GENERAL INFORMATION
SCIENTIFIC PROGRAMME
BOOK of ABTRACTS

14th International Conference on Nuclear Microprobe Technology and Applications

7 July 2014 - 11 July 2014

Palazzo del Bo and Centro Culturale San Gaetano - Padova, Italy

Organized by

INFN Istituto Nazionale di Fisica Nucleare

In cooperation with

IAEA International Atomic Energy Agency

Under the patronage of

Comune di Padova

UNIVERSITÀ DEGLI STUDI FIRENZE

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ICNMTA2014

14th International Conference on

Nuclear Microprobe Technology and Applications

and

International Workshop on Proton Beam Writing

July 7-11, 2014

Padova, Italy

WELCOME

On behalf of the International and Program Committee, we welcome you to the 14th International Conference on Nuclear Microprobe Technology & Applications, organized by the Italian National Institute for Nuclear Physics (INFN) under the patronage of the University of Firenze, Padova and Torino and in cooperation with the International Atomic Energy Agency (IAEA).

As in the tradition of ICNMTA's, the main goal of this conference is to provide an opportunity for researchers to present their most recent progresses, to exchange ideas and thoughts and to explore new research directions using nuclear microprobes. The more than 150 contributions collected demonstrate the vitality of the nuclear microprobe community, which, after 26 years, still consider this event as the most appropriate to present results to an international audience and to learn what's happening worldwide in the field of ion microscopy.

The conference, structured in plenary, oral and poster sessions, covers the many aspects of nuclear microscopy, regarding both the development of the micro/nano focused ion beam technology and the state of the art of its applications in many scientific fields, ranging from high-tech materials to biology, geology, art and archaeometry. On July 7th, a special plenary session with keynote lectures will be held at the Aula Magna, the Great Hall of the historical building (Palazzo Bo) of the University of Padova, one of the oldest, nearly 800 years history, universities in Europe, and famous for having had Galileo among its lecturers. Sessions with invited technical and oral presentations, a permanent poster session and industrial exhibitions will take place at the Centro Culturale San Gaetano in Padova, from July 7th to 11th. A round table on "*New strategies for fundraising to support projects in micro and nano ion beam technologies*" and the Proton Beam Writing Workshop, with a panel discussion on "*Nano positioning stages for Nano Probes used in Proton Beam Writing and Nuclear Microscopy*" are organized on July 10th.

We are grateful to all the Institutions and the Industrial sponsors, which supported this conference and we thank all the participants for their contribution. We hope that the scientific programme and our hospitality match your expectation and that the unrivalled charm of Italy, and in particular of Padova and Venice, can contribute to establish fruitful collaborations during and after the Conference.

We wish all participants a fruitful and successful Conference and a nice stay in Italy.

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Lorenzo Giuntini – University of Firenze – co-chair

Ettore Vittone – University of Torino – co-chair

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www.pelletron.com/

www.microbeams.co.uk/

CONFERENCE VENUE

Monday, July 7, 2014
8:30: registration
9:00: opening

Palazzo del Bo
University of Padova
via VIII Febbraio, 2 – Padova
ph. +39-049-827 5111
http://www.unipd.it/en/node/261
(letter A on Padova Map)

The first session, on Monday, will be held at the *Aula Magna* ("the Main Hall") of "Palazzo Bo" of the University of Padova , where Galileo Galilei taught experimental physics and astronomy.

From Tuesday, July 8 to Friday July 11

Centro Culturale Altinate San Gaetano
via Altinate 71- Padova
ph. + 39-049-8204715
http://www.altinatesangaetano.it/en
(letter B on Padova Map)

From Tuesday to Friday the Conference will be held in the Civic Center of Art and Culture Altinate / San Gaetano in the very center of Padova. It is a civic centre aimed at hosting and promoting cultural initiatives such as conferences, exhibitions, shows and art displays, with a coffee-shop and restaurant inside. The "Centro Culturale S.Gaetano", originally laid down in 1582 by the architect Vincenzo Scamozzi, has been completely renovated and improved in 2008, now bringing together the charm of a classical site with all the comforts of a modern structure.

SOCIAL EVENTS

Welcome Cocktail

Sunday, July 6, from 6:30 p.m. to 9:30 p.m.

Via VIII Febbraio, Padova
Ph. +39- 049-8205111
(letter W on Padova Map)

A rich welcome cocktail will be served at the *Loggia pensile* of the Padova City Hall (Palazzo Moroni).

Social Excursion

Wednesday, July 9, from 1:45 to 11:30 p.m.

Meeting point:
Centro Culturale Altinate San Gaetano
(letter B on Padova Map)

A Conference excursion to Venice and its lagoon (http://whc.unesco.org/en/list/394) has been organized. We will board near Venice for an extraordinary cruise around the Lagoon! The first stop will be in Murano, a lagoon island famous for glass manufacturing and blowing. A live demonstration of glass blowing will be offered by the *Maestri Vetrai* (glassblowing Masters) in one of the last furnaces still active on the Island. We will then go back to our ship and find a rich buffet cocktail while cruising towards St. Mark's place, where we will dock after a pleasant navigation. Free time at your disposal to visit St. Mark's place and its surroundings. At around 8 p.m. we will board the ship once again, where we will find a Venetian, fish-based dinner to wait for us. The navigation will continue until time to board the bus and finally return to Padova.

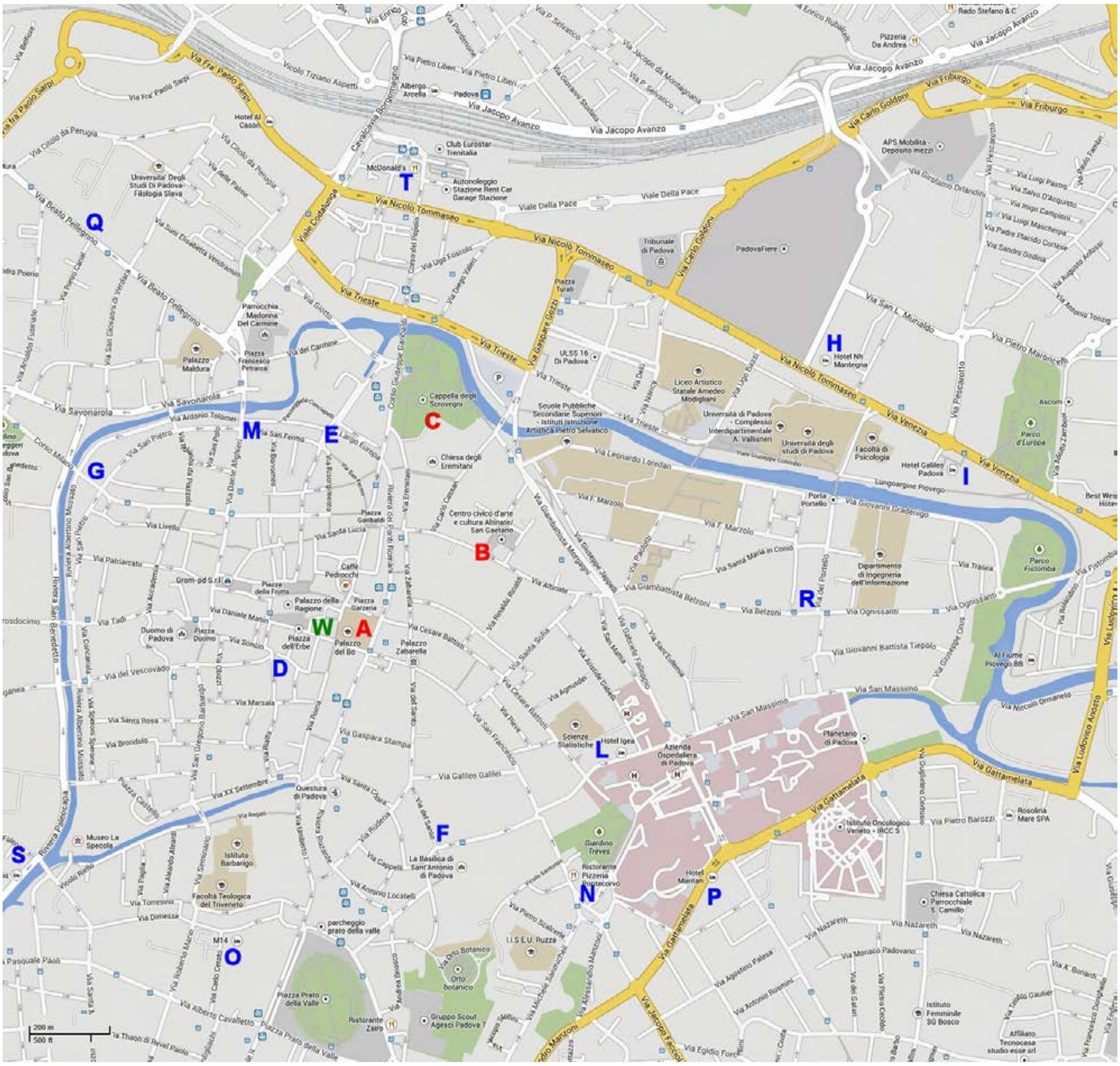
Social Dinner

Thursday, July 10, from 6:45 p.m. onwards

Piazza Eremitani 8, Padova
tel. +39-049-8204551
(letter C on Padova Map)

Padova is the undisputed capital of 14th century art, so why not take advantage of one of its most amazing museums for ICNMTA Social Dinner?
The setting will be the MUSEO CIVICO AGLI EREMITANI (Eremitani Civic Museum), with its magnificent Paleovenetian and Roman collections, as well as the Museum of Medieval and Modern Art with more than 3000 works.
The nearby CAPPELLA DEGLI SCROVEGNI hosts one of the masterpieces of the 14th Century, the frescoes painted by Giotto commissioned by Enrico degli Scrovegni, a wealthy banker from Padua, for the benefit of his family.
Both places will be opened to ICNMTA Social Dinner participants, and the dinner will be served in the magnificent *Magnolia Cloister*.

PADOVA MAP



A = Palazzo Bo’
B = Centro Culturale San Gaetano
C = Musei Civici Eremitani – Cappella Scrovegni
D = Hotel Toscanelli
E = Hotel Europa
F = Hotel Donatello
G = Hotel Plaza
H = Hotel Mantegna
I = Hotel Best Western Galileo

L = Hotel Igea
M = Hotel Sant’Antonio
N = Hotel Giotto
O = Hotel M14
Q = Hotel Patavium
R = Hotel Belzoni
S = Hotel Methis
T= Hotel Grand’Italia
W = Palazzo Moroni – Loggia Pensile

*The meeting point for the excursion to Venice is at Centro Culturale San Gaetano (**B**)*

PROGRAMME

Sunday 6th

18:30 – 21:30 - Palazzo Moroni – Registration and Welcome Party

Monday 7th

8:30-9:00 Registration

9:00-9:50 Opening

9:50-11:10 Plenary 1
Convener: Teresa Pinheiro

11:10-11:40 Coffee Break

11:40-13:00 Plenary 2
Convener: Dario Bisello

13:00-1350 Lunch Break

Group Photo

14:00-16:00 Plenary 3
Convener: Geoffrey Grime

16:00-16:30 Coffee Break

16:30-17:50 Plenary 4
Convener: Milko Jaksic

Tuedsay 8th

8:30-10:40 Session 1
Nuclear Microprobe Technology 1
Conv: Istvan Rajta

10:40-11:10 Coffee Break

11:10-13:10 Session 2
Nuclear Microprobe Technology 2
Conv: Chris Ryan

13:10-15:20 Lunch Break

15:20-17:00 Session 3
Nuclear Microprobe Applicarions Biology 1
Conv: Maria Dolores Ynsa

17:00-19:30 Poster Session
With Cheese and Wine

Wednesday 9th

8:30-12:40 Poster Exibition

8:30-10:30 Session 4
Nuclear Microprobe Applicarions Biology 2
Conv: Richard Ortega

10:30-11:00 Coffee Break

11:00-12:40 Session 5
Nuclear Microprobe Applications: Material Science
Conv: Tomihiro Kamiya

13:45-23:00 Conference Excursion to Venice Lagoon

Thursday 10th

8:30-17:45 Poster Exibition

8:30-10:10 Session 6
Nuclear Microprobe Applications: Ion Beam Material Micro-Modification
Conv: Paolo Olivero

10:10-10:30 Coffee Break

10:30-12:30 Session 7
Proton Beam Writing Workshop
Conv: Jeroen Anton van Kan

12:30-13:00 Panel Discussion

13:00-14:00 Lunch Break

14:00-15:10 Session 8
Nuclear Microprobe Applications: Microelectronics
Conv: Gyorgy Vizkeletny

15:10-17:45 Poster Session

15:10-16:00 Refreshment

16:00-16:30 Session 9 Inetrnat. Collab. and Initiatives

16:30-17:45 Round Table Conv. Valentino Rigato

18:45-22:30 Meeting and Visit & Social Dinner

Friday 11th

9:00-10:30 Session 10
Nuclear Microprobe Applications: Geology and Environmental Science
Conv: Per Kristiansson

10:30-11:00 Coffee Break

11:00-12:30 Session 11
Nuclear Microprobe Applications: Art and Archaeometry
Conv: Lisa Castelli

12:30-13:00 Closing Remarks

14th International Conference on Nuclear Microprobe Technology and Applications

Sunday 06 July 2014 - Friday 11 July 2014

Palazzo del Bo and Centro Culturale San Gaetano,
Padova, Italy

Programme

Sunday 06 July 2014

Palazzo Moroni: ICNMTA registration and welcome party - (18:30-21:30)

Monday 07 July 2014

Registration - (08:30-09:00)

Opening - Aula Magna (09:00-09:50)

Plenary 1 - Aula Magna (09:50-11:10)

- Convener: Pinheiro, Teresa

time	title	presenter
09:50	**Invited**: Cold beams and focused beams: new technology for new microprobes in the quantum era	JAMIESON, David
10:30	**Invited**: The functionalization of single atoms addressed by ion beam implantation	MEIJER, Jan

Coffee Break - (11:10-11:40)

Plenary 2 - Aula Magna (11:40-13:00)

- Convener: Bisello, Dario

time	title	presenter
11:40	**Invited**: Three dimensional silicon micromachining using a nuclear microprobe	BREESE, Mark
12:20	**Invited**: Radiation effects microscopy in microelectronic devices using a heavy ion nuclear microprobe	VIZKELETHY, Gyorgy

Lunch Break - (13:00-13:50)

Plenary 3 - Aula Magna (14:00-16:00)

- Convener: Grime, Geoffrey

time	title	presenter
14:00	**Invited**: Nuclear Microscopy of biological cells using MeV ions: A critical review.	WATT, Frank
14:40	**Invited**: Applications of nuclear microprobe imaging in neuroscience	ORTEGA, Richard
15:20	**Invited**: Radiobiological and medical research at the ion microprobe SNAKE	GREUBEL, Christoph

Coffee Break - (16:00-16:30)

ICNMTA2014 Group photo - (16:30-16:40)

Plenary 4 - Aula Magna (16:40-18:00)

- Convener: Jaksic, Milko

time	title	presenter
16:40	**Invited**: Ambient pressure molecular concentration mapping using simultaneous MeV-SIMS and PIXE	WEBB, Roger
17:20	**Invited**: The nuclear microprobe in cultural heritage studies: state of the art and challenges	CALLIGARO, Thomas

Tuesday 08 July 2014

Session 1 - Nuclear Microprobe Technology 1 - Auditorium (08:30-10:40)

- Convener: Rajta, Istvan

time	title	presenter
08:30	**Invited**: Development of microbeam technology to expand its applications at TIARA	KAMIYA, Tomihiro
09:00	**Invited**: The first interdisciplinary experiments at the IMP high energy microbeam	DU, Guanghua
09:30	**Invited**: Transmission diamond membrane detector and vacuum window for external microbeams	SKUKAN, Natko
10:00	The high-current mode performance of the proton beam at Ljubljana nuclear microprobe coupled to a multicusp ion source	PELICON, Primoz
10:20	Development of high brightness ion sources with an outlook to sub 10 nm beam spot size for a compact proton beam writing system	LIU, Nannan

Coffee Break - (10:40-11:10)

Session 2 - Nuclear Microprobe Technology 2 - Auditorium (11:10-13:10)

- Convener: Du, Guanghua

time	title	presenter
11:10	**Invited**: Deuterium Microscopy using Deuteron-Deuteron Scattering	REICHART, Patrick
11:40	**Invited**: MeV-SIMS with swift heavy ions at low pressure	MATSUO, Jiro
12:10	Miniature high resolution x-ray spectrometer for ion microprobe	FAZINIC, Stjepko
12:30	The DEFEL pulsed beam facility at INFN-LABEC, Florence: from millimetric to micrometric spatial resolution	FEDI, Mariaelena
12:50	Upgrade of the CSIRO Nuclear Microprobe aimed at high definition PIXE imaging	RYAN, Chris

Lunch Break - (13:10-15:20) (*14:20-15:20 International Committee Meeting*)

Session 3 - Nuclear Microprobe Applications: Biology 1 - Auditorium (15:20-17:00)

- Convener: Ynsa, Maria Dolores

time	title	presenter
15:20	**Invited**: Dual elemental and molecular imaging by MeV SIMS and micro-PIXE on biological tissue samples	OGRINC POTOCNIK, Nina
15:50	**Invited**: Ion beam induced fluorescence imaging in biological systems	BETTIOL, Andrew
16:20	Multimodal Correlative Microscopy: combination of ion beam micro-analysis with complementary microscopy techniques for the study of nanoparticles internalization	BARBERET, Philippe
16:40	The RIKEN microbeam facility: biological application using Fucci cells	PUTTARAKSA, Nitipon

Poster Session with Cheese and Wine - Agorà (17:00-19:30)

Wednesday 09 July 2014

Permanent Poster Exhibition - Agorà

Session 4 - Nuclear Microprobe Applications: Biology 2 - Auditorium (08:30-10:30)

- Convener: Ortega, Richard

time	title	presenter
08:30	**Invited**: Reduced side effects by proton microchannel radiotherapy – studi in a human skin model	DOLLINGER, Gunther
09:00	**Invited**: Automated high throughput analysis of metal atoms in biological macromolecules using Ion Beam Analysis	GRIME, Geoffrey
09:30	Chemical imaging of bone regeneration induced by bioactive glass implants in vivo: a multimodal and quantitative micro-ion beam analysis of mineralization and trace elements at the bone interface	LAO, Jonathan
09:50	In-vivo 3D PIXE-micron-CT imaging of Drosophila using contrast media	MATSUYAMA, Shigeo
10:10	Targeted irradiation of cellular substructures at SNAKE	SIEBENWIRTH, Christian

Coffee Break - (10:30-11:00)

Session 5 - Nuclear Microprobe Applications: Material Science - Auditorium (11:00-12:40)

- Convener: Kamiya, Tomihiro

time	title	presenter
11:00	When NMP meets ICF target	SHEN, Hao
11:20	Boron detection in diamond by the nuclear reaction $^{11}B(p,\gamma)^{8}Be$	YNSA, Maria Dolores
11:40	Assessment of dye distribution in sensitized solar cells by microprobe techniques	BARREIROS, M. Alexandra
12:00	Light element micro-analysis at AIFIRA facility	SORIEUL, Stephanie
12:20	Channeling Contrast Microscopy of GeSn virtual substrates	OSIPOWICZ, Thomas

Conference Excursion to Venice Lagoon - (13:45-23:00)

Thursday 10 July 2014

Permanent Poster Exhibition - Agorà

Session 6 - Nuclear Microprobe Applications: Ion Beam Material Micro-Modification - Auditorium (08:30-10:10)

- Convener: Olivero, Paolo

time	title	presenter
08:30	**Invited**: MeV ion beam mask lithography of parylene-C and parylene-F	WHITLOW, Harry J.
09:00	**Invited**: Ion beam writing on diamond in micrometer scale at the LIPSION nanoprobe	LUHMANN, Tobias
09:30	Deep Ion Beam Lithography in diamond: towards the nanoscale	PICOLLO, Federico
09:50	The use of focused ion beams for the realization of nano-structures in diamond	KALISH, Rafi

Coffee Break - (10:10-10:30)

Session 7 - 5th Proton Beam Writing Workshop - Auditorium (10:30-12:30)

- Conveners: van Kan, Jeroen Anton and Bettiol Andrew

time	title	presenter
10:30	**Invited**: Proton Beam Writing applications into DNA nano fluidics	VAN KAN, Jeroen
11:00	**Invited**: A flexible dielectrophoretic device with high-aspect-ratio pillar arrays fabricated by proton beam writing	NISHIKAWA, Hiroyuki
11:30	Development of embedded Mach-Zehnder optical waveguide structures in PDMS thin films by proton beam writing	KADA, Wataru
11:50	Fabrication of three-dimensional SU-8 microchannels by Proton Beam Writing for microfluidics applications: fluid flow characterisation	ALSHEHRI, Saad
12:10	Proton beam lithography in a new, liquid phase negative resist material	HUSZANK, Robert
12:30	**Panel Discussion:** Nano positioning stages for Nano Probes used in Proton Beam Writing and Nuclear Microscopy	

Lunch Break - (13:00-14:00)

Thursday 10 July 2014 (Cont.)

Session 8 - Nuclear Microprobe Applications: Microelectronics - Auditorium (14:00-15:10)

- Convener: Vizkelethy, Gyorgy

time	title	presenter
14:00	**Invited**: Development of diagnostic method for deep levels in semiconductors using charge induced by heavy ion microbeams	OHSHIMA, Takeshi
14:30	Radiation Hardness of n-type SiC Schottky Diodes	PASTUOVIC, Zeljko
14:50	Ion Electron Emission Microscopy at LNL	SILVESTRIN, Luca

Refreshment - (15:10-16:00)

Session 9 - International collaborations and initiatives - Auditorium (16:00-17:45)

- Convener: Rigato, Valentino

time	title	presenter
16:00	**Invited**: New initiatives to advance Accelerator-based Research & Development	SIMON, Aliz
16:30	**Round Table**: New strategies for fundraising to support projects in micro and nano ion beam technologies	

18:45 - Meeting at Eremitani Civic Museum

18:45-20:45 - Visit to Cappella degli Scrovegni and Eremitani Civic Museum

20:45-22:30 - Social Dinner at Eremitani Civic Museum Magnolia Cloister

Friday 11 July 2014

Session 10 - Nuclear Microprobe Applications: Geology and Environmental Science - Auditorium (09:00-10:30)

- Convener: Kristiansson, Per

time	title	presenter
09:00	**Invited**: High-Speed PIXE: Fast Elemental Analysis with a Colour X-Ray Camera	BUCHRIEGLER, Josef
09:30	Deuterium/Hydrogen microscopy in astrogeological material	ROS, Linus
09:50	Measurement of ratios of oxygen isotopes with pNRA at a microprobe beamline.	BORYSIUK, Maciek
10:10	Elemental compartmentalization changes of marine diatoms as a reporter of biogeochemical cycles	PINHEIRO, Teresa

Coffee Break - (10:30-11:00)

Session 11 - Nuclear Microprobe Applications: Art and Archaeometry - Auditorium (11:00-12:30)

-Convener: Castelli, Lisa

time	title	presenter
11:00	**Invited**: Evidences for an Afghan provenance of lapis lazuli utilized for glyptic by ancient Egyptian combining micro-PIXE and XRF results	RE, Alessandro
11:30	Correlation between ionoluminescence signal and the manufacturing conditions of the clay bodies of ancient tiles	CORREGIDOR, Victoria
11:50	Implementation of ionoluminescence in the IBA micromapping setup of AGLAE facility	PICHON, Laurent
12:10	A comparative study of Etruscan and Tartesic gold jewels by micro-XRF	SCRIVANO, Simona

Closing Remarks - Auditorium (12:30-13:00)

14th International Conference on Nuclear Microprobe Technology and Applications

Sunday 06 July 2014 - Friday 11 July 2014

Palazzo del Bo and Centro Culturale San Gaetano, Padova, Italy

Book of abstracts

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ORAL CONTRIBUTIONS

Plenary1

Cold beams and focused beams: new technology for new microprobes in the quantum era

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In many countries around the world there are emerging programs in a new field of technology that employs quantum mechanical principles in engineered devices. Remarkably, much of the expertise associated with the traditional nuclear microprobe technology and applications can find applications in the new field. Of crucial importance is the capability to engineer single atoms in silicon, diamond and other materials. These materials form the foundations of ultra-sensitive magnetic probes in nano-diamonds for reverse-engineering electromagnetic cellular processes, quantum communication and quantum computing. Over the past two years we have succeeded in implanting P-31 donor atoms into isotopically enriched Si-28 and used integrated nanocircuitry to read out the electron and nuclear spin. We find P-31 nuclear spin coherence times of greater than 30 seconds showing the exceptional promise of this system. The challenge of building large scale devices in this and other systems has triggered an avalanche of new ideas for ultra-fine probes of focused ion beams, scanned nanostencils and ultra-cold ion beams from new types of high brightness ion sources based on ion traps of cold atom arrays. This presentation will review the emerging new technologies of low and high energy ion probes to address the challenges of the quantum revolution.

Plenary1

The functionalization of single atoms addressed by ion beam implantation

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The ultimate goal of nanotechnology engineering in solids is the ability to prepare single atoms as full functional quantum devices in a scalable manner. Quantum effects enable to build extremely powerful sensors and still let the promise of a quantum computer. Devices based on defects in diamond play an increasing role in these fields, taking advantage of some recent technological breakthroughs and of the remarkable overall physical properties of diamond, allowing quantum information processing at room temperature. The nitrogen-vacancy (NV) defect center in diamond has attracted a lot of attention in the last decade. It consists of a substitutional nitrogen atom associated to a carbon vacancy as a first neighbor. Due to unique optical and spin properties, single NV centers are nowadays used as magnetometers or single-photon sources and are promising qubits for quantum computing at room temperature [1-3]. The development of new quantum devices, based on the interaction between the spins associated to single NV centers, requires the ability to create scalable arrays of such centers with high-resolution [4].

The key technology to fabricate and operate these devices is the positioning and addressing of single atoms in a solid with high lateral resolution. Whereas the manipulation of single atoms at the surface is possible since several years [5], the three dimensional addressing of single atoms needs more effort. The combination of surface manipulation and overgrowth is one possibility but it is technically very challenging and possible only for a few atoms like phosphorous in silicon. Ion beam implantation allows nowadays placing single countable atoms inside a given solid with a few nanometers lateral resolution. Focusing systems are more and more optimized aiming to reach a lateral resolution limit. Counting single ions is even more challenging, and the development of a deterministic single ion source based on an ion trap could solve this problem.
The paper discusses the hints and possible solutions for a road map of single ion implantation in the future.

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Plenary2

Three dimensional silicon micromachining using a nuclear microprobe

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At CIBA we have developed a process for fabricating 2 & 3D silicon and porous silicon components. This process is described and its uses in optics, photonics, microfluidics and nanoscale surface patterning are reviewed. The process is based on high-energy ion irradiation, with typically from 50 keV to 1 MeV protons and helium ions. The defects introduced by irradiation alter the hole current flow during subsequent electrochemical anodization, allowing the anodization rate to be slowed or stopped for low/high fluences. For moderate fluences the anodization rate is selectively stopped only at depths corresponding to the high defect density at the end-of-range, allowing three-dimensional silicon machining.
By combining different ion energies and fluences within the same area, we have created free-standing, large-area silicon nanostencils with feature sizes down to 50 nm. This equals to the minimum feature sizes which can be produced in conventional silicon nitride stencils and our capability to make nanostencils in thicker, stress-free crystalline silicon provides a new means of nanoscale patterning of many types of substrates. In another recent development of this process we have shown that low fluence irradiation can actually result in highly porous end-of-range regions. These zones can be selectively removed, leaving buried, hollow lines in porous silicon. After high temperature oxidation the remaining porous silicon forms a solid volume of glass, leaving the hollow micro/nano-scale channel buried beneath the surface. This new method of producing buried nanochannels in glass is being applied to DNA flow studies.

Plenary2

Radiation Effects Microscopy in Microelectronic Devices Using a Heavy Ion Nuclear Microprobe

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In microelectronic devices exposed to energetic radiation electron-hole pairs are created. The movement of these carriers in an electric field will lead to induced current in the electrodes of the device. While this is the basic mechanism of solid-state particle detectors, this current can lead to detrimental effects in microelectronic devices. This current can induce a variety of Single Event Effects (SEEs) that eventually can cause errors in the normal operation of the device or even the failure of the device. These effects can be catastrophic if they occur in microelectronic circuits installed in satellites and spacecraft when the devices are exposed to high level of radiation. Broad beam test can identify the problems and sometimes qualify the devices for radiation hardness but they are not able to pinpoint which element of the device is responsible for the failure of the circuit. Nuclear microprobes are just the right tools to study the mechanism of these SEEs on the microscopic level and help find mitigation techniques and methods.

In this talk we will review the various SEEs and how a nuclear microprobe can be used to study them. Several examples will be given using this technique to study SEEs. Among them a study on Single Event Burnout in Heterojunction Bipolar Transistors and radiation effects investigations in memristors will be presented. A brief review of effects of displacement damage in microelectronic devices will be discussed.

Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

Plenary3

Nuclear Microscopy of biological cells using MeV ions: A critical review.

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Nuclear microscopy has now reached the point where sub-100nm resolutions can be routinely achieved for low current application such as Scanning Transmission Ion Microscopy (1). Since simulations and experimental results have indicated that this resolution is maintained through a whole biological cell, then we now have the potential of imaging the interior of whole cells at unprecedented spatial resolutions (2,3).
This review will concentrate on the work carried out so far on both structural and elemental analysis of biological cells using a focused beam of MeV ions. The talk will include the strengths and weaknesses of nuclear microscopy in this field, and also include a description of competing techniques both for elemental analysis and structural imaging.

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Plenary3

Applications of nuclear microprobe imaging in neuroscience

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Essential trace metals such as Fe, Cu, or Zn are involved in biochemical processes required for the functioning of the nervous system, and as a corollary such metal based functions can be hampered by a dysregulation of metal homeostasis, or by the competition with exogenous neurotoxic metals. For instance most neurodegenerative disorders such as Parkinson's disease, Alzheimer's disease, amyotrophic lateral sclerosis, etc, are associated with an alteration of trace metal homeostasis. Therefore there is an increasing need for trace element imaging in neurobiology and neuropathology research. Such imaging of trace metals distribution in the nervous system is achievable by a variety of analytical tools, including the nuclear microprobe. This review will focus on the recent applications of nuclear microprobe in neuroscience research, with some emphasis on the complementary use of element imaging at different length scales: (i) the organ or tissue level, as illustrated by micro-PIXE imaging of element distributions in brains, (ii) the cellular level, used for example to image element localization in sub-cellular organelles of neurons, and (iii) the protein level, for trace metal quantification in metalloproteins, after protein separation using electrophoresis. The ability to derive the spatial distribution of elements on this diversity of length scales is a key to understanding the mechanisms involved in the etiology of neurodegenerative diseases.

