# Applied Nuclear Physics Conference



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# **Nuclear Physics Institute** of the Czech Academy of Sciences

NPI of the CAS, public research institution, conducts research in a broad field of nuclear physics, experimental as well as theoretical.



The properties of nuclear matter under the heavy ion collisions at high and intermediate energies, nuclear reactions important for astrophysics or power plants, beta decays of atomic nuclei including the problem of neutrino masses are especially studied in the NPI. The nuclear theory is oriented to nuclear structure, hypernuclei, interactions of elementary particles with nuclei, or mesonic degrees of freedom in nuclei.

NPI uses neutron scattering, mainly in the solid state physics and material research. A large complex of nuclear analytical methods based on charged particles and neutrons beams is used in the interdisciplinary research in collaboration with external specialists in chemistry, ecology, medicine, archaeology etc.

The dosimetry of ionizing radiation is oriented to the measurements of environmental and professional expositions, metrology, and biophysical aspects as DNA radiation damages. The research and development of radiopharmaceuticals, especially short lived positron emitters for the positron emission tomography (PET), is performed at the NPI, as well as physics for perspective methods of nuclear energetics like accelerator driven transmutation of nuclear waste.





#### Center of Accelerators and Nuclear Analytical Methods (CANAM)

Center of Accelerators and Nuclear Analytical Methods (CANAM infrastructure) offers to scientists and industry a unique experimental infrastructure in nuclear physics and neutron science. It comprises 3 major research laboratories of the Nuclear Physics Institute of the ASCR:

Laboratory of Tandetron (LT) Neutron Physics Laboratory (NPL)

operating an accelerator Tandetron 4130 MC providing facilities at the reactor LVR-15 Laboratory of Cyclotron and Fast Neutron Generators (LC & FNG)

operating the isochronous cyclotron U-120M

With funding from the Ministry of Education, Youth and Sports of the Czech Republic (project No. LM2011019) and Nuclear Physics Institute of the ASCR, experimental facilities of the CANAM infrastructure are proffered to the users in Open Access mode.



Nuclear Physics Institute of the ASCR public research institution



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### Laboratory of Tandetron (LT)

The Tandetron 4130 MC is a compact electrostatic tandem accelerator for production of ion beams with energies in the range 400 keV - 24 MeV of almost all elements of the periodic system.



The Tandetron accelerator and adjacent ion beam lines

#### LT offers a set of nuclear analytical methods

- RBS (Rutherford Backscattering Spectrometry)
- ERDA (Elastic Recoil Detection Analysis)
- TOF-ERDA (Time-of-Flight ERDA)
- PIXE (Particle Induced X-Ray Spectroscopy)
- PIGE (Particle Induced Gamma-ray Spectroscopy)
- PESA (Proton Elastic Scattering Analysis)
- NRA (Nuclear Reaction Analysis)
- RBS-channeling
- Ion implantation
- Ion microprobe
- External ion beam



## Laboratory of Tandetron (LT)

## RBS

- high sensitivity for heavy elements
- low detection limits (10<sup>11</sup>-10<sup>12</sup> at.cm<sup>-2</sup>)
- about nm depth resolution in elemental depth profiling

# **RBS channeling**

- channeling of charged particles in crystalline matrix
- structural studies

### **ERDA, TOF-ERDA**

· low detection limits for light elements

# **PIXE, PIGE and PESA**

- light elements analysis
- low detection limits down to 1 ppm (PIXE) and 1000 ppm (PIGE)

### **External beam**

 facility is necessary for the study of samples, which are unstable under vacuum conditions, or too large to be mounted in a vacuum chamber

## Ion Micro-beam

- focusing of ion beam with a spot size down to 0.5 µm and different scanning modes over the sample
- a versatile equipment that allows
  3D elemental mapping
  of microstructures











# Book of abstracts of the Applied Nuclear Physics (ANP) conference 2021

1<sup>st</sup> edition, September 2021

#### PUBLISHED AND DISTRIBUTED BY

AMCA, spol. s r.o. Vyšehradská 320/49 128 00 Prague 2, Czech Republic

#### **PRINTED BY**

Ivana Jakubcová Býkovice 679 71 Lysice Česká republika IČ: 76414248

ISBN 978-80-88214-25-0

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# **GENERAL INFORMATION**

**DATE** September 12 – 16, 2021

#### **CONFERENCE VENUE**

Faculty of Architecture, Czech Technical University Thákurova 9 166 34 Prague 6 Czech Republic

# ORGANIZER

The Conference is organized by

Nuclear Physics Institute of the Czech Academy of Sciences in cooperation with Nuclear Physics Board of EPS, Center of Accelerators and Nuclear Analytical Methods of NPI, Charles University, Institute of Nuclear and Particle Physics, and Faculty of Nuclear Sciences and Physical Engineering, Czech Technical University in Prague.

The main organizer is Tandetron Laboratory of CANAM NPI, Rez, Czech Republic.











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# NUCLEAR PHYSICS IN MEDICINE

#### APPLIED NUCLEAR PHYSICS AT NEW PARTICLE ACCELERATORS

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Several new large-scale particle accelerators are under construction all around the world (e.g. FAIR, SPIRAL2, ELI, SPES, SEEIIST in Europe; FRIB in USA; NICA in Russia; RAON in Korea). These machines offer new opportunities including high intensity, high energy, use of different ions, radioactive ion beams, ultra-short pulses. Many of these accelerators have cost in the range of  $10^9 \in$ , and the primary science objective is basic nuclear physics. However, most of them include programs in applied nuclear sciences, especially biomedical research. Applications to energy, medicine, materials, space, security and environment are indeed the fields with the largest expansion potential in nuclear physics [1]. They also give a social justification for the high investment cost of the new facilities. At the Facility of Antiprotons and Ion research (FAIR) in Darmstadt [2], applied physics has been always part of one of the four pillars (APPA [3]). The necessity to transform the FAIR Biophysics Collaboration into a truly International, worldwide collaboration stems from the interest in the same activities in many new facilities. An International Collaboration, linking the different new facilities, can guarantee the quality of the science and the complementarity of the activities. Even if the scheme is similar to the traditional highenergy physics collaborations, the Biophysics Collaboration can cover many different research topics, and therefore, rather than competition to reach first a specific goal, this The opportunities of the new accelerators can lead to breakthrough advances in space radiation research (where high energy is necessary) and medical physics (including particle radiography, minibeam radiotherapy, very high dose rate therapy, use of radioactive ion beam) and production of new radionuclides for medicine [4].

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#### PARADIGM SHIFTING OF MICRODOSIMETRY IN PARTICLE THERAPY

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The rationale for using ions in radiotherapy is supported by both physical and radiobiological advantages. Although clinical results have been encouraging, numerous treatment uncertainties remain major obstacles to the full realization of charged particles. One of the critical aspects emerging from medical experience is the lack of understanding of the relationship between dose delivered and corresponding biological effects. All existing treatment planning systems combine physical (e.g. absorbed dose or Linear Energy Transfer LET) and biological (Relative Biological Effectiveness RBE for cell killing) quantities to calculate the biological dose delivered in the treatment. Inaccurate determination of either the dose or the RBE can lead to an underdosage to the tumor, limiting treatment success, or an overdosage to normal tissue, increasing the complications probability.

Microdosimetry accounts for the stochastic nature of energy deposition, providing a description of the energy received by a cell superior to standard dosimetry. Existing RBE models assume that all variables follow a Poisson distribution, and thus can be described by their means. The assumption neglects stochastic fluctuations in energy deposition from cell to cell, and from dose fractionation, which can be especially significant in highly mixed radiation fields that occur at the beam edges and in the distal region.

Microdosimetry has been already applied to particle therapy for describing radiation quality. Here, we will present how the basic principles can be expanded to fully exploit the potential of microdosimetry. We have focused on developing wo aspects: i) an innovative two-stage detector (Hybrid Detector for Microdosimetry HDM), and ii) a new model (GSM<sup>2</sup> generalized stochastic microdosimetry model for radiobiological endpoints), for providing a general probabilistic framework to describe the damage formation and evolution. The combination of HDM and GSM<sup>2</sup> has the potential of improving ion therapy performance by increasing treatment effectiveness, as we as reducing possible toxicities.

#### NUCLEAR PHYSICS FOR REDUCTION OF RANGE UNCERTAINTIES IN CLINICAL AND PRE-CLINICAL APPLICATIONS OF ION BEAMS

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Ion therapy is rapidly emerging as a promising treatment modality in external beam radiation therapy. In particular, the favorable physical interaction properties of ion beams in matter enable concentrating the energy deposition in a well localized maximum in depth, so called Bragg peak, thus offering optimal coverage of the tumour target with better sparing of normal tissue and critical organs in comparison to the widely established photon therapy. However, full exploitation of the superior ballistic precision and advanced beam delivery strategies of modern ion beam therapy is still hampered by the issue of range uncertainties, i.e., uncertainties in the knowledge of the beam stopping position, related to the Bragg peak, in living tissue. To this end, in-vivo range verification during or after treatment is a very active research area exploiting several nuclear physics processes occurring in ion beam therapy range verification, which are just entering the clinic or are being investigated for possible clinical use in the near future, along with their downscaling to pre-clinical precision radiation research.

Parts of this work are funded by the European Research Council (ERC) under the grant agreement 725539

#### HADRONTHERAPY: PHYSICS MEETS ONCOLOGY IN THE FIGHT AGAINST CANCER

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#### Fondazione CNAO

Some elementary particles used for experiments of fundamental physics have properties useful to the treatments of patients affected by oncological pathologies. These are protons and carbon ions, collectively named hadrons, hence the term hadrontherapy. Hadrons, in particular carbon ions, are more precise than conventional X-rays on the tumour target and possess radiobiological characteristics suited to treat radio-resistant or inoperable tumours. Italy is at the forefront of these innovative techniques and in Pavia, a clinical facility called CNAO (Italian acronym that stands for National Centre for Oncological Hadrontherapy) has treated so far more than 3000 patients with very good results.

This talk will deal with the rationale of hadrontherapy and will give an overview of its status in the world. It will also present the technologies associated to hadron beams production and their use, including accelerators, the dose delivery systems, the patient positioning and the imaging devices. An overview of the most promising R&D topics is also included.

Hadrontherapy is a treatment modality suited for a growing - but still rare - and limited list of clinical indications. It is thus of paramount importance to properly select elective patients and, at the same time, to investigate combined treatments to improve its outcomes. A fundamental step in this direction is the creation of networks among hadrontherapy centres and clinical and research institutions to allow access to a larger number of patients and users. On the other side, it is crucial to develop new technologies at lower costs and dimensions and to make hadrontherapy more accessible.

The scientific community of hadrontherapy is multidisciplinary and many professionals are involved in the different steps of patient management and beams production. In this respect, educational programmes -providing hands-on experience to a new generation of researchers and thus allowing them to optimally access and exploit all the essential tools of the hadrontherapy research and clinical infrastructures - are of great importance.

# NOVEL RADIOISOTOPES FOR MEDICAL APPLICATIONS: THE CERN MEDICIS PROJECT AND BEYOND

Cocolios T.E.<sup>1</sup>, Duchemin C.<sup>1,2</sup>, Popescu L.<sup>3</sup>, Stora T.<sup>2</sup>, on behalf of the CERN-MEDICIS Collaboration and the Tb-IRMA-V project

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Nuclear medicine uses radioisotopes for fonctional imaging and medical treatment by injection of radioisiotopes in the body. Some elements have such targeted action, e.g. thyroid uptake of iodine, that their radioisotopes can be used directly. However, others have to be included in molecules, e.g. <sup>18</sup>F in sugar-analogue FDG, or complex vector molecules (peptides, hormones, antibody fragments) specific to cell markers. Radioisotopes which emit  $\gamma$  rays are used to image the location of the element or molecule in the body. Radioisotopes which emit charged particles such as  $\beta^{-}$ ,  $\alpha$ , or Auger electrons can be used to destroy target cells by in-body radiation therapy. The potency of such treatment is so high that damage to healthy cells has to be avoided as much as possible and the delivery vector has to be demonstrated with an imaging protocol first. The tandem between imaging and treatment is called theranostics.

In spite of the high promise that theranostics techniques have in the treatment of distributed cancer in particular, there are but a handful of radioisotopes that are currently used for this application. The main limitation is that novel radioisotopes that would have interesting properties are not commercially available, which limits their access for medical research. Meanwhile, so long that the medical research has not been performed, there is no commercial interest to produce them. In order to break out of this conundrum, CERN has established the Medical Isotope Collected from ISOLDE (MEDICIS) facility, which is dedicated to producing novel, promising radioisotopes available to the medical community to seed this initial research phase.

The terbium element is of particular interest as it features 4 radioisotopes which have the different properties of relevance: <sup>149,152,155,161</sup>Tb. CERN MEDICIS is currently the only facility worldwide that can provide the first three isotopes of this quadruplet reliably. Meanwhile, other facilities are exploring how to expand the production capacity for when these isotopes are needed in higher quantities, and in particular the ISOL@MYRRHA project at the SCK CEN in Belgium. In partnership between CERN, KU Leuven, SCK CEN and the university hospital UZ Leuven, the full chain from production to pre-clinical trials is currently being investigated under the Tb-IRMA-V project.

#### LET SPECTROMETRY IN RADIOTHERAPY AND RADIATION PROTECTION

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Track-etched detectors (TED) have been used as linear energy transfer (LET) spectrometers in heavy ion beams for many years. Even today, TEDs can be advantageously applied in research related to advanced radiotherapy techniques and/or radiation protection. To illustrate the capabilities of the technique, several representative studies are presented.

LET spectra and depth–dose distribution of a carbon ion beam were measured at Heavy Ion Medical Accelerator in Chiba, Japan. The measurements were performed along the monoenergetic beam with energy 290 MeV/u in different positions: (1) at beam extraction area, (2) at beginning, (3) maximum and (4) behind the Bragg peak region (0, 117, 147 and 151 mm of water-equivalent depth, respectively). The LET spectra inside and outside of the primary ion beam have been evaluated. TED record only heavy charged particles with LET above 8–10 keV/ $\mu$ m, while electrons and ions with lower LET are not detected. The Geant4 simulation toolkit version 4.9.6.P01 was used to estimate the contribution of non-detected particles to absorbed dose. In another studies, the TEDs were applied to measure the organ out-of-field doses in proton beam therapy, and for the description of radiation field perturbations behind hip or dental metallic implants. Presented results demonstrate the applicability of TED for microdosimetry measurements in therapeutic ion beams.

LET spectrometry can be applied with advantage to evaluate dose equivalent in complex environmental exposition conditions, such as in space. Cosmic radiation consists of primary high-energy galactic and solar particles. When passing through spacecraft walls and astronauts' bodies, the spectrum becomes even more complex due to generating of secondary particles through fragmentation and nuclear interactions. Total radiation exposure is contributed by both these components. The measurements using TEDs were part of different experimental campaigns on board the International Space Station (ISS). For instance, the measurements in Russian segment of the ISS in 2009 shown that the primary high-energy heavy ions with long range contribute up to 56% of the absorbed dose and up to 50% to the dose equivalent.

#### ION-BEAM THERAPY AT HIT: OPTIONS FOR MULTI-ION TREATMENT AND RESEARCH

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At the Heidelberg University Hospital the first dedicated proton-ion-therapy facility, the Heidelberg Ion-Beam Therapy Center (HIT), started its clinical operation in 2009. About 7.000 patients were treated with Protons or Carbon ions at HIT using the fully active 3-dimensional intensity-controlled rasterscan technique [1, 2]. An optimized linac-synchrotron combination generates libraries of energy-, focus- and intensity-variable pencil-beams for the dose-delivering scanning systems at two horizontally-fixed beam lines and a scanning ion gantry.

The availability of low-LET and high-LET beams at HIT ranging from Protons to Oxygen under identical conditions in combination with HIT's unique ion gantry optimally supports clinical trials aiming to clarify the question of which particle species is best suited for what indication. In order to pave the way for new therapy protocols HIT comprises a laboratory infrastructure and a dedicated research beam line offering low-LET proton and Helium beams and Carbon and Oxygen beams at the high-LET end.

Helium ions (<sup>4</sup>He) remained clinically unexploited worldwide since the shutdown of the clinical trials at the Lawrence Berkeley Laboratory (LBL) despite their favorable physical and biophysical characteristics. <sup>4</sup>He is characterized by minimal lateral scattering compared to protons, with a significantly reduced fragmentation tail compared to carbon ions and enhancement of bio-effects with relative biological effectiveness (RBE) values ranging between 1.3 and ~3. Comprehensive pre-clinical studies [3, 4], i.e physical beam characterization, radiobiological experiments, biophysical modelling and treatment plan comparisons in realistic clinical settings triggered an implementation project for this new modality at HIT. This research allows for the clinical translation of <sup>4</sup>He ion-beam therapy with state-of-the-art technologies.

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# RECENT TRENDS IN DEVELOPMENT OF PET RADIOPHARMACEUTICALS FOR NUCLEAR MEDICINE

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PET-CT is expanding rapidly in many countries and has quickly established its place in the diagnosis and management of several prominent diseases. This has come about through a growing and convincing evidence base in regard to its efficacy, combined with sound financial reasons. Taken together, these provide a firm argument for routine use of PET-CT in certain disease processes. Radioisotopes production for PET is generally performed by means of a cyclotron that is used to accelerate charged particles. These accelerated particles then go on to interact with a target to produce radioisotopes suitable for radiopharmaceuticals preparation and use in PET imaging. Impressive progress has been made recently in the radioisotope production. New achievement was done in the development of solid and liquid targets for metal radioisotopes production. This has allowed broader access to several new radionuclides suitable for the production of new radiopharmaceuticals in PET diagnostics, including gallium-68, copper-64 zirconium-89, scandium-44, yttrium-86.

# THEORETICAL STUDY OF <sup>47</sup>Sc PRODUCTION FOR THERANOSTIC APPLICATIONS USING PROTON BEAMS ON ENRICHED TITANIUM TARGETS

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Radionuclide <sup>47</sup>Sc represents a promising element for innovative radiopharmaceutical compounds suitable for theranostic applications, however an efficient and convenient production route has still to be identified. In this work we investigate its cyclotron production using enriched <sup>49</sup>Ti and <sup>50</sup>Ti targets. This study is developed within the REMIX project carried on at the LNL-INFN Laboratories of Legnaro, Padova, Italy. The first step implies an accurate modeling of the relevant cross sections, including the production of the main contaminants, which in this case is represented by the sole (long lived) <sup>46</sup>Sc. As shown in figure for the <sup>49</sup>Ti case, we describe statistically the results from the nuclear reaction code Talys, since a large variety of models can be selected with this tool. The solid black lines denoted BTE (Best Theoretical Evaluation), refer to the median of the interquartile band (gray band in the figure) which exhibits graphically the dispersion introduced by the variety of models included in the Talys package [1]. The large

discrepancies with the Levkovskij data for  ${}^{49}$ Ti(p, $\alpha$ ) ${}^{46}$ Sc compelled us to adjust the nuclear level densities in the Hartree-Fock-Bogoliubov description (ld5 models in figure). All these optimisations reflect in the prediction of the  ${}^{49}$ Ti(p,x) ${}^{47}$ Sc cross section where data are not yet available.

Starting from the <sup>47</sup>Sc and <sup>46</sup>Sc cross sections so determined, it is possible to select the energy interval which maximises the production yield and the purity, for both targets. Then, we numerically solve the coupled differential equations describing the decays of the relevant radionuclides, to calculate the evolution of the number of nuclei produced at any time from the beginning of the irradiation. In this way, one can derive the activity and the radionuclidic purity to be expected with these production processes, and from there, the dose-release assessments, such as the Dose Increase (DI) caused by these contaminants.

In this work, the full process of radionuclide production with the two enriched Titanium targets is analyzed, starting from the optimal modeling of the cross sections. It is clear that more experimental data are needed for the reactions involved as planned in the REMIX project.

[1] arxiv.org/abs/2102.12228
#### **TESTING A PCT SCANNER PROTOTYPE**

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Proton therapy is a cancer treatment technique that allows for a more selective application of dose to tumors in comparison with conventional radiotherapy with X- or grays. In order to extract the full potential of this technique, a high control of proton ranges is required for planning and treatment.

Currently, the treatment planning in proton therapy facilities is guided via X-ray computed tomography (X-ray CT) images. This requires the conversion "a posteriori" of the map of Hounsfield Units (HU) to Relative Stopping Powers (RSP) useful for proton therapy treatment plans [1]. This conversion induces a large uncertainty in the range of the protons (up to 5% in the abdomen and up to 11% in the head) [2-4]. In turn, treatment plans made using proton-CT (pCT) images would reduce the uncertainties of proton ranges to be below 1% and would provide a better control of the treatment [1].

In this context, we are building a prototype for pCT scanner using particle detectors extensively used in experimental nuclear physics. Those are the Double-Sided-Silicon-Strip-Detectors (DSSDs) used as proton trackers and LaBr<sub>3</sub>(Ce) and LaCl<sub>3</sub>(Ce) scintillation detectors as residual energy detectors. Mapping the energy losses of protons traversing the sample one can obtain a 3D distribution of Relative Stopping Powers (RSP).

A first test of the proton tracker was carried out at the CMAM tandem (Madrid, Spain) using low-energy protons (10 MeV). A second experiment at proton energies relevant in proton therapy (100-200 MeV) will be performed in Cyclotron Centre Bronowice (CCB) in Krakow (Poland) in June 2021.

In this contribution we report on the results from both experiments.

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#### A NEW NUCLEAR REACTION ROUTE TO PRODUCE <sup>52g</sup>Mn WITH HIGH PURITY FOR MULTI-MODAL IMAGING

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The <sup>52g</sup>Mn radionuclide decays with the emission of a positron ( $\beta$ + decay) and it is characterized by paramagnetic properties: these features make <sup>52g</sup>Mn suitable for the innovative MultiModal Imaging technique and in particular for a combined PET/MRI scan. The standard cyclotron-based production of <sup>52g</sup>Mn relies on the nuclear reaction <sup>nat</sup>Cr(p,n)<sup>52g</sup>Mn, which provides a sufficient yield but leads to the production of long-lived contaminants <sup>53</sup>Mn and <sup>54</sup>Mn. The production of both contaminants can be limited by irradiation of highly enriched <sup>52</sup>Cr targets, but this solution significantly increases the production costs.

For this reason, in the framework of the METRICS project at INFN-LNL we investigated the possibility of an alternative and competitive route to produce  $^{52g}$ Mn with high RadioNuclidic Purity (RNP) and high production yield. We identified this alternative route with the reaction  $^{nat}V(\alpha,x)^{52g}$ Mn, which has not been considered for this purpose so far. We investigated this production method through the use of state-of-art nuclear reaction codes, like TALYS, whose calculations were compared with experimental data taken from the literature.

However, we found some discrepancies between the theoretical calculations of the cross sections and the corresponding experimental data. Therefore we tuned the parameters governing the nuclear level densities in recent microscopic models implemented in TALYS, thus obtaining new cross sections that are in agreement with the data. This allowed us to obtain more reliable predictions for the subsequent calculation of the production yields and purities.

By studying the cross sections for  ${}^{52g}$ Mn and its contaminants, we identified an optimal energy region for the production of high purity  ${}^{52g}$ Mn around 40 MeV [1]. By solving Bateman equations we also calculated the time evolution of the number of nuclei of the different Mn isotopes, for an irradiation in this energy window. We determined that this reaction route leads to a very high RNP, that remains close to 1 for about 20 days after the irradiation (see figure). Moreover, we found that the production of the main contaminant,  ${}^{54}$ Mn, is expected to be lower than for the standard reaction  ${}^{nat}Cr(p,n){}^{52g}Mn$ .

The study suggests the reaction  $^{nat}V(\alpha,x)^{52g}Mn$  as a promising alternative route for  $^{52g}Mn$  production.



Radionuclidic Purity

# NUCLEAR FRAGMENTATION STUDIES FOR HADRONTHERAPY AND SPACE RADIATION PROTECTION WITH THE FOOT EXPERIMENT

Colombi S., on behalf of the FOOT collaboration

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The application of particle beams in cancer therapy is a well-established strategy and its combination with surgery and chemotherapy is becoming an increasingly effective approach for several clinical cases. Currently, protons and <sup>2</sup>C ions are used for patients' treatment due to their characteristic depth-dose deposition profile featuring a pronounced peak (the *Bragg Peak*) at the end of range. Nuclear interactions of the beam with the patient tissues always occur during treatment, leading to the production of both projectile and target fragments, which are responsible for changes in the primary radiation composition, energy and direction. Specifically, fragments produced along the proton beam path in the target fragmentation process may affect the proton Relative Biological Effectiveness (RBE), nowadays assumed as a constant value (i.e., RBE=1.1) in Treatment Planning System. Precise fragmentation cross section data would help to fill the gap in the available measurements for particle species and energies of therapeutic interest.

The FragmentatiOn Of Target (FOOT) experiment has been designed to measure with  $\sim 5\%$  accuracy fragmentation double differential cross sections with respect to the kinetic energy and the generation angle of the emitted fragments. The FOOT measurements campaign includes <sup>16</sup>O, <sup>12</sup>C and <sup>4</sup>He beams with energies spanning between 200 MeV/u and 700 MeV/u impinging on C,  $C_2H_4$  and Al targets. Target fragmentation induced by proton beams will be studied taking advantage of an inverse kinematic approach, while projectile fragmentation will be investigated using direct kinematics. The FOOT experimental setup consists of a "table-top" electronic setup, based on a magnetic spectrometer for the detection of heavier fragments (Z≥3), and an emulsion spectrometer to be employed alternatively for the identification of low Z fragments (Z≤3) that would not cross the whole magnetic spectrometer.

Space radiation protection will also benefit from such data to improve the design of the shielding of spaceships in view of long distance travels (i.e., Mars human exploration).

A performances study of the electronic setup in the cross-section evaluation based on FLUKA Monte Carlo simulations will be presented here, together with the analysis of experimental data acquired with the FOOT apparatus in 2019 and 2021.

#### IMPROVEMENT OF NUCLEAR REACTION MODELING FOR THE PRODUCTION OF <sup>47</sup>Sc ON NATURAL VANADIUM TARGETS FOR MEDICAL APPLICATIONS

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<sup>48</sup>Sc Cumulative of levels

In the context of the production of radionuclides for medical applications, the study of nuclear reactions plays a relevant role. In particular the analysis of the cross sections is extremely useful to calculate quantities like yields and purities with high accuracy, as demanded by dosimetry in order to compute the released and tolerated dose and to estimate the risks for the tissues.

