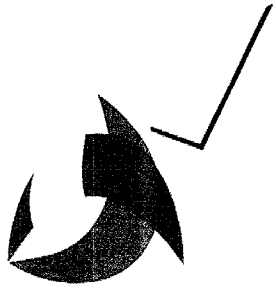
7th INTERNATIONAL CONFERENCE ON NUCLEAR MICROPROBE TECHNOLOGY & APPLICATIONS

Cité Mondiale, Bordeaux, France - September 10-15 2000

FINAL PROGRAM & ABSTRACTS

Conférence Internationale sur

la Technologie
et les Applications
des Microsondes Nucléaires.

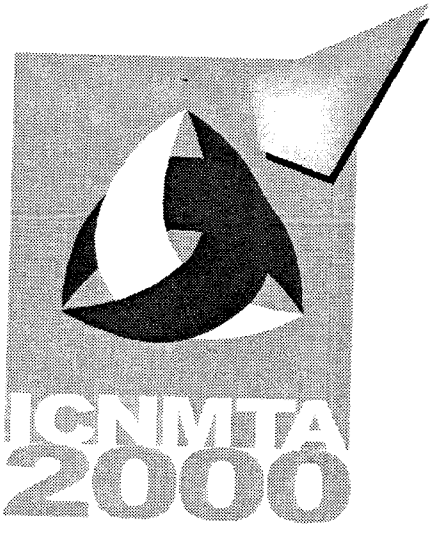


ICNMTA 2000

7th International Conference on Nuclear Microprobe Technology and Applications

Cité Mondiale, Bordeaux, France 10 – 15 September 2000

7ème Conférence Internationale sur la Technologie et les Applications des Microsondes Nucléaires



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Dear colleague,

we have pleasure in welcoming you in Bordeaux to attend the 7th International Conference on Nuclear Microprobe Technology and Applications (ICNMTA-2000).

The 7th ICNMTA is the first International Symposium of this series in the 21st Century. Therefore, it has a particular significance for the scientists coming from all over the world. This Symposium must give an up-to-the-date report on the continuously advancing applications and development of microbeam technology. The Symposium brings together internationally recognised scientists to conduct plenary and poster sessions, in a format that has already ensured the success of the latest conference editions, held respectively in Santa Fe (USA) in 1996 and Stellenbosch (South Africa) in 1998. After three meetings held outside Europe, the ICNMTA comes back on our continent. Thus we expect this event is going to stimulate fruitful discussions and new scientific projects, and to promote interdisciplinary approaches in our community.

The conference is being held at the "Cité Mondiale". This conference centre is located in the port of Bordeaux historical hearth, known as the "port de la lune" because it displays its recently renovated 18th century facades along a curve of the Garonne river. You will be able to recognise this symbol everywhere in Bordeaux, under the form of three overlapping crescents constituting the arms of the city. We have chosen to reproduce this symbol in the logo of the conference.

It was the aim of the organising committee to let young scientists speak. About 20 young European colleagues have been invited thanks to the financial support of the European Community, a large part of them having an oral contribution. We have entrusted them, together with young scientists from other continents, with the task of presenting most of "technical invited" contributions. We expect it is an efficient way to promote their career, the future of our discipline and also to ensure the scientific renewal for next ICNMTA conferences.

We have made particular efforts to propose the attendees and their accompanying persons exciting social activities, a part of them connected of course to the vineyards, but also to the cultural and architectural heritage of this region. We cordially invite you to enjoy the beauty of Aquitaine, its hospitality, its people, its food, its natural and historic attractions.

We would like also to take this opportunity to thank our sponsors, the members of the Organising Committee and all peoples who contributed to the organisation of the 7th ICNMTA, expecting that you will find such event to be a scientifically rewarding experience.

M. Bonnin-Mosbah & Ph. Moretto
Chairs of the ICNMTA2000 conference

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Cher collègue,

Nous avons le plaisir de vous accueillir à Bordeaux pour participer à la 7ème Conférence Internationale sur la Technologie et les Applications des Microsondes Nucléaires.
ICNMTA-20000 est le premier colloque de cette série ayant lieu au 21ème siècle. Il revêt donc une signification particulière dans l'esprit des scientifiques venant du monde entier. Ce colloque doit faire un bilan des applications sans cesse croissantes des microsondes nucléaires et mettre en évidence les avancées technologiques marquantes. Il réunit pour cela des scientifiques internationalement reconnus que ce soit au cours de sessions plénières ou dans le cadre de sessions posters et d'ateliers; un format qui a déjà assuré le succès des précédentes éditions qui se sont tenues à Santa Fe (USA) en 1996, puis Stellenbosch (Afrique du Sud) en 1998. Après trois conférences organisées en dehors de l'Europe, ICNMTA revient sur notre continent. Nous espérons que cet événement va être l'occasion de favoriser les contacts menant à de nouveaux projets et promouvoir une approche interdisciplinaire dans notre communauté.

La conférence se tient à la "Cité Mondiale". Ce centre de conférences est situé dans le cœur historique du port de Bordeaux, connu sous le nom de "port de la lune" car il étale ses magnifique façades du 18ème récemment rénovées le long d'une courbe de la Garonne. Vous pourrez retrouver ce symbole partout dans Bordeaux sous la forme des trois croissants entremêlés qui constituent les armoiries de la cité. Nous avons choisi de reproduire ce symbole sur le logo de la conférence.

Le but des organisateurs était de donner la parole aux jeunes scientifiques. Une vingtaine de jeune collègues européens ont été invités grâce au soutien financier de la Communauté Européenne; beaucoup d'entre eux ayant une présentation orale. Nous leur avons confié, ainsi qu'à d'autres jeunes chercheurs non-européens, la plupart des présentations "technical invited". Nous espérons que ce sera un moyen efficace de promouvoir leur carrière tout en assurant le futur de notre discipline et également d'assurer le renouveau scientifique dans la perspective des prochaines éditions d'ICNMTA.

Nous avons fait un effort particulier pour proposer aux participants et à leurs compagnons un programme social éclectique. Une partie est liée bien sur au vignoble bordelais mais également à l'héritage culturel et architectural de la région. Nous vous invitons cordialement à profiter du charme de l'Aquitaine en appréciant non seulement ses attraits naturels et historiques mais également son hospitalité et sa gastronomie.
Nous voulons enfin remercier nos sponsors, les membres du comité d'organisation ainsi que tous ceux qui ont contribué de près ou de loin à l'organisation de la 7ème conférence ICNMTA en espérant que cet évènement constituera pour vous une expérience marquante.

M. Bonnin-Mosbah & Ph. Moretto
Co-présidents d'ICNMTA2000

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Conference Address *(adresse de la conférence)*

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Conference Location *(lieu de la conférence)*

Cité Mondiale
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Conference Schedule

Monday, September 11

8h30-9h00 Opening Session

Session MF : Microprobe Facilities

Chair: G.W. Grime & K. Traxel

9h00-9h35 MF-O1 : Invited Lecture
NEW GENERATION NUCLEAR MICROPROBE SYSTEMS
D. N. JAMIESON

9h35-9h55 MF-O2
THE NEW CSIRO-GEMOC NUCLEAR MICROPROBE: FIRST RESULTS, PERFORMANCE AND RECENT APPLICATIONS
C. G. RYAN

9h55-10h15 MF-O3
THE MUNICH MICROPROBE SNAKE: FIRST RESULTS USING 20 MEV PROTONS AND 90 MEV SULFUR IONS
G. DOLLINGER

10h15-10h35 Coffee Break

10h35-10h55 MF-O4 Technical Invited
EXPERIENCE WITH THE HIGH RESOLUTION BEAMLINE ON THE OXFORD PROTON MICROPROBE FACILITY
G.W. GRIME

10h55-11h15 MF-O5
DEVELOPMENT OF AN AUTOMATED SINGLE CELL IRRADIATION SYSTEM COMBINED WITH A HIGH-ENERGY HEAVY ION MICROBEAM SYSTEM
T. KAMIYA

11h15-11h35 MF-O6
A NOVEL ULTRA-SHORT SCANNING NUCLEAR MICROPROBE WITH EXTERNAL BEAM: DESIGN AND FIRST RESULTS
S. LEBED

11h35-11h55 MF-O7
THE HEAVY ION MICRO PROJECTION SETUP AT BOCHUM
A. STEPHAN

11h55-12h15 MF-O8
MEDIUM ENERGY NUCLEAR MICROPROBE WITH ENHANCED SENSITIVITY FOR SEMICONDUCTOR PROCESS ANALYSIS
J. TAJIMA

13h30-15h00 Poster Session

Session AT: Analysis Techniques

Chair : K. Malmqvist & V. Prozesky

15h00-15h20 AT-O1 Technical Invited
3D HYDROGEN MICROSCOPY BY PROTON PROTON SCATTERING
P. REICHART

15h20-15h40 AT-O2 Technical Invited
THE AUTOMATIC ANALYSIS OF RUTHERFORD BACKSCATTERING SPECTROMETRY SPECTRA
J. PADAYACHEE

15h40-16h00 AT-O3
CHEMICAL STATE ANALYSIS OF Cu, Cu_2O AND CuO WITH WDX USING AN ION MICRO BEAM
K. KAWATSURA

16h00-16h20 AT-O4
NUCLEAR MICROPROBE ANALYSIS OF A GAS-SOLID INTERFACE
K. R. PADMANABHAN

16h20-16h40 AT-O5
LIGHT DETECTION WITH SPECTRAL ANALYSIS AT THE LEGNARO NUCLEAR MICROCROPROBE: APPLICATIONS IN MATERIAL AND EARTH SCIENCES
E. VITTONE

16h40-17h00 Coffee Break

Session IT: Imaging Techniques

Chair : M.B.H. Breese & M. Cholewa

17h00-17h35 IT-O1 Invited Lecture
ION-INDUCED EMISSION MICROSCOPIES
B. L. DOYLE

17h35-17h55 IT-O2 Technical Invited
THREE DIMENSIONAL ION MICROTOMOGRAPHY
A. SAKELLARIOU

17h55-18h15 IT-O3
IMAGING OF CHARGE TRANSPORT IN POLYCRYSTALLINE CVD DIAMOND USING IBIC MICROSCOPY
M.B.H. BREESE

18h15-18h35 IT-O4
IMAGING NICKEL SEGREGATION IN SYNTHETIC DIAMONDS USING IONOLUMINESCENCE (IL) AND PROTON INDUCED X-RAY EMISSION (PIXE)
A. A. BETTIOL

18h35-18h55 IT-O5
CHANNELING CONTRAST MICROSCOPY ON LATERAL EPITAXIAL OVERGROWN GaN FILM
E.J. TEO

Tuesday, September 12

Session MIBM: Micro-Ion Beam Modification of Materials

Chair : B. Doyle & F. Watt

8h30-8h50 MIBM-O1 Technical Invited
HIGH PRECISION 3D METALLIC MICROSTRUCTURES PRODUCED USING PROTON BEAM MICROMACHINING
J.A. VAN KAN

8h50-9h10 MIBM-O2 Technical Invited
OPTICAL PROPERTIES OF nc-Si MICROSTRUCTURES IN SiO_2
J. HEITMANN

9h10-9h30 MIBM-O3
CHARACTERIZATION OF MICRO-STRUCTURES FORMED ON MeV ION-IRRADIATED SILVER FILMS ON Si(111) SURFACES
B. ROUT

9h30-9h50 (to be scheduled)

9h50-10h10 Coffee Break

Session ME: Microelectronics

Chair : M. Takai & M. Jakšić

10h10-10h45 ME-O1 Invited Lecture
IBIC CHARACTERISATION OF DEFECT STRUCTURES IN POLYCRYSTALLINE SILICON
M. JAKŠIĆ

10h45-11h05 ME-O2 Technical Invited
INVESTIGATION OF BODY-TIE DESIGN ON ION BEAM INDUCED CHARGE COLLECTION IN SILICON-ON-INSULATOR FETS USING THE SANDIA NUCLEAR MICROPROBE.
D. S. WALSH

11h05-11h25 ME-O3
ION BEAM INDUCED CURRENT (IBIC) ANALYSIS OF HIGH POWER DEVICES
T. OSIPOWICZ

11h25-11h45 ME-O4
DIFFUSION TIME RESOLVED ION BEAM INDUCED CHARGE COLLECTION STUDIES ON STRIPE-LIKE JUNCTIONS USING HEAVY-ION MICROBEAMS
F.D. McDANIEL

11h45-12h05 ME-O5
SUPPRESSION OF FLOATING BODY EFFECTS IN SOI MOSFET STUDIED USING NUCLEAR MICROPROBES
S. ABO

13h30-16h15 **Workshops**

Session MS: Applications in Material Sciences

Chair : D.N. Jamieson & U. Wätjen

16h15-16h35 MS-O1 Technical Invited
INVESTIGATING NON-UNIFORM SURFACES OF THIN SIO_2 FILMS PRODUCED BY HIGH-TEMPERATURE NITRIDATION EXPERIMENTS WITH ION MICROSCOPY
A. MARKWITZ

16h35-16h55 MS-O2 Technical Invited
MICRO-RBS ANALYSES OF SURFACE TOPOGRAPHY OF Bi FILM PREPARED BY PULSED LASER DEPOSITION
A. SIMON

16h55-17h15 MS-O3
NUCLEAR MICROPROBE MEASUREMENTS ON LABELED POLYMER GRATINGS
C. M. LEEWIS

17h15-17h35 MS-O4
CHARACTERISATION OF Ti:SAPPHIRE WAVEGUIDES USING THE MELBOURNE NUCLEAR MICROPROBE
L. D. MORPETH

Wednesday, September 13

Session BM: Applications in Biology and Medicine

Chair : U. Lindh & M. Simonoff

8h15-8h50 BM-O1 Invited Lecture
THE IMPACT OF MICROBEAMS IN RADIATION BIOLOGY
M. FOLKARD

8h50-9h25 BM-O2 Invited Lecture
IS IRON A RISK FACTOR IN ATHEROSCLEROSIS?
F. WATT

9h25-9h45 BM-O3
CYTOLOGICAL AND HISTOLOGICAL STRUCTURES IDENTIFICATION WITH THE TECHNIQUES IBIL AND SEIM IN MICROPIXE ANALYSIS
P. ROSSI

9h45-10h05 BM-O4
$Na_4Lu(III)HDOTP$ AS EXRA-CELLULAR MARKER IN INDIRECT MAPPING OF INTRA-CELLULAR ION LEVELS IN HEART TISSUE VIA MICROBE-PIXE
P.H.A. MUTSAERS

10h05-10h25 BM-O5
ELEMENTAL ANALYSIS OF CELLULAR SAMPLES BY IN AIR MICRO-PIXE
K. ISHII

10h25-10h45 Coffee Break

10h45-11h05 BM-O6
NUCLEAR MICROBEAM IDENTIFICATION OF FUNCTIONAL NON-ORGANIC ELEMENTS IN PROTEINS IN EITHER LIQUID OR SINGLE-CRYSTAL FORM.
E. F. GARMAN

Session EPS: Applications in Earth and Planetary Sciences

Chair : C. Ryan & A.Z. Kiss

11h05-11h40 EPS-O1 Invited Lecture
NUCLEAR MICROPROBE ANALYSES OF LIGHT AND VOLATILE ELEMENTS (C, F, Br) IN MAGMAS
N. METRICH

11h40-12h00 EPS-O2 Technical Invited
MINERAL SPECIFIC TRACE ELEMENT CONTENTS OF INTERPLANETARY DUST PARTICLES
C. WIES

12h00-12h20 EPS-O3
TRACE ELEMENT ANALYSES OF APATITE FROM FOSSIL BONES OF MARINE ORGANISM AND ITS GEOLOGICAL APPLICATION
D. HABERMANN

12h20-12h40 EPS-O4
ANALYSIS OF URANIUM DISTRIBUTION IN ROCKS BY μ-PIXE
T. OHNUKI

12h40-13h00 EPS-O5
NUCLEAR MICROSCOPIC TECHNIQUES IN THE STUDIES OF HYDROGEN CHEMISTRY AND DYNAMICS IN DIAMOND
E. SIDERAS-HADDAD

Thursday , September 14

Session EB: Applications in Environment and Botany

Chair : W. J. Przybylowicz & A. Markwitz

8h30-9h05 EB-O1 Invited Lecture
ELEMENTAL MICROANALYSIS IN BOTANY: NUCLEAR MICROPROBE AND COMPETING METHODS
W.J. PRZYBYLOWICZ

9h05-9h25 EB-O2 Technical Invited
MANGANESE PROFILES IN FRESHWATER MUSSEL SHELLS
R. SIEGELE

9h25-9h45 EB-O3
WEATHERING AND METAMICTIZATION OF ZIRCON : NEW CONSTRAINTS FROM MICRO-PROBE MEASUREMENTS.
E. BALAN

9h45-10h05 EB-O4
NUCLEAR MICROPROBE ANALYSIS OF LICHEN SURFACES
B. M. CLARK

10h05-10h25 EB-O5
CHARACTERISATION OF ZINC IN A DREDGED SEDIMENT DEPOSIT AND IN THE SUBSOIL BY COUPLING μPIXE, μRBS, AND μEXAFS
M.P. ISAURE

10h25-10h45 Coffee Break

10h45-12h30 Workshops

13h30-16h45 Posters

Friday, September 15

Session AA: Applications in Art and Archaelogy

Chair : G. Demortier & J.C. Dran

8h30-8h50 AA-O1 Technical Invited
EXTERNAL MICROBEAM SETUP AT THE CNA (SEVILLA) AND ITS APPLICATION TO THE STUDY OF TARTESIC JEWELLERY
M. A. ONTALBA SALAMANCA

8h50-9h10 AA-O2 Technical Invited
ANALYSIS OF PREHISTORIC POTTERY FINDS FROM THE BALATON REGION, HUNGARY BY MICRO-PIXE TECHNIQUE
Z. ELEKES

9h10-9h30 AA-O3
ALBEMARLE IRON WORKS (1771-1772): WHY DID THIS OPERATION FAIL?
C .P.SWANN

9h30-9h50 AA-O4
PHOSPHORUS LOCALISATION AND QUANTIFICATION IN ARCHAEOLOGICAL IRON ARTEFACTS BY MICRO PIXE ANALYSES
P. DILLMANN

9h50-10h10 AA-O5
PIXE ELEMENTAL MAPPING ON ORIGINAL MANUSCRIPTS WITH AN EXTERNAL MICROBEAM. APPLICATION TO MANUSCRIPTS DAMAGED BY IRON GALL INK CORROSION
C REMAZEILLES

10h10-10h30 Coffee Break

Session ALT: Alternative Techniques

Chair : R.D. Vis & M. Bonnin-Mosbah

10H30-11H05 ALT-O1 Invited Lecture
HIGH RESOLUTION NUCLEAR AND X-RAY MICROPROBES AND THEIR APPLICATIONS IN SINGLE CELL ANALYSIS
M. CHOLEWA

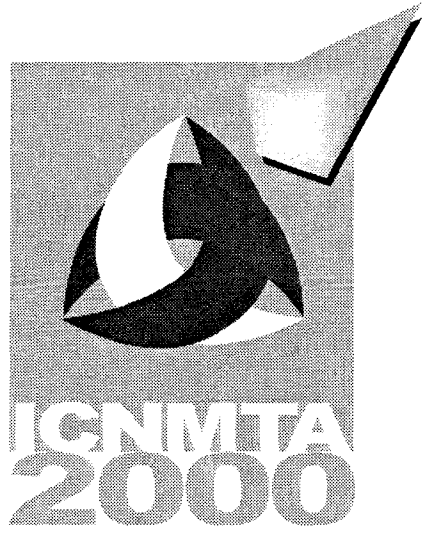
11h05-11h25 ALT-O2 Technical Invited
ANCIENT ARTEFACTS AND MODERN ANALYTICAL TECHNIQUES - LASER ABLATION ICP-MS EXEMPLIFIED ON ANCIENT GOLD COINS
S. A. JUNK

11h25-11h45 ALT-O3
COMBINED USE OF XPS, FTIR, NMA AND TEM TO STUDY SURFACE REACTIVITY OF HYDROXYAPATITE
F. MERCIER

11h45-12h05 ALT-O4
PROGRESS OF BEIJING SYNCHROTRON RADIATION X-RAY FLUORESCENCE MICROPROBE ANALYSIS
Y. HUANG

Closing Session :

15h00 CONCLUDING REMARKS
G. DEMORTIER



Abstracts
Oral Contributions

Session MF : Nuclear Microprobe Facilities

MF-O1

NEW GENERATION NUCLEAR MICROPROBE SYSTEMS

David N. Jamieson, The Microanalytical Research Centre, The School of Physics, The University of Melbourne, Parkville, 3010, Australia

Over that past 20 years, the minimum probe size for nuclear microscopy has decreased slowly below 1 micrometre. No breakthroughs in nuclear microprobe design have been forthcoming that have produced dramatic improvements in spatial resolution. The difficulties of reducing the probe size have been well recognised in the past. The most significant barriers have included:

- geometrical constraints in the specimen chamber due to the requirements of the detectors of the induced radiation or particles,
-limits to the strength of the focusing fields,
-limits due to lens focusing aberrations in particular chromatic and spherical aberration, the influence of stray (parasitic) fields or vibrations
-limitations in the brightness of the ion source.

Over the past 5 years it has become clear that some of these constraints may not be as limiting as first thought. For example, chromatic aberration clearly is not as significant as first implied from first order calculations. This paper reviews this and other constraints in view of the increased understanding of the past 5 years and looks at several new approaches, presently being evaluated in Melbourne and elsewhere, on how to progress beyond the original barriers. These approaches include modified RF ion sources for improved beam brightness and exploitation of the relaxed constraints on some lens aberrations allowing the use of high demagnification probe forming lens systems.

MF-O2

THE NEW CSIRO-GEMOC NUCLEAR MICROPROBE: FIRST RESULTS, PERFORMANCE AND RECENT APPLICATIONS

Chris G. Ryan[1,3], David N. Jamieson[2], William L. Griffin[3,1], Gary Cripps[1] and Roland Szymanski[2], [1] CSIRO Exploration and Mining, Australia, [2] School of Physics, Microanalytical Research centre, University of Melbourne, Australia, [3] GEMOC National Key Centre, Macquarie University, Australia.

Design plans and theoretical modeling for a new nuclear microprobe (NMP), targeted at high-resolution, high sensitivity analysis of geological samples, were unveiled at the previous ICNMTA conference in Cape Town. Central design goals of the NMP include high demagnification, low aberration and a slow growth in spot-size with increasing current. The instrument became operational in 1999 and this paper discusses its features, performance and some recent applications in geology.

Central features of the new NMP include: (1) a quadrupole quintuplet lens system with a demagnification of 67 and a unique quadrupole shape that permits a 80 mm working distance and with cut-outs in the return yoke for detectors; (2) a close detection geometry for X-ray , γ-ray and particle detectors; (3) a high quality Questar normal-viewing microscope, coaxial with the beam; (4) computer controlled beam-shaping and 5-axis sample stage; (5) scanning for imaging to >5 x 2.5 mm^2; and (6) a vibration isolated mounting.

The design goals have been successfully achieved. On the first day of operation, a spot-size of 1.3 μm was obtained at a beam current of 0.5 nA, suitable for fluid inclusion analysis and imaging. The spot-size grows to just 1.8 μm at 10 nA (3 MeV protons), despite the low brightness of our tandem accelerator (~1 $pA.\mu m^{-2}.mrad^{-2}.MeV^{-1}$). This beam current is ideal for geological samples with PIXE detection limits down to 0.2 ppm achieved in quantitative, high resolution, trace element images.

MF-O3

THE MUNICH MICROPROBE SNAKE: FIRST RESULTS USING 20 MEV PROTONS AND 90 MEV SULFUR IONS

Günther DOLLINGER, Gerd DATZMANN, Christian GOEDEN, Andreas HAUPTNER, Hans-Joachim KÖRNER, Patrick REICHART, Oliver SCHMELMER. Technische Universität München, Physik Department E12, James-Franck-Strasse, D-85747 Garching, Germany

The scanning ion microprobe SNAKE (Superconducting Nanoprobe for Applied Nuclear (Kernphysikalische) Experiments) was installed during 1999 at the Munich tandem accelerator. It is situated at the 0° beam line of the experimental hall I and mainly consists of high precision slits, a magnetic and electric scanning unit, a superconducting multipole lens and the target chamber which includes a four axis precision goniometer and an optical microscope for target and beam positioning. During the first experiments 20 MeV protons and 90 MeV ^{32}S ions were used to test all the equipment installed up to now. The superconducting lens worked without any problems. However, some field distortions were detected which are studied offline. Nevertheless, with a reduced divergence of the beam, a focus of 700 nm was achieved by scanning a gold grid with the focused beam and counting for transmitted ions with no energy loss.
In addition to the beam transport experiments the new detector equipment of the scanning ion microprobe was tested. The current instrumentation covers a stacked silicon detector (total thickness 3 mm) to analyse transmitted protons for their energy (for Scanning Transmission Ion Microscopy, STIM) and a similar structure to detect backscattered ions. Furthermore a 2.5 sterad silicon ring detector is installed 20 mm behind the target to detect forward scattered ions, especially proton-proton coincidences from elastic scattering events for hydrogen analysis. For PIXE (Particle Induced X-ray Emission) and PIGE (Particle Induced Gamma-ray Emission) experiments a HPGe detector is placed near the target as well as a channeltron detector for secondary electron detection. In order to perform high resolution STIM experiments with high count rate performance a large 90° vertical magnet situated behind SNAKE was used as a magnetic spectrograph in transmission geometry, by installing two additional quadrupole singlets in front of the magnet and a CCD linear detector at the focal plane of that magnetic arrangement. An overall energy resolution of $3.8 \cdot 10^{-5}$ fwhm was demonstrated.

MF-O4

EXPERIENCE WITH THE HIGH RESOLUTION BEAMLINE ON THE OXFORD PROTON MICROPROBE FACILITY

Geoff W. GRIME, Istvan RAJTA, Diane de KERCKHOVE and Mark B.H. BREESE[1] University of Oxford, Department of Materials, Parks Road, Oxford, OX1 3PH, UK, [1]University of Surrey School of Physics, Guildford, Surrey, UK

This paper presents a detailed description of the new high resolution beamline currently under development at Oxford. The design philosophy is presented from the point of view of both theoretical beam optical considerations and from a technological aspect. The results of preliminary tests and analytical measurements will be presented.

MF-O5

DEVELOPMENT OF AN AUTOMATED SINGLE CELL IRRADIATION SYSTEM COMBINED WITH A HIGH-ENERGY HEAVY ION MICROBEAM SYSTEM

Tomihiro KAMIYA, Watalu YOKOTA, Yasuhiko KOBAYASHI, Japan Atomic Energy Research Institute, Takasaki, Gunma 370-1292, Japan,
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An automated single cell recognition and irradiation system has been developed at a high-energy heavy ion microbeam. The system has been installed on a vertical beam line of the JAERI Takasaki AVF-cyclotron accelerator. The single-ion-hit technique, which had been established previously at the heavy ion microbeam system on the 3 MV tandem, has been applied to the higher-energy accelerator to irradiate individual biological cells in an air environment with energies up to 10 MeV/u by single ions.
The fully automated off-line system for cell positioning and recognition consists of high-precision stage, a computer network and a software package (AUTOSCOPE). The new system is able to obtain positional data from a cluster of individual samples randomly placed on a holder before irradiation and enable to inspect the same cells after irradiation with ions. Additionally, the on-line fully automated system with a software package (IRRADIATE) has been developed to perform irradiation of living cells with individual ions according to data obtained with the off-line system.
This paper will describe new systems, and show preliminary results from irradiation experiments with test samples.

MF-O6

A NOVEL ULTRA-SHORT SCANNING NUCLEAR MICROPROBE WITH EXTERNAL BEAM: DESIGN AND FIRST RESULTS

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The paper describes an optimized scanning nuclear microprobe (MP) with a ferrite-core post-lens beam scanning system and a new ultra-short (total length of 1.85m) probe forming system based on a divided Russian quadruplet of magnetic quadrupole lenses. The features of the design permit the MP operation in the high current (PIXE/RBS/FRS) and low current (STIM/IBIC/SEU) modes with a very short working distance (approx.8cm) and inexpensive adjustable lenses. The modern electrostatic accelerators have a rather high beam brightness of

about 10-25 pA/μm^2/$mrad^2$/MeV. This allows the proposed MP to provide a fairly high lateral resolution even with rather large (1%) parasitic (sextupole and octupole) pole tip field components in all lenses. The focused beam can be extracted to air through a thin (0.1-10μm) window.
The new MP is promising for studies of solids or biological samples with high resolutions (0.08–2μm) in both modes under normal (ambient) conditions. This optimised MP is simpler and much cheaper in construction than traditional long systems. Especially a vertical version of such a MP can be very useful for the single ion bombardment of living cells. In the present work a comparison of the calculated features of the new MP with first experimental results obtained with a similar system (total length of 2.30 m) at the INP in Cracow will be given. An adaptation of the MP in Cracow to work with an external beam is planned. We have started feasibility studies for the installation of an ultra-short MP with external beam for biomedical applications at the Leipzig laboratory LIPSION.