Plenary 3

Radiobiological and medical research at the ion microprobe SNAKE

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High energy ion microprobes allows a precise energy dose deposition, which makes them very attractive for irradiation of living, biological samples and open a wide range of application in radiation biology and neighboring fields, e.g. medicine. Beam size and targeting accuracy in the micron and submicron range allows structured and targeted irradiation on a dimension of the cell nuclei and below. Beyond the laterally very well defined irradiation with single or counted ions the amount of deposited energy is very reproducible. By using ions of different linear energy transfer (LET) the damage per single ion can be varied by several orders of magnitudes.
At the ion mircoprobe SNAKE installed at the Munich tandem accelerator 20 MeV protons are available. In new kinds of experiments these low LET protons (LET in water of 2.65 keV/µm) are focused to imitate and approximate spatial concentrated dose deposition of heavy, high LET ions to gain understanding of RBE effects [1]. In other kinds of experiments the potential of structured irradiation to reduce side effects in radiation therapy is investigated [2]. Structured irradiation with 55 MeV Carbon ions were used to induce well separated ionizing radiation induced foci. Subsequent measurement of foci distances reveals movement not compatible with normal diffusion, which might enhance rejoining probability of DSB end [3]. We cover these and other experiments to give an overview of the radiobiological research at the ion microprobe SNAKE.

Supported by the DFG-Cluster of Excellence 'Munich-Centre for Advanced Photonics' and by BMBF-project 02NUK031A "LET-Verbund"

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Plenary4

Ambient Pressure Molecular Concentration Mapping Using Simultaneous MeV-SIMS and PIXE

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Since the 1970s it has been known MeV heavy ions efficiently sputter insulating targets. An analysis technique, Plasma Desorption Mass Spectrometry (PDMS)[1], resulted in commercial instrumentation which employed Cf fission fragments as a source of MeV heavy ions. The technique was able to desorb large molecular secondary ions (>10kDa) from surfaces, which at the time was not possible with standard secondary ion mass spectrometry (SIMS) techniques using lower keV energies. In the 1980s, however, Laser Desorption techniques (such as Matrix Assisted Laser Desorption (MALDI) were also being demonstrated, which didn't require the presence of radioactive material or a large accelerator. The use of clusters in conventional SIMS also allowed much higher molecular masses (~10kDa) to be imaged. Consequently PDMS was all but forgotten.

There has been a resurgence of interest in the technique recently when it was shown the PDMS (renamed MeV-SIMS) can be used with a focussed beam[2] to produce images with a much higher spatial resolution than is currently possible with laser techniques such as MALDI. It has also been demonstrated that the technique can be performed simultaneously with Heavy Ion PIXE measurements[3]. One of the limitations of SIMS techniques (including MALDI) is the effects of the matrix on the secondary ion yield which can make even relative measurements difficult and isotopic isomers which can even make trace elemental identification difficult, the combination with the PIXE elemental signals removes some of the ambiguity in these measurements.

A further recent development has been the demonstration that the Mass Spectrometry can be performed at pressures above the vapour pressure of water enabling SIMS to be performed on wet samples[4].

The new equipment being commissioned at Surrey is described, which will allow simultaneous MeV-SIMS and PIXE to be collected in full ambient pressures with a micron beam resolution. SIMS spectra and images taken for the first time at fully ambient pressure are presented to demonstrate the potential of this new instrument.

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Plenary4

The nuclear microprobe in cultural heritage studies: state of the art and challenges

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At the crossing of physical sciences and humanities, the characterization of cultural heritage materials addresses central issues of art history, archaeology and conservation science, such as ancient manufacture technologies, raw materials procurement and artefact preservation. Among the wide panel of analytical techniques employed, ion beam analysis plays a prominent role due to an almost unique combination of excellent analytical features in terms of sensitivity and accuracy, joint with a non-destructive and even non-invasive character. The advantage of the nuclear microprobe, with respect to broad beam IBA, stems from its ability to take into account the frequent multi-scaled heterogeneity of materials constituting artworks and archaeological objects. The combination of multiple IBA techniques with a micron-sized beam provides a unique 3-D imaging tool for a large range of elements including light ones. If the full potential of the microprobe can only be realised under vacuum, thus on small items or on samples, external micro-beam setups featuring a degraded lateral resolution (20-50 µm) appear satisfactory for many applications, and fulfil the non-invasive character required for the study of precious artefacts. In addition to the ease of target handling and positioning in front of external micro-beam end-stations, the combination of a fast beam deflection with a mechanical translation of the target enables large scale high resolution mapping up to centimetres.
This communication will present the state of the art of nuclear microprobe applied to Cultural Heritage. The variety of activities in the field conducted at facilities throughout the world will be surveyed, with special focus on the shared transnational access to NMP to study cultural heritage offered in Europe (CHARISMA FP7 program). New developments targeted at such applications, both from instrumental and data processing viewpoints, will be depicted through recent examples conducted with the AGLAE external microprobe. The place of the NMP among other microprobes, ranging from laboratory instruments (Raman and electron probes) to the most advanced ones at large scale facilities (Synchrotron) will be reassessed. The pending challenges of the application to Heritage will be stressed, such as the development of high speed data acquisition system for the fast mapping of areas, and of software tools for mining the massive dataset generated. The study of delicate items such as paintworks constitutes another major defy. In this specific case, the reduction of the damage induced on sensitive constituent materials (lead white, varnish, bindings, etc) by the high fluences required by NMP calls for new damage monitoring and mitigation strategies.

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Development of microbeam technology to expand its applications at TIARA

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R of ion microbeam technology have been progressed at TIARA facility since 1990. In order to expand the variety of ion beam applications of analysing, and fabricating mesoscopic scale system, we have been developing three different types of ion microbeam systems connecting to the 3-MV single-ended accelerator, 3-MV tandem and AVF-Cyclotron (K=110) [1]. For micro-analyses, other than the micro-PIXE system, a micro-PIGE (Particle Induced Gamma-ray Emission) and a micro-IBIL (Ion Beam Induced Luminescence) [2] was also established on the light ion microbeam system, so as to expand the object of analyses to lighter elements, such as lithium, boron or fluoride, and also to the chemical bonding state between elements, respectively. For micro-fabrication, other than the standard PBW, techniques of mask-less patterning on materials without etching processes were studied aiming at development of optical, magnetic or other new types of micro-devices on the light-ion microbeam system [3-5]. For single-ion-hit technique, it was required to monitor every individual ion injection in real time, so as to improve the reliability and also the efficiency of the ion irradiation to living biological cells or semiconductor micro-devices. The method using a highly efficient scintillator and a high sensitivity camera has been developed [6], in addition the study on a method using thin film type particle detector has been started by international collaboration [7]. In this paper, the latest progress of the ion microbeam technology and applications at TIARA are summarised and a future prospect of them is discussed.

Acknowledgements

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The first interdisciplinary experiments at the IMP high energy microbeam

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The high energy beam of tens to hundred MeV/n ions possesses mm-to-cm penetration depth in materials and can be easily extracted into air without significant energy loss and beam scattering. Combination of high energy ions and microbeam technology facilitates the microprobe application to many practical studies in large scale samples. The IMP heavy ion microbeam facility has recently integrated with microscopic positioning and targeting irradiation system. In this work, we introduced the first interdisciplinary experiments performed at the IMP microbeam facility using beam of 80MeV/n carbon ions, including cell irradiation for radiobiology study, mouse irradiation for medical study, board irradiation for information security study and semiconductor irradiation for material science.

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Transmission diamond membrane detector and vacuum window for external microbeams

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In some external microbeam applications the knowledge of a precise number and timestamp of delivered ions is required. Among others, these include single event upsets, timing applications and investigation of radiation effects on living cells. Different triggering techniques have been developed to fulfill this requirement. In our approach, exit window acts as a trigger detector at the same time. Approximately 6μm thick membrane made of low cost optical quality scCVD diamond material was produced at CEA-Saclay. The membrane was mounted to a flange as a vacuum exit window and fully characterized at Zagreb microprobe facility. The negligible intrinsic noise of the device provides an excellent signal-to-noise ratio, even for energetic protons. In addition, the outstanding results of the radiation-hardness test indicate a wider possible range of device applications, including those that involve high currents of charged particles or long exposure to radiation. Production, characterization and applications in time resolved charge transport measurements of the newly developed membrane detector will be presented.

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The high-current mode performance of the proton beam at Ljubljana nuclear microprobe coupled to a multicusp ion source

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One of the key parameters influencing the performance of an ion microprobe is the brightness of the ion source. Traditionally, negative proton beams for injection in tandem accelerators are from von Ardenne type ion sources (duoplasmatrons). This type of ion source provides moderate normalized beam brightness of up to a value of 2 A m-2 rad-2 eV-1 [1,2], one order of magnitude lower compared to best single-ended particle accelerators, where positive hydrogen ion beam is extracted from e.g. an RF ion source located in the terminal of the accelerator.

A high brightness direct negative H- extraction multicusp ion source has been put in use at the Jožef Stefan Institute (JSI) Tandetron accelerator facility. This multicusp ion source and the related Tandetron injection system has been custom developed by High Voltage Engineering Europa. A series of quantitative measurements revealed that an achievable high-energy normalized beam brightness of 14 A m-2 rad-2 eV-1 is available at the microprobe lens, a high-energy proton beam brightness significantly higher than any other value reported on tandem particle accelerators [2]. The brightness value was obtained at only 18% of the total available ion source output current, as the microprobe object slits could not handle the beam power available by the multicusp and Tandetron accelerator.

Recent efforts at JSI were dedicated to improve the matching of the intense H- beams from the multicusp ion source to the properties of the nuclear microprobe beam line. Extraction aperture size was chosen to reduce the ion source output current to ~50 μA H- at optimal plasma conditions. Furthermore, newly home-built water-cooled object slits were installed, allowing intercepting beam power densities of more than one hundred watts/mm2. The Oxford Microbeams OM-150 quadrupole triplet was realigned. By using Focused Ion Beam (FIB) produced nanometer standards with edge irregularities below 20 nm, we were able to evaluate beam profile in detail. The achieved high-current beam mode performance, with proton currents of over 100 pA and the corresponding beam profiles in a sub-micrometer regime, suitable for micro-PIXE with high lateral resolution, is reported.

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Development of high brightness ion sources with an outlook to sub 10 nm beam spot size for a compact proton beam writing system

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In the recent past we have demonstrated the potential of proton beam writing (PBW) as a leading candidate for the next generation lithography technique [1,2]. We are now progressing towards sub-10 nm lithography in nuclear microprobe experiments. To achieve this goal, plans are being rolled out to improve the performance of existing low brightness (~15 – 70 A/m^2SrV) radio frequency (RF) ion source, used for the production of proton beams at CIBA, NUS. This RF ion source has potential to deliver higher brightness [3]. An Ion Source Test Bench (ISTB) set-up has been designed and commissioned in-house to extract the full potential of the existing RF ion-source. In future the ISTB will be used to test a novel ion source design, based on electron-impact gas ionization. Currently the ISTB coupled to an RF ion source has produced nitrogen and helium ions (ion current: ~ 5 μA) and can be operated at about 1-10 kV potential. In this paper we will discuss the integration of a Wien filter and the first brightness measurements in this ISTB.
Meanwhile we are developing a high brightness electron-impact gas ion source (with expected brightness of about 4 to 5 orders of magnitude higher than RF ion source), which will eventually be coupled to ISTB [4]. The idea, with this electron-impact gas ion source, is to create ion beams with small virtual source size of about 100 nm. The first experiments with small gas ionization chambers will be performed inside a Field Emission Scanning Electron Microscope in NUS. Different gases will be introduced into the source (e.g. helium, argon, and later hydrogen). The extracted ion currents for different gases will be studied as function of gas inlet pressure and injecting electron beam energy (200 to 1000 eV). In this paper we will present the experimental results and compare it with theoretical calculations. We will also give an outlook on the feasibility of developing a table top PBW system, capable of delivering 200 keV protons with sub-10 nm beam spot size.

We acknowledge the support from the US air force, Japan office

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Deuterium Microscopy using Deuteron-Deuteron Scattering

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Nearly background free analysis of light elements is possible using the coincidence pattern of elastic scattering reactions. This has been demonstrated for hydrogen analysis, by using proton-proton (pp) scattering [1]. Microscopy with sub-µm-resolution and sensitivity below 0.1 at-ppm for hydrogen analysis becomes possible at MeV energies due to the large elastic scattering cross section enhancement and lowest irradiation damage potential [2].

Using 17 MeV deuterons as the primary beam, we demonstrate Deuterium microscopy by using the deuteron-deuteron (dd) scattering reaction at the SNAKE microprobe. The high deuteron energies are advantageous for the analysis of freestanding samples, which may be easily prepared to a thickness of several tens of micrometers, so that the scattered particles are transmitted through the sample, to the 1 mm thick Si strip detector pairs covering about 2.5 sr solid angle of detection. The cross section for the dd-elastic scattering reaction is about the same as for pp-scattering (~100 mb/sr). The main background due to nuclear reactions is outside of the relevant energy window so that ppm sensitivity is also available for Deuterium microscopy. Deuteron-proton-scattering events give an additional signal for Hydrogen atoms, so the H/D-ratio is monitored in parallel [3].

This coincidence analysis becomes a valuable tool for studies of hydrogen incorporation or dynamic processes using Deuterium marking. The background from natural hydrocarbon or water contamination is eliminated. We present our first measurements on deuterated polyethylene sheets as well as 3D deuterium microscopy of Tungsten foils.

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MeV-SIMS with swift heavy ions at low pressure

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Secondary particle emission with swift heavy ion irradiation provides unique opportunities for further insight on ion collision with matter. Those phenomena are utilized in ion beam analysis techniques, and most of the techniques are for elemental analysis. However, strong demand for chemical analysis is growing these days, because of increased importance of organic and biological materials.
We have proposed molecular imaging technique with secondary molecular ions emitted by swift heavy ion beams (MeV-SIMS) for biological material analysis [1, 2]. In this technique, molecular ion emission from organic or biological molecules is utilized for chemical analysis. In conventional SIMS with keV-energy ion beams, elastic collisions occur between projectiles and atoms in constituent molecules. The collisions break the molecules and produce fragment ions, which makes acquisition of molecular information difficult. In contrast, MeV-energy ion beams excite electrons and enhance the ionization of high-mass molecules, which provide chemical information of molecules. Moreover, swift heavy ions produce secondary molecular ions at much higher yields than monomer ions with energy of a few tens keV.
A molecular imaging system combing with orthogonal acceleration time of flight (oa-ToF) mass spectrometer and electrostatic quadrupole focused lens has be developed to analyse biological samples with high lateral (<5 um) and high mass resolution(>10,000). The molecular distribution of small bio-molecules (up to 1 kDa) was clearly imaged with a lateral resolution of around 5 um, opening a new opportunity in organic and biological material analysis.
Furthermore, high energy ion beams (>MeV) have high transmission capability in matter and allows us to use this beam in low vacuum pressure (1,000 Pa) to analyse volatile sample, such as liquids, waters and wet biological samples. Mixture of fatty acid with various vapour pressure was measured, and accurate composition of the mixture was only measured at the vacuum pressure of 500 Pa.

Recent progress in this technique will be presented and discussed along with its possible applications for biological material analysis.

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Miniature High Resolution X-ray Spectrometer for Ion Microprobe

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Particle induced X-ray emission (PIXE) technique is usually used for trace element characterisation in combination with energy dispersive spectrometers like Si(Li), Ge and/or SDD detectors. Although related PIXE spectra are in principle chemically invariant, small influence of chemical effects could be observed [1]. However, if high resolution crystal X-ray spectrometers are used instead of energy dispersive detectors, related high resolution PIXE spectra clearly show chemical effects that could in principle be used for chemical speciation studies. For this purpose we designed broad beam simple high resolution PIXE spectrometer using flat diffraction crystals. Based on the promising results obtained with that spectrometer [2-5], we constructed miniature wavelength dispersive X-ray (WDX) spectrometer specifically designed for the use with ion micro-beams in order to explore the possibility for performing chemical speciation on microscopic samples utilizing micrometer beam size available at our ion microprobe. During the design stage an X-ray tracing program XTRACE was used to check the usability of a simple flat crystal as dispersive element and to optimize the spectrometer geometry [6,7]. Dedicated vacuum chamber, housing the diffraction crystal, sample holder and CCD x-ray detector, was constructed and positioned behind the main ion microprobe vacuum chamber. Here we present detailed description of the system, selected X-ray images collected by CCD camera obtained from various targets, and describe image processing procedure and algorithm for transferring X-ray images to energy spectra. We discuss advantages and limitations of our downsized spectrometer and the current work on the system developments and applications.

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The DEFEL pulsed beam facility at INFN-LABEC, Florence: from millimetric to micrometric spatial resolution

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The pulsed beamline DEFEL of the 3 MV Tandem accelerator at the INFN-LABEC laboratory in Florence has been significantly upgraded to complement the existing high temporal resolution with the newly implemented high spatial resolution.

At DEFEL beam pulsing is achieved by quickly sweeping the beam through adjustable slits. The fast beam deflection is actuated by a double system of deflecting plates. Particle bunch duration shorter than 1 ns is now routinely obtained. The average number of particles per bunch can be adjusted from less than one to many hundreds. This multiplicity depends, in our system, on the intensity of the initial continuous beam, on the intensity of the deflecting field, on the deflector-to-slit distance and on the slit apertures.

Spatial resolution of the order of 10 micrometers has been achieved by installing four new remotely-controlled x-y slits. Beam characteristics can now be precisely monitored by using our new CMOS sensor-based Beam Profile Monitor.

System stability was tested with pulsed beams of different ion species (H, Li, C, O, Ti, Fe and I). The updated facility opens up the possibility to map the response of semiconductor detectors with high spatial and temporal resolution, over a wide range of generated charge and ionization profiles.

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Upgrade of the CSIRO Nuclear Microprobe aimed at high definition PIXE imaging

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A major re-build of the CSIRO Nuclear Microprobe (NMP) in Melbourne was underway at the time of the 2012 ICNMTA conference, which aimed to exploit the construction of a purpose built Maia fluorescence detector for high definition PIXE imaging. Further refinement of the design of Maia has been necessary and the finalization of this project is very close and may be operational around the time of the ICNMTA in Italy. Hopefully, this paper will show the first data acquired using the new PIXE-Maia system. Notwithstanding Maia status, it will also feature the operation of the new data acquisition system and real-time processor, which can service 36 channels in addition to Maia and orchestrate a range of sample scanning modes on 6 axes with pixel advance based on constraints set on time, beam charge and/or counts. It will also outline features of the new Maia 384 element detector array [1,2], recently upgraded for better energy resolution, light element detection and life in the NMP vacuum chamber, as well as aspects of the reconstruction of the NMP built to accommodate Maia and precision in-vacuum stages, coaxial zoom microscope, and conventional X-ray and particle detectors. It will draw on the experience of Maia integrated into the X-ray Fluorescence Microscopy (XFM) beamline at the Australian Synchrotron [3], which can collect high definition SXRF element images of up to ~100M pixels using Maia [4]. Recent SXRF technique development of immediate application in PIXE mode includes the mapping of the depth of rare precious metal particles in geological samples, which exploits the deep penetration of X-rays or MeV protons, sample self-absorption of characteristic X-rays and the wide range of take-off angles to the Maia detector array elements to provide an imaging depth contrast and quantitative measures of individual particle depths [5].

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Dual elemental and molecular imaging by MeV SIMS and micro-PIXE on biological tissue samples

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In the last decade, there has been a growing interest in the elemental and biomolecular distribution on the tissue surface. These species are localized to a specific region of the tissue and play an important role in biochemical processes. Therefore, it is important to investigate the spatial distribution, structure and function of these biological species [1]. The current surface characterization techniques provide unique but limited information on lateraly-resolved elemental and molecular distributions. In order to overcome these limitations, we are using a dual imaging technique by combining micro-PIXE [2] and MeV SIMS [3,4] capable of providing us with the elemental and molecular distribution on the same tissue samples.

The MeV SIMS measurements are performed by a 5.8 MeV 35Cl6+ primary ion beam focused to a dimension of 20 µm x 20 µm. The acquired molecular maps are then correlated with the sequentially measured elemental maps by micro-PIXE, measured at the matched sample region. Micro-PIXE maps are acquired by a 3 MeV proton beam and the current lateral resolution limit of 700 nm x 700 nm. The current status of the dual imaging technique as well as the tissue sample preparation protocols will be presented at various plant case studies, with an emphasis on the leaves of Al-treated tea plants (Camellia sinensis). The correlated elemental and molecular distributions on other tissue types, including animal brain and human hair, will also be presented.

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Ion Beam Induced Fluorescence Imaging in Biological Systems

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Imaging fluorescence generated by MeV ions in biological systems such as cells and tissue sections requires a high resolution beam (< 100 nm), a sensitive detection system and a fluorescent probe that has a high quantum efficiency and low bleaching rate. For cutting edge applications in bioimaging, the fluorescence imaging technique needs to break the optical diffraction limit allowing for sub-cellular structure to be visualized, leading to a better understanding of cellular function. In a nuclear microprobe this resolution requirement can be readily achieved utilizing low beam current techniques such as Scanning Transmission Ion Microscopy (STIM). In recent times, we have been able to extend this capability to fluorescence imaging through the development of a new high efficiency fluorescence detection system [These proceedings].

Many of the fluorescent probes that have been developed for high-resolution fluorescence techniques such as confocal microscopy have been optimized for laser excitation. These probes are not necessarily useful for ion or electron excitation. In addition, biological systems typically have some degree of auto-fluorescence which most of the time results in an unwanted background signal that degrades the contrast of the image.

This paper discusses how we have addressed these issues for ion beam induced fluorescence imaging. We will also review previous work on fluorescence imaging in biological systems and show the current state-of-the-art in super-resolution fluorescence microscopy using focused MeV ion beams at the Centre for Ion Beam Application (CIBA), NUS Singapore.

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Multimodal Correlative Microscopy: combination of ion beam micro-analysis with complementary microscopy techniques for the study of nanoparticles internalization

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Assessing the cellular response to external factors (exogeneous compounds, nanoparticles…) often require the use of microcroscopy techniques to visualize the internalization of these factors in living cells. In many studies, imaging using different probes is performed to provide complementary data by combining the advantages of each individual technique. This is particularly true in the frame of nanoparticle (NPs) internalization studies at the level of single cells. One of the main actual challenges is to track these NPs in single cells and assess the number of NPs internalized per cell. In the frame of such studies, we have developped a multimodal correlative microscopy (MCM) approach to detect, track, and quantify NPs in single cells. This MCM is based on the complementarity of three individual techniques: fluorescence microscopy (FM), scanning electron microscopy (SEM), and ion microbeam analysis (IBA) coherently focused on the same targeted individual cell cultured and maintained on a specifically designed sample holder. In this approach, a unique preparation protocol is applied and allows multimodal analysis of the same cell at different microscopes/microprobes. The correlated data obtained by FM and IBA open a field toward toxicological study on native NPs. Complementary results from SEM and IBA provide both surface and in depth information on the cell reaction as well as on the exact localization of NPs. Finally, IBA contributes to high-sensitivity and in situ quantification of all the present chemical elements including NPs in a single cell.

In addition to these methodological developments, we have completed the equipment of the CENBG nanobeam line to allow i) the use of two X-ray detectors combined to improve the detection solid angle and thus reduce the time required for analysis, ii) secondary electron imaging using our proton microbeam, iii) improve the scanning capabilities to increase the number of pixels per image.

The latest developments performed on the nanobeam as well as the application of MCM to nanoparticle quantification in single cells will be presented.

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The RIKEN microbeam facility: biological application using Fucci cells

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A microbeam focused by a tapered glass capillary has been developed with a 1.7 MV Pelletron accelerator at RIKEN [1]. A few micrometer thick polymer window was inserted at the end of the capillary tip for evacuating, which allows it to be immersed in a liquid medium. In our facility, three bending magnets downstream of the beamline are used to deviate the trajectories of MeV ions (e.g. protons or helium ions) into the capillary holder at an angle of 45 degree to the vertical axis, above the targeted stage. This enables us to employ a standard 35 mm cell culture dish on an inverted microscope, which can visualize the capillary and cells during and post irradiation.

As one example for biological experiments at our setup, we present the irradiation of HeLa cells expressing fluorescent ubiquitination-based cell cycle indicator (Fucci) system [2]. By this, the fluorescent emission of the nucleus changes between red and green depending on the phase of the cell cycle. Thus, the influence of the cell cycle induced by the irradiation, or the phase in which the cell dies, can be directly visualized by the fluorescent color changes. Furthermore, it might be possible to observe the effects in the neighboring unirradiated cells. To examine this, we irradiated a nucleus of a single Fucci cell inside a cell colony with various doses of 1 MeV protons. After irradiation, time-lapse microscopy was used to monitor the irradiated and non-irradiated cells for about 48 hours, covering two cell cycles. Here, we will show the results of this investigation based on the RIKEN microbeam.

Acknowledgement

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Reduced side effects by proton microchannel radiotherapy – studi in a human skin model

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We propose a novel strategy to reduce the known side effects of radiotherapy by using proton microchannel irradiation. The goal is to minimize the risk of normal tissue damage by microchannel irradiation, while preserving local tumor control through a homogeneous irradiation of the tumor that is achieved because of beam widening with increasing track length. In order to prove the hypothesis of reduced side effects in normal tissue through microchannel proton irradiation, we report on a comparative study of microchannel and broad beam irradiation of artificial skin tissue.

20 MeV protons were administered to human skin models (EpidermFTTM) in 10 to 180 µm wide irradiation channels at the proton microprobe SNAKE on a quadratic raster with distances of 500 to 1800 µm between each channel (center-to-center) applying an average dose of 2 Gy. For comparison, other samples were irradiated homogeneously by protons at the same average dose. Widened channels as in deeper lying tissues were investigated in skin tissues as well.

Normal tissue viability was significantly enhanced after microchannel proton irradiation compared to homogenous irradiation (~80% vs. ~40% viability (MTT)). Levels of inflammatory markers, such as cytokines and chemokines, were significantly lower in the supernatant of the human skin tissue after microchannel irradiation than after homogeneous irradiation. Furthermore, genetic damage as determined by the measurement of micronuclei in keratinocytes was also significantly reduced after microchannel irradiation compared to homogeneous irradiation (0.015-0.030 micronuclei per divided cell for microchannel vs. 0.070 ± 0.007 MN/divided cell for homogeneous irradiation).

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Automated high throughput analysis of metal atoms in biological macromolecules using Ion Beam Analysis

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As we have described previously [1,2], microbeam PIXE combined with simultaneous RBS analysis is the only accurate method easily available for identifying and quantifying the small number of metal atoms commonly present in large biological molecules such as proteins.

This method has been in routine use at the Surrey Ion Beam Centre for several years, but is restricted in its range of applications because preparing, loading, positioning and analysing single samples by hand is time consuming and it is difficult to analyse more than ten samples in a typical working day. Many bio-science experiments are now designed on a 'high throughput' model where many samples are prepared in different ways or exposed to different reagents and the results analysed using automated methods such as optical fluorescence. Although metal atom determination using microPIXE would be desirable for many of these sample sets, analysing such large sample numbers by hand is totally impractical.

We have now developed a high-throughput method for analysing large sample sets which has significantly extended the application of the technique. The samples are deposited as arrays of spots using a non-contact ink-jet printer. These are then analysed sequentially using pattern recognition techniques to localise the spots under the beam, real-time spectrum evaluation to determine the end-point of the analysis and batch spectrum processing to determine the concentrations. This paper describes the method and presents some illustrative results.

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Chemical imaging of bone regeneration induced by bioactive glass implants in vivo: a multimodal and quantitative micro-ion beam analysis of mineralization and trace elements at the bone interface

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This communication will focus on the contribution of nuclear microprobes to the highly sensitive elemental analysis of bone tissues, in the context of evaluating the efficiency of bone regeneration induced by a bone substitute. Indeed the mineral composition of bone can give key clues on the maturity and the quality of the bone formed, the content in trace elements being especially meaningful.

Among all bone substitutes, bioactive glasses are particularly interesting due to their high biomineralization capabilities. When implanted, bioactive glasses endure major changes as a result of both the chemical reactivity of the glass and osteointegration, which lead to quick bonding of the implanted glass to bone with simultaneous stimulation of new bone growth. Here the presence of trace elements like zinc can indicate the maturity of bone tissues formed, as Zn is recognized as a co-enzymatic factor and is an essential component of a large number of enzymes.

Moreover recent advances in the field are the development of bioactive glasses able to release osteoinductive ions directly onto the site of implantation. The osteoinductive ions locally delivered help increase osteogenesis. Of special interest is the delivery of strontium (Sr) ions, since Sr has marked stimulatory effects onto bone cells resulting in strengthening of bone, stimulation of bone formation and decrease in bone resorption. Other trace elements of interest are Si and Mg due to their promotion of bone formation and cellular adhesion.

Studying bone traces in vivo is thus of high interest but calls for an extremely sensitive technique. An excellent spatial resolution is also required for characterizing the bone/bioactive glass interface at the micrometer scale. We propose here an original approach based on a multimodal analysis of bioactive glasses implanted in vivo. Histological studies of the bone/bioactive glass interface are coupled to micro-PIXE (Particle-Induced X-ray Emission) analysis for quantitative chemical imaging and a complete micro-analysis of the interface with a special focus on trace elements.

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In-vivo 3D PIXE-micron-CT imaging of Drosophila using contrast media

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For the research field of biology and medicine, it is very useful to develop the technology in which an interior of small animals can be observed in-vivo and with a high resolution of several micron meters. For this purpose, we have developed a 3D CT with micrometer resolution. The system comprises a point X-ray source, a rotating sample stage and a high-speed X-ray CCD camera. A microbeam system was used as a monoenergetic μ-X-ray source by bombarding pure metals. We called the system PIXE-micron-CT. Characteristic X-rays from a pure metal target bombarded with a few MeV proton micro-beams can be used as a point source of quasi-monochromatic X-rays.

We have applied PIXE-micron-CT to observe organs in a body of a living Drosophila, which is popularly used in various fundamental studies, such as gene research. The internal structure of living Drosophila was obtained in the different growth stages. The CT images of living Drosophila were much different from those of dead or formalin fixed ones [1]. Although the organs in thorax are clearly recognized in living Drosophila, the digestive organs in abdominal were not clearly seen except the rectum and the Malpighian tubule.

In this study, we tried to photograph digestive organs inside abdominal part, emphatically by applying radiographical contrast agent to Drosophila. Considering harmlessness and linear attenuation coefficient compared with physical tissues, BaSO4 compound was introduced as contrast agent. Iron was selected as an X-ray target instead of titanium which was used in the previous study considering its high linear attenuation coefficient to BaSO4. The 3D ventriculus and rectum in abdominal of living Drosophila images were obtained. The ventriculus images were never obtained without using radiographical contrast agent. The PIXE-micron-CT can provide in-vivo 3D-CT images that reflect correctly the structures of each living organ and is expected to be very useful in biological research.

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Targeted irradiation of cellular substructures at SNAKE

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With the development of a highly precise targeted irradiation at the ion microbeam SNAKE, installed at the Munich 14 MV tandem accelerator, cellular response after irradiation of subcellular or even sub nuclear structures can be studied. Based on the live cell setup at SNAKE [1,2] it provides a sub micrometer single ion irradiation facility in combination with a high-resolution optical microscopy. To reduce systematic errors, for beam spot verification and the target recognition the same light path is used. The optical microscope and the beam delivering system are controlled by a self-developed software which optional integrates the CellProfiler software [3] for automated target recognition.