In our work we investigate the reaction of protons on natural vanadium target for the production of <sup>47</sup>Sc [1], a radioisotope suitable for theranostic applications thanks to its decay characteristics, and we evaluate the co-production of the main contaminants. We perform the calculations using the nuclear reaction code TALYS, in which several theoretical models are implemented for the description of the different processes involved in a nuclear reaction. Unfortunately, in the case of the considered reaction, the default excitation functions do not reproduce the trend of the new experimental data obtained in the framework of the PASTA project.

In order to achieve an agreement between the theoretical curves and the data, we investigate the Nuclear Level Densities (NLD) of recent microscopic models provided by the code. They are mainly based on Hartree-Fock methods for the description of the manybody structure and they can be tuned to the data by varying two parameters. After a best fit optimization we achieve an improvement of the agreement with the new measurements, particularly around the evaporation peak not only for <sup>47</sup>Sc but also for its main contaminant, <sup>46</sup>Sc. The result, indicated as "Talys modified", is also evaluated by considering the effect on theoretical cumulatives of levels, as shown in figure for the <sup>48</sup>Sc case in comparison with Hartree-Fock and Goriely theoretical cumulative and with the experimental one.

[1] G. Pupillo et al., *Production of* <sup>47</sup>*Sc with natural vanadium targets: results of the pasta project*, Journal of Radioanalytical and Nuclear Chemistry, **322**, 1711 (2019), https://doi.org/10.1007/s10967-019-06844-8

#### CLINICAL RESULTS OF IN-VIVO INTER-FRACTIONAL MONITORING IN PARTICLE THERAPY BY MEANS OF THE INSIDE IN-BEAM PET

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Since an in-vivo treatment verification device is highly desired in particle therapy in order to thoroughly exploit the potential of charged particle energy deposition in depth, several range monitoring systems are currently under development or testing. Such type of devices could be mostly useful for some pathologies that may undergo to inter-fractional morphological changes in the patient during the treatment course. With an in-vivo verification system, morphological changes could be detected in an early phase in which they do not compromise the effectiveness of the treatment yet.

In the contest of the INSIDE project, a state-of-the-art technology in-beam Positron Emission Tomography (PET) scanner was developed and installed at the CNAO facility (Italy). This instrumentation relies on the reconstruction of the beta+ activity distribution generated into the patient body by nuclear interactions of the primary beam with human tissues. Thanks to this beta+ activity that is related to particle range it is possible to verify the compliance of the delivered and prescribed dose. In order to test the clinical performance of the INSIDE in-beam PET scanner and find the best indicator for clinician to identify regions in which morphological changes happened, a clinical trial started in July

2019 (ClinicalTrials.gov NCT03662373) including proton and carbon ion irradiations. Currently, 20 patients, affected by head-and-neck and brain pathologies, have been monitored and the analysis is reported in this study, considering the measurement reproducibility and the morphological changes detection sensitivity.

Results show that the measurement reproducibility depends on the primary particle type and also on the geometrical and dosimetric distribution of each irradiated beam. In general, PET images acquired during proton treatments have a better sensitivity in the range difference detection (6-8 mm FWHM) while, in the case of carbon ion therapy, it decreases (about 1.5 cm FWHM). In some patients, inter-fractional changes occurred. These variations are visible in the corresponding PET images, showing the capability to detect range differences before they become so critical to lead to replanning. Thanks to a detailed 3D statistical analysis, the critical regions can be reported to the clinicians.

# MICRODOSIMETRY MEASUREMENTS OF LOW ENERGY PROTONS WITH NEW SILICON 3D-MICRODETECTORS

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We have performed the first microdosimetry measurements on a low energy proton beam with therapeutic-equivalent fluence rates by using the second generation of new silicon-based 3D-microdetectors [1–3]. The sensors were designed with electrodes etched inside the silicon, as defined according to Parker et al. [4], which were manufactured in the National Microelectronics Centre (IMB-CNM, CSIC) at Barcelona, Spain. The second generation, with optimized technological parameters for a higher yield and improved charge collection performance, was manufactured on 4-inch Silicon-on-Insulator (SOI) wafers with a high resistivity n-type substrate and a thickness of  $10\pm0.5 \,\mu\text{m}$  or  $20\pm0.5 \,\mu\text{m}$ . Devices have leakage currents of 100 pA/cell and capacitances of 80 fF/cell at 5 V and show good diode behaviour with a depletion voltage of 5 V. Additionally, the set-up of the single channel readout electronics developed for optimizing the signal-to-noise ratio consists of two printed circuit boards to provide a portable and user-friendly device.

The irradiations were performed at the cyclotron facility (Cyclone 18/9 model) of the National Centre of Accelerators (CNA, Sevilla). The cyclotron produces 18 MeV protons and although this energy is below the energy range used in clinical proton therapy (up to 230 MeV), this study is acceptable as a first approximation since the 18 MeV protons are in the energy range arriving into the distal edge range of the clinical beams. Irradiations were performed at a therapeutic-equivalent fluence rate. Microdosimetry spectra of lineal energy were recorded at several depths up to the Bragg peak and the distal edge. Results were crosschecked with Monte Carlo simulations using a Geant4-based code.

This work demonstrates the capability of novel 3D-microdetectors to assess microdosimetric distributions at fluence rates as high as those used in proton therapy centres.

[1] C. Guardiola et al., Appl. Phys. Lett., vol. 107, no. 2, Art. no. 023505, 2015.

[2] C. Fleta et al., J. Instrum., vol. 10, no. 10, Art. no. P10001, 2015.

[3] Prieto-Pena et al., IEEE Trans. Nucl. Sci, vol. 66, no. 7, July 2019

[4] S. I. Parker, C. J. Kenney, and J. Segal, *Nucl. Instrum. Methods Phys. Res. A, Accel. Spectrom. Detect. Assoc. Equip.*, vol. 395, no. 3, pp. 328–343, 1997.

# DEVELOPMENT OF INTEGRATION MODE PROTON IMAGING WITH A SINGLE CMOS DETECTOR FOR A SMALL ANIMAL IRRADIATION PLATFORM

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A novel irradiation platform for pre-clinical proton therapy studies foresees proton imaging for accurate set-up and treatment planning. In integration mode, imaging at modern synchrocyclotron-based proton therapy centres with high instantaneous particle flux is possible. Commercially available detectors, such as large-area CMOS sensors, allow the determination of the object's water-equivalent thickness (WET). We present experimental results from two proton therapy facilities, supported by extensive Monte Carlo (MC) simulations, demonstrating the feasibility of this imaging modality for pre-clinical studies.

Image contrast is achieved by recording the proton energy deposition in the detector pixels for several incoming beam energies and applying a signal decomposition method

that retrieves the WET. A single planar  $114x65mm^2$  CMOS sensor (49.5µm pixel pitch) behind the imaged object was used. In experimental campaigns at two isochronous cyclotron-based facilities, probing energies suitable for small-animal sized objects were produced once with the built-in energy layer switching ability and the other time using a custom degrader wheel.

To assess WET accuracy, a micro-CT calibration phantom with 10 inserts of tissuemimicking materials was imaged. The phantom-to-detector distance was 0.3, 1.3 and 3.3cm.

The average relative WET error compared to the ground truth was <1% for 0.3cm spacing and the spatial resolution was 0.2mm. For 1.3cm spacing the results were <2% relative WET error and 0.4mm spatial resolution. For 3.3 cm proton scattering had considerable impact and WET relative error increased to 30%. Imaging time with the built-in energy switching was 95s, of which 82s are attributed to the limitations of the too slow (~4s) energy switching time (considerably reduced in new versions).

A pixelated CMOS detector and post-processing methods can enable fast proton radiographic imaging for small-animal-sized objects with high WET accuracy and spatial resolution.

Supported by ERC grant 725539.

#### METROLOGY FOR ADVANCED RADIOTHERAPY USING PARTICLE BEAMS WITH ULTRA-HIGH PULSE DOSE: TEST IN FLASH-LIKE ELECTRON BEAM AT MICROTRON MT 25

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The application of high therapeutic doses at very short pulses can reduce the risk of damage to healthy tissue. The practical use of this "FLASH effect" for treatment will be possible due to a new generation of accelerators (modified conventional electron accelerators, new laser-driven accelerators) enabling to generate pulsed beams of particles with very high doses in the pulse (UHD beams). However, the character of UHD beams requires development of new methods for dosimetry and metrology of both primary and secondary radiation outside the pulse beam. To develop and improve dosimetry standards for FLASH radiotherapy, very high energy electron (VHEE) radiotherapy and laser-driven medical accelerators is the aim of the European Joint Research Project "UHDpulse - Metrology for advanced radiotherapy using particle beams with ultra-high pulse dose". The project involves several national metrology institutes and research institutions from various European countries, including Czech Republic.

In this contribution we present the first results from the test in flash-like electron beam. Various passive detectors (different types of luminescent detectors and plastic nuclear track detectors) were irradiated in and outside the electron beam produced by microtron MT 25 of Nuclear Physics Institute CAS. The measured data are supported by Monte Carlo simulations.

This project 18HLT04 UHDpulse has received funding from the EMPIR programme cofinanced by the Participating States and from the European Union's Horizon 2020 research and innovation programme.

# FIRST IN SITU 2D-MICRODOSIMETRY MAPS AT A PROTON THERAPY CENTER WITH NOVEL SILICON 3D-MICRODETECTORS

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A new detection system for microdosimetry has been developed and successfully tested at the Institut Curie-Proton Therapy Center of Orsay (ICPO). It is based in silicon 3D-microdetectors having a cylindrical shape with a size comparable to that of human cells (20 µmthick, 25 µm-diameter). They were developed by the National Microelectronics Center (Spain), and have already shown a good performance for the measurement of microdosimetric distributions for hadrontherapy [1-3] (Fig. 1). The new system consists in an 11 x 11 array of 3D-microdetectors assembled to a multichannel readout electronics and a customized data acquisition system. The analysis of the data in real time was performed with an in-house software. The whole system is able to quantify the linear energy transfer (LET) at microscopic level. A proton beam with a clinically relevant energy (100 MeV) was used to irradiate this array as starting point.

Fig. 2 shows a representative 2D-map obtained when irradiating the array and interposing SP34 RW3 water-equivalent phantoms between the proton beam and the detector. Several thicknesses of RW3 were used in order to obtain the corresponding lineal energies (the microscopic parameter equivalent to the LET) deposited in the region of the Bragg peak. We will present the probability density functions of the lineal energy *y* and the dose, f(y) and d(y), respectively. Additionally, the whole ICPO beamline, including the detector set-up, was modelled using a Monte Carlo Geant4-based code to crosscheck the results.

We present the first 2D LET-maps ever obtained in situ during irradiation at a clinical facility. The potential clinical implementation of these measurements in the near future would allow for the RBE optimization of proton therapy treatments.



Fig. 1. a) Sketch of the 3D-microdetector cross section. b) SEM image of one of the microdetectors. c) Picture of the 11 x 11 array.



Fig. 2. Map of the energy deposited expressed in ADC units when the 11x11 3Dmicrodetectors array is irradiated. The corresponding ADC-to-energy conversion will be implemented in the near future.

- [1] C. Guardiola et al., Appl. Phys. Lett., vol. 107, no. 2, Art. no. 023505, 2015.
- [2] C. Fleta et al., J. Instrum., vol. 10, no. 10, Art. no. P10001, 2015.
- [3] J. Prieto-Pena et al., IEEE Trans. Nucl. Sci., vol. 66, no. 7, 2019.

### FIRST IN-BEAM TESTS ON SIMULTANEOUS PET AND COMPTON IMAGING AIMED AT QUASI-REAL-TIME RANGE VERIFICATION IN HADRON THERAPY

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Hadron Therapy (HT) with protons has advantages with respect to conventional radiotherapy because of the maximization of the dose at the Bragg peak. As a drawback, and because of different systematic uncertainty sources, a quasi-real time monitoring for the proton range verification is required to reduce safety margins and thus enhance its potential benefits. In this respect, a promising technique is prompt gamma-ray (PG) monitoring, which requires detection systems with large detection efficiency, high time resolution, compactness, fast response, low sensitivity to neutron-induced backgrounds and powerful image reconstruction capabilities. To a large extent, these criteria are fulfilled by the i-TED detection system. i-TED is an advanced array of Compton cameras originally designed for neutron-capture time-of-flight experiments. In this contribution we will demonstrate the suitability of i-TED also for PG monitoring in ion-range monitoring during HT. Furthermore, aiming at improved signal-to-noise Compton images in the highenergy gamma-ray range characteristic of HT, a novel Machine Learning (ML) methodology has been developed and applied for identification of full-energy events during the irradiations. Together with the use of GPUs, a quasi-real time PG monitoring can be achieved using i-TED.



Figure 1. Raw experimental images along the proton beam axis. Left: PET image of five 180 um thick Nylon layers obtained with two i-TED modules. Right: Experimental Compton image during the proton irradiation, reflecting mostly the position of a 2 mm thick graphite sample placed beyond the stack of Nylon layers.

At this conference we will present the first results from an experiment performed at the cyclotron of the CNA facility, Spain, where 18 MeV protons impinged on a 5-layers stack of thin Nylon foils separated by 1.6 cm and followed by a 2 mm thick graphite layer. The latter assembly was surrounded by two i-TED modules. The latter could be therefore simultaneously operated as a positron-emission tomography (PET) system for in-beam image reconstruction from 511 keV positron annihilation gamma-rays (Fig.1-Left). Also, using the high-energy prompt gamma-rays the i-TED modules were simultaneously used as a high-efficiency and large field-of-view Compton imager (Fig.1-Right). Finally, a short outlook will be presented on forthcoming plans with i-TED for simultaneous PET and Compton imaging at a HT facility under realistic clinical conditions.

\*This research has been funded by the European Research Council (ERC) under Grant Agreement 681740 HYMNS

#### DATA-DRIVEN MODEL OF CARBON ION FRAGMENTATION IN A FAST MC CODE (FRED) FOR TREATMENT PLANNING SYSTEM

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The Treatment Planning Software (TPS) commonly used in Particle Therapy (PT) adopts a simplified beam-body interaction model. Full Monte Carlo (MC) simulations, implementing the interaction of particles with actual human tissues, typically demand substantial computational resources. Thus they are used only to check treatment plans for a restricted and specific number of difficult cases. However, the advent of general-purpose programming Graphics Processing Units (GPU) has prompted the development of MC codes that can dramatically reduce the plan recalculation time with respect to standard MC codes running in CPU hardware. FRED (Fast paRticle thErapy Dose evaluator) is a fast MC code for ion beam treatment planning, developed to run on GPU, capable of rapidly recalculating a complete treatment plan within minutes.

Nowadays FRED is already used as a quality assurance tool in the proton clinical center of Maastricht and Krakow and as a research tool at several clinical and research centers in Europe (Krakow, Trento, Maastricht, Lyon and PSI). The adaptation of FRED for other therapeutically particle beams is under development.

When using Z>1 ions beam, the dose arising from the fragments produced in the collisions between the projectile and the target nuclei has to be taken into account. The projectile fragments, having on average the same energy per nucleon of the primary beam and a lower mass, can release dose also beyond the Bragg Peak causing the well-known fragmentation tail.

A data-driven model for carbon ions fragmentation, which balances accuracy and timeconsumption, has been developed. It is based on the available cross-sections data recently measured at GANIL (laboratory of CAEN, France), where the fragmentation of carbon ions on thin targets (H, C, O, AI and Ti) has been studied. The model has been benchmarked with full simulations performed with the FLUKA software, comparing the dose released in water phantoms. A level of agreement of the order of 2.5% of the total dose deposited is reached in the 50–300 MeV energy range. In this contribution, the model will be detailed and the performance within the FRED platform will be presented.

# NEW METHODS FOR THERANOSTIC RADIOISOTOPE PRODUCTION WITH SOLID TARGETS AT THE BERN MEDICAL CYCLOTRON

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The concept of theranostics in nuclear medicine is based on the use of a pair of radioisotopes to label radiopharmaceuticals for both diagnosis and therapy. For this reason, the production of novel medical radioisotopes is essential for the development of personalized nuclear medicine.

A research program is ongoing at the 18 MeV Bern medical cyclotron, equipped with a solid target station (STS) and a 6.5 m Beam Transfer Line (BTL) ending in a separate bunker with independent access.

The STS is installed on one out port of the cyclotron together with a mechanical transfer system developed by our group to load the target station without entering the bunker. A pneumatic transfer system (STTS) by TEMA Sinergie was installed to deliver the irradiated target either to one hot cell in the nearby GMP radio-pharmacy or to a receiving station in the BTL bunker.

To assess the activity at end of beam (EOB) by means of gamma spectroscopy, a system based on a 1 cm3 CdZnTe (CZT) crystal was designed and installed in the receiving station. The dector has been experimentally calibrated and allows measuring the produced activity with an accuracy of a few per cent.

To bombard itosope-enriched materials in form of 6 mm diameter compressed powders, a specific target coin was realized. To optimize the irradiation procedure, a novel ultracompact active irradiation system based on a specific magnetic lens and a twodimensional beam detector was conceived, constructed and tested. The system allows to control the size and position of the beam and to correct its characteristics by steering and focusing it and to keep it on target.

For an optimized production yield with the required radio-nuclide purity, precise knowledge of the beam energy and of the cross sections is crucial.

The beam energy measurement is on going with a method based on the stacked-foils technique, using the well known monitor reaction 48Ti(p,n)48V.

To measure the cross sections, a novel procedure based on the irradiation of a known target mass with a proton beam with flat profile was developed. The beam current passing through the collimator is measured by using a custom target station designed and built by our group.

Results on Er-165, Tb-155, Ga-68, Cu-61, Cu-64, Sc-43, Sc-44 and Sc-47 production are presented, with particular emphasis on the results obtained with the new irradiation system.

#### THE PAIR PRODUCTION IMAGING CHAMBER (PAPRICA)

Toppi M., Avanzolini I., Battistoni G., Calvi G., De Simoni M., Dong Y., Fantoni A., Fischetti M., Franciosini G., Marafini M., Mirabelli R., Muccifora V., Muraro S., Patera V., Ronchetti F., Sarti A., Sciubba A, Traini G., Valle S.M., Mattei I.

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In Particle Therapy, safety margins are applied when planning the treatment in order to account for the multiple sources of beam range uncertainty. Reducing safety margins is fundamental in the treatment of tumors close to organs at risk and paediatric patients. Many techniques based on prompt-gamma detection have been proposed, exploiting differ- ent strategy for the range monitoring (i.e. measurement of the emission spatial distribution, time of flight or energy spectrum). The PAPRICA (PAir PRoduction Imaging ChAmber) project proposes a novel detection strategy for the prompt-gamma. The chamber will reconstruct the prompt photons emission points 3D map exploiting the pair production mechanism, extending the technique of pair telescopes already used in astrophysics research at the energy of prompt-gamma emitted in particle therapy. The PAPRICA detector will be able to monitor proton and carbon ion treatments,

implementing neutrons background reduction strategies and profiting from the  $e^{+,-}$  pair clear topological event signature. No collimation nor time of flight information on the detected photons will be needed. The PAPRICA detector design and the expected performances evaluated by means of a Monte Carlo simulation in a real case scenario will be presented.

# MONDO: A SCINTILLATING FIBRE TRACKER FOR SECONDARY NEUTRON MEASUREMENTS IN PARTICLE THERAPY

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During a Particle Therapy (PT) treatments a considerable number of secondary neutrons is produced. Neutron can deposit a non-negligible amount of energy in- and out-of-field that contributes to an additional amount of dose that is organ dependent. To account for this contribution when optimising the treatment plans and to minimise the additional dose delivered to the healthy tissues it is necessary to know the secondary neutron energy and angular distributions.

Neutron measurements are challenging while the separation between the secondary neutrons from the ternary neutral component generated in the iterative interactions of fragmentation products with the treatment room and the patient itself is not trivial. To this aim a tracking detector is needed.

The MONDO is composed by a compact matrix of thin plastic scintillating fibres ( $250\mu$ m), assembled in orthogonal oriented planes (16x16x20cm3), optimised for the ultrafast neutrons. The tracking detection strategy exploits the double elastic neutron scattering. The tracking of both recoil protons allows for a complete neutron four-momentum reconstruction.

An innovative SPAD based CMOS sensor has been developed to readout the matrix system (SBAM sensor). SBAM has integrated electronics and has been designed to efficiently select proton tracks (99% efficiency for proton of 100MeV) with a low false positive rate.

The detector performance has been evaluated with a full MC simulation tool that implements the detector geometry, the pixel structure and the trigger strategy. An energy resolution of about 5-8 % is expected for neutrons in the energy range of 20-400 MeV with a detection efficiency of about  $10^{-3}$ , decreasing with energy because of proton not containment in the detector. The measurement of the energy loss by the protons along the track path allows to reconstruct the kinetic energy of the not contained recoil protons, thus, to detect efficiently neutrons up to 600 MeV with an energy resolution of about 10%. The expected back-pointing resolution is better than 3 mm for a neutron source placed at 20 cm. The secondary neutron spectra expected from a Carbon ion beam impinging on a

PMMA target has been convoluted with the detector neutron tracking capability. The expected results and efficiency will be presented as well as a preliminary SBAM readout characterisation.

#### <sup>225</sup>AC: FROM TARGET TO TEST TUBE TO TUMOR. DEVELOPMENTS ON HOW MUCH <sup>225</sup>AC CAN BE OBTAINED BY THE ISOL TECHNIQUE.

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Advances in the chelation of radionuclides to vector molecules have allowed for the targeted radio-immunotherapy of very specific cell types. If an alpha-emitting nuclide of the appropriate half-life is used, the resulting pharmaceutical can be used for highly effective treatment of small clusters of cancer cells. This technique is known as targeted alpha therapy (TAT). Although tested on many hundreds of patients [1], to date, no targeting molecule labelled with an alpha emitter has been awarded clinical approval. One of the few isotopes of interest for TAT is <sup>225</sup>Ac, due to its ten-day half-life and rapid emission of four alpha particles. However, by virtue of its desirable decay properties comes its rarity. The procurement of <sup>225</sup>Ac is the major bottleneck in developing TAT with this radionuclide, even in spite of increased production effort following a landmark study with <sup>225</sup>Ac-PSMA-617 demonstrating high treatment efficacy [2]. This is because almost all <sup>225</sup>Ac is obtained from only three <sup>229</sup>Th generators worldwide, with limited supply [3]. To solve this problem, new production routes for <sup>225</sup>Ac have been proposed, including the high energy proton spallation reactions: <sup>nat</sup>U, <sup>232</sup>Th (p, X) <sup>225</sup>Ac that can produce tens of Giga-Bequerel <sup>225</sup>Ac in target [4]. The challenge now remains to extract it with sufficient purity and efficiency, which is where the Isotope separation on-line (ISOL) technique can be applied. In this talk we present our first experiences in the off-line laser ionization and mass separation of <sup>225</sup>Ac at CERN MEDICIS. We discuss results on the laser ionization and overall collection efficiencies. Alongside this, we provide insight into the operating conditions that may be required for Ac release when oxidizing species are present in the heated target (e.g ThO<sub>2</sub>). The results are finally discussed in terms of the prospects for online <sup>225</sup>Ac collections at CERN MEDICIS, the first of which is planned for July 2021.

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#### SIMULTANEOUS NEUTRON AND GAMMA IMAGING SYSTEM FOR REAL TIME RANGE AND DOSE MONITORING IN HADRON THERAPY AND OTHER APPLICATIONS

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Hadron therapy in comparison to radiation therapy is able to target the tumor thanks to the maximum dose deposition at the end of the ion trajectory and its finite penetration in matter. However, this methodology faces two important limitations related to real-time (neutron and gamma) dose monitoring and ion-beam range verification which limit the potential benefits of protons over photons.

Compton imaging represents a promising technique for Prompt Gamma (PG) imaging for range verification in hadron therapy (HT) treatments. As for neutron monitoring, a drawback of most of the available systems is that only integral off-field neutron-fluence values are registered but no information is obtained from its spatial origin. In this work we will present GN-Vision, a novel dual gamma-ray and neutron imaging system, which aims at imaging, simultaneously to the PG, the spatial origin of the slow and thermal neutron dose (<10 eV) (see Fig. 1) generated during the treatment. The proposed device can also be of interest for industrial applications as well as in nuclear security.





The GN-Vision system has been designed following the technical developments of the i-TED detector, an array of Compton cameras that have been designed for neutron-capture

This research has been funded by the European Research Council (ERC) under Grant Agreement 681740 HYMNS. A PCT International patent has been filed.

experiments, in which  $\gamma$ -ray energies span up to 5-6 MeV, similar to the PG produced in HT. This contribution will first review the promising performance of the proposed system for PG imaging in proton therapy, studied on the basis of MC simulations of i-TED (see Fig. 1). The results indicate that this imaging system should be able to reconstruct the distal fall-off of the PG depth distribution with an accuracy of 2-3 mm for proton intensities as low as 10<sup>8</sup> protons, thanks to its high detection efficiency and the use of ML algorithms to compensate for the loss of full-energy events for high energy  $\gamma$ -rays. Then, we will describe the evolution from the i-TED detector towards the GN-Vision system and present the first conceptual results of its simultaneous neutron and gamma-ray imaging capability.

# STUDY OF THE INTERNAL PAIR-PRODUCTION DECAY OF THE 0<sup>+</sup> EXCITED STATE IN <sup>90</sup>ZR BY MAGNETIC SPECTROMETRY

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Yttrium-90 (<sup>90</sup>Y) radioembolization is a widely used internal-radiotherapy treatment of liver cancer. <sup>90</sup>Y is considered a pure beta-emitter, but a very small decay branch (~10<sup>-5</sup>) populates the first (0<sup>+</sup>) excited state of <sup>90</sup>Zr that decays by internal-pair production. The resulting positrons have been successfully used for PET/CT system to image <sup>90</sup>Y both in patients and phantom studies [1].

The accurate knowledge of the decay of <sup>90</sup>Y may open the possibility to use this isotope for quantitative image dosimetry with major consequence for health care and the possibility of tailoring therapy to an individual patient's response. A recent international effort has established the branching ratio of  $3.265\pm0.040\times10^{-5}$  [2] resolving discrepancies between older experiments. However, this consensus value has been reached between experiments that use the same technique of determining the emission rate of 511 keV  $\gamma$ rays produced following positron annihilation.

