UNIVERSITÀ DI TORINO

Fisica Torino Dipartimento di Fisica

nis Nanomaterials for Industry and Sustainability

INFN

NIS COLLOQUIUM ITACA

Ion-beam Techniques Applied to Cultural heritage and Advanced materials

12th-13th June 2023

Aula C - Dip. di Fisica
Via Pietro Giuria, 1 - Torino

PROGRAM

12th June 2023

9.15	**Opening**
9.35	Paolo Olivero (Università di Torino)
10.10	Natko Skukan (IAEA)
10.45	**Coffee break**
11.15	Damiano Giubertoni (FBK)
11.50	Jan Meijer (Universität Leipzig)
12.25	**Lunch**
14.00	Zdravko Siketi (RBI)
14.35	Javier Garcia Lopez (Universidad de Sevilla)
15.10	**Coffee break**
15.40	Matteo Campostrini (INFN-LNL)
16.15	Aleksandr Ponomarev (IAP- NAS of Ukraine)
16.50	Ettore Vittone (Università di Torino)
17.20	**Ion implanter inauguration**

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9.15	**Opening**
9.35	Alessandro Lo Giudice (Università di Torino)
10.10	Quentin Lemasson (C2RMF)
10.45	**Coffee break**
11.15	Roger Webb (University of Surrey)
11.50	Ziga Smit (Univerza v Ljubljani)
12.25	**Lunch**
14.00	Maria Dolores Ynsa Alcala (Universidad Autónoma de Madrid)
14.35	Luis Cerqueira Alves (Universidade de Lisboa)
15.10	**Closing**

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9.15 - 9.35 **NIS COLLOQUIUM OPENING**

9.35 - 10.10 **FABRICATION AND OPTICAL FUNCTIONALIZATION OF DIAMOND WITH ENERGETIC ION BEAMS**

The "Solid State Physics" Research Group of the University of Torino has a consolidated tradition in research activities focused on the use of energetic ion beams for both the characterization and controlled modification of wide-bandgap materials. In the last decade, the Research Group committed an ever-increasing focus on the employment of the use of energetic ions for the functionalization of artificial diamond, for applications in quantum technologies and biosensing. In the talk, the following activities will be overviewed: - development of ion-beam based lithographic techniques for the engineering of graphitic micro/nano-structures in diamond; - use of ion-beam lithography for the fabrication of in-vitro cellular biosensors based on diamond substrates; - use of ion beam irradiation for defect engineering of wide-bandgap semiconductors, with the purpose of developing quantum-light emitters with optimal properties in the field of quantum communication and sensing.

Paolo Olivero - Università degli Studi di Torino

10.10 - 10.45 **IAEA ACTIVITIES IN SUPPORT OF UTILIZATION OF ELECTROSTATIC ACCELERATORS**

Promotion of nuclear applications for peaceful purposes and related capacity building is among the missions of the International Atomic Energy Agency (IAEA). Accelerator applications are one of the thematic areas, where the IAEA supports its Member States in strengthening their capabilities to adopt and benefit from the usage of accelerators. Physics section of the IAEA actively supports its Member States in applying accelerator techniques using different mechanisms available within the Agency. These can span from support in establishing an electrostatic accelerator laboratory to leading Coordinated Research Projects (CRP) in the state-of-the-art electrostatic accelerator applications such as quantum technologies. This talk will give an overview of the IAEA support modalities with several examples, some of them in the applications of ion beams in materials science

Natko Skukan - International Atomic Energy Agency (IAEA)

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11.15 - 11.50 **NANOMETRIC MODIFICATION OF SOLIDS BY MULTI SPECIES ION COLUMN FIB**

Focused Ion Beam instruments (FIB) have represented a valuable tool for nanotechnology development in the last 40 years. Initially developed for the material and device characterization (imaging, cross-sectioning, lamella preparation), they were then adopted to directly pattern desired structures in every solid material. Exploiting the local sputtering induced by beams focused to <20 nm diameter, they allowed the realization of nanometric prototypes and devices. In the last 15 years, new opportunities arose from the introduction of liquid metal alloy ion sources (LMAIS) and multi-ion columns, making possible a wider variety of not only patterning strategies but also new opportunities of material modification. In this talk we will report examples of results obtained using a FIB instrument equipped with a LMAIS able to deliver focused beams of Au, Ge and Si ions: 'classical' patterning of Au films to create nanometric plasmonic structures on suspended Si3N4 membranes; modification of the Si resistance to Tetramethylammonium hydroxide (TMAH) etching to create suspended NEMS; deterministic ion implantation to create color centers in wide bandgap semiconductors.

