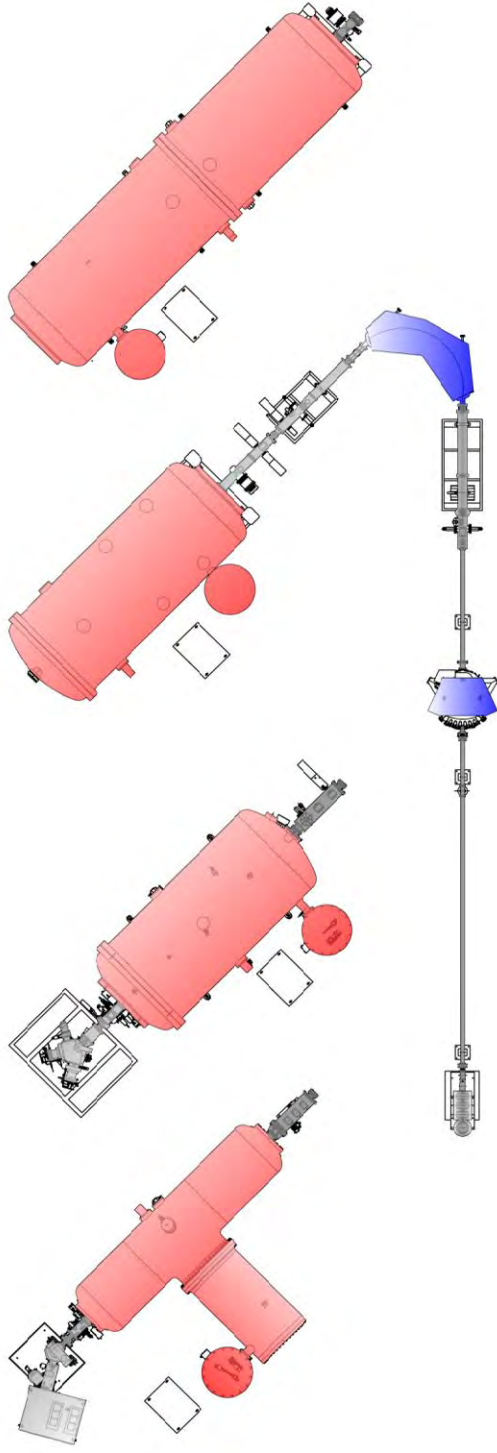
(1) University of the Bundeswehr Munich; Germany; (2) Helmholtz Zentrum Berlin; Germany; (3) University of the Bundeswehr Munich; Germany Compared to classic proton therapy, proton minibeam radiation therapy (pMBT) further spares normal tissue. To fully study this potential with small animal experiments focused minibeam with a  $\sigma$  of 50  $\mu\text{m}$ , a beam current of 1 nA and approx. 4 cm proton range (water) is needed. The MiniBEE located at the Helmholtz-Zentrum Berlin, is designed to fulfill all requirements of researchers for systematic studies on pMBT. The maximum beam energy of 68 MeV can be further reduced to approx. 35 MeV by a first degrader in vacuum after the cyclotron. A second degrader placed close before the target further reduces the energy, forming a spread-out Bragg peak in the target. Along the beamline, various slits shape the transverse beam profiles. A high magnetic field gradient triplet lens focuses the beam on the target while scanning magnets raster scan it over the target. A small animal radiation research platform (SARRP) is used for positioning and imaging of animals, it can be supplemented by an optical microscope to study cells, tissue but also scintillation targets. The Beamline can be used for magnetically focused but also collimated applications, together with therapy planning and a single field size of  $\sim 4 \text{ mm} \times 4 \text{ mm}$ , applied using beam scanning.



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## Research Infrastructure Access in Nanoscience & Nanotechnology

### Access to top-level research infrastructures through a single-entry point

Noelia Benito<sup>1\*</sup>, Gastón García<sup>1\*</sup>, Andrés Redondo<sup>1,2</sup>, Nuria Gordillo<sup>1,2</sup>, Jorge Álvarez<sup>1</sup>, Michael Stuckelberger<sup>3,4</sup>