MF-O7

THE HEAVY ION MICRO PROJECTION SETUP AT BOCHUM

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A new type of microbeam setup is presented allowing MeV ion projection imaging of large area masks structures using the final lens of a high energy microprobe. Key issue of this new technology is the use of high aspect and high resolution stencil masks capable of withstanding high beam power which are now available. While the theoretical lateral resolution limit for this setup should allow for the production of features well below 50 nm in size, the practical imaging resolution demonstrated so far is at 300 nm with heavy ions and typical implantation fluence. Microstructure formation is possible on substrates which do not allow the application of surface contact masks or in situations where such masks would result in sample contamination due to sputtering. A compact sample heater provides high substrate temperatures up to 1300 °C during implantation for minimum damage implantation of crystalline samples.
This setup opens a fascinating new field of applications in material science. The largely increased current density of a focused microbeam is combined with the possibility to produce many structures simultaneously. Fast structured implantation of stoichiometric doses of e.g. rare earth element ions gives access to the production of new materials of high purity at different substrate temperatures within reasonable time.
Applications where this new technique is used include synthesis of new materials, lateral doping, production of intentionally located Si micro crystals in SiO_2, production of structured metallic and semiconducting silicides and micro structurization of diamond.

MF-O8

MEDIUM ENERGY NUCLEAR MICROPROBE WITH ENHANCED SENSITIVITY FOR SEMICONDUCTOR PROCESS ANALYSIS

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A rapid shrinkage in a minimum feature size of integrated circuits (ICs) requires a localized analysis with complex real device structures. A nuclear microprobe for such analysis should meet the requirement of a lateral resolution of less than 10 nm with an enhanced sensitivity in the next 10 years.
In this study, medium energy ion probes of 100 - 400 keV with Be^+ and Si^+ beams have been used to enhance the scattering cross section for micro RBS. A large area detector with a multi-scaling time-of-flight (TOF) detecting system combined with a micro channel plate (MCP) was installed to obtain higher counting rate with enhanced mass resolution.
A chopped Si^+ ion beam at a frequency of 10 - 100 kHz could provide an enhanced yield without Si bulk signals, resulting in avoidance of stopping foils and, hence, low background analysis. The paper will describe the details of this multi-scaling TOF detecting system such as nano second time resolution and counting rate. A trade-off between high sensitivity and high lateral resolution down to nanometer range will be discussed.

Session AT: Analysis Techniques

AT-O1

3D HYDROGEN MICROSCOPY BY PROTON PROTON SCATTERING

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The new ion microprobe SNAKE (**S**uperconducting **N**anoscope for **A**pplied nuclear (**K**ern-) physics **E**xperiments) is a tool to focus e. g. 20 MeV protons to submicron beam spot size [1]. This facility establishes a sensitive 3D microscopy of hydrogen distributions by elastic proton proton (pp-)scattering [2]. The high proton energy enables analysis at a target thickness up to some 100 µm, e. g. carbon, without significant reduction of the lateral resolution. The scattered projectile and recoiled target protons are detected in coincidence using a 1 mm thick annular silicon strip detector with a hole diameter of 20 mm and an outer diameter of 70 mm. It covers scattering angles from 29° to 61° resulting in 2.5 sr solid angle of detection. The detector is devided into 16 sectors at the backside. 48 concentric rings at the frontside provide a 1° angular resolution. Therefore geometric effects are reduced to a minimum. E. g. a depth resolution better than 10 µm is calculated for a 200 µm thick carbon sample. Better depth resolution is expected for thinner samples.
The major advantage of pp-scattering is the lowest possible irradiation damage in ion beam analysis of hydrogen. Irradiation damage is dominantly produced via Rutherford scattering processes on electrons (ionisation) and on nuclei (atomic displacements). For 20 MeV protons the elastic pp-scattering cross section is enhanced 500 times above the Rutherford cross section. Therefore the ratio of detection cross section to damage cross section is larger than in any other ion beam analysis technique. The huge solid angle additionally allows analysis at proton currents of 100 pA. As a result, the detector installed at SNAKE gives the opportunity for 3D hydrogen analysis with submicron lateral resolution in any solid.
First tests of the new detector system and the readout electronics have been carried out. Both the coincidence analysis and the depth profiling worked successfully. Results of the tests and future applications will be presented.
[1] G. Datzmann, G. Dollinger, G. Hinderer and H.-J. Körner, Nuc. Instr. and Meth. B158 (1999) 74.
[2] B. L. Cohen and C. L. Fink and J. H. Degnan, J. Appl. Phys. 43 (1992) 19.

AT-O2

THE AUTOMATIC ANALYSIS OF RUTHERFORD BACKSCATTERING SPECTROMETRY SPECTRA

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It has been shown that Bayesian Statistics is ideal for application to the analysis of Ion Beam Analysis (IBA) data. Past work has shown its applicability to the deconvolution of the detector response function from micro-RBS and micro-PIXE spectra, subtraction of the background from PIXE spectra, the extraction of depth profiles from PIXE spectra using two detectors and the extraction of depth profiles from RBS spectra.

We have now applied the Bayesian theory to the automatic extraction of depth profiles from RBS spectra with the aim of creating an on-line RBS analysis program. We also present results from applying the Bayesian formalism to the batch analysis of many in-situ RBS spectra and single pixel spectra from microprobe maps.

AT-O3

CHEMICAL STATE ANALYSIS OF Cu, Cu_2O AND CuO WITH WDX USING AN ION MICRO BEAM

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In the previous paper[1], we showed that performance of a wavelength-dispersive X-ray spectroscopy system for PIXE (WDX-PIXE) consisting of an X-ray spectrometer with a PSPC and a heavy ion micro beam. In this paper, we describe the possibility of chemical state analysis with our micro WDX system using a light ion micro beam. High-resolution PIXE spectra of Cu, Cu_2O and CuO produced by MeV light ions were measured by the WDX-PIXE system at ONRI. Focused 2.0 MeV protons with a beam size of 80~100(H) x 30(V) μm^2 from the 1.5 MV tandem accelerator were used. In this experiment, Cu $L\alpha_{1,2}$, and $L\beta_1$ X-ray spectra were measured with an analyzing crystal of TAP(2d = 2.576 nm).

The Cu L X-ray spectra in the present X-ray energy region (900~1000 eV) show two clear peaks, the large one is the $L\alpha_{1,2}$ line and small one $L\beta_1$ line. Due to a high detection efficiency of our spectrometer equipped with PSPC for soft X rays, the change of the ratio, $L\alpha_{1,2}/L\beta_1$, which shows the chemical effects, can be detectable. The ratio is largest for pure Cu metal, and least for CuO which has a highest oxidation number. Moreover, $L\alpha_{1,2}$ X-ray spectrum for CuO show a large shoulder at the high energy side of the main peak, which is also considered to be due to the chemical bonding between Cu and O atoms.

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AT-O4

NUCLEAR MICROPROBE ANALYSIS OF A GAS-SOLID INTERFACE

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In situ analysis of gas-solid and liquid-solid interfaces have several important applications in the field of catalysis, plasma etching / damage and chemical corrosion. Analysis of interfaces using surface probe techniques such as AES generally provides information on only the surface composition and surface adsorption of atoms. However, if the investigation demands probing greater depths from the surface, it may be appropriate to use high energy microbeams.

This work involves the use of micro RBS, Channeling Contrast Microscopy (CCM) and Channeling Scanning Transmission Ion Microscopy (CSTIM) techniques as possible tools for studying gas-solid and liquid-solid interfaces. A special Thin Si window cell arrangement was used for either $4He^+$ or proton microbeam analysis of the interfaces. Channeling contrast maps obtained due to the distortion of the Si lattice as a function of gas pressure in the cell indicate that the dechanneling fraction is a linear function of the pressure increase inside the cell. CSTIM was used in conjunction with a double window cell arrangement to study the distortion of the Si lattice under pressure. Several applications of CCM and CSTIM with the cell arrangement for the study liquid-solid interface, plasma etching/damage in Si, in situ electrochemical corrosion and the lattice location of chemisorbed atoms on a single crystal surface will be discussed.

AT-O5

LIGHT DETECTION WITH SPECTRAL ANALYSIS AT THE LEGNARO NUCLEAR MICROCROPROBE: APPLICATIONS IN MATERIAL AND EARTH SCIENCES

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Among the numerous ion beam analytical (IBA) techniques available for material characterisation, ionoluminescence (IL) has not attracted the interest that it should deserve. Although the importance of IL technique, particularly if combined with other IBA techniques, has been widely proven, very few apparatuses to analyse light emission spectra have been installed at the microbeam facilities.

In this paper we present the new IL apparatus installed at the Legnaro (LNL) ion microbeam facility. The system is a modification of the OXFORD MONOCL2 apparatus for cathodoluminescence. Light collection is performed by using a retractable parabolloidal mirror located at a very short distance from the sample, with a small (2 mm) aperture to allow the ion beam to hit the sample. Accurate positioning of the retractable mirror directly coupled to a chamber mounted high-resolution monocromator (350-900 nm spectral range) allows high light collection efficiency.

This design assures that IL can be performed using low beam currents (<1 pA) with the consequent reduction of the radiation damage, which often occurs during ionoluminescence measurements.
A summary of some meaningful results obtained with such an apparatus are presented. The combination of IL/PIXE was used to characterise natural silica glass, known as Libyan Desert Glass and cubic BN grains; polycristalline CVD diamond has been studied by mapping both the IBICC signals and different spectral structures of ionoluminescence.

Session IT: Imaging Techniques

IT-O1

ION-INDUCED EMISSION MICROSCOPIES

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Nuclear microscopy normally involves focusing an MeV-energy ion beam to micron or even submicron dimensions, scanning this beam laterally (X,Y) across a sample specimen, and measuring an IBA signal(s) recorded as a pulse height (S). The data from such an experiment is usually collected in list-mode, i.e. a file of {X,Y,S} events, which is later binned into a Counts(X,Y,S) matrix of data for analysis and the creation of images. An alternative to this "Flying Spot" style of nuclear microprobe analysis emerged two years ago with the invention of Ion-Electron Emission Microscopy (IEEM). With IEEM the ion beam is only partially focused so as to fill the field of view of a Photo-Electron Emission Microscope lens fitted with a single electron Position Sensitive Detector (PSD). When a single ion strikes the sample, the emitted secondary *electrons* are projected at great magnification onto this PSD, and X and Y signals are generated. These signal are then put into coincidence with the IBA signal (S) made by this same ion, inside the sample, and an {X,Y,S} event file is created, just as with traditional nuclear microscopy.

In this paper, an update will be given on the state of IEEM including its application to Radiation Effects Microscopy of 256K radiation hardened SRAMs. In addition, a new type of full-field nuclear imaging is described: Ion-Photon Emission Microscopy or IPEM. With IPEM, *photons* generated in a phosphor layer coating the sample are projection imaged with a high power optical microscope onto a single photon PSD in a fashion completely analogous to IEEM. Preliminary results suggest that IEEM will have much better resolution (potentially 0.1 μm) than IPEM (one to a few μm) , but IPEM will be much easier to implement either in an accelerator lab or using radioactive sources. IEEM requires metal surfaces for the efficient production of secondary electrons, while IPEM requires thin short lifetime phosphor coatings, which are insulators, to make ion-induced photons. In some Radiation Effects Microscopy applications, it is preferable to have insulating rather than metallic surfaces. Both techniques require very high 1) IBA event probabilities and 2) secondary electron or photon generation and detection efficiencies, which limit both IEEM and IPEM to IBA techniques involving single ion effects such as IBICC.

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IT-O2

THREE DIMENSIONAL ION MICROTOMOGRAPHY

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The technique of ion microtomography (IMT) provides three dimensional distribution information about a sample's mass density and elemental composition. The required data is obtained by doing a STIM tomography experiment followed by a PIXE tomography experiment. The experiment times have been vastly reduced now that data is collected with MicroDAS, the new fast data acquisition system and the experiment is easier to perform because sample manipulation is automated via computer control. Also, the time to perform the PIXE tomography experiment has been substantially reduced now that a large solid angle between the sample and X-ray detector is used. To correct for the inherent three dimensional nature of such an experimental setup, a specially developed tomographic reconstruction technique is used to combine the STIM and PIXE tomography data sets to create an accurate quantitative tomogram of the sample. Examples are provided of a few samples, including a characterised "standard" sample that was used to test the efficacy of the entire IMT process. Data calculated in the tomogram agrees to within 2% of both the quantitative and structural information known about the standard sample. To interpret the three dimensional distribution information, a special volume rendering program is used to visualise various aspects of the tomogram. Each aspect is color coded to facilitate with the easy visualisation of multiple complex three dimensional structures.

IT-O3

IMAGING OF CHARGE TRANSPORT IN POLYCRYSTALLINE CVD DIAMOND USING IBIC MICROSCOPY

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This paper presents results of IBIC analysis of radiation detectors fabricated on polycrystalline CVD diamond films. These devices were designed as alpha particle and UV sensors, and were constructed with a co-planar electrode structure fabricated only on the growth side of the diamond film. This coplanar device structure is predominantly sensitive to charge transport close to the growth surface of the diamond film where the diamond crystallites are largest. Due to the shallow penetration of the electric field in the device, the focused MeV proton beam used for IBIC analysis only probes the charge transport in the portion of the diamond film nearer to the upper surface, where the charge drift lengths are maximised.
Results are presented in the form of video sequences of 200 x 200 μm^2 regions of the device with increasing bias voltage. These show the different threshold voltages at which individual 10-20 μm wide grains facilitate charge transport at different spatial locations away from the surface metallisation. From these the different collection efficiencies of the individual grains

can be quantified and related to the device structure as observed by optical and secondary electron imaging.
These results directly show that the charge collection efficiency in the inter-electrode region is limited by the size of the diamond crystallites. This study reinforces the need for further improvements in the size and quality of the crystallite structure in CVD diamond to enhance the performance of this type of diamond sensor for alpha particle or UV photon detection.

IT-O4

IMAGING NICKEL SEGREGATION IN SYNTHETIC DIAMONDS USING IONOLUMINESCENCE (IL) AND PROTON INDUCED X-RAY EMISSION (PIXE)

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Nickel is an optically active impurity in diamond which can be incorporated during growth, if a Ni catalyst is used, or by ion implantation. A number of optical centres attributed to nickel have been previously identified including absorption bands at 1.40eV, 1.883eV and 2.51eV, and two emission bands at 1.40eV and 2.56eV. Ionoluminescence (IL) performed with a nuclear microprobe is a highly sensitive method for imaging optically active impurities and defects in different materials. In this study we present the results of combined IL and Proton Induced X-ray Emission (PIXE) measurements performed with a nuclear microprobe on Sumitomo Electric Industries synthetic diamonds. Nickel impurities incorporated into different growth zones during the growth are identified with IL spectroscopy, and shown to segregate into the <111> growth zones with IL imaging. This finding is consistent with previously published luminescence and Electron Spin Resonance (ESR) results. PIXE analysis is used to unambiguously identify the presence of Ni and used to determine the lattice location from channeling measurements on the different growth zones. Results obtained using both techniques are compared.

IT-O5

CHANNELING CONTRAST MICROSCOPY ON LATERAL EPITAXIAL OVERGROWN GaN FILM

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Lateral epitaxial overgrowth (LEO) has been shown to significantly reduce extended defect densities in GaN films grown by metallorganic chemical vapor deposition (MOCVD). This reduction in threading dislocations has recently led to improved device performance. The knowledge of the lateral distribution of dislocations is potentially important for fabrication purposes. Frequently, studies on the crystallographic defects have been carried out using TEM due to its high resolution. However, tedious sample preparation makes the technique time-consuming. Here, we have used both channeling contrast microscopy (CCM) and broad beam channeling to investigate the quality of the epitaxial lateral overgrown GaN layer. A 100 nm SiN mask layer with 3um wide gaps that are 13 um apart is formed on GaN/Al_2O_3 substrate. Subsequently, 5um thick GaN stripes are epitaxially grown on the substrate by LEO. Broad

beam channeling and CCM measurements have been carried out using 1-2 MeV He^+ and H^+. Generally, very low minimum yields are found (χ_{min} =2.5-3%) in the axial direction with broad beam channeling, indicating the high quality of epitaxial growth. However, CCM images at a tilt angle of 0.3 degrees from the axial direction reveal stripes of dislocations which coincide with those of the mask structure. Low dislocation densities are observed elsewhere. The value of χ_{min} increases to 20% above the gaps. It is suggested that the dislocations originate from the underlying GaN seed layer in the 3um wide window region. The overgrown regions or "wings", in contrast, show a low density of dislocations. This is consistent with the TEM images, which confirm the presence of threading dislocations at the 3um wide window. The CCM images also reveal an increase in dislocation density with depth down to a few um below the GaN surface. Therefore, CCM is shown to be a quick and effective technique for 3 dimensional imaging of the dislocations in GaN.

Session MIBM: Micro-Ion Beam Modification of Materials

MIBM-O1

HIGH PRECISION 3D METALLIC MICROSTRUCTURES PRODUCED USING PROTON BEAM MICROMACHINING

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Microstamping and micromolding is a promising new field of application for Proton Beam Micromaching (PBM). The implementation of a post lithographic process step such as electroplating offers the possibility of cost effective batch production. In proton beam micromachining a focused MeV beam is magnetically scanned in a predetermined pattern over a resist (e.g. PMMA or SU-8), which is subsequently chemically developed. A proton traversing a sample follows an almost straight path, enabling the production of microstructures with well defined rectangular side walls. A resist layer with a thickness of typically 50% of the proton range is applied on a conductive substrate, so that the end of range damage does not occur in the resist. The substrate also acts as a seed layer for plating. Different substrates are discussed as a base for plating. A crucial step in the development of mechanically strong microstructures is the conversion of structures made from resist material of low hardness and strength, to metallic microstructures. In this current work the metallic microstructures are produced using either electrolytic Ni plating in a modified Watts bath or by electroless Ni plating. Clean Ni microstructures with a height of 5 to 20 □m are obtained after the resist is stripped. The plated Ni structures can be used as a resolution standard in nuclear microscopy. These first tests show that Ni grids produced with proton beam micromachining have typically better defined side walls than commercially available 2000 mesh Au grids. The preliminary data presented in this paper indicate that electrolytic plating of proton beam micromachined resist structures result in well defined and smooth metallic microstructures.

MIBM-O2

OPTICAL PROPERTIES OF nc-Si MICROSTRUCTURES IN SiO_2

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Arrays of Si nanoclusters have been produced by ion implantation of 500 keV Si^+ into SiO_2 using the heavy ion microprobe at the Ruhr University Bochum. The arrays consist of single dots with 2 μm and 700 nm diameter with a similar spacing in between. Their optical properties have been investigated and compared with normal implanted samples. Both, the structured and unstructured samples show significant photoluminescence in the red region of the spectra after annealing at 1100°C. The photoluminescence signal shifts towards longer wavelength with increasing implantation dose. However, with increasing annealing time the PL signal shows a shift towards shorter wavelength and an increase in intensity. These investigations show that origin of the photoluminescence of Si nanoclusters in the red region of the spectra is still not completely understood and cannot be explained by quantum confinement effects alone.

MIBM-O3

CHARACTERIZATION OF MICRO-STRUCTURES FORMED ON MeV ION-IRRADIATED SILVER FILMS ON Si(111) SURFACES

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Ag epitaxial layers on oriented silicon single crystal surfaces, upon MeV ion irradiation, undergo remarkable improvement in crystalline quality [1,2]. This is often associated with remarkable changes in surface morphology. Growth of micron sized (1- 30 microns) structures (blisters as well as islands) have been observed on silver films when silver thin films (~800Å), deposited on Br-passivated Si(111) surfaces, were irradiated with energetic Si ions (1 - 12 MeV). The micro-structures on the surface of Ag films, show a variation in the height, size and in the number density as a function of ion energy as well as dose. A detailed analysis of the height, shape, size distribution as well as the composition of these islands using micro ion beam will be presented. Also results of optical, SEM and AFM measurements will be presented, explaining the growth of these micro-structures. Thermal behaviour of these micro-structures will be compared with the vacuum annealed silver films grown on Br-passivated Si(111) surfaces.

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Session ME: Microelectronics

ME-O1

IBIC CHARACTERISATION OF DEFECT STRUCTURES IN POLYCRYSTALLINE SILICON

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The physics of silicon solar cell device imposes very stringent requirements on the electrical properties of material. A pressure to lower the cost of the substrate brings the novel material to be processed with a fairly inexpensive technology. Unlike integrated circuits that are surface devices, in silicon solar cell the entire volume of the cell is electrically active. This fact makes IBIC (Ion Beam Induced Charge) technique ideal for studies of defects, since the charge transport can be studied both close to the material surface and in its deeper regions. Deeply buried defects as well as contact imperfections are imaged using appropriate ion range. In addition to frontal IBIC measurements performed by protons, Li and O ions, lateral mode is used to determine contribution of the diffusion mechanism in charge collection. Defects were characterised using DLTS (deep level transient spectroscopy) technique as well.

Studies of defect structures were performed on edge-defined film-fed grown (EFG) silicon ribbons containing twin boundaries and disclocations. Parallel studies were performed on dislocated single crystalline float zone (FZ) and Czochralski (CZ) material, and several polycrystalline samples obtained with different casting processes as well as microcrystalline material deposited by RTCVD process. All samples are prior to the analysis cleaned and planar etched for about 10μm to remove subsurface layer additionally rich with structural defects. Schottky diodes were than formed evaporating gold (backside ohmic contact) and aluminium (front Schottky barrier).

ME-O2

INVESTIGATION OF BODY-TIE DESIGN ON ION BEAM INDUCED CHARGE COLLECTION IN SILICON-ON-INSULATOR FETS USING THE SANDIA NUCLEAR MICROPROBE.

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Silicon-on-insulator (SOI) technology exhibits three main advantages over bulk silicon technology for use in radiation environments. (1) SOI devices are immune to latchup, (2)the volume of the sensitive region (body) and hence total charge collection per transient irradiation is much reduced in SOI devices and (3) the insulating layer blocks charge collection from the substrate (i.e., no funneling effect). This effectively raises the single event upset threshold for the SOI device. However, despite their small active volume SOI devices are still vulnerable to single event effects (SEE). Inherent in the SOI transistor design is a parasitic *npn* bipolar junction transistor (BJT), where the source-body-drain acts as an emitter-base-collector BJT. An ion strike to a floating (not referenced to a specific potential) body can create a condition called snapback, where excess minority carriers in the

drain-body junction forward bias the source-body junction, causing the parasitic BJT to turn on and inject current into the drain. Tying the body to the source limits the emitter-base current and reduces the sensitivity of the device to single ion strikes. Unfortunately, the body-tie loses effectiveness with distance due to resistivity, and in regions far enough from the tie the BJT is still in effect.

Using the Sandia nuclear microprobe we have created charge collection maps of SOI FETs which have different body-tie designs. The effectiveness of single end, double end and body-under-source (BUSFET) ties on charge collection versus position of the ion strike are extracted from these maps. Comparisons of the experiment to DAVINCI simulations are also presented. Effects of body-ties and operating voltage on snap-back thresholds are also presented and predictions of performance in radiation environments made for the different designs.

ME-O3

ION BEAM INDUCED CURRENT (IBIC) ANALYSIS OF HIGH POWER DEVICES

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Ion Beam Induced Charge microscopy (IBIC microscopy) is used to investigate light-triggered 8 kV thyristors. The IBIC analysis allows the characterisation of electronic structures of these high power devices located several tenth of microns below the sample surface using a focused proton microbeam at an energy of 2 MeV. The large range (47μm) of the ion beam and the relatively high diffusion length of the induced charges make it possible to analyse deep and buried layers.
The depletion regions of pn-junctions located 100 μm below the surface were used for charge collection. Since very small ion doses are sufficient to generate good resolved IBIC images, destruction of the devices can be avoided. These preliminary studies suggest that IBIC microscopy could also be a powerful tool to analyse buried damage regions in power devices induced by stressing them to find the operational limits.

ME-O4

DIFFUSION TIME RESOLVED ION BEAM INDUCED CHARGE COLLECTION STUDIES ON STRIPE-LIKE JUNCTIONS USING HEAVY-ION MICROBEAMS

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Ionizing radiation can lead to undesirable generation and migration of charge within electronic devices. To design more robust ICs, it is essential to create and test accurate models

of charge collection dynamics with microcircuits. A new technique is demonstrated to measure the average arrival time of the diffused charge, which can be related to the first moment (or the average time) of the arrival carrier density on the junction. The order of average arrival time for diffusive charge collection can be crucial to understanding and mitigating radiation induced circuit malfunctions during normal IC operations. In the present work, specially designed stripe-like junctions were experimentally studied using microbeams (12 MeV carbon and 28 MeV silicon) with a spot size of 1 μm produced at the Sandia National Laboratories. The relative arrival time is measured along with the charge collection using a multiple parameter data acquisition system. A 2D-device simulator, the MEDICI code, is used to simulate the charge collection dynamics on the stripe-like junctions. The simulations compare very well with the microbeam experimental results. These results show the importance of the diffusive charge collection by junctions, which is especially significant in accounting for Single Event Upsets (SEUs) and Multiple Bit Upset (MBUs) in digital devices. Another, unanticipated, use of this technique will be discussed: Using DTRIBICC on a stripe junction target geometry as a high resolution ion position detector useful for focusing microbeams.
Work supported by the National Science Foundation, the State of Texas Coordinating Board - Texas Advanced Technology Program, and the Robert A. Welch Foundation. Work at Sandia supported by the U.S. DoE under contract DE-AC04-94AL85000.

ME-O5

SUPPRESSION OF FLOATING BODY EFFECTS IN SOI MOSFET STUDIED USING NUCLEAR MICROPROBES

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A strong demand for high speed and low power operations for the next generation of ULSI (ultra large scale integration) for PDA's (Personal digital assistants) and hand-held PCs (personal computers) application accelerates a development of structures with MOSFET's (metal oxide semiconductor field effect transistors) on SOI (silicon-on-insulator) substrates. SOI MOSFET's have various advantages over conventional MOSFET's on a bulk such as SEU (single event upset) free, high speed and low power consumption, though there exists a critical issue of floating body effect, i.e., the SOI body potential is floating, resulting in SEU in a special case.

In this study, the suppression of the floating body effects in partially depleted SOI MOSFET with and without body-contact electrodes has been investigated using proton nuclear microprobes. Transient SOI MOSFET behavior during ion incidence has also been investigated by three dimensional computer simulation. The transient drain-source current as a function of incident microprobe position with various gate lengths from 300 -700 nm has, for the first time, been clarified.

Session MS: Applications in Material Sciences

MS-O1

INVESTIGATING NON-UNIFORM SURFACES OF THIN SIO_2 FILMS PRODUCED BY HIGH-TEMPERATURE NITRIDATION EXPERIMENTS WITH ION MICROSCOPY

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Non-uniform surfaces of thin silicon dioxide films produced by high-temperature nitridation experiments were studied with ion microscopy. Thin SiO_2 layers were produced by thermal oxidation of Si wafer material. To study the nitridation effect of the oxide layers, the specimens were nitrided in a furnace at high temperature. Surprisingly, a non-uniform artificial pattern on the surface of samples annealed at 1200 °C was found. Non-destructive ion beam analysis was performed to determine changes in the elemental concentrations and depth profiles of the major components. Two dimensional maps of N and O were measured with the nuclear reactions $^{14}N(d,\alpha)^{12}C$ and $^{16}O(d,p)^{17}O$, respectively. RBS was used to measure Si maps. In the centre of the non-uniform pattern, only silicon was found. Additionally, bands consisting of Si, O, and N surrounding the patterns were discovered. The results are in agreement with electron microscopy measurements.

MS-O2

MICRO-RBS ANALYSES OF SURFACE TOPOGRAPHY OF Bi FILM PREPARED BY PULSED LASER DEPOSITION

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Pulsed laser deposition (PLD) is a well developed technique for preparation of thin films of metals, semiconductors, insulators, ceramics etc., and multilayers based on these films. For fundamental and technological reasons a lot of work have been performed to improve the understanding of the mechanisms involved both in the ablation and deposition steps. Various theoretical and experimental works tried to correlate the material distribution collected onto a given substrate with the irradiation conditions such as the laser fluence, irradiation spot size and the composition of the target.
The greatest drawback of PLD seems to be the emission of particles from the laser irradiated surfaces so that they are transferred to the substrate and are incorporated into the growing film. Several methods have been reported to minimise the droplet formation. In case of ablation of a liquid target instead of a solid one the target deterioration is almost completely solved for different metals, but for Bi films some peculiarities have been arisen. Sub-micrometer and micrometer size drops are observed on the film and the drop formation could not be totally avoided.
The anomalous behaviour of Bi made important the quantitative description of PLD film composition with micrometer lateral resolution. Rutherford backscattering spectrometry combined with microbeam was applied to analyse the distribution of Bi, the areal density of

separate species and the surface topography. The separated RBS spectra of rough and smooth surfaces provide more precise data for the theoretical calculations than the spectra of total area which were used before.