Damiano Giubertoni - Fondazione Bruno Kessler (FBK)

11.50 - 12.25 **MOBILE QUANTUM COMPUTER**

In the talk we present a mobile quantum computer based on NV. The physical properties of the NV centres allow use at room temperature and mobile use for industrial applications. Gate operations are demonstrated live.

Jan Meijer - Universität Leipzig

12.25 - 14.00 **LUNCH**

14.00 - 14.35 **APPLICATION OF CONVENTIONAL AND UNIQUE ION BEAM TECHNIQUES AT RBI FOR MATERIAL CHARACTERISATION AND MODIFICATION**

Laboratory for Ion Beam Interactions (LIBI) at the Ruđer Bošković Institute (RBI) is equipped with two ion beam accelerators, 6 MV Tandem Van de Graaff and 1 MV Tandentron, capable of delivering ions with MeV energies to the nine different end- stations.

Conventional techniques such as Particle Induced X-ray Emission (PIXE) or Elastic Backscattering (EBS) are routinely used for elemental characterisation and depth profiling of various samples. In addition to the broad beam, a focused ion beam is also used, which allows 2D mapping of samples with micrometre spatial resolution using the above-mentioned techniques. In addition to PIXE and RBS, more advanced techniques such as ion beam induced charge (IBIC), Time-of-flight Elastic Recoil Detection Analysis (TOF-ERDA) and MeV Secondary ion mass spectrometry (MeV SIMS) are developed for the detector characterisation, depth profiling of thin films and mapping of organic samples. In addition to material characterisation, MeV ion beams are also used for material modification using high-current dual beam capability (DiFu end-station) or controlled single ion implantation using single or dual microbeam end-stations. In this contribution, examples of the application of PIXE and RBS for the analysis of cultural heritage samples will be shown. Recent results of the application of the unique techniques IBIC, TOF-ERDA and MeV SIMS will be presented. Finally, some examples of study of materials used in fusion reactors and the creation of colour centres in diamond will be shown.

Zdravko Siketić - Ruđer Bošković Institute (RBI)

14.35 - 15.10 **ANALYSIS OF RADIATION DETECTORS USING MEV ION BEAMS**

In this talk, we will briefly present the infrastructure available at the Centro Nacional de Aceleradores of the University of Seville, based on a 3 MV tandem accelerator, which is used for several Nuclear Physics applications, as characterization and modification of materials using ion beams, irradiation of electronic devices, neutron research and development of nuclear instrumentation. In the following, we will describe how we can use monoenergetic ion beams to study the behaviour of different radiation detectors. Some of the devices, developed at the Institute of Microelectronics of Barcelona, are prototypes that represent the state of the art in the field of semiconductor detectors. Among the illustrative examples, the characterization of 3D Si microdosimeters of interest in hadrotherapy, the analysis of low gain avalanche detectors (LGAD) based on Si, developed for high energy physics experiments due to their excellent timing properties and the high

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temperature spectroscopic response of SiC diodes, with interest in nuclear fusion research, will be discussed.

Javier Garcia Lopez - Universidad de Sevilla

15.10 - 15.40 **COFFEE BREAK**

15.40 - 16.15 **LNL VAN DE GRAAFF ACCELERATOR FACILITIES: IBA AND APPLICATIONS**

The MeV Legnaro National Laboratories Van De Graaff accelerators facilities continue to play an essential role in INFN's research in the fields of nuclear physics, astrophysics, medical physics, and in the study, development and characterisation of materials, cultural heritage and environmental samples, with specific applications in detectors and electronic components testing, irradiation of spacecraft materials and more recently in precision targeting for quantum technology applications. In recent years, new applications in both nuclear medical physics have been pushing to the development of new isotopic nuclear targets with considerable improvements on material purity. Ion Beam analysis characterisation techniques are been used as a fundamental tool to drive the production methods and to allow for precise nuclear cross section measurements. Parallel to IBA applications the LNL MeV accelerators are used to irradiate spacecraft materials on large area following Space Agency prescriptions to emulate the lifetime of satellite components, and are deeply involved in single particle irradiation for testing new detectors and for the creation of color centres and defects in semiconductors.