This motivates our new measurement using a different approach, which employs the doubly-focussing magnetic spectrometer of the Ecole Polytechnique Fédérale de Lausanne (Switzerland), to detect positrons directly, filtering out the much larger amount of  $\beta$ <sup>-</sup> radiation emitted in the ground-state-to-ground-state transition. This approach will also allow to measure the positron-energy spectrum for the first time. The details of the latter inform on the interaction between leptons and the Coulomb potential of <sup>90</sup>Zr that strongly deforms leptons wave functions and lead to an angular correlation between emitted positron and electrons.

In this contribution we will present the experimental setup, it accurate characterization, the result of the pair-production branching ratio measurements and the comparison with nuclear-theory predictions. We will also discuss the future plans to test <sup>90</sup>Y quantitative dosimetry using phantoms at NPL.

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# AUGER ELECTRON SPECTROSCOPY STUDIES AT THE NATIONAL PHYSICAL LABORATORY FOR MEDICAL APPLICATIONS

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When an atom is ionized by removing an electron from an inner atomic shell, the residual atom is in an excited state. Atomic relaxation back to the ground state occurs rapidly via radiative (X-rays) and non-radiative processes involving emission of Auger electrons.

Auger-emitters hold great potential for future targeted radiotherapies. This is because the Auger electron emissions, while low in energy, deposit that energy in a very short distance (comparable to the chromosome size), making these emissions of high linear energy transfer.

Despite this great potential, only a few Auger emitters have reached the clinical trial stage, and none are presently licensed for clinical use. This is because of several open questions involving radiobiology, dosimetry, and the lack of robust decay data.

At the National Physical Laboratory, we have recently started a program of Auger emission probabilities measurements essential to assess the biological outcome of Auger emission and to validate theoretical models of atomic relaxation.

Advancement in the field require improvement of detector technologies but most urgently of techniques to produce high quality radioactive sources that must consist of a single atomic layer of electron emitters, stable against the recoil of the decaying atoms and deposited on a substrate made of a low-Z made material.

This contribution will present the recent efforts at the National Physical Laboratory in the direction of source production, with a focus on Iodine and Platinum radioisotopes. The first has proven to be an ideal isotope to benchmark atomic theories, while the second is among the most promising candidate for future Auger therapy thanks to the large number of electrons emitted per decay.

#### MEASUREMENT OF THE FRAGMENTATION CROSS-SECTION OF OXYGEN IONS ON CARBON AND POLYETHYLENE TARGETS WITH THE EMULSION SPECTROMETER OF THE FOOT EXPERIMENT

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Hadron Therapy uses charged particle beams for the treatment of deep-seated tumours, profiting of the much larger dose released in the tumour region (Spread-out-Bragg-peak position) compared to the surrounding healthy tissues. The measurement of the fragments produced in the nuclear interactions of charged particle beams with human tissues is a key task to improve the clinical treatment plans.

The FOOT (FragmentatiOn Of Target) experiment is an international project whose focus is to study nuclear fragmentation of the target induced by proton beams. Fragmentation of <sup>16</sup>O and <sup>12</sup>C targets induced by 150-250 MeV/n proton beams will be studied via the inverse kinematic approach, where <sup>16</sup>O and <sup>12</sup>C therapeutic beams collide on graphite and hydrocarbons targets. The complete characterization of fragments produced will provide double differential cross sections in kinetic energy and emission angle. FOOT detection system consists of an electronic setup for the identification of Z ≥ 3 fragments and of an emulsion spectrometer to measure Z ≤ 3 fragments.

The first data taking was performed at the GSI facility in Darmstadt (Germany) by exposing the emulsion spectrometer to 200 and 400 MeV/n <sup>16</sup>O beams. Emulsion spectrometers were designed according to the Emulsion Cloud Chamber technique which consists of nuclear emulsion films alternated with passive material plates. Each chamber is composed of three sections: (i) the most upstream provides vertices reconstruction and it is made of emulsion films acting as micrometric tracking devices alternated with passive layers acting as target; (ii) a second section devoted to ion charge measurement, made of emulsion films which underwent dedicated treatments to expand their dynamic range of sensitivity; (iii) the most downstream section made of emulsion films alternated with passive materials of increasing density to measure the particle range and momentum.

In this talk, we will report the results obtained by analysing the emulsion chambers exposed to 200 MeV/n  $^{16}O$  on  $C_2H_4$  and C targets. Charge measurement will be described in detail and first cross section evaluation.

# MEASUREMENT OF THE PRODUCTION CROSS SECTION OF $\beta \text{+}$ emitters for range verification in proton therapy

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In proton therapy, PET range verification requires a comparison of the measured and expected  $\beta^+$  activity distributions produced by the proton field in the body, looking at the short-lived  $\beta^+$  emitters (online monitoring) or the long-lived  $\beta^+$  emitters (offline monitoring). The reliability of the expected activity distributions depends on the Monte Carlo simulations and hence on the accuracy of the underlying cross section data <sup>[1]</sup>. Several studies confirm the need for more and better measurements and evaluations of these cross sections <sup>[2,3]</sup>, especially for the short-lived nuclides, for which there are no data above 55 MeV <sup>[4]</sup>.

In this work, we have developed a method to measure the production yields of the long-lived  $\beta^+$  emitters  $^{11}C$  (t<sub>1/2</sub>=20min),  $^{13}N$  (t<sub>1/2</sub>=10min) and  $^{15}O$  (t<sub>1/2</sub>=2min) in C, N, and O in the full energy of interest. This method has been successfully used below 18 MeV at CNA (Spain) to obtain the differential cross sections for these reactions (paper submitted to

Rad. Phys. and Chem. <sup>[5]</sup>). The method combines the multi-foil activation technique with the subsequent measurement of the induced activity in a clinical PET scanner. The experiment was carried out at CNA (Spain) <sup>[5]</sup> and WPE (Germany). The experimental set up and preliminary results will be presented.

Regarding the short-lived nuclides ( $t_{1/2}$ <19s of  ${}^{10}$ C), the most widely produced isotopes are  ${}^{12}$ N ( $t_{1/2}$ =11ms) from C,  ${}^{29}$ P ( $t_{1/2}$ =4.14s) from P and  ${}^{38m}$ K ( $t_{1/2}$ =924ms) from Ca  ${}^{[4]}$ , which will be measured, in principle, at HIT (Germany). The set-up is designed to minimize the number of irradiations placing thin target films between thicker converter/degrader foils and measuring the activity induced with a miniPET made of four 10x10 cm<sup>2</sup> i-TED  ${}^{[6,7]}$ modules. In this way, a single irradiation provides the production yield at several energies, one for each target. The experimental setup, its validation at CNA below 18 MeV, and the corresponding simulations to measure the production yields of interest up to 200 MeV will be presented.

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# INTER-FRACTIONAL MONITORING IN PARTICLE THERAPY TREATMENTS WITH <sup>12</sup>C IONS EXPLOITING THE DETECTION OF SECONDARY PARTICLES

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In Particle Therapy (PT) the morphological changes occurring inside the patient body could lead to a significant difference in the released dose with respect to the planned one, with an impact on the treatment efficacy and the Normal Tissue Complication Probability. Nowadays, a system capable of checking on-line the conformity of the released dose to the target volume with the TPS prescription is missing in the clinical routine, and the treatment is kept under control performing a CT scan after a fixed amount of fractions (varying accordingly to the specific pathology) is delivered.

In PT the production yield of secondary charged fragments is highly correlated to the density of the tissues crossed by the beam. By comparing the fragments 3D production maps related to different treatment fractions it should be hence possible to spot morphological modifications occurred after the acquisition of the initial CT scan. The above-mentioned technique has been exploited to build an in vivo verification device operating in the Z>1 PT treatments, in which a significant fraction of the projectile fragments has a sufficient kinetic energy to escape from the patient body.

The device is the Dose Profiler (DP): a scintillating fibers based detector designed to detect and track the secondary charged particles produced in carbon ion treatments at CNAO (Centro Nazionale di Adroterapia Oncologica, Pavia, Italy). The DP has been developed within the INSIDE collaboration as a part of a bi-modal monitoring device including also a PET system capable of measuring the beam-induced  $\beta$ + activity. The DP is operational in the CNAO centre, since august 2019, as a monitoring device of <sup>12</sup>C ion. By comparing the reconstructed maps of the fragment emission points between the different fractions, it is exploring the feasibility of a technique capable of providing a reliable experimental feedback to support the physicians treatment re-planning decision.

The DP sensitivity is currently under investigation using the in-vivo data collected during the INSIDE clinical trial started in 2019 at CNAO (ClinicalTrials.gov Identifier: NCT03662373). A first set of (10) patients included in the trial involving head-neck pathologies has been analyzed, observing significant differences in some of the reconstructed emission maps consistently with the control CT scans information. In this contribution the final results, benchmarked with a MC simulation performed with the FLUKA software, will be presented.

#### A FEASIBILITY STUDY ON HIGH-Z METAL (OXIDE) NANOPARTICLES FOR CONTRAST ENHANCEMENT IN PROTON IMAGING AT A SMALL ANIMAL IRRADIATION PLATFORM

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Precision in proton therapy can be improved by pre-treatment proton imaging, as direct determination of the relative (to water) stopping power of tissue can reduce uncertainties in treatment planning. The use of contrast enhancement agents in proton imaging can augment tumour visualization, however only little research is available in this field. This study aims to assess the feasibility of high-Z metal-(oxide) nanoparticles (NPs) as constrast agents for proton imaging at a small animal irradiation platform.

Proton radiographies of a 2 cm thick PMMA block with a 1 cm cylindrical insert were taken with 70 MeV protons at the experimental beamline of the Trento Proton Therapy facility. The insert was filled with oil-gadolinium(III)-oxide NPs solutions of varying concentration. A miniaturized Timepix detector (MiniPIX) was placed behind it to record position of individual protons after traversing the block, along with their energy deposition inside the 300  $\mu$ m thick silicon sensor chip. Energy deposition was converted to water-equivalent thickness (WET) of the traversed object. To complement experimental radiographies, a FLUKA Monte Carlo simulation framework of a pre-clinical proton computed tomography (pCT) system [1] was used to evaluate contrast enhancement by gold, gadolinium(III)- and bismuth(III)-oxide NPs. A cylindrical water phantom containing inserts with varying concentration of homogeneously distributed metal-(oxide) as a surrogate for the NPs, was considered for simulated pCT imaging.

WET values of experimental radiographies indicate that contrast enhancement can be observed already with 4%wt gadolinium(III)-oxide NPs. This is in agreement with simulated pCT images, where contrast enhancement was found at 4%wt for all three NP materials, (contrast-to-noise ratios between 2.2 and 2.7). The investigated NPs may hence provide a viable way to enhance tumor visualization in proton imaging.

Supported by ERC grant 725539.

[1] Meyer et al 2020 Phys. Med. Biol. 65 155008

# STUDY OF THE CHARGE COLLECTION EFFICIENCY IN NOVEL SILICON 3D-DETECTORS FOR MICRODOSIMETRY

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The measurement of microdosimetric parameters, such as the lineal energy (y) or the probability density functions f(y), d(y) is of utmost importance to optimize proton therapy treatments since they will determine, among other parameters, the relative biological effectiveness (RBE) of the irradiation. For this purpose, new silicon 3D-microdetectors with size and shape equivalent to those of human cells have been developed and manufactured at the Centro Nacional de Microelectrónica (IMB-CNM, CSIC) in Barcelona, Spain. Their good performance as microdosimeters has already been demonstrated somewhere else [1], [2]. In this work, the charge collection efficiency (CCE) of the microdetectors has been studied by means of the ion beam induced charge (IBIC) technique at the microprobe beamline of the Centro Nacional de Aceleradores (CNA, Seville, Spain). Although the devices that we use for the microdosimetry experiments consist of multiple-microdetector arrays, here we have investigated the CCE in the individual cylindrical-cells. Two silicon 3Dmicrodetectors with different thicknesses (10  $\mu$ m and 20  $\mu$ m) and a diameter of 25  $\mu$ m (including the n<sup>+</sup> diffusion region) have been studied. Fig. 1 shows the 2D CCE-map and the CCE profile obtained for the 20  $\mu$ m-thick microdetector. For both microdosimeters, we observed that the active volume extends to the very edge of the detector. In the region from the center up to a radial distance of 10 µm, the CCE was greater than 93 %, whereas for distances between 10 µm and 12.5 µm the mean CCE was 75 %.



Figure 1. 2D CCE-map and CCE profile of the 20 μm-thick microdetector. [1] F. Gómez et al., "Measurement of carbon ion microdosimetric distributions with ultrathin 3D silicon diodes,"

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# ENHANCING GAMMA PRODUCTION FOR ONLINE DOSE VERIFICATION IN PROTON THERAPY

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The main rationale for using protons in cancer treatment is based on the depth-dose distribution, which translates into a superior sparing of normal tissue compared to conventional radiotherapy. This advantage can be fully exploited only if the match between the Spread-Out-Bragg-Peak (SOBP) and the tumor position can be accurately monitored. A mispositioning potentially translates into an under-dosage of the tumor as well as an over-dosage of the normal tissue, which can significantly hinder the treatment efficacy.

We present a novel strategy for real time dose verification. The methodology is based on the detection of prompt gammas (PG), whose production is artificially enhanced with a non-radioactive element transported selectively to the tumor with a drug carrier. Nuclear interactions of this element with the proton beam generate a signature PG spectrum, from which the tumor position can be reconstructed.

To provide a proof-of principle of the methodology and to identify potential candidate elements, we performed Monte Carlo calculations with Geant4, simulating the interaction of protons at clinical energies with 9F, 31P, 63Cu and 89Y targets. These elements were chosen because of their low abundance in the human body, which minimizes the background influence, and the existence of a compatible drug carrier, which guarantees an applicability in the clinic.

In addition, we measured the PG energy spectra emitted by solutions of water and the stable elements of 63 Cu (CuSO<sub>4</sub>+H2O), 31P (NaH<sub>2</sub>PO<sub>4</sub>+H<sub>2</sub>O) and 89Y (Y(NO<sub>3</sub>)<sub>3</sub>+H<sub>2</sub>O), when irradiated with 70 MeV protons. We also investigated the minimum element concentration in water to detect the PG enhancement. Both simulations and experiments indicated that 63Cu and 31P are the most promising elements, ass they produce signature PGs in the 800 keV -1400 keV range, with an enhancement of about 7% (31P) and 15% (63Cu) at a 0.5% concentration.

# INVESTIGATING A POTENTIAL HEALTH RISK DUE TO RADIATION FROM SAMPLES COLLECTED IN CHAD

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Radioactivity is well known and understood, but its usefulness in industrial applications to optimise processes or increase economic viability is not yet fully utilised by many industries. A study to ascertain the radioactivity levels of primordial and man-made radionuclides from samples collected in Chad is ongoing. The calculated activity concentrations are determined for the radionuclides: 226Ra, 214Pb, 214Bi and 228Ac, 208Tl, 212Pb following the decays of the primordial radionuclides as well as 40K and 137Cs (anthropogenic). This present study uses gamma-ray spectroscopy in a low background measuring system employing a high resolution Hyper-Pure Germanium (HPGe) detector. To estimate the levels, weighted averages for the radionuclides of interest will be used to assess the radiation risk. The analysis results and conclusion will be presented at the conference.

# ION AND NEUTRON BEAM IRRADIATION OF MATERIALS
# ION IMPLANTATION AND RADIATION EFFECTS IN GROUP-III NITRIDE SEMICONDUCTORS

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Group-III nitrides namely, GaN and related alloys, became famous beyond the nitride community due to their applications in light emitting diodes and lasers for solid-state lighting and data storage. Furthermore, these wide bandgap semiconductors are promising materials for radiation resistant electronics. A better understanding of radiation damage is required to assess their potential for space and nuclear applications as well as for the implementation of ion implantation as processing technique for nitride electronics.

The defect accumulation in GaN upon medium energy ion implantation was investigated using a combination of Rutherford backscattering spectrometry/channelling (RBS/C) and transmission electron microscopy (TEM). Interestingly, the damage accumulation is strongly influenced by the surface orientation of the samples [1]. In particular, basal stacking faults are the dominant implantation defects in c-plane GaN while dislocation loops predominate in a-plane samples. Challenges of RBS/C data analysis in the presence of extended defects will be discussed. Implantation into GaN nanowires, on the other hand, causes a much smaller density of extended defects compared to thin films [2]. This fact, together with the superior crystalline quality of as-grown wires, makes these one-dimensional structures interesting building blocks for small and flexible radiation sensors [3].

In space, materials will be subjected to intense radiation fields including a significant flux of heavy energetic ions with high ionisation potential. Upon irradiation with so called swift heavy ions, cylindrical tracks with amorphous cores are formed. Nevertheless, even for very high fluences, the crystal is never completely amorphised. Combining experiments with Molecular Dynamics simulations the reason for this strong radiation resistance was shown to be strong in-track and inter-track recrystallization of the region melted by the ion passage and even beyond [4].

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# UNIQUE HIGH ENERGY NEUTRON BEAMS AT ITHEMBA LABS

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iThemba Laboratory for Accelerator Based Sciences (iThemba LABS) is a national facility of the National Research Foundation (NRF) in the Republic of South Africa. Activities at the facility are based around a number of subatomic particle accelerators. The largest, a K-200 separated sector cyclotron, accelerates protons to a maximum energy of 200 MeV and heavier particles to much higher energies. The facilities provide opportunities for modern research, advanced education and the production of unique radioisotopes.

The iThemba LABS neutron beam experimental facility is one of the few facilities available to provide quasi-monoenergetic neutron beams in the energy range, 30 MeV to 200 MeV [1]. Quasi-monoenergetic neutron beams in this range are produced in the neutron experimental vault via the <sup>7</sup>Li(p,xn) or <sup>10</sup>Be(p,xn) reactions [2] for varying thicknesses of Li and Be targets. Collimated fan beams are possible at neutron emission angles amongst others of 0° and 16°. These neutron beams at iThemba LABS have been well characterized, refer to [3,4,5]. The facility is to be designated by the National Metrology Institute of South Africa as an entity responsible for providing traceability for the medium and high-energy neutron measurements in South Africa. Thus, the facility is intended to support neutron physics and metrology communities for calibrations of neutron detectors and radiation protection instruments. Cross-section measurements for neutron-induced reactions in the medium to high-energy region can also be performed.

For this contribution, we present some of the ongoing developments and future plans of the facility.

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#### SILICON QUANTUM TECHNOLOGIES WITH IMPLANTED DONORS

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Donor spin qubits in silicon are one of the most promising platforms for building a quantum computer. Single-spin readout, long coherence times and quantum gate fidelities approaching the error correction thresholds have all been experimentally demonstrated. Nevertheless, a convenient coupling mechanism between spins that would also be compatible with current ion implantation capabilities is still lacking. This is a crucial requirement for scaling up the single qubit demonstrations into a true quantum computing platform.

In this talk I will first provide an introduction to donor spin qubits in silicon and explain the state-of-the-art. I will then discuss the current ideas for scaling up and especially present the work done in my group aiming to solve the scalability issue. I will also focus on what are the requirement for ion implantation that will be needed in different scaling-up schemes.

### ION BEAM MODIFICATION OF DIAMOND FOR BIOSENSIG APPLICATION

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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 compart [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 nanobeam [5].

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#### SOLID STATE PHYSICS AT ISOLDE-CERN

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ISOLDE-CERN is a large-scale facility for the on-line production of exotic radioactive isotopes, with high yields, high elemental selectivity and isotopic purity. Solid-state physics research at ISOLDE-CERN profits from the delivered radioactive ion beams since 1976 to conduct experiments using various material characterization techniques such as self-diffusion, emission Mössbauer spectroscopy, time-differential perturbed angular correlations, photoluminescence and emission channeling. The quality of the obtained results have long attracted interest from researchers beyond the core nuclear physics community, and have established an extremely active programme in applied nuclear physics. This presentation describes the current status of this research programme along with recent illustrative results, predicting a brilliant future for the unique employed research methods and collaborations.

# NUCLEAR MATERIALS AND ION IRRADIATION STUDIES USING THE JANNuS-ORSAY *IN SITU* DUAL ION BEAM TRANSMISSION ELECTRON MICROSCOPE

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*In situ* Transmission Electron Microscopy (TEM) with ion implantation/irradiation is a key tool to investigate a wide field of topics, such as phase transition far from the equilibrium, solid-state nucleation/growth, defects recombination, and ageing of materials used in nuclear or space industries.

In the 1980's our laboratory was a pioneer in operating this kind of facility [1]. Thanks to this long experience, we were able to extend our *in situ* experimental potential to multiple simultaneous rastered beams, by building our first *in situ* dual ion beam TEM setup. Based on a 200 kV Tecnai G<sup>2</sup> 20 Twin FEI microscope coupled with two ion accelerators [2], it offers a large choice of ions and energies (range from 10 keV to 6 MeV) and complementary analytical equipments (EDX, STEM and EELS) associated with different specimen holders (from LN<sub>2</sub> temperature up to 1000°C). Launched in 2009 as an open facility, it allows each year 6 to 8 worldwide teams through the EMIR&A French accelerators network [3] to process their *in situ* experiments.

In this presentation, in the light of a selection of scientific results obtained throughout the past years, I will give an overview of the JANNuS-Orsay *in situ* TEM facility along with my own feedback as a local contact. Examples will be given on different materials studied for fission and fusion nuclear applications, and especially on recent results obtained (i) on the behavior of boron carbide under irradiation as a function of crystallographic orientations and temperature [4], and (ii) on uranium dioxide matrix destabilisation induced by incorporated impurities to get some insight on the early steps of the High Burnup Structure [5].

The JANNuS-Orsay/SCALP technical staff (C. Bachelet, C. Baumier, P. Benoit-Lamaitrie, J. Bourçois, S. Hervé, S. Picard), the other local contacts (C. Baumier, B. Décamps, S. Jublot-Leclerc), are all gratefully acknowledged for their unfailing assistance.

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### HIGHLY CHARGED ION INTERACTION WITH SURFACES

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Technologies and experimental techniques based on ion physics typically make use of the ion's energy and mass for sputtering and damage creation, angle of incidence for scattering and analysis techniques, beam focus in lithographic approaches, and many others; omitting one parameter unique to ions: the charge state. In contrast to electron beam techniques, ions can alter their charge state typically considered as an obstacle e.g. when magnetic and electrostatic detectors are used. The charge fractionization depends on the ion energy as well as electronic target properties, making a simple predictive model challenging.

We use a different approach and investigate charge exchange of ions in freestanding 2D materials as those enable us to study the dynamics on the natural time scale of charge transport. Varying the ion kinetic energy in the 1-500 keV range and the material thickness on the atomic level allows us to probe charge exchange and corresponding secondary processes in the (sub-)femtosecond regime [1]. Slow highly charged ions are herein an ideal tool, because they yield a far-from equilibrium initial charge, whose change can be monitored easily in experiment.

We observe that charge equilibration and the transition to the atomic/ionic ground state of the projectile is surprisingly fast, also for highly charged ions, which cannot be explained by conventional intra-atomic Auger cascades [2]. Taking into account inter-atomic (Augerlike) de-excitation channels, we elaborated a model for both charge transport and ion deexcitation (necessary to stabilize the captured electrons) [3]. With the help of this model we can now use charge exchange spectroscopy to probe material porosity in the limit of single scattering. This charge exchange spectroscopy is particularly helpful for short-range ordered molecular nanosheets, where the atomic structure cannot be deduced from electron- or x-ray-based methods.

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# DEVELOPMENT OF THE TAGGED NEUTRON METHOD FOR ELEMENTAL ANALYSIS AND NUCLEAR REACTION STUDIES – THE TANGRA PROJECT

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The TANGRA (<u>TAgged Neutrons & Gamma RAys</u>) project was started at JINR in collaboration with several other institutes [1]. The aim of the project is to continue the development of the tagged neutron method (TNM) and to use this method for applied tasks, as well as for experimental studies of neutron-induced nuclear reactions. The main idea of the TNM is the registration of the  $\alpha$ -particles, which are produced in the d(t, $\alpha$ )n reaction and emitted simultaneously with the 14.1 MeV neutrons, but in opposite direction. Using position sensitive  $\alpha$ -particle detector can help to identify precisely the direction of emission of the corresponding tagged neutron. The use of the coincidence between  $\alpha$ -particles and  $\gamma$ -rays, emitted from the interaction of the tagged neutrons with a target can reduce the background-to-signal ratio by more than 200 times, which significantly improves the quality of the measurements. Another important advantage of the TNM is the possibility of monitoring the flux of tagged neutrons incident on the target, with a high efficiency.

We're using a portable neutron generator ING-27 [2] and a set of  $\gamma$ -ray and neutron detectors in order to investigate the applicability of this method for elemental analysis (e.g., determination of elemental content of geological samples and ores, searching of diamonds in kimberlite ores, etc). The results of various test measurements and Monte Carlo simulations will be presented.

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# A GROUND-BASED EVALUATION OF THE IMPACT OF NEUTRON DOSE RATE ON HEALTH EFFECTS DURING SPACE TRAVEL

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The lack of information on how biological systems respond to low-dose and low dose-rate radiation makes it difficult to accurately assess the corresponding health risks. This is of critical importance to space radiation, which remains a serious concern for long-term manned space exploration. Therefore, a growing number of particle accelerator facilities implement ground-based analogues to study the biological effects of simulated space radiation. In this presentation, we will introduce first results of the 'INVEST' project at iThemba LABS, which aims to implement a first ground-based set-up to study space health effects in Africa. The focus of this work is on neutron irradiation, which is considered to be an important secondary component in space radiation fields. In a first set of experiments, the effect of neutron dose rate on immune system alterations and DNA double-strand break (DSB) induction and repair was investigated. Blood samples of adult volunteers were exposed to p(66)/Be(40) neutron neutron irradiation (fluence-weighted average energy: 29.8 MeV) at a lower dose rate (LDR) of 0.015 Gy/min or a higher dose rate (HDR) of 0.400 Gy/min. DNA DSB formation was 40% higher at HDR exposure compared to LDR exposure. The DNA DSB levels decreased gradually to  $1.65 \pm 0.64$  foci/cell (LDR) and  $1.29 \pm 0.45$  (HDR) at 24 h post-irradiation, remaining significantly higher than background levels. The impact of neutron dose and dose rate on immune alterations was studies using the in vitro cytokine release assay. Recall antigens and mitogens were used to activate lymphocytes post-irradiation and dose rate effects on the cytokine production capacity of the cells were observed under specific conditions. The results give a first indication that the dose rate should be taken into account for health risk estimations related to neutron irradiation.