Targeting accuracy was determined by 55 MeV carbon ion irradiation of fluorescence labeled cells carrying red chromatin domains as targets and GFP-tagged MDC1 protein for damage response. Analysis of hit positions relative to the target in 140 irradiated red chromatin cells shows a systematic shift of 1.1 µm and a random distribution relative to the mean of about 3 µm fwhm.

First application of the targeted irradiation is the investigation of the accumulation of GFP-tagged Ku70 and XRCC4 protein, which are reported to form foci in the cell nucleus after UV Laser irradiation [4]. This accumulation is not observed after sparsely ionizing radiation or single carbon ion irradiation. In the targeted irradiation mode specific irradiation patterns with an exact number of 1-1000 carbon ions per point are applied less than 5 s. Hereby in U2OS cells expressing XRCC4-GFP after 100 carbon ions per point foci were formed after 13 s, in stably transfected HT1080 cells the Ku70-GFP protein with 300 carbon ions per point after 4 s. These results show that an enormous amount of DNA lesions is responsible for the foci formation of Ku70 and XRCC4 after UV Laser irradiation.

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When NMP meets ICF target

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The Inertial Confinement Fusion (ICF) program is investigating the conditions to achieve controlled thermonuclear fusion. ICF target fabrication quality is as vital as driving laser to ensure that energy deposition from the lasers results in uniform compression. Therefore Targets must be characterized for size, concentricity, surface roughness and distribution of dopant concentration, layer thickness and fuel pressure as well. An ideal analysis method would be able to obtain such information simultaneously and non-destructively.
Nuclear Microprobe (NMP) could be the one. Typical ICF targets are spheres less than a millimeter in diameter, several tenth micrometers in thickness. With the ability to focus MeV ion beams down to micron spot sizes, NMP with particle-induced X-ray Emission (PIXE), Rutherford backscattering (RBS), Scanning Transmission ion microscopy (STIM) and Elastic Recoil Detection Analysis (ERDA) enables us to image the shape, density and depth profile, to map trace elements like S, Br, Au, Cu, etc. of target, and Hydrogen isotopes in the target. Due to the importance of ICF program, precision and accuracy should be extremely carefully considered. Nevertheless the technique of NMP is a powerful method, which is proving to be very useful in the field of ICF target characterization.

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Boron detection in diamond by the nuclear reaction $^{11}B(p,\alpha)^{8}Be$

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The diamond is an especially attractive material because of its gemological value as well as its unique mechanical, chemical and physical properties. One of these properties is that diamond is an electrically semiconducting material at practically any desired value when doped with boron (p-type). This property makes it possible to use diamond for multiple industrial and technological applications.

Although boron doped p-type diamond exists in nature, the boron can be incorporated into pure diamond by different techniques such as implantation. Generally, the typical energies used to dope diamond by ionic implantation are about 200 keV though some implantations have also been achieved with high energies. The CMAM internal microbeam line has demonstrated to be a powerful setup to implant boron with high energies. An 8 MeV boron beam with a size of about 5 x 5 μm2 and a beam current higher than 500 pA has been implanted controlling the beam position and fluence at all irradiated points. The subsequent mapping of the implanted boron in diamond has been achieved using the strong and broad nuclear reaction $^{11}B(p,\alpha)^{8}Be$ at Ep = 660 keV. This reaction has a high Q-value (8.59 MeV for α0 and 5.68 MeV for α1) and thus is almost interference free. The sensitivity of the technique and the best experimental conditions are studied in this work.

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Assessment of dye distribution in sensitized solar cells by microprobe techniques

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Dye sensitized solar cells (DSC's) have received considerable attention once this technology offers economic and environmental advantages over conventional photovoltaic (PV) devices. A DSC photoanode typically consists of a nanocrystalline porous TiO2 film, endowed with a large adsorptive surface area. Dye molecules that capture photons during device operation are attached to the film nanoparticles. The effective loading of the dye in the TiO2 electrode is of paramount relevance for controlling and optimizing solar cell parameters. In particular, the cell short-circuit current density (Jsc) is directly proportional to the light harvesting ability of the photoanode, which in turn is strictly dependent on the dye concentration on the TiO2 adsorptive surface. In addition, the dye adsorption behavior affects the cell open circuit voltage (Voc).

Relatively few methods are known today for quantitative evaluation of the total dye adsorbed in the film. In this context microprobe techniques come out as suitable tools to evaluate the dye distribution and dye depth profile in sensitized films.

Electron Probe Microanalysis (EPMA) and Ion Beam Analytical (IBA) techniques using a micro-ion beam were used to quantify and to study the distribution of the ruthenium organometallic (N719) dye in TiO2 films, making use of their different penetration depth and beam sizes. Two different types of films were prepared and sensitized, mesoporous nanoparticles and 1D nanostructured TiO2 films (about 4 µm thickness).

The high sensitive analytical techniques used allowed to assess dye surface distribution and depth profile, by means of Ru signal, despite the low concentration of this element. X-ray mapping by EPMA/WDS technique made possible to visualise the dye distribution in sample cross-section. PIXE maps of Ru and Ti indicated an homogeneous surface distribution. The assessment of ruthenium depth profile by RBS showed that some films have homogeneous Ru depth distribution while others presented up to half of the Ru concentration in the top layer (2 µm thickness) when compared to the lower one.

Dye load evaluation in different TiO2 films by two different techniques (µPIXE and EPMA/WDS) provided similar results of Ru/Ti.

The assessment of the dye distribution and quantification across an oxide semiconductor film by microprobe techniques can lead to a better understanding of the device performance.

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Light element micro-analysis at AIFIRA facility

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The AIFIRA facility (Applications Interdisciplinaires des Faisceaux d'Ions en Région Aquitaine) in operation at CENBG (Centre d'Etudes Nucléaires de Bordeaux-Gradignan) may be a reliable tool adapted to a wide range of scientific fields. Indeed, ion beam analysis and imaging techniques, material characterization and irradiation (fast neutrons and charged particles), all are carried out at AIFIRA to conduct an interdisciplinary research program. The main activity of the platform is driven by the microbeam dedicated to the analysis, imaging, and characterization of biomedical samples and advanced materials. Its last version was recently presented to the community [1] [2]. Since, the microbeam continuously underwent developments and improvement especially of the target chamber. This constant evolution is due to the great flexibility offers by the beam line, which potentially allows a wide range of applications (chemical analysis at the sub-cellular scale, micro-tomography, nuclear reaction analysis, proton beam writing, etc.).
A recent development is about Elastic Recoil Detection Analysis (ERDA), which is known as one of the ultimate technique for the quantification of the elusive Hydrogen. Furthermore, the long working distance quadrupoles configuration can focus deuterons, as well as protons and alpha particles in the MeV energy range. Taking all of those specificities into consideration, it is possible to perform quantitative analysis of light elements at sub-micrometre scale through the use of ERDA, NRA and PIGE techniques.

The communication will focus on the analysis of light elements through a description of the available set-ups on the target chamber. We will also illustrate the performances of the microbeam with selected examples, for instance the analysis of 3D bioactive scaffolds with ERDA and NRA

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Channeling contrast microscopy of GeSn virtual substrates

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Recently, interest in the semiconductor research community has switched, to some degree, from Si based devices to materials with higher carrier mobilities, such as germanium, germanium-tin, and compound semiconductors. This is also driven by the need to extend the functionalities of silicon IC technology to enable optical interconnect systems, i.e. find a direct bandgap material that can be integrated with Si process technology. Germanium-tin (GeSn) alloys promise to deliver on both these aspects; they may become technologically important materials.
Here we report on Channeling Contrast Microscopy GeSn thin films grown by Molecular Beam Epitaxy (MBE) at temperatures of 170-150°C. This technique allows epitaxial growth of thin films with sub monolayer precision. The films were grown with Sn concentrations up to 16% and display excellent crystal quality.
Broad beam conventional and high resolution RBS was used to determine the depth profiles, and angular scans around the (100) axis were carried out to assess crystal and interface quality. Channeling Contrast Microscopy (CCM) at sub-micron resolutions in an optimized detector geometry was used to acquire depth resolved images of lateral in homogeneities, surface morphologies and lattice tilts, in order to optimize the growth methodology.

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MeV ion beam mask lithography of parylene-C and parylene-F

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Lithographic patterning with 16O+ ions has been demonstrated by us, and others in polytetrafluoroethylene (PTFE). Motivated by the molecular level similarity of PTFE with parylene-C and parylene-F; we have investigated the possibility of using 0.6–2 MeV oxygen ions to pattern these polymers using aperture mask lithography for prosthetic applications in inner-ear surgery.

Parylene-C and –F could be patterned in a similar way to that reported for PTFE. However, the removal per unit fluence for 1 MeV 16O+ was ~5% that for PTFE. Both films deposited on glass and silicon substrates and self-supporting films of Parylene-C could be patterned. We also investigated irradiation in a atmosphere of up to 10-3 mbar oxygen and observed any increase in removal rate for the polymers in question. Increasing the energy of the 16O+ beam from 600 keV to 2 MeV increased the removal rate of parylene-C ~1.5×.

Biocompatibility tests using murin Spiral Ganglia cells (SG) showed that there was no significant difference in SG cell proliferation between irradiated and unirradiated areas. Moreover, laminin and poly-L-lysine applied after ion irradiation completely dominate the cell attachment.

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Ion beam writing on diamond in micrometer scale at the LIPSION nanoprobe

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Diamond has a broad field of applications due to their extraordinary properties like the pronounced thermal conduction and hardness. In mineral science the hardness is used at high pressure laboratories in diamond anvil cells (DACs). Here an intrinsic heater could be useful to generate high temperatures besides the high pressure. Furthermore diamond is one of the most prominent candidates for the realization of a quantum computer at room temperature via nitrogen vacancy (NV) color centres, where it is essential to control the charge state of the NV centres e.g. by electric fields [1,2].
The construction of micrometer scale intrinsic electric wires, coils or heaters in diamond by ion beam induced graphitization is a method to realize such devices directly under the surface [3].
For this reason diamond single crystals (optical grade, Element Six) were irradiated by 1.9 MeV focussed He+ particles at the LIPSION nanoprobe with a spot size of 3 µm. Graphitization was obtained for fluences between 1016 cm-2 to 1018 cm-2 and subsequently annealing [4]. As a consequence of the relatively high ion energy the created structures are located about 3 µm deep under the diamond surface and protected to their surroundings.
The scope of this study is the optimization and characterization of such structures for implementation in diamond devices without using a mask e.g. as micrometer-size heater for NV centre creation by build-in annealing or wires for charge state control of the fabricated centres. Different feed-trough processes between graphite layer and diamond surface for electrical contacts were investigated. First measurements of resistance and surface resistivity as well as ellipsometry were used.

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Deep Ion Beam Lithography in diamond: towards the nanoscale

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In the present work we report about an innovative ion beam fabrication technique whereby sub-superficial graphitic nanostructures can be created in diamond.
Ion beam implantation is an effective tool to modify diamond. In particular ion-damaged diamond can be converted to graphite, which is electrically conductive and displays an higher reactivity to chemical etching with respect to the chemically inert pristine structure. This phase transition occurs in sub-superficial layers because it is due to the peculiar damage profile of high energy (MeV) ions, which mostly damage the target material at their end of range.
In the last years, micro beam lines were employed in diamond ion beam implantation, thus opening the way to the fabrication of micro-structures in this material. Femtosecond laser can be also employed to induce the diamond graphitization both on the surface and in the bulk. The mentioned techniques are versatile tools for diamond modification but offer a spatial resolution limited to some micrometers.

The presented strategy consists in the combination of ion beam lithography and focused ion beam (FIB) milling. FIB milling plays a key role in the fabrication process, indeed it is employed to create the masks through which ions are implanted in diamond. The masks are realized on thin metal layers (<10 μm) deposited directly on the crystal in order to reach nanometric resolution (no mask banding, no scattering, high thermal dissipation, etc.). Moreover, masks with variable thickness profiles are feasible tuning opportunely the FIB ion milling doses; these masks allow to modulate the ions penetration range for the creation of structures (i.e. conductive paths, fluidic channels, etc.) at the desired variable depths.

We will present the electrical and structural characterization of the sub-superficial graphitic nanochannels.
Moreover, a prototype of single cell biosensors realized with the above described technique will be shown. The biosensor has 16 electrodes converging inside a circular area of 20 μm diameter (typical neuroendocrine cells size) for simultaneous recording of amperometric signals.

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The use of focused ion beams for the realization of nano-structures in diamond

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Diamond is a unique material with outstanding electrical, optical, chemical and mechanical properties. Recently, the negative nitrogen-vacancy color center (NV-) in diamond with its specific luminescence properties has been identifies as being the most suitable candidate for realization of scalable solid-state quantum computing systems (qubits) operating at room temperature. The quantum information in such devices is carried by the photons, emitted from the luminescing center. These, however, must be propagated and manipulated to permit their application in a large variety of most promising future devices.

In order to enhance the photon-atom coupling, specific optical structures must be realized in the diamond that contains the emitting NV center. For example, optical cavities in which a qubit (NV-) is located, and the cavities are interconnected via waveguides. All these should preferably be realized in a single diamond slab. The fabrication and processing steps commonly used for implementation of photonic crystals in semiconductors are inapplicable to diamond due to its extreme chemical and mechanical properties. This makes Focused-Ion-Beam (FIB) a very attractive tool for realization of photonic structures, such as membranes, cavities, waveguides and mirrors in diamond.

Here we describe how the use of low energy (30 keV), well focused Ga ions enables the fabrication of various photonic devices suitable for enhancing and guiding the quantum information carrying photons from the NV center in diamond. Several photonic devices, such as planar photonic crystals in diamond membranes and triangular cross sectional nano-beams were realized by the use of 30 keV Ga ions. Their design, fabrication and optical characterizations are described. The inherent problem of shallowly implanted Ga+ ions following FIB processing has detrimental influence on diamond optical properties. We show how post processing techniques, relying on H plasma treatment and wet chemistry, can be used to remove the Ga and graphite containing outermost layers and to finely tune the dimensions of the photonic devices on the nm scale.

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Proton Beam Writing applications into DNA nanofluidics

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Proton beam writing (PBW) is an ideal technique to fabricate lab-on-chip devices with features down to the nano scale. PBW exhibits low proximity effects coupled with the straight trajectory and even energy deposition its path will results in sidewall smoothness of a few nm RThe high penetration depth of the proton beam enables the production of high aspect ratio, high density 3D nano structures with smooth sidewalls, ideal for high quality mold production for nano-imprint-lithography (NIL) applications. In PBW experiments proton beams can now be focused down to 13 x 29 nm^2, revolutionizing applications of MeV proton beams [1] e.g. high aspect ratio lithography down to 19 nm in HSQ.
HSQ, PMMA and SU-8 are all suitable resist material for PBW with a high resolution and smooth sidewall [2]. The comparison of these three photoresist under the same fabrication condition has never been done. Here we will compare the resolution of these three different resist at sub 100 nm level with the optimized proton dose andthesamebeamsize.
The improved PBW performance opens up new ways of mold production for NIL applications. Using HSQ resist as a mold material an easy method is introduced allowing fast replication of complicated nanofluidic lab on chip devices featuring cross-sections down to 60 nm in polydimethylsiloxane (PDMS). These HSQ molds can be used more than 200 times to replicate nanofluidic devices capable of handling single DNA molecules [3]. A novel mold design will be presented which allows the manipulation of DNA molecules inside PDMS nanochannels as well as large scale DNA genome mapping.
PDMS replication is not compatible with sub 50 nm lab on chip fabrication. To achieve polymer nanofluidic circuits down to 19 nm, PBW fabricated resist structures will be replicated in OrmoStampTM (Micro Resist Technology GmbH) to form hard stamps which can be used in thermal NIL. In our experiments, different types of resist will be tested on the compatibility with Ormostamp replication. Initial test with Ormostamp molds are used in NIL.

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Session7- Proton BeamWritingWorkshop/114

A flexible dielectrophoretic device with high-aspect-ratio pillar arrays fabricated by proton beam writing

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Dielectrophoresis (DEP) has been widely used as a technique for manipulation or trapping of micro particles and microbes such as E.coli for prevention of food poisoning diseases. We have previously reported the introduction of high-aspect-pillar arrays into DEP devices for improved trapping capability by modulating the electric field to enhance the DEP force to attract micro particles [1, 2].
In this study, we have developed flexible DEP devices on a plastic film equipped with high-aspect-pillar arrays by proton beam writing (PBW) for better convenience of the DEP device. The PBW was performed with a proton beam focused to 1.0 micro meter at beam energy of 1.0-1.7MeV using beamlines at the Center for Flexible System Integration, SIT and TIARA, JAEA Takasaki. The high-aspect-ratio pillar arrays were written and developed with a SU-8 resist layer on a flexible polyethylene terephthalate (PET) film. Improved convenience and trapping capability of the flexible DEP devices on PET films as an electric micro-filter will be demonstrated. Application of cleaning and drying techniques using supercritical CO2 (40 degree C and at 14 MPa) to avoid the collapse of the pillar arrays due to capillary force will be also discussed.

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Session7 - Proton Beam Writing Workshop/128

Development of embedded Mach-Zehnder optical waveguide structures in PDMS thin films by proton beam writing

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Proton beam writing (PBW) technique has capability to fabricate micro-optical devices which will be utilized in high-speed optical communications. In the previous study, we have demonstrated the fabrication of Mach-Zehnder (MZ) optical waveguide structures in poly-methyl-methacrylate (PMMA) [1]. The optical switch based on this MZ optical waveguide was successfully developed, however, results indicated that enhancement in optical transmittance of the MZ waveguide structures is required for the actual applications. On the other hand, interests on optical properties of poly-dimethyl-siloxane (PDMS) has been emerged as a potential candidate for such micro-optical devices [2].

In this study, we have demonstrated embedded MZ optical waveguide fabrication on PDMS by precise control of scanning proton microbeam probe and mechanical sample-stage. PDMS films (Toray Dow Corning SYLGARD184) were formed on a silicon wafer (40 mm× 20 mm × 0.5 mm) by spin-coat process with thickness of approximately 30 μm. 750 keV proton microbeam probe was employed to fabricate embedded optical waveguide structure in the PDMS films with the depth of 18 μm. Embedded MZ waveguides were fabricated with different beam fluence from 40 nC/mm² to 100 nC/mm² and evaluated by 1.55 μm fibre-laser irradiation with different light propagation conditions. The results indicated that embedded structure in PDMS films have successfully equipped single-mode light propagations.

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Fabrication of Three-dimensional SU-8 Microchannels by Proton Beam Writing for Microfluidics applications: Fluid Flow Characterisation

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The proton beam writing (PBW) technique has significant advantages over other lithography techniques in the fabrication of three-dimensional structures with a high aspect ratio, straight and smooth sidewalls. These features potentially facilitate the rapid prototyping of microfluidic systems with some crucial improvements in the quality. A network of polymeric buried channels was fabricated in SU-8 as a part of project to develop functional microfluidic networks. Using protons with energies between 2.5 MeV and 0.75 MeV buried channels have been fabricated with minimum feature size of around 1 µm and depths of 40 µm and their properties have been evaluated. Straight-walled channels with surface roughness of around 2nm for the sidewalls of the channels were measured. The exposure regime, post processing and examples of functional networks fabricated using PBW are described. In addition, the paper will present theoretical modelling using COMSOL multiphysics developed to predict the flow pattern properties of fluid in the SU-8 microchannels structured by PBW. This modelling focused on the investigation of the impact on the functionality of microfluidics system of enhancements in the microchannel texture and structural engineering. The results are compared with some microchannels in literature fabricated by conventional lithography techniques.

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Proton beam lithography in a new, liquid phase negative resist material

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To extend the applications of the proton beam writing (PBW) technique it is important to explore potential new resists materials. Poly(dimethyl-siloxane) (PDMS) is a promising material for many applications nowadays because of its advantageous properties. In cross-linked, elastomeric form it has been widely used to fabricate Micro-Electro-Mechanical Systems (MEMS), microfluidic devices, micro-stamps by moulding techniques [1,2], or in micro optical devices using developer-less PBW method [3]. However, the PDMS can also be crosslinked directly induced by ion beam irradiation [4].

In this work we investigated the applicability of liquid PDMS polymer as a negative resist material for direct proton beam writing technique. We irradiated the polymer in liquid phase, spin-coated on different substrate materials creating various microstructures. PDMS pre-polymer was crosslinked just by PBW. As the crosslinking process increases, the irradiated area becomes more solid. The rate of the solidification strongly depends on the deposited ion dose. The effects of fluence, beam current, substrate type and developer solvent was investigated. Furthermore, at the irradiated areas the adhesion, the wettability and the rigidity also changes due to the chemical change of the PDMS polymer. This effect makes the possibility to form microstructures in PDMS with tunable adhesion and wettability properties. In practical viewpoint, the PDMS resist can also have some advantages compared to other resists such as easy stripping, very fast developing (as the un-crosslinked PDMS is soluble in many organic solvents), not sensitive to light or high current.

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Panel Discussion / 162

Nano positioning stages for nano probes used in Proton beam writing and nuclear microscopy

Session 8 - Nuclear Microprobe Applications: Microelectronics / 13

Development of Diagnostic Method for Deep Levels in Semiconductors using Charge Induced by Heavy Ion Microbeams

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Deep-level defects which act as carrier traps are created in semiconductors during crystal growth, device fabrication and also operation under radiation conditions, and they negatively affect the device characteristics. Therefore, it is important to clarify deep levels in semiconductors. Deep Level Transient Spectroscopy (DLTS) is known as one of the most famous techniques to investigate deep levels [1]. However, DLTS does not well work for samples with high resistivity. For deep level investigation in samples with high resistivity, Photo Induced Current Transient Spectroscopy (PICTS) was proposed [2]. However, PICTS has a disadvantage that we need to fabricate semi-transparent contacts since the injection of laser into samples is required to generate carriers (electrons/holes). To overcome this issue, DLTS using charge generated by ion incidence was proposed [3]. Recently, we developed a deep level evaluation system based on Charge Transient Spectroscopy using alpha particles from 241Am (Alpha Particle Charge Transient Spectroscopy: APQTS) and reported the effect of deep levels in 6H SiC pn diodes generated by electron irradiation on the characteristics as particle detectors [4]. In this study, we will report the development of Charge Transient Spectroscopy using Heavy Ion Microbeams (HIQTS). The HIQTS can detect deep levels with micron meter spatial resolution since microbeams are applied. Thus, we can clarify the relationship between deep levels and device characteristics with micron meter resolution. When a 6H-SiC pn diode was irradiated with12 MeV-oxygen (O) ions at 4x109 and 8x109 /cm2, the charge collection efficiency (CCE) decreased to 71 and 52 %, respectively. HIQTS signals obtained from those damaged regions using 15 MeV-O microbeams increased at measurement temperature ranges above 350 K, and the signals are larger with increasing 12 MeV-O ion fluence. On the other hand, the increase in HIQTS signals at temperature range between 250 and 300 K was obtained from 6H-SiC pn diodes irradiated with 1 MeV-electrons although no significant increase was obtained for 12 MeV-O ion irradiated samples. This indicates that deep levels created by O ions are different from those created by electrons.

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Radiation Hardness of n-type SiC Schottky Diodes

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The results of recent IBIC and DLTS studies of radiation damage in silicon carbide (SiC) diodes will be presented. n-type Schottky diodes prepared on an epitaxial grown 4H-SiC thin wafers have been irradiated by a raster scanned alpha particle microbeam (2 & 4 MeV He2+ ions separately) in order to create patterned damage structures at different depths within sensitive volume of tested diodes suitable for Ion Beam Induced Current (IBIC) microscopy. Deep level transient spectroscopy (DLTS) was used to characterize defects created in SiC after implantation of single alpha particles. Robust and proven IBIC experimental protocol [1] has been used to determine a degradation of the charge collection efficiency over a wide fluence range of damaging alpha particle. The radiation hardness of these SiC wafers is compared with the hardness of n-type silicon wafers grown by the Floating zone and Czochralski methods obtained by the same experimental protocol. A suitability of as prepared SiC diodes for the light ion detection and spectroscopy in the MeV range will be discussed from the perspective of applications in harsh radiation environments.

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Ion Electron Emission Microscopy at LNL

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Ion Electron Emission Microscopy (IEEM) at SIRAD, the heavy ion irradiation facility at the XTU Tandem on LNL, is used to obtain micrometric sensitivity maps of electronic devices to Single Event Effects (SEE). The electronic Device Under Test (DUT) is located one millimeter behind a very thin (100 nm) self-standing Silicon Nitride (Si3N4) membrane with a 40 nm Au deposition. The secondary electrons extracted by the ions impacting the Au surface normally are collected and the electron emission microscope, a system of electrostatic lenses coaxial with the ion beam, focuses them onto a chevron MCP and a fast phosphor stack. The ion-impact signals, light spots on the phosphor screen, are regenerated by an image intensifier and finally imaged by a high-rate and high-resolution position detector purposely developed for the IEEM system. The Au layer and the thin Si3Ni4 membrane, used to ensure a copious and uniform emission of secondary electrons, are thin enough not to significantly perturb swift ion beaThe SEE detected on the DUT are time correlated with the position of the ion impact reconstructed by the IEEM system and a sensitivity map is thus constructed.

We give a detailed description of the SIRAD IEEM setup in its present configuration, highlighting possible improvements. To this end we briefly describe the varied experience we have had with the IEEM at the SIRAD: (1) the first tests mapping Single Event Upsets (SEU) in a SDRAM memory; (2) a time-resolved Ion Beam Induced Charge Collection (IBICC) experiment with a power MOSFET device to map out regions with different sensitivity to the impinging ions; (3) the SEU sensitivity of a Shift Register used to address the lines of a monolithic pixels detector fabricated in a commercial silicon on insulator technology; (4) a study of the origin of supply current spikes and destructive events in NAND flash memories under heavy-ions by investigating the role of charge-pump capacitors, previously considered the most probable cause of the phenomenon.

Session 9 - International collaborations and initiatives / 140

New initiatives to advance Accelerator-based Research & Development

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The IAEA Physics Section is pursuing efforts on utilizing particle accelerators to support fundamental and applied research, characterize and qualify materials of nuclear interest and provide education and training. Investigation of radiation damage and radiation hardness with ion beam irradiations is one of the priorities both for nuclear and semiconductor materials. Recent coordinated efforts to develop a protocol for characterisation of radiation induced damage includes both experimental, theoretical and software developments. Other efforts focus on the improvement of the quality of the analytical data from the infrastructural, nuclear data and software development points of view. The support of the recent development of molecular concentration imaging with focussed heavy ions is also part of the Physics Section Programme.

Furthermore, it is also recognized that establishment and operation of accelerator facilities require more attention because of their complexity and remarkable costs involved. Operation schemes of various types of accelerator facilities in response to the users' requirements, standardisation of the beamlines, performance indicators and figures of merit are only a few aspects which require coordinated and joint actions in order to improve the reliability of the service in a cost effective way. One of the new initiatives is that we support to develop and coordinate aspects of management strategies for accelerator facilities.

The IAEA Physics Section is launching a new Accelerator Knowledge Portal (AKP) for the benefit of accelerator scientists, accelerator users and service providers worldwide. The knowledge portal offers not only a database of MV particle accelerators in the world, but it has several networking and community features in an attempt to bring together the accelerator community, as well as provide information to accelerator users and policy makers, too.
The AKP is a community driven website with some features which are opened for the public and some of them exclusively for registered users.

For more information, registration and update your accelerator facility please visit the AKP: http://nucleus.iaea.org/sites/accelerators/.
The different mechanism to achieve our objectives and new initiatives in the field of "Accelerators" which define our programme in the 2014-15 cycle will be presented in this talk.

Session 10 - Nuclear Microprobe Applications: Geology and Environmental Science / 105

High-speed PIXE: fast elemental analysis with a colour X-Ray camera

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A new PIXE-beamline equipped with a full-field energy dispersive X-ray camera [1,2] has recently been put into operation at HZThis so-called SLcam® comprises poly-capillary optics guiding the proton-induced X-ray fluorescence radiation towards a 264×264 pixel pnCCD-chip, each with an energy resolution of 156 eV (@Mn Kα). Two X-ray optics are available, with a magnification of one and six, allowing a field of view of 12×12 mm² and 2×2 mm², respectively. Attached to a large sample analysis vacuum chamber containing a precision sample manipulator, high throughput of even large samples is feasible. Additionally, a beam broadening system ensures a homogeneous illumination of the detection area and an optical microscope allows correlative superimposition of the PIXE maps with optical images. The single CCD pixel size is 48×48 µm² leading to a lateral resolution better than 100 µm for the 1:1 optics. By using sub-pixel resolution algorithms imaging of single capillary channels (25 µm) is expected.

The new setup is mainly developed for the investigation of geological samples for resource technology research which comprises the analysis of grain composition and intergrowths as well as the determination of rare earth element distributions. The simultaneous measurement of a huge array of pixel enables a fast overview over a large region of the sample with first results becoming visible almost immediately. Together with the PIGE implementation at the classical micro-beamline at HZDR this new approach allows analysis of most of the elements of interest in mineralogy.

First results concerning lateral resolution and detection limits on geological samples are encouraging. Due to the low background in the PIXE spectra investigation of trace elements with concentrations below 0.1 at.% is achievable.

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Session 10 - Nuclear Microprobe Applications: Geology and Environmental Science / 58

Deuterium/Hydrogen microscopy in astrogeological material.

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Many primitive meteorites show elevated D/H-ratios relative to terrestrial material. It is believed that this is due to the preservation of the organic molecules which were formed in the presolar molecular cloud [1]. The D/H-ratio varies considerably between different classes of meteorites. This isotopic variation is due to different degrees of mixing of the presolar material with solar system materials [1]. Isotopic measurements of extraterrestrial material collected at Earth provide a way to compare the degree of mixing of the primordial molecules among different solar system material [1].

In recent years a quantitative technique for D/H-ratio microscopy has been developed at Lund Ion Beam Analysis Facility (LIBAF). The technique is derived from the proton-proton scattering technique and has been proven to have the same beneficial features, namely low detection limit, high lateral resolution, and insignificant matrix effects [2, 3]. In this work we present and discuss the results from a measurement on samples from the Tagish Lake meteorite, which is suggested to be one of the most primitive solar system material yet studied [4]. We also present an evaluation of the technique, with results of measurements on a geological standard.

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Session 10 - Nuclear Microprobe Applications: Geology and Environmental Science / 56

Measurement of ratios of oxygen isotopes with pNRA at a microprobe beamline.

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Isotopic fractionation of light elements such as carbon, oxygen and nitrogen is a basis of many analytical tools in hydrology, geology, paleobiology and paleogeology. The goal of the current experiment is to investigate if a nuclear microprobe could be utilized for those applications.

Particularly we focus on measurement of oxygen isotopic ratio. The measurement of stable isotopes of oxygen has a number of applications. The one driving the current investigation is astrogeology or specifically evaluation of fossil extraterrestrial material [1].