Matteo Campostrini - INFN-LNL

16.15 - 16.50 **NUCLEAR MICROPROBE ACTIVITIES IN INSTITUTE OF APPLIED PHYSICS OF NATIONAL ACADEMY OF SCIENCES OF UKRAINE**

The nuclear microprobe at the Institute of Applied Physics (IAP) was put into operation in 2008 on the basis of a single-ended Van de Graaff-type electrostatic accelerator with a maximum energy

of 2 MeV. All the main elements of the microprobe: the accelerator, integrated doublets of magnetic quadrupole lenses, collimators, magnetic scanning system, data acquisition system, target chamber and beam line, were made in Ukraine. The focusing system of the microprobe is a separated orthomorphic quadruplet with demagnification of 23. The resolution of the microprobe in high current mode is about 2 μm, in low current mode it is 0.6 μm.

In connection with the growing scale of construction in Ukraine before the war and the discovery of historical heritage artifacts, it became necessary to study them. A number of such studies were carried out on our microprobe in the micro-PIXE technique. Sumy State University conducts research on solar energy converters. A nuclear microprobe was used to determine the uniformity of the distribution of chemical elements over the surface of the absorber layer of thin film solar cells.

For solid-state joining of stainless steel with zirconium alloy, thin interlayers of copper and niobium are used on a special hot vacuum rolling equipment at the Kharkov Institute of Physics and Technology. During cyclic heating at the joint boundary using a microprobe, sulfur segregation was detected at the grain boundaries of the copper interlayer.

The nuclear microprobe was used to determine the distribution of chemical elements in geological samples of uraninites from various deposits. The use of a microprobe was due to the micrometric dimensions of the samples, which does not lead to the need of using a hot chamber.

The IAP is working on the creation of a phase-contrast X-ray unit based on an electrostatic accelerator. To create coherent X-ray radiation, it is necessary to make X-ray diffraction gratings. For these purposes, a microprobe with focusing into a thin line was used using proton-beam writing technique.

In the 1990s, our institute collaborated with A.D. Dymnikov. As a result of this collaboration, a group of employees was formed, which mastered the matrix method - the matricant method, which is subsequently used in research on ion optics in a nuclear microprobe.

Aleksandr Ponomarev - Institute of Applied Physics of National Academy of Sciences of Ukraine

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16.50 - 17.20 **FUNCTIONAL CHARACTERIZATION AND FUNCTIONALIZATION OF MATERIALS BY ION BEAMS**

Ion beam techniques (IBTs) are well-established techniques for both the analysis and the modification of solid-state materials, whose potential results from the peculiarities of the interaction of ions, in the keV-MeV region, with matter. Besides the traditional use for the structural or compositional analysis of materials, IBTs play important roles also to investigate material features, which are directly involved in the functions of related devices, i.e. for material functional characterization. An example is given by the Ion Beam Induced Charge (IBIC) technique, which allows the electronic characterization of semiconductor materials and devices. Concurrently, ions can act as active agents, capable to induce controlled modifications of the structural and functional properties of materials as well as changing their chemical composition, as happens in the doping processes of semiconductors. The coupling of defects generated by ion-material interaction with different chemical species introduced in a solid material by ion implantation, combined with the analytical functional features of IBTs, opens the possibility to deterministically manipulate the material's optical/electronic properties at the micro/nanoscale. Since the '90s, the Solid State Physics group has exploited the analytical capability of IBTs as well as their potential for material modification in activities spanning from the mapping of the charge collection efficiency to the assessment of the radiation hardness of semiconductors, from the micro/nano modification of silicon to the creation of color centers in high band gap materials. This contribution is aimed at presenting the main results achieved by the group in this field and the future direction of research.