MS-O3

NUCLEAR MICROPROBE MEASUREMENTS ON LABELED POLYMER GRATINGS

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Polymers with an ordered molecular structure can be applied in optical systems for e.g. data transport, data storage and displays. Patterned UV photo-polymerization is used to prepare polymer gratings from a mixture of two acrylate monomers. A 3 MeV proton microprobe is used to study these gratings, consisting of two different monomer units, both of which contain different easily detectable label elements, e.g. Cl, Br, Si or F.
During the preparation process, the difference in reactivity and mobility of these two monomers in combination with polymer-monomer interaction results in diffusion of monomers. Since this diffusion process takes place on length scales of micrometers, a scanning ion microprobe is a powerful tool for the quantitative analysis of the polymer films, obtained after complete polymerization. The microprobe is equipped with PIXE, PIGE, NBS, and NFS, to quantify both the label elements and C, O and H. This makes it possible to determine the concentration of monomer units as a function of position and to study the diffusion process.
Several combinations of different monomers are studied. In the case of a mixture of a monofunctional and a difunctional monomer, both monomers migrate to the illuminated areas and large thickness variations are observed. When a mixture of two difunctional monomers is used, opposite migration of the two monomers is observed, while the film shows no variation in areal density.

MS-O4

CHARACTERISATION OF Ti:SAPPHIRE WAVEGUIDES USING THE MELBOURNE NUCLEAR MICROPROBE

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The creation of a titanium sapphire waveguide laser would yield a highly tunable and robust light source useful for spectroscopy and optical telecommunications. However, before these devices are to be realized, high quality guides with controllable geometries must be produced. We have successfully used high-energy ion implantation of titanium and oxygen into c-axis oriented sapphire to fabricate waveguides with micron size dimensions.
Following fabrication, microbeam characterization was performed on the Melbourne nuclear microprobe. Evolution of ion beam induced defects, and hence recovery of the crystal quality as a function of annealing temperature, has been investigated using Rutherford Backscattering and Ion Channeling. Substitutionality, lateral position and homogeneity of Ti within the sapphire substrate were determined with the application of scanning Proton Induced X-ray

emission with a focussed ion beam. This was necessitated by the absence of Ti signal in the RBS spectra as a result of the implantation depth and low dose regime used in this project. We have also studied the photoluminescence intensity as a function of implantation and annealing conditions and have found the optimum conditions for creation of the Ti^{3+} state which is required for laser operation. This project encompasses the application of ion implantation to waveguide creation in addition to the use of ion beam techniques for characterization.

Session BM: Applications in Biology and Medicine

BM-O1

THE IMPACT OF MICROBEAMS IN RADIATION BIOLOGY

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Cellular micro-irradiation is now recognised as a powerful technique for understanding how ionising radiation interacts with living cells and tissues. Charged-particle microbeams are uniquely capable of delivering single, or counted multiple particles to selected sub-cellular targets. This capability is particularly useful for studying the risks associated with environmental exposures to α-particle emitting isotopes (such as radon) where exposed cells within the body are unlikely to receive more than one particle traversal. Microbeam methods are also seen as highly appropriate for the study of the so-called 'bystander-effect'. It has always been thought that direct damage of the DNA within the cell nucleus is required to observe radiation-induced effects. In recent years however, it has emerged that unirradiated cells can also respond to signals transmitted by irradiated neighbours. Clearly, the ability of a microbeam to irradiate just a single cell, or selected cells within a population is well suited to study this phenomenon. Using the Gray Laboratory microbeam, we have been able to demonstrate a significant increase in the levels of apoptosis and chromosome damage in a population of cells after just a single cell has been exposed. Also, by targeting the cell cytoplasm, we have also shown that intra-cellular signalling between the cytoplasm and nucleus can cause DNA damage, indicating that direct DNA damage is not required to observe radiation induced effect in cells.

The use of microbeams in a radiobiological application presents the designer of such a system with a unique set of problems, not least, the requirement for the target (i.e. the cells) to be in a humid environment at atmospheric pressure. There is also a considerable practical advantage in using a vertically-oriented beam, rather than the horizontal configuration common to microprobes used in physics applications. This requirement has severely hindered attempts to adapt existing microprobes for radiobiological use. The Gray Laboratory has developed a charged-particle microbeam uses a 1μm diameter bore glass capillary to collimate protons, or $^3He^{2+}$ ions accelerated by a 4MV Van de Graaff. We have used track-etch plastic to determine the targeting accuracy of our facility. The overall accuracy is determined by the properties of the collimator, scattering by the vacuum window and detector (usually an 18μm thick plastic scintillator) and by the positioning accuracy of our automated cell alignment system. Our measurements show that for protons, we can target 90% of cells (at a rate of 4000 cells per hour) with an accuracy of ±2μm, or 96% of cells with an accuracy of ±5μm. Using $^3He^{2+}$ ions (which are less easily scattered), 99% of cells are targeted with an accuracy of ±2μm.

BM-O2

IS IRON A RISK FACTOR IN ATHEROSCLEROSIS?

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Iron is believed to play a part in atherogenesis (coronary heart disease) by promoting the formation of free radicals, leading to oxidative stress. Previous investigations using nuclear microscopy, which have shown a seven-fold increase in iron concentrations within atherosclerotic lesions in hypercholesterolemic rabbit models compared to healthy artery tissue, support this theory (1). In a follow-up time sequence study, we have also shown that iron accumulation occurs early at the onset of lesion formation and that blood iron depletion (induced by weekly bleeding) can reduce the uptake of iron in the lesion, and delay the onset of atherogenesis (2). These results provide support for the role of iron in initiating atherogenesis.
In a continuing study, we are investigating the effect of the iron chelator *desferal* on the development of atherosclerotic plaques. A review of the work to date, including preliminary results of the iron chelation investigations will be presented, together with a critical appraisal of the evidence for and against the involvement of iron in atherogenesis.
(1) PSP Thong, M Selley and F Watt. Elemental changes in atherslerotic lesions using nuclear microscopy. Cellular and Molecular Biology (1996) 42 (1), 103-110.
(2) D Ponraj, J Makjanic, PSP Thong, BKH Tan, and F Watt. The onset of atherosclerotic lesion formation in hypercholesterolemic rabbits is delayed by iron depletion. FEBS letters 459 (1999) 218-222.

BM-O3

CYTOLOGICAL AND HISTOLOGICAL STRUCTURES IDENTIFICATION WITH THE TECHNIQUES IBIL AND SEIM IN MICROPIXE ANALYSIS

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A general purpose detection system, inclusive of the PIXE, RBS and STIM techniques, is operational in the Legnaro nuclear microprobe since several years. More recently an IBIL (Ion Beam Induced Luminescence) capability has been added both for panchromatic analysis with high sensitivity (based on a couple of Hamamatsu single photon counting PM's) and for spectral analysis with reduced sensitivity (based on a self-standing monochromator of the Oxford ltd). Finally a channeltron detector for SEIM (Secondary Electron Ion Microscopy) has been acquired since few months.
While the micro-PIXE, possibly together with STIM and RBS, are of widespread use in tissues analysis, IBIL and SEIM are seldom quoted in this field. Our intent was to explore the support these last techniques could offer to a traditional PIXE analysis of histological and cytological samples, frozen-dryed and placed in vacuum.
We used IBIL to identify structures marked or stained with traditional dyes, like the fluoresceine (green-yellow), connected to cytoplasm, and the rodamine (red), connected to nucleus, capable to discriminate cells of different nature or different parts of a cell. The knowledge of the dyes IBIL-spectrum, measured with our monochromator, enabled us to develop an original high accuracy positioning method for the microbeam. A low dose IBIL

map, obtained with our high sensitivity PM system filtered on the colour of interest, allows a subsequent beam aiming for a traditional elemental micro-PIXE analysis.
Eventually, SEIM, being extremely sensitive to surface roughness, appeared to be able to detect surface structures present in biomedical samples, if adequately prepared. Also in this case an extremely low dose is enough to produce a map.
These methods have been applied to a micro-PIXE analysis of breast tissues to study correlation between trace elements and neoplastic pathologies.

BM-O4

$Na_4Lu(III)HDOTP$ AS EXRA-CELLULAR MARKER IN INDIRECT MAPPING OF INTRA-CELLULAR ION LEVELS IN HEART TISSUE VIA MICROBE-PIXE

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Accurate determinations of intracellular element concentrations in cryo-sections of cardiac tissue requires the measurement of intracellular compartments only, thereby excluding extracellular compartments. However, the determination of intracellular element concentrations can be hampered by redistribution of mobile ions during cryo-fixation. Use of a rather small microbeam could solve the first problem, although he minimum diameter with a sufficiently high current in our microprobe facility remains too large (3 μm × 3 μm). To be able to solve both problems, we tried to measure intracellular elements indirectly, *viz.* by using an extracellular marker, which allows to split the measured elements into contributions from the intracellular and the extracellular compartments. In isolated hearts perfused with crystalline buffer solutions with known composition, the extracellular contribution to the measured total dry-weight mass fraction of each element can be derived from the mass fraction of a foreign element incorporated in the perfusion buffer.
We have investigated the practical feasibility of $Na_4Lu(III)HDOTP$ as an extracellular marker in the determination of Na, Mg, Cl, K and Ca in cryo-sections of cardiac tissue. This marker proved to meet various physiological and analytical requirements to be set to a marker. For a proper application of the marker, the PIXE production cross-sections for Lu M lines (energy range 1.228 - 2.060 keV) have been determined. The marker was used to determine intracellular elements in ventricle transmural cryo-sections of normoxic and low-flow (20%) perfused isolated rat hearts. The transmural scans of cryo-section of the left ventricle walls of the low-flow perfused rat hearts show a gradual increase of both intracellular Na, and Cl and a decrease of the K dry-weight mass fractions. The inner cell layers of the endocard seem to be unaffected.

BM-O5

ELEMENTAL ANALYSIS OF CELLULAR SAMPLES BY IN AIR MICRO-PIXE

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For the purpose of applying the micro-PIXE analysis to studies of biomedicine, we have developed a micro-PIXE system at the division of Takasaki Ion Accelerator for Advanced Application (TIARA) in Japan Atomic Energy Research Institute (JAERI), which consists of a micro-beam system combined with a 3 MV singled-ended electrostatic accelerator, a beam control system, and a data taking system of x-ray detection[1),2)]. We call our micro-PIXE system micro-PIXE camera since it presents spatial distributions of elements as pictures and this name is generally understandable for people.

Up to the present, the method of micro-PIXE analysis has been well studied, its technology has been also well developed and it is now widely applied[3)]. In many cases of its applications, samples are put in a vacuum chamber. Therefore, the samples had to be completely dried. Our system, the micro-PIXE camera was designed so that the samples can be put in the atmosphere. We fixed a cellular sample on the Mylar film of 5 μm thickness and set it as a beam window of a vacuum chamber. The sample side of the film was in the atmosphere and the another side was in vacuum. The sample was cooled in the helium gas. Micro-beams passed through the 5 μm film and the sample, and went in the helium gas. The spatial resolution of micro-beam became slightly bad due to the thin film. The x-rays were measured with a Si(Li) detector settled at the vacuum side. The x-ray energy and the beam position were simultaneously measured for each event.

As cellular samples for application of the micro-PIXE camera, we analyzed cultured bovine aortic endothelial cells, and cancer cells, liver cells, brain cells of the rat. It was not possible to analyze the cellular samples direct, since the water in the samples lowered the detection limit of PIXE. The samples were freeze-dried in a vacuum chamber, but it was not perfect so that it might not destroy the cells. Elemental mappings in the region of 60μm×60μm for main component elements contained in the cultured bovine aortic endothelial cells were obtained. It was confirmed that the shapes of elemental distributions coincide with those observed by a light microscope and the elements of P and Cl are concentrated in the cell nuclei. Bovine aortic endothelial cells were cultured in a medium with bromodeoxyuridine (BrdU). It was observed that the element of Br was concentrated in the nucleus. BrdU and cisplatin were administered to the rat. Concentration of Br and Pt were confirmed on the elemental mappings of cancer cells and liver cells.

The x-ray detector of the present system had the sensitive area of 12.5 mm^2 that was not large enough to obtain the images of trace elements in the cell. We are now developing a multi-detectors system to increase the detection efficiency, with which one sample can be analyzed in several minutes.

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2. T.Sakai et al., Nucl. Instr. And Meth. in Phys. Res. B 136-138, 390-394(1993).
3. G. W. Grime and F. Watt, Nucl. Instr. And Meth. in Phys. Res. B75, 495-503(1993).

BM-O6

NUCLEAR MICROBEAM IDENTIFICATION OF FUNCTIONAL NON-ORGANIC ELEMENTS IN PROTEINS IN EITHER LIQUID OR SINGLE-CRYSTAL FORM.

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Unambiguous identification and accurate quantitation of non-organic elements (e.g. Ca, Fe, Cu, Zn), bound to proteins is an important challenge for structural biologists. These elements (whose identity is often unknown) are present typically at the level of 1 atom per organic molecule of 10 –100 kD in samples which may be crystals 50 – 500 μm in size or small volumes of solution at low protein concentration. The non-organic atoms are present either as part of the natural molecular structure or may have been added during crystallisation to aid 3-D structure determinations, and so accurate identification and quantitation can be vital to the understanding of the protein function or can be an aid to crystal characterisation.
The low concentration of the target atoms and the small size of the samples means that microbeam PIXE is one of the few reliable techniques available for this type of analysis and this application has been developed over several years using the Oxford Scanning Proton Microprobe facility. Using the elemental mapping capability, small crystals can be identified, even when embedded in non-crystalline residues, and this then allows the locations of representative points for quantitative analysis to be selected.
A high accuracy of elemental concentration is obtained by forming the ratio of the target element to the sulphur which is contained in two of the amino acids (methionine and cysteine) found in proteins. The number of these is known from the primary amino acid sequence and this provides a convenient internal standard, allowing the atomic ratio of the unknown elements to be determined with an accuracy of 6 – 10%. The accuracy of this ratio is enhanced by the use of simultaneous RBS, which provides the crystal thickness for use in self absorption calculations.
Measurements on over 18 proteins as well as on a virus have been performed over the last few years and the method and results are discussed. The technique is now a valuable and reliable addition to those available to the structural biologist.

Session EPS: Applications in Earth and Planetary Sciences

EPS-O1

NUCLEAR MICROPROBE ANALYSES OF LIGHT AND VOLATILE ELEMENTS (C, F, Br) IN MAGMAS

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The major volatile elements exsolved during the ascent of magmas are H_2O, CO_2, S and halogens to different extents. The melt inclusions (MI) trapped during the crystal growth, at high temperature, allow to study the volatile behavior in magmas prior to the eruption and at different steps of the magma evolution.

In order to assess the volatile abundance in magmas, an effort has been done during the last 5 years to analyze different elements (C, F, Br) in reference silicate glasses and MI in natural olivine crystals using the nuclear microprobe. Carbon and F were analyzed using the nuclear reactions $^{12}C(d,p)^{13}C$ and $^{19}F(p,p'\gamma)^{19}$, respectively and Br by PIXE. The same samples were also analyzed by other methods as Infra-Red Spectroscopy (FTIR) for C and electron microprobe for F and Br. The interests and limits of the methods will be discussed for these elements.
The detailed results will be presented in the case of carbon in MI trapped in olivines (Fo_{89-82}) from Stromboli volcano (Aeolian islands), for which the concentrations decrease from 1650 to <40ppm CO_2, as the CaO/Al_2O_3 ratio varies from 0.85 to 0.36. The variation in carbon concentrations is discussed as the result of the both compositional effect of the silicate glass and degassing process during magma ascent.
The second application of nuclear microprobe in analyzing minor elements in silicate matrices will be detailed in the case of Br. In order to assess the Br degassing during the volcanic eruptions and the solubility of Br in silicate magmas, we have combined results on Br concentrations in albitic synthetic glasses whose concentrations vary from 890 and 5 ppm [1] and new results obtained on silicate glasses and MI from volcanic crystals.
[1] Bureau et al., Earth. Planet. Sci. Letters (submitted).

EPS-O2

MINERAL SPECIFIC TRACE ELEMENT CONTENTS OF INTERPLANETARY DUST PARTICLES

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Interplanetary dust particles (IDPs), collected in the stratosphere, are small extraterrestrial particles (10-20 μm) with micron and submicron sized mineral grains. Relative to solar abundances certain volatile trace elements are strongly enriched in IDPs. Two possible reasons for these enrichments are currently discussed:
1) IDPs represent a new type of solar material that is more primitive than CI chondrites [1], or 2) the enrichments are due to atmospheric contaminating processes [2].
Identifying the location of the volatile trace elements will help to quantify the relative importance of the two causes. In case (1) one would expect to find native trace element contents in specific host phases, whereas contaminating processes that were demonstrated to exist [3,4] tend to concentrate enriched trace elements at the surface regions. In addition, the determination of their host mineral(s) allows to compare these phases with those in carbonaceous chondrites that are the most *primitive* solar system material available for laboratory analysis.
We analysed thin sections of individual IDPs with PIXE, STIM and RBS. These new results will be presented in comparison to data obtained with TOF-SIMS and TEM of the same particles. In addition technical aspects of the sample preparation method and of the data reduction will be discussed.
Acknowledgement: We thank Ursula Heitmann, Institut für Planetologie, Münster, for preparing the IDP thin sections.
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EPS-O3

TRACE ELEMENT ANALYSES OF APATITE FROM FOSSIL BONES OF MARINE ORGANISM AND ITS GEOLOGICAL APPLICATION

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The francolite, a carbonate-fluorapatite with >1% F and appreciable amount of CO_2, from fossil bones of marine organism is applied as one important tracer mineral in reconstructing the isotope composition of paleo seawater. Isotopes of the structural components O, C and S of francolite are used for reconstructing the temperature of formation, the age and the diagenetic environment, respectively. The paleo land - sea distribution and oceanography can be reconstructed by isotopic work using, e.g. neodymium. It is the purpose of this work to evaluate structural and chemical alteration of fossil apatite using the combination of:
- micro-PIXE for quantitative REE analyses,
- CL-spectroscopy/-microscopy for analysing the REE incorporation apatite/francolite lattice position and mapping the relative REE distribution
- ESR-spectroscopy for determining structural defects achieved by REE^{3+} incorporation in divalent cation position of apatite/francolite
The micro-PIXE analyses of fossil francolite samples from conodonts of Triassic age and shark teeth of quaternary age indicate that most of the REEs are significantly enriched in Triassic conodonts comparative to the average shale standard (NASC). The PIXE analyses of fossil conodonts revealing the REE-distribution not to be in balance with the REE-distribution of seawater and recent fish bone debris. Strong inhomogen lateral REE-distribution in fossil conodont material is shown by CL-mapping and most probably not being a vital effect. It is not clear on which scale the documented enrichment occurs, but high REE-concentrations and enrichment of Ce and some REEs (Sm, Eu) are most probably affected by the chemical composition of surface water and/or burial ground water. Hence, the resulting REE signal from fossil francolite used in this study is the sum of vital and post-mortem incorporation. The latter is basically controlled by local chemical and physical properties of the fluids. Consequently, if fossil francolite from marine organism does not show the REE pattern of seawater, these samples are most probably chemically affected by diagenetic processes and certainly not useful for reconstructing the isotope composition of paleo seawater.

EPS-O4

ANALYSIS OF URANIUM DISTRIBUTION IN ROCKS BY μ-PIXE

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The mechanism of fixation and migration of U in groundwater is key to understand the earth surfacial U cycle. Many kinds of electromagnetic waves and particle beams are used as analytical probes for the investigation of uranium migration. Particle induced X-ray emission (PIXE) is one of the possible methods detecting the elements in the level of ppm. A micro-PIXE (μ-PIXE) analyzing system was developed in the TIARA facility of JAERI for various elemental analyses with sub-micron level spatial resolution. We have performed the studies to analyze the distribution of U in rock samples collected at the Koongarra uranium deposit, Australia. The traditional probes of SEM-EDS and EPMA were also applied to measure the U distribution in the rock samples to compare the advantages and disadvantages of μ-PIXE – EDS analysis with those of traditional methods.
The spatial resolution of μ-PIXE was higher than that of SEM-EDS, and was comparable to EPMA. The detection limits of the probes are in the order of μ-PIXE < EPMA < SEM-EDS. These results indicate that μ-PIXE can give us sufficient data on U migration not only in natural rock samples but in solid samples obtained in the laboratory.

EPS-O5

NUCLEAR MICROSCOPIC TECHNIQUES IN THE STUDIES OF HYDROGEN CHEMISTRY AND DYNAMICS IN DIAMOND

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Hydrogen and oxygen are known to play a significant role in the growth of diamond (CVD), and hydrogen has been reported to influence its electrical properties. Resonance RBS and Forward Scattering in Channeling Transmition mode with thin diamond single crystals were used to determine the concentration and lattice site occupancy of surface oxygen. Despite some successes made in studying hydrogen in diamond, the configuration, Chemistry and behaviour are not yet well established. Ttrapping and diffusion of hydrogen in a range of different types of natural an synthetic diamond have been investigated under different conditions such as high dose hydrogen implants after creation of vacancies, plasma loading of hydrogen in both intrinsic and semi-conducting diamond. ERDA and NRA were used to map the hydrogen distrbution in three dimensions in polycrystalline CVD diamond with respect to trapping at grain boundaries.

Session EB: Applications in Environment and Botany

EB-O1

ELEMENTAL MICROANALYSIS IN BOTANY: NUCLEAR MICROPROBE AND COMPETING METHODS

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Botany is one of the research disciplines where microanalytical analysis of minor and trace elements can substantially contribute to the existing knowledge and to the results obtained

using a more classical methods. This applies to solving problems in research areas such as plant physiology, agriculture and environmental pollution. An overview of recent applications is presented. Special emphasis is made on an update of nuclear microprobe applications, following earlier review on that subject [1]. Problems solved using other microanalytical techniques such as EDX, SIMS, LMMS, SXRFM and EELS are also reported. Quantitative capabilities of each method, including quantitative mapping, are reviewed. Proper methods of specimen preparation are described and discussed.

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EB-O2

MANGANESE PROFILES IN FRESHWATER MUSSEL SHELLS

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The pollution of the marine environment and rivers is of growing concern, however many pollution events, particularly in remote areas, remain undetected. Bones and shells of organisms living in the aquatic environments can be used as monitors for both contemporary and historical pollution events. In order to use these records, various factors such as patterns of bioaccumulation over the life span of the animal need to be understood.

Ion microprobes offer a tool to measure metal (impurities) at very low concentrations with a high lateral resolution. This paper describes the use of the heavy ion microprobe at ANSTO to measure Mn profiles in freshwater mussel shells. The ANSTO heavy ion microprobe can focus 9 MeV He, 25 MeV C as well as 35 MeV Cl ions to spot sizes down to 5μm. With the microprobe manganese profiles across mussel shells were taken after 10 days exposure of the shells to increased Mn concentrations in the water, in order to determine the pattern of accumulation in the shells. Additionally 2-dimensional imaging was used to investigate the accumulation of trace elements in particular areas.

EB-O3

WEATHERING AND METAMICTIZATION OF ZIRCON : NEW CONSTRAINTS FROM MICRO-PROBE MEASUREMENTS.

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Zircon (tetragonal $ZrSiO_4$) is the oldest mineral found at the Earth's surface. Its resistance to dissolution is exceptional compared to that of other silicate minerals. Because of this important property, zircon is widely used in tracing and dating geological processes and it is considered as potential waste matrix for the plutonium. However, zircon may loose its resistance in some environments (hydrothermal fluids, tropical soils) and the use of zirconium as an immobile element in weathering processes has been questioned. In addition, metamictization, i.e. the loss of crystal periodicity due to the accumulation of radiation induced defects, strongly modifies the properties of zircon.

Surprisingly, little attention has been paid on the role of metamictization on the weatherability of zircon in natural environments. Here, we report new results obtained on zircons sampled in the sediments and the soils of the Amazonian Basin (Brazil). These zircons originate from the Precambrian Guyana shield (0.5-3 Ga). Their actinide (U, Th) concentrations were determined between 10-7000 ppm by proton induced X-ray emission (PIXE) micro-analysis. This range of concentration is consistent with that commonly reported for crustal zircons. Thus, the studied series can be regarded as representative. Their metamictization degree, expressed as a radiation dose and determined by Raman micro-probe, is between 5.10^{14} - ~3.10^{15} alpha-decay/mg. The maximum degree of metamictization coincides with the degree for which radiation damages become predominant. It is also significantly lower than that expected for the more actinide enriched zircons. This strongly suggests that more damaged zircon disappeared during weathering processes. Otherwise, for the observed metamictization range, the soil formation does not appear to affect the zircon population. Since Rutherford backscattering (RBS) measurements shows that no protective Zr-enriched layer forms at the surface of these zircons, their resistance to weathering can be attributed to the zircon crystal structure.Consequently, our results suggest that metamictization has a major control on zircon weathering at the Earth's surface.

EB-O4

NUCLEAR MICROPROBE ANALYSIS OF LICHEN SURFACES

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Elemental analysis of upper and lower cortical surfaces of the lichen *Xanthoparmelia chlorochroa* have been performed using microbeam proton induced x-ray emission (PIXE). The analyses demonstrate that element distribution patterns on lichen surfaces are heterogeneous and complex. The unique characteristics of lichens present some interesting analytical challenges that will be addressed. The abundance of inorganic particles and diversity in particle size and composition confirm the long-standing hypothesis that particulate entrapment and decomposition is a primary mechanism for nutrient accumulation employed by lichens. The implications of this particulate study to the role of lichens as biomonitors of air pollution will also be discussed.

EB-O5

CHARACTERISATION OF ZINC IN A DREDGED SEDIMENT DEPOSIT AND IN THE SUBSOIL BY COUPLING μPIXE, μRBS, AND μEXAFS

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In flat lands, sediments deposited in ship-canals are periodically dredged for maintenance and are generally disposed on agricultural soils along banks. In industrial areas, the canal sediments are often polluted by heavy metals and these practices are hazardous because toxic metals can migrate to the underlying soil and groundwater. The chemical risk depends on the mobility and bioavailability of metals, which themselves depend on their speciation.
The aim of this study is (i) to identify the speciation of zinc in a contaminated dredged sediment (6000 ppm of zinc) collected in a highly polluted area in the North of France, (ii) to determine which particular Zn-containing mineral species are able to release zinc from the sediment to the non polluted subsoil, and (iii) to identify the mechanisms of Zn immobilisation in the subsoil. Owing to the highly heterogeneous nature of sediment and soil, bulk analyses should be completed by investigations at the micron scale. μPIXE and μRBS are well adapted to our study because (i) μPIXE gives information on the spatial distribution of elements with Z>12, (ii) μRBS allows to obtain the distribution of the elements with depth of the sample, with a good selectivity for thin heavy element layers deposited on light matrix (iii) both techniques present multielemental analytical capabilities and high sensitivity. μEXAFS allows to obtain structural information on micron-scale particles.
Coarse particles of the sediment (500-2000 μm) contain high amount of zinc (~1.5%) and were studied by μPIXE and μRBS. μRBS results showed that Zn, Fe and S are concentrated at the surface of the grains and μPIXE elemental maps allowed to identify an association between Zn and S, irregularly distributed on the particles. Sometimes, Fe is associated with S and Zn. Sphalerite (ZnS) was identified by μEXAFS as the major Zn-bearing phase.
After 18 months of deposit, the zinc concentration in the subsoil increased from originally ~ 70 ppm to ~ 300 ppm. This enrichment at least partly results from a direct transfer of particles from the sediment to the soil, but a migration of dissolved Zn, released by the weathering of ZnS, followed by its immobilisation in neoformed phases can not be excluded. μPIXE, μRBS and μEXAFS experiments are in progress to distinguish the transferred particles from the neoformed phases.

Session AA: Applications in Art and Arcahelogy

AA-O1

EXTERNAL MICROBEAM SETUP AT THE CNA (SEVILLA) AND ITS APPLICATION TO THE STUDY OF TARTESIC JEWELLERY

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A new commercial external microbeam system, from Oxford Microbeams Ltd., has been installed recently at the +45° beam line of the 3 MV Pelletron accelerator at the Centro Nacional de Aceleradores (CNA). The system includes quadrupole doublet focusing lenses, two sets of slits, a faraday cup and an accurate positioning system based on an optical microscope with a CCD camera, a laser and a video controlled positioning machine that allows motorised displacements on the three axes. Also a BPM (beam profile monitor) and a quartz viewer have been added to facilitate the microbeam production.

The first PIXE application of the new facility was made in the field of Archaeometry, on the study of Tartesic gold artefacts (700-500 B.C.) from Ébora, Mairena and other archaeological sites. Preliminary analyses, mainly devoted to identify the soldering procedures of small details in gold jewels, were performed on the Scanning Proton Microprobe Unit of Oxford University (Oxford, UK), under the frame of the European COST-G1 Action. The study has been continued at CNA in order to collect a significant number of measurements on several artefacts relative to the soldering procedures that were employed by our ancestors along the valley of Guadalquivir River after the Phoenician colonization.