There are a number of methods within Ion Beam Analysis, IBA that are sensitive to isotopic and not elemental concentrations. The method chosen by us is photon tagged Nuclear Reaction Analysis pNRA, which is a gamma-particle coincidence method developed at Lund Ion Beam Analysis Facility, LIBAF [2].

The pNRA method was used with a 2 MeV deuteron beam; this setup can potentially be used to detect all 3 stable oxygen isotopes. Three samples of aluminum oxide (Al_2O_3) were irradiated. One with a natural oxygen isotopic fractionation which is 99.8% ^{16}O, 0.2% ^{18}O, 0.04% ^{17}O as well as two enriched samples, one with 70% of ^{18}O and one with 10% of ^{17}O.

Lines belonging to all three isotopes were identified in the particle gamma plots of the enriched samples. The results of the two experimental runs will be presented with a discussion of how well this method can be adapted to geological and astrogeological applications.

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Session 10 - Nuclear Microprobe Applications:Geology and Environmental Science / 99

Elemental compartmentalization changes of marine diatoms as a reporter of biogeochemical cycles

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Studies of phytoplankton elemental composition constitutes a direct measurement of environmental changes, allowing anticipating consequences of anthropogenic alterations to organisms, ecosystems and global marine geochemical cycles. Traditional bulk size-fractionation techniques used to measure phytoplankton elemental composition may present ambiguous results due to considerable detrital matter occurrence. Alternatively, nuclear microprobe techniques are a powerful tool, allowing qualitative imaging of distribution, and quantitative determination of intracellular concentrations.

Coscinodiscus eccentricus are pollution tolerant diatoms that often dominate the phytoplankton community in coastal zones. They are efficient scavengers of trace elements being used as biomonitors of metal pollution. The present study aimed at evaluating metal content and compartmentalization changes in whole cells of Coscinodiscus eccentricus exposed to different metal loads in vitro. Control and metal exposed diatom cells were analysed with particle induced X-ray emission (PIXE), Rutherford backscattering spectrometry (RBS), and scanning transmission ion microscopy (STIM). The STIM image information about density variations of the sample together with the mapping of the major elements, such as C and Si using RBS, and minor elements, such as S and K using PIXE, enabled to identify intracellular compartments. Metal exposure caused drastic alterations in cell morphology. Quantitative elemental transepts across cells also enabled us to identify changes in metal compartmentalization after exposure.

Results on metal mapping of diatoms gave clues about transport, toxicity, and fate of metal in the ecosystem. These aspects highlight the value of diatoms as biomonitors of environmental quality and allowed estimating consequences of metal pollution in marine biogeochemical cycles.

Session 11 – Nuclear Microprobe Applications: Art and Archaeometry / 142

Evidences for an Afghan provenance of lapis lazuli utilized for glyptic by ancient Egyptian combining micro-PIXE and XRF results

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Lapis lazuli is a semi-precious blue stone widely used for different purposes since the antiquity but, at present, there are still some lacking information about both its trade in ancient times and the quarries exploited from different civilisations. Due to the restricted compositional and physical constrains in which lapis lazuli can form, only few sources of this rock exist in the world [1], so that the possibility to associate the raw material to man-made objects is helpful to reconstruct trade routes. Historical sources are in hardly accessible places, such as Afghan and Pamir Mountains, and stones were transported for thousands of kilometres, during periods for which the knowledge of trade routes is still largely incomplete. This is especially true for ancient contexts where there is an absence or scarceness of written records. Although the Badakhshan quarries in Afghanistan (the most famous being Sar-e-Sang) are widely considered until now as the only source of lapis lazuli in ancient times [2], other sources have been considered: Tajikistan (Lyadzhuar Dara, Pamir Mountains), Pakistan (Chagai Hills), Siberia (Irkutsk, near Lake Baikal).

A systematic study of lapis lazuli rocks is carried out by our group since 2007 [3-5], using a protocol based on a multitechnique approach. We present here the analyses on archaeological finds from the Egyptian Museum of Firenze based on the characteristic markers for provenances found in previous experiments. We studied few carved objects made in lapis lazuli from the first millennium BC.

Using the combination of micro-PIXE and portable-XRF analysis we obtained a scientific indication that lapis lazuli used by ancient Egyptians in the first millennium BC presents the same chemical-physics characteristics of the Afghan rocks. In particular we compared the analysed objects with our database taking into account the trace elements content in pyrite crystals, determined by using the in air proton microbeam at the LABEC laboratory in Firenze, and the amount of some minor and trace elements in the whole object found by means of portable-XRF.

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Correlation between Ionoluminescence signal and the manufacturing conditions of the clay bodies of ancient tiles

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The first uses of tiles appeared in the region of Mesopotamia, Egypt and Persia, being the beginning of an enduring tradition. From there, the tile manufacturing technology and utilization spread worldwide, usually through commercial circuits, and consequently the tile evolved and adapted to each culture and local styles.

Tiles are composed of a ceramic body covered by a vitreous glaze layer, which is usually coloured. The ceramic body acts as support of the glaze and its quality is essential for the good conservation and preservation of tiles along the centuries. Some of the factors which will affect the ceramic body quality are the kiln temperature during the manufacturing process and also the raw materials used. In this sense, underfired bodies will tend to be soft, and brittle when they are overfired. The initial clays will also affect the final composition of the ceramic body, with influence for example in its hardiness or final colour.

In this work we propose the combination of non destructive IBA techniques to assess the manufacturing conditions of ancient tiles, specially the ceramic body. The conditions to be determined are:
-the identification of the raw materials through the study of the elemental composition by means of PIXE and PIGE techniques;
- the firing temperature through the study of the compounds or particular mineral phases present in the ceramic body by means of IL measurements.

In this work a set of tiles with quite different chronological production (from the XVII to the XX centuries) were analysed and the obtained results will be presented and discussed.

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Implementation of ionoluminescence in the IBA micromapping setup of AGLAE facility

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Ion beam induced luminescence (IBIL) is a powerful technique that provides information beyond those obtained by IBA methods. For instance, it can be used for the detection of rare earth elements, differentiation of mineral polymorphs and visualization of crystalline defects.
The coupling of IBA and IBIL is very promising for the study of complex targets such as Cultural Heritage artefacts. Moreover, the characterization of these often heterogeneous samples requires imaging at various scales. A new IBIL imaging system has been fully integrated to the external microbeam line of the AGLAE facility.
The IBA microimaging system where the IBIL setup has been implemented exhibits specific features. Fast elemental mapping of large areas (mm to cm) is obtained by combining a vertical magnetic deflection of the beam with a horizontal mechanical translation of the target and data are acquired in list mode. Both specifications are hardly compatible with the collection of an IBIL spectrum. This work details the IBIL setup, gives its performances and explains the technical solutions retained to couple IBIL with IBA in order to produce fast and large IBIL-IBA maps.
The luminescence signal is collected through a 1mm diameter fiber without focusing optics to an Ocean Optics QE65000 spectrometer. It records spectra from 200 to 1000 nm with a minimum collection time of 8ms thanks to an USB 2.0 port. The frequency of the magnetic vertical deflection has been adjusted so that dwell time on each position allows a proper IBIL acquisition without notably slowing down IBA mapping. Imaging of a 1-cm^2 area with a 40-µm resolution can last less than one hour.
The IBIL acquisition is made through specially developed software which, for every position, saves the spectrum, creating a data cube file. Then IBIL information can be mapped in 2D and visualized with the AGLAEmap program which also permits data extraction from coupled IBA-IBIL maps.

Examples of coupled mapping on Cultural Heritage materials will be presented to emphasise the interest of performing simultaneously IBA and IBIL large mappings.

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A comparative study of Etruscan and Tartesic gold jewels by micro-XRF

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Tartesic and Etruscan civilizations were coevals and show similar characteristics in jewel materials and manufacture. The aim of this work is to extend our studies on Tartesic jewellery carried on in the last 15 years by comparing these results with data obtained on some selected Etruscan gold works. Such an intercomparison can help to elucidate the early evolution of gold jewellery manufacturing technology and the geographical spread of this knowledge in ancient times.

A set of Etruscan jewels dating between the 8th and the 4th centuries BC of the National Archaeological Museum of Florence has been studied by means of XRF, a multi-elemental, non-destructive, non-invasive, highly sensitive, fast and portable technique.

As the analyzed Etruscan jewels are characterised by fineness and elaborate decorations, with details of typical dimensions down to tens of microns, a high spatial resolution is required to study the soldering technique and the features of the objects. The micro-XRF set-up developed at the Centro Nacional de Aceleradores (Sevilla) is equipped with a polycapillary mini-lens in the excitation channel providing a micro- beam of 30 µm FWHM spot size. The design of the spectrometer allows for easy and fast measurements and the measuring head can be mounted both horizontally and vertically, depending on the sample.

We characterized the bulk composition of the alloys, the welding/soldering zones and the decorations of each jewel. These results allow identifying the manufacturing method and comparing the Tartesic and Etruscan techniques.

POSTERS

Poster Session - Board 1 / 23

P01 - Study of ion probe formation with high current density for micro irradiation techniques

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Impurity segregation is one of the main effects in structural materials in nuclear power engineering under radiation loads. Impurities migrate along the various types of defects in the materials of the reactor facility during operation. It's lead to generate zone with increased concentration of the impurity on the grain boundaries. Nuclear scanning microprobe has unique capabilities to imitate radiation loads and mapping of the impurity evolution in process of the irradiation step-by-step. The paper discusses the formation proton beam in regime of irradiation microscopic region of the material, which includes several grains. For the purpose of getting uniform dose, the profile of current-density distribution on the target should be close to rectangular. Beam formation regimes which provide maximum current density were considered in order to decrease time for collection necessary dose. Two probe-forming systems based on doublet and separated Russian quadruplet of magnetic quadrupole lenses were discussed. Current density dependence on total current value in the focused beam, when current on FWHM to total current ratio is more than 90% was determined. Total current were varied from 10 to 100nA. Non-uniform distribution of the proton beam of nuclear microprobe, which was measured by method of two slits, was taken into account. Optimized formation beam regimes obtained by numerical simulation were realized into the experiment. The influence of beam energy variation on the spot was considered. Parameters of focused beam was determined by means of calibrated grid scanning and registration secondary electron emission.

Poster Session - Board 2 / 25

P02 - Ion optics of probe forming systems on the base of magnetic quadrupole lenses with conical aperture

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One crucial factor in the nuclear microprobe resolution improvements is the beam optical performance of the focusing system with an acceptable ratio between demagnification and aberration. One of the way resolution improvement may be using magnetic quadrupole lenses (MQL) with a conical aperture. The optics of such lenses is described in the paper [1]. The main difference of MQL with a conical aperture from conventional quadrupole lenses consists in that, the poles are not parallel to the lens axis and positioned at a certain angle. The conical angle influences on the longitudinal distribution of the field gradient, which leads to a changing of the optics of such type quadrupole lens. In MQL with a conical aperture the longitudinal field distribution profile is not symmetric relative to the geometric center of the lens. The focal plane of such lens can be shifted by means of the variable conical angle. Due to this fact the lens aberrations value can be changed by the conical angle.

Investigations of the ion-optical properties of probe-form systems (PFS) on the base of doublet and triplet of MQL with a conical aperture are presented in this work. The dependence of PFS optics from the conical angles and geometric parameters of the system was determined. There is a possibility to increase the acceptance of PFS to use MQL with a conical aperture for a doublet in one and a half times, and for triplets - more than double as opposed to conventional systeThis result confirms a more favorable ratio between demagnification and aberration for analyzed PFS.

Those magnetic quadrupole lenses with a conical aperture are expected to be useful for the focusing system, and there is an opportunity to design quadrupole lenses with a permanent magnet poles. The conical angle variation leads to a change of the focal power lens and allows adjustment of the system.

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Poster Session - Board 3 / 36

P03-Improvement of compaction microbeam focusing with the hundreds-keV three-stage acceleration lens system by optimizing the divergence angle of an incident beam

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A several hundred keV compact ion microbeam system with a three-stage acceleration lens system is under development at Japan Atomic Energy Agency. This system consists of a duoplasmatron-type ion source and the three-stage acceleration lenses. The microbeam system was designed to form a hundred nanometer sized ion beam with an energy of 300 keV. In the previous studies, we had shown that the three-stage acceleration lens system worked as a focusing lens as we designed. A proton beam with an energy of 143 keV could be focused down to 17 Dm in diameter in the performance test [1,2]. The next goal is to improve the demagnification of the lens system by forming an ion beam with several micro-meters in diameter using 150 keV energy proton beam. Because the microbeam system is placed in the open air at this stage, lower energy operation is required to prevent the lens system form occurring electric discharge. The beam size measurements were carried out by changing the divergence angle of an incident ion beam from the extraction of the ion source to the first acceleration lens. As a result, the demagnification of the lens system was improved by optimizing the incident beam divergence angle. We will report the experimental setup and the result of beam formation test in this paper.

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Poster Session - Board 4 / 21

P04-High voltage scanning ion microscope: beam optic and design

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This article focuses devotes to a conceptual design of a high voltage ion microscope. This setup is a further development of compact ion microprobe. Probe formation after a ion source is the main distinction of the proposed design, like it in electron microscopes. Traditional microprobes use only a small fraction of a beam current from source. Most of it is lost on a object and angular collimators. Therefore it is advisable to use a high intensive ion sources in the high-voltage ion microscope. Small amount of the sources current will be compensated by it more fully using and higher acceptance of probe forming system.
Constructively ion optical system is divided into two parts: the ion injector and probe- forming system. The structure of the injector includes an ion source with a small emission aperture, a Wien filter and an axial lens. The lens forms an output crossover of the injector and controls its size. This crossover plays an object collimator role function. The final formation of the beam is made by the probe forming system. It consists of an accelerating tube and multiplet of quadrupole lenses. Inclusion accelerating tube in the probe forming system will reduce the size of the microprobe and improve its performance.
We studied the ion-optical properties of the proposed probe forming system taking into account the influence of the chromatic and third order aberrations energy spread of the beam at the exit of the injector and the nonlinear properties of the probe forming system. Possibility using of high brightness ion sources of existing structures as part of the injector was considered. The design of high-voltage scanning ion microscope based on optimization calculations was proposed and the ion optical characteristics were determined.

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P05-Five magnetic quadrupole lenses with four separated power supplies as one stage probe forming system of nuclear microprobe

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The present work is aimed at creating new types of microprobe systems by means of upgrade of existing ones. We propose one stage probeforming system with five magnetic quadrupole lenses and four power supplies. First two lenses with separated supplies are single unit doublet [1], which is easy to adjust. A final three lenses are a conventional high excitation triplet, which also have two supplies. For such pentuplet an optimization problem was solved in order to get at the target a probe with the best parameters. Excitation for the triplet was specified by a condition of stigmatic beam focusing. Free parameters determined during the optimization were values and variants of a doublet excitations and its position along a beam line. The optimization criteria was based on a maximum normalized acceptance – acceptance normalized to value of a given probe size.

The results of our numerical simulation have shown that proposed pentuplet has more than twice higher maximum normalized acceptance than conventional triplet. Regimes when the system has demagnification coefficients equal in x and y directions and in four times greater than triplet demagnifications for the same normalized acceptance are determined.

The analysis also confirms that the value of the system acceptance increases with a decrease in the working distance. At the same time a position of the single unit doublet along beam line effects insignificantly on the characteristics of probeforming system. Advantage of using single unit doublet as the first two lenses at the pentuplet is confirmed by calculations of tolerances for lens positioning accuracy.

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Poster Session - Board 6 / 50

P06 - New Microbeam Slit System for High Beam Currents

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For ion microprobe materials analysis the brightness of the beam source is the most critical requirement in order to achieve best sensitivity with micrometer resolution and acceptable imaging time. We have installed a multicusp source [1] for negative hydrogen ions in order to optimize beam brightness at SNAKE and thus optimize also sensitivity and lateral resolution of 3D hydrogen microscopy by pp-scattering. As a result of beam transport simulations, we find that it is necessary to inject beam currents as high as 10 µA to meet the required phase space volume at SNAKE without a reduction of the transported beam brightness. This beam current means a thermal load of 250 W for 25 MeV protons mainly deposited at the microslits of SNAKE. Thus, our existing microslit system has to be replaced by a new, high power resisting, water cooled and temperature controlled microslit system.

Using finite element simulations we investigated the requirements for the new microslit system under maximum power input [2]. We use optimum heat conducting materials and a water cooling capable of up to P = 600 W heat dissipation [3]. The geometry of the water cooling is configured to avoid turbulence-induced pressure fluctuation and therefore parasitical vibrations, which would limit the achievable resolution. The slits are electrically preheated to keep constant temperature profiles and thus minimal changes in slit size. A very low uncertainty of the object size of < 1.7 µm and a position accurateness of < 0.3 µm is achievable [2]. The whole slits-system is electrically isolated to allow the tandem stabilization by using the current difference of the ion beam on the slits.

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Poster Session - Board 7 / 52

P07 - Beam Transport of High Brightness Beam through a Tandem Accelerator for a High Energy Ion Microprobe

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For running an ion microprobe a high brightness beam is the basic requirement for sub-µm beam focus in order to fill the small phase space that is accepted by a usual ion microprobe with enough ion current for the desired application. Raytracing simulations of the lens design for the ion microprobe SNAKE at the Munich tandem accelerator demonstrate, that a brightness of B = 1 µA mm-2 mrad-2 MeV-1 is at least required for a 100nm beam focus with 100pA ion current under ideal conditions [1]. So far we achieved 10 times less brightness and a beam spot size of about 1-2µm at 30pA at SNAKE that is installed at the Munich tandem accelerator [2-3]. In order to increase the beam brightness and thus reduce beam spot size we installed a multicusp ion source for negative hydrogen ions manufactured by HVEE [4] at the Munich 14 MV tandem accelerator [5]. At the ion source exit we measure a beam brightness of B = 27 µA mm-2 mrad-2 MeV-1 that in fact represents the space charge limit for conventional ion sources at 30 kV extraction potential. However, the beam brightness is reduced to B = 2.3 µA mm-2 mrad-2 MeV-1 at the high energy side of the accelerator at 17 MeV proton energy.
In order to understand the brightness reduction due to the beam transport through the tandem accelerator we performed beam transport simulations to elucidate the requirements for even larger beam brightness at SNAKE. A main limit for the beam transport is set by additional constraints that the injected beam current into the accelerator is limited to 10-50 µA although the ion source would deliver up to 1 mA into the full acceptance of the accelerator. We discuss the effects of the stripping foil in conjunction with the intrinsic astigmatism in the beam transport and possible parasitic influences. From the calculations we obtain the optimum ratio in the angular and object apertures to form the optimized phase space volume of the beam to keep as much as possible the beam brightness of the source. In addition we present first consequence in our beam transport system and future suggestions on possible improvements for transporting a stable high bright beam through a tandem accelerator.

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Poster Session - Board 8 / 159

P08 - Setup and First Results of the New External Micro-beam of the 5SDH Tandem Accelerator at LAEC

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Recently, a new external micro-beam was commissioned at the 1.7 MV tandem accelerator of the Lebanese Atomic Energy Commission. Despite the use of a RF ion source, it was possible to steer a measurable beam and extract it into air. The setup is performed using an assembly of object slits, collimating slits and two quadrupole magnets from “Oxford Microbeams”. A description of setup and its performance will be shown, as well as some preliminary results of two case studies, such as the localization of nanoparticles in superconducting materials and metals retention in micro-porous polymers.

Poster Session - Board 9 / 94

P09 - The FUDAN proton microbeam for sub-cellular irradiation

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Biological proton microbeam for sub-cellular irradiation is constructed in Fudan University, on a 2×3MV tandem accelerator (NEC-9SDH2). With a 14 meters beam line (8.6m horizontal and 4.5m vertical), energetic protons is chosen by a 30°magnet analyzer and bended vertically up by a 90°bending magnet, and finally focused and pre-collimated to the beam exit at the endstation. To produce an external proton beam down to a sub-cellular size, a capillary collimator of 1.5μm ID and 1mm length is used, though which a collimated proton beam of 3MeV (FWHM<40keV) can be acquired with a spatial resolution <2.2μm in air. The precise detection of the protons are performed by a scintillation detector using a 13μm scintillator film (BC400) at the beam exit together with a photomultiplier (Hamamatsu R7400U) mounted above the target living cells cultured on a 3μm Mylar dish. The counting signals from the photomultiplier is discriminated by NIM electronics and counted by a computer-based counter, which sending the feedback signal to a fast beam deflector on the horizontal beam line for a fast beam cutting in 1μs, when preset number of protons is delivered to target cells. Imaging system are also set up with a cooled CCD camera mounted to Olympus microscope (BHT), and the image processing and cell irradiation steps are automatically performed by a central computer with home-developed program.
The current performance of the Fudan microbeam is summarized as: external proton beam (3MeV, FWHM<40keV) produced by capillary collimator, with a beam size of 2.2μm in air; CR39 tracking method has demonstrated a single-hitting precision >95% and a targeting discrepancy within ±2μm.

Poster Session - Board 10 / 89

P10 – Upgrading the external microbeam facility at INFN-LABEC in Florence: PIXE with carbon microbeams and the forward scattering setup

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At the external scanning microbeam in Florence successful tests have been carried on with carbon ion beams of 10 MeV energy (charge state +4, terminal voltage 2 MV), adopting ultra thin Si3N4 windows (50 and 30 nm thick) for beam extraction: in these conditions, we obtained about 1 nA electrical current (100-200 pA of particles) and beam spot size on sample within 30 micron. First results of the Carbon-PIXE techniques in external (He atmosphere) are presented.

We developed a system for the detection, out of the vacuum, of the forward scattered particles (i.e. at scattering angles in the (-90°, 90° interval)), compatible with the existing set-up, for the implementation of complementary analytical techniques, such as the external-STIM (Scanning Transmission Ion Microscopy) and external-FS (Forward Scattering), now both available at our beamline. First successful tests are shortly presented.

Poster Session - Board 11 / 92

P11 – Fifteen years of the microbeam facility at the INFN-LABEC laboratory in Florence

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2014 is a very significant year for the applied nuclear physics group in Florence, as twenty-five years have passed since the start of the activities in Ion Beam Analysis (IBA), fifteen from the birth of our external microbeam and ten since the new accelerator installation.

The first electrostatic Van de Graaf accelerator arrived in the early 70s and allowed for the beginning of nuclear physics experiments in Florence. From the mid of the 80s, some of the younger members started to work in applied physics and the first IBA studies were accepted for publications in 1989. Since the late 80s and throughout the 90s, IBA activity developed in both environmental and cultural heritage fields; in the latter, main results were obtained in the characterisation of medieval and Renaissance pigments and for the reconstruction of the chronology of Galileo's writings by PIXE.

At the end of the 90s, the EMBE (External MicroBEam) experiment was financed by the Italian National Institute of Nuclear Physics. In 1999 a strong focusing doublet, allowing for the extraction of the first microbeam, was installed, followed in 2000 by a beam scanning system. In 2004, the new Tandem machine in the new laboratory in Sesto Fiorentino allowed for a better beam quality, as regards energy definition, stability, transport and minimum dimensions. Beam transport became easier, faster and highly reproducible, thanks to the installation of a computer-controlled diagnostic system, based on 5 beam profile monitors and 3 monitoring stations along the beam path.

The 2-detector PIXE setup of the early 2000s has been gradually upgraded by making the PIGE, BS and IL techniques also available. By the end of 2000s, the IBIC and STIM techniques have been operative; to obtain this result, it has been necessary to reduce in a controlled way the beam intensity from the nA range down to few thousand of particles per second. This has been achieved through a setup for the detection of the forward-scattered particles in the [-90, 90°] angular range. Detector positioning has been remotely actuated, which has allowed us to quickly adjust the beam intensity to any desired value.

The installation of a second independently-positioned detector in the forward configuration opened up to the possibility of detecting hydrogen in external, even in coincidence mode, to enhance the sensitivity. The most recent developments regard the feasibility study of extracted carbon microbeam.

Poster Session - Board 12 / 8

P12 - Use of a capillary microprobe for heavy ion microbeams in Ion Beam Analysis and MeV SIMS

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The capillary microprobe at the Laboratory of Ion Beam Physics at ETH Zurich has recently been demonstrated as a useful tool to generate microbeams, even for heavy ions and in air. As ions are collimated to micrometer diameters without focusing, the microprobe is neither limited in particle mass nor energy. Recent results for STIM in air showed its applicability and the advantage of heavy ions e.g. in experiments with thin samples, where contrast could be increased using iodine ions.

This prior work with heavy ions encourages the use of our capillary microbeam in a MeV SIMS setup. Heavy projectiles with MeV energies increase secondary ion yields from the bombarded surfaces and cause a decrease in fragmentation of secondary ions [1]. Such high molecular yields combined with the microprobe's micrometer level spatial resolution will enable MeV SIMS imaging for medical and biological applications, where molecular information is most valued.

While typical quadrupole microprobes are limited in particle mass and energy due to the beam rigidity, the capillary microprobe has no such limits and is expected to allow for a further exploration of secondary ion yields with heavier MeV ions than demonstrated so far. For now we will present our results on the investigation of the capillary microprobe's performance as well as show the planned setup for MeV SIMS, which is currently being built.

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Poster Session - Board 13 / 9

P13 - Current measurement for low current microprobe techniques including MeV SIMS

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Reliable measurement of ion beam currents can be a challenging task in experiments utilizing low microbeam currents (for example lower then 1 pA). This is in particular the case for heavy ions that produce a high number of secondary electrons. Normally the current is measured directly from the sample itself or by using a Faraday cup behind the sample. Unfortunately these methods are not reliable under the earlier mentioned circumstances especially if the sample holder for the MeV SIMS setup is on a high kV potential (5 kV at the RBI setup).

In order to enable reliable measurement of beam currents down to a fA range that was needed to investigate the dependence of MeV SIMS yields on different ion microbeam parameters (ion mass, energy, current, etc.), we have considered different approaches. One alternative is indirect determination using RBS. This is only effective for beam currents that are relatively high for MeV SITherefore a more reliable and reproducible method was proposed by measuring the current directly using a PIN-diode that occasionally intercepts the ion beam.

In order to make this possible a new system has been constructed at the RBI microprobe beamline using a stepper motor driven PIN diode holder. In this work we present the constructed system and direct beam intensity measurements that have been compared to indirect measurements using RBS to show the different capabilities of the two methods. Furthermore first yield measurements are shown as well, preparing the way for a better understanding of the processes occurring during MeV SIMS.

Poster Session - Board 14 / 27

P14 - Progress in development and application of MeV TOF-SIMS technique at the Zagreb Heavy Ion Microbeam Facility

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In 2012, ToF-SIMS setup is constructed and installed at the Heavy Ion Microbeam Facility at the Ruđer Bošković Institute in Zagreb. In this method, secondary molecular ions are extracted from the sample after impact of heavy MeV ions, using an acceleration potential difference between the sample surface and a grounded extractor (± 5 kV). Tip of the extractor is positioned perpendicular to the sample surface at a distance of several mm. Dedicated Multi-Stop Time to Digital Converter (TDC) pulse processing electronics have been developed based on the FPGA card, enabling microbeam-scanning control, incoming ion microbeam pulsing and molecular mapping.

Imaging of heavy molecules (>300 Da) with submicron resolution is possible due to the enhanced yield of intact secondary molecular ions desorbed by MeV ions. Considering that this technique is actually the first among all ion beam analysis techniques able to provide information about molecular content of the sample, application perspectives seem to be extremely wide. Initial measurements have shown excellent sensitivity which was demonstrated in the analyses of several organic molecules [1].

In this work further development concerning enhancement and application of the method will be shown. Heavy ion beams from both RBI tandem accelerators as well as different focusing arrangements have been tested. Also, dedicated system for charge normalization, based on the PiN diode, has been developed.

We applied MeV TOF-SIMS for the analysis of modern paint materials. Degradation (stability) of the paints under different aging conditions is studied. In addition, first measurements and application of the MeV TOF-SIMS technique on the investigation of diabetes disease will be shown.

This work is supported by the UKF project „Study of modern paint materials and their stability using MeV SIMS and other analytical techniques“, bilateral project between Austria and Croatia „Application of MeV SIMS for identification and characterization of ageing properties of synthetic painting materials used in contemporary art“ and Marie Curie Actions - Initial Training Networks (ITN) as an Integrating Activity Supporting Postgraduate Research with Internships in Industry and Training Excellence (SPRITE) under EC contract no. 317169.

[1] T. Tadić, I. B. Radović, Z. Siketić, D. D. Cosic, N. Skukan, M. Jakšić and J. Matsuo, Development of a TOF SIMS setup at the Zagreb Heavy Ion Microbeam Facility, accepted to NIMB

Poster Session - Board 15 / 6

P15 - Investigations into doped NaYF4 nanocrystals as novel probes for ion beam induced fluorescence imaging

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Fluorescent probes that can elucidate biochemical mechanisms inside cells are paramount in understanding cell behaviour. We have demonstrated that proton induced fluorescence (PIF) has the capability of producing fluorescent images of cells at super-resolutions (ie below the diffraction limits of light) [1]. However, preliminary results have indicated that high levels of cell auto-fluorescence may mask the signal from conventional organic probes when using PIF, and any fluorescence induced using alpha particles appears to quench very quickly. We are therefore investigating probes which offer both high brightness and resistance to quenching for both PIF and alpha induced fluorescence. Candidates for a suitable probe include doped NaYF4 nanocrystals, which appear to be very bright when irradiated with both MeV proton and alpha beams, and moreover, they have a resistance to quenching even when irradiated using 1.6 MeV alpha particles.

We present results of a spectral study of these potential probes, with the aim of optimising the fluorescence yield for MeV ions, and show that both downconversion by direct atomic excitation and upconversion by energy transfer from sensitizers (Yb3+) to activators (Tm3+) contribute to the fluorescence emission. In this process, delta rays play an important role by raising both the sensitizers and activators to their exited states. Using a focused beam of 1.6 MeV alpha particles, we have achieved sub-40 nm lateral resolutions for fluorescence by imaging 1500 ×150 nm doped NaYF4 nanorods. These NPs exhibited good resistance to quenching and enough brightness to make them promising candidates for future fluorescence probes in PIF and alpha induced fluorescence studies.

[1] R. Norarat, V. Marjomäki, X. Chen, M. Zhaohong, R. Minqin, C. Chen, A.A. Bettiol, H.J. Whitlow, F. Watt. Ion-induced fluorescence imaging of endosomes. Nuclear Instruments and Methods in Physics Research B 306 (2013) 113–116.

Poster Session - Board 16 / 17

P16 - Reconstruction of relief by means of stereo-PIXE for curved target

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This paper demonstrates an innovative simple technique to reconstruct the irregularities on the surfaces with curved geometry in stereo-PIXE (Particle Induced X-ray Emission) set-up. The method assumes that local inclination of the topographical structure of the object results in X-ray yield asymmetries in two spectrometers mounted on both sides of probing beam. The relief on curved target could be quantitatively reconstructed by comparing the detected yield asymmetries and those calculated from a known non-structure cylindrical sample model of target. The results of measurement on a cone seashell at different position of tip are presented.