<u>Funding acknowledgement</u>: Funding for the collaborative INVEST project was made available by the joint SA-NRF and Belgian Federal Science Policy Office (BELSPO) funding program under grant reference number BELS180425324336. Research consumables and beam time costs were funded by NRF iThemba LABS' institutional grant.

### RECENT CALCULATIONS FOR D<sub>2</sub>O MODERATED <sup>252</sup>Cf REFERENCE FIELDS AT PTB

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# Abstract

The neutron reference field produced by a heavy-water (D<sub>2</sub>O) moderated <sup>252</sup>Cf source is used routinely at the Physikalisch-Technische Bundesanstalt (PTB) for calibrating neutronmeasuring devices for radiation protection purposes. Knowledge of the precise neutron spectrum is very important for investigating operational quantities. Recently, a new fission spectrum has been proposed based on the latest cross section data and a new transport code. At PTB, the last calculations for the D<sub>2</sub>O moderated <sup>252</sup>Cf source were prepared more than 20 years ago, thus updated and more detailed calculations are required. To this end, a detailed simulation model of source assembly has been prepared and investigated using new spectral data and new Monte Carlo Transport codes, MCNP6 and PHITS 3.22, accompanied by a latest cross section data libraries, ENDF/B-VIII.0 and JENDL 4.0, respectively. The neutron spectra at different distances from the source and at the reference point were calculated. The ambient dose equivalent using the fluence to dose equivalent conversion coefficients, provided by ICRP74, and ambient conversion coefficient for the current PTB assembly were evaluated. The values were compared to the previous calculations performed at PTB and to the recent available published results. In addition, the effect on the calculated conversion coefficient of using different versions of cross section data libraries were studied.

#### RADIATION DEFECT DYNAMICS IN $\beta$ -Ga<sub>2</sub>O<sub>3</sub>: ION FLUX VS IRRADIATION TEMPERATURE

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Gallium oxide ( $Ga_2O_3$ ) belongs to a class of ultra wide bandgap semiconductors and, currently, it is considered as one of the most promising materials for the next generation power electronics. However, insufficient understanding of intrinsic defects limits bright applications of  $Ga_2O_3$ . Here, we use general approach to investigate radiation defect dynamics in  $Ga_2O_3$  and measure the dose-rate effect on structural disorder by varying such irradiation parameters as the ion flux and the irradiation temperature.

More specifically, monoclinic (010)  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> single crystals were implanted with 400 keV Ni ions in wide ranges of ion fluxes (8×10<sup>10</sup> - 5×10<sup>12</sup> at.cm<sup>-2</sup>s<sup>-1</sup>) and irradiation temperature (25-300 °C) keeping the total accumulated ion dose constant at 6×10<sup>13</sup> cm<sup>-2</sup>. Disorder build-up in the samples was analysed by a combination of the Rutherford backscattering spectrometry in channeling mode and x-ray diffraction. The results are summarized in Fig. 1 demonstrating a strong dose-rate effect in the temperature range of 25-250 °C. Furthermore, using methodology [1] we determined a critical temperature for each ion flux and extracted the activation energy of the dose-rate effect (see the inset in Fig. 1). The obtained value of  $E_a$ =0.8±0.1 eV is correlated with migration barrier for gallium vacancy, consistently with the theoretical data [2].



Figure 1 Maximum relative disorder as a function of irradiation temperature for three different ion fluxes. The inset shows the Arrhenius plot used to extract the activation energy of the dose-rate effect in  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> [3].

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#### **ONE-STEP 3D MICROSTRUCTURING OF PMMA USING MEV LIGHT IONS**

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The conventional procedure for creating 3D microstructures in resists by ion beam lithography consists of two stages – exposure and developing. However, single stage of manufacturing 3D structures in resist is also possible. Irradiation of Polymethyl methacrylate (PMMA) can cause it to shrink. This feature of the polymer can be used for one-step three-dimensional microstructuring, which simplifies the manufacturing process. The shrinkage of PMMA film on a substrate has been extensively studied, while research on free-standing film is not comprehensive. The use of free-standing PMMA film allows the creation of a flexible material with 3D microstructures, which can be used in medicine, optics, and electronics. The question here is whether the results obtained for the PMMA film on the substrate are applicable to the free-standing film. Since the nature of shrinking is outgassing of volatile products, the film on the substrate has only one surface for the release of gases, while in the free-standing film, gases can be released from the sample from both sides. Therefore, the shrinking in the free-standing film occurs on both sides. The aim of this work is to study the shrinkage of the free-standing film and compare it with that of the film of the same thickness coated on the substrate.

#### **RESPONSE OF DEFECTIVE KTAO3 TO IONIZING ION IRRADIATION**

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Pre-damaged KTaO<sub>3</sub> has been irradiated with18 MeV Si and 91.6 MeV Xe ions at 300 K to examine the effects of electronic energy loss (Se) on damage evolution, and compared with previous results obtained for 21 and 358 MeV Ni ions. The results unambiguously show that the pre-damage states have a significant synergistic effect on the response of  $KTaO_3$  to the dissipation of S<sub>e</sub> that enables the enhanced creation of linear amorphous nanostructures or tracks in the regions with pre-existing damage. Experimental characterization and computer simulations reveal that the size of these latent ion tracks increases with S<sub>e</sub> and level of pre-existing damage. These results further reveal that the threshold Se value (Se<sup>th</sup>) for track creation increases with decreasing predamage level. The values of Se<sup>th</sup> increase from 5.02 keV/nm for a pre-existing fractional disorder of 0.53 to 10.81 keV/nm for pristine  $KTaO_3$ . Above these thresholds, amorphous latent tracks are produced due local melting and rapid quenching. Below a disorder fraction of 0.07 and  $S_e \le 6.68$  keV/nm, the synergistic effect is not active, and damage accumulation is suppressed due to a competing ionizing-induced damage annealing process. These results indicate that, depending on Se and the amount of preexisting damage, highly ionizing ions can either enhance or suppress damage accumulation in KTaO<sub>3</sub>. Comprehending the conflicting roles of highly ionizing ions in defective ABO<sub>3</sub> oxides is vital for understanding and predictive modeling of ion-solid interactions in complex oxides, as well as in the microfabrication of waveguides and design of electro-optical devices resistant to cosmic radiation.

#### ENERGETICS, MIGRATION AND TRAPPING OF Zn INTERSTITIALS IN ION IMPLANTED ZnO

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ZnO is a direct wide band gap semiconductor having numerous potential applications in optoelectronics and power electronics. However, insufficient understanding of intrinsic defects hinders realization of ZnO-based devices. Previously, it was demonstrated that the behavior of residual Li in ion implanted ZnO depends on the preferential localization of the implants [1], in particular forming characteristic Li depleted, or Li pile-up regions for Zn O sublattice occupation of the implanted impurity.

In the present contribution we use this methodology in a combination with conventional RBS channeling measurements to study damage annealing kinetics in ZnO implanted with elements residing predominantly on Zn-sublattice (Er, Si, B) and molecular BF<sub>2</sub> ions. We demonstrate that Li behavior depends on the defect annealing kinetics which is a strong function of the implanted fluence and ion species. In particular, the formation of Li depleted and Li piles-up regions, or even combinations of the two, were observed. This is illustrated by Fig. 1, showing a dramatic decrease of the Li lean region accompanied with Li pile-up around  $R_{pd}$  for BF<sub>2</sub> as compared to single B implant. It is discussed how the observed Li redistribution trends can be used for better understanding of the energetics and migration/trapping properties of Zn interstitials (Zn<sub>i</sub>) in ion implanted ZnO [2]. Furthermore, the obtained results give a possibility to control and manipulate the Zn<sub>i</sub> flux during post-irradiation thermal treatments by co-irradiation schemes.



Figure 1 Li depth profiles in ZnO implanted with  $B^+$  and  $BF_2^+$  ions to  $3 \times 10^{15}$  ions/cm<sup>2</sup> after 800 °C anneals. The inset shows an Arrhenius plot of effective  $Zn_i$  diffusion obtained for B ion implantation.

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#### ION BEAM SYNTHESIS OF HIGH OXIDATION STATE PALLADIUM OXIDE NANOPARTICLES

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The well-known catalytical properties of Pd and PdO may be enhanced by increasing its oxidation state [1].  $Pd^{(IV)}O_2$  is of particular interest, yet it is virtually missing on the map of transition metal oxides, where only a single study reports its successful synthesis in bulk form in reasonable amounts [2], most approaches stabilize  $Pd^{(IV)}$  cations by additional species or oxides in the system or report  $PdO_2$  as a monolayer on PdO nanoparticles [3]. We used radiofrequency sputtering with a high purity Pd wire acting as the cathode of the oxygen plasma discharge to deposit polycrystalline layers of PdO on Si (100) substrates, which were subsequently oxygen ion implanted using plasma immersion ion implantation (PIII) to reach the 1:2 ratio between Pd and O in the implanted layer. Results of the first experiments and characterization of the layers are presented and discussed.

This research was funded by the European Regional Development Fund under contract No. ITMS2014+: 313011W085 and the Slovak Research and Development Agency under contract No. APVV-18-0168.

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# STOPPING FORCE OF DIAMOND LIKE CARBON AND SILICON NITRIDE FOR BERYLLIUM AND BORON IONS

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The energy loss of ions in materials is of crucial importance in fields such as Ion Beam Analysis (IBA) and ion beam modification of materials. Diamond like carbon and silicon nitride thin-films are regularly used in IBA and Accelerator Mass Spectrometry (AMS) as detector window, filter, and electron stripping foils. <sup>10</sup>Be AMS analysis is of increasing interest for geological and environmental applications. Isotopes of Be and B are very similar from an AMS measurement perspective, where contaminant B beams have to be separated from Be. The B isobars can be separated in the detector system or with the use of degrader foils, which requires knowledge of stopping forces. During the development of <sup>10</sup>Be AMS at University of Helsinki, we have become aware of the paucity in experimental stopping force data of Be and B ions in diamond like carbon and silicon nitride in the literature. Accurate stopping force values of these materials for Be and B ions would be of benefit to Be AMS development, as it would help in reducing the isobaric interference of <sup>10</sup>B with <sup>10</sup>Be isotopes.

In this work, we determined stopping forces of  $Si_3N_4$  and C for B and Be ions. We measured the energy loss of Be and B ions for the energy range of 500 keV to 15 MeV in diamond like carbon and silicon nitride foils. Measurements were made with a modified Elastic Recoil Detection Analysis setup using a time-of-flight detector enabling us to measure the entire energy range at once. Energy losses were measured for different thickness of diamond like carbon and silicon nitride foils using a stopping foil holder with three foils, making possible to measure several samples without breaking the vacuum. The effect of the different isotopes on stopping forces was studied using <sup>10</sup>B and <sup>11</sup>B beams. Experimental stopping forces from this work were compared against SRIM predictions and experimental values from literature. The results agree well with SRIM, differing at most by 15%. We did not observe any difference in the stopping force for different projectile B isotopes within experimental uncertainty.

# ION TRACK FORMATION IN SAPPHIRE STUDIED BY SEQUENTIAL SWIFT HEAVY ION IRRADIATION

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Small size of ion tracks in sapphire has been difficult to accomodate within thermal spike framework [1]. Prompt ion track recrystallisation has been proposed to explain this feature of the sapphire [2,3]. To test this hypothesis, we have irradiated sapphire sequentially by two different swift heavy ion beams. In the first step, ion tracks have been formed by 83 MeV Xe ion beam. In the second step, 18 MeV Cu has been used to simulate prompt annealing because its electronic stopping power is just below the threshold for ion track formation. Experimental results obtained by the RBS/c are discussed within the thermal spike framework.

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#### ENERGY RETENTION IN SWIFT HEAVY ION IRRADIATED THIN FILMS

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Swift heavy ion (SHI) beams are a unique tool for materials nanostructuring with diverse uses such as nanomembrane production, catalysis, sensing or optical waveguide fabrication. All of these applications are enabled by the material damage produced along the SHI path. Current understanding of this damage formation in insulators and semiconductors is based on the thermal spike model, that intense electronic excitation caused by SHI passage can lead to material heating via electron-phonon coupling. If deposited energy is sufficiently high, this can lead to material melting and ultimately to formation of permanent damage, as defects remain "frozen" during rapid cooling. SHI beams have also been found to be very useful in 2D materials nanostructuring. For example, perforation of graphene has been found in wide range of energies [1-3] and production of graphene nanomembranes in this way has been successfully demonstrated [4]. Still, it remains an open question if damage formation in graphene and similar 2D materials can proceed in the above manner. More specifically, SHI impact in 2D material and thin film targets, could be less damaging because deposited energy can dissipate away by emitted secondary electrons. Here we present results of our analyses, based on Geant4 simulations, of energy dissipation in thin graphite and silicon targets. We found that even for the thinnest targets in our study, the majority of the deposited energy still remains within the target.

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# ZNO NANO-PILLARS DECORATED WITH AU NANOPARTICLES PREPARED BY ION BEAM IMPLANTATION – STRUCTURE MODIFICATION AND OPTICAL PROPERTIES

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ZnO nanopillars of 500 nm in length deposited on Si wafer substrates were implanted with Au 400 keV ions at various ion fluences and sub-sequently annealed at 750°C for 15 minutes. The Au coalescence and/or incorporation into ZnO nanopillars interplay with radiation damage was followed before and after annealing in connection to structural modification and optical properties – luminescence and surface plasma resonance. ZnO nanorods have been implanted with Au ion-fluences  $1x10^{15}$  cm<sup>-2</sup>,  $5x10^{15}$  cm<sup>-2</sup> and  $1x10^{16}$  cm<sup>-2</sup>. Rutherford backscattering spectrometry (RBS) was used to follow Au distribution in ZnO nanorods, Raman spectroscopy have shown the damage accumulation in the irradiated nanorods partially recovered after the annealing. Scanning electron microscopy (SEM) followed a complex morphology of modified ZnO nanorods decorated with Au as-implanted and as annealed, simultaneously XRD analysis corroborated recovery of radiation damage after the annealing. Photo-luminescence measurements have shown surface plasmon resonance (SPR) response, developing progressively with the increasing Au-ion fluence and annealing in ZnO nanopillars.

# PROPERTIES OF GRAPHENE OXIDE, POLYIMIDE, POLYETHERETHERKETONE AND POLYETHYLENETEREPHTHALATE IMPLANTED BY MULTI-ENERGETIC AU IONS

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Multi-energetic ion implantation was employed as a method for production of 3D structures of Au nanoparticles in insulating graphene oxide (GO) and polymers matrix. (PI), Graphene oxide (GO), polyimide polyetheretherketone (PEEK) and polyethyleneterephthalate (PET) were implanted using Au ions with energy in range from 800 keV to 3.2 MeV and with fluences 1.875 and 3.75x10<sup>14</sup> cm<sup>-2</sup>. The Au depth profiles were determined by RBS and compared with simulated ones using SRIM code. The changes in structure and elemental composition of the irradiated area were characterized by Raman spectroscopy, X-ray Photoelectron Spectroscopy, Rutherford Back-scattering Spectroscopy and Elastic Recoil Detection Analysis spectroscopy. As follows, the shape of the Au nano-structures was studied using SEM and TEM analysis. The surface morphology of the implanted samples was studied using AFM. The electrical properties of the structures were studied in relation to the used ion fluence, by standard two point method. We can conclude that the resulting electrical conductivity depends on the used ion fluence and type of insulating matrix.

The research has been realized at the CANAM (Center of Accelerators and Nuclear Analytical Methods) infrastructure LM 2015056 and has been supported by project GACR 19-02482S. This publication was supported by OP RDE, MEYS, Czech Republic under the project CANAM OP.

# THE STRUCTURAL AND OPTICAL RESPONSE OF THE AU NANOPARTICLES EMBEDDED IN YSZ MODIFIED USING HIGH-ENERGETIC ION IRRADIATION

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We tried to deform gold nanoparticles (NPs) implanted in crystalline yttria-stabilized zirconia (YSZ) using high-energetic silicon ion irradiation. YSZ is a highly resistant material to radiation that exhibits different resistivity in different crystallographic orientations. The (100)-, (110)- and (111)-oriented YSZ samples were implanted with 1 MeV Au<sup>+</sup> ions at room temperature and fluences of  $1.5 \times 10^{16}$  cm<sup>-2</sup>,  $5.0 \times 10^{16}$  cm<sup>-2</sup> and  $7.5 \times 10^{16}$  cm<sup>-2</sup>. Subsequent annealing for 1h at 1100°C was provided in an ambient atmosphere. Deformation by 10MeV Si<sup>3+</sup> implantation with fluence  $5 \times 10^{14}$  cm<sup>-2</sup> was attempted to modify Au distribution and the structural modification of the YSZ host lattice. The structure modification, as well as Au particle incorporation, was studied using Rutherford backscattering spectrometry (RBS) and RBS in channelling configuration (RBS-C). The RBS spectra indicate that the implanted impurity concentrates near the surface of the YSZ. Optical characterization was done using ellipsometric measurement. The formation of gold NPs in the YSZ can be judged from the characteristic surface plasmon resonance band in the reflectance spectra of the samples implanted to a fluence higher than  $5.0 \times 10^{16}$  cm<sup>-2</sup>.

#### Acknowledgement

The research has been carried out at the CANAM (Centre of Accelerators and Nuclear Analytical Methods) infrastructure LM 2015056 and supported by GACR 18-03346S project.

#### PLASMA IMMERSION ION IMPLANTATION INDUCED SURFACE PATTERNING

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Ion irradiation surface nanopatterning is a well-established technology to induce selforganized nanopatterns even on large surfaces [1]. Commonly employed are Kaufmann ion sources with monochromatic broad beams, where size of the ion source is limiting the surface size being processed and the technology is limited to the processing of flat surfaces only. Plasma immersion ion implantation (PIII) on the other hand, enables ion bombardment of non-flat surfaces and is easily scalable, yet studies on using PIII for surface patterning are missing. Our investigation focuses on the feasibility of using PIII for surface (nano)patterning, particularly the effect of the polychromatic nature of the irradiation on the pattern evolution. We investigated the surface morphology of various single-element metallic surfaces irradiated under normal incidence using Ar ions and 1 kV accelerating potential. First experiments on Al(111) surfaces resulted in kite-like structures, however with significant deposition of SiO2 eroded from the quartz tube serving as a protector for the cathode of the radiofrequency discharge.

This research was funded by the European Regional Development Fund under contract No. ITMS2014+: 313011W085.

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#### ASSESSING ELECTRONIC EXCITATIONS IN SINGLE-CRYSTALLINE SIC FOILS BY KEV IONS

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Accurate knowledge of the energy deposition of energetic ions in matter is crucial for fundamental as well as applied science and technology. In fundamental research, studying the stopping of slow ions (around the Bohr velocity) is the first step in understanding how dynamic processes such as charge exchange can contribute to target excitation. In applications, knowledge of the specific energy loss is a necessity for materials characterization and modification based on energetic ions, e.g. in ion beam analysis, ion implantation, irradiation, sputtering and for hadron therapy.

Silicon carbide (SiC) is a material of high technological interest since it exhibits excellent physical and electronic properties e.g. wide bandgap and high radiation tolerance. Such properties render SiC an ideal material for use in high-temperature power electronics and in harsh environments, e.g. fusion devices or space applications [1,2]. Data on the specific energy loss in SiC at ion energies below the Bragg maximum is, however, scarce and furthermore largely obtained from a combination of ion implantation and secondary ion mass spectrometry experiments [3].

Experiments were performed using the Time-of-Flight Medium Energy Ion Scattering System at the 350 keV Danfysik Implanter at the Uppsala University [4]. Pulsed beams of H, He and heavier ions, with typical pulse length of ~1 ns, were directed on the SiC foil with energies of 60 - 300 keV. Transmitted ions were detected by a large-angle, position-sensitive detector. The target was a single-crystalline, self-supporting SiC (100) foil (Norcada Inc.) with nominal thickness of 200 nm. The recorded angular distribution is shown in Fig. 1 for transmitted He projectiles with a primary energy of 60 keV.

The energy loss was measured along random and several different channelling trajectories. The specific energy loss was extracted from experimental data and compared for all ions. Data is found to deviate from SRIM predictions [5]. Ions heavier than protons, show a similar discrepancy between electronic stopping along random and channelling trajectories as earlier observed for Si (100) [6]. The experimental results provide an ideal dynamic system for benchmarking theoretical models which predict equilibrium (DTF) and non-equilibrium (TD-DFT) electronic structures for solids.

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Figure 1. 2D distribution of 60 keV He ions transmitted through a SiC self supporting foil (100) with 200nm nominal thickness.

# CAPABILITIES OF THE ION BEAM MICROPROBE IN THE STUDY OF DIFFERENT POLARIZATION QUENCHING TECHNIQUES APPLIED TO SC-CVD DETECTORS

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In high resistivity materials, such sc-CVDs, the trapping of free carrier by defects during their motion produces an asymmetry in the space charge distribution in the sensitive volume of the diamond crystal. This asymmetry induces an internal electric field which is opposite to the electric field induced by the bias applied to the detector who reduce the intensity of local electric field. The reduction of the electric field increases the probability of free carriers recombination, which results in a significant deterioration of the charge collection efficiency (CCE). This is the polarization phenomena and produces serious disadvantages for diamond detectors use in practical experimental applications [1].

The feasibility of the application of diamond crystal as sensor material involves investigating approaches to suppress or attenuate these phenomena. In the literature, different techniques have been explored [2], including application of alternate bias polarity, thermal and optical excitation and irradiation by other types of ionizing radiation. In this work, the lon beam induced current technique (IBIC) has been used to study the properties of the polarization phenomena under the influence of these quenching techniques in different sc-CVD detectors.

The experiments were carried out at the ion beam microprobe at the Ruđer Bošković institute and the temporal evolution of the charge collection efficiency has been studied as a function of the ion beam species, the applied bias, the operating temperature and the illumination conditions.

Our experimental results indicate that switching bias Off during continuous irradiation can be used as a satisfactory method to recover the CCE to prepolarized state. In addition, the heating of the sample is also a particularly promising method for the reduction of the strength of the polarization. The experiments carried out also have shown a certain influence of light on polarization, suppressing the intensity of polarization induced by hole traps. Finally, probing the CCE using combinations of frontal and lateral IBIC in thick detectors have shown to be useful to quantify the effects of polarization.

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# IN-SITU TOF-LEIS STUDY OF TUNGSTEN SURFACE ENRICHMENT IN EUROFER97 BY ANNEALING TO ELEVATED TEMPERATURES

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EUROFER97 is a reduced activation ferritic martensitic (RAFM) steel developed in Europe as the candidate material for the structural components and parts of the first wall in future fusion devices [1]. We address a specific property of EUROFER97, namely the enrichment of alloying elements near the surface and grain boundaries after annealing. Such enrichment may modify the rate at which the steel is eroded under plasma exposure and can thus affect the lifetime of plasma-facing components [2]. This phenomenon, particularly, has been studied by different techniques like ToF-RBS [3], ToF-MEIS, ToF-ERDA [4] and ESA-LEIS [5].

In this work, we employed the ToF-LEIS technique which is a surface-sensitive method with the ability to measure backscattered neutral particles in addition to the ions. We used 8 keV Ne<sup>+</sup> projectiles to study the evolution of the near-surface composition during different temperature treatments. As a result, we detect a surface enrichment of W+Ta in a depth interval of less than 2 nm thickness after annealing the sample up to 670°C and maintaining it for one hour. Additionally, we observe surface segregation of chromium and sulfur. Moreover, these results confirm the existence of tungsten diffusion/segregation dominancy in specific temperature regions in EUROFER97 which was claimed in a previous study [2].

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# PROPERTIES OF POLYAMIDE 6 AND POLYVINYLIDENE FLUORIDE NANOFIBERS IRRADIATED USING C AND H IONS

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Polymer nanofibers have gained considerable attention in recent years due their excellent properties, such as large surface area, flexibility and toughness, for which can be used practically in many sensory, medical as well as electronics applications. In this work, the polymer nanofibers of polyamide 6 (PA6) and polyvinylidene fluoride (PVDF) were formed by electrospinning on a Nanospider<sup>™</sup> device. Subsequently, these nanofibers were irradiated using 5 MeV C<sup>3+</sup> and 1 MeV H<sup>+</sup> ions with different ion fluences  $(1,8; 3,6; 5,7; 7,5 \times 10^{14} \text{ cm}^{-2})$ . The purpose of ion irradiation of polymeric nanomaterials was to observe change of electric conductivity and sensory properties. The change in physical and chemical properties of polymers was studied by nuclear and spectroscopic analytical techniques (RBS and ERDA, X-ray photoelectron spectroscopy XPS and infrared spectroscopy FTIR). Changes in surface topography were examined by SEM and AFM, simultaneously electrical properties were examined by two-point method. The humidity sensing ability of the ion-irradiated nanofibers has been studied in atmospheric chamber with variable humidity. The results showed after ion irradiation significant changes of the interior polymer structure, surface nanofiber topography as well as electrical and sensory properties depend on the type of used ions and the ion fluence.

The research has been realized at the CANAM (Center of Accelerators and Nuclear Analytical Methods) infrastructure LM 2015056 and has been supported by project GACR 19-02482S. This publication was supported by OP RDE, MEYS, Czech Republic under the project CANAM OP.

# IRRADIATION OF (111)-CaF<sub>2</sub> USING A MODERNIZED BEAMLINE IN UPPSALA

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An overview of the irradiation beamline setup at Uppsala University's 5 MV pelletron tandem accelerator laboratory is presented and recent modifications are highlighted. The layout of the target chamber is shown along with the sample transportation system using a magazine able to accommodate up to 20 samples for automated irradiation sequences. Samples up to 10x10 cm<sup>2</sup> can be mounted. The specifications of the raster scanner unit are given along with a summary of available projectiles from H to Au, energies from 1 to 50 MeV (the higher end of the range for heavy ions) and charge states from 1+ to 25+, scanned over areas of between approximately 1 and 140 cm<sup>2</sup>. Fluences from 10<sup>9</sup> cm<sup>-2</sup> to 10<sup>16</sup> cm<sup>-2</sup> can be achieved within a reasonable time and irradiation footprint homogeneity is shown. A modified control unit for the setup is introduced along with new possibilities for monitoring pressure and beam current during and between irradiations. Potential applications of the beamline include high energy implantations, nanostructure and ion track formation e.g. engineered nanopores mimicking the function of channels in biological membranes, resilience tests of electronics for radiation-rich environments and space applications as well as material modification on the nanoscale affecting optical and electromagnetic properties of thin films, nanodots and nanowires.