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

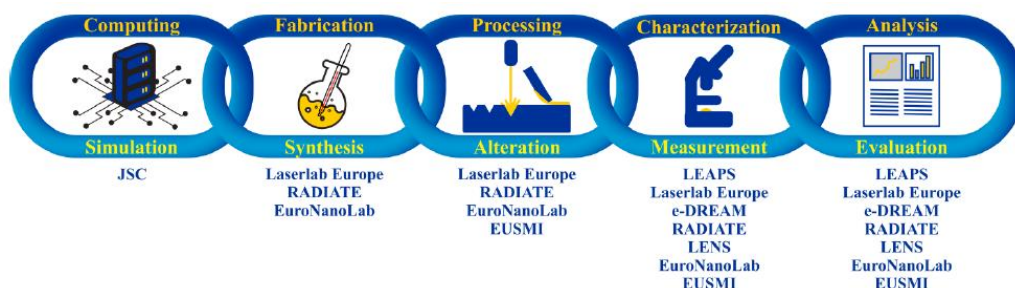
<sup>2</sup> Universidad Autónoma de Madrid, Departamento de Física Aplicada, Madrid, Spain

<sup>3</sup> Deutsches Elektronen-Synchrotron DESY, X-Ray Nanoscience and X-Ray Optics, 22607 Hamburg, Germany

<sup>4</sup> on behalf of the RIANA project team

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RIANA is a Horizon Europe funded project which supports curiosity-driven research in nanoscience with open research questions for long-term impact, and challenge-driven research in nanotechnology with targeted research questions for short- and mid-term impact. A consortium of 7 European networks of top-level research infrastructures (RI) to cover the most advanced techniques relevant for synthesis, nanofabrication, processing, characterization, analytics, as well as simulation capacity. RIANA offers access to simply the best research infrastructures for nanoscience and nanotechnology [1]



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More details of the project, and how to apply, can be found at: <https://riana-project.eu/>.

#### References:

[1] RIANA proposal - grant agreement No. 101130652

[2] [RIANA-project.eu](https://riana-project.eu/)



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# a hub for materials research

**Circular materials characterisation and recycling process optimisation: free support to academia and industry from European project ReMade@ARI**

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ReMade@ARI aims to boost the circular economy by helping research into recycling and recyclable materials through support and assisted access to Europe's foremost analytical research infrastructures.

The project is funded with over 15 million EUR from Europe, UK and Switzerland; it started on 01 September 2022 and will run for 4 years. The coordinator is HZDR and there are 48 partners providing access to synchrotrons, neutron sources, laser facilities, electron microscopes, ion and positron beams, and high magnetic field facilities.

The project offers assistance that goes beyond the usual services offered at research infrastructures. A network of over 150 instrument scientists will guide projects from the initial idea, to selection of complimentary techniques at over 50 facilities across Europe. A team of 18 junior scientists has been employed by the project to assist users with materials characterisation and experiments at the facilities. Training can be requested to help new users begin their first experiment or for existing users to learn new techniques.

There are three access methods provided by ReMade@ARI:

- **ReMade-TNA:** Transnational access is open to researchers from academia and industry. A pre-proposal can be submitted to obtain a review of feasibility and guidance on suitable techniques. Users are expected to request two or more techniques. As this is EC funded TNA, there is an obligation to request facilities in another country, and results have to be reported/published. Access to the facilities is free and travel expenses will be covered by ReMade@ARI.
- **ReMade-SME:** Small and medium enterprises can benefit from support for fast experiments. Additional support will be provided for data analysis. There is no obligation to report/publish results, and a local facility can be requested. Mail-in services will be prioritised. This is an opportunity to test a particular technique or carry out a first experiment before submitting a more complicated project via other tracks (TNA or IND).
- **ReMade-IND:** Any industry can apply in collaboration with a knowledge provider such as a Research and Technology Organisation (RTO) or an academic expert. The knowledge provider will help to translate the industry problem into experiments at the facilities. A grant of 30,000 EUR per project is available to cover costs associated with the experiments. IND can be combined with ReMade-TNA for free access, or the industry partner can pay for confidential access to facilities to ensure retention of IP.

We expect access to range from routine materials analysis by sample mail-in to intricate *in situ/operando* experiments involving dedicated sample environments. A single application through the ReMade@ARI portal can request experiments at multiple sites across Europe.

More details of the project, and how to apply, can be found at: [remade-project.eu](http://remade-project.eu).

# Funded by the European Union as part of the Horizon Europe call HORIZON-INFRA-2021-SERV-01 under grant agreement number 101058414 and co-funded by UK Research and Innovation (UKRI) under the UK government's Horizon Europe funding guarantee (grant number 10039728) and by the Swiss State Secretariat for Education, Research and Innovation (SERI).

Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or the UK Science and Technology Facilities Council or the Swiss State Secretariat for Education, Research and Innovation (SERI). Neither the European Union nor the granting authorities can be held responsible for them.