Poster Session - Board 17 / 41

P17 - Development of a new light collection and detection system optimized for ion beam induced fluorescence microscopy

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Ion beam induced fluorescence microscopy can potentially outperform existing light microscopy imaging techniques due to its high resolution (sub-50 nm) and the ability to combine it with other quantitative techniques such as STIM. Such high-resolution fluorescence microscopy is vital for understanding the function of sub cellular structures within a single cell. Although the current state-of the-art spatial resolution using MeV ions is of the order of 20 nm [1], the same spatial resolution has not been achieved using ion beam induced fluorescence imaging techniques. The main limitation is in the collection and detection of emitted photons from the sample. In this work, a new light collection system based on a custom made parabolic mirror is employed to improve the efficiency thus allowing us to reduce the beam current and achieve higher resolution. A detailed simulation study has been carried out to optimize the mirror design. This custom made mirror not only enhances the collection of fluorescence signal but also allows us to perform the simultaneous structural (Scanning Transmission Ion Microscopy) imaging. A detailed simulation and the experimental results will be presented and compared with existing systems.

[1] Watt, F., Chen, X., Chen, C.-B., Udalagama, C.N.B., Van Kan, J.A., Bettiol, A.A. Whole cell structural imaging at 20 nanometre resolutions using MeV ions, (2013) Nuclear Instruments and Methods in Physics Research, Section B: Beam Interactions with Materials and Atoms, 306, pp. 6-11.

Poster Session - Board 18 / 84

P18 - Variation in the uptake of Nanoparticles by Monolayer Cultured Cells using High Resolution Ion Beam Imaging

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Nanoparticles (NPs) are being increasingly used in a wide range of biological applications, for example, as imaging probes or therapeutic agents1. Understanding the mechanism of cellular uptake of NPs is therefore important. It is known that the efficiency of internalization of NPs by individual cells depends on many factors, including the shape, size, and surface modification of the particles2,3 as well as the timing of the cell within its cell cycle4. Previous work using PIXE has indicated that there can be as much as ten times variation in NP uptake in individual cells in the same cell population5.

We have demonstrated that Scanning Transmission Ion Microscopy (STIM) can image NPs in individual cells with a spatial resolution of around 20nm 6, and this technique therefore offers a way of characterizing and quantifying the number of nanoparticles each cell has internalized. Here we present preliminary results on the variability of uptake of NPs in individual human-derived cells that have been grown under the same controlled conditions.

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Poster Session - Board 19 / 118

P19 - Performance of a gas flow ionization detector filled with He-iC4H10 mixtures for STIM-T

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A cylindrical gas flow ionization chamber has been developed for measuring particle energy for Scanning Transmission Ion Microscopy Tomography (STIM-T) experiments. Due to its ability to withstand the direct beam this type of detector is of great importance for efficient performance of on-axis STIM-T. The response of a He-iC4H10 filled ionization detector to 2 MeV H+ and He+ beams irradiation was studied. Different operating parameters such as concentration of isobutane (iC4H10) (in the range of 55% to 100%), anode voltage, amplifier shaping time, proton scattering at the detector entrance canal and the solid angle of the detector were investigated. The stable operating plateau and the anode voltage at which the best energy resolution is attained were also determined for every gas mixture. The best energy resolution achieved so far for 2 MeV H+ and He+ particles was 1.3%, which is comparable to that of Si PIN diode detectors (in the range of 15-30 keV). Computed tomography (CT) was applied to a set of STIM projections acquired with the gas ionization chamber at the IST/CTN microprobe beam line in order to visualize the 3D-mass distribution in a test structure.

Poster Session - Board 20 / 132

P20 - Improving the lateral resolution in ion beam analysis by deconvolution of the point spread function of a nuclear microprobe

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The precise knowledge of the point spread function (PSF) of a nuclear microprobe should allow to improve the lateral resolution of maps created in ion beam analysis (IBA) using mathematical deconvolution. Therefore, a resolution standard for high current ion beam applications like e.g. particle induced X-ray emission or Rutherford backscattering spectrometry was developed, fabricated and characterized. The standard consists of two concentric structures made of thick titanium film on a glassy carbon substrate in order to ensure elemental and topographical contrast in the IBA method used for imaging with a sufficiently high yield to avoid unreasonably long measurement times. In addition, the deconvolution software 'PSFinder' was developed and optimized for the resolution standard. Both were used to determine the PSF of the ion nanoprobe LIPSION at the University of Leipzig. The dimensions of the PSF were cross-checked with measurements of Cu- and Ni-meshes, where good agreement was obtained. Furthermore, 'PSFinder' was used to increase the resolution of several element maps leading to a decrease of the edge full width at half maximum by up to 45 % after a deconvolution for structures of known dimensions. Moreover, different element maps with a low signal-to-noise ratio were deconvolved using a deconvolution algorithm based on the Wiener filter in order to investigate the limits of this approach. Here, the result was a clear improvement of the contrast and the resolution of the structures in the map. A quantification of the improvement, however, was difficult in these cases due to the low signal-to-noise ratio in the original images. Enabling the improvement of the resolution of elemental maps, the developed resolution standard and the associated deconvolution software 'PSFinder' are now integrated in the measurement protocol of the ion beam laboratory LIPSION.

Poster Session - Board 21 / 53

P21 - A compact gas ionisation direct-STIM detector for MeV ion microscopy

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Scanning Transmission Ion Microscopy is a powerful technique that yields structural information in sub-cellular whole cell imaging [1,2]. Usually, a Si p-i-n diode is normally used in direct-STIM measurements as a detector. However, this is sensitive to radiation damage because when used to image deep sub–µm areas the ion fluence is high even for moderate numbers of ions per pixel. This leads to a shift in charge carrier collection efficiency which appears as an apparent degradation in energy resolution [3,4]. Gas ionisation detectors are intrinsically insensitive to radiation damage. Therefore, a compact gas ionization direct-STIM detector [4] is based on the Geiger-Muller geometry without a Frisch-grid is being developed for use in a MeV ion microscope based on a standard Oxford triplet lens and scanning system. In this work, the critical issues in designing the detector are a large the entrance window area to accept a scanned beam and obscuring as small a solid angle as possible to facilitate installation of proton induced fluorescence detector [2] while maintaining a small capacitance (10.2 pF) to achieve low noise. Tests with isobutane against a calibrated capacitor show that a resolution of about <~20 keV fwhm could be achieved which allowed imaging of Ren cells on a Si3N4 support and an effective ionisation energy of 21 eV at pressures above 600 mbar and bias voltage >600 V. The resolution is most probably limited by the charge sensitive amplifier resolution which is estimated to be ~180 e- rms in the current configuration.

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Poster Session - Board 22 / 113

P22 - Identification and reduction of acoustic-noise influence on focused ion beam (FIB)

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The use of focused ion beam (FIB) for research or processing of nanostructures requires very accurate beam positioning. However, numerous reasons for beam-position fluctuations exist. When FIB is used for specimen imaging, then these beam fluctuations cause the image jitter, blur or specimen-edge deformation. Similarly, beam fluctuations decrease the spatial resolution of FIB-based technological processes of milling or deposition. The sources of fluctuations are electromagnetic interference (EMI), floor vibrations and airborne acoustic noise.

Our work concerns acoustic noise impact on focused ion beam fluctuations. The measurements were carried out on Helios NanoLab 600 DualBeam system with ion and electron beam columns. Reference specimens were imaged by electron or ion beam while acoustic waves of different frequency, magnitude and direction were intentionally generated nearby the system.

It was found that while EMI-related distortions are caused by a wide and continuous spectrum of frequencies, for acoustic noise the strong deformations of image occur only at several resonant frequencies (mainly in the range 100-400 Hz). Comparison of results obtained for either electron or ion beam allowed to attribute different resonant peaks to various FIB-system components (ion column, electron column, specimen stage). Spectral analysis showed that resonant components of the acoustic noise surrounding the system cause beam-position fluctuations in the range of several nanometers, highly unfavorable for nanotechnological works on FIB. The noise is generated mainly by various parts of the system itself.

A method was also developed to identify whether the observed beam-position fluctuations originate from acoustic noise or from electromagnetic interference. It was possible because electromagnetic field impacts charged particles along their entire path while the acoustic vibrations act only on the mechanic elements of the system. Therefore the electromagnetic fluctuations are dependent on the particle velocity (i.e. the beam energy) while the acoustic fluctuations are independent of it.

It was found that distortions (of FIB image and of patterns performed by FIB technological processing) caused by ion-beam position fluctuations due to acoustic noise can be reduced. The reduction can be achieved by selection of appropriate parameters of FIB process e.g. working distance and scanning parameters (like scan rate and scan direction).

Poster Session - Board 23 / 152

P23 - A Fully Digital Data Acquisition System for Nuclear Microprobe Applications

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Nuclear microprobe analyses involve the spectrometry of signals from a set of different detectors, e.g. for particle induced X-ray emission (PIXE); particles from backscattering (BS), transmission (STIM), or forward scattering; electron detection; or even for ion beam induced light (IBIL). Besides data acquisition from the preamplifiers with subsequent digital pulse processing, a data acquisition system also needs beam scanning with data synchronization, and desirably, beam and stage control.

We report on the design and development of a scalable multi-channel data acquisition system that is based on 1-GHz, 12-bit digitizers with on board FPGA digital pulse processing. The digital pulse processing uses a parameterized transfer function that enables flexible and user friendly filter setup for pulse shaping to handle the various preamplifier signal shapes. Build into a compact PXIe chassis the digitizers communicate and synchronize their data via the backplane with a two channel, 16-bit DAC scan module with on board FPGA scan control. The system is connected via a PXIe-PCIe controller to the control PC with the user interface. The digital data acquisition system is discussed and performance tests are presented.

Poster Session - Board 24 / 86

P24- A segmented detector for airborne gamma-ray spectroscopy

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The Airborne Gamma-Ray Spectrometry (AGRS) allows to measure the radioactivity content of large areas of topsoil. The results of surveys are exploited in many fields like homeland security and geological, mining and hydrocarbon explorations.
Following the IAEA guidelines the first Italian prototype for AGRS has been recently developed and extensively tested for many investigations. In [Guastaldi et al., Remote Sens. Environ., 2013] a detailed radiometric survey of Elba Island (Italy) performed with an autogyro is reported. The energy spectrum information is analyzed offline using the Full Spectrum Analysis (FSA) technique with the Non-Negative Least Square (NNLS) constraint to achieve the concentration of potassium, uranium, and thorium in the topsoil. A multivariate estimation method for interpolating the primary under-sampled airborne gamma-ray data considering the well-sampled geological information as ancillary variables (Collocated Cokriging) is applied for the first time. The maps of U, Th and K distribution with relative uncertainties are the final products. These experiences encouraged to develop a segmented instrument for investigating airborne gamma ray directionality. The great innovation is the new layout of sixteen NaI detectors, which work independently. The peculiar configuration is set in order to generate asymmetries in the signal acquisition, that can be used to study the anisotropy of radioactivity distribution. The potential identification of orphan sources with respect to the natural background has been confirmed by Monte Carlo simulations, specifically developed for modeling the expected signals.
The communication focuses on the mechanical layout, the main electronic features and the performances of the segmented detector for airborne gamma-ray spectroscopy. We show how the constituents can be easily and rapidly mounted by a single operator on most common helicopters. The acquisition system can manage the charge integration and multi-channel analysis of the signals from each detector. Data registration is flexible: both list-mode and full energy spectra can be selected as recording modes. Preliminary feasibility studies have been performed to test the mechanics and the hardware of the whole system, which is designed to work without any human attendance. The first flights are planned in 2014, with the aim to detect the artificial point sources having intensities on the order of 10^8 Bq and natural enriched fields already monitored.

Poster Session - Board 25 / 5

P25 - Proton Beam Writing combined with controlled subsequent electrochemical etching for the three-dimensional microstructuring of p-GaAs and p-InP for MEMS applications

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Nowadays, an increasing demand on microelectromechanical systems can be found in the field of capacitive accelerometers, pressure sensors or energy harvesters [1,2].
Three-dimensional microstructures needed for those applications have already been fabricated with the lithographic technique Proton Beam Writing [3].
In particular, just by variation of the irradiation fluence, Proton Beam Writing in combination with fluence depending electrochemical etching proved to be promising for three-dimensional semiconductor microstructuring [4].
Recently, a controlled fabrication of free-standing or undercutted structures was possible due to finite element simulations of the electrochemical etching rates [5].
We are going to present our latest results regarding the microstructuring of p-GaAs and p-InP.

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Poster Session - Board 26 / 76

P26 - Proton beam writing of dye doped polymer microlasers

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Proton beam writing, a high resolution direct write lithographic technique, is becoming increasingly interesting both because of its continuous improvement in spatial resolution as well as its applicability to wide range of materials [1, 2]. It is also capable of fabricating three dimensional high aspect ratio structures and this technique is well suited for optical applications because of the straight and smooth sidewalls of the fabricated structures. In this work, proton beam writing is applied to the fabrication of microlasers in dye doped polymer layers. Interestingly, the dye is not bleached by the irradiation process. Whispering gallery mode microlasers of different cavity designs are fabricated and their laser characteristics studied using optical pumping. Directional laser emission as well as low pumping thresholds are obtained using these laser cavities. The details of the cavity designs and their performance will be presented.

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Poster Session - Board 27 / 38

P27 - Creation of double tilted pillar structures for microfluidic applications

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Exploiting the advantages of P-beam writing lithographic method to make irradiations into tilted samples, doubly tilted pillar microstructures are created for microfluidic applications.

SU-8 negative tone resist spin-coated on glass substrate was irradiated with 2 MeV proton microbeam. The created structures consisted of pillars standing in rows inside an appropriate frame. The fluid (water, blood, etc.) can be coupled through an inlet into the chip and extracted through an outlet. The goniometer feature of the Atomki microbeam facility enabled us to tilt the pillars relative to the surface normal (in this case odd rows were tiled by +20 an even rows by -20 degrees).

The aim of tilting the pillars is to increase the functional surface of the pillars with which the fluid can interact with the stationary phase, and to improve the fluid dynamics properties. With the help of this promising method we are able to create microfluidic chips that can be used for an improved efficiency cell capture device.

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P28 - Automatic beam focusing in the 2nd generation proton beam writing line

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Proton beam writing (PBW) can fabricate three-dimensional, high aspect ratio nano structures with vertical, smooth sidewalls and low line-edge roughness [1]. This technique has been used in many areas like photonics, micro or nano-fluidics, nano imprinting, silicon machining and mask for x-ray lithography.But as so far, unlike electron beam lithography or electron microscopy, proton beam writing technique is still under development. Focusing is achieved by manually adjusting the currents of the Oxford triplet lenses to get the minimum beams spot size. An automatic focusing system based on C++ had been developed and a sub-micrometer beamspot of approximately 600 nm × 700 nm was achieved in the first generation proton beam writing system [2].

Recently the 2nd generation PBW system has shown improved focusing performance down to 13 nm x 30 nm and a fine lithographic HSQ patterns with 19 nm line width and 60 nm spacing have been fabricated [3,4]. In order to make this system more convenient, a new automatic focusing program based on Labview is being developed. A high quality resolution standard with a sidewall projection of less than 5 nm is used to focus down the beam [5]. Scanning Transmission Ion Microscopy (STIM) and proton induced secondary electron microscopy provide the imaging information to characterize the beam. During the experiment, the beam is first manually focused on quartz to a spot of ~10 µm × 10 µm. Then the beam is scanned over the sharp edge of the Ni resolution standard to form an image. After that the user defines two lines, which cross the edge of the image in x and y directions. The data obtained is fitted by a modified Gauss error function to give the beam spotsize. The minimum beam size is found in each direction by changing the currents of the quadrupole power supplies. With this method, beam spot sizes of 30 nm x 80 nm have been achieved.

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Poster Session - Board 29 / 71

P29 - Comparative Study of Microstructured Polymer Foils using STIM with H, He and Li ions

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Scanning Transmission Ion Microscopy (STIM) provides structural images based on the energy loss of swift ions passing through the sample and therefore it depends on local mass density. That quality makes the technique useful for morphological analysis of microstructures fabricated on homogeneous substrates such as polymer foils.

Proton Beam Writing (PBW) is an important technique for fabrication of various devices with applications on different areas such as microfluidics, tissue engineering substrates and microphotonics, among others. Microstructures obtained by PBW can be an interesting object of study by STIM when fabricated on homogeneous substrates. In this case, STIM can be an important tool for morphological characterization.

In this work we present a study of two different structures obtained by PWB and subsequently submitted to STIM measurements using three different ions, namely protons, alpha particles and Li+3 ions, in order to evaluate the differences between the analysis by each particle beam considering the energy resolution achieved by them and the effects on the resulting images. The results indicate that ions heavier than protons may provide distinct information about the microstructures under study.

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P30 - Ion micro-beam and pulsed-laser beam techniques for the micro-fabrication of diamond surface and bulk structures

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Micro-fabrication in diamond by using highly focused ion beams is involved in a wide set of emerging technologies, exploiting the exceptional characteristics of this material for application in bio-physics, photonics, radiation detection. Micro ion-beam irradiation, in particular, allows the micro-patterning and fictionalization of surface and/or bulk material, modifying its optical, electrical and mechanical characteristics. Complementary techniques, such as those employing pulsed laser irradiation, permit the implementation of more complex geometries, useful for the integration of electrical and optical devices.
In this contribution we summarize our work concerning high resolution ion beam modification of the optical properties of diamond and pulsed laser beam fabrication of 3D diamond detectors. Preliminary results about the integration of the two approaches for the production of complex 3D-structures in diamond bulk and surface are also discussed.

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P31-Ion-beam-fabrication of buried graphitic electrodes for the excitation of electroluminescent NV centers in diamond

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Focused MeV ion beams with micrometric resolution are suitable tools for the direct writing of conductive graphitic channels buried in an insulating diamond bulk. Their effectiveness has been shown for the fabrication of multi-electrode ionizing radiation detectors [1] and cellular biosensors [2].
In this work we investigate such fabrication method for the electrical excitation of color centers in diamond, aiming at the development of quantum technologies based on single-photon sources [3].
Differently from optically stimulated light emission from color centers in diamond, the electroluminescence requires a fairly high current flowing in the diamond subgap states between the electrodes. With this purpose, buried graphitic electrode pairs with a spacing of less than 10 μm were fabricated in the bulk of a single-crystal diamond sample using a 6 MeV C microbeam.
The electrical characterization of the structures showed low currents at low applied bias voltage, due to residual radiation associated with the fabrication process; a significant current increase was observed above an an effective voltage threshold of several tens of volts.
The light emission from the sample was imaged both in electroluminescence and photoluminescence regime using a confocal microscopy setup, in order to identify the active regions of the device and to investigate the role of the residual vacancy density in the diamond sample due to ion irradiation.
A bright electroluminescent emission from native neutrally-charged nitrogen-vacancy centers (NV0) was observed; the presence of light emission associated with residual vacancy clusters associated with radiation damage (A-band) was not observed in electroluminescence regime, indicating a potential application of the electrodes fabrication method for the electrical control of isolated single-photon sources.
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Poster Session - Board 32 / 96

P32 - Writing and Imaging Nanostructures of Single Defects in Diamond

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In the last years quantum information processing developed very fast and progressive. One promising way to realize a solid state quantum computer is doping solids with single atoAmong the variety of doping possibilities one prominent candidate is the Nitrogen Vacancy (NV) centre in diamond.

This long-known defect (present in most of natural diamonds) can easily be created by nitrogen implantation followed by thermal annealing. It posesses strong optical absorption and flourescence which enabels the optical imaging of single NV centres.
The NV centre exists in three different charge states: neutral (NV0), negatively (NV-) and positively charged (NV+). The electron spin associated to the negative NV- can be polarized and read out optically. Moreover, the coherence time of the electron spin can reach ms at room temperature in ultrapure diamond samples. Therefore, the NV centre can be used as a qubit. The entanglement of NV-qubits has already been demonstrated at room temperature [1, 2].

In order to create scalable structures based on coupled NV centres one needs to place single NV centres within distances of a few tens of nanometers. To realize the addressing of single NVs within this resolution, we developed a unique technique to implant single ions with an accuracy of below 10 nanometers in all three dimensions.

The nano implanter is a combination of an atomic force microscope (AFM) with a pierced hollow tip and a low energy ion source (keV range). This results in a very small collimated ion beam about a few nanometers in diameter. An ion gun with a gas source provides a broad range of ion types and an integrated electron multiplier allows the detection of single ions. This contribution gives the actual status and discusses the advantages and limitations of the system.

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Poster Session - Board 33 / 102

P33 - Resolution intercomparison in microscopy and lithography using light and ion beam imaging

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An important question for MeV ion beam microscopy and lithography is how the
spot size in MeV ion beam microscopy relates to the Franhofer-diffraction (FD) limited resolution of an optical microscope or lithography tool. The comparison is not straightforward because not only are the resolving powers measured in different ways but also the achievable contrast depends on the imaging mode.

We have undertaken a numerical study of the broadening of edges by numerical convolution of 2D symmetric Gaussian, rectangular box, and Airy disc shaped PSFs. They correspond to the extreme cases of objective aperture size as in programmable proximity aperture lithography (PPAL); and aberration limited resolution in a MeV ion microprobe and FD limited light microscopy/lithography. The test objects were grid bars, δ-functions and gratings as well a natural sharp image of neural cells. These correspond to common objects for focussing MeV ion microscopes, optical microscopes, determining the modulation transfer function in an image and a real common object.

The results reveal that in general the shape of the PSF does not have a strong influence on the image. The Gaussian and Airy disc PSD approximate each other and the fwhm measured from edge broadening is a factor 1/3 of the measured fwhm of the central peak in the PSF measured using a δ-function object. However, the Airy disks for FD have a notable effect with fringe effects and give a background at large distances from the open region of a gridbar. The statistical noise from counting statistics has a very significant effect on the visible perceived resolution.

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P34-Quantitative Hydrogen Microscopy

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Proton-proton scattering at the Munich microprobe SNAKE gives the unique possibility for sensitive 3D hydrogen microscopy [1]. Quantification of the hydrogen content without the need of any reference sample, a sensitivity of few or even less than one atomic part per million, a lateral resolution of about 1 µm and a depth resolution of a few micrometers are the main characteristics. We use proton energies between 10 MeV and 25 MeV for analyzing any kind of unsupported samples with thickness between 10 µm and 250 µm depending on the atomic density of the investigated material [2].

Two major aspects of the quantification of the method are presented in this paper: (1) Improving the sensitivity without loss of lateral resolution requires maximum beam brightness that we have optimized by a new Multicusp proton source [3] and improved temperature stabilized water-cooled microslits. (2) Precise quantification requires (a) scattering cross sections [4], but also (b) efficiency analysis of the coincident signal. The latter will be discussed as a main topic: Strict filters has to be applied on the selection of the scattering plane as well as a strict 90° angular sum for the coincident proton pair in order to avoid accidental coincidences. However, multiple (small angle) scattering effects on the path of the protons through the sample disturb this angular conditions of the detected protons. This decreases the detection efficiency depending on the atomic number Z and thickness (in at/cm2) of the scattering event. In order to understand this decrease and correct for a precise quantification, we use the CORTEO [5] Monte Carlo code. This code allows simulating coincident events with variable detector geometry by efficient numerical routines. We present simulation of multilayered sandwich targets compared to analytical calculations and experimental data. The results are applied to quantification of current studies of geological samples as well as heavy metals.

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Poster Session - Board 35 / 54

P35 - Quantitative reconstruction of PIXE-Tomography data for thin samples using GUPIX X-ray emission yields

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Characterisation of microscopic specimens using PIXE Tomography (PIXET) has long been restricted by experimental and computational difficulties, mainly: i) the sample must be mounted on a rotation axis to collect data on at least 180°; ii) the long duration of data acquisition; iii) very few reconstruction algorithms available. In recent years, several attempts have been made to design reconstruction algorithms to overcome these difficulties. A major trend is to implement algorithms other than filtered back projection (FBP), in order to use fewer projections. This trend has been recently extended to quantitative reconstruction. A MLEM code, JPIXET, has been designed at the IST/CTN in Lisbon [1]. PIXET data are processed together with Scanning Transmission Ion Microscopy Tomography (STIMT) data. The process is long, which was solved using GPU programming

At CENBG, PIXET was implemented to study biological samples. The major composition of these samples is uniform, whereas the internal structure is complex and not as contrasted as in most inorganic specimens. This led us to develop a specific algorithm, TomoRebuild. We present here a new development of this software package, to perform quantitative PIXET reconstruction. X-ray yields are obtained from the GUPIX code. The GUPIX data base is available for protons up to 5 MeV and also in the 20-100 MeV energy range, deuterons up to 6 MeV, 3He and alphas up to 12 MeV. The main features of the TomoRebuild code were kept: user-friendly design, modular C++ implementation and portability on Windows and Linux operating systems.

In this version, X-ray yields are calculated for thin samples, i.e. without simulating X-ray attenuation. This task is foreseen for the coming months. The distribution of element content (in g/cm3) can be obtained from PIXET data ; only the last step requires STIMT data to get normalised concentrations in µg/g. Images misalignment can be corrected, as well as the difference in beam size between the two experiments. We will give here reconstruction examples on biological specimens of Caenorhabditis elegans nematodes, analysed at the microbeam line of the AIFIRA facility of CENBG. The experimental conditions and the different steps of data processing will be discussed. The reconstruction results will be compared between the different codes TomoRebuild, DISRA and JPIXET.

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P36 - Improvement of spatial resolution and detection efficiency by control of secondary-electron in single-event three-dimensional time-of-flight Rutherford backscattering spectrometry

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A single-event three-dimensional time-of-flight (TOF) Rutherford backscattering spectrometry (RBS) system has been developed using 150 keV focused Be+ beam [1, 2]. Signals from a secondary electron detector (SED) and a micro channel plate (MCP) were used for the start and stop triggers in single-event TOF-RBS. The achieved time resolution was 4.4 ns, corresponding to the depth resolution of 12 nm, which was slightly longer than that with a beam chopping system [3]. The fluctuation of the secondary-electron flight-path affects the time resolution in single-event TOF-RBS. When the secondary electron is not detected, the stop signal is uncounted to the TOF-RBS data, resulting in the prolonged measurement time. Therefore, the flight-path and detection efficiency of the secondary electron are important factors for single-event TOF-RBS. In our recent study, the shorter time resolution and measurement time were achieved with positive sample bias voltage [1]. However, the reason of the improvement was not clarified. In this study, the secondary-electron flight-path from the sample to SED were simulated using a boundary element method for clarify the reason of the improvement and the simulated results were compared with the experimental them.

Applied voltages used in the simulation were +10 kV, -1.6 kV, and 0 V for SED, four MCPs and the outer part of the focused ion beam (FIB) column, respectively. Sample bias voltages were ranging from -200 to + 200 V. When negative sample bias voltage was applied, electric field between the sample and the outer part of the FIB column accelerates the electrons, most of the electrons impinge the outer part of the FIB column and few electrons were detected at SED with long flight paths, resulting in the long time resolution and low detection efficiency. When positive sample bias voltage was applied, the secondary electrons were not spread around the sample and pulled by high voltage at SED before electron return to the sample, resulting in the short time resolution and high detection efficiency. In the experiment, with sample bias voltage of +100 V, the depth resolution of Pt stripes under a SiO2 layer was 6 nm smaller and the measurement time was 65 % shorter than those without sample bias voltage. These results were good agreement with the simulated them.

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P37 - A 17th century glass collection from Monastery of Santa Clara-a-Velha in Coimbra, Portugal: Exploratory results using PIXE

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During the archaeological excavations carried out at the Monastery of Santa Clara-a-Velha in Coimbra (Portugal), an enormous set of glass fragments (including millefiori and filigrana glasses) were found and dated from 16th to 17th century. This Monastery was occupied by the Order of Poor Clares from 1317 until 1677, when it was abandoned due to the repeated flooding, a consequence of its proximity to the Mondego river.

The analyzed glass set comprises fragments with several colours (green, blue, red, brown, yellow and uncolored glass), shapes (bowls, gourds, bottles, cuppingglasses, inkpots and flasks) and decoration techniques (mould blown patterns, filligrana, gilding and engraving). With the aim of studying and characterizing this collection, thirty-six glass fragments were analyzed by means of micro Particle Induced X-ray Emission (PIXE). The glass fragments were first embedded in a resin mold and then polished in order to ascertain that a smooth and corrosion free surface could be analyzed. All the glasses were found to be of soda-lime-silica type. The relatively high amounts of K2O, MgO, P2O5 and the presence of chlorine suggest the use of coastal plants as the source of alkali. µPIXE analysis also allowed to distinguish three groups regarding the alumina contents where it was possible to identify a group with low alumina contents (Al2O3 < 2 wt%), a group with high alumina contents (3< Al2O3 <6wt%) and a group with very high alumina contents (Al2O3 > 6 wt%). This alumina content clustering suggests the use of three different silica sources which can be related to the existence of different production centers.

Results will be further compared with glass compositions dating to the same period from Portugal and from several European centers, highlighting differences and similarities, and discussing the possible origin of the finds.

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P38 - Micro_PIXE and SEM-EDX Studies for Archaeological Metal Findings Characterization

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For the analysis of cultural heritage materials Proton Induced X-ray Emission (PIXE) spectroscopy and Scanning Electron Microscopy with Energy dispersive X Ray spectrometry (SEM –EDS) hold an important position in the field of non destructive techniques. The chemical characterization of the material composition facilitate the determination of their provenance, age, technology of production, intended purpose and, therefore, their authenticity.

For metal objects the concentration levels of the basic constituents can be used for drawing conclusion concerning the economic situation of an area or a period.

The present study concerns the investigation of two different archaeological metal findings, probably dated to the Iron Age period and found on the Piedmont region in northern Italy, in order to ascertain the types of alloys used and whether there is a differentiation in composition depending on the type of object.

Preliminary SEM-EDX assays and subsequent macro and micro PIXE experiments were conducted on the same tiny fragments detached from the finding internal and external surfaces.

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P39 - Withdrown.

Poster Session - Board 40 / 104

P40 - Light element analysis and imaging using Particle Induced Gamma-ray Emission

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Spatially resolved analytical methods are important in many fields of application. One area of research is the interdisciplinary field of geometallurgy, which combines geology with extractive metallurgy to explore and exploit ore bodies and extract valuable minerals. The characterisation of the chemical composition and structure of ores and intermediate products is important for the material- and energy-efficient utilization of primary and secondary resources of minerals and metals.

Information on “mineralogical light” elements, i.e. hydrogen to fluorine, is needed over a broad concentration range from traces to major elements. These elements can be a useful resource in itself, like lithium. Besides, they provide information about the genesis of e.g. ore deposits and rocks or have a strong influence on the mechanical behaviour of rocks.

The analysis of such elements, especially at the trace level, is a challenge for many standard micro-analytical methods. Particle Induced Gamma-ray Emission has the advantage of 1) obtaining quantitative results without matrix-matched standards, 2) being non-destructive and 3) wide applicability. In addition, it can be combined with other ion beam analysis methods like Rutherford Backscattering Spectrometry and Particle Induced X-ray Emission, for which a new set-up has been developed (see the presentation of Josef Buchriegler).