Ettore Vittone - Università degli Studi di Torino

17.20 - 18.00 **ION IMPLANTER INAUGURATION**

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9.15 - 9.35 **NIS COLLOQUIUM OPENING 2ND DAY**

9.35 - 10.10 **ION BEAM ANALYSIS FOR ARCHEOLOGICAL MATERIALS:PROVENANCE STUDY OF LAPIS LAZULI**

It has been more than 15 years since the start of activities in physics applied to archaeometry and cultural heritage at the Physics Department of the University of Torino and at the INFN (National Institute for Nuclear Physics) Torino division. The important impact on society can be seen in the collaborations that have been established not only with national and international academic and research institutions, but also with museums, superintendencies and restoration centres. In Torino, the main activities involve the use of X-ray, proton and neutron beams for the analysis of materials and objects of interest in archaeology and art history. Analysis with proton and neutron beams are provided by means of transnational access programs at medium and large scale facilities. The main information that can be obtained concern the conservation state of artworks, the types of materials used, the construction techniques, the provenance of raw materials, etc. This talk is focused on the use of Ion Beam Analysis, in particular μ-PIXE (Particle Induced X-ray Emission) and μ-IBIL (Ion Beam Induced Luminescence) techniques, employed for lapis lazuli provenance studies to reconstruct ancient trade routes. The research, started more than 15 years ago, is carried out in collaboration with archaeologists, geologists and material scientists and many of the analysed objects belong to museums. The analyses have been performed both in vacuum at INFN-LNL (Legnaro, Italy) and with an extracted beam at NewAGLAE (Paris, France) in the framework of the IPERION-HS program. Moreover, in 2023 the in vacuum beamlines at both Surrey Ion Beam Centre (in the framework of RADIATE program) and Ruđer Bošković Institute (in the framework of CERIC program) are to be used for the first time by our group.

Alessandro Lo Giudice - Università degli Studi di Torino

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10.10 - 10.45 **IBA FOR HERITAGE SCIENCE AT NEW AGLAE**

Quickly after the development of ion beam analysis (IBA) techniques in the 1970s, they were adapted to heritage materials. The aim is to provide answers to questions of materials science as a part more general Humanities issues. Knowing how an artefact was made provides information on the production centre where it was made, the date of manufacture and possibly the materials used and their provenance, thus providing information on the trade routes in place at the time of manufacture. Other more contemporary issues concern the detection of fakes or the conservation of materials. The IBAs provide fundamental elements for understanding the structure of materials, their composition, their properties and their transformations over time, thus providing valuable information for answering the aforesaid questions. The AGLAE accelerator has been installed at the Palais du Louvre for over 30 years and is dedicated to the non-invasive analysis of heritage materials. The Equipex NewAGLAE project has reinforced this mission. The accelerator now provides IBA imaging capabilities and allow night-time operation. During these thirty years of operation, countless objects from the collections of French museums, archaeological excavations and European collections have been analyzed by IBA, adding new stones to our knowledge of heritage.

Quentin Lemasson - C2RMF - Centre de Recherche et de Restauration des Musées de France

10.45 - 11.15 **COFFEE BREAK**

11.15 - 11.50 **MULTIMODAL IMAGING FOR THE COLOCATION OF ORGANIC MOLECULES AND METAL MARKERS USING SPATIALLY RESOLVED MASS SPECTROMETRY WITH ELEMENTAL MAPPING**

The colocation of elemental species with organic molecules provides new insights into complex biomedical materials and could be of use in understanding and provenancing cultural heritage samples. In biomedicine alkali metals have been the subject of extensive research and are implicated in various neurodegenerative and infectious diseases and are known to

disrupt lipid metabolism. Desorption electrospray ionisation (DESI) is a widely used approach for molecular imaging, but DESI delocalizes ions such as potassium (K) and chlorine (Cl), precluding the subsequent elemental analysis of the same section of tissue. Modifying the solvent can prevent the delocalization of elemental species and thus enables elemental mapping to be performed on the same sample section post-DESI. A robust multimodal imaging workflow can then be applied to a lung tissue section containing a tuberculosis granuloma, showcasing its applicability to elementally rich samples displaying defined structural information. This same technique could be readily adapted for cultural heritage samples.
Roger Webb - University of Surrey