The first project carried out at the new beamline consists of irradiation experiments on single crystal  $CaF_2$  samples with the aim to investigate formation of nanohillocks for yet unexplored combinations of projectile charge state and energy. Hillocks have been observed after irradiation of  $CaF_2$  with heavy ions at energies around 100 MeV [1]. It has also been shown that a threshold for hillock formation exists near charge state 20+ for 5 MeV Xe and near 25+ for 3 MeV Xe [2]. For 180 MeV Au projectiles in charge state equilibrium, on the other hand, clusters in the size range of several nm are ejected from the material [3]. We present results obtained with heavy ions at energies from a few to tens of MeV, and charge states from 1+ to 15+.

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#### CHARGE STATE DEPENDENCE OF THE DAMAGE ONSET DEPTH IN SELF-IRRADIATED Ge

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We recently investigated the dependence of the energy loss experienced by heavy ions passing through this self-supporting foils on the initial ion charge state [1]. It is well known that the stopping power exhibits a charge state dependence [2,3] and scales approximately with  $Z_1^2$  [4]. Together with a model for the electron exchange between target and projectile, the energy loss could be used to draw conclusions about the length scale over which charge state equilibration occurs for projectiles with different velocities. In this contribution, we present the next logical step of the investigation; using the charge state dependence of the stopping power for heavy ions in the MeV range to affect the modification of an irradiated material.

Germanium samples are self-irradiated with ions in low charge states at intermediate energy (e.g. 2.5 MeV <sup>74</sup>Ge<sup>+</sup>) both directly and through foils of C and/or Au with a few tens of nm thickness. Charge state equilibration of the irradiating ions thus happens either in the foil or in the sample, with different damage onset depths as a result. Possible applications for creation of embedded nanostructures in single- or multilayer material systems are discussed.

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# EXPERIMENTAL ALPHA-PARTICLE MODIFICATIONS OF THE NATURAL RESINS.

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We have investigated alpha-particle damage from natural U-decay series in natural fossil resins (ambers) on a microscale. To understand chemical changes within the alpha-range it is necessary to obtain large enough specimens of irradiated resins for most of the analytical techniques. Thus, we designed two kinds of experiments. The first one is based on He<sup>+</sup> ion sputtering using Tandetron accelerator at the Nuclear Physics Institute of the Czech Academy of Sciences in Řež (NPI CAS). The second one is based on boron compound diffused within the bulk of devolatilized coniferous resin irradiated in LVR-15 research reactor at NPI CAS to obtain various alpha-doses via <sup>10</sup>B(n,  $\alpha$ )<sup>7</sup>Li reaction at several different irradiation times. The irradiated resins were submitted to vibrational spectroscopy as well as chromatography and mass spectrometry methods.



Fig. 1 Infrared spectra of analyzed ambers including non-irradiated sample.

Our results confirm the increase of aromatization and decarboxylation of resins in accordance with previous studies on naturally irradiated amber from Křižany and Hamr uranium mines.

The study has been supported by the Czech Science Foundation (GAČR) project 19-05360S "Radiolytic alteration of organic matter in uraniferous environment".

It has been performed within open-access infrastructures CANAM and SUSEN funded by the Ministry of Education, Youth, and Sports of the Czech Republic within project LM2015056 and by the Ministry of Industry and Trade, respectively.

# RAMAN SPECTROSCOPY INVESTIGATION AND MOLECULAR DYNAMICS SIMULATIONS OF ION TRACKS IN GRAPHENE

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Swift heavy ions have been used to introduce damage into supported single layer graphene. Immediately after irradiation with 23 MeV iodine beam, Raman spectroscopy has been undertaken to quantify defects due to ion bombardment. For the fixed ion irradiation parameters (i.e. ion beam type, energy and fluence), we observe significant change in the number of defects introduced into the graphene, when only ion beam charge state is varied. This is accomplished by comparing the samples irradiated with 6+ charge state beam directly delivered from the accelerator, with samples irradiated with beam having equilibrium charge state of 14+ after it has been stripped by passing through the thin carbon foil.

Following the experiment, we have used classic MD simulations based on the LAMMPS code to clarify mechanism of the ion track formation in graphene. Close to the threshold, we observe efficiency for ion track formation is less than one. With increasing energy loss of the projectile, we also observe increase of the ion track size and various trends in abundancy of the different types of defects.

### AN OPTIMIZED DT-NEUTRON GENERATOR IRRADIATION FACILITY FOR PROMPT NEUTRON ACTIVATION ANALYSIS OF LIGHT ELEMENTS

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Compact neutron generators are nowadays used in a growing number of applications such as non-destructive elemental analysis of various types of materials including CBRNE materials (chemical, biological, radiological, nuclear and explosives) or drugs [1]. Deuterium-tritium (DT) neutron generators emitting 14 MeV neutrons are especially suitable for detection of light elements (C, O, N) using the prompt neutron activation analysis in which characteristic gamma rays following the inelastic fast neutron scattering are detected [2]. The sensitivity of the method is closely linked to the reduction of detector background caused by primary or secondary neutrons and gammas emitted by surrounding materials (shielding, laboratory walls, soil, etc.). Neutron background can be reduced mainly by shielding primary neutrons and by pulse shape gamma/neutron discrimination [3].

There are two approaches to solve the gamma background problem: (i) to shield the gamma detector and/or (ii) to completely shield the neutron generator leaving only one irradiation channel open to reduce the activation of surrounding materials. Using Monte Carlo MCNP6 simulations for the DT MP320 neutron generator and two gamma spectrometers (3"x5"x16" NaITI or 30% HPGe) in the Laboratory for Neutron Activation Analysis and Gamma Spectrometry at VSB-Technical University of Ostrava [4] we investigated different optimized systems of materials (SM), homogeneous and sandwiched, to minimize the gamma background within the two approaches. In particular, we compare two SMs shielding the neutron generator: the first one composed of heavier elements to reduce the fast neutron flux while the second one composed of light elements to moderate and then to absorb neutrons.

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# PHOTOLUMINESCENCE AND EPR STUDIES OF SINGLE DIAMONDS WITH GeV-COLOR CENTERS FORMED BY ION IMPLANTATION

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At present, a large number of color centers in the diamond which could be used as single-photon sources are known. These include color centers such as NV-, SiV-, SnV-, NE8- centers, etc. [1]. However, among these color centers, only NV- and SiV-centers show reproducible results. Recently, room-temperature photoluminescence of GeVcenters had been observed in the diamond implanted with Ge ions at energy ranged from 150 to 260 keV [2]. The single photon emission capability of GeV-center with a ZPL line at around 602 nm had been demonstrated at first time. In the given work, Ge<sup>+</sup> ions were implanted in single crystalline (001)-face oriented plates of synthetic diamond (type IIa, NDT Ltd) at more low energy of 40 keV and the fluences in the range of (0.05-1.0)  $\times 10^{15}$  ion/cm<sup>2</sup>. The implanted diamond plates were then annealed at high temperature of 1273 K under high vacuum conditions (10<sup>-9</sup> mbar). A home-made confocal microscope and laser diode excitation at a wavelength of 520 nm were used to look for GeV-centers in diamond. The spectra of photoluminescence (PL) was taken at room temperature. In addition to the intense Raman line (558.6 nm), PL spectra are characterized by a narrow emission line at a wavelength of 601.5 nm related to GeV-centers. Electron paramagnetic resonance (EPR) was used to determinate the structural position and the crystalline symmetry of GeV-centers under study. However, EPR spectra reveal only one nonstructured line with g-factor of 2.003 (except very weak lines from NV-centers) at room and liquid nitrogen temperatures in both rf X- and Q-bands. Moreover, there not angular dependence of EPR spectra on the orientation of diamond crystalline plate with respect to magnetic field direction. The last suggests that the GeV-center has a more symmetrical position in crystalline structure of diamond than, for example, a SiV-center similar to it (point symmetry  $D_{3d}$ ).

This work was supported by the Mega-grant of the Government of the Russian Federation (Agreement No. 14.W03.31.0028).

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# OPTICAL PARAMETERS STUDY OF AMORPHOUS GERMANIUM ( $\alpha$ -Ge) BY SPECTRAL ELLIPSOMETRY

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Block justified text (in 300, max 2000 characters not including spaces) At ion implantation of Ge a porous layer is formed on its surface the thickness of which is several times more than the average ion range [1]. The possibility of creating a nanoporous structure on the Ge surface has led to a trend in recent years for the use of Ge in new areas of technology: in lithium-ion batteries as an anode material, as new materials for gas sensors and optoelectronic devices. As previously shown by the example of single-crystal silicon (c-Si) implanted with various types of ions in a wide energy range, spectral ellipsometry (SE) is an informative technique for studying partially amorphized semiconductor layers [2, 3]. This work presents the dispersion dependences of the optical parameters of  $\alpha$ -Ge obtained by the study results of a thick film of  $\alpha$ -Ge (1000 nm) grown by magnetron sputtering technique on a corundum substrate. These dependencies were used for SE study of c-Ge plate implanted with fluence  $4 \times 10^{16}$  ion/cm<sup>2</sup> of argon ions 300 keV energy. The established value of the disordered layer (amorphous layer) thickness during ion irradiation corresponds to the thickness of the layer obtained by simulating irradiation in the SRIM-2008 program [4].

The reported research was funded by Russian Science Foundation (grant No 19-79-10216).

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# ION BEAM ANALYTICAL METHODS IN MATERIAL SCIENCE
### RECENT ACHIEVEMENTS – AND CHALLENGES – IN ION BEAM ANALYSIS FOR MATERIALS CHARACTERIZATION

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In the late fifties and throughout the sixties, when nuclear physicists started abandoning their small accelerators, the field of ion beam analysis began to flourish. Techniques such as nuclear reaction analysis, particle induced X-ray emission, Rutherford backscattering spectrometry as well as ion channeling rapidly established themselves as key characterization techniques for studying, e.g., nuclear materials or ion-implanted semiconductors, two emerging research fields in that era.

Obviously, a lot has changed in the past sixty years. Amongst others, significantly better detectors have been developed, researchers have access to much more powerful algorithms for data analysis, etc. However, at the same time, the customer's demand has become much more stringent, e.g., aiming at analysis of nano-systems, extremely dilute dopant concentrations, or measuring in an extreme environment, thus challenging ion beam techniques in terms of sensitivity, resolution and accuracy.

Hence, a legitimate question is whether ion beam analysis will remain competitive for materials characterization, or whether we are reaching the limits. Will ion beam analysis continue to play a forefront role in a research era, which largely focuses on structures and properties at the nanometer-scale? If so, which role, and which technical advances do we need to achieve this goal?

In my talk, I will illustrate some specific capacities – and specific limitations – of current ion beam analysis using a selected set of classical and more challenging examples. In many cases, the combination of advanced equipment with a clever experimental design can provide nanoscale information, complementary to – and competitive with – other advanced characterization techniques.

### ION BEAM MODIFICATION FOR SI PHOTONICS

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Si quantum dots (Si-QDs) embedded in dielectric materials have been the subject of intense research due to their potential applications in optical and optoelectronic devices. A number of research studies have focused on Si-QDs made by implanting Si ions in SiO<sub>2</sub>. However, the mechanism by which Si-QDs luminescence is not fully understanding due to the difficulty in distinguishing between luminescence originating from defects in the SiO<sub>2</sub> matrix and Si-QDs.[1-3] Gaining an understanding of these processes would aid in the development of a Si- based light source. It is for these reasons that we have chosen to study the luminescence of Si implanted  $Al_2O_3$  films, for better interpretation of the luminescence spectra and its transparency makes it suitable of the fabrication of transparent devices. [4]

We have implanted Si ion into crystalline and amorphous Al<sub>2</sub>O<sub>3</sub> followed by an annealing process. The amorphous Al<sub>2</sub>O<sub>3</sub> films used were synthesized by anodization of Al foils, and crystalline samples were commercially acquired. Seevral ion beam analysis techniques were used to the study the composition of the samples produced these techniques include particle induced x-ray emission (PIXE), Rutherford backscattering spectroscopy (RBS), elastic recoil detection analysis (ERDA). Other complimentary techniques such as photoluminescence (PL) spectroscopy, scanning electron microscopy (SEM), powder x-ray diffraction (XRD and x-ray absorption near edge spectroscopy (XANES) were also utilised to study the structure, optical properties and I-V characteristics of the samples.

Their properties were explored using I-V measurements, photoluminescence, and X-ray photoelectron spectroscopy. Rutherford backscattering spectroscopy was used for depth analysis to accurately quantify Si atomic fractions and distributions. Different electrical transport mechanisms, including Fowler-Nordheim tunneling, Poole-Frenkel emission and hopping conduction were explored to understand how an ensemble of Si quantum dots or wells behaves in a matrix, with conduction via oxide tunneling and hopping effects between silicon interstitials and silicon QDs.

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### IBIC MICROSCOPY FOR SEMICONDUCTOR DETECTORS RESEARCH

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In this talk we will present the work we are currently doing at the Centro Nacional de Aceleradores (CNA) to study semiconductor detectors with the Ion Beam Induced Charge (IBIC) technique. These experiments are carried out using the microbeam line of our 3 MV Tandem accelerator, to take advantage of both its good lateral resolution and its capacity to produce very low intensity beams that allow for studies to be carried out without damaging the devices.

First, we will describe a methodology developed to study spectrometric and transport properties in semiconductor detectors, based on the drift and diffusion of charge carriers in the active and neutral regions of the detector. This methodology has been developed in the framework of a Coordinated Research Project of the IAEA: "Space-time structural evolution of materials induced by ion beams: Accelerators for a new technological era" to explore the ion dose rate dependence in the generation of point defects in Si photodiodes. The same procedure has been employed to analyse the radiation hardness of SiC detectors when irradiated with alpha particles, to investigate the possible use of this wide gap material in the high radiation, and high temperature environment of future fusion reactors.

Finally, we will explain on the capacity of the IBIC technique to characterize different Si detectors, manufactured at the Institute of Microelectronics in Barcelona, that are at the frontier of technology, i.e, i) the Low Gain Avalanche Detectors (LGADs), that will be included in the ATLAS and CMS experiments after the high-luminosity LHC upgrade because of their excellent timing properties and ii) new 3D-silicon microdosimeters that will allow a more accurate measurement of the dose received by patients in the hadrontherapy treatments.

### ION BEAMS AND SYNCHROTRON LIGHT IN PERSPECTIVE

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Ion beams have been traditionally by a very stable community, clustered around a large number of limited-size facilities and with a strong national dimension for each of such facilities. Synchrotrons and more recently free electron lasers are instead very large and costly facilities, which work for a very large user community with a strong international dimension. Ion beam analysis techniques are complementary to photon-based ones and become unique tools for some particular analytical challenges. In addition modification of materials is a unique niche of ion beam facilities. However it is not frequent to see experimental works bridging over these too worlds. CMAM (at Madrid Autonomous University) will be described as an example of an ion beam facility and some examples will be given of experimental work in which ion beam and photon-based techniques are or might in the future be working together in synergy. Finally the process in which all European accelerator-based photon facilities have adopted a common strategy by establishing the alliance LEAPS will be explained, as a model which may be useful in some aspects for ion beam facilities.

# ELEMENTAL MAPPING ON THE NM SCALE: SECONDARY ION MASS SPECTROMETRY IN THE HELIUM ION MICROSCOPE

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Helium ion microscopes (HIM) are commonly used high resolution imaging devices. They deliver superior resolution on the (sub) nanometer range as well as a highest depth of field on conductive and insulating samples [1]. Contrast generation in HIM imaging is realized by evaluating the number of secondary electrons (SE). Identification of secondary ions produced by the impacting He<sup>+</sup> or Ne<sup>+</sup> projectiles was rarely taken into consideration so far, although it provides an additional mechanism for contrast generation.

Here, we report on the implementation of Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) in a HIM. We demonstrate that pulsing the primary ion beam represents an efficient way to integrate SIMS into a HIM without disturbing its excellent imaging capabilities [3]. This technique can be easily retrofitted to existing HIMs [2,3].

Our setup uses a straight secondary ion extraction optics that has been designed and optimized for highest transmission. The high efficiency is the most crucial parameter to collect enough signal from nanoparticles prior to their complete removal by ion sputtering.

ToF-SIMS represents a versatile add-on that can reveal details about the sample and is therefore beneficial for many applications. Our setup allows to obtain SIMS data (elemental composition) from a certain region of interest or can be used in imaging mode to obtain elemental line profiles and maps of the surface. The beam resolution has been evaluated to 8 nm using the knife edge method, an un-pulsed beam and a 75%/25% criterion. The system has been qualified on test and real life samples from different fields like lithium ion battery research, aerosol analysis, thin film coatings. Selected results will be presented.

Latest attempts on upgrading our system with a delayed secondary ion extraction will be presented as an outlook.

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# NOVEL DEVELOPMENTS OF ION-BEAM TOOLS FOR NON-DESTRUCTIVE COMPOSITION ANALYSIS

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Ion-beam analytical (IBA) techniques have strongly contributed to the rapid advancement of our high-tech society in the last half-century. Being often more quantitative and straightforward to interpret than using information based on photon or electron beams results on chemical composition and sample structure from IBA nowadays are standard tools in materials research.

As many other techniques, the ongoing miniaturization, as exemplified in the development of electronic devices, but omnipresent last-but not least also due to a society striving for resource and cost-effective materials manufacturing, IBA has been challenged in terms of both sensitivity and depth resolution. As relevant length scales in thin film technology reach length scales of around a nanometer, novel approaches are requested. Such thin samples require methods with higher depth resolution, but also often require the capability of *in-situ* analysis of the material system to be characterized.

In this contribution we present a series of new instrument developments performed at the Tandem Laboratory at the university of Uppsala. Specifically, for MeV primary ion energies, we will present a novel-approach characterization of thin-film batteries using coincidence transmission ERD as well as a new-set-up for *in-situ* analysis of materials growth and modification. At keV ion energies we will present a series of modification made to our Time-Of-Flight Medium Energy Ion Scattering System as well as a new set-up for low-energy IBA. The capabilities of our instrumentation will be exemplified by a series of recently conducted materials science studies.

### MEV SIMS APPLICATIONS IN MATERIAL SCIENCE

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Time-of-flight Secondary Ion Mass Spectrometry using MeV heavy ions (MeV TOF-SIMS) is a newly developed mass spectrometry technique used for molecular identification and imaging of various organic materials. In MeV TOF-SIMS, MeV ions interact with the surface layers of the sample mostly through the electron scattering, causing desorption of intact molecular ions and making the determination of sample molecular composition much easier. Several orders of magnitude larger yields, as well as less fragmentation, are expected for larger molecular masses when MeV ions are used for the excitation, which is especially important for imaging of organic samples with a micrometer lateral resolution. Due to the technique potential to analyse high-masses with high-sensitivity, and fact that is surface sensitive, MeV TOF-SIMS can be employed to analyse organic samples such as fingerprints, paints, inks, tissue, body fluids, etc. Since the MeV TOF-SIMS is done with MeV ions, standardly used for the Ion Beam Analysis (IBA), it is a complimentary technique to the other IBA techniques such as PIXE, EBS, ERDA providing information about molecular composition of the analysed samples.

Recent results on the application of MeV TOF-SIMS in the analysis of biological, forensic and cultural heritage samples will be presented. MeV TOF-SIMS was successfully applied for the 2D imaging of the various lipids in the mouse liver and brain tissue and investigation of chemical changes in the body fluids (serum and urine) of the healthy and diabetic mice. Secondly, MeV TOF-SIMS in combination with Particle Induced X-ray Emission (PIXE) was applied to determine deposition order of the different writing tools for forensic document examination. In addition, MeV TOF-SIMS was demonstrated to be successful for the identification of synthetic organic pigments and binders in contemporary art objects.

# EXPERIMENTAL ION BEAM - MATTER INTERACTION PARAMETERS AT 0.1 MEV/U – 1.0 MEV/U ENERGIES FOR HEAVY ION NUCLEAR ANALYTICAL TECHNIQUES

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Validation of theoretical models that describe various ion beam-matter interaction phenomena for accurate ion beam analysis (IBA) depends on the availability of experimental data to test these models. The use of heavy ion beams (Z>2) in IBA techniques has been shown to enhance the sensitivity of these techniques when compared to using light ions (H, He). One of the factors inhibiting widespread implementation of heavy ion IBA techniques, apart from infrastructural limitations, is the scarcity of experimental data of basic parameters such as stopping force and X-ray production cross sections. The work presented here describes measurements carried out to determine heavy ion induced X-ray production cross sections and stopping force in thin solid films for applications in Heavy Ion ERDA and Heavy Ion PIXE. Stopping force data is compared to predictions of the *ab initio* CasP code and the semi-empirical SRIM, and X-ray production cross section measurement results are compared to the plane wave Born approximation (PWBA) and ECPSSR-UA calculations.

#### ENHANCED THIN FILM ANALYSIS VIA HRBS USING THE NEC CARBS SYSTEM

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Advancements in thin film deposition techniques can now produce films of only a few monolayers in thicknesses, with multiple applications emerging. This maturing manufacturing technique is driving the need for diagnostics tools able to accurately measure depth profiles. To meet this need, the Compact Automated Rutherford Back-Scattering (CARBS) system is under development at National Electrostatics Corp. (NEC) to provide a focused system with a 4 x 4-meter footprint for surface layer analysis using Highresolution RBS (HRBS). CARBS has a Pelletron, beamline, high-resolution end station, and software purpose built for this technique. Optimization of the HRBS magnet has improved energy resolution to 1keV and consequently provides depth resolution of 5 Angstroms. Improved solid angle acceptance has reduced the time needed to reach statistical confidence. The Pelletron developed for CARBS is a single-ended 500 kV accelerator which has demonstrated an ion beam with an energy ripple of 30 eV (RMS) at energy. The CARBS system also provides a 500 keV beam high level of autonomous operation and demonstrated beam on target efficiencies of ~100% with minimal intervention. Finally, modernization of the data analysis software suite simplifies the generation of an elemental depth profile.

### DEMANDS AND CHALLENGES FOR STOPPING POWER TABULATIONS

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"Ion Beam Analysis" (IBA) subsumes experimental techniques using different types of ions in a wide energy range. What all IBA techniques have in common is the need of stopping powers to convert ion energy losses to depth scales. Thus, reliable Stopping Powers are needed for a wide range of experimental parameters. We judge that, in order not to limit the accuracy of IBA results by the quality of the stopping powers used, an accuracy of  $\sim 2$ - 3% is required. In this contribution, we will discuss to which extend these demands have been met and what are the reasons for splendor and misery of the striving for more accurate predictions of stopping powers.

There are different approaches to provide stopping power data, either as a tabulation based on experimental results and on theoretical scaling [1] or by collecting all available data [2]. For ions with energies in the range of several MeV/A (with mass number A) the required accuracy is easily met for elemental targets, and for compounds Bragg's additivity rule yields reliable stopping powers. At low and intermediate energies up to the stopping maximum, the spread of experimental data is embarrassingly large despite the fact that transmission and backscattering experiments have been shown to yield concordant stopping power data, when performed properly.

Another challenge for any data-based stopping power tabulation is that by far the majority of experimental studies concentrated on noble metals plus a handful of other elements like C, Al, Si, Ni. Maybe it was wise not to touch chemically active materials to avoid an even larger spread of data. On the other hand this means, however, that tabulations have to interpolate for transition and rare earth metals, thereby ignoring band structure effects [3] – despite the fact that at low and intermediate energies stopping powers of compounds have been shown to exhibit strong chemical effects, which may lead to the breakdown of Bragg's rule [4][5] and even vanishing electronic stopping in extreme cases [6]. Charge state effects ions heavier than protons will hamper a simple scaling of proton stopping to heavier ions. There is certainly no simple recipe how to overcome these problems, but one should be aware of these problems.

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# SPATIAL AND TIME CHARACTERIZATION OF TANDETRON MICRO-BEAMS WITH TIMEPIX AND TIMEPIX3 PIXEL DETECTORS

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Delivery of particle beams can be verified and examined on-line in high precision with the pixel imaging detectors Timepix and Timepix3. These hybrid semiconductor pixel detectors provide single particle counting sensitivity, high granularity and per-pixel spectrometric and time response. Contamination and component admixtures in the delivered beams can be also resolved and determined. The spectral, spatial and time distribution of proton and light ion micro-beams are selectively measured and characterized in wide range of beam species, energy and particle fluxes. Results are presented for various geometries and beam delivery configurations. A novel detector chip array architecture consisting of four Timepix3 chips and single common sensor with a 2 mm hole opening in the centre allows to probe micro-beams in close proximity in radially symmetric geometry. This architecture allows to detect scattered and secondary reaction particles close to the delivered primary beam and to the target. Application include novel measuring methods for XRD (X-Ray Diffraction), SAXS (Small Angle X-ray Scattering), SEM (Scanning Electron Microscopy) and other Nuclear Analytical techniques (RBS, PIXE, ERDA). All these techniques use secondary effects occurring when a pencil beam interacts with a sample surfaces causing that particles to change direction of trajectory and deviate from the primary beam. These particles need to be registered by imaging detector with maximal information about their position, energy and time of arrival. Research has been realized at the CANAM (Center of Accelerators and Nuclear Analytical Methods) infrastructure LM 2015056.

### NOVEL APPLICATIONS OF 3D ION TRANSMISSION EXPERIMENTS AT KEV ENERGIES

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Medium energy ions have proven to be a powerful tool in both fundamental research and industrial applications. Although keV ions are widely used for ion beam analysis, ion irradiation, implantation, and sputtering, the complex physics behind the interaction of ion and target material at these energies leaves still room for further improvement of our understanding of the underlying processes which may, in turn, provide new or improve existing analytical techniques.

Recently, a new imaging technique, the helium ion microscope (HIM) based on keV ion beams has been introduced. In transmission geometry



**Fig.1:** 3D surface map of the mean energy loss for 50 keV He transmitted through a 200 nm single-crystalline silicon foil.

contrast in such imaging can be obtained not only from intensity caused by scattering, but as Lohmann *et al.* showed, at energies typically employed in the HIM, the final energy of transmitted ions depends strongly on the ion trajectories [1]. Contrast can therefore be visualized as a function of projectile energy loss during their passage through the sample.