The nuclear microprobe of the Helmholtz-Zentrum Dresden-Rossendorf has been upgraded with a Gamma-ray detector (HPGe) to facilitate the spatially-resolved analysis of light elements (lithium to phosphorus). This upgrade is presented in this work. Extensive calibrations have been performed. Next, the implementation of the analysis and imaging procedures are discussed. Finally, the first results of the application on mineralogical samples are shown.

Poster Session - Board 41 / 55

P41 - An evaluation of the proton-proton scattering method for hydrogen measurement in geological samples.

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We performed a comparison of the facility for hydrogen measurement available at the microprobe facility in Munich (SNAKE) with the one developed at Lund Ion Beam Analysis Facility (LIBAF).
The motivation for study of trace amounts of hydrogen in geological samples comes from Nominally Anhydrous Minerals (NAMs). They are a class of geological materials containing water although no water is expected. The presence of water in a material, even in trace amounts changes a number of physical properties of materials and constitutes an important field of study for geologists, but commonly used methods like IR-, Raman- or Secondary Mass Spectrometry require calibration by IBA methods at these low concentrations of hydrogen [1].
The method used by us for hydrogen evaluation is pp-scattering with a Double Sided Silicon Strip Detector (DSSSD). This has been shown to be a very sensitive method [2]. In fact it is the only method for 3D hydrogen microscopy with a sub-μm resolution and at sub-ppm sensitivity. Such a setup for hydrogen measurement is available at SNAKE where it has been tested extensively. A similar detector setup based around a circular DSSSD has been developed at LIBAF [3]. We tested and compared our two setups. As test samples we used a number of hydrogen rich and hydrogen poor geological samples.
The geological samples used for the evaluation are the minerals zoisite and orthopyroxene. Samples have been previously tested with SIMS [4]. The current experiment provides a validation of the setup and measurement methodology used at LIBAF.

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Poster Session - Board 42 / 10

P42 - Archaeometric studies of Byzantine pottery from Harsova (Carsium), Romania

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A set of ceramic shards excavated from Hârsova (Carsium), Romania, dated to the 11th century A.D. were subjected to archaeometric investigations, aiming to reveal the manufacturing techniques and raw materials employed by the potters from Low Danube region during the Middle Byzantine period.
The initial division of the shards into fine and coarse ceramics was refined by a subsequent petrographic study. Optical microscopy observations detailed the potteries fabric, identifying - up to a certain point - the mineralogical composition. Petrographic investigations agree with the original separation of the shards into fine and coarse fabric, but indicate an important variability in terms of mineral composition, homogeneity and porosity. Two main categories of shards were found: one defined by the use of kaolinitic clays, occurring in Southern Dobrogea, approximately 80 km away from the archaeological site, and a second more heterogeneous one, based on the use of several sedimentary sources. The coarse inclusions present in some samples were identified as sand grains (quartz). The firing took place either in reducing or oxidizing conditions. The surface treatment of the fine fabric shards consists in a very thin shimmering golden engobe, a rare occurrence in Byzantine pottery, while other fragments were decorated with a green-olive glaze. Some fragments of coarse fabric have a fine engobe located both on the internal and external side of the shard. Micro-PIXE measurements of the ceramic shards performed with the nuclear microprobe facility of AN2000 accelerator of LNL, INFN Italy allowed the identification of their chemical composition. The statistical analysis of the PIXE data evidenced two main categories of pieces with distinct compositional signatures. Thus, the fragments from kaolinitic clays were clearly dissimilar from the rest of the samples. Micro-PIXE scans of the interfaces between the decorated surfaces and the ceramic bodies provided hints about the minerals present in the golden engobe and the green glaze. Clear changes in the chemical composition were revealed for the glazed shards, characterized by a strong enrichment of the lead oxide content compared to the corresponding ceramic body.
This study on Byzantine ceramic will continue with micro-PIXE analyses on shards from other archaeological sites, trying to identify possible commercial exchanges in the Low Danube region during the 11th century A.D.

Poster Session - Board 43 / 95

P43 - Full field x-ray fluorescence for the two-dimensional micro imaging of painted artworks

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X-ray based techniques are particularly suited for the non-destructive investigation of Cultural Heritage and Archeological materials. An interesting case concerns the analysis of pigments that are often distributed on artworks in painted decorations of sub-millimeter dimensions. Moreover, the chemical associations among elements composing a given pigment can be often identified by investigating the elemental distribution in the micrometric scale.
Generally, the two-dimensional elemental mapping is obtained by the scanning micro-X-Ray fluorescence. The merits and limits in using this experimental approach were largely demonstrated. The recent advances in the development of the Full Field X-ray Fluorescence (FF-XRF) instruments provided a novel and promising alternative for performing the two-dimensional elemental imaging avoiding the scanning approach.
Recently we developed a novel Full Field X-ray Pinhole Camera (FF-XPC) presenting high-energy and high-spatial resolution. The FF-XPC consists of a back-illuminated and deep-cooled CCD detector coupled to a 50 micron pinhole-collimator, coaxially positioned between the sample and the CCD. A low-power X-ray tube is used as primary source for inducing the characteristic X-Ray fluorescence in the samples under investigation. A multi-image acquisition in single-photon counting and a real-time processing of each image-frame have enabled the use of the FF-XPC for the energy dispersive X-ray fluorescence imaging with an energy resolution down to 133 eV at 5.9 keV and spatial resolution down to 25 micron.
The FF-XPC can work with different magnifications (M) depending on the dimensions of the sample under measurement. This approach allows both the micro-FF-XRF imaging of small dimension samples (down to 2x2 mm2 at M=6) and the macro-FF-XRF of large samples (up to 5x5 cm2 at M=0.4).
The potential use of the FF-XPC for the two-dimensional imaging of pigments in polychrome archaeological pottery and paintings has been tested and verified. The macro-FF-XRF approach was used to obtain fast information on the global composition of the painted decorations on the samples; the micro-FF-XRF imaging and spectroscopy was applied with the aim of characterizing the pigment and to investigate the painting technique.

Poster Session - Board 44 / 16

P44 - Some applications of micro-PIXE in the study of ancient bronze, silver and obsidian artifacts

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A study concerning the copper provenance of some Bronze Age items (axes, sickles, daggers, swords, celts) found on Romanian territory was performed at AN 2000 accelerator in Legnaro . The problem consists in their classification from the Bronze Age regional mines point of view – North-East Bulgaria [Ai Bunar – "fingerprints" minor-trace elements As (up to 2-3%), Ni and Sb (hundreds of ppm)], Serbia [Rudna Glava and Majdanpek – "fingerprints" trace elements As, Sb, Ni, Ag, Se (thousands-hundreds ppm)] or Transylvania (e.g. Baia-Mare – "fingerprints" trace elements Sb and Ag – thousands of ppm). We analyzed 15 very small samples (less than 500 microns diameter) from different archaeological sites in south Romania – especially axes and sickles. The majority presents relevant traces of arsenic and antimony, suggesting the use of copper from north Bulgaria. For silver items, we performed a study on some Silver (drachms) issued between V and III Century BC by Greek colony Histria – situated on Romanian Black Sea coast and on Dacian silver imitations of Greek silver tetradrachms to detect trace elements which can be used as fingerprints for Silver provenance (e.g. bismuth for South-Balkans deposits, antimony for Carpathian deposits, gold, lead) and to determine copper content which is an indicator of the metallurgical procedure - copper was used to increase the mechanical properties of silver. We also used micro-PIXE to study some Neolithic micro-tools (blades) found in Romania to determine the obsidian provenance: Tokay Mts. (Hungary, Slovakia), Melos (Greek islands), Lipari, etc. Micro-blades found in Magura - an important Early Neolithic site from Teleorman county, Muntenia, approx. 100 km South-Vest of Bucharest – were analyzed. To identify the obsidian sources we used the group Rb-Sr-Y-Zr, a "pattern" specific for each source as illustrated in PIXE spectra. Our results suggest for Magura Early Neolithic – Crish Starcevo culture (6200 – 5200 BC) samples some Aegean sources, obsidian arriving from Macedonia crossing the Balkans.

Our studies demonstrated that micro-PIXE is a good analytical tool to investigate the composition of ancient artefacts in order to determine their provenance, especially the geological sources of metals and obsidian.

Poster Session - Board 45 / 49

P45 - Ion beam analysis of golden threads from Romanian medieval textiles

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Technical studies by classical analytical techniques routinely used for analysis of cultural heritage materials (XRF, SEM-EDX) and recently, more sensitive techniques like AES, XPS, SIMS and laser-ICP/ MS, have been done previously in order to characterize the chemical nature and morphology of the metal threads from historical textiles. As these techniques proved to be limited for the study of the very thin, possible multilayered metal threads, further in-depth investigation was needed to be carried out by using the IBA techniques which are ideal by their features for this kind of research. The aim of our study carried out within the CHARISMA - Fixlab IBATEX and IBATEX 2 projects was to demonstrate the necessity of integrating the advanced IBA methods with the classical techniques, frequently used in museums, for an in-depth applied interdisciplinary research that brought new developments and rich accurate information on historical golden threads constituent materials, especially the trace elements, and their ancient production technologies. Samples taken from gold-brocaded velvets and religious embroideries (15th - 18th cent.) were wires and strips wrapped around a dyed or undyed silk core yarn and wires with no core yarns (IBATEX), also cross-sections obtained by embedding the metal threads in epoxy resin (IBATEX 2). Compared to classical techniques, IBA nuclear methods allowed a precise detection of the layered structures and an accurate identification of the trace elements (detection limits of few tens of ppms). PIXE results confirmed that both types of the metal threads studied – wires and strips – have a layered structure being made of gilded fine silver (refined by cupellation) and not of Au-Ag alloy, or gilded Ag-Cu alloy or Au-Ag-Cu alloy, as resulted from the previously performed SEM-EDX analysis. The elemental maps allowed us to identify the areas from which the metal threads structure and quantitative composition could be precisely determined. RBS results revealed that, in some cases, the gold layer is separate from the silver bulk by an interface layer that resulted by atomic diffusion of silver into the gold layer, which lead us to the conclusion that probably the methods used for gilding were the diffusion gilding or the fire gilding.

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Poster Session - Board 46 / 80

P46 - Searching for Late Bronze Age soldering techniques: µPIXE analyses of the gold bracelets from Herdade do Álamo (Beja, Portugal)

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Several technological changes emerged in the Atlantic façade of the Iberian Peninsula during the Late Bronze Age. Goldwork gained new forms by joining hollow casted pieces. Two bracelets from the collection of the National Archaeological Museum in Lisbon, produced by joining 10 round sections solid casted thin rings, are the most representative gold items of a transition period when the ancient solid casting techniques are used simultaneously with the new techniques.

No analytical study was carried out on the very rare objects representative of this transition period. In this work we present the first analytical results obtained for the two bracelets from the find of Herdade do Álamo (Moura, Beja, Portugal) using non-destructive quantitative elemental tecnhiques: portable XRF and µPIXE. Both bracelets were analysed in situ by portable XRF. The equipment comprises an Eclipse IV X-ray source with a Rh anode and a XR-100 SDD Amptek X-ray detector at 90º geometry. The identification of the joining technology requiring higher spatial resolution was carried out through µPIXE analysis using a 2 MeV proton beam focused down to 100x100 µm2 at the external microbeam setup of the IST/ITN van de Graff accelerator comprised of a OM150 quadrupole triplet system and a 30 mm2 Bruker SDD X-ray detector.

The results indicate that the solid thin rings were produced with two different alloys with an Ag/Au ratio of 0.20 for 12 of the rings and 0.26 for the other 8 rings. The latter show the same composition as the two hollow gold neckrings from the same find, analysed by portable XRF. Micro-PIXE elemental distribution maps and point spectra analysis shows that the bracelets were soldered together using gold alloys with different melting points. Based on this data we could reveal the manufacture technologies employed and propose the first mounting scheme for the bracelets of the Herdade do Álamo find.

Poster Session - Board 47 / 116

P47 - Simultaneous micro-PiXE and micro-EBS analysis applied to XVI century silver and copper coins

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A coin database containing information about major, minor and trace elements contents and correlation to different eras is an essential tool in: reconstructing the metal and monetary circulation; detecting forgeries; and recognition of different metallic surface enrichment processes.
Ion Beam Analytical (IBA) techniques like Particle Induced X-ray Emission (PIXE) and Elastic Backscattering Spectrometry (EBS) play an important role in the construction of this database as they are non-destructive and can determine the coin chemical composition fingerprint down to the ppm range, which in some cases can be related with the ore provenance or the metal purification process. However, surface inhomogeneities (thickness and composition) created by centuries of corrosion growth may hinder a proper quantitative analysis unless a micro beam is used, which allows us to select small regions with different degrees of corrosion.

Using 1.0 and 2.0 MeV proton beams from the nuclear microprobe (resolution 3x4 um2) located at the Laboratory of Accelerators and Radiation Technologies at CTN (Sacavém - Portugal), micro-PIXE and micro-EBS spectra were taken simultaneously, for two copper and four silver XVI century Portuguese coins. 2D-PIXE maps were acquired for all coins, followed by point analyses in selected regions with higher or lower corrosion extension.

A self-consistent solution is hard to reach analysing the PIXE and EBS spectra with separate software tools, as for these coins a single PIXE or EBS spectrum is not enough to obtain a unique solution describing a concentration profile changing with depth. Thus, a simultaneous analysis of these spectra was performed using DataFurnace software [1] which allowed differentiating the superficial corrosion layer from the uncorroded volume underneath. Grazing incidence XRD was also performed on these coins in order to identify the compounds present in the corrosion layer, namely oxides, carbides, etc, and that were used as input for DataFurnace.

The results obtained using this simultaneous fitting procedure are presented and compared with the results obtained when fitting only the PIXE spectra.

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Poster Session - Board 48 / 117

P48 - Micro-PIXE and micro-XRF applied to ancient coins

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High-tech replication technology combined with old-school craftsmanship raise new questions on the authentication of ancient coins. As such, numismatists are aware that the traditional methods used to determine coin's authenticity, like visual inspection, are often insufficient, and look for different approaches that may give a conclusive answer.

Particle Induced X-ray Emission (PIXE) or X-ray Fluorescence (XRF) are becoming common analytical techniques applied to cultural heritage artefacts, including coins, and that may help to distinguish a true coin from a modern fake.

Surface inhomogeneities (thickness and composition) created by centuries of corrosion growth in the most varied environments may hinder the results obtained by these techniques. However, if a micro beam is used it is possible to select small regions with different degrees of corrosion in the coin surface, and to decouple the corroded from the uncorroded volumes.

This work presents the results obtained by micro-PIXE and micro-XRF for two copper and two silver XVI century Portuguese coins. A comparison in terms of trace elements detected and quantified by both techniques is very important as they can give information about the ores provenance or purification processes used. Micro-PIXE spectra were acquired using 1.0 and 2.0 MeV proton beams from the nuclear microprobe (resolution 3D4 Dm2) located at the Laboratory of Accelerators and Radiation Technologies at CTN (Sacavém - Portugal). The used 80 mm2 Link X-ray detector has a 145 eV energy resolution and it is positioned at 135o to the beam direction. As for the XRF spectra, they were acquired using a M4 Tornado micro-XRF spectrometer fitted with a Rh-tube (Bruker AXS) and a X-Ray poly-capillary optic offering a spot size down to 25 µm combined with high excitation intensity. Detection of fluorescence radiation was performed by an energy dispersive Silicon-Drift-Detector with 30 mm² sensitive area and energy resolution of 142 eV for Mn-Kα.

Poster Session - Board 49 / 130

P49 - Application of nondestructive analytical techniques to the study of Iron Gall inks

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The study of cultural heritage objects is a delicate issue that needs well defined experimental conditions when applying any analytical technique. When dealing with historical valuable documents it has to be taken into account many factors as for example the fragility of the pieces or their size, and so the use of non-destructive techniques are usually mandatory.

The use of an external proton beam is one of the best options to characterize them under the experimental conditions that safeguard the documents.

In this work, handwritten documents with iron gall inks were characterized by means of X-ray diffraction (XRD) and by ion beam (IBA) techniques using an external proton beam, mainly Rutherford backscattered spectrometry (RBS) and proton induced X-ray emission (PIXE) which were simultaneously recorded.

Through XRD analysis it was possible to identify the presence of different types of cellulose, anhydrite and calcite as part of the constitution of the paper. These analyses helped to understand and fit the RBS spectra obtained for paper and inked areas. However, it was not possible to obtain information about the ink composition neither by XRD or by RBS because of the low quantity of material in comparison with the compounds belonging to paper support. Another constraint in RBS is the impossibility of distinguishing between the Ca and Fe signals due to the close kinematic factor.

Yet, PIXE analysis provided the identification of the major elements in iron gall inks (Fe and S) and also the presence of trace elements, such as Cu, Zn, Sr or As, which made possible to differentiate inks, even in the same document [1]. Moreover, PIXE results revealed differences in terms of paper elemental composition between documents with the same date and place which suggests different manufacturing processes [2].

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Poster Session - Board 50 / 135

P50 - Lost image recovery for stained glass panels from the Rosslyn Chapel

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Rosslyn Chapel, built in 1446, is both a place of Episcopalian worship and an ancient monument of international significance. Having survived both the Protestant Reformation and the English Civil War, the chapel is now one of Britain's most important sites for romantic tourism; displaying suspected Templar ritual within its mediaeval carving and influences of Scottish Tractarianism within its stained glass. The iconography of both stone and glass continues to be hotly debated, but there is no doubt that the glass imagery was intended to have sacred significance.

Manufactured by Clayton and Bell of London between 1867 and 1887 with a "grisaille" technique, many panels have suffered extreme image loss due to an aggressive internal environment. Grisailles are the Fe-rich monochrome paints fired into the glass and used for shading or outlining the image. This work aims to uncover the lost imagery by revealing the original artists' paint strokes.

Using Total-IBA methods we have demonstrated that the lost grisaille leaves an invisible residue in the glass in the form of a surface enrichment of Fe. The Fe concentration is low, but can be detected and mapped using PIXE at a high spatial resolution.

New external microbeam facilities at the Surrey Ion Beam Centre incorporate a large-range motorised sample manipulator and a high count-rate spectrometry system for both particle and X-ray detectors. Using this we have analysed deteriorated stained glass from Rosslyn chapel; we show that the lost image can efficiently be recovered by PIXE with high acuity, which provides invaluable guidance for the restorers and conservators.

This project is partially funded by The Rosslyn Chapel Trust (Registered Charity number SC024324) Lord and Lady Rosslyn, Owners and Trustees, and WREN Heritage Fund (www.wren.org.uk)

Poster Session - Board 51 / 137

P51 - The collection of Hispano-Moresque tiles from the Museum of the Roman Theatre, in Lisbon: chemical characterisation by μ-PIXE

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The great earthquake that partially destroyed the city of Lisbon in 1755, brought to light the ruins of a Roman theatre that was used as a structure for later constructions up until the 18th century. Those constructions included private middle-class houses, some exhibiting interesting decorations. One of such houses was decorated with Hispano-Moresque tiles dated from the 15th-16th century. The collection includes flat monochromatic, arista and cuerda-seca tiles.

The chemical characterisation of these Hispano-Moresque tiles was performed by particle-induced X-ray spectrometry (μ-PIXE) on small polished fragments so to avoid surface corrosion and/or contaminants contribution. Both the glaze and the ceramic paste were analysed. Five colours were identified: white, blue, green, amber and brown, which are in agreement with the typical colour palette for these type of tiles. All glazes contain high contents of lead oxide (ca. 40-50 wt%), with higher amounts for white and blue glazes. Also, two groups of colours can be distinguished based on the SnO_2 content, used for opacifying white and blue glazes and, therefore, with higher contents in these colours (ca. 5-10 wt%) than in the others (0-2 wt%).

Calcitic clays were used for the ceramic bodies of the tiles, as the chemical composition shows: all samples display a similar composition, with CaO contents between 15-27 wt% and relatively low Fe_2O_3 contents (< 5 wt%).

The archaeological collection of the Museu do Teatro Romano is the first collection from Lisbon to be characterised and the results are part of a wider study that aims at comparing several Portuguese and Spanish collections.

Poster Session - Board 52 / 144

P52 - Paintings on copper by the Flemish artist Frans Francken: PIXE characterization by external micro beam

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The use of copper as support for oil paintings had its major expression in the XVI century, at first in Italy and then in the Netherlands, probably due to the interest that artists revealed for painting on unconventional supports and also due to the accessibility of copper plates for etching, engraving and enamelling [1]. Copper offers a flat and rigid surface, where it is possible to perform more detailed images than in a traditional canvas or wooden support, providing also a particular finishing and colour brightness.

Through the external beam micro-PIXE analysis of three paintings dated from the XVII century and attributed to the Flemish artist Frans Francken, the present work aims to contribute to the study of the painting materials used by the painters in this type of support. The experiments were performed using a 2MeV proton beam at the external proton beam set-up installed at the CTN/IST [2]. The beam, with dimensions of 70x70 um2, can raster a surface of 800x800 um2.

Several spots in each of the paintings were chosen for analysis in order to cover almost all the pigments used in the colour palette and at the same time highlight some of the painting techniques used. Lead was detected in almost all the analysed regions and its distribution is usually presented as small agglomerates. In previous works lead has been related with the presence of lead white, used as ground layer [3]. Regarding the colours composition, preliminary results point to specific combination of elements in the characterization of each colour. For example, the red colour or carnations exhibit a high concentration of Hg and S, suggesting the use of vermillion.

Small quantities of gold were also detected in two of the three paintings: in “The conversion of the gentiles” the gold is restricted to the Christ’s halo, while in “Marriage at Cana”, gold was also detected in a wall drapery pleats. Gold was usually employed on renaissance paintings to embellish some details of the representations [3].

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Poster Session - Board 53 / 151

P53 - Micro-PIXE Analysis of Ancient Roman Coins

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The presence of surface silver-enriched layer is quite common in objects made of a silver-copper alloy. In this work, some silver coins have been sectioned to study their microstructure and especially to assess the presence or absence of corrosion layers, enriched layers and non-homogeneities between surface and bulk. Some papers in literature have already discussed the topic [1-2]. Coins presented here are victoriati and quinarii minted by the Roman republic between the II and the I century B.C.

Micro-PIXE measurements were carried out on cross-sections at the AN2000 microbeam facility using 2 MeV protons. The beam was focused to a spot size of ~5 μm and raster-scanned over the samples both on parts close to the surface and in several areas of the bulk. Quantitative analysis has been carried out by means of the Gupixwin software (version 2.1.3).

The presence of a very thick surface silver-enriched layer (100-150 μm) has been confirmed on the victoriati, while other analysed coins do not appear to be affected by this phenomenon. Profile measurements carried out on victoriati show that silver content is clearly higher in the surface layer, suggesting an intentional depletion occurred with acid chemicals during minting operations, as reported in [1]. The most interesting results, however, concern the distribution of minor elements along the section. Elements like chlorine and iron, commonly present in soil and water, are detected mainly in the silver enriched layer close to the surface and can be therefore linked to alteration phenomena due to the bury conditions. On the other hand, gold is clearly linked to silver as it appears mostly present in the silvery layer. Nickel and zinc seem, on the contrary, to be correlated to copper, since their presence is concentrated in the bulk.

In conclusion, the strong different elemental distribution between surface and bulk implies that compositional analyses carried out with surface techniques on untreated surfaces on silver-copper alloys are not reliable to provide fineness of ancient coins. These measurements confirm also that the victoriati series is strongly characterized by the presence of thick surface silver-enriched layers, as shown in recently published data [3].

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Poster Session - Board 54 / 68

P54 - Micro-PIXE and micro-NRA: associated tools for materials characterization

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Ion-based analytical techniques are widely used for modification, characterization and analysis of materials. One of the motivations for the continued development and application of these techniques part of its high sensitivity for determining and quantifying trace elements in the order of parts per million (ppm), and create images of their distributions and depth profiles.

The elemental characterization of homogeneous samples is performed in, general, by combining two complementary techniques: PIXE (Particle Induced X-ray Emission) and RBS (Rutherford Backscattering) with stationary beams of the order of mm2. However, when there are structures in the sample surface and differences in its elemental distribution, we should use the scanning microprobe system, where the beam spot size is reduced to the order μm2. Micro-PIXE provides the spatial distribution of elements with Z> 12, while micro-RBS allows the study of multilayered samples with good selectivity for thin layers of heavy elements on light elements substrates.

Despite having lower cross sections, the technique of nuclear reaction analysis (NRA - Nuclear Reaction Analisys) can also be used in the microprobe system. The positive point is that through NRA is possible to determine light elements and their isotopes on any substrate.

This study shows the possibility of associating micro-PIXE and micro-RNA for elemental characterization of materials. As an example, we present the elemental characterization of a human hair. Samples were irradiated at the Laboratório de Implantação Iônica (IF-UFRGS) with the 3MV tandem accelerator. Proton beams with energy of 1,75 MeV were selected for analysis of particular micro regions of the hair sample with micro-PIXE and micro-NRA. The selected regions were scanned with focused proton beam of 4 - 5 μm spot size and currents of ~80 pA. The size of the scan was 200 μm x 200 μm.

Through the micro-RNA was possible to determine the presence of carbon in human hair, while micro-PIXE showed the presence of elements such as S, K, Ca and Zn, which are in agreement with other studies [1].

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Poster Session - Board 55 / 11

P55-Withdrawn

Poster Session - Board 56 / 19

P56 - Nuclear microprobe analysis of leaves from tropical nickel hyper-accumulators growing in Sabah, Malaysia

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Understanding the ways in which hyperaccumulator plants take up and store metals is critical to optimizing their use for/in phytoremediation and phytomining. However, to date very little work has focussed on tropical nickel hyperaccumulators, which have the greatest potential for application in future phytomining operations. Three plant species with high phytomining potential (because of their high growth rate and high nickel accumulation) are Phyllanthus balgooyi, P. securinegoides (Phyllanthaceae) and Rinorea bengalensis (Violaceae) that occur in Sabah, Malaysia on ultramafic soils. Phyllanthus balgooyi has been found to have 17% (dry weight) of nickel in the phloem sap, and 8,600 mg/kg in leaves, while P. securinegoides has 23,250 mg/kg of nickel in leaves and 10,800 mg/kg in phloem tissue, and Rinorea bengalensis 12,800 mg/kg in leaves and 22,600 mg/kg in phloem tissue. Plant material was cryo-fixed in the field using specially designed procedure. Micro-PIXE quantitative elemental mapping of leaves and stems of these species was performed using the nuclear microprobe at the Materials Research Department, iThemba LABS (South Africa). Analysis of stem sections of Phyllanthus balgooyi shows that nickel is very high in the major vascular bundles, whereas calcium is high in cortex and collenchyma, but low in the vascular bundles. In P. balgooyi leaves nickel is located mainly in the lower epidermis extending into the spongy mesophyll, and extremely high in central vascular bundles. Calcium is also high in the lower epidermis and spongy mesophyll, but lower in the vascular bundles. Similar elemental distribution patterns in the leaves apply to P. securinegoides, except that nickel is higher in the upper epidermis. In Rinorea bengalensis leaves nickel is highest in both the lower epidermis and upper epidermises. Calcium is located mainly in leaf vascular bundles (xylem and phloem) in the mid-vein as well as in spongy mesophyll tissue.

Poster Session - Board 57 / 22

P57 - Fluorine uptake into human enamel surface from fluoride-containing sealing materials during cariogenic pH cycling

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For prevent the caries and the hypersensitivity dentin, sealing materials with/without fluoride were applied on the tooth. Fluoride contains sealing materials also made the acid resistant layer made from the sealing materials were physically protected the acid attack. Fluoride in the tooth structure will prevent the acid attack. This study evaluated the demineralization prevention and fluorine (F) uptake into human enamel of sealing material using an automatic pH cycling. Five 150µm sections were prepared from each tooth. Fluoride containing sealing materials (i.e. “MS coats F” (MSF)) and fluoride free sealing materials (i.e. “Hybrid coats 2” (HI)) were used in this study. Each material was applied to the original tooth surface and cut surface were covered with sticky wax. The automatic pH-cycling (pH6.8 - pH4.5) simulated daily acid challenges in the oral cavity was carried out for 4 weeks. Caries progression was analyzed using the difference of integrated mineral loss (ΔIML) calculated from transverse microradiography (TMR) taken before and after 4 weeks pH-cycling. The fluorine and calcium distributions in the carious lesion in each specimen were evaluated using PIGE (Proton Induced Gamma Emission) technique at TARRI (Takasaki Advanced Radiation Research Institute), Japan. The surface margin of enamel was defined as the 5% of calcium concentration point. 1000 µm × 1000 µm area at the surface of enamel was scanned and constant width region at an arbitrary position in the area was analyzed. The average amount of F in the outer 70 µm of each specimen was obtained.
The ΔIML of MSF and HI are significant lower than control. From the PIXE analysis of the fluorine uptake in the enamel surface, MSF only showed significant difference than HI and control. These results suggest that the MSF will make the acid resistant layer and provide fluoride into the enamel surface. HI and MSF prevent the demineralization both. The fluoride existence in the enamel surface suggested that MSF will prevent the demineralization, even if the layer was removed, in clinical. The PIGE and PIXE techniques were useful in understanding the benefit of fluorine by fluoride-containing sealing material for preventing caries.

Poster Session - Board 58 / 66

P58 - Contribution of micro-PIXE to investigate the toxicology of soluble and particulate cobalt on human lung cells

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The mechanisms of toxicity of metal oxide particles towards lung cells are far from being understood. In particular, the relative contribution of intracellular particulate versus solubilized fractions is rarely considered as it is very challenging to assess, especially for low-solubility particles such as cobalt oxide (Co_3O_4). We used micro-PIXE analysis to quantify the intracellular particulate and solubilized fractions of Co in human lung cells (BEAS-2B) exposed in vitro to cobalt oxide. Single cells were imaged and elemental quantification was carried out in whole cells, either excluding the particles (giving the solubilized fraction) or including only the cobalt oxide particles (giving the particulate fraction).

Quantitative determination was assessed by simultaneous micro-PIXE and micro-RBS analysis performed with a proton beam of 3.0 MeV energy at AIFIRA facility, CENBG. The proton beam was focused down to a 0.8 µm spot size, resulting in a 350 pA beam current on target, to determine the trace element content (Mg, P, S, K, Ca, Fe, Co and Zn) at the subcellular level.

The Co solubilized fraction determined by micro-PIXE was in good agreement with ICP-MS measurements obtained on bulk analysis of cell lysates. In addition, mico-PIXE analysis enabled to quantify the intracellular particulate fraction of Co which could not be achieved by ICP-MS, since the samples preparation for ICP-MS involved a fraction of extracellular cobalt oxide particles sedimentation together with the cells, preventing the intracellular cobalt particle fraction assess.

Our study shows that cobalt oxide particles, of very low solubility, are readily incorporated by BEAS-2B human lung cells. Combination of micro-PIXE and ICP-MS techniques allowed demonstrating that they are partially solubilized at low pH within lysosomes. Solubilized cobalt was detected within the cytoplasm and the nucleus. As expected from these low-solubility particles, the intracellular solubilized cobalt content is small compared with the intracellular particulate cobalt content, in the part-per-thousand range or below. However, we were able to demonstrate that this minute fraction of intracellular solubilized cobalt is responsible for the overall toxicity. Cobalt oxide particles are readily internalized by pulmonary cells via the endo-lysosomal pathway and can lead, through a Trojan-horse mechanism, to intracellular release of toxic metal ions over long periods of time, involving specific toxicity.