11.50 - 12.25 **ARCHAEOMETRIC MEASUREMENTS AT THE TANDETRON ACCELERATOR IN LJUBLJANA**

Studies in archaeometry are part of activities at the Tandetron accelerator of the Jožef Stefan Institute in Ljubljana. Experiments are performed by 3 MeV protons at the in-air beamline, using spectroscopies of proton-induced X-rays (PIXE) and gamma rays (PIGE). The materials include archeological metals, glass, (semi) precious stones, as well as artworks from the point of using pigments and their degradation. In the field of metals, we studied the beginning of the use of brass at the beginning of the 1st c. BC, and several problems in numismatics, like the trade routes of silver in medieval silver coinage. For the analysis of glass, we developed a combined PIXE-PIGE analytical method, with which we studied glass of almost all historical periods. Our initial interest was if raw glass was produced in the 16th century Ljubljana. We also contributed to the studies of Roman glass, which should point to major trade routes as reflection of political influences. Transition from natron-type to plant-ash-type glass may be used for dating of the early medieval graves. The National Museum of Slovenia keeps a donation of glass vessels from 1834. Is the origin of this glass consistent with the record? Among the artworks, which artworks were not made by Hans Georg Geiger, a popular baroque painter? Is it possible to identify a mid-19th c. photographic technique? Where did garnets and emeralds come

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from? Are some pigments damaging to paper? How was the cream-colored ceramics of 1800 was made? What were the techniques for gilding Late Antique bronze and silver brooches? The questions to these answers will be reviewed, together with further technical development of the beamline.
Žiga Šmit - Univerza v Ljubljani

12.25 - 14.00 **LUNCH**

14.00 - 14.35 **SUITABILITY OF ION BEAM ANALYSIS (IBA) TECHNIQUES FOR THE CHARACTERIZATION OF ARCHAEOLOGICAL METALLIC OBJECTS**

The adequate restoration and conservation treatments of archaeological metallic pieces require the accurate determination of the elemental composition and distribution within the objects, as well as the identification of the nature and distribution of the corrosion products. Due to their non-destructive character, Ion Beam Analysis techniques such as PIXE (Particle Induce X-ray Emission) and RBS (Rutherford Backscattering Spectrometry) have proved to be of great utility for this purpose. In order to illustrate these capabilities of IBA techniques, two different works carried out at the CMAM external microbeam line have been chosen: the application of IBA techniques for the assessment of laser cleaning on a mediaeval gilded copper cross and the study of ancient Islamic metallic pieces.
Maria Dolores Ynsa - Universidad Autónoma de Madrid

14.35 - 15.10 **OLD AND NEW MATERIALS PROBED BY ION BEAMS**

Material analysis of Cultural Heritage artefacts aims to be able to answer to questions like, what were the raw materials used, how were they made (production techniques), which is their probable provenance or if they are forged or looted goods. This analysis often requires the use of non-destructive analytical techniques for which MeV ion beams are well suited. They gather high sensitivity with extended information obtained when combining different

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spectrometry techniques as PIXE (Particle Induced X-ray Emission), RBS/EBS (Rutherford/Elastic Backscattering Spectrometry), PIGE (Particle Induced Gamma-Ray Analysis) and NRA (Nuclear Reaction Analysis). With the use of a scanning nuclear microprobe lateral spatial information and imaging can also be obtained. But, as in a recent past, where both the development of ion beams transport and techniques were closely interconnected with the development of the Si based semiconductor industry, new functional materials can profit not only from the ion beam characterization capabilities with important feedback to the manufacturer but also from ion beam materials modification or defect engineering as a means to improve or tune device performances. Here we present some case studies carried out at, or mainly conducted by researchers from the Laboratory of Accelerators and Radiation Technologies from IST where the advantages of the combination of IBA techniques are explored and applied to the study of historical metal and glass samples together with some work performed in solar cell materials and wide band gap semiconductors.
Luis Cerqueira Alves – Universidade de Lisboa

15.10 – 15.30 **NIS COLLOQUIUM CLOSING**

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