We present a number of applications using 3D ion transmission experiments, i.e. simultaneous measurements of angular distributions and flight time of particles exiting thin self-supporting foil targets. Analysis of primary ions exiting the thin self-supporting single-crystalline silicon foils, allows for mapping of intensity and different energy loss moments [2]. In addition, by employing heavier projectiles, recoils can be detected with high sensitivity for light elements, which gives the possibility to study surface and bulk composition.

Our results can be utilized to interprete implantation profiles in single-crystalline materials by visualization of the reduced energy loss in specific crystal directions. They also provide a basis for interpretation of data to be obtained in prospective transmission studies when for example using a helium ion microscope.

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### TRAJECTORY-DEPENDENT ELECTRONIC EXCITATIONS OF KEV IONS

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We present experiments directly demonstrating the significance of charge-exchange events for the energy deposition of ions with velocities below the Bohr velocity. The observed effects lead to a drastic trajectory-dependence of the specific energy loss.

Experiments were performed with the time-of-flight medium energy ion scattering set-up at Uppsala University [1]. We employed pulsed beams of singly charged ions with masses ranging from 1 ( $H^+$ ) to 40 u ( $Ar^+$ ) and energies between 20 and 350 keV. Ions were transmitted through self-supporting Si(100) nanomembranes and detected behind the sample. We assessed the energy and angular distributions of deflected particles for different alignments of the initial beam direction with the crystal axes and planes. A set-up for measuring the exit charge state was constructed to support the analysis [2].

For all ions we observe lower electronic stopping for channelled trajectories as compared to random ones as shown in Fig. 1 [3]. For protons, this difference is explained by increasing contributions of core-electron excitations more likely to happen at small impact parameters accessible only in random geometry. For heavier ions, core-electron excitations at employed ion velocities are inefficient and we, therefore, explain these results by reionisation events occurring in close collisions of ions with target atoms [4]. These events in turn result in trajectory-dependent mean charge states, which heavily affects the energy loss, and could be confirmed by first qualitative measurements of the trajectory dependence of exit charge states. The simplicity of our experimental geometry leads to results that can serve as excellent benchmark systems for calculations using time-dependent density functional theory.





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# PROTON RADIOGRAPHY USING THE TIMEPIX3 PIXEL DETECTOR APPLIED AT TANDETRON

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The goal of the work is to investigate the ability of using low-energy proton beams for radiography of thin samples. The main advantage of proton radiography is a low radiation dose delivered to the samples. Although the biological proton radiation effects are high, a radiography can be created using only one proton per pixel. In this study, the application of the Timepix3 detector for particle radiography of thin samples using monoenergetic low-energy proton beams was evaluated. The Timepix3 detector equipped with 500  $\mu$ m Si sensor is readout with an AdvaPix readout electronics interface. Data were collected at the Tandetron light-ion accelerator of the NPI-CAS equipped with 3 MeV proton beams. Measurements were performed with various samples such as thin mylar and aluminum foils.

A proton micro beam was used to scan the samples placed in front of the detector's sensor. In addition, calibration of the detector was performed in the air. The principle of proton radiography is based on measuring energy decrease of protons, cluster size of impacted protons traces, maximum pixel value of clusters and other parameters. Each mono-energetic proton should create a similar cluster into an active layer of the detector but after passage through sample it scatters and its cluster becomes smaller. The final image is created based on these parameters that are localized.

Proton radiographies were reconstructed based on individual parameters. The radiographic image of staired aluminum foils with thicknesses of 0-air, 5, 10, 25 and 30 mm was reconstructed using Cluster area parameter, see Fig. 1. Color scale represents number of clusters pixels. Further plans involve radiographies of organic samples.



**Figure 1** (Left) Proton Radiography of Aluminum foil staired sample. Image generated based on size of proton clusters. (Right) The photo of aluminum stairs sample with thicknesses of 0, 5, 10, 25 and 30 mm.

### BORON QUANTIFICATION AND DEPTH PROFILING BY ION BEAM ANALYSIS FOR CHARACTERIZATION OF NOVEL BORIDE MATERIALS

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Boron, besides being the most common p-type dopant in the silicon technology, is also used in transition metal borides (such as CrB<sub>2</sub>, WB<sub>2</sub>, and TaB<sub>2</sub>), a new class of promising materials for applications such as superconductivity and corrosion-resistance coating [1]. Accurate characterization of elemental composition depth profiles with high resolution is essential for these materials, since thermal stability and mechanical properties can be changed substantially by subtle modifications in composition [2]. Ion beam-based analytical techniques based on MeV primary particles are frequently used in the investigation of such materials due to their capability of non-destructive, depth-resolved and highly accurate elemental composition analysis. However, analysis of light elements present in a heavy matrix can be challenging, which frequently requires a combination of different techniques. Here, in order to assess the different methodologies to detect, quantify, and depth profile materials containing boron, several samples were investigated by different ion beam analytical techniques: nuclear reaction analysis using the reaction  $^{11}$ B(p, $\alpha$ )<sup>8</sup>Be at 163 keV and 2.6 MeV, elastic backscattering spectrometry using the <sup>11</sup>B(p,p)<sup>11</sup>B reaction at 2.6 MeV, and coincidence time-of-flight elastic recoil detection analysis using heavy primary ions such as  $^{127}I^{8+}$  and  $^{79}Br^{8+}$  at 36 MeV [3].

Different samples were prepared by ion implantation of <sup>11</sup>B<sup>+</sup> with a wide range of energies and doses in high and low-Z substrates. Also, boron containing substrates such as boron nitride were used. Au thin films were deposited by sputtering to create a multi-layered structure, allowing for an evaluation of interface roughness. The depth resolution, probing depth, film homogeneity, and sensitivity achieved by the individual techniques will be presented and discussed, as well as the pros and cons of using these techniques for each specific group of samples. Finally, the use of these techniques to investigate composition, distribution and migration of boron in thin films of transition metal borides as used in e.g. hard coatings, will also be discussed.

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# PULSED LASER DEPOSITION OF THIN FILMS ON POLYDIMETHYLSILOXANE FOR BIOMEDICAL APPLICATION

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Keywords: Pulsed laser deposition, polydimethylsiloxane, graphene oxide, bioactivity

The realization of material satisfying technical and biological functions is still a challenge although remarkable progress has been made on biomaterial research. Ion implantation and deposition is an attractive method for the modification of surface properties of material because its capability of modifying objects with irregular shapes and its good control of coating components and thickness. Presently, titanium and carbon-based material were deposited using laser ablation on polydimethylsiloxane substrate to modify its surface features and to allow its clinical applicability. A nanosecond Nd:YAG laser was employed to ablate solid targets of titanium and carbon-based material in vacuum generating hot plasma and depositing a thin films of laser irradiated material on substrates of polydimethylsiloxane located inside the vacuum chamber in appropriate The thickness of the deposited material was evaluated by Rutherford positions. backscattering spectrometry. Depending on the desired applicability, the grain size, porosity, uniformity, wetting, hardness and adhesion of the obtained films were investigated using different surface techniques such as IR spectroscopy, Scanning electron microscopy as well as wettability and hardness.

### THE SYNTHESIS OF Au-NPs BY ENERGETIC ION IMPLANTATION INTO THE CRYSTALLINE GaN AND CHARACTERISATION OF THEIR OPTICAL PROPERTIES

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The modification of wide band semiconductors with noble metal nanoparticles (Au, Ag, Pt) is an attractive way for a manipulation with the optical properties of material with perspective application in optoelectronics, photocatalytic processes or as a material for Surface Enhanced Raman spectroscopy (SERS). The implantation of energetic ions offers an interesting way for metal nanoparticle preparation without use of additional chemicals that offers precise control of nanoparticle composition and depth distribution. The aim of this study is a synthesis of the gold nanoparticles (Au-NPs) in GaN by implantation of Au ions with energy 1.85 MeV into the crystalline *c-plane* GaN with implantation fluences in the range from  $1.5 \times 10^{16}$  to  $7 \times 10^{16}$  cm<sup>-2</sup>. The projected range  $R_p$  of Au ions with this energy is 230 nm with standard deviation  $\Delta R_{\rho} \sim 63$  nm. Implanted crystals are annealed at 800 °C in ammonia atmosphere for 20 min to support Au-NPs nucleation and growth. The structural characterisation, carried out by Rutherford backscattering spectroscopy in channelling mode (RBS-C), showed formation of two separated regions - surface region and buried layer with implanted Au ions. The lower implantation fluences induce damage mainly in buried layer, however, the increase of the Au-ion implantation fluence leads to the defect diffusion towards the surface region and leads to the increase of surface damage. The Au depth distribution characterised by RBS-C exhibited typical Gaussian depth profile for the lowest implantation fluence. With increasing fluence, the Au dopant has been shifted to the surface and create multimodal depth profile. XRD and TEM analyses have been used to follow GaN structure modification and Au NPs coalescence in the as-annealed samples to follow the formation of Au-NPs at various Au-ion implantation fluences. Luminescence properties and optical absorbance of the prepared GaN:Au nanostructured layers were characterised and discussed in connection to the structural changes.

# HYDROGEN STORAGE ON THE NANOSCALE: VISUALIZING INTERSTITIAL HYDROGEN IN NANOSTRUCTURED METALS WITH MEV ION BEAMS

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Metal hydrides represent a promising class of materials for reversible solid-state storage of hydrogen for energy applications [1]. Hydrogen atoms absorbed from molecular gas (H<sub>2</sub>) occupy octahedral or tetrahedral interstitial sites, which results in volume expansion in the crystalline early transition metals forming compounds existing as multiphase systems ( $MH_n$ ). To meet requirements for applications, such as fast delivery at ambient temperatures and high absorption rates, nanostructures can be used to improve reaction kinetics and reduce the enthalpy of formation [2].

Real space location of hydrogen sites in single crystalline two-dimensional superstructures is achieved by <sup>15</sup>N(6.6MeV)-ion channelling in combination with <sup>1</sup>H(<sup>15</sup>N,  $\alpha\gamma$ )<sup>12</sup>C nuclear reaction analysis. The detection of characteristic  $\gamma$  emission discloses absolute hydrogen concentration and relative lattice location when measurements are performed angular-resolved. Detection of elastically backscattered ions allows not only identification of heavy matrix elements and alignment towards crystallographic axes, but also exploration of hydrogen induced lattice expansion [3]. Monte-Carlo simulations of ion trajectories verify the exact location and reveal for the first time thermal vibrational amplitudes of subsurface hydrogen. In this contribution the effect of elastic boundaries, finite size and proximity on the site occupancy and phase formation of metal hydrides, e.g. vanadium, is studied on the nanoscopic to atomic scale.

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# IN-SITU CHARACTERIZATION OF ULTRA-THIN NICKEL-SILICIDE FILMS USING LOW-ENERGY ION SCATTERING

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Metal-silicides play an important role as contact materials in thin film electronics, with nickel-silicides showing especially advantageous properties, including low resistivity, low silicon consumption and low formation temperature [1]. Here, growth and annealing of ultra-thin (less than 10 nm) nickel-silicide films were characterized *in-situ* utilizing Time-of-Flight Low Energy Ion Scattering (ToF-LEIS). Using ions with primary energies of 1-10 keV the employed ToF-LEIS set-up, capable of detecting both ions and neutrals, provides high surface sensitivity to study composition and structure of the outermost atomic layers [2].

Ni films were deposited on clean Si(100) substrates at pressures lower than  $5.2 \times 10^{-10}$  mbar by e<sup>-</sup>-beam evaporation, ensuring high film purity. Annealing was performed at a temperature of 290°C, known for the formation of an intermediate nickel-silicide phase [3]. ToF-LEIS experiments were complemented by *in-situ* Auger Electron Spectroscopy (AES) and Low Energy Electron Diffraction (LEED) measurements and *ex-situ* Rutherford backscattering spectrometry (RBS), Time-of-Flight Medium Energy Ion Scattering (ToF-MEIS), Transmission Electron Microscopy (TEM) and Scanning Transmission Electron Microscopy (STEM). These methods provide additional information on total areal densities, crystallographic structures and phases present in the film.

Our results comprise a detailed characterization of Ni growth on Si(100) and the phase transition observed at 290°C. ToF-LEIS and ToF-MEIS confirm the formation of a nickel-silicide layer which is shown to consist of two phases by TEM. Structural analysis using nanobeam electron diffraction indicates the dominant phase to be Ni<sub>2</sub>Si and the sub dominant phase, epitaxial to the Si substrate, to be NiSi<sub>2</sub>. The absence of an intermediate phase between the two observed phases indicates a direct phase transition from Ni<sub>2</sub>Si to NiSi<sub>2</sub>, which, to the best of our knowledge, has not been earlier reported. Furthermore, the dominant Ni<sub>2</sub>Si phase is shown to be oriented axiotaxial regarding the Si substrate.

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# CHARACTERIZATION OF TITANIUM ALUMINUM NITRIDE FILMS USING LOW-ENERGY ION SCATTERING

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The thermal stability of (Ti,Al)N, a metastable compound widely used as hard coating on cutting and forming tools, is limited by spinodal decomposition into TiN and AlN at temperatures above 860°C [1]. We investigate the behavior of (Ti,Al)N films at elevated temperatures in ultra-high vacuum and in an oxygen atmosphere of  $1.0 \times 10^{-6}$  mbar *in-situ* using Time-of-Flight Low-Energy Ion Scattering (ToF-LEIS). Using ions with primary energies of 1-10 keV the employed ToF-LEIS set-up, capable of detecting both ions and neutrals, provides high surface sensitivity to study composition and structure of the outermost atomic layers [2]. Supplementary *in-situ* Auger Electron Spectroscopy (AES) as well as *ex-situ* Rutherford Backscattering Spectrometry (RBS), Time-of-Flight Elastic Recoil Detection Analysis (ToF-ERDA), and Transmission Electron Microscopy (TEM) measurements complement the ToF-LEIS results with additional information on composition, total areal densities, and structure of the films. (Ti,Al)N films, with a thickness of 3 µm, were grown on MgO (001) substrates by co-sputtering Ti and Al in reactive Ar/N<sub>2</sub> atmosphere using a hybrid process [3].

Our results comprise a sub-nm depth characterization of the effects observed during annealing of (Ti,Al)N coating surfaces. ToF-LEIS and AES measurements confirm the formation of an Al-enriched surface layer starting at annealing temperatures of 650°C in high-vacuum. At temperatures below 650°C, no change in the surface composition and structure is observed. Identical measurements after annealing a (Ti,Al)N film for 90 min at 850°C in  $1.0 \times 10^{-6}$  mbar of oxygen show similar behavior and no further subsurface oxidation of the sample. The ability of our method to detect surface and subsurface oxidation is shown by comparative measurements performed on TiN films. Together these results show the high stability of (Ti,Al)N towards both phase decomposition as well as oxidation and the analytical power of the here employed approach for characterization of phase transitions and reactions on the sub-nm depth scale of thin-film surfaces.

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# NUCLEAR ANALYTICAL METHODS FOR ENVIRONMENTAL AND CULTURAL HERITAGE STUDIES

### SMALL ACCELERATORS FOR CULTURAL HERITAGE - ANALYTICAL CAPABILITIES AND HISTORICAL OVERVIEW

### Fedi M.

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The importance of scientific diagnostics in supporting study and restoration of Cultural Heritage has by now become widely recognised. Small particle accelerators have played an important role both in material analysis and in dating issues since the 1970s. During this decade, in fact, first experiments in Ion Beam Analysis (IBA) and Accelerator Mass Spectrometry (AMS) demonstrated that, in spite of appearing perhaps quite old for the emerging needs of high energy physics, such machines were useful for new applications in applied research. Electrostatic tandem accelerators and AMS allowed us to measure the radiocarbon concentration, thus dating artefacts and findings, using small samples, even during short counting times. The introduction of external beam set-ups for PIXE (Particle Induced X-ray Emission) measurements opened the possibility to investigate the elemental composition of almost all the artworks without any need of sampling or preparation, even though with some limitations (consider, for example, that only elements with Z≥11 can be detected in those conditions, and that the studied artwork has to be brought into the laboratory). In the most recent times, R&D interests have been directed towards optimizing the information we can get from the measurements, for instance simplifying the set-ups or minimizing the possible invasiviness - if present - of the technique. At the same time, accelerator-based composition analyses have started to face the competition of easy and portable new systems, as, for example, the X-ray fluorescence (XRF) ones.

After an historical overview and a brief introduction of the main basic principles of IBA and AMS, the presentation will discuss some of the highlights of the most recent research in the field of Cultural Heritage applications, focusing especially on microgram samples for radiocarbon dating and innovative movable accelerators for PIXE.

# NUCLEAR PHYSICS FOR THE ENVIRONMENT AND CULTURAL HERITAGE: THE LABEC EXPERIENCE

Chiari M.

### Istituto Nazionale di Fisica Nucleare (INFN), Division of Florence, Sesto Fiorentino, Italy

The LABEC, the ion beam laboratory of nuclear techniques for environment and cultural heritage of the National Institute of Nuclear Physics (INFN), located in the Scientific and Technological Campus of the University of Florence, Italy, started its operational activities in 2004, after INFN decided in 2001 to provide our applied nuclear physics group with a large laboratory dedicated to applications of accelerator-related analytical techniques, based on a 3 MV Tandetron accelerator. This new accelerator greatly improved the performance of existing Ion Beam Analysis (IBA) applications, for which an old single-ended Van de Graaff accelerator was used since the 1980s. At LABEC we have a great expertise in IBA measurements with external beams, performed while maintaining the targets/objects to be analysed in atmosphere at ambient pressure, avoiding the need of picking up samples, reducing ion beam induced damages and greatly enhancing the measurement throughput.

This presentation describes the IBA facilities presently available in the LABEC laboratory, their technical features and some success stories of recent applications, mainly focused on studies of cultural heritage and atmospheric aerosol composition, but including also applications to biology, geology, material science and forensics.

#### DEVELOPMENT AND APPLICATION OF THE FIRST AMS FACILITY IN AFRICA

### Woodborne S.

### iThemba LABS, Johannesburg, South Africa

In 2013 iThemba LABS upgraded the Tandem EN accelerator to include an accelerator mass spectrometry (AMS) capability. The facility has subsequently commissioned methods for <sup>14</sup>C, <sup>10</sup>Be and <sup>26</sup>Al measurements. It has also commissioned preparation laboratories for the pretreatment of samples and production of cathodes. Performance of the AMS system is not simple, and several compromises in the design were overcome, but the overall performance is determined against international inter-comparison samples. With the merit of the system demonstrated, the facility was immediately wellsubscribed by local archaeologists, and the demand for <sup>14</sup>C analyses is the dominant operation. The facility hosts post-graduate students and local researchers as part of their mandate for training and research, and a unique, no-cost financial model has been implemented. The outcome has been the stimulation of a new way of addressing chronology in heritage studies in which programs that involve substantially larger numbers of dates than were previously affordable are now conducted. In addition, iThemba LABS scientists have been leading in-house research focused on climate reconstruction for the last 1000 years through tree ring proxies. A record of climate change spanning the entire African subcontinent from Namibia to Madagascar has been completed, and this demonstrates systematic shifts that are relevant in understanding future climate change in the region. The research team has also focused on the global  $\Delta^{14}$ C record for the last 50 000 years. Using a speleothem for which uranium series dates are available, it has been possible to reconstruct a well-resolved record for the southern hemisphere. Expansion plans for the facility aim to open the access to the broader African research community, but for this it will be necessary to overcome capacity issues and further investment is required.

# IBA AND AMS TECHNIQUES FOR CULTURAL HERITAGE STUDIES: EVIDENCING ANCIENT AND RECENT FORGERIES

Beck L.

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Forgeries have existed in many fields. Money, goods, artworks have been imitated since centuries to deceive and make profit. In this talk, I will present two cases where nuclear analytical methods have been used to study past and recent forgeries.

The first example will deal with ancient counterfeit coins. Long considered uninteresting by numismatists, counterfeit coins are nevertheless archeological evidence of ancient metallurgy technologies as well as counterfeiting practices. Many techniques were used to fool customers with false coins representing circulating coins. One of them was the use of cheaper metals than those of the official production, while respecting the visual aspect - color, engraving, size - as well as the weight which was directly connected to the value of the coinage. Silver coinage was imitated in the past by replacing silver, partly or entirely, by copper or iron and the visual silvery surface was produced by various manufacturing processes. The technology of the unofficial production of silvered coins was investigated by using fast neutron activation analysis and PIXE for the non-destructive and quantitative determination of the metal content and by Rutherford backscattering spectrometry (RBS) for the surface analysis. The silvering processes used for silver-plated coin forgeries of the 3rd and the 16th centuries will be described.

The second example will deal with recent art forgeries. Investigations to authenticate paintings rely on an advanced knowledge of art history and a collection of scientific techniques. However, accelerator mass spectrometry (AMS) radiocarbon (14C) dating is the only technique that gives access to an absolute time scale. AMS radiocarbon dating was applied to paintings of the 20th century to identify potential forgeries in the context of an ongoing police investigation. 14C measurements show that the plants used to make the canvas were harvested after 1955, that is to say at least 10 years after the death in the 40s of the supposed artists. These results demonstrate that the paintings are recent forgeries.

Finally, the new attribution of the Flora bust of the Bode-Museum recently <sup>14</sup>C AMS dated will be presented.

### RECENT ACHIEVEMENTS IN NAA, PAA, IBA, AND AMS APPLICATION FOR CULTURAL HERITAGE INVESTIGATIONS

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Nuclear analytical techniques comprising neutron and photon activation analysis (NAA and PAA, respectively), ion beam analysis (IBA) have been used within the Centre of Nuclear Analytical and Accelerator Methods (CANAM), among other applications, in cultural heritage studies. A new laboratory of Accelerator Mass Spectrometry (AMS) will further enhance our capabilities in this field.

In one earlier study we determined 35 major, minor and trace elements in sandstone samples taken from building blocks of 19 Angkor temples in Cambodia and from 2 quarries using instrumental NAA (INAA). However, using cluster analysis, we found no straightforward correlation between the chemical/petrological properties of the sandstones and a presumed period of individual temples construction. The possible reasons for the poor correlation will be discussed. In another study, we used a combination of instrumental PAA (IPAA) and radiocarbon dating to determine the matrix composition of an ancient bracelet recently found in ruins of a fortification in the vicinity of Karlovy Vary (Carlsbad) made of an organic material and its age. The nitrogen determination by IPAA revealed that the bracelet was made of tree bark (not leather), while radiocarbon dating assessed its age to 1408–1266 years BC. The radiocarbon analysis was also used to date charcoal paintings in Kateřinská cave of the Moravian Karst. One graffiti was dated to Neolith (around 5000 BC) and three others to Eneolith (around 4300 BC), making them the oldest known drawings in the Czech Republic. A new gentle method of wipe-sampling was developed to preserve contours of the original drawing. In the most interesting study we applied INAA, radiochemical NAA (RNAA) and IBA methods to analyse remains of the famous Danish astronomer Tycho Brahe, who died in Prague in 1601 aged 54 years. By analysis of his 5-mm sectioned hair samples and of his bones we found that Tycho Brahe was not poisoned by Hg, as was previously speculated, because the Hg concentration in last weeks of his life were decreasing. In the hair samples, we also found similar time trends for the elements Fe, As, Ag and Au indicating possible exposure from Brahe's alchemical activities. Concerning our future activities in AMS, which will start soon, we expect an increase of archaeological applications using namely radiocarbon dating.

### DEVELOPMENT OF NOVEL INSTRUMENTATION FOR MATRIX INDEPENDENT ULTRA-TRACE DETECTION AND QUANTITATION OF RADIONUCLIDES USING COLINEAR RESONANCE IONISATION SPECTROSCOPY

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The easy incorporation of certain radionuclides into the biosphere renders their determination a key issue in decommissioning. The classification and quantitation of decommissioning waste materials can be challenging, especially if the sample is complex. High throughput elemental analysis techniques such as inductively coupled plasma tandem mass spectrometry (ICP-MS/MS) often lack the abundance sensitivity and selectivity required for some radionuclides such as <sup>90</sup>Sr.

Collinear resonance ionisation spectroscopy (CRIS) offers a solution combining high resolution spectroscopy with high sensitivity ion detection. Developed at CERN for probing the energy structure of radioactive elements, this technique employs tuneable lasers overlapped with a radioactive ion beam to resonantly ionise the element of interest. At the ISOLDE facility in CERN, CRIS continues to be used for measuring fundamental properties of radioactive nuclei. The high isotopic selectivity, made possible due to the kinematic shift induced when using accelerated beams, makes this technique suitable for measuring trace radioactive elements in environmental samples.

The development of ICP-MS-CRIS has the potential to reduce current analysis times of  $^{90}$ Sr to <30 minutes compared with the current Cherenkov counter-based analysis times of ~28 days. The universal benefits of CRIS lies in the fact that it can be applied to any element on the periodic table with unrivalled sensitivity and selectivity.

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[2] R. P. de Groote *et Al.*, Dipole and quadrupole moments of <sup>73–78</sup>Cu as a test of the robustness of the Z=28 shell closure near <sup>78</sup>Ni, Phys. Rev. C 96 (2017) 041302(R). https://doi.org/10.1103/PhysRevC.96.041302

#### STUDYING UV AGEING EFFECTS IN MODERN ARTISTS' PAINTS WITH MeV-SIMS

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MeV-SIMS is a highly surface-sensitive technique that can provide information of the chemical composition in the uppermost layers of materials under investigation. By irradiating the sample with an ion beam in the MeV energy range, intact molecules are desorbed from the surface through electronic sputtering which leads to less fragmentation and a more straightforward interpretation of the mass spectra compared to keV-SIMS. For this reason, MeV-SIMS has emerged as a powerful tool with application to many fields, one of which is cultural heritage studies where it has been successfully applied in identification of synthetic organic pigments (SOPs) in paint materials [1] and for molecular imaging of modern paints [2]. In this work artificially aged alkyd and acrylic self-made paints, containing SOPs from different chemical classes (phthalocyanine, guinacridone and diketopyrrolo-pyrrole) were studied using MeV-SIMS with the aim to investigate chemical changes that are taking place in the surface layer due to paint ageing. Two-component mock-up samples were prepared and exposed to accelerated artificial UV ageing conditions for two and four months. The obtained results show that the investigated SOPs are stable under accelerated ageing conditions and can be easily identified in aged paints. New chemical species were detected in the aged paints compared to the unaged samples which were identified as the degradation products of the binder portions of the paints, both alkyd and acrylic. Enrichment of the pigment relative to the binder on the sample surface was also observed. Acrylic binder exhibited slightly higher stability to photodegradation compared to the alkyd medium, while the kinetics of the degradation processes were roughly found to be dependent on the pigment present in the paint, with phthalocyanines having the highest photo-stabilizing effect among the studied pigments. This study will significantly facilitate identification of modern paint materials in real artworks that have been exposed to environmental conditions, having undergone chemical transformation in the surface layer.