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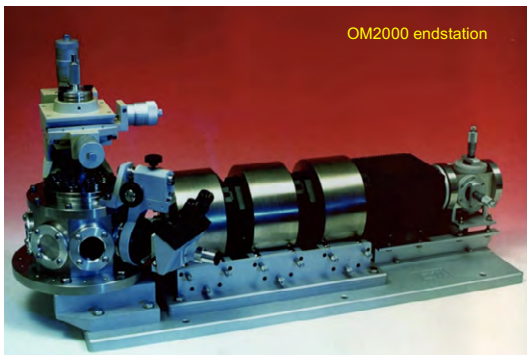
### Application areas:

- PIXE and PIXE mapping
- RBS and RBS mapping
- Proton Radiobiology
- MeVSIMS mapping
- Targeted single ion irradiation
- Proton beam writing
- External beams for cultural heritage studies
- Scanning Transmission Ion Microscopy
- Heavy ion microbeams (to 150 MeV.amu/q<sup>2</sup>)
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- Ion Beam Induced Charge microscopy

### Performance of OM systems:

**OM2000 Oxford triplet endstation:**  
**300 x 450 nm at 50pA** (2MeV protons).  
**900 x 900 nm at 1 nA** (2.5MeV protons)

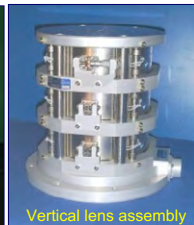
**Oxford triplet configuration of OM-52 lenses:**  
Spot sizes of **20 x 25nm** have been achieved for low current applications, and **20nm** high aspect ratio structures have been written using proton beam writing.



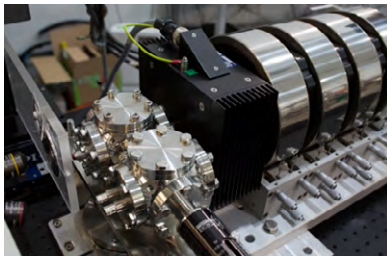
OM2000 endstation



OM-52 Miniature triplet



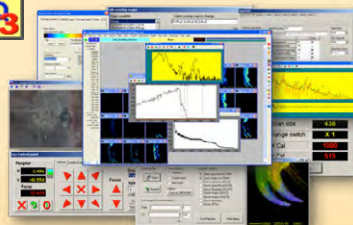
Vertical lens assembly



Radiobiology  
Endstation using  
OM post lens  
scan coil and  
Oxford triplet  
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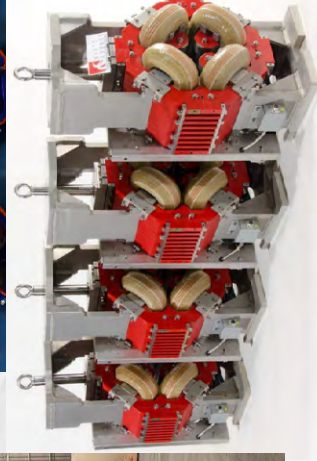
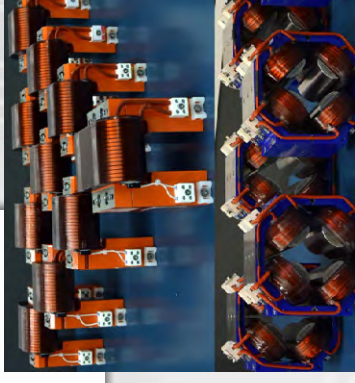
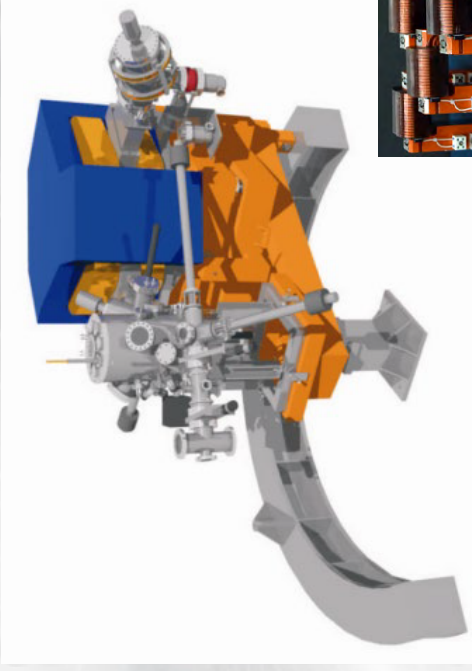


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