Poster Session - Board 59 / 73

P59 – Enhanced RBE of submicron focused low LET protons

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Due to their physical and radiobiological properties, in particular their increased relative biological effectiveness (RBE), high linear energy transfer (LET) radiation qualities are of special interest for tumour therapy. To study the influence of spatial dose distribution on the biological effectiveness we use the ion microprobe SNAKE to concentrate the deposited dose in cells by focusing a certain number of low LET protons or ions to a submicron spot which approximate a high LET dose distribution. By changing the spot size and the number of focused ions one has opened a wide field to study RBE effects in dependence of dose distributions on submicron scale.

For our experiments we compare homogeneous proton irradiation with spots of focused 20 MeV protons (LET in water: 2.65 keV/µm) or 45 MeV Lithium ions (LET in water: 60 keV/µm) in a matrix pattern with certain numbers of ions per matrix point resulting in a constant dose of 1.7 Gy. For induction of chromosome aberrations we observed an increased effectiveness for higher number of ions per point and thus for a higher ionisation load per spot. 1.7 Gy irradiation with 541 protons per spot increases induction of dicentrics by a factor of 2.7 with respect to homogeneous proton application. However, the ionisation load per spot, the product of LET times the applied ions per spot, is not enough to understand RBE. At constant ionisation load at a single point of the matrix the effectiveness for the yield for dicentrics increases with LET. At the same mean dose of 1.7 Gy and the same matrix pattern 5 Li ions increase the yield for dicentrics by 45%, one Carbon (LET in water: 310 keV/µm) ion by 130%, with respect to 117 protons per spot.

The results indicate that at least two effects cause the increased RBE of high LET particles. On the one hand the focused dose application leads to an locally enhanced DNA double strand break (DSB) density, which increases the probability for misrepair, e.g. joining wrong ends of DSB. On the other hand the very high doses in the core of a heavy ion lead to an higher amount or more complex DNA damage, e.g. due to nonlinear

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Poster Session - Board 60 / 81

P60 - Analysis of erythrocyte elements in chronic hepatitis C patients treated with interferon and ribavirin by in-air microPIXE

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Chronic hepatitis C (CHC) patients treated with pegulated-interferon and ribavirin (Peg-IFN+RBV) and telaprevir (Peg-IFN+RBV+TPV) are frequently associated with anemia, whereas the precise mechanism of anemia is not determined. This study is aimed to analyze the elemental changes in erythrocytes to investigate the pathogenesis of anemia caused by combination of Peg-IFN and anti-viral drugs.

Subjects and Methods: Thirteen CHC patients (3 cases without IFN therapy, 7 cases treated with Peg-IFN+RBV, 3 cases treated with Peg-IFN+RBV+TPV) and 4 healthy controls were enrolled in this study. Whole blood was collected via peripheral vein, and the sample for PIXE analysis was prepared according to our method (T. Nagamine, et al. IJPIXE 22, p 249-258, 2012). Elemental levels in erythrocytes were determined by the method of Iwata et al. Elemental distributions of erythrocytes were analyzed by in-air microPIXE at JAEA- Takasaki, Japan.

Results: 1. Elemental map of erythrocytes

The shape of erythrocyte, so-called the donut-like, fitted well with the counterplots of Cl, S, and K dots in healthy controls. Erythrocyte shape became blurred in Peg-IFN+RBV and Peg-IFN+RBV+TPV cases. In Peg- IFN+RVB cases, Cl, S, and K dots aggregated modularly. These elements tended to diffuse spreading over the erythrocytes in Peg-IFN+RBV+TPV cases. In addition, Na dots were apparently detected in Peg- IFN+RBV+TPV cases compared to Peg-IFN+RBV cases and healthy controls. Fe dots were distributed granularly dividing into 3-4 peace in normal erythrocytes, and Fe dots were not changed by Peg-IFN+RBV or Peg- IFN+RBV+TPV.

2. Elemental levels in erythrocytes

HCV cases without IFN showed increased levels of Ca, Fe and Cu and decreased level of Zn in erythrocytes compared with healthy controls. Peg-IFN+RBV cases showed increased level of Zn and decreased level of Ca compared with controls. Every element tended to increase in Peg-IFN+RBV+TPV cases.

In conclusion, the elemental distributions in erythrocytes were changed in CHC patients received Peg-IFN+RBV. In addition, erythrocytes elements were altered by Peg-IFN+RBV+TPV, suggesting that the pathogenesis of anemia was different between Peg-IFN+RVB therapy and Peg-IFN+RBV+TPV therapy.

Poster Session - Board 61 / 91

P61 - Study of the elemental distribution in the stigma of a Ni hyper-accumulator plant

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The knowledge of the metal concentration and distribution in hyperaccumulator plants is of great interest in order to understand how and the amount of metals, the plant is able to absorb and store. Nuclear microprobe set-ups are good tools to carry out these studies.

Up to now the elemental distribution and concentration in roots, leaves and seeds of different hyperaccumulator plants have been studied, but there is less information about other vegetal organs, as flowers or fruits. Flowers are temporary organs responsible of the sexual reproductions in plants. Among other parts, flowers carry the masculine (stamens) and feminine (gynoecium) reproductive organs. The gynoecium is typically divided in ovary, stile and stigma. The stigma is the pollen receptor and the way in to the ovary for the pollen tube that eventually will reach the ovary, fecundate an ovule and produce a fruit with its corresponding seeds.

The present work studies the elemental distribution and concentration in the flower stigmas of the Iberian Ni hyperaccumulator species Alyssum serpyllifolium subsp. malacitanum. In order to study the elemental distribution in the flower stigmas nuclear microprobe techniques were used.

Significant S, Cl, K, Ca, Ti, Mn, Fe, Co and Ni concentrations non-homogenously distributed, have been found in this part of the flower. While Cl, K, S and Ni are preferentially localized at the stigma Ca, Mn and Co are mainly in the upper part. In this region, Ca distribution is clearly grainy with a concentration of about 2%, whereas Co concentration is higher than 150 ppm.

Poster Session - Board 62 / 101

P62 - Impact of inflammation on tissues stores of iron

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Psoriasis is a severe inflammatory and hyper proliferative condition of human skin whose aetiology remains poorly understood. Accumulating evidence supports a role for cellular iron in cell proliferation, inflammation, and disease tolerance. Herein, we utilized nuclear microscopy techniques to quantify with cellular resolution and high sensitivity the concentration of iron in involved (psoriatic plaques) and non-involved skin of psoriatic patients.

In non-involved skin of psoriatic patients the iron distribution across skin depth showed a peak at the basal layer of epidermis. The iron concentrations at the basal layer were of the order of several hundreds of μg/g contrasting with upper epidermal regions where concentrations did not exceed a few tenths of μg/g. At the inflammatory sites (plaques) the epidermal profile of iron distribution was profoundly changed. An even distribution of iron across the epidermal depth was observed, from basal layer to the outer most layers, possibly reflecting the hyper proliferative state of psoriatic epidermis. Also, the concentrations of iron found in the epidermis of non-involved skin areas of psoriatic patients were significantly increased when compared to those in the epidermis of healthy individuals [1].

In conclusion, we found significantly increased iron deposits in the epidermis of psoriatic patients, particularly in areas of inflammation (and epidermal hyper proliferation). These findings suggest an important role for iron in the pathogenesis of psoriasis. They also raise the possibility that manipulation of iron levels in the skin may become relevant for the clinical management of psoriasis.

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Poster Session - Board 63 / 109

P63 - Evaluation of caries progression in dentin treated by fluoride-containing materials using PIGE/PIXE system

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Fluoride in some fluoride-containing materials (FCMs) was demonstrated as a great benefit for preventing dental caries. Although it is well known that fluorine (F) from FCMs penetrates directly into tooth structures, whether F penetrated from material inhibits caries progression is a matter of debate. The purpose of this study is to investigate rates of caries progression of dentin in various amount of F uptake using PIGE/PIXE system at the Wakasa Wan Energy Research Center.

The dentin sections of six extracted human teeth were prepared to being in various amount of F uptake by three kinds of FCF uptakes into dentin from FCMs were analyzed using PIGE/PIXE system. 1000 µm × D1000 µm area at the surface of dentin was scanned and two line analyses at an arbitrary position in the scanned area were subsequently performed. F and calcium distribution of specimens were obtained. The surface margin of dentin was defined as the spot containing 5% of calcium concentration in intact dentin. The average amount of F in the outer 100 µm of each specimen was obtained.

After evaluation, the specimens were immersed for 7 days in 10 ml of demineralizing solution (pH 4.5) for simulating caries attack. To estimate caries progression rates, the same area of the specimens were evaluated again using PIGE/PIXE system after caries attack treatment. The outermost surface and the innermost portion of the carious lesion were defined as the position containing 5% and 95% of the calcium concentration in intact dentin. Calcium and F distribution of specimens was calculated every 10 µm distance from the defined surface. As the caries progression rates, the area of calcium loss with the caries attack was calculated by an average of calcium loss and the depth of the caries lesion. The results suggest the negative correlation between the F uptake in dentin and its rate of caries progression. Therefore, caries progression was inhibited with increasing the amount of F uptake from FCWe will discuss the potentiality with the analysis for evaluation of preventive effects of FCMs.

Poster Session - Board 64 / 110

P64 - A new ImageJ plugin for ion beam imaging and data processing at AIFIRA facility

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Quantification and imaging of chemical elements at cellular level require the combination of techniques such as micro-PIXE, micro-RBS, STIM, secondary electron imaging associated with optical and fluorescence microscopy techniques employed prior to irradiation [1]. Such a numerous set of methods generates an important amount of data per experiment. Typically, for any acquisition the following data has to be processed: chemical map for each element present with concentration above detection limit, density and backscattered maps, mean and local spectra corresponding to relevant region of interest such as whole cell, intracellular compartment, nanoparticles, etc... These operations are time consuming, repetitive and as such could be source of errors in data manipulation.

In order to optimize this first step in data processing, we developed a new tool for batch data processing and imaging. This tool has been developed as a plugin in imageJ [2], a versatile tool for image processing that is suitable for treatment of basic IBA data operation [3]. Because ImageJ is written in Java, the plugin can be used under Linux, Mas OS X, Windows in both 32-bits and 64-bits environments [2], which may interest scientists working on open-access ion beam facilities like AIFIRA. The main features of this plugin will be presented here: listfile processing, spectroscopic imaging, local information extraction, quantitative density maps and database management using OMERO [4].

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Poster Session - Board 65 / 138

P65 - The role of microPIXE in the study of the distribution and function of trace elements in the retina and cornea of the rat eye.

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Trace elements, particularly iron, zinc and copper, are known to be important in many biochemical processes essential to the functioning of living organisIn the case of vision, zinc is known to be particularly important; deficiency can cause night blindness while excess zinc can cause retinal damage. However little is known about the spatial distribution and transport mechanisms of metals in the eye and a more detailed knowledge will contribute both to the understanding of fundamental processes in the retina and the treatment of vision dysfunction due to disorders affecting metal metabolism, for example diabetes and haemochromatosis.

In an earlier study [1] we demonstrated that microPIXE was able to observe large changes in zinc concentrations in certain layers of the rat retina in response to dark adaptation. Now we have extended this to include other metals in the retina and, for the first time, the distribution of metals in the cornea. The retina study was a collaboration where microPIXE was deployed in parallel with micro-Synchrotron XRF at the Diamond Light Source and ICP-MS analysis of whole retina. The metals of interest are at relatively low concentrations (5 - 50 ppm) in the retina, requiring long analysis times to achieve significance and an automated method was developed which permitted sequential runs of several hours on a series of samples. Spectra for each layer in the retina were extracted using masks derived using multichannel overlay maps of the major elements to provide false-colour discrimination of the different layers.

This paper outlines the methods used to obtain the microPIXE data and summarises the results. A comparison of PIXE and SR-XRF is also presented.

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Poster Session - Board 66 / 147

P66 - Evaluation of the effects of Kolaviron (Garcinia kola) on the elemental metabolism in the rat liver and kidney using PIXE, RBS and SEM.

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Poor or no antioxidant activity has been implicated in the aetiology of various pathologies. Since antioxidants are mostly derived from natural resources, the search for medicinal plants, that can either cure or alleviate ailments, has been phenomenal over the past decades. One plant, Garcinia kola, the oil of which is termed kolaviron, has been identified to have possible antioxidant activity [1]. Trace elements such as Fe, Mn, Cu, Zn and Se, form an integral part in antioxidant activity, especially in organ metabolism. In this study organ (liver and kidney) metabolism of major such as C, O, N, S, and trace elements is investigated. The kolaviron was dissolved in corn oil. Two groups (control and experimental) of Wistar rats were selected. The animals were housed in accordance with WHO animal regulations. Both groups had ad libitum access to standard rat chow and potable tap water. The control group was fed, by gavage, with 200 microlitre of the solution of kolaviron in corn oil once per day for a period of 4 weeks. Afterwards the animals were sacrificed by intraperitoneal injection. The organs were excised and homogenised into smaller parts which were freeze-dried -80°C. The freeze-dried organ was then pulverized and press into a palette. Concentrations of trace elements were determined with proton-induced X-ray emission (PIXE). PIXE was selected since with this technique concentrations downto minimum detection limits (MDLs) of parts per million (ppm) can be determined. A beam of 3 MeV protons was used for bombardment and a Be filter of 125 micrometer thickness for absorption. Backscattering spectrometry (BS) and Scanning Electron Microscopy (SEM) were used to determine the matrix composition. PIXE and BS measurements were performed simultaneously. Scanning electron microscopy was used both as complementary and supplementary to PIXE and BS. Statistical tests of $p < 0.5$ were considered significant. References

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Poster Session - Board 67 / 148

P67 - Investigation of intracellular Multilayer decomposition of Layer-by-Layer self-assembled particles by means of ion beam analysis

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Layer-by-Layer (LbL) microcarriers represent a novel group of drug delivery systeThe modular design of the polymer multilayer in nanometer thickness provides a multifunctional transport system: The step-by-step assembly of oppositely charged biopolymers on a dissolvable core allows the integration of active substances into different layers. Further functionalizations of the surface facilitate a local, targeted transport and time- controlled release of active agents into cells.

The understanding of uptake and processing of the carriers in cells and organs play a major role concerning the development of new drug delivery systeHence, the integration of a reporter for visualization of cytoplasmic processing was aimed allowing the time-dependent investigation of multilayer decomposition within cytoplasm by means of an element-sensitive method, Proton Induced X-Ray Emission (PIXE). As basis multilayer protamin sulfate and dextran sulfate has been assembled onto a CaCO3 core (5 µm). Fe3O4 nanoparticles (NP) were then integrated into the multilayer in different layer numbers. To select microcarriers which are already released into cytoplasm, cell staining was applied by LysoTracker-FITC and confocal images of the regions of interest were obtained. Release profiles were then taken by PIXE after co-incubation of the carriers with Vero cells for 24 h, 48 h, 72 h, and 120 h. Comparing the Ca profiles of the cores with the Fe profiles of the NP in the multilayer, after 72 h and 120 h an increasing difference between the profiles could be detected indicating an increasing decomposition of the multilayer components and release of the NP.

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P68 - Investigation of elemental distribution in human femoral head – studies of the Paget disease of bone

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Paget's disease of bone (PDB) is the second most common rheumatic disease and it is a condition of unknown etiology characterized by excessive and abnormal bone remodeling rate [1]. Trace elements are important indicators of bone pathology since they have an important role on bone metabolism and calcium homeostasis. Therefore, ion beam analysis techniques were applied to determine the concentration of the major and trace elements in order to find some characteristic abnormalities on pagetic bone. Bone samples removed after hip replacement in patients affected by the PDB were investigated by Particle Induced X-Ray Emission (PIXE) and Rutherford Backscattering Spectrometry (RBS). Micro X-Ray fluorescence Spectroscopy (MXFRS) was also applied to the bone analysis. For micro-PIXE and RBS analysis bone cross-sections were irradiated, under vacuum conditions, with a 2.0 MeV proton beam produced by the 2.5 MV Van de Graaff Accelerator of CTN/IST. An Oxford Microbeams-type nuclear microprobe was used (OM150 triplet system) [2], which allowed the proton beam to be focused on the sample with a spatial resolution of 3x4 μm2. The proton beam scanned a selected area of the sample in order to obtain elemental distribution maps for major elements. From these maps, line scans were defined in order to extract concentration profiles of these elements along the bone cross-sections and the concentrations of trace elements were also obtained. Both cortical and trabecular bone were analyzed. The MXRFS analysis was carried out using the Bruker's M4 Tornado spectrometer. The quantification was based on the fundamental parameters method using WinAxil code and the compare mode considering standard reference materials. Calibration against a series of standard samples has been carried out. Results were compared with concentrations referred in the literature for healthy bone of each type [3, 4, 5], and interpreted regarding the comparison and the functional role of elements in the bone metabolism.

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Poster Session - Board 69 / 43

P69 - Calibration and application of molecular imaging with MeV SIMS in positive and negative mode on plant tissue

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The introduction of new biomolecular imaging techniques into biomedical research is of high importance for improving our understanding of living organisms and their processes. These processes are often governed by large molecules, which are undergoing relocation and chemical modifications. The ability to provide their molecular maps can give us an excellent insight into the organisms' metabolism.

At the nuclear microprobe of Jožef Stefan Institute, the established sample preparation protocols and micro-PIXE method is optimized in order to provide quantitative elemental maps in biological tissue [1]. To extend our analytical capabilities with molecular imaging, a linear Time-Of-Flight mass spectrometer for MeV Secondary Ion Mass Spectrometry (MeV SIMS) spectrometer was constructed and incrementally added to the existing detection setups. A 5.8 MeV 35Cl6+ beam was focused to the size of 20 µm × 20 µm and pulsed to provide start signal for the time measurement. Ions were detected with double stack microchannel plate detector. The achieved spectrometer mass resolution of 500 is dominantly determined by the duration of the primary ion pulse.

For calibration purposes, we prepared several reference samples. The cholesterol standard solution was left to dry on Si wafer, opposed to Arginine and Glycine amino acids solution which were spin-coated on the Si wafer. We present the calibration spectra and discuss the issues connected with the beam pulsing and secondary ion detection [2]. To demonstrate the imaging capabilities, series of thin plant tissue cuttings were prepared by standard shock-freezing and freeze-drying protocol [3] and deposited on the Si wafer. By inverting the target bias voltage the system allows the detection of positively as well as negatively charged secondary ions. Maps acquired in the negative ion mode are compared with those acquired in positive ion mode. To simplify the sample positioning and to identify the sample morphology, the MeV SIMS maps were correlated with the maps acquired by heavy ion induced X-ray emission.

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P70 - Molecular imaging using micro-MeV-SIMS

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The fully ambient pressure micron lateral resolution secondary ion mass spectrometry (MeV-SIMS [1]) device at the Surrey Ion Beam Centre has recently had its analytical capabilities assessed. A 2 MV tandem accelerator and magnetic quadrupole lenses are used to accelerate and focus heavy primary ion beams through a thin exit window to exploit the electronic sputtering phenomenon in air. By simultaneously exciting and measuring characteristic X-rays both molecular and elemental analysis can be performed. A presentation will be given of data acquired of a wide range of samples, which include polymers, organics, and fine aerosol particles collected on PTFE filters.

A presentation of research being undertaken at the INFN Facility, Florence, Italy, outlines how MeV-SIMS is being applied in the field of cultural heritage. Determining the ideal conditions for performing MeV-SIMS on the precious samples that are often studied in this field is of paramount importance. A sample damage minimization campaign will determine primary ion parameters that simultaneously maximize the intact molecular ion yield as well as the heavy ion particle induced X-ray emission production cross section values. This work will offer a better understanding of the sensitivity and useful lateral resolution of MeV-SIMS.

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Poster Session - Board 71 / 48

P71 - Elemental distribution and sample integrity comparison of freeze-dried and frozen-hydrated biological tissue samples with nuclear microprobe

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The analysis of biological samples in frozen-hydrated state with micro-PIXE technique at Jožef Stefan Institute (JSI) nuclear microprobe has matured to a point that enables us to measure and examine frozen tissue samples routinely as a standard research method.

Cryotome-cut slice of frozen-hydrated biological sample is mounted between two thin foils and positioned on the sample holder. In first experiments, silicon nitride windows were used [1] and later replaced by polymer foils stretched over aluminium frames. In this way, we avoid the Si background peak from the acquired PIXE spectrum, which now enables us to detect trace elements in the low energy range from F to Cl. In addition, the use of polymer windows results in less demanding sample mounting procedure on the sample holder. The temperature of the cold stage in the measuring chamber is kept below 130 K throughout the insertion of the samples and the proton beam exposure. Matrix composition of frozen-hydrated tissue is consisted mostly of ice. Sample deterioration during proton beam exposure is monitored during the experiment.

The aim of this experiment was to determine differences and similarities between two kinds of sample preparation for micro-PIXE analysis, namely freeze-dried and frozen-hydrated sample preparation.

In the presented work, a standard micro-PIXE configuration for tissue mapping at JSI was used with five detection systems operating in parallel, with proton beam cross section of 1.0 x 1.0 μm2 and a beam current of 100 pA. The comparison of the resulting elemental distributions measured at the biological tissue prepared in the frozen-hydrated and in the freeze-dried state revealed significant differences in elemental distribution of particular elements at the cellular level due to the morphology alteration in particular tissue compartments induced by water removal in the lyophilization process.

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Poster Session - Board 72 / 150

P72-Elbowdysplasia:an unsolvedproblem

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Elbow dysplasia happens most commonly in dogs between 4 and 6 months in medium and large dogs, during the period of high growth velocity. In Portugal the disease appears frequently and affects mainly the large dogs. The term Elbow dysplasia includes different entities including fragmented medial coronoid process, osteochondrosis dissecans and incongruity of the elbow joint. All of the above can cause lameness and osteoarthritis. Fragmented medial coronoid process is now called medial coronoid process (MCP) disease since tomographic and histopathological studies showed that bone disease might be present without fragmentation of the process, this finding implies that simple fragment removal, via arthrotomy or arthroscopy, may be an incomplete treatment [1]. This disease may be caused by different factors, being genetics associated with environment influences the most important. In order to achieve a better phisiophatological knowledge to assert a more efficient and less expensive therapy, elemental and structural studies at the bone–cartilage interface in normal and diseased elbow joint affected by MCP were performed.

Micro-Proton Induced X-ray Emission (u-PIXE), Elastic Backscattering Spectrometry (RBS), micro-Proton Induced Gamma-ray Emission (u-PIGE) and Scanning Electron Microscopy (SEM) were applied for qualitative and quantitative analysis of the bone-cartilage interface removed after arthroscopy.

For micro-PIXE, RBS analysis and micro-PIGE analysis, the experiment was performed at the Van de Graaff accelerator facility of CTN/IST in Lisbon. An Oxford Microbeams-type nuclear microprobe was used (OM150 triplet system) [3], which allowed the proton beam to be focused on the sample with a spatial resolution of 3x4 μm2.

SEM observations have been carried out with backscattered electrons (BSE) using a JEOL JSM 7001F microscope equipped with an INCA Oxford Instruments EDS spectrometer for point analyses and X-ray mapping.

The combination of these techniques proved to be a viable approach for the bone-cartilage interface characterisation and the results were compared to the concentrations for healthy bone-cartilage interface. The role of major, minor and trace elements (Na, Ca, P, S, K and Zn) as well as cartilage organisation at the bone–cartilage interface, implicated in MCP disease were interpreted.

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Poster Session - Board 73 / 145

P73 - Design for RARAF nanoprobe

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We are planning to install a sub-100 nanometer single-cell irradiator at RARAF of Columbia University. It will consist of an electrostatic multiplet first stage and a superconducting solenoid final lens. In this paper I will discuss the various design calculations and expected performance of the system. Extensive ray-tracing calculations including grid-shadow studies will be reported leading to an optimized system with well understood intrinsic aberrations and potential parasitic ones.

Poster Session - Board 74 / 14

P74 - Trace element mapping of pyrite from gold deposits – A comparis on between PIXE and EPMA

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The chemical zoning of pyrite can record the evolution of mineralising fluids at widely varying P-T conditions ranging from diagenesis to medium-grade metamorphism [1,2]. If preserved, zoning can reveal growth textures, brecciation and veining, resorption and recrystallisation events, thus shedding light on the processes that contributed to ore formation [3]. Chemical zoning of sulfides is invisible in optical microscopy, but can be studied by chemical etching, high-contrast back-scattering electron images, and compositional maps (e.g. [4]). In this study, we present proton-induced X-ray emission (PIXE) and electron probe micro analysis (EPMA) data on the chemical zoning of pyrite in mineralised veins from the Sheba and Fairview gold mines, South Africa, and compare the two techniques. The compositional maps show complex distribution of trace elements, which suggest multiple events of pyrite crystallisation and gold deposition. EPMA maps show fine-scale variations reflecting growth and recrystallisation textures marked, in particular, by variations of As, Ni, and Co. Up to three events of pyrite formation have been distinguished. In PIXE maps, gold occurs both as finely-distributed and as discrete inclusions, suggesting incorporation in the pyrite structure as solid solution, and deposition as electrum inclusions, respectively. This study shows that trace element mapping of pyrite by PIXE and EPMA can provide complementary information, so that the two techniques can be used in conjunction as a powerful tool to obtain information of chemical zoning of pyrite in ore deposits.

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Poster Session - Board 75 / 15

P75 - Geological Information on Transylvanian Native Gold Using micro-PIXE

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Very small samples (hundreds of microns) of native gold from Rosia Montana (Apuseni Mts) and from Cavnic (Baia-Mare district) were scanned by micro-PIXE to obtain information on electrum structure - values of ratio Ag/(Au + Ag), on presence of Au and Ag minerals (Te and Sb compounds) – many as micro-inclusions and on other characteristic elements as Hg. Rosia Montana is one of the oldest and most interesting gold deposit with both veins and stockworks. Cavnic deposit is located in the eastern part of the Carpathian belt, one of Europe's major metallogenic provinces. Analyzed samples are from recovery obtained by inhabitants from landfills of former mining plants. Micro-PIXE analyses were performed at LNL AN2000 accelerator from Legnaro and at AGLAE Louvre Accelerator in Paris. On Rosia Montana samples we detected micrometric areas (5um X 5um) rich in Ag and Sb and without Au, Sb/Ag ratio being 1/5-6, indicating the presence of stephanite - Ag5SbS4. In one sample we detected the micro-presence of mercury, suggesting Hg could be used as a "fingerprint" for Rosia Montana gold deposit. On Cavnic-Roata samples, to investigate Au and Ag minerals, we analyzed areas of approx. 50 microns diameter. The study was focused on Sb and Te presence and on the variation of Ag/(Au+Ag) ratio which characterize electrum's metallogeny. The ratio varies from 0.221 to 0.395 - average value of 0.27, with big differences from point to point illustrating electrum's inhomogeneities. Ratio values are significantly lower than those given for neighbour mines Cavnic-Boldut 0.47-0.53, but approaching over Nistru data - 0.25 or rather Herja - 0.36. An interpretation in terms of electrum metallogeny is discussed. One micro-area revealed an important presence of Te (16657 ppm), a significant presence of Sb (2861 ppm) and an increase of Ag content (Ag=32.75% versus Au =50.05%), indicating the presence of a Ag telluride containing also Sb. The high As content (6.64%) could indicate benleonardite - Ag8(Sb,As)Te2S3 highlighted in Kremnica, Slovakia. A comparison with a micro-mineralogical study using a SEM (Scanning Electron Microscope) associated with EDX (Energy-dispersive X-ray spectroscopy) facility is presented. Some cassiterite (Sn oxide) micro-grains extracted from Valea Pianului alluvial gold samples were also analyzed and traces of Zr, Nb, Ta, W were identified.

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P76 - Combined PIXE-PIGE measurements of quartz- and topaz-hosted melt inclusions from the Ary-Bulak ongonite massif of Siberia

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Analysis of melt and fluid inclusions in minerals is an important way to understand ore formation processes. Nuclear microprobe provides direct non-destructive methods to determine their composition. Methodology related to micro-PIXE fluid inclusion analyses has been mastered by the CSIRO-GEMOC nuclear microprobe group [1] and is now available to the users of the GeoPIXE software [2].

We performed simultaneous PIXE-PIGE analyses on multi-phase inclusions (containing glass, crystals, vapour) hosted in quartz and topaz from the Ary-Bulak complex in Mongolia, complementing Raman spectroscopy, EDS-EPMA and LA-ICP-MS analyses. Previous studies of Ary-Bulak rocks have revealed the presence of silicate glass with extremely high F contents (up to 7-8 wt.%) [3]. Given the multi-phase nature of the inclusions, the PIXE-PIGE analysis was the only method possible to analyse the entire inclusions without homogenising them, and to prevent the loss of fluid. PIXE and PIGE spectra (for the detection of F) were collected using the same HPGe detector.

PIXE of topaz-hosted inclusions indicates the presence of K, Rb, Ca, As, Cs, Fe, Mn, Cu and Zn. A combination of PIXE, Raman spectroscopy and EDS allowed the identification of cryolithionite [Na3Li3Al2F14], mica, alkali-feldspar, and an Fe-Mn mineral (possibly an oxide) as daughter phases (formed after melt entrapment), and Nb-Ta-W oxides co-trapped with the melt. The obtained results revealed that the two melts with different F contents were trapped at different stages by quartz and topaz phenocrysts, and bear important implications on the magmatic evolution of F-rich silicic magmas and the deposition of Nb-Ta-W ores.

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Poster Session - Board 77 / 29

P77 - Application of IBA in the comparative analyses of fish scales as biomonitors of pollution

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Application of IBA in the comparative analyses of fish scales as biomonitors of pollution.
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Many natural resources have on a world-wide scale been contaminated by major industrial concerns. More so, most of these natural resources have been destroyed beyond remediation. However, only a few of these incidents are noticeable, such as the Exxon Valdez (1989), oil spillage, the Thor Company, 1988, Manica Province Mining, Mozambique, 2005 and mercury release into the environment. Alarming is the fact that most environmental pollution events occurred or are occurring over extended periods of time. It is thus difficult to pinpoint the sources of pollution and therefore also holding the perpetrators legally responsible. More alarming, fish found in the resources are used as dietary supplements, especially by individuals that reside near the natural resources. The scales of fish have been proven to be applicable in monitoring contamination of the natural resources. However, the morphology and chemical composition of the scale of various species differ to a significant degree. Consequently, the incorporation of contaminants into the scale structure will be different. There is a need of pilot study for contaminants which can harm the biota. The composition of the fish scales is different. To quantify the degree of incorporation onto the scale matrix we have analysed, using PIXE, RBS and SEM, the scale of four types of fish scales, that is, Pomadasys kaakan; Lutjanus gibbus; Pinjalo pinjalo and Lithognathus mormyrus. In this work we report on the viablility of using various fish scales as monitors of natural resource contamination. These compositional data will then be used to pinpoint the dates on which the pollution events occurred.