AA-O2

ANALYSIS OF PREHISTORIC POTTERY FINDS FROM THE BALATON REGION, HUNGARY BY MICRO-PIXE TECHNIQUE

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The investigated site, Vörs-Máriaasszonysziget lies in the middle of the Little Balaton marshes, on a long sandy peninsula protruding the beach of former Lake Balaton, the largest fresh-water lake in Central Europe. The area belonging to the village has been intensively studied by archaeologists due to water management constructions in the past decades. The site itself was also subject to rescue excavations several times. Remains of Early Neolithic settlement have been identified recently [1]. New excavations were started in 1999 to save more parts of this valuable site. Archaeological units belonging to several cultures were found in clear stratigraphical position. Samples from all closed units (with various ages, i. e. Early Neolithic, Early Copper Age, Late Copper Age and Early Bronze Age) were taken for sieving for scientific analysis of the small and microscopic facies of the material which is otherwise lost for analysis. Pottery fragments of coarse fraction of these sieved samples were selected for analysis.

The scientific investigations were taken by micro-PIXE technique at the Institute of Nuclear Research of the Hungarian Academy of Sciences (ATOMKI). The reason for the use of microbeam was that in several cases the determination of bulk composition of the potteries is not enough to explore relations between the elemental concentration data and the provenance and/or manufacturing method and/or the age of the samples. Therefore, both the bulk composition and the microstructure of the specimens were studied. In order to determine the concentration of light, main and heavier, trace elements in a single run the x-rays were detected by two Si(Li) detectors simultaneously. The correctness of our hypothesis, i.e. there is a correlation between the microscopic contents of the potteries and the raw material source and/or preparing technique for the vessels is discussed in this work.

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AA-O3

ALBEMARLE IRON WORKS (1771-1772): WHY DID THIS OPERATION FAIL?

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In December of 1770 the Albemarle Iron Works, Albemarle County, Virginia, U.S.A. was formed to build a blast furnace for producing cast iron. This furnace was put into operation in September of 1771 but was closed permanently in June of 1772; no usable iron was ever produced. The reason for this failure is not clear, but it has been suggested that the cause was the use titaniferous iron ore. The presence of high Ti would result in a very high viscosity slag, which in turn would not allow for the separation of the iron from
the slag and, thereby, slow down the operation of the furnace. Slag, recently taken from the site, has been analyzed by both PIXE and SEM, and the results confirm the high Ti content. Another indication that the furnace was having difficulties was that the slag was not typical glassy slag. Furthermore the iron prills entrapped in the slag showed a high phosphorus content which would result in an iron too brittle for practical use.

AA-O4

PHOSPHORUS LOCALISATION AND QUANTIFICATION IN ARCHAEOLOGICAL IRON ARTEFACTS BY MICRO PIXE ANALYSES

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Because it segregates at grain boundaries, phosphorus increases the embrittlement of iron and steels. Thus, it is important to eliminate it from the metal during the ironmaking process. Several recent studies showed that ore containing important amount of phosphorus were used in Europe to obtain iron with the direct process (solid state reduction) but also with the indirect process (liquid state reduction that conducts to cast iron and refining of this cast iron during a second stage to obtain iron or steel). The phosphorus quantification in archaeological iron artefacts is of great importance to understand ancient ironmaking processes. It also provides information about the quality of iron obtained with these processes using phosphorus ores.

The metallographic structure of ancient iron (before the end of the 19^{th} century) is very heterogeneous and often contains an important amount of non metallic slag inclusions coming from the reduction stage for the direct process and from the refining stage for the indirect process. Thus, bulk analyses of ancient iron artefacts is not sufficient. It is necessary to use a microbeam to detect the minor elements like silicon, sulphur or phosphorus in the iron matrix. Moreover, to understand ancient ironmaking processes it is fundamental to study the role played by slag that can entrap special elements. Particularly, for phosphorus, the determination of the slag inclusion composition (iron content, basicity) is of great significance. We will present the study of several archaeological artefacts representative of the direct and the indirect processes containing high phosphorus slag inclusions. This will be completed by the analyses of reconstituted samples by experimental archaeology. For the direct process, ironsmelting was proceeded with oolithic ore from Lorraine (France) containing about 2% of phosphorus oxide. For the indirect process, phosphorus cast iron was oxided under the condition of an ancient refining hearth to obtained dephosphorized iron. For this purpose, slags of different compositions were used.

For all these samples, phosphorus distribution in the metal and in the slag inclusions were analysed using the micro-PIXE technique.

AA-O5

PIXE ELEMENTAL MAPPING ON ORIGINAL MANUSCRIPTS WITH AN EXTERNAL MICROBEAM. APPLICATION TO MANUSCRIPTS DAMAGED BY IRON GALL INK CORROSION

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Metallogallic inks were largely in use for writting untill the beginning of the twentieth century. All these inks are basically prepared with a mixture of a metallic salt and a solution of gallic acid. Plenty of recipes have been reported by different authors, mentioning a large panel of ingredients, each of them being more or less pure. All these ingredients may have an influence on the degradation process of the paper. A great work has been done to explain the role of acidity and iron content on the paper degradation. Yet the variety of visual aspects of original inscriptions suggests that many side effects may occur, and very little knowledge has been collected through scientific analysis in order to explain these effects.
In this article we report some preliminary measurements carried out with a 3 MeV external proton microbeam on original valueless manuscripts. The non-destructive aspect of the method has been tested, as well as its reproducibility. Then, PIXE analysis have been performed on 200x200 micronsquare spots to identify the elemental composition of all inscriptions. The visual aspect of the ink and its iron, zinc, lead, and copper contents will be commented.
PIXE mappings have also been performed on some particular samples corroded by iron gall ink. The migration of some elements such as calcium, sulfur, iron and copper out of the inscriptions has been observed in some cases. These migrations can be related to halos which are visually observed around inscriptions. Several mappings will be presented and the role of each detected element will be discussed.

Session ALT: Alternative Techniques

ALT-O1

HIGH RESOLUTION NUCLEAR AND X-RAY MICROPROBES AND THEIR APPLICATIONS IN SINGLE CELL ANALYSIS

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Investigations of uptake of different chemical compounds by single cells has become a very popular topic and has been performed with different microanalytical techniques using electron, laser, ion and X-ray beams. With the advent of nuclear microprobes with submicron

resolution it has been possible to investigate distribution of different elements inside individual cell with sensitivity of parts-per-million (ppm).
Recently, with X-ray beams focused to below 100 nm and high intensity obtained from third gereration synchrotron facilities it become possible to perform analysis on single cell with sensitivity of parts-per-billion (ppb). Synchrotron radiation offers also a unique possibility to investigate an oxidation state for different elements.
In this paper authors will present and discuss data from experiments on single cells performed with high resolution nuclear and X-ray microrobes.

ALT-O2

ANCIENT ARTEFACTS AND MODERN ANALYTICAL TECHNIQUES - LASER ABLATION ICP-MS EXEMPLIFIED ON ANCIENT GOLD COINS

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One important question in archaeology is the origin of ancient precious metals, e.g. the provenance of Celtic gold. In principle, provenance determination should be possible by comparing trace element concentrations of sources and artefacts. However, the analyses should be performed with the smallest sample consumption possible and, moreover, previous attempts in this direction have not been conclusive.
Laser ablation (LA) is a sampling technique which is well suited for such precious artefacts. Using the example of Celtic gold coins - so called Regenbogenschüsselchen - the sampling technique is described. In addition, the benefits of LA attached to an ICP-MS with a sector field mass spectrometer and multiple Faraday detectors are reviewed. It will be shown that Osmium isotope ratios determined with this method are a valuable guide for determining the provenance of Celtic gold.
The precision and accuracy achieved with LA-ICP-MS are discussed. Corrections for chemical interferences and mass bias are exemplified. Within this scope, the requirement for a self consistent set of isotope masses and ratios is shown.

ALT-O3

COMBINED USE OF XPS, FTIR, NMA AND TEM TO STUDY SURFACE REACTIVITY OF HYDROXYAPATITE

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Apatite minerals are envisaged as components of the clay engineered barrier for the storage of the nuclear waste. The apatite minerals are known to exhibit strong retention properties towards dissolved ionic species. Our work is devoted to the determination at a macroscopic and microscopic scales of the surface sites of hydroxyapatite (HAP) and of its chemical reactivity towards heavy trace elements (fission products of ^{235}U or chemical analogues). A spectroscopic approach is developed in this work by using different complementary

techniques as : X-ray Photoelectron Spectroscopy (XPS) and Fourier Transform Infra Red spectrophotometry (FTIR) to identify reactive surface sites of the mineral at a macroscopic scale ; Transmission Electron Microscopy (TEM) to evidence the nanostructure of the mineral and Nuclear Microprobe Analysis (NMA) by coupling μPIXE and μRBS to determine concentrations and distribution (sorption or incorporation) of heavy elements sorbed onto HAP after contact.These different techniques have been applied to the granulometric fraction 63-80 μm of a synthetic and purified hydroxyapatite ($Ca_{10}(PO4)_3(OH)_2$) from Biorad® put in contact of different aqueous solutions containing cadmium or selenium. From the nuclear microprobe results, it appears that the fixation of cadmium and selenium corresponds to an incorporation into the hydroxyapatite mineral. Considering the TEM images, this incorporation seems to be conditioned by the nanostructures of the hydroxyapatite mineral. According to XPS and FTIR data, the chemical environment of phosphorus at the surface and into the mineral seems be dominated by the species CaHPO4.
Further studies would be oriented to XPS and FTIR investigations concerning the chemical environment of selenium that presents the interest to be absent from the structure of HAP.

ALT-O4

PROGRESS OF BEIJING SYNCHROTRON RADIATION X-RAY FLUORESCENCE MICROPROBE ANALYSIS

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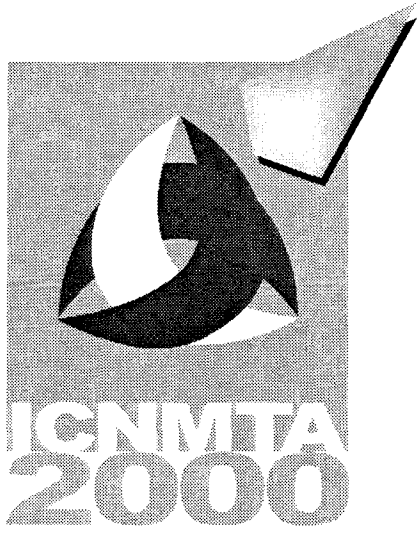
In this paper, the new progress of Beijing synchrotron radiation X-ray fluorescence microprobe analysis facilities was presented, and minimum detection limits of trace elements were tested by using several kinds of standard reference specimens. Some new experimental results of the applications on biology, medical science, petroleum, geology and materials science were also reported here. The first successful nondestructive analysis of single fluid inclusion by this method in China and scanning analysis of human bone tumor slice were detailed.



Abstracts
Poster Sessions

Session MF : Nuclear Microprobe Facilities

MF-P1

A LABVIEW™ BASED SCANNING AND CONTROL SYSTEM FOR PROTON BEAM MICROMACHINING

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LabVIEW™ is steadily gaining in popularity as the programming language of choice for scientific data acquisition and control. This is due to the vast array of measurement instruments and data acquisition cards supported by the environment, and the relative ease with which advanced software can be programmed. Furthermore, virtual instruments that are designed for a given system can be easily ported to other LabVIEW™ platforms and hardware. This paper describes the new LabVIEW™ based scanning and control system developed specifically for proton beam micromachining (PBM) applications. The new system is capable of scanning figures at 16-bit resolution with improved sub-microsecond scan rates. Support for electrostatic beam blanking and external dose normalization using a TTL signal have been implemented. The new software incorporates a semi-automated dose calibration system, and a number of novel dose normalization methods. Improved algorithms for generating and designing input files have also been added. These additions have allowed us to make major improvements to side-wall definition and smoothness for an arbitrary proton beam micromachined shape. Limitations of the current beam scanning hardware are discussed in light of new results obtained from micromachining experiments performed in SU-8 photoresist.

MF-P2

THE USE OF SOLENOID LENSES IN A TWO STAGE NUCLEAR MICROPROBE PROBE-FORMING SYSTEM

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Typical demagnifications which have been produced using quadrupole multiplets in nuclear microprobes are between 20 and 100, with working distances of 10 to 20 cm. In comparison, a scanning electron microscope might achieve a demagnification of several thousand using several solenoid lenses to progressively demagnify the focused beam spot, resulting in a final spot size as small as 10 nm.
Whilst quadrupole lenses provide a strong focusing field, they also have higher intrinsic spherical and chromatic aberrations than solenoid lenses, and also higher parasitic aberrations which arise from the difficulty of adequately aligning the several quadrupole lenses in a probe-forming multiplet to the required tolerances. Given the fundamentally superior spatial resolution which is attainable using solenoids, it is worth studying further to see whether a suitable solenoid lens can be constructed for use in two stage microprobes.
This paper studies the optics of a two stage microprobe system comprising a magnetic quadrupole quadruplet followed by a strong solenoid lens. The orthomorphic Melbourne

magnetic quadruplet is chosen as the first probe-forming stage. Results are presented showing the performance of this two stage system and compared with a two stage system comprising two sets of quadrupole lenses.

MF-P3

AN ELECTROSTATIC BEAM ROCKING SYSTEM ON THE SURREY NUCLEAR MICROPROBE

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The use of two sets of magnetic dipoles, producing opposite fields, to rock a focused MeV ion beam over the surface of a crystalline sample is now well established in several nuclear microprobe laboratories. Such a `beam rocking' system allows ion channeling analysis from micron-size regions of the sample to be measured, with a beam displacement over the sample surface of a few microns, and no requirements for an automated goniometer.
While magnetic beam rocking systems are ideal for many applications, they are limited in the speed at which the beam can be rocked in angle owing to hysteresis effects, which also cause problems of non-reproducibility of the beam displacement on a micron scale. Also, heavier ions are more difficult to rock with a magnetic beam rocking system, whereas an electrostatic beam rocking system gives a rocking angle which is mass independent.
This paper describes the construction and uses of a fast electrostatic beam rocking system, which uses two sets high voltage plates driven in opposition at high frequencies. The optics of this beam rocking system, in which both sets of deflection plates are located before the quadruplet lens formation are discussed. The uses of this system to rapidly image the location of crystal planes and axes, and to carry out rapid channeling analysis is presented.

MF-P4

CHARACTERISATION OF THE SUPERCONDUCTING MULTIPOLE LENS OF SNAKE

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The main demagnification of the ion beam at the Munich nuclear microprobe facility SNAKE (Superconducting Nanoscope for Applied nuclear physics (Kernphysikalische) Experiments) is done by a superconducting multipole lens. It is designed to focus 25 MeV protons and heavy ions with energies up to 200 MeV q^2/A to a submicron beam spot [1]. The intrinsic aberrations of the magnetic lens due to the large aperture of ±7 mm and the contribution of the fringe fields are compensated by higher order correction fields, determined via ray tracing. In order to get an accurate information of the fringe field the 3 dimensional shape of a pole edge is formed as an equipotential surface of an analytical fringe field function. Additionally, superconducting correction mechanisms, such as foils and current loops, for reduction of parasitic aberrations are introduced.
All important magnetic properties of the lens have been tested offline with a special designed magnetic multipole detection device [1]. A complete set of multipole components along the

magnetic axis of the three singlets have been measured. This contains information about rotational and axial alignment, fringe field components and higher order correction components and the focussing strength. The achieved quadrupole field gradient is 1T/cm at a current of 8A. Detailed results as well as the investigations on the superconducting correction mechanisms will be presented.
In addition to this multipole field mapping a novel type of online ray tracing has been developed. Placing a tungsten grid (65 μm hole diameter, 350 μm spacing, 3% transparency) in the beamline behind the divergence slits provides the possibility to prepare a series of pencil beams entering the focussing lens. Observing the pattern - produced in a scintillator crystal in the focal plane - with an optical microscope, is a fast and easy way to find a focus and to study intrinsic and parasitic aberrations. First experiments with a 90 MeV ^{32}S beam will be shown.

[1] G. Datzmann, G. Dollinger , G. Hinderer and H.-J. Körner, Nuclear Instruments and Methods B158 (1999) 74.

MF-P5

SOME EXPERIENCES ON THE WAY TO BIOLOGICAL SINGLE ION EXPERIMENTS

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One of the experimental cornerstones on the way to the irradiation of single biological cells with single, precisely aimed high energy ions is the reliable detection of hits. Another one is the necessity to keep the cells wet during irradiation. Therefore, the cells have to be separated from the microprobe vacuum by an thin window.
This window should be extremely thin to minimise scattering. At the same time it should be strong enough to withstand atmospheric pressure and it should be radiation hard.
During a previous conference Cholewa et al.[1] proposed to use a thin diamond foil to serve as vacuum window and as part of the hit detection system. Later, Kamiya et al. [2] tested the hit detection efficiency for Alpha-particles using B doped diamond. To confirm these results for heavy ions we have checked the relative merits of diamond foils compared to that of other materials. In addition to that we have looked for other sources of problems which might prevent us from hitting single cells with the desired accuracy of 0.1%.
[1] M. Cholewa, A. Saint, G.J.F. Legge, and T. Kamiya, NIM B, 130(1997) 275-279
[2] T. Kamiya et al., Appl. Phys. Lett. **71** (13) 1997, 1875-1877

MF-P6

THE NEW EXTERNAL BEAM FACILITY OF THE OXFORD SCANNING PROTON MICROPROBE

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This paper describes the development of a high spatial resolution external beam facility on one of the beamlines of the Oxford Scanning Proton Microprobe tandem accelerator. Using a magnetic quadrupole doublet to focus the beam through the Kapton exit window a beam diameter of < 50μm fwhm can be achieved on a sample located at 4 mm from the exit window. The facility is equipped with two Si-Li x-ray detectors for PIXE analysis of light

and trace elements respectively, a surface barrier detector for RBS analysis and a HP-Ge detector for gamma ray detection. The mechanical and beam-optical design of the system is described and a selection of results from test samples are presented. An example of the application of the system to study surface layers on large museum objects is described

MF-P7

DEVELOPMENT OF THE ULTRA-FINE MICROBEAM APPARATUS

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Semiconductor processing technique is glowing rapidly. Line width of relief patterns is reaching to 0.1 μm. The microbeam having beam size of 0.1 μm should be a useful tool for a non-destructive inspection of a fine structure of the semiconductors.
The method to produce ultra-fine microbeam was designed employing electrostatic field lens, so-called "acceleration lens", which has double function of a single aperture lens and simultaneous beam acceleration [1]. The ultra-fine microbeam apparatus was developed on the basis of this design so as to produce beams of 0.1 μm order for H_2^+ with energy of 100 keV.
The apparatus consists of the duoplasmatron-type ion source, the acceleration lens system and the beam measurement system. The ion source was made to supply 100 eV H_2^+ with 1 eV energy width. The lens system was designed to minimize a spherical aberration to be less than 0.1 μm involving computer calculations. The beam current measurement by cutting a beam path using the knife-edge was applied to estimate a beam size accurately. The positioning of the Faraday cup suppression electrode and the sharpness of the knife-edge were improved to obtain better reproducibility of the current measurement and higher resolution in the estimated beam size, respectively. The H_2^+ beam of about 30 keV with the size of 0.5 μm order has been produced using this apparatus so far.
[1]A. Isoya, Proc. Int. Conf. on Application of Nuclear Techniques. Crete, Greece, (1990)P.334

MF-P8

PARALLEL BEAMS FROM NUCLEAR MICROPROBE LENS SYSTEMS

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A nuclear microprobe lens system is normally used to focus a diverging beam into a fine probe. It does this by making the astigmatism terms in the first order transfer matrix: (x/θ) and (y/ϕ) zero. Thus the position of a ray vector in the image plane (x, y) will not depend on the divergence of the ray vector in the object plane (θ, ϕ). This is accomplished at the expense of magnifying the object divergence by factors (θ/θ) and (ϕ/ϕ) in the xoz and yoz planes respectively leading to relatively steep convergence angles at the Gaussian image plane where the specimen is located.

In some applications of ion beam analysis, a parallel beam is required. Precision channeling measurements are one example. In this case the magnified convergence angle of the beam can degrade the channeling measurement because the steeply converging ions may not be

guided into the channels of the crystal. The problem is particularly acute for measurements involving planar channels that are narrower than axial channels.
An alternative operation mode of the nuclear microprobe lens system is possible where the convergence angle of the beam after the lens system is made independent of the divergence angle of the beam prior to the lens system. In this case the demagnification terms in the first order transfer matrix: (θ/θ) and (ϕ/ϕ) are zero. This mode of the lens system does not produce a focused beam, so is most suitable for crystals without lateral structure. As an example, we present measurements from the <0001> alpha-Al_2O_3 axis which was channeled using various configurations to compare the relative improvement on the channeling depth and width of the sapphire axis and planes with the parallel beam. We find that there is indeed improvement with the parallel beam even compared to a collimated beam.

MF-P9

THE PIERRE SÜE LABORATORY NUCLEAR MICROPROBE AS A MULTIDISCIPLINARY ANALYSIS TOOL

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The Nuclear Microprobe at the Pierre Süe Laboratory is a facility exclusively devoted to microanalysis. The microprobe consisting in a single stage 3.75 MV Van de Graaff accelerator and two microbeam lines will be described. Simultaneous detection (x-rays, γ-rays, charged particles...) and imaging are routinely performed by a PC-based multi-parameter data acquisition system. Telescope mounting is frequently used for particle identification particularly when performing Nuclear Reaction Analysis.
A unique feature of the Pierre Süe Laboratory Nuclear Microprobe is the ability to analyze radioactive samples. One of the two beamlines has been specifically designed for that purpose. Radioactive environment requires suited target handling and detection setups.
Main application fields of the Microprobe are material, earth, planetary, environmental sciences and electronuclear related topics.

MF-P10

A NEW TEMPERATURE CONTROL AND DATA COLLECTION SYSTEM FOR TRANSIENT-IBIC MEASUREMENTS

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The new target chamber and data collection system for temperature controlled Transient or Time Resolved-IBIC measurements is outlined. The system developed at JAERI has been designed for both ultra-fast and relatively slow Transient-IBIC measurements as a function of temperature from 77K to 400K. The control system, implemented in the Labview environment, allows single ion scanning and transient acquisition on a range of oscilloscopes for an array of temperatures and bias. The modularity of the system allows its use for a broad range of experiments from SEU Scanning Transient Current measurements to SIDLTS charge transient measurements. In this paper, we discuss the software and hardware implementation and illustrate the use of the system on several technologically relevant samples.

MF-P11

THE NEW CRACOW SCANNING NUCLEAR MICROPROBE

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A new scanning nuclear microprobe with a short-length probe forming system has been designed and installed at the 3MV Van de Graaff accelerator in Cracow. The optimized probe forming system includes a divided Russian quadruplet of magnetic quadrupole lenses. A novel beam scanning system with ferrite-cored magnetic coils is used. Preliminary experimental results and some features of the MP are described. The design developed is promising for the next generation of compact and vertical nuclear microprobes.

MF-P12

THE HIGH-ENERGY HEAVY ION NUCLEAR MICROPROBE AT THE UNIVERSITY OF NORTH TEXAS

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The high-energy heavy ion nuclear microprobe at the University of North Texas has been in operation since Sept 1999. A high demagnification factor (~60) has been achieved in the system, using a probe-forming lens system (from MARC, Melbourne), which has a new Russian quadruplet configuration. The microprobe beam line is installed on a 3MV NEC 9SDH-2 Pelletron tandem accelerator, which provides an ultra stable high-energy for heavy ions ($\Delta E/E \sim 10^{-5}$). A negative ion-sputtering source produces a wide variety of ion species (H, Li, C, Si, and etc.). Due to the electron-stripping process in the tandem accelerator, the beam brightness achieved is relatively low, about one or two orders of magnitude lower than the best values found in single-ended machines. The spatial resolution of 2-3 microns has been achieved for 4.0 MeV carbon beams with a beam current of about 100 pA. The spatial resolution has been measured by RBS imaging over a 1000 lines/inch mesh copper grid. The direction for further improvement of the entire system and future applications in microelectronics are outlined.

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MF-P13

DEVELOPMENT OF A SINGLE ION IRRADIATION SYSTEM AT CENBG FOR APPLICATIONS IN RADIATION BIOLOGY

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Current trends in Radiation Biology are experiments at the cell level. This methodology remains one of the only possible ways to gather evidence of the response of an individual cell to a very weak irradiation dose, including extreme situations like exposure to a single ionising particle. A single event facility is currently under development at CENBG for applications in this field. The aim is to target individual living cells with an exact number of ions, the beam being delivered ion by ion in specific cell compartments (nucleus, cytoplasm...).

The system takes advantage of the focusing properties of the existing microbeam line at CENBG. Since the beam is positioned on individual cells by means of fast electrostatic deflection plates, it allows cell targeting with a higher frequency than collimated beams usually used for such applications. The microbeam line has been equipped with a removable final stage placed inside the lower part of the analysis chamber in order to constitute a versatile system working on demand, either in external beam mode or for classical analysis under vacuum. By this way, it was not necessary to construct a new line.
For single event control, the beam is deflected by fast response electrostatic plates triggered by the path of each ion through a transmission detector. For this purpose, two detectors have been designed. The first is based on a thin plastic scintillator foil optically coupled to two photo-multipliers tubes working in coincidence. However, because of the limited range of ions in the energy range of a few MeV, this set-up can be only used for irradiation in proton mode. For utilisation in alpha mode, a very thin low-pressure gas detector equipped with Si_3N_4 windows has been designed. The efficiencies of these detectors have been measured for MeV protons and alphas at levels higher than 99 %.

The equipment of the irradiation stage is currently under development. In this design, the aim is to target adherent cells cultured on thin polymer foils stretched on dedicated culture flasks. During the irradiation, cells will be kept in their culture medium, in a wet chamber positioned by a high precision x-y-z stage. Each cell of the monolayer will be localised on line by a fast optical recognition system coupled with a fluorescence microscope and a computer-controlled high sensitivity CCD camera. The whole automatic procedure is expected to be fast enough to allow a targeting rate of 10 cells per second.

MF-P14

STATUS OF ION MICRO-BEAM FACILITY AT INSTITUTE OF PHYSICS, BHUBANESWAR, INDIA

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The first ion micro-beam facility in India has been developed in the Institute of Physics, Bhubaneswar, around its 3 MV tandem (9SDH-2, NEC, USA) Pelletron accelerator facility in collaboration with the State University of New York at Albany, USA. The system uses a set of micro-polished slit assembly and a magnetic quadrupole doublet lens with $ME/q^2 \sim 16$ (MS 104-T slit assembly, QL300 lens, Dyer Energy Systems, USA) to focus the ion beam to the desired spot with a demagnification ratio between 10-15. The setup has the following capabilities; (a) RBS and PIXE elemental maps (four maps), (b) SEM in point mode, line mode as well as in area mode (maximum scanning area is about 500μm × 500μm with multi-zoom) (c) video image of area under investigation, (d) computer controlled x-y stage for the movement of the sample with 0.5 micron resolution. The necessary hardware and software have been developed for control of the beam, data acquisition and auto-pumping/venting of the target chamber, using a 486 PC. Although the design goal is to get close to 1 μm, at the moment it has been possible to focus a 3 MeV He^{2+} beam to a spot size of 4 μm with a current of about 80 pA.
As a first application of this ion microprobe we analyzed self-assembled epitaxial gold silicide islands, which show a shape transition and gold fractal structures [1] formed on Si(111) and Si(110) surfaces, using both RBS and PIXE maps. Because of asymmetric diffusion on Si(110) surfaces, long (~100) μm narrow (≤ 10 μm) self-assembled parallel silicide wires have been formed. A status report with detailed description of the system along with some of the results will be presented.

[1] K. Sekar, G. Kuri, P. V. Satyam, B. Sundaravel, D. P. Mahapatra, B. N. Dev, Phys. Rev. B51 (1995) 14330; Solid State Commun. 96 (1995) 871.

MF-P15

A FAST DATA ACQUISITION SYSTEM USING COMPLETE DEAD TIME CORRECTION AND CHARGE NORMALISATION

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The design and characteristics of a fast data acquisition system that uses simple electronics to interface the NIM electronics to a personal computer is discussed. By using the Linux operating system, fast data acquisition of up to 100k counts per second per station can be easily achieved. To correct for the large dead times that are implicitly inherent with such high data rates, dead time information is collected for each station. In fact, since the count rate varies as different regions of a sample are scanned, dead time information is collected for each pixel of each station. To generate quantitative charge normalised maps of spectral features, charge information is also collected using an ultra sensitive charge-to-frequency converter. To evaluate the data acquisition system, called MicroDAS, examples are provided that demonstrate the improvement to maps when dead time and charge information is used to correct the original raw energy data. These capabilities are implemented with the flexibility to use various types of scan and trigger modes. These include *raster, triangle, shape* and *line* scan modes in which scan progression is triggered by one of the *clock, charge, external, data* or *smart data* trigger modes. We conclude that the most quantitative accurate maps are generated when charge triggering with beam blanking is used.