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# NON-DESTRUCTIVE MASS SPECTROMETRY OF SINGLE HOT PARTICLES FROM THE CHERNOBYL EXCLUSION ZONE BY RESONANT LASER SNMS

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In the Chernobyl accident of 1986, hot particles were dispersed in the area now known as the Chernobyl exclusion zone (CEZ). These particles, ranging from 3-50 µm and composed of spent nuclear fuel elements, have remained in the environment for the past 35 years. Their precise composition and behaviour in the environment is key to understanding their impact on radiation protection. Of particular relevance are the isotopic composition of the actinides U, Pu and Am that indicate the type of reactor used, initial enrichment, and burnup of the fuel.

Due to isobaric interference and organic background, a full analysis of multiple actinides is limited in many mass spectrometric techniques. Additionally, such techniques can destroy the particle in the process of measurement, which precludes further analysis. Element selective ionization through multi-step resonant laser excitation allows for the separation of the element of interest and subsequent measurement through mass spectrometry.

Here we present multi-element analysis (U, Pu, Am) of hot particles from the CEZ using our unique resonant laser SNMS system. Measurements are performed using a commercial TOF-MS (IONTOF 5) with a pulsed Ti:Sa laser system capable of performing two-, and three-step laser excitation on the sputtered secondary neutrals<sup>(1)</sup>. These results show how this system can easily be used to quickly switch between U, Pu and Am. The ultra-trace, non-destructive analysis of these microscopic particles provides information on the range of compositions found in the CEZ, and how they relate to their formation.

(1) M. Franzmann et al., J. Anal. At. Spectrom., 33, 730-737 (2018)

### **RECENT DEVELOPMENTS IN IBA ANALYSIS AT CENTA, BRATISLAVA**

Ješkovský M.<sup>1</sup>, Kaizer J.<sup>1</sup>, Kontuľ I.<sup>1</sup>, Kvasniak J.<sup>1</sup>, Pánik J.<sup>2</sup>, Zeman J.<sup>1</sup>, Staniček J.<sup>1</sup>, Povinec P. P.<sup>1</sup>

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In the CENTA laboratory, there is a tandem accelerator Pelletron (NEC, USA) in operation, which is used among other applications, for development of ion beam analysis (IBA) techniques. Two ion sources are installed for production of ion beams from solid and gas targets. After mass and energy separation, the ion beam is accelerated with up to 3MV terminal and deflected to a vacuum chamber for IBA analysis. At the moment, the vacuum chamber is equipped with PIXE, PIGE and RBS detectors. BEGe detector (Canberra) is used for PIGE analysis, and since it can operate from 3 keV, also for high energy X-ray analysis. For lighter elements, 70 mm<sup>2</sup> FAST SDD<sup>®</sup> (Silicon Drifted Detector; Amptek, USA) with energy resolution of 129 keV for 5.9 keV <sup>55</sup>Fe line is used for PIXE analysis.

Backscattered protons and alphas are detected by Si PIN-diode Hamamatsu S3590 in front of the sample. Recent results from measurements of environmental, astronomical and biomedical samples, together with development of IBA methods at the CENTA laboratory will be presented.

### NATURAL RADIOAVTIVITY AND IMPORTANCE FOR SOIL: A REVIEW ON CRITICAL FINDINGS IN TURKEY

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The term of radiation is defined as energy dissipation or transfer in the form of electromagnetic waves or particles. It is impossible to live isolated from radiation nowadays. Since the creation of the universe, all living creatures are exposed to radiation. About 96% of the total radiation dose is from natural sources exists. There is radioactivity naturally in the soil, air, sunshine and in the foods we consume. Soil is the most common source of radiation because of mineral content. The soil radioactivity is a good indicator of the accumulation of radioactivity in the environment. A group of elements such as Potassium-40, uranium-238, and thorium-232 as well as their decay products are essential natural elements, which lead to a significant portion of the human radiation dose. Radionuclides can be quickly accumulates into the food supply chain in terrestrial strata. Natural radiation and soil radioactivity are becoming a significant source of concern for study through humans at various rates based on the radioactive minerals found globally in each area. On the other hand, many different local studies have been performed in terms of determination of natural radioactivity. In this review study, the basic information about the radiation and natural radiation is explained and then effect of natural radiation in soil is investigated. Moreover, some critical local findings on natural radiation and soil radioactivity levels in Turkey have been evaluated. The obtained results were discussed and compared with those invevstigated similar studies and with internationally recommended radioactivity levels.

Keywords: Radiation, sources of natural radiation, soil.

# RADIATION EXPOSURE OF MICROORGANISMS LIVING IN RADIOACTIVE MINERAL SPRINGS

Kolovi S.<sup>1a</sup> for the TIRAMISU collaboration, Fois G.R.<sup>1</sup>, Lanouar S.<sup>1</sup>, Chardon P. <sup>1a</sup>, Miallier D.<sup>1</sup>, Rivrais G.<sup>1a</sup>, Alain E.<sup>2a</sup>, Baker L-A.<sup>2a-3a</sup>, Bailly C.<sup>4a</sup>, Beauger A.<sup>2a</sup>, Biron DG.<sup>3a</sup>, He Y. <sup>4a</sup>, Holub G.<sup>5a</sup>, Le Jeune A-H.<sup>3a</sup>, Mallet C.<sup>3a</sup>, Michel H.<sup>6a</sup>, Montavon G.<sup>4a</sup>, Schoefs B.<sup>7a</sup>, Sergeant C.<sup>5a</sup>, Maigne L.<sup>1a</sup>, Breton V. <sup>1a</sup>

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<sup>a</sup>Members of Long-Term Socio-Ecological Research (LTSER) «Zone Atelier Territoires Uranifères (ZATU)»

The TIRAMISU collaboration gathers expertise from biologists, physicists and radiochemists within the Zone-Atelier Territoires Uranifères in France to analyze the response of microorganisms living in naturally radioactive mineral springs [1]. Mineral springs are small waterbodies that are extremely stable over geological time scales and display different physicochemical and radiological parameters compared to their surroundings.

Water and sediment samples collected in 27 mineral springs of the volcanic Auvergne region (Massif Central, France) have been studied for their microbial biodiversity and analyzed using  $\alpha$  (to measure, for instance, radium and polonium activities) and  $\gamma$  spectroscopy (to measure radon activity), together with ICP-MS and ICP-MS-HR techniques to identify trace elements. Among the microorganisms present, microalgae (diatoms), widely used as environmental indicators of water quality, have shown to display an exceptional abundance of teratogenic forms in the most radioactive springs studied (radon activity up to 3700 Bq/L) [2].

The radiological risk to freshwater biota was estimated using the ERICA tool [3]. Most of the sampled mineral springs were highly above the risk threshold of 10  $\mu$ Gy/h due to the large concentrations of radium in the sediments (up to 50 Bq/g).

The complete radiological data on water and sediments are used as inputs to the Monte Carlo simulations [4] at micro- (GATE) and nano-(Geant4-DNA) scale in order to assess the direct and indirect damages on the diatom DNA by simulating not only the direct energy deposition but also the water radiolysis and the respective Single and Double Strand Breaks (SSB, DSB).

In an effort to further disentangle the radiation stress from other environmental stresses (for instance heavy metals) that could induce deformations, diatoms collected from the mineral springs are isolated, grown in vitro and exposed to doses up to 50 Gy using an X-Ray irradiator.

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# MICRO PROTON INDUCED X-RAY EMISSION SPECTROSCOPY APPLICATION IN ENVIRONMENTAL STUDIES

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Micro proton-induced x-ray emission (micro-PIXE) spectroscopy is a highly sensitive ion beam analysis (IBA) technique based on the inelastic collision of charged particles with electrons of target atoms resulting in the emission of characteristic x-rays. It offers information on the quantitative distribution of elements within a matrix with a lateral resolution of approximately 1µm and the capability of quantifying trace elements down to ppm level from Aluminum (Al) to Uranium (U). This technique can be used for the detection of trace elements such as heavy metals originating from geogenic, anthropogenic sources, or uncontrolled waste disposal that may contaminate soil, air, and water indirectly contaminating agricultural crops and other food sources including marine reserves. These heavy metals such as Cadmium (Cd) and Lead (Pb) do not only have a devastating impact on the environment but may also lead to the development of health problems. Possible differences in the mineral composition of hyper-accumulating plants and insect species feeding on hyper-accumulating plants from various locations closer to industrial and mining communities were investigated and the results were published in many reports. These plants can be used for phytoextraction purposes on contaminated soils. Here, we present key features of the micro-PIXE analytical technique at iThemba LABS and some of the results from these investigations.

# DEVELOPMENT OF A PHOTOIONISATION MASS SPECTROMETER FOR MEASUREMENT OF $^{85}\mathrm{Kr}$

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<sup>85</sup>Kr is a volatile radioactive fission product that is released into the atmosphere during civil nuclear operations and plutonium breeding, making it a useful isotope for monitoring reprocessing activities and detecting unreported plutonium production. Additionally, <sup>85</sup>Kr measurement can aid leak detection in the assessment of nuclear waste. A photoionisation mass spectrometer is being developed for rapid measurement of atmospheric <sup>85</sup>Kr, reducing costs and improving on the sensitivity of existing radiometric techniques for trace isotope detection. The proposed device makes use of an ECR ion source for the production of positive ions from atmospheric samples, resonant ionization for selecting <sup>85</sup>Kr from interference mass species and an EPT Magnetof for detection. A pre-existing ECR ion source will be optimized for the production of Kr+ ions. Using this ion source, it is possible to continuously sample the atmosphere at a rate of 1 cc / min, detecting changes in the concentration of atmospheric <sup>85</sup>Kr over 10 minute intervals. The design utilizes the technique of collinear resonance ionization spectroscopy (CRIS) [1] developed at the ISOLDE facility in CERN for the study of short-lived nuclei. In this implementation of the technique, the ion beam will be mass separated and bunched in a compact gas-filled linear Paul trap (currently under construction and simulation). The ion bunch will be neutralised by passing it through an alkali metal vapour. The resulting atoms can then be resonantly excited into Rydberg states with a laser beam, from which they can be ionised with an electric field and detected. Work at CERN has demonstrated that the CRIS technique is capable of reaching sensitivities of below 1 part in 10<sup>16</sup>, representing an enhancement factor of over 100 when compared with ICP-MS. By changing the frequency of the resonant lasers used, other isotopes can be selected for measurement, making the CRIS spectrometer a versatile tool for environmental monitoring.
### ELEMENTAL AND PHASE MAPPING OF SWORD FRAGMENTS FROM 2<sup>nd</sup>-1<sup>st</sup> CENTURY BCE CHINA

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Scientific investigations and archaeometric studies have played a major role in the field of archaeology, especially with regard to materials transformed through human activity, like metals. Metals are generally investigated through metallography and Scanning Electron Microscopy (SEM), which required sampling or surface preparation. Neutron techniques instead are able to provide the bulk properties of metals in a non-invasive way.

Use In this work we present a neutron imaging study of two Chinese bimetallic sword fragments from 2nd-1st century BCE. In particular, white beam Neutron Tomography (NT) and Neutron Resonance Transmission Imaging (NRTI) have been applied, using the IMAT and the INES beamlines of the ISIS pulsed neutron source in the UK, respectively.

The earliest example of bimetallic weapons in China dates as early as the Shang Dynasty (1600–1100 BCE), where meteoric iron and bronze were combined to forge weapons. With the discovery of iron smelting technology during the Spring and Autumn Period (770–473 BCE), bimetallic swords with bloomery iron and bronze became more common. They have been found in many parts of central China.

The sword fragment investigated has an iron blade mounted on a studded bronze grip (probably for a twine binding) and a ricasso with three long spikes protruding on each side. The object resembles two published examples with similar form of hilts [3, 4] listed as originating from burials investigated in the mountainous regions of Longpaozhai, in the Min River Valley (Central Sichuan), dating from the 2nd or 1st century BCE. Similar swords are also found further north and may have been introduced from further west.

NT allowed us to study the inner morphology of the sword, revealing details of its conservation status and the forging and/or casting of the different components. NRTA provided a 2D map of the elemental composition of the artefact, indicating the nature of the bronze alloy of the grip (whether tin bronze, leaded tin bronze, or arsenical tin bronze) and of the iron blade.

The study presented was complemented by Neutron Diffraction, Neutron Resonance Capture Analysis (NRCA), and by negative muons, providing a full characterisation of the object in terms of alloy composition, microstructural characterisation and elemental information, in a non-destructive way.

### NON-DESTRUCTIVE TECHNIQUES APPLIED TO THE CHARACTERIZATION OF ANCIENT GLASS MOSAIC TESSERAE

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A set of glass mosaic tesserae, dating from 2nd to the 11th century AD and coming from different geographical areas (Greece, Italy, and Syria), has been investigated via nuclear techniques to trace back the bulk composition in terms of crystalline phases and elements. Glass tesserae are composed of amorphous matrix with the addition of various raw materials, generally minerals, with different functions like colorants and/or opacifiers. The process of colouring glass tesserae is a demanding task, peculiar of the geographical area and historical period.

One way forward exploring the manufacturing technique employed is through analysis of the composition of glass tesserae themselves. The goal of this study is the application of non-destructive analytical methods to give a qualitative and quantitative description of the glass composition. At first, micro-Raman spectroscopy has been applied, which allowed for qualitative identification of the mineralogical phases acting as opacifiers. Since Raman spectroscopy is a surface and single spot technique, an investigation of the bulk properties was then provided by neutron analysis performed at the ISIS Neutron Source (UK). Neutron Resonance Capture Analysis (NRCA), Neutron Activation Analysis (NAA), and Neutron Diffraction (ND) have been chosen.

In particular, NAA led to the quantification of the isotopes and elements concentration of the samples, while NRCA returns qualitative information about the elemental composition of the bulk. Finally, ND analysis gives information about the crystalline phases dispersed in the whole bulk of the tesserae, and it can be compared to Raman spectroscopy results, which are limited to the surface.

The combined use of different non-destructive analysis returns an in-depth characterization of the mosaics, highliting the potentiality of neutron-based techniques for inhomogenous samples characterization.

#### X-RAY SPECTROSCOPY STUDY ON THAI AMULET: PHRA KRU NADUNE

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Thai Amulets exist in many forms in Thailand and were made by many different traditions and ethnic groups. Phra Kru Nadune was one of the important amulet created in the Dvarati period that found at Amphoe Nadune, Mahasarakham province, northeastern Thailand. This amulet mostly contained 2 components; powders and glass ornaments. Selected samples of the ancient amulets and the imitation ones were characterized microstructure and composition using a scanning electron microscopy coupled with an energy-dispersive X-ray fluorescence spectroscopy (SEM-EDS) and proton-induced X-ray emission spectroscopy (PIXE). It was shown that oxides of iron, magnesium, and copper were present as the main compositions of the enamel. These non-destructive and nonsampling techniques showed the information, especially the comparison of composition which was obviously differed between the ancient and new amulets.

#### PRECISE DETERMINATION OF U-235 AND RA-226 PHOTOPEAK INTENSITIES IN NATURALLY OCCURRING RADIOACTIVE MATERIALS USING OPTIMIZATION SUBROUTINE FUNCTION

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The need to determine the U-235/U-238 isotopic ratio in a naturally occurring radioactive material (NORM) is growing in order to detect uranium leakage. The gamma-ray intensities were discovered to be a significant source of confusion during the quest for a nondestructive way of testing natural matrices for the uranium isotopes U-235 and U-238. Ra-226 and U-235 are always found in the presence of each other in soil samples, and each has primary gammas whose energies differ by only 0.496 ± 0.014 keV. The least squares fitting method, using the MIGRAD function optimization subroutine in ROOT framework was implemented to obtain reliable values for the two photopeak intensities of Ra-226 and U-235. This framework was developed to examine the effect of increasing or decreasing the number of degrees of freedom in the fit on the confidence intervals of the fitted intensity parameters (heights of the two Gaussians). With the sample in a Marinelli beaker geome attempt, an ORTEC HPGe detector was used to count an IAEA U1GX natural uranium reference content considered to be in secular equilibrium. The researchers discovered that using a higher number of degrees of freedom (i.e., a smaller number of free parameters) produces more precise photo peak intensities, with differences between the calculated ratio of U-235 to Ra-226 intensities and the predicted ratio for natural uranium in secular equilibrium ranging from 0.27 to 2.8 percent.

Keywords: Uranium, HPGe, NORM, Gamma-ray.

### DEVELOPMENT OF A COMPACT X-RAY MULTI-TECHNIQUE DEVICE FOR CULTURAL HERITAGE APPLICATIONS

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The role of non-invasive non-destructive X-ray based analysis techniques is well established in the field of heritage sciences. INFN-CHNet, the network of the Italian National Institute for Nuclear Physics (INFN) devoted to Cultural Heritage, has been created for developing instruments and methods for heritage science.

Within the INFN-CHNet collaboration, a MA-XRF (Macro X-Ray Fluorescence) scanner was built-up for both elemental imaging and spectroscopy [1]. The main features of the instrument are easiness of use, high portability, good performances and ultra-low radiation dispersion. The instrument has been designed as an open system, easy to integrate with further developments.

Among the others, one intent is to upgrade the scanner allowing other X-ray based techniques for applications to cultural heritage, using only one tunable X-ray source. For this scope, two techniques planned to be added are X-Ray Luminescence (XRL) and Radiography (RX). The potentiality of XRL in cultural heritage field was recently tested to study the provenance of lapis-lazuli as reported in [2]. The usefulness of RX technique is well established, for instance to study the conditions of panel paintings and wooden statues [3].

According to the purpose, another bench-top device, based on the INFN-CHNet MA-XRF scanner, is under development within the INFN-CHNet collaboration. For XRL measurements, a CCD spectrometer, with a bandwidth between 250 nm and 1000 nm, will be mounted. For RX, in the framework of the NEXTO project funded by the Compagnia di San Paolo, a flat-panel detector with a resolution of 50  $\mu$ m was tested and is now in use. A motorised system similar to the one of the MA-XRF will allow elemental mapping and radiographies for objects with a size up to 30x30 cm<sup>2</sup>. Tests of components are ongoing and first results will be presented.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under the Marie Skłodowska-Curie grant agreement No 754511 (PhD Technologies Driven Sciences: Technologies for Cultural Heritage – T4C).

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#### GAMMA RAY TRANSMISSION TECHNIQUE WITH A BARIUM SOURCE FOR THE STUDY OF COPPER-BASED ARCHAEOLOGICAL OBJECTS

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The transmission of gamma rays from a radioactive source has been used for the estimation of the bulk composition of archaeological metal objects. This technique is useful in the study of corroded samples that cannot be polished, cut or damaged in any way because of their value such as archaeological objects. Indeed, coins and other relatively thin pieces have been studied with this technique employing a 241-Am source [1,2]. The procedure consists in measuring the attenuation coefficient at the gamma emission line energy and using this information to correct the surface elemental composition already obtained by any X-ray fluorescence technique. In this case, the thickness and the density of the material in the analysed spot must be also determined, since only a single gamma emission line can be used. Consequently, this method presents several difficulties: it requires the immersion of the objects in some fluid for density determination, which is not very welcomed by most curators, and large uncertainties may be introduced in the calculations.

In this work, we explore the advantages of using a 133-Ba source, with several useful gamma emission lines, thus avoiding the necessity of determining the density and thickness of the sample. A new special system has been built for this barium source including a CdTe detector. This set-up has been designed as a portable shielded device to be moved to museums or institutions, together with a portable XRF system. Certified reference materials and several copper-based binary alloys have been analysed to test the procedure.

Copper-based archaeological samples are good candidates to be analysed with this system because they usually present strongly altered thick corrosion patinas. Therefore, the procedure has also been checked with four bronze roman fibulae previously analysed by PIXE, providing good results with copper-tin alloys.

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#### IDENTIFICATION OF <sup>90</sup>SR IN ENVIRONMENTAL SAMPLES VIA THE HYPHENATION OF ICP-MS WITH COLLINEAR RESONANCE IONISATION SPECTROSCOPY

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The reported total volume of nuclear waste in the UK inventory is  $4.56 \times 10^6$  m<sup>3</sup> with 94% of the volume classified as low-level waste, LLW, and very low-level waste, VLLW. However, the inventory of LLW-VLLW cross-boundary waste at  $1.56 \times 10^5$  m<sup>3</sup> would benefit from an estimated saving of more that £1000 per m<sup>3</sup> in disposal processes if the characterisation of radionuclides are correctly identified. The isotope <sup>90</sup>Sr, is chemically similar to calcium and upon consumption becomes a surrogate in bones leading to an

increased risk of developing bone related cancers. Therefore, detection of <sup>90</sup>Sr abundance in the soil, groundwater and building materials near nuclear power plants serves to (i) reduce the cost of disposal due to correct identification of cross-boundary waste, and (ii) provide an accurate means of monitoring the abundance of carcinogenic radioisotopes in the environment. Both are of significant importance for the safe operation, maintenance, and decommission of a nuclear reactor site.

Using ICP-MS, parts per trillion characterisation of <sup>90</sup>Sr should be possible from soil and groundwater samples. However, isobaric interference from <sup>90</sup>Zr produces a signal 10<sup>6</sup> time greater than <sup>90</sup>Sr. The Collinear Resonance Ionisation Spectroscopy (CRIS) experiment at ISOLDE CERN has demonstrated the removal of isobaric contamination by a factor of 10<sup>7</sup> and has a significant advantage over traditional reaction/collision cells in ICP-MS for isotope discrimination. The aim of the PhD project is to develop a rapid and cost-effective method for the detection of radionuclides from environmental samples. This is to be achieved by hyphenating the CRIS method with ICP-MS by designing and constructing a prototype ICP-MS-CRIS unit that will provide proof-of-concept and validate the gain in

sensitivity via the flagship isotope <sup>90</sup>Sr. Once the system has been validated it would then be optimised to identify various other radionuclides of interest to the nuclear industry.

### HIGH SOLID ANGLE RBS DETECTION OF HEAVY ELEMENTS WITH LOW CONCENTRATIONS IN SILICON

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Rutherford backscattering spectrometry is one of the most well studied techniques for material characterisation, it is a non-destructive and model-free method with a high absolute accuracy even without the use of sample-matched standards. The sensitivity of the technique, although depending on the matrix of the sample under study, can be very good for heavy elements of the order of parts-per-million (ppm); however, the choice of the ion beam particle and energy can increase considerably the cross section, with combination with high detection solid angle, the limit of detection can achieve a few parts-per-billion (ppb).

In this work we used 2.5 MeV Si<sup>3+</sup> microbeam to measure the depth profile of very low Pt concentrations diffused in 400  $\mu$ m thick Si wafers with different annealing temperatures in order to determine the best thermal treatment. It has to be stressed that Si beam has been used to reduce possible contribution from backscattered ions, which is in the case of Si beam on Si target impossible. In that way, beam currents can be increased significantly without increasing the count rate of the large area detectors.

To maximize the detection solid angle, two large area pin diodes  $(10 \times 10 \text{ mm})$  were used in an optimized configuration. A beam collimator of 2 mm diameter was used to shield the pin diodes from the forward scattering due to the beam halo and minimize the background due to the scattering of Si ions at different components in the reaction chamber. A lateral scan of the silicon wafers was performed by focussed Si ions (of approximately 10 um size and of nA ranged currents). Two signals were gain-matched and summed. In this way maps representing the Pt depth profile were obtained with a sensitivity as low as of 3  $10^{11}$  atms/ $cm^2$  (about 3 ppb).

### STUDYING OXYGEN MOBILITY IN PHOTOCHROMIC YTTRIUM OXY-HYDRIDE FILMS BY ISOTOPIC LABELLING

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Yttrium oxyhydride (YHO) thin films show a reversible and color-neutral photochromic effect when exposed to visible light, which makes the material a promising candidate for application in smart windows[1]. The films are typically manufactured by depositing  $YH_2$ using either reactive sputtering or e<sup>-</sup>-beam evaporation which is subsequently oxidized in air[2]. The magnitude of the photochromic response strongly depends on the oxygen concentration, therefore understanding of the oxidation process of YH<sub>2</sub> is essential for the production of photochromic films. Besides, it has been hypothesized that oxygen migration is responsible for photo-induced darkening[3]. In this regard, we have performed a study of oxygen mobility in photochromic YHO films. Hydride films were produced by e-beam evaporation on carbon substrates in a scattering chamber that allows for in-situ oxidation and IBA characterization [4]. At first, a bilayer film consisting of an YH<sub>2</sub> layer on YHO was prepared. RBS measurements using 2 MeV He<sup>+</sup> conducted after deposition and repeated after 30 minutes did not reveal oxygen diffusion into the O-free layer, indicating a low oxygen mobility. Following exposure to <sup>18</sup>O<sub>2</sub> at the pressure of 3×10<sup>-3</sup> mbar led to a near-surface oxidation. While the film was fully oxidized over its whole thickness after exposure to air, the distribution of <sup>18</sup>O remained unaffected. The distribution of constituents has been subsequently verified by ToF-ERDA using 36 MeV  $^{127}$  I<sup>8+</sup>, whereas a depth profile of <sup>18</sup>O has been measured by NRA using  $^{18}$ O(p,  $\alpha$ )  $^{15}$ N reaction that features a sharp resonance at 152 keV. The result indicates that oxidation happens through grain boundaries and intercolumnar voids without long-range diffusion inside the film. Finally, the film was illuminated by 455 nm LED to trigger photochromic effect. RBS measurents conducted before and after illumination did again not show any detectable changes in chemical composition, thus excluding light-induced oxygen transport over long distances.

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### APPLICATION OF THE RUTHERFORD BACKSCATTERING METHOD IN POWDER NANOTECHNOLOGY

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Rutherford Backscattering Spectrometry (RBS) is an ion scattering technique used for compositional thin film that are less than 1 $\mu$ m thick analysis. During an RBS analysis, highenergy He<sup>2+</sup> ions with energies in the region from several hundred kiloelectron-volts to 2 -3 MeV are directed onto the sample and the energy distribution and yield of the backscattered He<sup>2+</sup> ions at a given angle is measured. Since the backscattering cross section for each element is known it is possible to obtain a quantitative compositional depth profile from the RBS spectrum obtained.