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P78 - Elemental Characterization of Gunshot Residues Generated by Brazilian Manufactured Ammunition

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The findings of GSR (waste shooting of a firearm) in the hands or clothing of a suspect is of high interest for police authorities. These residues identified by the presence of Pb, Ba and Sb in a single particle of generally spherical geometry stem from the condensation process of material from the primer, projectile, cartridge case and the gun barrel. Therefore, its composition varies according to the gun and the ammunition used.
The most accepted technique for the detection of GSR in Brazil nowadays is the Scanning Electron Microscope Energy Dispersive X-Ray Spectrometry (SEM-EDS) since it is a non-destructive technique and capable of specifying whether the characteristic elements (Pb, Ba and Sb) are in the same particle, thus discarding the possibility of contamination from another source. However, in most cases, is not possible to discriminate waste from different sources using SEM, because this technique is not sufficiently sensitive to trace elements.
The objective of this study is to establish the elemental characterization of GSR generated by ammunition made in Brazil, and determine the level of dependence of its composition with the firearm and the corresponding ammunition used during the firing. To that end, PIXE and micro-PIXE experiments were carried out.
GSR samples from the firing of a Taurus (gauge 38 special), with cartridges CBC (38 SPL+P+) were collected on paper which served as an aim. The measurements were performed at the Ion Implantation Laboratory of the Physics Institute (UFRGS). The samples were irradiated with an average current of about 100 pA and 2 nA for the micro-PIXE and PIXE experiments. Preliminary results show a moderate degree of correlation between the GSR and the ammunition.

Poster Session - Board 79 / 20

P79 - Elemental Quantification of Gunshot Residues

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Gunshot residues (GSR) constitute a relatively easy way to identify an event where a firearm was discharged. Usually, GSR are deposited near the firing event including surfaces, objects and the body of the shooter. Such particles are characterized by a mixture of elements present in the primer, projectile, gun powder, case and projectile. The aim of the present study is to measure the elemental concentration of a large number of particles ejected during the discharge of a firearm in order to establish a correlation between the GSR and the components that participate in the generation of them.

The samples of ammunition used in this work were provided by the Ballistics Section of the Department of Criminalistics (Instituto General de Perícias - IGP) located in Porto Alegre. The ammunition under study is the 38 SPL caliber, ogival lead type (CHOG), which was chosen with the criterion of being one of the most widely used both by police and by criminals.

The analysis were carried out at the microprobe facility at the Ion Implantation Laboratory. A 3 MV Tandetron accelerator delivered 3.0 MeV protons at the Oxford microprobe chamber. An Oxford triplet system focused the proton beam to 2 x 2 Dm2. Characteristics X-rays were detected by a Si(Li) detector.

The results indicate that the relative elemental concentrations vary from particle to particle. A possible correlation between particle size and relative elemental concentration is discussed.

Poster Session - Board 80 / 26

P80 - Comparative study of the charge collection efficiency decrease on Si and SiC diodes after irradiation with high energy protons

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The study of radiation effects in semiconductor electronics and detectors is fundamental to evaluate the lifetime and performance deterioration of the semiconductor devices working in high radiation environments like nuclear reactors, particle accelerators and outer space.

In this work, we present a comparative study of the charge collection efficiency (CCE) degradation on a series of Si (n-type and p-type) and SiC (n-type) diodes after irradiation with high energy protons. The CCE was determined by the Ion Beam Induced Charge (IBIC) technique using 4 MeV protons. Moreover, the transient behavior of the collected carriers was analyzed at various reverse bias voltages.

The diodes were irradiated in our cyclotron with 17 MeV protons and fluences ranging from 3.3x1011 to 3x1014 p/cm2. At this energy the proton stopping power across the samples is practically constant, leading to a uniform vacancy profile with depth.

From the analysis of the IBIC results, using the simple drift-diffusion model, the change in the diffusion length of the minority carriers has been evaluated for different proton fluences. In addition, it has been observed an increase of the leakage current with ion dose, from which the damage coefficient of the samples has been calculated.

Keywords
Si diode, SiC diode, IBIC, proton irradiation, charge collection efficiency, transient analysis

Poster Session - Board 81 / 2

P81 - Self-consistent depth profiling of GaN-based high electron mobility transistors

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High energy (MeV) ion microbeams provide unique capabilities to carry out both in-depth and lateral analysis of electronic devices and circuits [1]. However, these studies are scarce because the complex architecture of the devices (including several heterostructures) and their reduced dimensions preclude a reliable characterization in many cases. A self-consistent way to analyse these heterostructures consist in the simultaneous fitting of RBS and PIXE spectra, the so-called total-IBA approach [2], where the mass-depth ambiguity can be reduced and the sensitivity to both light/heavy elements increased. In this work we address the characterization as-processed AlGaN/GaN high electron mobility transistors (HEMTs) under such methodology. HEMTs are key pieces for the implementation of amplifiers working in the microwave range and, therefore, for the development of faster and more extended telecommunications.

RBS and PIXE experiments were carried out in the nuclear microprobe at Lisbon. In order to have an accurate analysis of the layers, the experiments were performed with 2 MeV He+ and 2 MeV H+ beams in consecutive runs. This procedure ensures a good depth resolution of the shallow layers (with He+ ions) but also a direct quantification of the atomic content of the heavy metals forming the contacts (due to the well-separated signals with H+ in the RBS spectra). In addition, the use of He+ and H+ warrants a high sensitivity to low and high energy X-ray lines in PIXE.

The individual parts of the device (wafer, source/drain, and gate) were analyzed and fitted self-consistently with NDF software [2]. The concentration and thickness of the active AlGaN layer was determined in isolated regions of the wafer. The depth-profile of the in-diffused Au/Ni/Al/Ti ohmic contact was extracted in the source/drain, including the detection of the Si3N4 passivant layer. Finally, the thickness of the Schottky Au/Ni gate contact was also quantified. The elemental RBS mapping of shallow Au and N signals at the gate was used to prove the lack of a passivant layer and, taking advantage of the well-separated Au and N energy windows, the 1µm-width gate could be resolved laterally from the near source and drain contacts. The results show that a complete (lateral and in-depth) characterization of such processed devices can be done by IBA and, consequently, be used as a quality control.

[1] Jamieson et al., NIM B 158, (1999) 628. [2] Jeynes et al., NIM B 271, (2012) 107.

Poster Session - Board 82 / 34

P82 - Hypervelocity dust impact craters on photovoltaic devices imaged by ion beam induced charge

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Hypervelocity dust has a speed of greater than 5 km/s and is a significant problem for equipment deployed in space such as satellites because of impacts that damage vulnerable components. Photovoltaic (PV) arrays are especially vulnerable because of their large surface area and the performance can be degraded owing to the disruption of the structure of the junction in the cells making up the array. Satellite PV arrays returned to Earth after service in orbit reveal a large number of craters larger than 5 micron in diameter arising from hypervelocity dust impacts. Extensive prior work has been done on the analysis of the morphology of craters in PV cells to understand the origin of the micrometeorite that caused the crater and to study the corresponding mechanical damage to the structure of the cell. Generally, about half the craters arise from natural micrometeorites, about one third from artificial Al-rich debris, probably from solid rocket exhausts, and the remainder from miscellaneous sources both known and unknown. However to date there has not been a microscopic study of the degradation of the electrical characteristics of PV cells exposed to hypervelocity dust impacts. Here we present an ion beam induced charge (IBIC) study by a 2 MeV He microbeam of craters induced on a Hamamatsu PIN diode exposed to artificial hypervelocity Al dust from a dust accelerator. Numerous 5 micron diameter craters were identified and the charge collection efficiency of the crater and surrounds mapped with IBIC with bias voltages between 0 and 20 V. At highest bias, it was found the efficiency of the crater had been degraded by about 20% compared to the surrounding material. The speed achieved in the Al dust accelerator was 15 – 20 km/s compared to 11 – 68 km/s for dust encountered in low Earth orbit. We are able to extrapolate the charge collection efficiency degradation rate of unbiased cells in space based on our current measurements and the differences in the structure of the targets.

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Poster Session - Board 83 / 37

P83 - Geiger mode mapping: a new imaging modality for focused ion microprobes

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Geiger mode detector structures in substrates intended for the construction of single atom nano-scale devices leads to a new form of deterministic ion implantation. Single photon detection is possible in a solid state detector operating in Geiger mode where a single electron-hole pair can trigger an irreversible avalanche current that is quenched with a gated bias potential. Our detectors that exhibit this phenomenon are fabricated with an architecture based on the p-i-n diode structure and operated with a transient bias voltage that activates the Geiger mode. After the avalanche breakdown triggered by ion impact and diffusion of an electron-hole pair the device is quenched by removal of the transient bias voltage which is synchronised with a beam gate. Incorporation of such a device allows the exceptional sensitivity of Geiger mode to register an electron-hole pair from sub-10 keV donor atom implants needed for the deterministic construction of shallow arrays of single atoms only 10 nm deep in the substrate required for emerging quantum technologies. In this paper we describe the development of an imaging system for a nuclear microprobe system that allows micron-scale mapping of the Geiger mode zones fabricated into substrates designed for the development of quantum computer devices from deterministic sub-10 keV P ion implants. Our system exploits the large breakdown current in the device to remove the need for a pre-amplifier. It also incorporates a fast electrostatic ion beam switcher gated by the transient device bias, duration 800 ns, with a time delay, duration 500 ns, that allows for both the ion time of flight and the diffusion of the electron-hole pairs in the substrate into the sensitive region of the device following ion impact of a scanned 1 MeV H microbeam. We demonstrate images of the micron-scale Geiger mode zones in silicon substrates engineered with this ultimate-sensitivity detector structure.

Poster Session - Board 84 / 44

P84 - IBIC mapping of anomalous polarity pulses in a multi-electrode diamond detector

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In multi-electrode detectors, the evolution of charge carrier densities generated by ionizing radiation induces a charge at all the surrounding electrodes. As the intensity and the polarity of the charge pulses depend on the geometrical, electrostatic and carrier transport properties of the device, the occurring of charge sharing effects may lead to bipolar currents and anomalous polarity pulses affecting the response of the device to ionizing radiation.
In this work, we report on the investigation in the formation of anomalous polarity pulses in a multi-electrode diamond detector with buried graphitic electrodes fabricated by a 2 MeV He microbeam.
The detector was investigated by the Ion Beam Induced Charge (IBIC) microscopy, using a 4 MeV He beam. The IBIC signals were recorded with both polarity configurations; the acquired charge collection efficiency maps evidenced the height and the position of ion incidence associated with the occurring of anomalous polarity pulses [1].
The experiment was interpreted according to a purely electrostatic model based on the Shockley-Ramo-Gunn theory [1], suitable for a general application in multi-electrode devices and detectors, providing a deeper insight in charge sharing phenomena and in the description of the observed anomalous polarity pulses.

[1] J. Forneris et al., "Measurement and modelling of anomalous polarity pulses in a multi-electrode diamond detector" EPL, 104 (2013) 28005

Poster Session - Board 85 / 67

P85 - Sharing effects in the inter-strip gap of DSSSDs

GRASSI,Laura [1]; TORRESI, Domenico [2]; DI PIETRO, Alessia [3]; FIGUERA, Pierpaolo [3]; FISICHELLA, Maria [3]; FORNERIS, Jacopo [4]; GRILJ, Veljko [5]; JAKSIC, Milko [5]; LATTUADA, Marcello [3]; MIJATOVIC, Tea [5]; MILIN, Matko [6]; PREPOLEC, Lovro [5]; Mr. SKUKAN, Natko [5]; SOIC, Neven [5]; TOKIC, Vedrana [5]; UROIC, Milivoj [5]; ACOSTA, Luis [7]

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Double Sided Silicon Strip Detector (DSSSD) are commonly used for detection of charged particles in nuclear and particle physics experiments. The front and back side surfaces of these devices are segmented into strips with a strip-to-strip separation, called "inter-strip gap".
Phenomena as charge sharing, recombination and trapping effects are observed for charged particles hitting the region nearby the inter-strip gap. Moreover, inverted polarity pulses can be produced by adjacent strips affecting the charge induced on electrodes. Previous studies of the inter-strip effects have been performed by using alpha-particles of low energies [1, 2], 3 MeV protons [2], 59.5 keV gamma-rays from a 241Am source [3], laser beams [4, 5] and, more recently, 7Li and 16O beams of energies between 6 and 50 MeV [6]. These experiments confirmed that the efficiency for full energy reconstruction is sensitive to the experimental working conditions.
The response of two DSSSDs (75 µm and 998 µm thick) was studied by the Ion Beam Induced Charge (IBIC) technique at the Ruđer Bošković Institute in Zagreb. It was found that protons of different energies hitting the inter-strip gap induce different effects on the generation of signals. Results show that the behaviour of signals induced in the inter-strip region is related with the penetration depths of the protons and the detector bias voltage. The effective inter-strip width is defined and measured and the trend is discussed.

[1] Y. Blumenfeld, et al., NIM A 421 (1999) 471.
[2] J. Yorkston et al., NIM A 262 (1987) 353.
[3] S. Takeda, et al., NIM A 579 (2007) 859.
[4] T. Poehlsen, et al., NIM A 700 (2013) 22.
[5] V. Eremin, et al., NIM A 500 (2003) 121.
[6] D. Torresi, et al., NIM A 713 (2013) 11.

Poster Session - Board 86 / 18

P86 - Micro-IBA analysis of Au/Si eutectic "crop-circles"

VITTONE,Ettore [1]; AMATO, Giampiero [2]; BATTIATO, Alfio [1]; CROIN, Luca [3]; JAKSIC, Milko [4]; SIKETIC, Zdravko [4]; VIGNOLO, Umberto [1]

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When a thin (few tens of nanometers) gold layer is deposited onto the native oxide of a silicon wafer and is annealed at temperatures greater than 600°C, peculiar circular features with a regular polygon at the centre of each circle, reminiscent of so called "alien" crop circles, can be observed.
In a recent paper [1], a systematic investigation on the role of the annealing temperature and of the Au layer thickness allowed the formation mechanism of these circular nano-structures to be properly modelled: at high temperatures, the native oxide, which is a natural diffusion barrier isolating the deposited film from the substrate, weakens and narrow channels open; the resulting Au/Si interdiffusion lead to an AuSi eutectic layer with a thickness determined by the thickness of the deposited Au film.
The central polygons occur in correspondence of the oxide channel openings and represent regions filled with a AuSi alloy which, during cooling, segregated into pure Au and Si. The shape of this central polygon is dependent on the Si wafer orientation, e.g. a square for (100) or a triangle for (111) orientations. Their typical dimensions vary in the range of few micrometers and are related to the diameters of the circles, which extend up to few tens of micrometers.

In order to get more insight into the "crop circles" formation mechanism, micro ion beam analyses were carried out at the Ruder Boskovic Institute of Zagreb on samples prepared on Si (100) substrate covered with 50 nm Au thin film and annealed at 600°C. 4 MeV C ion raster scanned beams focused down to few micrometer spot size were used to image the elemental composition of the eutectic Au/Si circles and of the Au central regular structure.

[1] T. S. Matthews et al. Phys. Rev. Lett. 108, 096102 (2012)

Poster Session - Board 87 / 61

P87 - Monitoring of the vacancy recombination rates and defect formation processes in Si and diamond during the irradiation by MeV energy ions

JAKSIC,Milko [1]; Mr. SKUKAN, Natko [1]; Mr. GRILJ, Veljko [1 1]

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Ion microprobe technique IBIC (Ion Beam Induced Charge) is established method for microscopic monitoring of the charge collection properties in semiconductor materials and devices. Here we applied IBIC to observe kinematics of defect creation processes in semiconductors. For this purpose silicon and diamond radiation detectors have been irradiated by MeV ion microbeam (Si, O, C) using different ion fluencies and wide range of ion rates. Observed changes in IBIC responses, indicate that simultaneous irradiation of material and on-line IBIC monitoring can be used to study the kinematics of defect creation processes. These were further studied by the new dual beam irradiation setup at RBI. This setup enables alternated irradiation by a damaging beam from the 6.0 MV tandem accelerator and by a probing beam from the 1.0 MV tandem accelerator. Results will be presented and discussed.

Poster Session - Board 88 / 90

P88 - Degradation of the charge collection efficiency of an n-type Fz silicon diode subjected to MeV proton irradiation

FORNERIS,Jacopo [1]; GRILJ, Veljko [2]; JAKŠIĆ, Milko [2]; RAISANEN, Jyrki [3]; SKUKAN, Natko [2]; SIMON, Aliz [4]; BARBERO, Nicolò [1]; VITTONE, Ettore [1] [1] *Physics Department, NIS research Centre and CNISM, University of Torino, via P. Giuria 1, 10125 Torino, Italy*

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The International Atomic Energy Agency (IAEA) Coordinated Research Project entitled “Utilization of ion accelerators for studying and modelling of radiation induced defects in semiconductors and insulators” is aimed to gain a deeper understanding of how different types of radiation influences the short and long term electronic properties of materials and devices. Within this project, an experimental protocol [1] has been implemented to determine the key parameters for the characterization of radiation damage effects on semiconductors. The protocol is based on the selective damage of small regions by focused ion beams at different fluences and, subsequently, on the measurement of the charge collection efficiency (CCE) degradation by means of the IBIC technique, using probing rarefied light ions.

This report describes the application of this experimental protocol to evaluate the radiation damage on an n-type Fz silicon detector developed at the Helsinki Institute of Physics . Proton beams of 1.3, 2.0 and 3.0 MeV focused down to 2 μm, were used to irradiate (0.1x0.1) mm2 areas with fluences ranging from 1011 to 3D1013 cm-2. Therefore, the radiation induced damage was probed by evaluating the degradation of the CCE at different bias voltages, using focused 4.5 MeV Li ion beaThe IBIC experiments were carried out at the nuclear microprobe facility of the Ruder Boskovic Institute in Zagreb.

At low proton fluences, the CCE behaviour as function of the proton energy, and diode polarization state is compatible with a general linear model based on the Shockley-Read-Hall recombination mechanism, whereas, at high fluences, the degradation is strongly influenced by the variation of the effective doping profile due to (hydrogen related) defects induced by irradiation.

[1] Ž. Pastuović, E. Vittone, I. Capan, M. Jakšic, Applied Physics Letters 98 (2011) 092101.

Poster Session - Board 89 / 131

P89 - Electric Force Microscopy Characterization of Ion Beam microfabricated Graphitic Channels in single crystal diamond

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The possibility of fabricating sub-superficial graphitic microchannels in diamond offers several promising opportunities in the fields of cellular bio-sensing [1] and particle radiation [2].
In this work, we present an investigation by Electric Force Microscopy (EFM) of a graphitic microchannel fabricated by using a 1.8 MeV He+ microbeam scanning over the surface of a single-crystal diamond. A linear pattern (50 Dm wide and 1 mm long) was irradiated at a fluence well above the graphitization threshold. Before the implantation process, the diamond's surface were covered with a copper layer in order to reduce the ion penetration depth without modifying the beam energy, choosing in this way the depth of the highly-damaged layer from the diamond surface. Further metal deposition of variable-thickness masks was then realized in order to implant channels with emerging end-points [3]. High temperature annealing was performed in order to induce the graphitization of the highly damaged buried region.

The presence of a conductive channel, buried at a depth of 1 Dm, was clearly evidenced, in the presence of current flowing in the channels, by maps of the electrical potential of the surface region overlying the channel. The electric potential is measured collecting for every position the potential that minimize the electrical force probed by the conductive microtip. Moreover, the electric potential profiling shows regions of opposite contrast located at different distances from the channel' endpoints. This effect is attributed to the dissimilarity between the electrical resistance path on the graphitic microchannel and the electrical resistance path on the superficial conductive layer induced by the high thermal annealing.
The results have significant implications for future fabrication of all-carbon graphite/diamond devices, both in the fields of cellular biosensing and radiatieon detection.

[1] F. Picollo et al., Advanced Materials 25 (2013) 4696
[2] J. Forneris et al., Nuclear Instruments and Methods in Physics Research B 306 (2013) 169
[3] F. Picollo et al., New Journal of Physics 14 (2012) 053011

Poster Session - Board 90 / 40

P90 – Withdrawn

Poster Session - Board 91 / 108

P91 - Nuclear Microbeam Analysis of Germanium doped GDP from Thin Film to ICF Target

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Since the National Ignition Facility (NIF) in America starts to be carryed out in 2010, for the Inertial Confinement Fusion (ICF) target, Germanium doped Glow Discharge Polymer (GDP) have become the preferred target material[1]. The nondestructive measurement of elements content in the ICF target has become a significant work in recent years. The paper presents the compositional and distributional results of the Germanium doped GDP analysis from the thin film to ICF target investigated by the Rutherford Backscattering Spectroscopy (RBS) combined with the Particle induced X-ray emission(PIXE) and the Elastic Recoil Detection Analysis(ERDA). The samples are thin film with 13-15 um thickness and ICF target with 500-2000 um radius. The calibration and geometrical arrangement in the analysis from the thin film to spherical target should be carefully considered in order to acquire the accurate result. In the work, the uniformity of the ball is shown and the ratio of carbon, hydrogen and germanium has been measured. The result of ratio value is in good agreement with the combustion method. Besides, the difference of the composition from thin film to ball is also disscussed. Nuclear microbeam analysis is one of ideal methods to assess the ICF target qualify.

Poster Session - Board 92 / 24

P92 - Investigation of Deep Levels in Silicon Carbide using Ion-Induced Charge Transient Spectroscopy

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Silicon Carbide (SiC) is a promising candidate of particle detectors for its high radiation resistance. For such a SiC application to particle detectors, it is necessary to clarify the relationship between radiation induced deep-level defects and degradation factors in thier device performance. However high resistivity of SiC prevent to investigate these defects by conventional deep level transient spectroscopy (DLTS). On the other hand, there is an arise of charge transient spectroscopy using charge induced by particle irradiation into Si and SiC detectors [1,2]. In this study, we have applied charge transient spectroscopy using charged particles such as alpha particles from an americium (241 Am) radio isotope and also heavy ion microbeams (the details of evaluation techniques are presented in elsewhere [2,3]) to the measurement of the deep-level defects in SiC particle detectors, in order to investigate effects of radiation-induced damage on their Charge Collection Efficiency (CCE) . Firstly, 4H SiC Schottky Barrier Diode (SBD) particle detectors were irradiated with 1 MeV electrons or 3 MeV protons to create the radiation damage inside of the detector. Then irradiated and non-irradiated 4H SiC SBD detectors were both sequentially probed with charge transient spectroscopy systems with a probe of 5.5 MeV alpha particles and focused heavy ions of 10.5 MeV oxygen. The details of the charge transient spectra obtained from SiC SBD detectors with defects created by different radiation exposures will be discussed in the presentation.

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Poster Session - Board 93 / 46

P93 - Study of the oxygen depth profile of welded joints using PIGE, RBS and NRA techniques at various deuteron energies

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The selection and optimization of the proper welding technology is a key factor in the production of steel structures, for which, the understanding and specific utilization of the physical processes is necessary. The oxygen content of the applied shield gas is one of the most important parameter in welding process, which significantly influences the mechanical properties of a welded joint.
In order to study the oxygen content and characterize the oxygen depth profile on the surface of welded joints, Particle Induced Gamma-ray Emission (PIGE), Rutherford Backscattering Spectrometry (RBS) and Nuclear Reaction Analysis (NRA) were used. The measurements were carried out on several deuteron energies (1.0-1.8 MeV) by the scanning nuclear microprobe of MTA Atomki using HPGe and ion-implanted silicon detectors. From oxygen and carbon elemental maps, suitable regions were chosen for depth profile determination. Instead of using standards the oxygen and carbon depth profiles of the surface were calculated on the basis of the gamma-ray and particle production cross sections [1,2].
This study demonstrates the advantages of the deuteron induced ion beam analytical techniques by nuclear microprobe, utilized in oxygen depth profiling in steel.
The work was supported by the IAEA Coordinated Research Project “Reference Database for Particle-Induced Gamma-ray Emission (PIGE) Spectroscopy” (Contract No. 16967/R1).

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P94 - Precision differential cross sections of the $^{12}C(p,p)^{12}C$ elastic scattering in the vicinity of the resonance at 1.726 MeV

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More precise and based on the previously published data, differential cross sections of the 12^C(p,p)12^C elastic scattering in the vicinity of the resonance at 1.726 MeV for the 170° scattering angle were obtained in present work. New data were compared with the similar literary cross section values as well as theoretical excitation function estimations. A shape of our excitation function fits very good experimental data from [R.Amirikas, D.N.Jamieson, S.P.Dooley. NIMB77 (1993) 110-116] and has some shift in energy with respect to our data. As an example of the cross section application, a result of the analysis of thin film structure with multiple element composition (H, C, N, O, Ti, Cu, soda lime glass substrate (SLG)) is shown. The sample is a thin film TiO based coating on the SLG matrix obtained using Ion Beam Assisted Deposition (IBAD) of Ti^n+ (n=1; 2; 3) ions, namely, simultaneous Ion Deposition and MEVVA Ti Ion Implantation. Total IBA analysis of thin film structure was performed using three complimentary ion beam techniques such as RBS, Resonant Elastic Back Scattering of 1^H^+ and 4^He^+ ions, and ERDA. A buried C_3N_4 stoichiometric sub-layer in the TiO coating and gaussian like H depth profile in coating-matrix interface were observed. Additionally, scratch test reveals an increase in the micro hardening of the coating with respect to the original SLG matrix. Most probably, TiO and trace CuO composite based hydrogen diffusion barrier on SLG substrate was formed after IBAD treatment.

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P95 - Tomographic examination of ion tracks by ion microbeam energy loss analysis

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A nondestructive tomographic approach (i.e., ion microprobe energy loss spectroscopy) for the study of density inhomogeneities (e.g., etched nuclear tracks) in thin solid films (e.g., polymer foils) is introduced. In the method, the tomographic data are obtained by analysis of the energy loss of ions passing through the spatial micro-inhomogeneities (vacancies or densifications) and registering by solid state detector placed behind the target foil. The energy spectra from the scanning areas (with the size of ~ 1 or more micrometers) are subjected to the MC simulation - anlysis by a set of codes corresponding to the various shape patterns. The optimal fit of the experimental data is obtained by gradual variation of the microobject spatial parameters. The method enables determination of the 3D form of the pore, study of dynamic processes, such as pore gradual evolution in the etching procedure, or filling of the pore hollow with materials of different densities. For randomly-shape material inhomogeneities, the method has to carry out a set of energy loss tomographic scans from different inclining angles. Here, an application of ion microprobe in tomographic examination of ion tracks (process of their evolution and filling) is presented and discussed.

Poster Session - Board 96 / 111

P96 – Withdrawn

Poster Session - Board 97 / 82

P97 - Imaging of Li Distribution in Li ion batteries by direct elemental detection tecnique of PIGE and NRA combined with proton microbeam at TIARA

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The ion microbeam analysis is a powerful tool to characterise the distribution and the behaviour of specific elements in the three dimensional space in materials. For the development of advanced lithium ion batteries, especially for large scale applications such as vehicle and energy-storage uses, precise diagnostics of Li especially in the electrode material is strongly desired. The distribution of light elements, in particular Li in Li ion battery materials, have been characterized by combining nuclear reaction analysis (NRA) and particle induced gamma-ray emission (PIGE) techniques. NRA and PIGE measurements were carried out by using the {^{6}Li(p,α)^{4}He} and the {^{7}Li(p,p'γ)} nuclear reactions, respectively. The characterization of other elements different than Li was simultaneously done, by using particle-induced X-ray emission technique (PIXE). Measurements were done at the the proton microbeam system with the spatial resolution of 1 μm at Takasaki Ion Accelerators for Advanced Radiation Application (TIARA) of Japan Atomic Energy Agency (JAEA). The Li distribution was analysed for specially fabricated electrode samples containing micro particles based on metal oxides such as Li_xNiO_2 ($0.75 \leq x \leq 1.0$) [1]. Although some other advanced Li- ion batteries cathodes materials were also investigated. The Li distribution is studied as a function of charge conditions and of materials parameters. This paper describes the analysis system dedicated to the Li-ion battery characterization and the main results of some of the experiments.

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(2012) 79–84

Poster Session - Board 98 / 146

P98 - An observation of the open pore formation and ion guiding effect in corundum implanted with Ti ions and irradiated with 90 MeV Kr ions

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Open pore formation and ion guiding effect was detected in a modified polycrystalline corundum (α- Al_2O_3). Modification was done via ion implantation (MEVVA implantation) with irradiation using swift heavy ions (SHI irradiation). MEVVA implantation was performed at energies of 50-150 keV, fluence of about 10^17 Ti^n+/cm^2 (n=1,2,3), room temperature (RT). Parameters of SHI irradiation are energy of 90 MeV, fluence of about 10^12 and 10^14 Kr^15+/cm^2, residual vacuum of (6-8)×10^-4 Pa, RT. Ti, Al, O were depth-profiled using RBS and ResBS ^4He^+ ions (elastic resonance in scattering of ^16O(^4He,^4He)^16O at energy in the vicinity of 3.045 MeV). SEM and AFM microscopy was applied for an analysis of the surface morfology. MEVVA as-implanted samples were thin-film layered composite, electrically conducting structures at a dielectric surface with nanoparticles of metallic Ti nanoparticles after SHI irradiation. But at the same time, considerable oxygen enrichment (by half) of the buried modified layers was observed. Last observation is in a good agreement with one of the mechanism of surface oxidation of Fe thin films subjected to SHI irradiation under different regimes described in literature. At normal incidence of a ^4He^+ probe beam onto the sample surface of the irradiated corundum a spectrum energy shift was observed in a resonant yield of ^4He back-scattered on nuclei of ^16O in the buried layers. Similar shift was not observed at grazing incidence of the beam onto the surface. Energy shift may be explained by a so-called “ion guiding-effect” described in detail, for example, in one of the multiple recent reviews in the literature. AFM analysis revealed a formation of “hillocks” of 250 nm in diameter and about 50 nm in height on some areas of the MEVVA as implanted samples. SEM analysis showed a presence of the open pores on irradiated sample surface (MEVVA+SHI) that are charged under an analysing electron beam bombardment. The results of SEM microscopy unexpectedly coincided with observation data of one the experimental work in literature where open stoichiometric pores were observed in an ion crystal (NaCl) after being subjected to MeV electrons at high dose and the simple mechanism of their formation (similar to for Al_2O_3) is theoretically proved. Obtained results suggest that MEVVA+SHI processing may form oriented perpendicular to surface closed cylinder shaped pores of few nm in diameter and several tens of nm in lenght.

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P99 - Effect of Cobalt Doping on Titanium Dioxide Thin Film Prepared by Ion Layer Gas Reaction method

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Ion layer gas reaction (ILGAR) method was used to deposit Cobalt doped Titanium dioxide thin films from precursor salts of Cobalt Chloride and Titanium Isopropoxide respectively. The Cobalt concentration in Titanium dioxide was varied between 5% and 50% in the precursor salts. The effect of Cobalt doping of Titanium dioxide thin films was investigated by Rutherford backscattering spectroscopy where the depth profiles were determined from the simulated spectra. The activation energies and exponential pre-factors of Cobalt diffusion determined. Cobalt occupies Ti sites leading to a change in the stoichiometry and therefore variation of thickness of the film.

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