MF-P16

THE SIMULATION OF BEAM TRANSPORT IN TANDEM ACCELERATOR BEAMLINE

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A WindowsNT[#] Delphi[##] application has been developed in order to simulate the behavior of the ion beam in the electrostatic tandem accelerator. The application enables us to follow the central beam and the envelope from the stripper channel, high energy acceleration tube, electrostatic quadrupole triplet and switching magnet to 30°, 10° and 0° beamlines, where additional optical elements such as slits and quadrupole lenses act on the beam. The program is used both to determine the optimal parameters of the tandem focusing system in its daily use and while designing new beamline configurations. The whole calculation is based on the linear approximation[1]. The initial coordinates of the rays are chosen randomly in the elliptically shaped phase space, which dimensions are determined by user. Up to 10000 rays is traced in order to calculate the envelope of the beam and the transmission through various slit systems. The pattern of the transmitted rays at the target cell plane can also be observed. The application is designed to be as fast and friendly as possible. It reacts promptly on every change of the parameters, which can be made from the menu or toolbars. It also allows us to save data, parameters, and print graphs and zoom regions of interest.

[1] Klaus G. Steffen, High Energy Beam Optics, John Wiley & Sons, New York, 1965.
[#] WindowsNT is a registered product of Microsoft Corporation.
[##] Delphi is a registered product of Inprise Corporation.

MF-P17

THE HEIDELBERG PROTON MICROBEAM GROUP 2000 - TECHNICAL DEVELOPMENTS AND APPLICATIONS

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We summarize the technical developments of our microprobe since the last paper [Traxel et al. 1995]. A new ion source and minor improvements in the hardware of the ion optics resulted in a brightness improvement of a factor 100 and enhanced stability. Further changes include the addition of a RBS detecting system and new features in our data acquisition system.
An overview on the current activities in biological, cosmochemical and geochemical field will be given.

[Traxel et al. 1995] *K. Traxel, P. Arndt, J. Bohsung, K.U. Braun-Dullaeus, M. Maetz, D. Reimold, H. Schiebler and A. Wallianos:* THE NEW HEIDELBERG PROTON MICROPROBE: THE SUCCESS OF A MINIMAL CONCEPT, Nucl. Instr. and Meth., B104, 1995, 19-25.

MF-P18

THE NUCLEAR MICROPROBES FACILITY AT IPAS

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A new dedicated high energy nuclear microprobe was installed at the Institute of Physics, Academia Sincia, Taiwan, in winter 1999. In this contribution we report on the main points of the installation procedure, the optical properties of the Oxford quadruple triplet lens system, and the results of the performance tests. Using a focused beam, the powerful techniques of ion beam analysis can be combined with spatial information to generate structure and elemental maps, line profiles or point composition analysis for elemental throughout the periodic table. This facility serves adverse range of application, from medical research to micro-electrons, in both academic and industry environments.

Session AT : Analysis Techniques

AT-P1

SURFACE CONTAMINATION BY SMEARING DURING POLISHING.

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Preparation of samples for PIXE analysis follow certain relatively standard procedures in order to present a smooth, uniform surface for analysis. Reports on the quality of the surface preparation are scant. We report on investigations of preparation of metallic samples for microprobe analysis. These samples are typically 1-5 mm^2 in area and are mounted either individually, or multiply as a square matrix, in a resin base for suitable support. The surface was polished using accepted standard procedures. Smearing of the sample material over the base has been considered negligible or non-existent due to the polishing method applied, however evidence has arisen indicating that this is not true, especially if several elements are present in the primary sample having greatly different hardness characteristics and/or are very ductile.

We report on microprobe studies of metals, including platinum-group metals prepared in the standard manner indicating evidence of smearing occurring. The potential influence of beam halo and beam scattering in the resin mount is also discussed.

AT-P2

ANALYTICAL APPROACH FOR DETECTING Pt, Pd AND Rh IN MATRICES RICH IN Cu AND/OR Ni.

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We discuss the approach used when detecting trace amounts of Pt, Pd and Rh in matrices that are rich in S as well as Cu and/or Ni, when using the NECSA PIXE microprobe. Due to the fact that light elements are present in high concentrations, an aluminium filter is essential to provide scope for the detection system to observe the trace amounts of platinum group elements. A description of the experimental system and results of various measurements and analysis procedures followed, will be presented.

AT-P3

ACCURACY OF PIXE ANALYSES

Sophie Gama, Marcel Volfinger, Claire Ramboz and Olivier Rouer - ISTO, Orleans

As part of the Geofrance 3D program, we analyse trace elements in various minerals from the La Chataigneraie mining district (Massif Central, France) with the proton miniprobe at CERI laboratory (Orleans, France) and with the proton microprobe at LPS laboratory (Saclay, France). The PIXE characterization of trace element patterns allows us to define the different sources of the W-mineralization. In order to evaluate the accuracy of our analyses, we have checked the different parameters of the standardization by irradiating various standards (silicates, oxides, metals, sulfides) containing various elements from Al to Ba.
Our measurements were performed using a Si(Li) detector placed at 45° to the target surface with the distance between the target and the detector being 45mm at Saclay and 30mm at Orleans. The filters between the detector surface and the X-ray source were a 135µm-thick Be and an Al funny filter: each Al filter (100 or 200 or 300µm) has a hole in its center (0,5mm diameter). The quantitative analysis is performed using the code and database "GUPIX", which requires the determination of the instrumental constant H. However, we observe that H usually varies with the X-ray energy. This behaviour can be explained making simulations using GUPIX database: we found that it is necessary to input the formal geometrical parameters which we measured (hole/detector surfaces ratio, filter thicknesses, detector/target distance) and then to vary them until we reproduce the experimental data. In the presented case study, we have entered the following parameters:
a hole ratio of 0,0069, Al 100µm and Be 135µm-thick filters and a distance of 31mm. The parameters that allow the experimental data to be reproduced are 0,0185 - 95 - 140 - 44, respectively. Our methodological approach allows the true geometrical parameters to be determined and thus, to ensure the accuracy of the quantative results. Some applications are given in examples.

AT-P4

MEASUREMENT OF LATERAL STRAGGLING USING A MICROBEAM

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There is today an increasing number of external beams and single event facilities under construction. Their design requires a precise estimation of lateral straggling in different materials, in order to estimate the degradation of the spatial resolution of the microbeam. This evaluation is of primary importance in the development of transmission particle detectors as

well as in the choice of thin exit windows used for the extraction of ion beams in air. Up to date, very few experimental data were available for lateral beam spreading measurement in the microscopic range, thus making difficult to ensure the reliability of simulation codes.
An original way has been developed to measure the lateral straggling of submicron proton and alpha beams after passing through polymer foils of different thickness. For this purpose, the microbeam line at CENBG has been used in STIM (Scanning Transmission Ion Microscopy) configuration (low fluence, submicron resolution) to deliver the ions in normal incidence through the polymer foils. To measure the lateral spreading of the beam passing through the foil, a metal collimator (10 μm aperture) was placed in front a silicon particle detector, centred on the beam axis and placed 6 mm behind the polymer foil. The whole surface of the detector was sealed by a gold film except the collimator aperture. When horizontally scanned in front of the collimator, the lateral distribution of the beam was measured step by step detecting particles passing trough the small aperture. Regular gaussian shapes were obtained for the lateral distribution of the beam.
Experimental data obtained during this experiment were compared to the result of simulations carried out using the SRIM Monte-Carlo code for 2.5 MeV protons and alphas in mylar and formvar thin films.

AT-P5

ON THE USE OF MICRO-CAPILLARIES FOR HIGH RESOLUTION NUCLEAR MICROSCOPY

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The use of micro-capillaries and micro-light guides have revolutionised optical microscopy, enabling the spatial resolution to become orders of magnitude less than allowed by the diffraction limit. This has led to a new field of application or Near-field Optical Microscopy. These capillaries consist of glass tubes with exit apertures of the order of some tens of nm. Micro-capillaries have been tested as ultra-collimators for nuclear microscopic applications. The obvious application is for use in single event studies, both in semiconductors and biological systems. The contribution also reports on the application of micro-capillaries in high-resolution imaging using traditional ion-beam techniques, such as PIXE and RBS.

AT-P6

DEVELOPMENTS IN DYNAMIC ANALYSIS FOR QUANTITATIVE PIXE TRUE ELEMENTAL IMAGING

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Dynamic Analysis is a method for projecting quantitative major and trace element images from PIXE event data-streams (off-line or on-line) obtained using the Nuclear Microprobe [1]. The method un-mixes full elemental spectral signatures to produce images that strongly reject artifacts due to overlapping elements, detector effects (such as escape peaks and tailing) and background. The images are also quantitative, stored in ppm-charge units, enabling images to be directly interrogated for the concentrations of all elements in areas of the images. Experience using the *Dynamic Analysis* method extensively with the new CSIRO-GEMOC Nuclear Microprobe has identified a number of areas where approximations in the basic method breakdown necessitating investigation of refinements to the technique. These areas include: (1) X-ray yield variation due to changing major element composition across a

complex sample, (2) background treatment and the spatial variation of background, and (3) the spatial variation of pileup "element" components. Furthermore, geological applications have demanded additional tools to: (1) extract average concentrations from areas enclosed by flexible arbitrary shapes to study melt inclusions, (2) project element concentrations onto lines or radii to extract line profiles and mineral zonation data, and (3) reduce noise in images.
The purpose of this paper is to provide an update of research on these issues, introduce new software designed to make *Dynamic Analysis* more accessible, and to illustrate the application of the method to many areas of geological application.
[1] C.G. Ryan, D.N. Jamieson, C.L. Churms, J.V. Pilcher, Nucl. Instr. Meth. B104 (1995) 157.

AT-P7

ERDA WITH AN EXTERNAL HE MICRO-BEAM: ADVANTAGES AND POTENTIAL APPLICATIONS

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ERDA based on He beam of a few MeV is now widely applied to various issues of materials science for which hydrogen or deuterium depth profiling can be of interest, e.g. the durability of silicate glasses or the properties of fusion reactor walls. To our knowledge, this analytical technique has been until now always implemented under vacuum. Since our IBA facility is entirely dedicated to the study of objects of cultural heritage, an external beam line has been specially built to perform any type of analysis, whatever PIXE with protons, RBS with helium ions or NRA with deuterons, directly on the object in an helium atmosphere. We thought that our set-up could work as well in ERDA mode. We have consequently designed a stage allowing an accurate scattering geometry (incident beam at 15° with respect to sample surface and emerging protons or deuterons at 15° in the forward direction). In the classical vacuum set-up, the scattered He ions are stopped by a thin foil. In our experiments, the He atmosphere between the beam spot and the surface barrier detector acts as the absorber. The results on standards made of silicon wafers on which known doses of H and/or D ions have been implanted, indicate that the analytical capability is as good as under vacuum. As a first application, we have checked that incorporation of H or D during mechanical polishing of sapphire is below the detection limit. The advantages of operating at atmospheric pressure will be stressed as well as several possible applications in the field of cultural heritage.

AT-P8

HIGH RESOLUTION 0°-SPECTROGRAPH AT THE ION MICROPROBE SNAKE

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As a new component of the Munich ion microprobe SNAKE (Supraleitendes Nanoskop für Angewandte Kernphysikalische Experimente) [1] a high resolution spectrograph has been installed behind the target chamber in 0°-geometry. The main part of the spectrograph is a

vertical 90° magnet with a mass energy product of 200 MeV A/q^2. Two independent quadrupole singlet magnets are installed in front of the 90° magnet to optimise the ion beam optics. The maximal angular acceptance of the arrangement is about ±5mrad in both transversal directions. However, to reduce high order aberrations of the beam optics the acceptance is reduced to ±1mrad in normal operation. Finally, a position sensitive detector (CCD linear image sensor) providing a lateral resolution of 14μm is mounted in the focal plane of the spectrograph. For minimum ionising particles (e.g. 20MeV protons) this detector offers the possibility to count many ions within one readout cycle leading to a counting rate of more than 100kHz. Due to its small dimensions (28.6mm active length) the accepted bin of relative energy shift amounts 0.6% which will be enlarged to the maximum value of 1.5% in the future.
First tests of the arrangement have been done using 20MeV protons and 90MeV sulphur ions. The achieved relative energy resolution of the whole setup was 3.8×10^{-5} FWHM. The sharp beam energy was prepared by cutting down the beam with object and image slits [2] in front and behind the analysing magnet of the tandem accelerator. The derived relative energy dispersion is $2.3 \times 10^{-4} mm^{-1}$. Measurements on thin foils show the principle power of analysing transmitted ions using the 0°-spectrograph.
[1] G. Datzmann, G. Dollinger, G. Hinderer and H.-J. Körner, Nuc. Instr. and Meth. B158, (1999) 74.
[2] O. Schmelmer, G. Dollinger, G. Datzmann, C. Goeden and H.-J. Körner Nuc. Instr. and Meth. B158 (1999) 107.

AT-P9

SCANNING ION MICROBEAM ANALYSIS OF DIFFUSION

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A scanning $^3He^+$ ion microbeam technique has been developed at the University of Surrey Van de Graaff accelerator laboratory. It allows the measurement of important instances of diffusion hitherto inaccessible to other techniques.
To date it has been applied to:
- Water and drug diffusion in drug-release polymers
- Shampoo diffusion into hair
- Water diffusion into fibre optic cables
- Water and chlorine diffusion in cements/concrete.

Present plans are to extend it to the study of:
- Water/cosmetics into skin
- Drugs into mucosae.

Many other applications of this technique, presently in unique use in the world, are envisaged.

AT-P10

NOVEL TEST SAMPLE FOR SUBMICRON ION BEAM ANALYSIS

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A new test sample with nanometer-structures for high resolution ion beam analysis has been developed by the University of Leipzig and the Institute of Surface Modification (IOM). In order to determine the beam spot size, scan size, and scanning properties of a microprobe system in the sub-μm-regime the test sample provides structures, which range down to 250 nm in size. The periodic structures consist of Au, Ti, and Cr on Si produced by electron beam lithography and various deposition techniques. A special semiconductor heterostructure with a thickness of a few micrometer permits the detection of transmitted particles, which allows the determination of the beam spot size in the low current mode used for STIM and IBIC. The test sample was investigated with an SEM and the high-energy ion nanoprobe LIPSION. It is our aim to introduce this novel test sample to microprobe laboratories worldwide.

AT-P11

REALIZATION OF THE SIMULTANEOUS MICRO-PIXE ANALYSIS OF HEAVY AND LIGHT ELEMENTS AT A NUCLEAR MICROPROBE

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A new micro-PIXE experimental set-up has been elaborated for the simultaneous detection of light ($Z \leq 14$) and heavier ($Z > 14$) elements at the scanning proton microprobe facility of ATOMKI. The microprobe made by Oxford Microbeams Ltd. was previously equipped only with a conventional Be-windowed Si(Li) detector (Canberra-type, area = 80 mm^2, thickness = 5 mm) to which a new ultra thin windowed (UTW) Si(Li) detector has been installed. Detectors have been aligned symmetrically at an angle of 135 degrees to the incident beam. This way elements from U down to C can be detected with high sensitivity by one of the two detectors in a single irradiation process. The installation procedure involved a significant development of the original measuring chamber of the microprobe. Special mechanical interfaces were made for the detectors for their precise positioning and to hermetically separate them from the measuring chamber when necessary (e.g. during maintenance). In order to protect the light element detector from scattered protons a special magnetic deflector and collimator unit was made. By the development of filter holder units, different types of x-ray filters (C, Al, etc.) can be easily placed between the sample and the detectors making it possible to use the optimal conditions for x-ray detection. Carbon filter is placed in front of the UTW detector especially when optical fluorescence of a sample disturbs the operation of the UTW detector. A new port had to be created for the optical microscope on the top of the measuring chamber, because, one of the 135 ports previously used for the visual observation of samples had been occupied by the new Si(Li) detector. The capability of this new experimental setup has been tested by different types of standard samples (biological, geological and industrial). Measured detection limits for light elements will be presented as well as experiences gained with the operation of this new experimental setup will be discussed at the conference in detail.

Session IT : Imaging Techniques

IT-P1

STIM TOMOGRAPHY: A POTENTIAL TOOL FOR THE NON-DESTRUCTIVE CHARACTERISATION OF SiC MICRO-COMPOSITE MATERIALS

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Despite the fact that Ion Micro-Tomography techniques were initiated several years ago, useful applications are still rare in Material Sciences. This work is a preliminary study to perform the non-destructive characterisation of a thin BN inter-phase embedded in a metal matrix composite material using STIM (Scanning Transmission Ion Microscopy) tomography. The technique, initially developed at CENBG for applications in the biomedical field, was used here to investigate several fibres produced under different processing conditions.
The data reduction codes, based on analytic reconstruction methods, made it possible to determine the spatial extension of the BN inter-phase in transversal planes at different positions along the thin fibres (25 μm in diameter). This measurement was performed taking advantage of the density contrast between SiC and BN even for a layer thickness down to 3 μm. A first attempt was also made to compare the initial BN inter-phase to the same structure after thermal oxidation.

IT-P2

THE USE OF ELECTRON BACKSCATTERING AS FAST IMAGING TECHNIQUE WITH MOLECULAR BEAMS

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The novel use of molecular beams has been demonstrated by using DH^+ beams for imaging using single-ended accelerators. The use of molecular hydrogen (H_2^+) in imaging presents not only standard PIXE imaging that can be performed, but the electron in the molecule has enough energy to offer a backscattering signal similar to the backscattering technique of the Scanning Electron Microscope. The electron backscattering signal has a much higher yield compared to traditional nuclear techniques, such as PIXE and RBS, and this suggests that these signals can be used as fast imaging tool.
Electron backscattering has been performed using a Centaurus detector, developed especially for low-energy electron backscattering. A H_2^+ beam of energy 5 MeV was used, with each proton having 2.5 MeV, and the accompanying electron having an energy of 1.3 keV. The backscattered electrons were measured simultaneously with the PIXE and RBS signals, as complementary information. Results show that the electron backscattering signals indeed offer elemental information in imaging at least an order of magnitude faste than PIXE. This may result in novel applications in imaging of geological grains, with subsequent trace element analysis.

IT-P3

MATHEMATICAL RECONSTRUCTION OF SAMPLE MICROSTRUCTURES OBTAINED FROM PIXE ELEMENTAL MAPS

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In many cases quantitative characterization of microstructures cannot be achieved from the PIXE signal alone but a mathematical treatment of the distribution maps is required. In this work a numerical algorithm is proposed to reconstruct geometrical sample shape and composition of microstructured materials from the PIXE signal. The precision of reconstruction and space resolution is discussed. Using the novel reference material IRMM-301 "micro-structured RM" and its earlier prototypes, the results of applying the developed numerical technique to experimental distribution maps are compared with the known properties of the RM. It is shown that the method allows an unambiguous reconstruction of the microstructure patterns.

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IT-P4

AN ION-ELECTRON EMISSION MICROSCOPE WITH MICROMETRIC RESOLUTION AT SIRAD, THE IRRADIATION FACILITY AT THE 15MV TANDEM OF THE INFN LEGNARO LABORATORY.

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The SIRAD facility at the 15 MV Tandem of the INFN Legnaro Laboratory is routinely used for radiation damage studies (bulk damage, total dose and Single Event Phenomena) on semiconductor detectors, electronic devices and systems.

Nevertheless in SEP studies SIRAD can only characterize the global sensitivity of the device under test to single ion impacts. To map out the sensitivity of the DUT with micrometric resolution we are building an Ion-Electron Emission Microscope (IEEM) that reconstructs the impact coordinates of an energetic ion by imaging those of he secondary electrons emitted by the DUT, following an idea first developed by B.L. Doyle and collaborators at SANDIA.

In our configuration the electron microscope will be axial with the ion beam. The photoelectrons will be accelerated up to 15 keV and then will impact on a thin (15 μm) scintillator tile which acts as image amplifier. Finally photons will be detected by a multichannel plate and the image read out by a position sensitive detector. Both the multichannel plate and the position sensitive detector will be positioned at 90^0 respect to the beam axis, out of the vacuum chamber. In this way electron microscope and image detector will be mechanically and electrically decoupled, that also allowing for a better flexibility and an easier maintenance.

We will discuss the basic principles and design constraints of the IEEM at SIRAD and the expected performance of such a system.

Session MIBM : Micro-Ion Beam Modification of Materials

MIBM-P1

INVESTIGATION OF LATTICE DAMAGE DURING MICRO-CHANNELING MEASUREMENTS

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The degree of postimplantation damage in various semiconductor crystals depend on the applied dose rate. In particular strong effects have been observed at dose rates up to 10^{19} $cm^{-2}s^{-1}$ which are typical for FIB devices with sub-μm beam diameters.
For a systematic study of damage accumulation at such high dose rates only small implanted areas with dimensions of some ten micrometers are feasible. For RBS/channeling measurements a nuclear microprobe must be employed, and consequently the fluence of the analysing beam of light ions is also orders of degree higher than usually in RBS/C experiments. Therefore the damage generation by the analysing beam must be considered.
We have studied the near-surface damage accumulation at room temperature in Si, 6H- SiC and other materials by H, He and Li ions of various energies both for channeling and random incidence. The nuclear energy loss near the surface ranged from 0.003 to 0.3 $eV/10^{15}$ cm^{-2}. Distinct dose rate effects have been observed. The results are used for a phenomenological model.

MIBM-P2

OBSERVATION OF INDIVIDUAL RADIATION DAMAGE USING AN AUTOMATED SINGLE ION HIT TECHNIQUE AT THE JAERI HEAVY ION MICROBEAM SYSTEM,

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In JAERI Takasaki, we are developing a technique for the purpose of investigating individual interaction between a high-energy ion and material in local area of it using such techniques as single event transient current (SETC) measurement. The pulse height of SETC for Si PIN photodiodes was measured using a single ion hit technique combined with the JAERI heavy ion microbeam system. The reduction of the pulse height was observed according to accumulation of radiation damage introduced by single ion injections in the same micro areas. A series of single ion irradiation experiment was made with changing conditions such as the ion species and the irradiation area in micron scale. Reduction trends of the pulse height obtained in these measurements showed different features from those by large area irradiation. This is due to the dimensions of charge collection in a sample, which is comparable to the spatial resolution of microbeams to irradiate confined area of the sample. In order to extract information about the lateral extent of an individual charge collection and radiation damage, the obtained data were analyzed with a statistical function. Monte Carlo simulation was also performed with applying a simple model to reproduce measured data.
This paper will give details of measurements using the heavy ion microbeam with the single ion hit system, and results of data analyses and simulation.

MIBM-P3

PHOTOLUMINESCENCE AND FIRST-ORDER RAMAN SPECTROSCOPY OF IRRADIATED, IMPLANTED AND ANNEALED GaN/SAPPHIRE

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GaN/sapphire (GaN/S) has been subjected to α-particle irradiation from both GaN and S sides at doses 2.5-5.0x10^{13}cm^{-2}. Another sample has been implanted by N_2^+ions at 1.0x10^{19}cm^{-2} (5keV).

Photoluminescence (PL) excitation using the frequency doubled (514.5/2 nm) Ar^+-ion laser line show good optical properties for the as-grown samples. We find all prominent transitions BX, L_1, L_2, L_3, DAP1, DAP2, together with the dominant yellow band (YB) centered at 2.21eV. Exciting with the 514.4nm line, we resolve the red band (RB) at 1.73eV and a broad weak band at 1.5153eV. From the S side, we detect the 1.513eV band, RB (weakly resolved), YB and, in addition, a band at 2.82eV.

Upon alpha irradiation, all transitions experience a drop in intensity due to the opening of new (non-radiative) channels. These come from irradiation-induced extended defects. Also the YB shifts towards high energy by about 0.055 eV, while DAP2 shifts towards low energy. The PL signal from S side confirms the shift of the YB but no optical transition could be detected after 5x10^{13} cm^{-2} alpha particle irradiation. Upon implantation, the PL (514.5nm) response of GaN does not show any shift of the RB. Its intensity become very low, while the weak band at 1.513eV (as grown) is enhanced and dominates the spectrum.

First order <z(...,...)z> Raman spectra collected from as grown GaN yields all prominent active phonons previously reported. Irradiation does not change the energy peak positions of E_2 (high), A_{1g}(S) and A_1 (LO) modes, but reduces their intensity. Implantation shifts E_2 and A_1 (LO) towards higher energy, while A_{1g}(S) disappears. After alpha particle irradiation at a dose 5x10^{13} cm^{-2} and annealing at 400°C (1h) the S side loses all frequencies. Instead we observe bands at 207, 316 and 538 cm^{-1}. Further annealing at 500°C and 700°C (1h) generates a series of modes at 150, 215, 430 and 606 cm^{-1}. At higher excitation intensity we observe well-resolved (rather luminescence-like) bands at 151, 266, 406 and 600 cm^{-1}.

Forthcoming polarization experiments and some calculations on activation energy of the new transitions will clarify their origins.

MIBM-P4

STRUCTURAL AND ELECTRONIC PROPERTIES OT THE n-GaAs:Si AS-GROWN, ALPHA PARTICLE IRRADIATED AND ANNEALED

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The effect of alpha-particle irradiation on the electronic properties and LO and TO phonons of n-GaAs:Si (10^{19} cm^{-3}) was investigated by means of Raman {z(..,...)z} and photoluminescence (PL) spectroscopy.

In the Raman spectrum of the as-grown sample we observe the TO (266.4 cm^{-1}) and LO (287.2 cm^{-1}) phonons, as well as a weakly resolved band at 154 cm^{-1}, which at certain excitation intensities and polarization can be split into bands at 138.9, 157.2 and 165.8 cm^{-1}. These bands are frequently related to single particle excitations (SPE).

The energy peak positions of the LO and TO phonons do not change upon irradiation with doses between 1.6 - 7.8x10^{13} cm^{-2}. However, the FWHM decreases from 15 to 7 cm^{-1} and the ratio of the LO to TO mode increases essentially. That would be due to a possible formation of donor-acceptor-pairs (DAP), these were carefully worked out in J. Appl. Phys. 81, 6948 (1997). The DAP formation is related to a decrease of the free carrier density of electrons in the conduction band and therefore causes a weakening of the coupling between plasmon and the LO phonon. At higher irradiation doses the SPE band disappears, which might support our explanation.
In the PL spectrum of the as-grown sample we observe the impurity band transition at 1.5176eV. Irradiation with a dose of 2.4x10^{13} cm^{-2} shifts the band up to 1.53eV and decreases its intensity. The FWHM have also increased to twice that of the band in the as-grown sample. Annealing at 400°C (1h) shifts the band to 1.50eV, with an accompanying intensity drop. Further annealing at 500°C (1h) shifts the band to 1.49 eV, Moreover, at the higher energy side we observe two overlapping bands at 2.0eV and 1.9eV (well above the E_g energy gap).
Our quantum mechanical calculations on the LO irradiated phonon strength, together with further annealing and polarization experiments might clarify the origin and behaviour of the new bands.

MIBM-P5

WHITE ELECTROLUMINESCENT NANOSTRUCTURES IN SILICON BY FOCUSED ION IMPLANTATION

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The use of an accelerator fed microbeam as an implanter enables highest flexibility in doping substrates. Dopant, implantation dose and pattern can be chosen individually and specifically adopted to each structure if implanted sequentially. On the other hand, high throughput of individually shaped but identically doped samples, as needed in semiconductor device fabrication, can be achieved in conjunction with the Bochum ion projector. This machine is capable of focusing an ion beam down to below 300 nm and allows both, prototyping and production of small series of devices without the need of a coating mask on the substrate surface.
A method for producing nanoscale point or curve shaped, unpolarised white light emitting areas in doped crystalline silicon using focused ion beams is reported. Focused ion beam implantation yields lateral npn (or pnp) junctions emitting light from the depletion region in (reverse biased) breakdown operating mode, depending on voltage polarity on either side of the implanted area. The actual beam diameter is not a limiting factor for submicron width light sources because the radiation origins only from depletion zones with lateral sizes in the order of 100 nm. Compatibility with standard semiconductor processes for silicon allows for monolithic integration of the nanoscale light sources in conventional circuit designs. The emission spectrum covers the whole visible range without sharp peaks. However, three pronounced maxima whose positions and relative heights vary with implantation dose and applied voltage can be observed. In contrast to light emission from porous silicon these samples are high-voltage and high-temperature stable as well as insensitive to environmental influences.