The capabilities of this method can be significantly expanded. In particular, the method can be used in powder nanotechnology to study elemental composition in microscopically small objects.

The application of methods based on Rutherford Backscattering Spectrometry is extremely interesting for adsorption energy devices, in particular, these methods can be used with maximum efficiency for various chemoelectronic converters.

A unique opportunity is to study the elemental surface of adsorbates on the surface phase separation in functional nanostructured layers.

For this reason, the preparation of planar-distributed chemoelectronic converters and the study of the elemental composition of adsorbates using the Rutherford Backscattering Spectrometry technique was the purpose for the investigation.

The tasks of this study included: development and optimization of the technology for producing planar chemoelectronic converters a functional layer in the form of rounded drops containing monodisperse nanosized (7.5  $\mu$ m) particles of a solid solution of the ZrO<sub>2</sub> system - 3 mol% Y<sub>2</sub>O<sub>3</sub> (YSZ) in the PVA polymer matrix, study of the theoretical characteristics of the obtained chemoelectronic converters [1], study of the elemental composition of the obtained chemoelectronic converters using Rutherford Backscattering Spectrometry.

The atomic and chemical composition of these layers has been studied using nuclear and atomic methods.

The thickness of the oxide layers was found to be approximately the same for all implanted samples. These values were determined on the basis of Rutherford Backscattering Spectrometry and nuclear reactions (RBS/NR).

The study was performed in the scope of the Poland-JINR and the RO-JINR Projects within the framework of themes FLNP JINR: 04-4-1140-2020/2022, 04-4-1143-2021/2025, and 03-4-1128-2017/2022.

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## NUCLEAR PHYSICS FOR ENERGY AND SPACE TECHNOLOGIES

#### SEMICONDUCTOR MATERIALS FOR RADIATION DETECTION – CURRENT STATUS AND AND FUTURE DEVELOPMENT

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For radiation detection, semiconductors have a number of distinct advantages over other detection media arising from the shear range of physical and electronic properties available. For example, wide-gap materials offer the ability to operate in a range of hostile thermal and radiation environments while still maintaining spectral resolutions <2% FWHM at X and gamma-ray wavelengths. Narrow-gap materials offer the potential of exceeding the spectral resolution of Ge by a factor of three. Currently, almost any compound can be grown, but in general, detector performances are still plagued by material problems caused by micro-crystallinity, high defect and impurity densities, stoichiometric imbalances and fabrication issues. However, some significant advances have been made - particularly in material development and the manipulation of materials at the microscopic level. For example, in defect engineering in which the mechanical and electrical properties of materials can be tailored by the precise manipulation of defects within a crystalline solid and in lattice hybridization/ dimensional reduction which allow a whole new class of compounds to be synthesized with essentially tailored properties.

In this paper we summarise the current status of research in semiconductor material development for radiation detection and discuss where the future may lie. In the near term, an increase in detector performance can be most easily achieved with improved material growth and processing techniques and with the controlled and directed manipulation of charge - for example employing single carrier sensing techniques to neutralize the degrading effects of the poorest carrier. In the intermediate term, by exploiting quantum effects at the nano-scale. In the longer term, it is clear that conventional detection techniques based on manipulating the electrons charge using electric fields will reach an impasse in terms of sensitivity. A major step forward would be to look beyond simply "collecting electrons" and exploit other obscure internal degrees of freedom of the electron in addition to its charge for non-volatile information processing. For example, utilizing its spin, or alternately, the valley degree of freedom implicit in the band structure of some semiconductors.

### PLASMA FACING MATERIALS FOR INERTIAL CONFINEMENT NUCLEAR FUSION REACTORS

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Nuclear fusion is a promising option to provide clean energy fulfilling the requirements of our increasing energy demand. The main advantages of nuclear fusion compared to other clean energy sources are: high power density and reliable power supply.

There are two main approaches to fusion energy: magnetic confinement (MCF) and inertial confinement (ICF) namely by laser (laser fusion). Important technological differences arise in ICF depending on the use of direct drive targets (laser beams directly illuminate the target) or, indirect drive targets (laser energy is converted to x-rays that illuminate the target). The most advanced projects to demonstrate the viability of fusion energy are ITER (International Thermonuclear Experimental Reactor) for MCF, and NIF/LMJ/OMEGA/FIREX for ICF. DEMO in MCF and HiPER (high-power laser energy research) for shock-direct drive in Europe and KOYO-F for fast ignition in Japan will follow.

One of the bottlenecks to make nuclear fusion a reality is the lack of materials able to withstand the combined effects of large thermal loads and radiation environments taking place in this kind of reactors.

In this talk, we will focus on the study of plasma facing materials (PFM) working in nuclear fusion reactors operated in the direct drive concept using a dry wall evacuated chamber such as HiPER. In HiPER, PFMs have to protect the chamber from X-rays, high energy neutrons (MeV) and ions (keV-MeV) generated after the burning of the target. Among all threats, ions produce the largest detrimental effects in PFM. Pulses of high fluxes ( $10^{22}$ - $10^{25}$  m<sup>-2</sup>s<sup>-1</sup>), high energy (keV-MeV), mainly light ions (He and H-isotopes) will reach the PFM producing Frenkel pairs at the depth where they are implanted. In this frame, light species will accumulate in vacancies forming bubbles which can easily grow, becoming overpressurised leading to surface blistering, cracking and exfoliation.

Focus of our study will be the performance, under these conditions, of coarse-grained W (CGW) that, nowadays, is the first candidate to be PFM. The capabilities of nanostructured W also will be discussed as one of the proposed alternatives to GCW. For nanostructured W we show the capabilities of combining ion beam analysis experiments with computational methods to study the role of grain boundaries on light species retention.

#### NEW CHALLENGES FOR EXPERIMENTAL DATA DEDICATED TO REACTOR PHYSICS

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Researches on nuclear reactors both for optimization of current generation or for study of next generations require simulations. Indeed reactor operation parameters, fuel burning, waste production, etc. can be studied by simulation with Monte Carlo or deterministic codes. These codes simulate the fundamental interaction of nucleons or ions with the matter and use as inputs nuclear data like reaction cross sections, angular distributions, fission yields, decay information ... These inputs are called evaluated nuclear data, they are compiled in evaluated nuclear databases and they are determined from experimental data and state of the art nuclear reaction codes. The increase of calculation power allows today precise sensitivity studies which reveal that the one major limiting factor for accuracy simulations of reactor parameters is the accuracy of evaluated nuclear data used as inputs. International community is thus continuously working on the improvement of evaluated nuclear data libraries like the European - JEFF (Joint Evaluated Fission and Fusion), the US - ENDF (Evaluated Nuclear Data File) or the Japanese - JENDL (Japanese Evaluated Nuclear Data Library), ... The quality of evaluated nuclear data bases can be improved with efforts both from the experimental and theoretical sides as reliance on nuclear models is common today for nuclear data evaluation. In some cases, where experimental data are scarce or known with low precision, new measurements are mandatory to provide new and relevant constraints for nuclear modeling. Moreover, experimental integral data are also used in the evaluation cycle as validation. In this presentation, after the description of the context and issues of nuclear data for reactor physics, I will focus on the new challenges we have to face, for microscopic experimental data (used for theoretical modeling improvement) but also for integral experimental data (used for validation) in the frame of the development of modern evaluated data bases.

### ION BEAM ANALYSIS IN STUDIES OF FIRST WALL MATERIALS IN CONTROLLED FUSION DEVICES

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First wall materials in controlled fusion devices undergo serious modification by several physical and chemical processes arising from plasma–wall interactions. Materials eroded from the wall are ionised, transported along the magnetic field lines in the vacuum vessel of a tokamak and then re-deposited in another location than the place of origin. This leads to the formation of mixed-material layers which contain both eroded species and hydrogen isotopes, i.e. fuel atoms such as deuterium and tritium. Consequently, the list of isotopes of interest encompasses a broad spectrum of masses from low-Z species (H,D,T), via He, light wall materials (Li, Be, B, C) to medium (steel) and heavy metals such as Mo, W. The effectiveness of material migration studies is enhanced by tracer techniques based either on the exposure marker PFC tiles, injection of a rare isotope gas or volatile compounds of high-Z metal, e.g W and Mo hexafluorides. Conclusive characterisation of wall materials requires a number of methods among which ion beam analysis techniques play a prominent role.

The aim of this contribution is to provide an overview of experimental procedures and results obtained in the examination of materials from JET, TEXTOR and COMPASS tokamaks. The role of <sup>3</sup>He-based NRA, RBS, PIXE (standard and micro-size beam) and HIERDA in fuel retention and material migration studies is presented of especially using tracer techniques with rare isotopes (e.g. C-13, N-15, O-18) or marker layers on wall diagnostic components. The application of ion beams in the modification of diagnostic components (so-called first mirrors) will be presented and the development of equipment to enhance research capabilities and issues in handling of contaminated materials are addressed.

\*See the author list: J. Mailloux et al, 28<sup>th</sup> IAEA Fusion Energy Conference, 2020 (2021).

#### REAL TIME DOSIMETRY WITH RADIO-CHROMIC FILMS

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Radio-chromic film dosimetry is a technique particularly suitable for dose measurements in medical application, industrial radiation processing and radiation hardness assurance tests. The main characteristics of radio-chromic films are the precise, accurate and permanent dose values, ease of handling and data analysis, high spatial resolution and wide range of dose. However, they are used only on passive mode and measurements of the trend of the dose in time with radio-chromic films are very difficult by means of commercial reading tools. In this work we propose a new method for the determination of the dose in real-time and by remote control with radio-chromic films. This method, based on optoelectronic instrumentation, is able to read in real-time the changes of optical properties of the films due to the darkening induced by ionizing radiation. By mean of optical fibers, the electronic instrumentation can be positioned far from the maximum radiation field. The method has been applied to different types of radio-chromic films and demonstrated to be useful in different range of doses. In addition, the analysis of the light spectra allowed to increase the dynamic range of the radio-chromic film declared by vendor itself (Fig. 1). The proposed method is an ideal dosimetric tool in environments with high radiation levels as in nuclear and high-energy collider experiment where the radiation monitoring represents a crucial issue to be carefully investigated for understanding of the beam-induced background, the replacement of detector parts and the overall experiment lifetime. A miniaturized version of this dosimeter is going to be installed in Belle II experiment at KEK laboratory in Japan.



Fig. 1: Observed variation of the transmitted light spectra through HDV2 radio-chromic film observed in real time

#### CALIBRATION CHALLENGES OF PIN DIODE SILICON DETECTOR

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The key factor influencing acquisition of reliable dosimetric values using solid state silicon dosimeters in mixed radiation fields is the accuracy of the energy calibration. Selected calibration methods are discussed with CANDY detector developed at NPI for measurement in mixed radiation field on board aircraft. The CANDY detector houses a commercially available planar silicon PIN diode as a radiation detector, a transimpedance amplifier and a pulse shaper as main components (see Figure 1). The device is battery operated for minimizing noise as much as possible.



Figure 1: CANDY detector - mechanical setup.

The device is connected to a digital oscilloscope and data acquisition is done by tailormade scripts running on a PC. This measurement setup is suitable for an experimental work and investigation of properties of the PIN diode as a detector. CANDY detector provides records about all ionisation events (each pulse is recorded including its shape). A visualisation method was developed for displaying obtained events/pulses in 2D histogram manner respecting the shape and amplitude of individual pulses (see Figure 2).



Figure 2: Example of response of detector for different bias voltages and different angles of irradiation.

Using this technique we discuss challenges in detectors calibration – namely spatial distribution of ionising radiation around the sensor, boundary effects, pulse-height energy approximation error and bias voltage dependency. The work comprises measurements with proton microprobe at Tandetron accelerator at the Nuclear Physics Institute of the Czech Academy of Sciences and HIMAC (Heavy Ion Medical Accelerator in Chiba) at National Institute of Radiological Sciences in Japan. Our findings can be used for selection of calibration methods and for selection of design principles of detectors which can be less sensitive to calibration errors.

#### APPLIED NUCLEAR PHYSICS FOR THE VERIFICATION OF NUCLEAR WEAPONS DISARMAMENT

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Today, nine countries own more than 13,000 nuclear weapons. Although states proclaim the goal of a nuclear-weapon-free world, progress is slow. A key challenge is disarmament verification; that is, monitoring disarmament activities to provide confidence that disarmament actually takes place. Here, applied nuclear physics plays an important role. Verification approaches often rely on trusted measurements of radiation emitted from fissile materials.

This contribution discusses two research projects in the field of disarmament verification. Both projects focus on passive gamma-ray spectroscopy for nuclear warhead verification and address the question: Can we trust the results?

To increase confidence in the data processing hardware, a prototype vintage verification system has been build around a MOS 6502 processor. CPUs designed in the distant past, when their use for sensitive measurements was never envisioned, drastically reduce concerns that the other party implemented back-doors or hidden switches. Their simplicity allows for easier hardware authentication. Using a custom designed digitization circuit, the device records gamma spectra from a sodium-iodine detector.

To protect sensitive information in the process, it is in the interest of nuclear weapon states to use low-resolution detectors. These detectors could enable imitations of nuclear weapons signatures. To deter such manipulations, the second project investigates the extent to which the radioactive signature of a nuclear warhead can be imitated by replacing isotopes. For selected measurement systems, detector response matrices resulting from Monte Carlo Simulations using OpenMC are used to explore whether gamma spectra of nuclear weapons or weapon-usable material can be replicated with radioactive materials.

Measurement and simulation results for both projects will be presented. The contribution will conclude with a broader overview of future research challenges and potential additional topics for applied nuclear physics.

### MEASUREMENT OF THE $^{235}$ U(N,F) CROSS SECTION RELATIVE TO N-P SCATTERING UP TO 500 MEV AT THE N\_TOF FACILITY AT CERN

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Neutron cross section standards are fundamental ingredients for both measurements and evaluations of neutron-induced reaction cross sections. This is the case of <sup>235</sup>U(n,f) cross section: one of the most important standard cross sections at thermal neutron energy and between 0.15 MeV and 200 MeV. Above 200 MeV this reaction plays an important role for several applications, ranging from biological effectiveness to nuclear technology, as well as for fundamental nuclear physics.

However, no cross section standard exists for neutron energies above 200 MeV. This led to a request for a new absolute measurements of  $^{235}$ U(n,f) cross section, in order to extend the precision and possibly to establish it as a standard up to 500 MeV.

The n\_TOF facility, at CERN, offers the possibility to improve the situation thanks to the wide neutron energy spectrum available in its experimental areas, from thermal to 1 GeV. A dedicated measurement campaign was carried out to provide accurate and precise cross section data of the <sup>235</sup>U(n,f) reaction in the energy region from 10 MeV to 500 MeV. The experimental setup consisted of two chambers to detect the <sup>235</sup>U fission events, while the number of neutrons impinging on the <sup>235</sup>U samples was simultaneously measured by exploiting the neutron-proton scattering process. To this end three Proton Recoil Telescopes were used to detect the protons emitted from two polyethylene samples placed along the neutron beam, downstream of the fission chambers.

An overview of the experimental setup will be presented, together with the first results of the data analysis.

### EXTENSION OF THE BIANCA BIOPHYSICAL MODEL UP TO FE-IONS AND APPLICATIONS FOR SPACE RADIATION RESEARCH.

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BIANCA (Blophysical ANalysis of Cell death and chromosome Aberrations) is a biophysical model, implemented as a Monte Carlo code, which simulates the induction of chromosome aberrations and cell death by different monochromatic ion beams (i.e., different ion types and energy values), as well as photons. In previous works, the model predictions for cell survival along therapeutic-like ion beams have been successfully benchmarked against experimental data, considering ions up to oxigen.

With the aim of evaluating the biological damage induced by Galactic Cosmic Rays (GCR), in this study, BIANCA was extended up to Fe-ions. Concerning cell death, experimental data available in the literature for V79 cells irradiated by ions heavier than oxygen (Si, Ne, Ar, Fe) were reproduced, and a radiobiological database describing V79 cell survival as a function of ion type ( $1\le Z\le 26$ ) and energy, as well as dose, was constructed. Following an approach developed in a previous work, analogous predictive databases for other cell lines were generated, and good agreement was found between the model predictions and the data. Concerning the induction of chromosome aberrations, which are regarded as biomarkers of radiation risk, BIANCA was applied to reproduce dicentric data in lymphocytes irradiated by different ions up to iron, as well as photons as a reference. This allowed producing a radiobiological database that predicts lymphocyte dicentric induction as a function of dose, ion type ( $1\le Z\le 26$ ) and energy.

Finally, interfacing BIANCA with the FLUKA Monte Carlo transport code, a feasibility study was performed to calculate the Relative Biological Effectiveness (RBE) of the different components of the GCR specttrum, both for dicentrics and for cell survival. Overall, following this work BIANCA can now provide RBE predictions of cell killing and lymphocyte dicentrics not only for hadrontherapy, but also for space radiation exposure.

#### EXPERIMENTAL STUDY OF SPACE RADIATION SHIELDING MATERIALS: MEASUREMENT OF SECONDARY RADIATION BEHIND THICK SHIELDING AND ASSESSMENT OF ITS RADIOBIOLOGICAL EFFECT

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The harmful effects of space radiation are a major limiting factor for long-duration human space missions beyond low Earth orbit (LEO) [1]. Space agencies have recognized the risks of exposure to space radiation and are developing complex model-based risk mitigation strategies. Space radiation risk models are based on an interplay between physical and radiobiological effects. In both fields, there are still significant gaps of knowledge [2, 3] which need to be addressed by means of high-quality experimental data, corresponding theoretical models, and the validation and improvement of simulation codes used for radiation risk assessment. One significant gap is the lack of experimental data on neutron and light ion production by galactic cosmic rays (GCR) in thick shielding.

A research cooperation that includes PTB, GSI and TIFPA-INFN has been set up to investigate the complex secondary radiation field produced by GCR-like radiation in a thick shielding. The aim is to develop a novel method for producing high-quality experimental data on neutron and light ion production in shielding materials relevant for space radiation protection. Four complementary detector systems are used to determine the energy spectra and angular distributions of high-energy secondary neutrons and light ions. In addition to the physical component of the approach, a biological component looks at the biological effectiveness of the secondary radiation, determined by irradiating samples of human peripheral lymphocytes behind the shielding. In this way, genetic effects can be studied under conditions approximating the radiation field to which an astronaut's body is exposed to e.g. an in-situ planetary habitat.

In the presentation, the experimental method and preliminary results will be discussed.

This experiment is performed at the heavy ion synchrotron SIS18 at the GSI Helmholtzzentrum für Schwerionenforschung, Darmstadt (Germany) in the frame of FAIR Phase-0. The beam time is funded by the European Space Agency (ESA) in the frame of the IBER project. This experiment is funded by the German Space Agency at the German Aerospace Center (DLR) under the funding number 50WB2125.

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#### PERFORMANCES OF A COMPACT NEUTRON DETECTOR USING HIGH PURITY <sup>10</sup>B-ENRICHED PLD-GROWTH FILMS

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Nowadays several fields require an accurate measurement of neutron flux and the development of dedicated solutions plays an important role for many applications, from nuclear waste monitoring to medicine. Different materials can be employed to convert the neutrons into charged particles, which are later detected, through very well known nuclear reaction such as  ${}^{10}B(n,\alpha)^{7}Li$ . The INFN project BoLAS-NEXT investigates the properties of very thin boron films, enriched in <sup>10</sup>B, to be employed as neutron converters, which are produced by room temperature Pulsed Laser Deposition. These films, coupled with silicon detectors, represent a very compact and cost effective solution for neutron flux monitoring. In this context, the production of high quality and uniform <sup>10</sup>B is essential and for such a reason the films have been carefully characterized in term of contamination and homogeneity. An initial simple configuration for this detector has been tested with an intense Am-Be neutron source  $(2.2 \cdot 10^6 \text{ n/s})$  for 5 different thickness of the <sup>10</sup>B film (0.5, 1, 1.2, 1.5 and 2 2m), aiming to determine the optimal value. The setup has been simulated with a Monte Carlo code in GEANT4, in order to calculate the detection efficiency and a good agreement is observed with the experimental data. During the talk the results of the preliminary test and the characterization with the simulations will be presented, together with the perspectives and the complete design of the detector.

### FACILITIES FOR COMPLEMENTARY PHYSICS EXPERIMENTS AT THE IFMIF-DONES FUSION NEUTRON SOURCE

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IFMIF-DONES – a powerful neutron irradiation facility for studies and qualification of materials to be used in fusion reactors – is planned as part of the European research roadmap to the realisation of fusion energy. Its main goal will be to study properties of materials under severe irradiation in a neutron field similar to the one in a fusion reactor first wall. It is a key facility to prepare for the construction of the DEMOnstration Power Plant envisaged to follow ITER.

At IFMIF-DONES, neutrons with fusion relevant energy spectrum and required flux intensity will be produced through Li(d,xn) stripping reactions with a D+ beam at an energy of 40 MeV impacting on a flowing liquid Li target. A unique high-current - high duty-cycle accelerator and liquid metal target technology are being developed for this facility. The preliminary engineering design of the facility has been completed by a collaboration of research institutes and industrial partners within the framework of the EUROfusion consortium. Besides of the systems and equipment devoted to the main fusion-related objectives, the present design of IFMIF-DONES also includes facilities for complementary nuclear and applied physics experiments.

In this contribution, opportunities offered at the IFMIF-DONES neutron source for experiments using a collimated beam of neutrons in the MeV energy range, slow energy neutron beams, and a fraction of the deuteron beam extracted from the main accelerator line will be presented. Some preliminary assumptions on the implementation of complementary experiments, such as neutron scattering, nuclear spectroscopy, nuclear reactions induced by neutrons, production of radioisotopes, and studies of radiation effects in electronic devices, will be discussed.

This work has been carried out within the framework of the EUROfusion Consortium Early Neutrons Source work package (WPENS) and has received funding from the Euratom research and training programme 2014-2020 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

#### ELEMENTAL ANALYSIS OF CONCRETE VIA FAST NEUTRON TRANSMISSION AND SCATTERING SPECTROMETRY

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Within all nuclear installations concrete is used for its structural properties and the shielding of radiation. Concrete is a composite material of cement, sand, large aggregate and water, although various additives may also be introduced to alter the structural or shielding properties. Standard concrete mixtures comprise largely of hydrogen, oxygen, aluminium, silicon, calcium and iron. Developing methods to independently and non-destructively verify the composition of such materials is of high priority for the regulation of existing and future nuclear installations. Of importance to the neutron shielding properties of concrete is the water content, which has further significance as nuclear installations age.

We report on the development of neutron-based techniques to non-destructively measure the composition of concrete at the fast neutron laboratory, n-lab, within the Department of Physics, University of Cape Town. Two neutron sources are currently available, a D-T sealed tube neutron generator (STNG) and a radioisotopic <sup>241</sup>Am<sup>9</sup>Be source. Measurements were made of the transmitted and scattered neutron energy spectra from various of elemental and composite samples using an EJ301 organic liquid scintillator. These measurements were used to determine the various ratios of water, sand and cement in a simple mortar mix, in addition to the elemental compositions of each. These data are compared to XRF measurements of the major elements. Monte-Carlo based radiation transport calculations were performed with MCNP6 to support these measurements and verify the analysis procedure. We present the analyses of measured neutron energy spectra, and MCNP6 calculations, which offer new opportunities to non-destructively determine the composition of concrete samples of unknown origin.

#### DEVELOPMENT OF DIAMOND BASED CRYOGENIC NEUTRON DETECTORS FOR NUCLEAR FUSION APPLICATIONS

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Neutron damage in materials is one of the key areas of research in the roadmap to the fusion DEMOnstration Power Plant (DEMO). This work presents the current status in the development of a compact neutron detector based on a CVD diamond, which is designed to work at cryogenic temperatures near the superconductive solenoid magnets in the next generation material research facility DEMO Oriented Neutron Source (DONES). Due to their size and radiation hardness, detectors based on CVD diamonds are key candidates for micro-loss monitors envisioned to monitor the neutron environment around the accelerator beam tube.

To gain a better understanding of the response of a diamond detector at cryogenic temperature, a detector was created using a 300  $\mu$ m CVD diamond metalized with 200 nm W electrodes bonded to a ceramic PCB. The detector was irradiated from room temperature down to 38 K with 2.4 MeV protons carried out at the Ruđer Bošković Institute (RBI) nuclear microprobe. Moreover, the response was also studied in the aforementioned temperature range with  $\alpha$  particles, obtained by a triple  $\alpha$  source containing <sup>239</sup>Pu, <sup>241</sup>Am and <sup>244</sup>Cm. Furthermore, the diamond was also bombarded with 14 MeV neutrons at the RBI neutron generator. The charge collection efficiency (CCE), which is defined as the ratio of the measured charge to the total generated charge, was shown to follow a logistic function (Fermi function) with temperature. Full CCE (100%) was observed at higher temperatures followed by a drastic decrease in the temperature range from 110 K to 70 K. Below this temperature, no change in the CCE was observed down to 38 K. Additionally, it was also observed that the CCE decrease varied for each impinging particle type.

The obtained results are crucial for the study of the detector behavior at cryogenic temperatures with the aim of developing a method for neutron-gamma discrimination, yielding an accurate measurement of neutron flux in mixed radiation environments.

### POROUS POLYDIMETHYLSILOXANE COMPOSITE FILLED WITH GRAPHENE OXIDE AND GOLD NANOPARTICLES

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**Keywords:** Nanoparticles, laser ablation in liquid, biomaterial, polydimethylsiloxane, graphene oxide

The design of new materials with improved mechanical and biological properties is a major interest in tissue engineering, nanotechnology and material science. This paper presents the synthesis of macro-porous polydimethylsiloxane (PDMS) sponges composites with pore sizes ranging between 50 and 900  $\mu$ m and further decorated with nanoparticles. The sponge was realized using the sugar templating process. Nanoparticles of gold, graphene oxide and gold linked to graphene oxide were produced by laser ablation in liquid. Graphene oxide is a biocompatible material and its ability to grasp gold nanoparticles preventing their aggregations represents a green alternative on the production of metallic nanoparticles without the use of surfactants. The production of the three different solutions containing the nanoparticles was studied by ion beam analytical methods. The control of the size, shape and the uniform dispersion of produced nanoparticles was monitored by scanning and transmission electron microscopies. The realization of the gold nanoparticles with size of about 10 nm was monitored by UV–Vis–NIR absorption spectroscopy.





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