MIBM-P6

RADIATION STUDIES OF A NOVEL MATERIAL FOR ULTRA HIGH DENSITY NON-VOLATILE MEMORY FOR SPACE

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Properties of binary chalcogenide based materials such as GeTe, AsTe, SbTe have been known and studied for over 30 years and have found applications that exploit their optical properties. By injecting the correct amount of thermal energy into these classes of alloys, the material will undergo a phase transformation from an amorphous to one or more crystalline states. This process is rapid and fully reversible. The thermal energy required to induce vitrification or crystallization can be supplied by, either, injecting optical or electrical energy. A prototype design has already proven to be an excellent approach to manufacture a very high density, non-volatile memory. However, single event effects play a severe role in the long-term survivability of these materials in space. For example, a low energy proton ion with an energy of 1 MeV will have an energy loss of 58 eV/nm in $(GeTe)_2(Sb_2Te_3)_2$. The majority of the energy for a low energy ion will be deposited in a linear track with an effective diameter in the order of only 10 nm. The deposited energy will be approximately 1.2×10^{-19} J/nm^3. The energy density required to reset a single memory cell based on the chalcogenide material will require only an energy density of 6.2×10^{-20} J/nm^3. Clearly, a low energy proton will have enough energy to produce an amorphous columnar track with a diameter of approximately 7 nm that induces a localized resistance change that can lead to loss of stored information. Ongoing studies using an ion beam, focused to about a micron attempt to determine the single ion induced radiation effect as a function of position.

MIBM-P7

SILICIDE SYNTHESIS BY HIGH ENERGY ION PROJECTION

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The increasing complexity of semiconductor devices stimulates the research on new materials. Silicides are an interesting class as they allow to produce either direct band gap semiconductors for optoelectronics or single crystal magnetic or metallic conductors. We are interested in buried silicide structures. On (100) Si as preferred for semiconductor applications they can be produced only by ion beam synthesis.
For the ion beam synthesis of silicides stoichiometric doses have to be implanted. The samples have to be heated during the implantation in order to form high quality silicide layers and to avoid amorphisation of the silicon substrate because of the high dose implantation. Masking of the substrate as necessary for the synthesis of structured silicide layers is highly sophisticated as sputtering of mask material during the high dose implantation might contaminate the sample. Because of these problems only a small fraction of possible silicides have been investigated up to now.
With our high energy ion projector we have an unique machine to overcome the problems of the ion beam synthesis of buried silicide structures. The apparatus combines a technique to implant structures at a lateral resolution of 300 nm without a direct mask on the sample, the possibility to use a wide range of high energetic heavy ions, a procedure to implant high doses

in small structures in short time and the possibility to heat the samples during the implantation.

In this setup we have implanted various ion species into silicon substrates at implantation temperatures of (300 – 450) °C. The samples were subsequently annealed. Ostwald ripening during the annealing forms a silicide layer. With this technique we were able to get Rh_3Si_4 and $MnSi_{1.73}$ structures by ion beam synthesis for the first time.
Properties of the synthesized silicide layers, RBS and spreading resistance measurements will be discussed.

MIBM-P8

NUCLEAR REACTION ANALYSIS OF HELIUM DIFFUSION IN TANTALUM AFTER ION IMPLANTATION AND HIGH-TEMPERATURE ANNEALING

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The behaviour of helium in heavy metals plays a major role in the ageing of divertor materials for controlled fusion reactors exposed to α particle irradiations. Some of the key parameters are the helium diffusion constants and activation energies of the processes involved in helium atom migration at high temperatures. However, such data are difficult to obtain due to the small helium atomic number and atomic mass especially in heavy metal hosts which give a huge background precluding the use of standard X-ray scattering and conventional Rutherford backscattering techniques.

We therefore used the nuclear reaction analysis (NRA) of ^3He based on the $^3He(d,p)^4He$ "resonant" reaction which yields a specific signal regardless of the matrix in order to study the thermally-activated diffusion of ^3He implanted in tantalum. Excitation curves were obtained by measuring the 14-MeV proton yield as a function of the incident deuteron energy for as-implanted and annealed Ta samples. These curves are convolution integrals of the ^3He depth profiles with the reaction cross-section which has a broad "resonance" centred at around 430 keV.
The as-implanted depth profile is assimilated to a Gaussian curve which is fitted to match the experimental excitation curve. The same process is used for the data of samples isochronously annealed at high temperature in a range between 0.12 T_m and 0.54 T_m, where T_m=3269 K stands for the tantalum melting temperature. A decrease of the amplitude and increase of the full width at half maximum (FWHM) of the fitted depth profile is found for increasing annealing temperature. The diffusion constant is then deduced from the amplitude and FWHM at a given annealing temperature. The activation energy for ^3He migration is then derived from an Arrhenius plot of these data. A simpler analysis is also given by plotting directly the excitation curve maximum versus the inverse temperature. No effect of the helium concentration ranging from around 200 to 40000 appm is seen in the present results which are discussed with respect to available diffusion data of helium in b.c.c. metals.

Session ME : Applications in Microelectronics

ME-P1

GATE RUPTURE OF OXIDE GROWN ON 6H-SiC USING ION BEAM INDUCED CHARGE

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Investigation of oxide charge trapping and interface state generation in SiO_2 on 6H-SiC was performed using Ion Beam Induced Charge and the high frequency capacitance-voltage method. These trapped charges were related to the defects either existing in the oxide or generated during irradiation. Large flatband shift in capacitance-voltage measurements indicated that high density of positive charges are trapped near the interface of SiC/SiO_2. The issue of oxide rupture caused by alpha particles on the metal-oxide-p-type SiC device will be addressed. The radiation hardness of SiC electronic devices will be reported.

ME-P2

INVESTIGATION OF THIN PHOSPHOR COATINGS ON PIN-DIODES BY IBICC/IBIL

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A good-quality luminescent thin layer deposited on the top of microelectronic chips is required for the application of Ion Photon Emission Microscopy (IPEM) for microelectronics applications. Coatings of thin layers of phosphors on semiconductor devices have been investigated for the optimization of the luminescence efficiency and the homogeneity of the layers through the combined use of the techniques of Ion Beam Induced Charge Collection (IBICC) and Ion Beam Induced Luminescence (IBIL) in a nuclear microprobe. In search for a high efficiency of luminescence emission and a minimum layer thickness with a high homogeneity, the coatings of the different types of phosphors are produced by various deposition processes. The quantitative results are compared and discussed.

Acknowledgements:
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ME-P3

MICROPROBE RBS ANALYSIS OF LOCALIZED PROCESSED AREAS BY FIB ETCHING AND DEPOSITION

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Impurity incorporation due to the localized beam processing by focused ion beams (FIBs) and electron beams (EBs) such as physical sputtering, gas-assisted etching (GAE) using iodine gas and beam-assisted deposition using [$MeCpPt(Me)_3$] precursor gas has been investigated by a medium energy nuclear microprobe. RBS mapping on incorporated impurities has been performed using 300 keV beams with a beam spot size of 50 nm. RBS mapping images for physically sputtered and GAE areas indicate the localized Ga and I atoms distributions from the Ga FIB and from the etchant gas, respectively. The amount of Ga in the GAE area was estimated to be ~2.7 times smaller than that in the physically sputtered area. RBS mapping images for FIB assisted Pt deposited areas revealed that Pt and Ga atoms distributed in the processed areas, while C atoms resided beyond the processed areas.
The analyzed results have been compared with those by EDS analysis using electron beam. 300keV Be^{2+} microprobe RBS analysis shows that iodine residues exist in the area of FIB assisted etching in iodine gas which cannot be detected by EDS analysis. It is confirmed that microprobe RBS analysis has higher sensitivity than EDS analysis.

ME-P4

INVESTIGATION OF SINGLE-EVENT CHARGE COLLECTION FROM ANGLED ION STRIKES

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Single event upset's (SEU) are a major in concern for DRAM and SRAM devices installed in space based satellites. Recently, large-scale integration of memory devices has made then even more susceptible to multiple-bit upsets (MBU). MBU generally results when an ion strike passes through, or interferes with, multiple memory cells at the same time. However, the fundamental processes behind MBU have not yet been fully clarified. In order to investigate MBU, we examined the relationship between the amount of collected charge and both the spatial position and the angle of the incident ion. The samples used in this experiment were Si pn junction diodes. The samples were irradiated with a variety of heavy ions and energies. Charge collection and transient current measurements were made as a function of the incident beam angle compared to TCAD simulation. Transient current measurements were made with the wide bandwidth oscilloscope system described elsewhere.

ME-P5

FRONTAL IBIC OF CdTe RADIATION DETECTORS AND RESPONSE TO LOW ENERGY GAMMA RAYS

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Within a continuous international effort in developing the non-cryogenic semiconductor detectors for gamma ray spectroscopy, various wide gap materials were considered. With a best performance achieved, CdTe and CdZnTe based detectors became today widely accepted and commercially available. In addition to possible future use of such detectors for PIGE, nuclear microprobes are in recent years applied more as their characterisation tool using IBIC technique.

Several CdTe detectors of 2x2x1 mm^3 size were used in this study. On the basis of frontal IBIC measurements of charge collection efficiency distribution, a spectral response to low energy gamma rays was simulated. These simulated spectra were compared with experimental peak shape measurements. Different modes of frontal IBIC (ions, energy) were used in order to find depth dependence of charge collection efficiency distribution. Further degradation of charge collection efficiency and the downward trend in peak position were studied by on-line irradiation of CdTe samples with 3 MeV protons up to 10^{10} p/cm^2 radiation dose.

ME-P6

SINGLE ION INDUCED TRANSIENT MEASUREMENTS AND 3D SIMULATION OF PARTIALLY DEPLETED SOI

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Electronic components built on Silicon on Insulator (SOI) have been part of the mainstream technology for space electronics for many years. The charge collection mechanisms for bulk or epitaxial CMOS components have been well researched in the past and are at this point well understood. The understanding of the charge collection mechanisms in fully or partly depleted SOI is still sketchy.

Our 3D, time dependent simulation results of single ion strikes through partially depleted SOI structure exhibit a suppressed charge collection volume for diffusive charge collection and a complete suppression of funneling assisted charge collection. The simulation results are substantiated through Time Resolved IBICC [1] measurements using 25 MeV carbon and copper ions at a position resolution of less than 1 micron. Furthermore, the results show a clear sensitivity of the charge collection on the ion strike location within a single FET and angle of ion incidence. It is also shown, that the parasitic bipolar effect induced by an ion strike can be significantly suppressed in partially depleted SOI by using properly designed body ties.

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ME-P7

METHOD BASED ON THE EXTENDED RAMO THEOREM TO INTERPRET CHARGE COLLECTION EFFICIENCY PROFILES AS DETERMINED BY LATERAL IBICC

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An efficient method for calculating charge collection profiles in semiconductor devices as determined by lateral IBICC measurements is presented. The method is based on the extended Shockley-Ramo theorem that provides a rigorous mathematical tool for the calculation of the induced charge under the assumption of a quasi-steady state operation of the semiconductor device. The time dependent charge collection efficiency as a function of the ion impact distance from the electrodes is accomplished by calculating the current induced at the electrodes by the motion of the injected charges as evaluated using the weighting potential method.
The method turns out to be suitable to interpret lateral IBICC measurements (where funnelling effects are considered negligible) carried out on fully or partially depleted semiconductors, whichever is the contact quality of the electrodes (ohmic, injecting, blocking).
The method is applied to analyse charge collection efficiency profiles measured on virgin and damaged Si diodes with different doping profiles.

ME-P8

MICRO-IBICC AND MICRO-IL ANALYSIS OF CVD DIAMOND MICRODOSIMETERS

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Diamond is an ideal material to fabricate dosimeters because of its tissue equivalence, chemical stability, non-toxicity, mechanical and radiation hardness. Chemical vapour deposition technique (CVD) technique allows the fabrication of small diamond detectors, which can be considered for in vivo dose measurements. We have fabricated microdosimeters by depositing thin diamond films (<20 μm thick) on tungsten wires (Ø = 200 μm). Such devices work as solid state ionisation chamber where the electrodes are the W substrate and a thin gold layer evaporated on the diamond surface. The ion beam induced charge collection (IBICC) and ionoluminescence (IL) techniques turn out to be very suitable to characterise such small and irregularly shaped microdosimeters. The uniformity of the electronic quality of diamond has been evaluated by mapping the charge collection efficiency as obtained by IBICC measurements, whereas IL was used to map radiative recombination centres as well as to study radiation damage effects in combination with the microRaman technique.

Session MS : Applications in Materials Science

MS-P1

STUDY OF SOLID STATE REACTIONS OF SiC/SiC$_f$ COMPOSITES USING MICROBEAMS

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Among the advanced materials with potential interest for applications in fusion technology the SiC/SiC$_f$ composites are considered to be one of the most promising. We used microbeam RBS and PIXE techniques to follow the structural changes of several SiC/SiC$_f$ samples in contact with lithium orthosilicate and lithium titanate breeders at 800°C for different time intervals. After 216 h of exposure the strongest reaction taking place was the oxidation of the composites. The oxidation rate was faster in the samples exposed to the orthosilicate. Simultaneously, lithium diffuses into all the composites and there is a noticeable loss of carbon. For longer exposure the thickness of the oxide layers extends to values above the depth of ion beam analysis. The surface oxidation was attained trough the formation of SiO_2 and Li_2SiO_3, which was revealed by XRD analysis. The formation of Ti rich precipitates was also observed in the samples in contact with the Li titanate.

MS-P2

OXYGEN DIFFUSION STUDIES IN OXIDE SCALES THERMALLY GROWN OR DEPOSITED ON MECHANICALLY LOADED METALLIC SURFACES

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The high temperature growth of oxides on metallic surfaces mechanically in loading is not well understood yet. The knowledge of the growth of oxides on static surfaces and of the mechanical behavior of the metal/oxide system do not give account of the synergetic effects between the load and the growth of the oxide.

The formation of cracks on the oxide scales, their healing and the role of the load on the oxygen diffusion processes has been studied on pure nickel and zirconium samples in creep. The use of oxygen-18 to study the oxygen diffusion and the determination of local oxygen-18 diffusion profiles with use of nuclear reaction $^{18}O(p,\alpha)^{15}N$ shows a sharp influence of the load. The application of the load induces an increase of the oxygen diffusion coefficients until two orders of magnitude (typically from around 10^{-15} $cm^2.s^{-1}$ to around 10^{-13} $cm^2.s^{-1}$ in NiO thermally grown on nickel monocrystals). However, this enhancement decreases with the increase of the load.

As the oxide scales are multilayered and as spalling and regrowth may occur, RBS mapping of the local thickness of the oxide stripes is also performed. This technique helps in

understanding the formation of the microstructure and of the damaging process during the mechanical loading.

MS-P3

ION BEAM ANALYSIS OF THE OXIDATION AND HYDROGENATION OF SWITCHABLE MIRRORS

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The hydrogenation of Y, La or rare-earth metals induces a remarkable metal-insulator transition in these metals, e.g. YH_2 being metallic reflecting and YH_3 insulating and transparent. A typical sample geometry contains an active layer on a transparent substrate (SiO_2 or Al_2O_3). A capping layer of Pd is used to prevent oxidation of the active layer and to enable the dissociation of molecular H_2 in atomic H. In this paper the hydrogenation and oxidation of switchable mirrors is investigated using ion beam analysis techniques.
The nuclear reaction $^{15}N(^1H,\alpha\gamma)^{12}C$ enables the determination of H concentration depth profiles (depth resolution ~ 5 nm). The lateral resolution in this kind of experiment is around 3 mm^2. Combination with Elastic Recoil Detection of lateral H concentration profiles using a microbeam yields a good insight in the 3D H distribution in the samples.
The 3.045 MeV resonance in the $^{16}O(\alpha,\alpha)^{16}O$ scattering reaction is used to probe the amount of oxidation of the active layer. Typical sensitivities in the order of $5{\cdot}10^{15}$ at/cm^2 and depth sensitivities in the order of 30 nm are found from an analysis of the resonance yield as a function of energy.
These techniques have been applied to the analysis of $Al_2O_3/LaH_x/Al_2O_3/Pd$ and $Al_2O_3/YH_x/Pd$ multilayered samples.

MS-P4

MATERIAL CHARACTERIZATION USING THE MICRO-PIXE TECHNIQUE – A CASE STUDY

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One of the main features of the PIXE technique is its deep particle penetration into the sample matrix. If combined with a nuclear microprobe for lateral distribution analysis, the so-called micro-PIXE technique can characterize layered structures in all three dimensions. This technique may be useful for the characterization of wear, coatings and thin films, and buried layers in microelectronics applications.
In this work lateral micro-PIXE distributions are generated from the rake face of an uncoated WC-Co cutting tool insert, which was used for machining aluminum. These data are utilized to numerically produce a layer thickness profile from the rake face including a narrow, Si, Mn and Fe rich deposit in the vicinity of the cutting edge and located between the tool base and the adhered workpiece material. The method to numerically produce a layer thickness profile is verified by electron beam induced X-ray mapping of the cross-sectional area of the same cutting tool, which also allows to evaluate the limitations of the method. Moreover, the combination of the layer thickness profile with the X-ray maps from the corresponding cross-sectional area offers a tool to obtain additional information such as layer densities.

MS-P5

MICROBEAM CHANNELING INVESTIGATION OF Rb DOPED KNbO3 AND Zn DOPED LiNbO3 SINGLE CRYSTALS

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KNbO3 and LiNbO3 crystals due to their high non-linear optical coefficients are promising materials for frequency conversion. Their high electroptic coefficients,their transparency and the possibility to grow good quality crystals of these Niobates also make them attractive for optoelectronic and wave guide applications. the non linear and photorefractive properties of these crystals are strongly influenced by the doping of impurities. It is therefore important to understand the lattice site of the dopant like Rb in LiNbO3 and Zn in LiNbO3 to predict the location of energy levels in such doped crystals.

This paper is primarily concerned with the structural investigation of Rb doped KNbO3 and Zn doped LiNbO3 single crystals. Micro RBS/-C and Channeling Contrast Microscopy (CCM) was employed to analyze the as grown crystal quality. 7Li(p,α)4He nuclear reaction channeling analysis was used of the Li atoms in LiNbO3 crystals. Micro PIXE was used for the lattice location of the doped impurities in the crystal. A channeling simulation program was employed for the determination of the crystal quality by comparison of the experimental channeling spectra with the simulated. Results indicate deterioration of crystal quality with higher doping concentration of Rb in KNbO3. In the case of LiNbO3, Nb and Zn dips show perfect substitution of Zn with Nb rows which constitute the <0001> channel. However, Zn dips are found to be narrower than Nb dips indicating that the Zn atoms probably occupy Li sites.

MS-P6

ION MICROPROBE STUDY OF THE SCALE FORMED DURING HIGH TEMPERATURE OXIDATION OF HIGH SILICON EN 1.4301 STAINLESS STEEL

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A study of the oxide layer formed on the surface of high silicon (0.8 %) EN 1.4301 (AISI-304) stainless steel after 125 hours oxidation in air at 1273 K has been performed by means of scanning electron microscopy (SEM), X-ray diffraction (XRD), RBS and proton microprobe. Oxidation experiments in synthetic air were performed in a thermobalance and the kinetic curve is compared to that of a standard EN 1.4301 austenitic stainless steel. These results show that the high silicon steel presents an enhanced oxidation resistance. XRD experiments show that the only crystalline species present in the scale is Cr_2O_3. Nevertheless, transversal section studies of the scale using proton microprobe show the development of a multilayered scale formed by a vitreous silicon rich layer in the scale to alloy interface and a Cr_2O_3 oxide layer in the external scale. Those results are confirmed by SEM experiments. The formation of the silica layer can be the responsible of the increase in the resistance to high temperature oxidation in this steel.

MS-P7

MICRO-RBS STUDY OF NICKEL SILICIDE FORMATION

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A 2 MeV He^+ microbeam is used to obtain information on silicide formation in patterned nickel silicide samples under different annealing conditions. It is important to characterize silicide formation processes in such laterally non-homogenous samples in order to investigate the resistivity variation that is observed when the MOSFET (metal oxide silicon field effect transistor) gate length is reduced, and also when the silicidation temperature is changed. Nickel monosilicide (NiSi) has been shown to have the following advantages: low contact resistivity, low resistivity (~15 μΩ.cm), low Si consumption, low formation temperature (~400-600°C). In addition, it exhibits little or no resistivity degradation on narrow lines (no linewidth dependence), and it can be produced in a single annealing step.
The patterned samples investigated consist of an array of square shaped bond pads (50 x 50 μm^2) of the structure $NiSi_x$ / Poly Si / SiO_2 (2000 Å) / Si and narrow lines of 100 μm length and linewidths ranging from 2 to 0.35 μm. Pt (~4at%) was added for some samples to delay the formation of $NiSi_2$, which is undesirable because of its higher resistivity. Microbeam RBS (~5μm^2 beam spot) was used to obtain the thickness and stoichiometry of the silicide films for the bond pads. This beam size was chosen because a beam current of typically 500 pA is then available. The beam was focused to submicron sizes for the scans of the narrow lines. Micro-RBS results for the different silicide structures are presented and correlated with micro-Raman data.

MS-P8

MICROBEAM STUDIES OF GEL-POLYMER INTERFACES WITH Li ANODE AND SPINEL CATHODE FOR Li ION BATTERY APPLICATIONS USING PIGE AND PIXE SPECTROSCOPY

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Characteristics of Li-ion batteries are determined by the interfacial resistance at interfaces of gel-polymer electrolyte with a Li-anode and with a spinel-cathode, respectively. Interfacial resistance depends on the spatial distribution of Li and other chemical species in the vicinity of interfaces of gel-polymer with cathode or anode. Studies of gel-polymer interfaces in samples for Li-ion battery applications have been performed by proton microbeam scanning of the sample cross section. PIGE spectroscopy was used for Li and F detection and PIXE spectroscopy for the detection of S and other heavier elements of interest. Distribution of Li and other elements of interest have been studied at gel-polymer interfaces in a symmetric cell (Li/gel-polymer/Li) and semicell (spinel ($LiMn_2O_4$)-cathode/gel-polymer) samples containing Liimide Li-salt solution in a gel-polymer electrolyte.

MS-P9

THE CHEMICAL DURABILITY OF ZIRCON CONSIDERED AS A POTENTIAL HOST MATRIX FOR THE IMMOBILIZATION OF ACTINIDES

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Zircon ($ZrSiO_4$) is one of the oldest mineral present on earth : samples of more than 4 x 10^9 years have been found and characterized [1]. Its strong structural affinity for uranium and thorium together with its very high chemical durability [2] make it a potential crystalline host matrix to immobilize actinides issued from separation of nuclear wastes. Ewing and coworkers were the first to propose the use of zircon for the disposal of plutonium from nuclear weapons [3]. They have performed a lot of studies concerning as well aqueous alteration as irradiation behaviour of natural and synthetic zircons [4]. Irradiation induces amorphization of the crystalline structure (the metamictization process) and thus decreases the chemical durability of the material.

Leaching tests have been conducted on natural zircons from Brazil at 96°C for a period of 6 months, using deionized water buffered at pH = 9.5 with sodium hydroxyde and doped with EDTA, a strong complexing agent of zirconium. Leachates have been analyzed by ICP-MS and spectrophotometry. Zircon solid surfaces have been investigated by coupling Scanning Electron Microscopy and X-ray Microanalysis with Nuclear Microprobe Analysis (μPIXE and μRBS).

This paper is focused on the first experimental results obtained in this study. From the mass balance between leachates and hydrated surfaces, the probable mechanisms of zircon aqueous alteration are presented and discussed. The dissolution rates of silicium and zirconium are higher than in deionized water due to the basic pH of EDTA and the stabilization of Zr in solution by strong complexing effects.

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MS-P10

BEHAVIOUR OF THE INTERFACE LANTHANIDE-ALUMINOSILICATE GLASS/WATER

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Lanthanide-aluminosilicate glasses have been developed for their high mechanical properties. The substitution of a few atomic percent of oxygen by nitrogen, introduced in the glass as AlN, strengthens the glass structure [1]. Nevertheless, little is known about the chemical durability, the role of rare earth-elements and the eventual influence of nitrogen on the dissolution mechanisms of such glasses [2]. This type of glass appears to be a suitable complement waste forms of borosilicate glass for the specific immobilization of trivalent actinides [3].

The aim of this work is to improve our knowledge on the relationships between the chemical durability and the irradiation resistance of aluminosilicate glasses. The first part deals with the synthesis of glasses in the system SiO2 – Al2O3 (AlN) – Y2O3 – La2O3 and the study of their leachability using deionized water in a reflux column at 100°C. Hydrated glass surfaces have been examined with scanning electron microscopy coupled with X-ray microanalysis, infra-red microspectrometry, X-ray photoelectron spectroscopy and nuclear microanalysis. Leachates have been characterized by using ICP-MS, ion chromatography and spectrophotometry. In a second part, the same " leaching tests plus characterization " approach is applied to glass samples previously submitted to irradiation tests using 5.5 MeV α-particles or 100 – 200 keV heavy ions (mass around 200).

The formation of a surface layer enriched in Y and La and depleted in Si and Al is shown. The further dissolution of the glass seems to be controlled by the solubility of (oxo)hydroxy or complexed species such as carbonates or phosphates, depending of the aqueous solution composition.

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MS-P11

MICROPROBE DEPTH PROFILING OF LONG RANGE DIFFUSION OF Cr IN Ti

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Service range and lifetimes of titanium based alloys can be significantly extended if corrosion and wear resistance can be improved without loss of the high elastic limit-to-density ratio. We used ion implantation to promote the formation of Ti-Cr intermetallic particles in the surface region of Ti, and conventional and microbeam techniques to follow the diffusion behaviour of Cr. 140 keV Cr^+ ions were implanted into Ti with doses in the range 0.8 – $4x10^{17}/cm^2$ at different temperatures. Samples implanted at lower temperatures were further annealed at 600 and 1000 °C, under pressures of 10^{-6} mbar, for periods of 1 to 6 hours.

Implantations with the highest doses, $4x10^{17}/cm^2$, at temperatures in excess of 900 °C lead to a loss of Cr, as seen by RBS. The same effect was observed upon annealing at similar temperatures. In these situations standard MeV RBS spectrommetry is completely unable to yield a depth profile and thus to answer the question whether Cr was actually lost from Ti or diffused inwards at very fast pace. In the present work we used a proton microbeam to obtain step wise cross-sectional concentration profiles of Cr. The technique successfully allowed to obtain profiles over depths more than 2 orders of magnitude larger than those probed by RBS, and to show that, rather than being lost through the surface, chromium diffused inwards at a very high rate. Diffusion coefficients for Cr diffusion in Ti at these temperatures are derived from data and presented.

Session BM : Applications in Biology and Medicine

BM-P1

MICROPIXE AND RAMAN SPECTROSCOPY IN ATHEROSCLEROTIC PLAQUE CHARACTERISATION

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Raman spectroscopy can be used for in vivo characterisation of atherosclerotic plaque composition in human coronary arteries. This technique is particularly useful in predicting plaque rupture and selecting proper therapeutic intervention. Raman spectroscopy is applied in situ on animal models to optimize this technique and study atherogenesis. For this purpose APOE*3 Leiden transgenic mice — which over express the dysfunctional human apolipoprotein E variant — are fed a high cholesterol diet and are sacrificed after 5 or 6 months. These mice develop a diet induced hyperlipidemia and atherosclerotic plaques, similar to those in humans. Raman spectroscopy is used to create maps of the chemical composition of the atherosclerotic plaques at various disease stages.
However, Raman spectroscopy is a non-quantitative technique that only yields the relative amount of the arterial components. For quantitative measurements an additional technique must be utilised. Due to its high resolution and low detection limit, scanning μPIXE is capable of producing quantitative elemental maps with a resolution similar to that of Raman spectroscopy. These elemental maps are produced using Dynamic Analysis software designed for atherosclerotic tissue. For quantification and verification purposes the same segments of atherosclerotic mice aortas were measured using both Raman spectroscopy and PIXE. Whereas Raman spectroscopy measures molecular components, PIXE measures elemental distributions. Therefore, PIXE can be used to study the homogeneity of the arterial components — for example the Ca/P ratio in calcifications — verifying whether refinements in the model will result in a significant improvement in clinical applications. Additionally, the low detection limit of PIXE allows this technique to measure the amount of trace elements such as Fe, Cu and Zn. These results may contribute in studying the atherogenesis.

BM-P2

NMP APPLICATION TO EVIDENCE TEETH INHOMOGENEITY

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The present work is part of a study to investigate whether tooth elemental contents reflect environmental influence, dietary habits and health status.

The distribution of major and trace elements along human teeth can reflect metabolic alterations associated with chronic pathologies, such as diabetes, hypertension and kidney insufficiency.
However to assess the elemental distribution variations in disease conditions it will be relevant to know the homogeneity of the teeth mineralised tissue at different regions.
Nuclear microprobe technique was used to analyse teeth from subjects with diabetes, hypertension and kidney insufficiency. The used NMP is installed at ITN Van de Graaff accelerator and provides a resolution of 1 μm.
An X-ray fluorescence set-up with microprobe capabilities, installed at the LURE synchrotron (France) was also used for elemental determination in teeth.
The resolution of the synchrotron microprobe was 100 μm and the energy of the incident photons was 18 keV.
The NMP concentration profiles for P, Ca, Fe, Cu, Zn, Sr and Pb in the different regions are discussed and correlated with the results obtained with the synchrotron microprobe.
In addition, the teeth in-homogeneity obtained at 1 μm and 100 μm level, for the elemental distributions both for disease conditions and healthy subjects, will be examined.

BM-P3

PIXE INVESTIGATION OF TRACE ELEMENTS IN SOFT TISSUES AND NEAR INTERFACES AROUND DIFFERENT IMPLANTED BIOMATERIALS.

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When prosthesis or generally biomaterials are implanted in a body, they are submitted to physiological fluids reactions. If metallic ions or fragments are emitted by a prosthesis, they can induce toxicity in surrounding tissues. As small quantities are involved, PIXE method allows to detect and evaluate the nature and quantity of these elements. We have made these measurements on human soft tissues around metallic prosthesis. Concentrations in metallic elements in inflammatory tissues are significantly higher than in reference healthy tissue. Some results are indicated for Fe and Cr in the table.
without prosthesis; Fe = 176 ± 9 ppm and Cr < 3 ppm
with prosthesis; Fe = 812± 26 ppm and Cr = 200±8 ppm
In other grounds, we have studied metallic compound Ti6Al4V coated with bioglasses of different bioactivities, implanted in cortical sites of ovine thighbones. Cartographies of major and trace elements are made near interfaces to evaluate the osteoconductivity and the properties of bioglasses to protect the implants against corrosion.
To illustrate the possibilities, we show here a great correlation between Ca and Sr distributions in a neoformed bone around the site of implantation.

BM-P4

COMPARISON OF STIM AND PARTICLE BACKSCATTERING SPECTROMETRY MASS DETERMINATION FOR QUANTITATIVE MICROANALYSIS OF CULTURED CELLS

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In biological samples microanalysis, a mass-normalization method is commonly used as a quantitative index of elemental concentrations determined by PIXE (Particle Induced X-Ray Emission). The organic mass can either be determined using particle backscattering spectrometry, or STIM (Scanning Transmission Ion Microscopy). However, the accuracy of quantitative microanalysis in samples such as cultured cells is affected by beam-induced loss of organic mass during analysis. The aim of this paper is to compare mass measurements determined by particle backscattering spectrometry or by STIM and to show how PIXE results can be corrected from these results.

Non-damaging STIM analysis of samples before PIXE irradiation is certainly one of the most accurate ways to determine the sample mass, however this requires strong experimental handling. On the other hand, particle backscattering spectrometry, performed simultaneously with PIXE, appears to be the simplest method to determine the local mass in cultured cells.
In order to compare STIM and particle backscattering spectrometry analysis, we measured by both techniques the thickness of standard polymer foils of polycarbonate (3 and 6 μm), Mylar® (4 μm), Kapton® (7.5 μm) and Nylon® (15 μm), as well as the organic mass loss induced during PIXE microanalysis of cultured cells.

BM-P5

TRACE METALS AND CANCER: THE CASE OF NEUROBLASTOMA

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More than 50% of patients with neuroblastoma, a young children solid tumor, present a metastatic dissemination at the moment of diagnosis, with a survival rate inferior to 15%. N-*myc* oncogene amplification is one of the most established prognostic factors in neuroblastoma, associated with advanced stages of the disease. Many works showed that amounts of ferritin, an iron storage protein, are abnormally increased in serum of patients with advanced stages disease. Moreover, N-*myc* amplified neuroblastoma cells can synthesize zinc metalloenzymes allowing tumoral invasion and metastases formation.

The aim of this study was to find a relationship between N-*myc* amplification and trace metals in human neuroblastoma cells. Coupling PIXE (Particle Induced X-ray Emission) and RBS (Rutherford Backscattering Spectroscopy) techniques, Nuclear Microprobe allowed to analyze elemental distributions and to determine trace metal concentrations within neuroblasts. Firstly, Fe, Cu and Zn distributions have been observed and their contents have been measured in 3 human neuroblastoma cell lines characterized by an increasing degree of N-*myc* amplification (1, 25 and 60 copies). Secondly, the relationship between trace metals and tumor formation in nude mice, after injection of cells from the same lines, has been studied.
Our data allowed to establish a relation between trace metal contents and mechanisms of neuroblastoma oncogenesis, both amplified cell lines representing more aggressive phenotypes of the disease. They should be confirmed with analysis of cultured neuroblasts and tumors issued from a cell line transfected with the N-*myc* oncogene.

BM-P6

BIO-NORMALYZER® : AN ANTI-CANCER DRUG FOR NEUROBLASTOMA ?

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Iron is an essential micronutrient that is required for cell division and growth. Incorporation of iron into cells is achieved by endocytosis of its transport protein : transferrin. Then, iron may be stored in ferritin and hemosiderin, its two main storage proteins. Increased intracellular iron concentrations may promote malignant cell growth and high levels of serum ferritin are frequently observed in patients suffering from cancer. Patients with advanced-stage neuroblastoma (NB), a young children solid tumor, show abnormally high levels of serum ferritin, very likely synthesized and secreted by NB tumor *in vivo* and consistent with a frequent accumulation of iron in ferritin in NB tumor tissues. In a previous study, we showed that there is no iron accumulation in ferritin of NB cultured cells, and intracellular iron concentrations, determined by Nuclear Microprobe Analysis on these cells, proved to be especially low compared to normal values.
Bio-Normalyzer® (BioN) is a natural antioxidant containing papain, a proteolytic enzyme which has proved to be able to digest transferrin on cell surface. Thus, papain limits iron-transferrin endocytosis and prevents iron incorporation into cells. In the present study, we firstly tested cell viability after treatment with BioN, with papain and with dextrose (the main constituent of BioN) at concentrations ranging from 0.1 to 100 mg/ml and found that BioN, probably thanks to papain, presents an anti-proliferative effect on NB cultured cells. Coupling PIXE and RBS techniques, we secondly measured iron concentrations in NB cells treated or not with BioN, with nuclear microprobe. The preliminary results tend to prove that BioN could present interesting effects on trace metal concentrations in NB cultured cells. Such a compound may be useful in treatment of this pathology.

BM-P7

A COMBINATION OF NUCLEAR MICROSCOPY AND INDUCTIVELY-COUPLED PLASMA MASS SPECTROMETRY (ICP-MS) AIDS IN DIAGNOSING CONSEQUENCES OF METAL EXPOSURE

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Twenty-seven consecutive patients with health problems associated with dental amalgam were recruited to the present study. A healthy age and sex matched control group with dental amalgams but without symptoms was also recruited. In addition to monitoring of metals through blood plasma measurements by ICP-MS, nuclear microscopy of isolated individual blood cells was undertaken. Although there were significant differences in many of the trace elements in plasma between the two groups, no single element seemed to be of decisive value. In contrast, nuclear microscopy provided a sharper means of differentiating between the groups. However, nuclear microscopy is not readily available to clinicians. Therefore, an attempt to make use of the plasma trace element data was tried. By using multivariate statistics it was shown that plasma data could be used to discriminate individuals between the groups with only three being misclassified.

BM-P8

MEASURING TWO-DIMENSIONAL MAPS OF CALCIUM AND STRONTIUM OF SECTIONS OF FISH OTOLITHS WITH A HEAVY ION MICROPROBE AND SIMS

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The fish otolith is a calcium carbonate (usually aragonite) crystal that grows continuously by accretion over the life of the fish and unlike bone is not continuously re-metabolised. Consequently, the otolith has long been regarded as a potential store of information about the life history of an individual fish, and its information is encoded in the deposition pattern of trace elements in the otolith. The code has been difficult to crack. However, recent developments have shown that: 1) Sr is one of the few non-mobile trace elements in the otolith; and, 2) the pattern of Sr deposition summarises the effects of environment changes that effect the growth rate of the otolith crystal. The remaining difficulties in cracking the chemical code in the otolith hinged about making reliable micro-measurements of the stable Sr content at spatial resolution of 10 micrometer or less. This interval represents about 4 – 6 days of otolith growth in most species of fish. Sections of otoliths from the Jurel, or Peruvian Jack mackerel, *Trachurus murphyi* (Carangidae: Teleostei) are measured with the new heavy-ion microprobe at ANSTO to investigate the possibility of analysing otoliths with heavy ions for the first time. The results are compared with 'conventional' microprobe measurement using 2.5 MeV protons and high-resolution SIMS. The aim of the contribution is to achieve reliable two dimensional maps of Sr and Ca and to investigate the beam damage caused by the heavy ion beam.

BM-P9

FLUORIDE UPTAKE MEASUREMENT USING MICRO BEAM PIGE

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Investigating the optimum fluoride concentration for the inhibition for caries, it may be one of key steps to know the fluorine distribution in such a micro domain as acid-resistant layer. The methods as PIGE, EPMA and SIMS can be used. Here the micro-beam PIGE was carried out to obtain high resolution.
Class V cavity in human tooth was filled with light-cured composite resin (Teethmate F-1, Kuraray), with and without etching (K-etchant, Kuraray, time for 40 seconds), then immersed in the normal saline solution at 37°C. After one month, the tooth was bisected longitudinally through the center of the cavity surface perpendicular to the axial wall.
The 1.7 MeV proton beam accelerated by the TIARA single-ended accelerator at JAERI Takasaki was delivered to a micro-beam apparatus. The beam spot size was about 1μm with a beam current of about 100pA.
A nuclear reaction $^{19}F(p,\alpha\gamma)^{16}O$ is used for measuring fluoride density. The gamma rays of this reaction were detected with a 5" NaI(Tl) detector. Proton induced X-rays are detected with a Si(Li) detector for measuring calcium density. Photon yield from reference materials

$Ca_{10}(PO_4)_6(OH)_{2-2x}F_{2x}$ (x = 0, 0.1,0.5), which has similar chemical compounds as a tooth has, were measured to calibrate the absolute density. Beam intensity was monitored with X-ray yield from the copper foil, which is scanned in every 30 seconds.
In the etching teeth, the fluorine distribution shows steep increase and reached maximum at the cavity wall surface. The maximum fluoride uptake textured a band of 6μm width, and gradually decreased inward. The calcium distribution also showed rapid increase near at the cavity wall surface and kept constant after reaching the maximum density. The calcium reached at maximum at the location that was about 5μm inside from the fluorine maximum position. Whereas in the non-etching teeth, the maximum concentrations of both fluorine and calcium were observed at the cavity wall surface.
The small beam spot makes possible to resolve fine structures in fluoride distribution. Observed fluoride concentration in the narrow area can be seen only with sub-micron beam lines. The sub-micron beam line at TIARA might bring a new tool in dentistry

BM-P10

IN AIR SCANNING TRANSMISSION ION MICROSCOPY OF CULTURED CANCER CELLS

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Scanning transmission ion microscopy (STIM) enables either transmission imaging with high spatial resolution and measurement of energy loss which provides information about local sample thickness. When applied to tissue cultured cells, STIM can be used for sub-cellular imaging and organites identification, specimen damage monitoring, and sample mass determination. So far it has been only applied to the analysis of freeze-dried cultured cells under vacuum. With the development of external micro-beams STIM can now be performed in air and sample drying processes can be avoided. In this paper we report the STIM analysis of living cultured cells using 2.5 MeV protons with the nuclear microprobe of Bordeaux-Gradignan.
A thin, 100 nm, silicon nitride window was used to extract the micro-beam in air. Human ovarian cancer cells were cultured as 70% confluent monolayers onto 150 nm thick Formvar films. Samples were removed from the culture medium and placed immediately behind the silicon nitride window. The distance between the window and the sample was approximately 2 mm. The same distance separated the sample from the particle detector. With this experimental setup, STIM could be performed on hydrated cultured cancer cells, and transmission images were obtained with a spatial resolution in the micrometer range. Examples of cell imaging will be presented as well as corresponding energy loss spectra of wet and dried samples. Potential applications of in air STIM in cell biology will be discussed.

BM-P11

IRON DISTRIBUTION IN CANCER CELLS FOLLOWING DOXORUBICIN EXPOSURE USING PROTON AND X-RAY SYNCHROTRON RADIATION MICROPROBES

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Doxorubicin is a well established and highly efficacious anticancer agent. Despite the extensive and long-standing clinical use of this drug, its mechanism of action is still uncertain. Chemical studies have shown that doxorubicin is a powerful iron chelator and the resultant iron-drug complex is an efficient catalyst of the conversion of hydrogen peroxide to the highly reactive hydroxyl radical. However the intracellular complexation of anthracyclines with endogenous iron is still debated.
Using scanning proton X-ray microanalysis, we observed in human ovarian carcinoma cells exposed to 20 μM iodo-doxorubicin (IDX) that iodine and iron cellular distributions were spatially correlated, suggesting a mechanism of intracellular iron chelation by the anthracycline. Furthermore, as iron followed the preferential nuclear localization of the DNA-intercalating agent IDX, it also suggested that drug-metal complexes could play a critical role in the cellular pharmacology of doxorubicin compounds. Thanks to the high sensitivity of X-ray micro-fluorescence induced by synchrotron radiation (μ-SXRF) at the European Synchrotron Radiation Facility, intracellular iodine and iron mapping at decreased drug concentrations down to pharmacological doses (2 μM) were performed. In addition, preliminary μ-XANES (X-ray Absorption Near Edge Spectroscopy) experiments for iron speciation in cultured cells will be presented.

BM-P12

CHROMIUM MAPPING IN MALE MICE REPRODUCTIVE GLANDS EXPOSED TO $CrCl_3$ USING PROTON AND X-RAY SYNCHROTRON RADIATION MICROBEAMS

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Preconception exposure of male rodents to certain carcinogens may increase risk of tumors in offspring, and a similar phenomenon is suspected in humans, especially with regard to occupational metals. In a recent study, a significant increase of tumors in offspring was observed after exposure of male Swiss mice to Cr(III), a constituent of welding fumes. The mechanism of chromium trans-generation carcinogenicity remains unknown. There is a need for the determination of tissue and cellular distributions of chromium in mice reproductive glands to better understand the chemico-biological interactions involved in this process.
Using scanning proton probe X-ray microanalysis we have been able to detect chromium in testes sections from mice 24 h after intraperitoneal administration of 1 mmol/kg $CrCl_3$. Chromium concentration was about 5 μg/g dry mass in average, round distributions of iron, and presumably chromium, of 30 μm diameter were observed in the tunica albuginea. In addition, μ-SXRF (Synchrotron radiation X-ray Fluorescence) measurements clearly demonstrated the presence of chromium in the tunica albuginea and within isolated cells from the interstitial connective tissue.

BM-P13

THE ROLE OF ZINC IN THE DARK ADAPTED RETINA STUDIED DIRECTLY USING MICROPIXE

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Zinc deficiency is known to cause night blindness, which can be reverted by the administration of zinc. The exact function of zinc is not understood but zinc seem to play a unique role in the phototransduction process and/or photoreceptor/retinal pigment interaction. The localisation of free (histochemically reactive) zinc within the photoreceptors changes with light stimulation. In the dark-adapted retina chelatable zinc can be visualised primarily in the perikarya, whereas after photostimulation free zinc is mainly associated with the inner segments. MicroPIXE has been used to analyse the total (free and bound) zinc distribution in each retinal layer and a difference was seen between light- and dark-adapted retinas (preliminary data). Following light stimulation the content of zinc increased in the inner segments of the photoreceptors and the layer containing the photoreceptors perikarya (outer nuclear layer). The increment in the inner segments was several times greater than in the outer nuclear layer.

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BM-P14

CORRESPONDENCE ANALYSIS EVALUATION OF LINEAR NUTRIENT DISTRIBUTION IN ROOT TIPS OF THE TROPICAL FORAGE *Brachiaria Brizantha*

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The technique of Correspondence Analysis was applied to a set of data obtained from X-ray elemental analysis by Nuclear Microscopy. Samples analysed, form part of an ongoing research project at the Materials Research Group (MRG), National Accelerator Center (NAC), Faure and the International Centre for Tropical Agriculture (CIAT), Colombia. Hydroponic experiment simulating tropical acid soil conditions were carried out to determine possible mechanisms of Al-toxicity stress by specific varieties of the genus *Brachiaria*. In particular the species *B. brizantha* was tested for gradient variation along the central cylinder of selected root tips. Single-point irradiations by Nuclear Microscopy gave some indication of a possible trace element profile gradient along the root axis. To be able to extrapolate to a more confident level the possible correlation and trace elemental concentrations gradients the data matrix from Nuclear Microscopy was analysed by Correspondence Analysis. A clear gradient on the plot of the first two axes of the Correspondence Analysis was found. The correlation of Ca and Cu as well as that K and Cl was established.

BM-P15

MOBILISATION OF TOXIC ELEMENTS IN THE HUMAN RESPIRATORY SYSTEM

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This paper reports ongoing studies of the localisation and chemical characterisation of particulate matter in post-mortem tissue from the trachea, bronchi, lung tissue and lymph nodes of the lung periphery for healthy individuals who lived in an urban environment.
The fate of respired particles in the respiratory system is inferred through the chemical characterisation of individual particles in the epithelial regions of trachea and bronchi, and the accumulation of toxic elements in lung alveoli and lymph nodes.
The particles and tissue elemental distributions were identified and characterised using micro-PIXE elemental mapping of thin frozen sections using the ITN Nuclear Microprobe facility.
Apart from the visualisation of respired aerosols and elemental characterisation of isolated particles and macrophage inclusions, the elemental concentrations for the tissues surrounding deposited particles at epithelial region of the air ducts, as well as for lung and lymph node tissues can be achieved.
Higher concentrations of Si and Ti can be found in lung tissue and in lymph nodes relative to upper regions of the respiratory system, i.e., epithelial regions of trachea and bronchi. The cortical region of lymph nodes constitutes the preferential accumulation site for elements, such as Al, Si, Ti, Cr and Cu. The elemental distributions and accumulation patterns for toxic and essential elements such as, Al, Si, Ti, Cr, Fe, Cu, Zn, at different levels of the respiratory system, may express different mechanisms for removal and mobilisation of toxic elements and may provide information about the toxicity of particulate matter.

BM-P16

THE USE OF EXTERNAL MICRO-PIXE TO INVESTIGATE THE FACTORS DETERMINING THE Sr:Ca RATIO IN THE SHELLS OF FOSSIL ARAGONITIC MOLLUSCS

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The ratio of strontium to calcium in the shells of fossil marine molluscs has been proposed as an indicator of seawater temperature in past epochs, since this is less affected by other environmental conditions (such as variation of salinity) than the more conventional seawater palaeotemperature proxy $\delta^{18}O$. However, in this paper, we demonstrate that the Sr:Ca ratio is influenced by several factors including temperature and growth rate.
Two fossil molluscs, a large spiral gastropod, *Clavilithes macrospira* and a large bivalve, *Venericarda planicosta*, from the Eocene-aged deposits of Southern England were selected for study. These were mounted in such a way that they could be positioned precisely under the high-resolution beam of the Oxford external-beam facility, and the variation of the Sr:Ca ratio was measured in a direction perpendicular to the growth direction using PIXE. In the case of *Clavilithes macrospira*, analysis took place directly on the outer surface of the spiral, while

the shell of *Venericarda planicosta* was sectioned and polished prior to analysis. Small samples were also taken from both shells using a fine drill to allow $\delta^{18}O$ and $\delta^{13}C$ profiles to be measured.
The Sr:Ca profiles show a significant increase of Sr with age, as well as seasonal, possibly temperature related variations. Comparison of the Sr:Ca profiles with those for $\delta^{18}O$ and the spacing of the growth rings suggests that Sr incorporation is controlled primarily by metabolic activity, which in turn is influenced by factors such as temperature, salinity, age and growth rate.

BM-P17

VISUALISATION OF COLLAGENEOUS FIBRILS IN JOINT CARTILAGE USING STIM

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Nuclear Magnetic Resonance Microscopy can visualise the anisotropic water mobilty in joint cartilage. It is assumed that the anisotropic dipole-dipole interaction is determined by the arrangement of the collageneous fibrils. Thus, regions of radial and transversal alignment of the collageneous network with respect to the articular surface can be distinguished. There is a theory of cartilage degradation which assumes a calcium induced network transformation in the early stage of arthrosis.
Scanning Transmission Ion Microscopy was employed to investigate the collageneous network in tibial and femoral cartilage of pig's knee joint. The cartilage was cryosectioned into 30 μm thick cross sections and freeze-dried. It could be clearly proofed that the NMR-visible structures originate from aligned collageneous fibrils. The tibia has a relatively wide zone of radially aligned fibrils, whereas the femur shows a narrower ribbon of transversally aligned fibrils. Below the articular surface of both epiphyses the network consists of transversally aligned fibrils. The calcium induced network transformation is discussed in a separate contribution to this conference.
In the freeze-dried cross sections unexpectedly high ratios in areal mass density (up to a factor of four) were found between the zones of aligned and randomly distributed fibrils. The origin of this heterogeneity is not yet understood. Further investigations are under way.
Additionally, high resolution STIM images (20 μm × 20 μm, resolution 100 nm) were taken from collageneous fibrils surrounding cartilage cells. This possibility to visualise single fibrils is a first step to perform STIM tomography for a 3D visualisation of the cartilage collageneous network.

BM-P18

SPATIALLY RESOLVED ELEMENTAL DISTRIBUTION IN ARTICULAR CARTILAGE

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Cartilage research is focused on the degradation of joint cartilage. Biochemical and biophysical methods reveal principle approaches to the processes that lead to cartilage degradation but yield limited information on elemental distribution, only. In this study, the Nuclear Microprobe technique is employed to provide the lacking knowledge. The distributions of the elements P, S, Cl, K and Ca were investigated. Both femoral and tibial cartilage were analysed. The samples were taken from pig's knee joint. 30 μm thick cross sections were prepared by means of cryosectioning and freeze-drying.
PIXE and RBS analysis revealed information on the elemental distribution with respect to the internal structure of the cartilage which originates from aligned fibrils of the collageneous network. The main components of the organic matrix are C, N and O. It was surprising that their ratios vary with the cartilage structures. It could be shown that zones with aligned collageneous fibrils contain less sulphur and potassium but more chlorine. The higher chlorine concentration is remarkable because latest biochemical studies found that hypochloric acid is involved in cartilage degradation.
Furthermore, the calcium distribution is of great interest. Its correlation to structural changes inside the cartilage is still being discussed. However, our studies do not support the idea that zones of higher calcium concentration are related to the aligned structures of the collageneous network. It is more likely that the main part of calcium in joint cartilage is in the form of a calciumphosphate instead of Ca^{2+} ions, as has been assumed earlier.

BM-P19

A NUCLEAR MICROSCOPY STUDY OF IRON-RICH GRANULES IN THE PARKINSONIAN SUBSTANTIA NIGRA OF AGED AND YOUNG PRIMATE MODELS

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Parkinson's disease (PD) is a progressive neuronal degenerative brain disease of the elderly, which can be experimentally induced in primates by the neurotoxin 1-methyl-4phenyl-1,2,3,6-tetra-hydro-pyridine (MPTP). MPTP induces Parkinsonism in human and primates by selectively destroying dopaminergic cells in the Substantia Nigra region of the brain. Iron has been linked to cell death in Parkinson's disease (PD) because of its potential to promote free radical production, leading to oxidative stress. An increase in total bulk iron in the Parkinsonian substantia nigra (SN) of both human and animal models has been confirmed by several research groups using a variety of methods. We have also confirmed this result, and have also shown that dopaminergic cell death precedes the increase in bulk iron. This tends to suggest that the increase in bulk iron may be a secondary effect in PD.
More recently we have focused our attention on iron-rich neuromelanin granules in the SN, to determine whether or not they act as localised centres for free radical production. Since Parkinson's disease is an age-related illness, the current study focuses on the differences between aged and young monkeys to the MPTP-lesioning. A group of 3 old monkeys (older than 7-years) and a group of 3 young monkeys (younger than 7 years) were MPTP lesioned and sacrificed after 35 days (corresponding to an average 30% loss of dopamine producing cells). We have observed that only the SNs of the older monkeys appear to have iron rich granules, indicating that these may be storage sites that accumulate iron with age. In addition we have also observed that the focal iron distributions are anti-correlated with the surviving neurons as identified using Nissl staining. These and other histochemical studies tend to

implicate localised iron as a factor in cell death, and further, that cell death due to iron-catalysed free radical production is more probable with age.

BM-P20

MICRO-PIXE STUDIES ON GALLIUM INCORPORATION IN MINERALIZED TISSUE

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Gallium has been found to be a potent inhibitor of bone resorption and is clinically effective in controlling cancer-related hypercalcemia. To elucidate gallium's mechanism on mineralization we have examined its effects on mineral formation in a cell culture model. The mineralized nodules formed in this culture system have diameters of about 100 μm. Minerals are irregularly distributed over the nodule area. Mineralization is observed more at the central portion compared to the peripheral portion of the nodule. In-situ investigations have be performed with the use of experimental methods which enable investigations of the microstructures. The aim of the presented experiment is to determine the elemental composition of inorganic deposits formed within the nodule. The model of rat bone marrow stromal cells was adopted for experiment. The cells were seeded in a cell culture insert at a density of $4x10^6$ cells/insert and cultured for 27 days. Gallium was added to the medium. The doses of Ga ranged from 0 μg/ml to 500 μg/ml. The bottom foil of the insert was mounted on a plastic frame and used for further investigation. To determine the distributions of element contents at a spatial resolution of about 3 μm, micro-PIXE (microprobe in combination with Proton Induced X-ray Emission) was applied. It was demonstrated that the amount of incorporated gallium correlates with the concentration of the element in the cell culture medium. However, a nonlinear relation was observed. The correlation between P, Ca and Ga distributions was observed for gallium concentration in the medium lower than 50.0 μg/ml. It indicates that Ga is incorporated within Ca-P deposits. For higher Ga concentrations (>50.0 μg/ml) the precipitation of a separate gallium compound was detected. The influence of Ga on mineral formation is a dose-dependent processes. Gallium treatment stimulates Ca-P mineral formation at very low (< 1.0 μg/ml) doses. At high doses the impaired mineral formation is detected.

BM-P21

MINERALIZATION OF INORGANIC DEPOSITS IN AORTIC VALVE CUSPS

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Mineralization of the aortic valve homografts due to degenerative changes is the most common lesion encountered in patients who were subjected to the aortic valve replacement. The primary pathogenic mechanism(s) responsible for calcific degeneration of the aortic valve still remain unclear. Therefore the elucidation and potential elimination of the factors involved in the pathogenesis of aortic valve mineralization are of a great importance. The aim of the presented experiment is: (1) to determine the elemental composition and structure of

the inorganic deposits formed within the aortic valve cusps and (2) to check the correlation between the development of the mineralization process in the aortic valve cusps and in the aortic wall. A proton microprobe in combination with proton induced X-ray emission and with proton induced gamma-ray emission were used to determine the elemental composition while the structure of the deposits were investigated with the use of a X-ray microprobe in combination with X-ray diffraction method and a Fourier transform infrared microscopy. The investigations were carried out on 10 μm thick sections of the human aortic valve cusps and aortic wall. In the first step the concentrations of F, P, S, Cl, K, Ca Fe and Zn were determined at the cellular level. The presence of inorganic deposits was determined on the basis of the distributions of Ca concentrations. In the second step of the studies the structural data were collected for well defined regions of the sample. It was found that deposits in the aortic valve cusps are composed of Ca-P compounds. Fluorine is accumulated in the inorganic phase, however, the mechanism of F deposition is more complex and awaits further investigations. The only phase detected by the X-ray diffraction was a defective hydroxyapatite. The Fourier transform infrared microscopy revealed that the crystals were contaminated with carbonate groups. A strong correlation was observed between the development of mineralization process in the aortic valve cusps and the aortic wall. The latter observation may have a clinical implication in the assessment of the aortic valve cusp susceptibility to mineralization which is of a fundamental importance for the choice of valves for transplantation.

BM-P22

IRON IN RETINA OF RATS DURING THE PHASES OF DEVELOPMENT AND OF HEREDITARY RETINAL DEGENERATION

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The retina as well as other tissues needs iron to survive, but modifications in iron metabolism have also been suggested to contribute to cerebral neurodegenerative diseases. Our study was intended to investigate iron distribution in the retina of normal and RCS (Royal College of Surgeons) rats affected by hereditary degeneration of the retina at different developmental stages (from 25 to 55 days after birth).
Iron (Fe) distribution was determined by PIXE (Proton Induced X-ray Emission) microanalysis on retinal sections and debris collected from the space under neural retina (in the RCS rats), and compared to other tissues (cornea, liver, spleen) and to other elements (K, Ca, Cu, Zn and Br). Elemental concentrations were determined in different retinal layers especially the photoreceptors, which are progressively altered and disappear in the RCS rats.
Iron is unevenly distributed throughout the rat retina. The highest concentration is observed in the choroid and the retinal pigmented epithelium and in the inner segments of photoreceptors. Iron content is lower in the outer segments but still significant. It increases during both the development and the disease at the level of the segments and inside the debris. This last localised iron increase can result in an overproduction of free radicals and be correlated with the photoreceptor cell loss.

BM-P23

MICRO-PIXE ANALYSIS OF LUNG BIOPSY TISSUE OF A PATIENT SUFFERING FROM HARD METAL LUNG DISEASE

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Hard metal lung disease is an occupational respiratory disease that occurs in susceptible workers exposed to dust generated from the production or use of tungsten carbide. It is widely believed that cobalt is a causative agent of such a disease. Micro-PIXE analysis of a lung biopsy specimen taken from the right lung of a patient diagnosed to have hard metal lung disease confirmed the presence of a significant amount of tungsten and some titanium. The concentrations of Fe and Ca in the specimen were also higher than those in normal lung tissues. However, the concentration of cobalt was below detection limit and the cobalt-to-tungsten concentration ratio was estimated to be no more than one part in 500. Elemental maps obtained from a tungsten-rich region of the tissue revealed that the profiles of tungsten, iron and titanium are similar, implying that these elements are co-localised. PIXE analysis of airborne dust particles collected from the workplace where the patient performed his duty of grinding tungsten carbide edges of blades and cutters for the past ten years showed that the concentrations of cobalt and tungsten particles in the ambient air of the workplace during working hours were both high, and their ratio was about 5 to 100. These results suggest that cobalt-rich tungsten carbide particles were initially accumulated in the lung tissue of the patient, but in time the cobalt had leached out to leave only tungsten.

Session EPS: Applications in Earth and Planetary Sciences

EPS-P1

EXPERIENCE WITH THE KING FAHD UNIVERSITY SCANNING MICRO-PIXE FACILITY IN GEOLOGICAL APPLICATIONS

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The scanning microprobe facility on the 3 MV Tandetron accelerator at King Fahd University of Petroleum & Minerals has been applied extensively in various fields including geology. This paper summarizes some of the geological applications that were undertaken in the past few years. A brief account of the scanning proton microprobe set up is given. Elemental composition and distribution maps across single mineral grains, fluid inclusions, grain boundaries and matrices of some gold-bearing rocks and phosphate deposits in Saudi Arabia as well as several volcanic sediments are reviewed. The results reveal unique microstructural features of particular significance to fundamental geological processes, ore genesis and ore body recognition in the samples studied.

EPS-P2

$^{14}N(d,po)^{15}N$ MICROANALYSIS OF SILICATE PHASES IN THE RENAZZO (CR) CHONDRITE

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Nitrogen contents were determined in olivines, pyroxenes and in a glass inclusion in olivine of the Renazzo chondrite using the nuclear microprobe facilities of the Pierre Süe Laboratory (Saclay, France).
In the glass inclusions, the N content was measured using either a scanning mode over an area of ~ 20x20 μm^2 or punctual analysis. Three areas in the same inclusion gave a mean content of 90 ppm N. Punctual analysis in three small-size glass inclusions show contents of 500, 180 and 240 ppm. For olivines and pyroxenes the N contents are below the detection limit (< 20 ppm).
The recent advances in the technique for measuring Mg-rich matrices allow us to conclude that N is present only in low concentration in olivines and pyroxenes of the Renazzo carbonaceous chondrite. However, it is present in highly variable amounts in glasses of glass inclusions in olivine. This behaviour of N is similar to that observed for C [1]. Thus, under conditions prevailing during condensation of olivine and pyroxene in the early solar nebula [2] (at temperatures of 1300 K and 10^{-3} at pressure) N seems not to enter the structure of these phases. However, it can be stored in glasses of glass inclusions contemporaneously formed with the host. The original as well as the current speciation of N are unknown but trapping of a condensed refractory N species which subsequently was oxidised to N_2 appears to be a likely process for N incorporation into glasses. The detection of high contents of N in the bubble of a glass inclusion in the Vigarano meteorite [3] supports the presence of N as a volatile species
1) Varela et al., (1999), Geochim Cosmochim Acta (submitted); 2) Grossman (1972) Geochim: Cosmochim Acta, 36, 597-620; 3) Varela et al., (2000), Lunar Planet. Sci. Conf. 1620.

EPS-P3

DISTRIBUTION OF VARIOUS COMPONENTS IN A HYDROGENETIC FERROMANGANESE NODULE AND AN AFANASIY NIKITIN SEAMOUNT CRUST FROM INDIAN OCEAN - A GEOCHEMICAL STUDY USING MICROPIXE.

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The present study emphasizes the geochemical features pertaining to the distribution of the major and minor elements in a hydrogenous ferromanganese nodule and a seamount crust originating from the Indian Ocean. The micro-PIXE elemental maps indicates the successive layer formation of Fe and Mn in these deposits. A Ni associa on with Mn has been further confirmed by observing a Ni - Mn hot spot. In addition, the core or the nucleus of the ferromnaganese nodule has been found to be rich in Fe and not Mn, which strengthens the assumptions made earlier that the nodule formation s rted with Fe deposition which catalyzed the growth of a ferromnaganese nodule or crust by successive deposition of Mn and Fe. The irregular patterns of Mn and Fe layers observed, has been outlined. A few Ti - Fe correlations

have been observed, which are tributed to Fe-Ti phase. It strengthens the conjecture that there are multiple Fe phases as discused in a Mossbauer study by one of the present authors.

EPS-P4

PROTON MICROPROBE STUDIES ON THE DISTRIBUTION OF SIDEROPHILE METAL IN 144 MA MOROKWENG IMPACT MELT SHEET

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The ~145 Ma Morokweng structure, western North West Province, South Africa, has a diameter [3] 150 km and ranks among the largest confirmed asteroidal impact scars known on our planet. The core of this structure consists of lenticular melt sheet, some 25-30 km in diameter and >500 m thick of granophyric quartz norite. This body shows evidence of chemical differentiation from a more granitic upper part to intermediate compositions at greater depth. Siderophile metals, namely, Ni, Co, and the platinum group metals [PGMs]) which are thought to be of meteoritic origin are highly enriched in the quartz norite, and largely concentrated in sulphide-metal oxide
intergrowths never before observed in impact melt sheets. In this study the authors have undertaken to map out the distribution and mineralogy of the siderophiles in the intergrowths. This study not only has metallogenic applications, but also addresses fundamental questions on the behavior of the PGMs at high temperature and very reducing conditions.

EPS-P5

MAGNETIC SPHERULES AS MARKERS OF PALEOZOIC IMPACT IN HUNGARY

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Microtektites and cosmic spherules are generally small and rounded glass or magnetic objects. They occur on widely separated territories of the Earth [1]. Glassy and magnetic spherules from river beds and by continuous cores of drillings Miocene, Pliocene and Quaternary sediments were uncovered in Hungary and studied by micro-PIXE and SEM-EDAX techniques earlier [2]. The origin of these findings, which can be of extraterrestrial, volcanic or antropogenic, are not unambiguously cleared yet. However, from a drilling hole spherules different from the above ones were found in the south part of Hungary. They were explored from sediments attached to the surface of Paleozoic granitoid rocks. It was pointed out by micromineralogical analysis that the sediments embedding the spherules contained moissanite (SiC) which mineral is known as an ingredient of meteorites. Therefore, investigations were started to prove the extraterrestrial origin of these spherules.
The scientific studies was done by using micro-PIXE techniques. The microanalysis was carried out by the nuclear microprobe unit of the Institute of Nuclear Research of the Hungarian Academy of Sciences (ATOMKI). The x-rays were detected with two Si(Li) detectors determining the concentration values of light and heavier, trace elements at the same time. On the basis of the morphology and composition of the samples it was concluded that signs of a meteoritic impact could be found.

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EPS-P6

STUDY OF MINERAL INCLUSIONS IN OBSIDIAN GLASSES

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From several point of view interesting representatives of natural glasses are the obsidians which are silicic, volcanic products [1]. Our investigations in the recent past primarily focused on the chemical composition of these glasses and related to the comparison of various glass structures with archaeometrical aspects. The subject of the current, geological study is the mineral inclusions found in the obsidian glass matrices. First, thin sections of obsidians were produced in order to identify the mineral phases by polarizing microscope. The compositional analysis was then carried out by micro-PIXE method. The microprobe measurements were done at the Institute of Nuclear Research of the Hungarian Academy of Sciences. Samples from Armenia, Hungary, Island, Italy, Mexico, Slovakia and Turkey were included in the analysis. The following inclusions having various sizes (10-500 μm) could be identified and measured: quartz, rutile, chalcopyrite, pyrrhotine, olivine, zircon, biotite, amphibole, feldspars, sanidine, monazite, apatite and anhydrite.
On the basis of the quartz and siliceous minerals petrological and petrogenetical processes are detailed and we could indicate the tectonical settings and the properties of the magma chambers. Moreover, by the identification of some accessory minerals (such as anhydrite, chalcopyrite, pyrrhotine, monazite) further geological processes could be concluded.
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EPS-P7

INDIVIDUAL FLUID INCLUSION ANALYSIS AS A TOOL FOR PREDICTING PERMEABILITY BARRIER IN OIL RESERVOIR

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Understanding fluid circulation in the Earth's crust is a major issue in numerous geological processes. Fluid inclusions are little amounts of trapped and preserved water, brine or oil in rocks. Therefore, they represent a unique and direct sampling of paleo-crustal fluids. Our group has developed a method of quantification of single fluid inclusion composition based on the complementary use of nuclear and photon high-resolution microprobes (PIXE-PIGE and Synchrotron X-ray fluorescence; Philippot et al, 1998, Chem. Geol., 144, 121-136 ; Menez et al., 1999, NIM B, 158, 533-537).
The recognition of oil reservoir heterogeneities responsible for field compartmentalisation, is a key issue to reach the production targets and to prevent or reduce expenses involved by unrecognised barriers to fluid flow. During exploitation of Dunbar field, North Sea, a connectivity discontinuity was found, preventing extraction from the lower part of the

reservoir. Sr residual salt profiling showed a major break in $^{87}Sr/^{86}Sr$ ratio corresponding to a coal layer that behaved as a first order permeability barrier. In order to test if individual fluid inclusion analysis can be used to characterize impermeable barriers in oil reservoirs, primary fluid inclusions lining overgrowth zones of sandstone quartz were investigated using PIXE-PIGE and SXRF techniques in samples collected on both sides of the barrier. Our preliminary results clearly show the occurrence of two different fluid populations on both sides of the barrier. Among important compositional discrepancies is a high Zn concentration in the upper part of the reservoir contrasting with Fe enrichment and strong Zn depletion below the barrier. These results are discussed in terms of different degrees of organic matter evolution and/or distinctive reservoir infilling events.

EPS-P8

STRUCTURAL POINT DEFECTS IN "ISLAND SPAR" CALCITE

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The standard of calcite ($CaCO_3$) is the well known Island spar from the historical locations of Helgustadir,. Stedji-Berg and Hvalfjoerdu in East Island and Hoffel in South-East Island.
We analyze Island spar samples from Helgustadir, Stedji-Berg, Hoffel and two samples of unknown locations using the combination of:
- micro-PIXE for quantitative trace element analyses,
- CL-spectroscopy/-microscopy for analyzing the Mn^{2+} lattice position and mapping the relative Mn^{2+} distribution and
- ESR-spectroscopy for determining paramagnetic point defects.

Although Island spar is one of the best chemically and structurally investigated natural minerals, there are still some unclear phenomena of structural defects. The CL of island spar frequently varied from dark blue (intrinsic) to bright orange caused by Mn^{2+} built in the Ca^{2+}-position of the calcite structure. However, Barsanov & Sarsembaeva (1962) described additionally an uncommon green luminescence - which was also found in one of our samples- and attributed this to Mn^{2+} in interstitial lattice position. The CL-spectroscopy data of our sample reveals a band at 520 nm being the reason for this uncommon green CL of calcite. At present, our experimental results of ESR do not support the assumption of Barsanov & Sarsembaeva (1962). On the basic of PIXE-, CL- and ESR-analyses and defect model simulations we discuss different point defects as possible explanation of the uncommon green CL of Island spar. We give new data of structural point defects in Island spar from different sample locations.

Lit: Barsanov & Sarsembaeva (1962). Akadmiya Nauk S.S.S.R., 13. 147-152.

EPS-P9

IMAGING FLUID INCLUSION CONTENT USING THE NEW CSIRO-GEMOC NUCLEAR MICROPROBE

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During ore formation, samples of ore fluids are trapped as fluid inclusions in minerals such as quartz. The analysis of these trapped fluids holds the key to understanding the origins of ore metals, fingerprinting fluid sources, identifying fluid pathways, and proving insight into ore

formation processes. PIXE analysis of fluid inclusions using the nuclear microprobe provides a direct non-destructive method to determine the composition of these trapped fluids. However, some results have been controversial, such as the strong partitioning of Cu into the vapour phase in populations of coexisting brine and vapour inclusions, and high concentrations of Pb (~ 5 wt%) and Ba (~10 wt%) in some ore-related fluid inclusions.
In order to demonstrate the reliability of the PIXE approach, and eliminate spurious sources of important elements (e.g. S, Cu, Pb, Zn, Ba), the internal contents of fluid inclusions have been imaged using the new CSIRO-GEMOC Nuclear Microprobe in a method that uses identification and quantitative integration of the inclusion signal from PIXE images. Examples of this approach show clearly that these elements reside within the fluid inclusions. The images also show, and allow discrimination against, solid phases outside the inclusions, that may be consumed along with the inclusion by destructive methods of analysis and thereby bias those results.
The observation of high Cu partitioning into vapour inclusions reinforces earlier results and points to vapour-separation and transport as an important factor in the formation of Cu, and probably Au, ore deposits. The high Ba result places an important constraint on some ore fluids suggesting very S deficient conditions that prevent the loss of Ba from the fluid as barite.

EPS-P10

NUCLEAR MICROPROBE ANALYSIS OF MELT INCLUSIONS IN MINERALS: WINDOWS ON METASOMATIC PROCESSES IN THE EARTH'S MANTLE

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Samples of melts and fluids, responsible for metasomatic change and evolution of the earth's upper mantle are often preserved as inclusions in minerals carried to the earth's surface in volcanic eruptions and provide unique windows on metasomatic processes in the mantle. However, because these melts crystallize as heterogeneous assemblages they pose special problems to quantitative analysis. High field-strength elements, such as Zr and Nb, as well as Th and the rare-earth elements can become highly concentrated into rare minor phases on cooling, and these minor phases can be interspersed within a complex network of quench crystals.
Ideally, in order to reconstruct the composition of the trapped melts it would be necessary to integrate the entire inclusion volume. A practical alternative is to analyze melt composition directly using inclusions identified in polished thin-sections. However, the minor phases are easily overlooked using surface analysis techniques, and a more accurate approach is to use Nuclear Microprobe analysis due to the sensitivity of PIXE and the deep penetration of MeV protons which samples inclusion content, including minor quench phases, to ~40 μm depth.
A method has been developed using the CSIRO-GEMOC Nuclear Microprobe to image the minor and trace element components of melt inclusions, and integrate all contributions across and into each inclusion, using the *Dynamic Analysis* method developed at the CSIRO. Examples show that this approach can quantitatively image spatial variation in component elements at 1-2 μm resolution and at levels of ~2 ppm and determine reliable melt composition with detection sensitivities down to 0.2 ppm.

EPS-P11

SORPTION MECHANISMS OF AQUEOUS IONS ON HYDROXYAPATITE SURFACE : ANALYTICAL CHEMISTRY AND NUCLEAR MICROPROBE RESULTS

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In the concept of geological repository for high level nuclear waste, mineral assemblies of the near field are used to delay the migration of radionuclides through the geosphere. Hydroxyapatite surfaces, as well as clay materials, exhibit potential high retention properties for aqueous ions (cations or anions) and especially fission products or actinides.
Previous studies devoted to the behaviour of mineral barriers such apatites, clay, and other type of rocks versus aqueous ion sorption have been only considered from the point of view of analytical chemistry supported by thermodynamics calculations [1, 2]. Or from the point of view of sorbed specie characterization using appropriate physical techniques [3, 4].
In this work, we have intent to combine these two complementary approaches. Hydroxyapatite powder has been suitably conditionned and then put into contact with aqueous solutions containing ions covering a large interval of oxidation degrees and ion nature from Cd(II) to U(VI) through Am(III), Se(IV) and Np(V). Radioisotope tracing and conventional analytical chemistry procedures have been applied to measure sorption isotherms versus the pH and the ionic strength of the solution. Nuclear microprobe experiments have been carried out, by coupling μPIXE and μRBS, in order to quantify the sorbed quantities and determine their depth distributions. This paper is focused on the behaviour of Se
Results from this " two approach methodology " are presented and discussed.
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Session EB : Applications in Environmental Sciences and Botany

EB-P1

SORPTION MECHANISM OF Pb (II) ONTO ZEOLITE X BY ION-BEAM TECHNIQUE

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A variety of minerals are proposed as sorbent phases in the treatment of waters or of industrial wastewater containing metal toxic species. Detailed metal sorption results using different

minerals have been reported by several authors. Angove *et al*[1] reported the adsorption aqueous of cadmium (II) on kaolinite by two distinct processes while Zachara *et al*[2] investigated the adsorption aqueous of chromate on kaolinite. Ma *et al*[3] tested the apatite to immobilize heavy metals *in situ*. Badillo-Almaraz[4] studied the adsorption aqueous of cadmium (II) and selenium (IV) on hydroxyapatite by nuclear microprobe techniques.
A serious contamination by lead in groundwater, soils, sediments and the most dangerous in children has been reported at north of Mexico. In this work we proposed to use zeolite X in order to remove Pb and to minimize bioavailability in these environments because the lead is a heavy metal and is potentially toxic to mammals.
In the present study, the sorption mechanism of Pb has been studied by nuclear microprobe experiments by coupling μPIXE and μRBS based on the sorbed quantities of lead and the depth profiles of this element in zeolite from the surface. Two types of experiment were used to study the sorption of Pb (II) onto zeolite X samples at 25 C. Sorption isotherms at constant pH and variation of concentration of element and sorption isotherms at concentration of lead constant and pH variable. Electron microscopy TEM and SEM confirmed the mechanism. The sorption mechanism did not alter the morphology of the zeolite grains.

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EB-P2

APPLICATIONS OF NMA AND XPS TO THE CHARACTERIZATION OF THE CHEMICAL SPECIES FORMED ONTO OXIDIZED PYRITE SURFACES

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Pyrite (FeS_2) is the most widespread metal sulfide at the surface of Earth and particularly at contents comprised between 1 to 4 weight per cent in clayey minerals. The oxidative dissolution of pyrite by O_2 and Fe(III) (the main oxidizing agents present in natural medium) of pyrite contributes to the formation of sulfuric acid. This acid release induces drastic consequencies in environmental concerns: acid mine drainage, formation of acid sulfate soils and migration of radionucleides from a nuclear waste package stored in a clayey environnement.

Pyrite alteration in oxidative conditions concerns both oxidation and dissolution . The aim of our work is to determine if oxidation occurs in solid state or after dissolution. For this purpose, pyrite dissolution experiments in different conditions of pH and Eh are conducted, and the originality of this study (relatively to similar studies reported in the literature) is based on the use of two complementary approaches: solid and solution characterization. Nuclear Microprobe Analysis (NMA) with the two resonant reactions $^{12}C(p,p)^{12}C$ at 1.725 MeV and

$^{16}O(\alpha,\alpha)^{16}O$ at 3.05 MeV give informations about atomic ratios and the distribution of oxidation products (clusters or covering film?) at the pyrite surface. X-ray Photoelectron Spectroscopy (XPS) indicates the redox degrees and the nature of oxidation products of Fe and S onto the pyrite surface. The analytical techniques in solution bring iron and sulfur speciation. The NMA results on a pyrite oxidized in a HCO_3^- medium shows that oxidation products are heterogeneously distributed and under the form of clusters of variable thickness. Moreover, different S to O ratios have been evidenced, traducing the formation of intermediate species of sulfur onto the surface. In the same way XPS results indicate that the oxidation products are the following: : Fe(II)-OH-SO_4-CO_3, Fe(II)-S-S_xO_y-SO_4. The "two approach methodology" already indicates that pyrite dissolution mechanisms in oxidative conditions occurs both in solid state and in solution.

EB-P3

ALTERATION OF GLASSES BY MICROORGANISMS

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Microorganisms are suspected to play an undeniable role in materials alteration. Obviously, they will be present in nuclear waste repositories, either introduced by technological activity or laid by fluids circulation. Their metabolism may induce chemical modifications to the surrounding media and then affect the chemical durability of storage materials.
Biodegradation of glasses is studied in the Pierre Süe laboratory. Experiments with various species of bacteria and fungi are carried out with the collaboration of a microbiologists team interested in stained glass alteration. Ion beam techniques (PIXE, RBS) are performed to quantify surface modification of glasses and elemental incorporation in microorganisms. Analyses of the solutions will lead to a complete assessment of elemental exchanges between glass sample, culture media and microorganisms. In this presentation, we will present preliminary results of characterization of glasses and microorganisms by nuclear microprobe analysis.

EB-P4

PIGE AND XRF ANALYSIS OF A NANO-COMPOSITE PILLARED LAYERED CLAY MATERIAL FOR NUCLEAR WASTE APPLICATIONS

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A special tailored aluminum pillared montmorillonite, was prepared for radio-strontium removal in complex ionic environment. In a first series of experiments, the methods of PIGE and XRF were used to characterize the material as a cation exchanger. Furthermore, the charge carried by the pillars, a critical quantity of the pillaring process, was estimated during all the steps of its preparation. A qualitative model for the organization and structural evolution of the clay lamellas from the nm to the μm scale, was developed. The possible use of a micro-beam for quantitative studies of pillared clays, will be discussed.

EB-P5

APPLICATIONS OF MICRO-PIXE TO ATMOSPHEIRC AEROSOL STUDIES

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Last several years, the importance of global environmental problems has been recognized at the worldwide. Atmospheric aerosols play an important role in global warming/cooling and acid deposition as well as local pollution problem. The physical and chemical properties of aerosol particles are fundamental to understand the effects of aerosols on the atmospheric environment. The characteristics of aerosol particles are described by a number of physical and chemical factors. In general, the most important factors are the concentration, particle size and chemical composition.

The properties of aerosols are usually evaluated as the average of a large number of particles, namely as a bulk sample. It is ultimately recommended that the properties of the single particles be revealed. In this study, Micro-PIXE technique was applied to characterize the physical and chemical properties of the single aerosol particles.

Atmospheric aerosols were collected onto the Nuclepore filter using a two-stage filter pack sampler that can classify into fine and coarse fractions or a low-pressure Cascade Impactor that can classify the aerosols from about 0.01 to 30 micro-meter into 13 size ranges. Micro-PIXE analysis was carried out with the micro-beam system at the Advance Radiation Technology Center, Japan Atomic Energy Research Institute. It consists of a 3 MV single-ended electrostatic accelerator, a beam control system and a data taking system from X-ray detector. Micro-PIXE measurements were performed with a scanning 2.5 MeV H^+ beam having the beam diameter of 1-2 micro-meter and the beam current of >100pA.

Only fairly large particles larger than several micron-meter were measured here because of the limitation of the size of micro-beam. The main elements of large particles were Si, K, Ca and Fe that were main components of soil dust. More than 90 % of S exists in the fine fractions in most cases. But during Kosa dust that is transported from deserts in central Asia, S was also found almost in all single large particles. The information of elements of single particles allows estimating the origin of each particle. Further study to determine the absolute concentration or amount of each element is required to understand the behavior of aerosols and to evaluate the effects of aerosols on atmospheric environment.

EB-P6

CAPABILITIES OF NUCLEAR MICROPROBE AND COMPLEMENTARY TECHNIQUES AS XPS, NAA AND TOC ANALYSIS TO STUDY A MODEL TERNARY SYSTEM : SILICA/HUMIC ACID/IODINE

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The presence of organic matter as humic substances present at contents comprised between 1 and 10 µg/g in natural waters may affect the behavior of radiotoxic elements in the geosphere. Indeed these organic substances possess important complexation properties towards the

metallic elements and present an affinity for the mineral surfaces. Due to their colloidal form, they can contribute to the migration of the radioelements. The understanding of the behaviour of iodine potentially released in the environment is the subject of many researches (medical, nuclear fields) because of the radiological hazard posed by two of its radionuclides (^{129}I and ^{131}I of respective half-lives $1.57.10^7$ y/8.04 d). Moreover, according to the conditions prevailing in natural waters, iodine would be present as the mobile anion I^-. Our results obtained by Neutron Activation Analysis and PIXE on natural humic substances (from different geochemical environments), have evidenced non negligible iodine concentrations (from some hundreds of μg/g to more than one weight per cent). Our scientific aim is oriented to the understanding of the associations humic substances/iodine: role of the organic matter in the retention of iodine onto the mineral surface in a model ternary system silica of high purity/commercial humic acids/iodine? nature of humic substances/iodine bonds? The nuclear microprobe is particularly suitable for this study by coupling microPIXE and the $^{12}C(p,p)^{12}C$ reaction at the resonance energy of 1.725 MeV. Indeed, PIXE permits to quantify iodine and trace elements scavenged by humic acids and $^{12}C(p,p)^{12}C$ reaction allows the evidence of the thickness of humic acids films sorbed on silica slide. The results have showed a heterogeneous distribution of the humic acids and mappings of silica slide surfaces by the beam have evidenced associations between iodine and humic acids. Our complementary results by X-ray Photoelectron Spectroscopy on model iodinated molecules and natural humic substances rich in iodine have showed that iodine is attached to the organic matter by covalent bonds.

EB-P7

NUCLEAR MICROPROBE STUDIES OF ELEMENTAL DISTRIBUTION IN SEEDS OF *Biscutella leavigata* L. FROM ZINC WASTES IN OLKUSZ, POLAND

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Biscutella laevigata L. (Brassicaceae), a rare perennial metallophyte plant species, known mainly from Central and Southern Europe, has a very restricted distribution in Poland. This investigation focuses on the only known lowland location of *B. laevigata* on zinc dumps near Olkusz, Upper Silesia. The results presented are a continuation of earlier studies to understand mechanisms of plant adaptation to a hostile environment which is rich in heavy metals. Previous investigations showed metal distributions in seeds of *Silene vulgaris* (Moench.) Garcke and *Gypsophila fastigiata* L. (Caryophyllaceae) - species with different seed structure and different ecological characteristics. The NAC nuclear microprobe was used to obtain elemental distributions by the simultaneous use of PIXE and proton backscattering spectrometry (BS).

Quantitative elemental maps showed that differing seed tissue had well-defined specific elemental distribution patterns. Zn was the only heavy metal present in relatively high concentrations and its highest concentration was found in the endosperm (i.e. the non-embryonic tissue). This correlates with the distribution of other heavy metals. Within embryonic tissue, the highest concentration was found in the radicle while the Zn distribution throughout the remaining parts of the seed was relatively homogeneous. Artefacts caused by the geometry of specimens are discussed.

The results show that plants specifically and actively select both the elements and the respective concentrations taken up in different tissues. This mechanism is probably responsible for their survival.

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EB-P8

STUDY OF ZIRCALOY-4 FUEL CLADDINGS CORROSION USING ION BEAMS APPLICATION TO LONG TERM DISPOSAL OF NUCLEAR WASTES

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In the pressurised water reactors the cladding tubes in zircaloy-4 are oxidised on several micrometers on the internal face by direct contact with the UO_2 pellets. At the same time, fission products such as ^{129}I are implanted by recoil. During reprocessing the cladding tubes are cut up and washed with nitric acid to dissolve the spent fuel. Until 1995, the cladding tube pieces called hulls were embedded in concrete. The concrete medium being very basic, this study simulates the corrosion of hulls in the perspective of a long term disposal in these severe pH conditions. This paper is dedicated to the study of the partial dissolution of the oxide layer, which is the responsible mechanism for the activity release. In order to follow the solid-liquid interface, europium was implanted at the material surface as a marker. Next, the corrosion induced in autoclave at 300°C, 140 bars and in alkaline water was studied. The determination of europium profiles using Rutherford Backscattering Spectrometry analysis (RBS) allows to deduce the fraction of dissolved ZrO_2. It was shown that this dissolution is inhomogeneous and that it gives rise to the formation of crevices. These results are confirmed by Scanning Electron Microscopy. Using the nuclear microprobe of Pierre Süe laboratory, a more precise study of the specimen surface was performed.

EB-P9

STUDY ON UPTAKE OF EUROPIUM BY THE MIXTURE OF APATITE AND SMECTITE USING μ-PIXE AND RBS

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Uptake of rare earth elements (REEs) from a contaminated groundwater is an important issue in environmental remediation. Here we report the uptake mechanism of europium, one of the REEs, by the mixture of apatite and smectite. Thin film of the mixture of apatite and smectite was contacted with 1 mM Eu, one of the REEs, solution for 2 days at 25 C and at initial pH 4.5. After separating the film from the solution, the surface distribution of europium was determined by μ-PIXE developed in the TIARA facility of JAERI. Depth profiles of elements

in the film were also obtained by Rutherford Backscattering Spectroscopy developed in the TIARA facility.
RBS spectrum shows that Eu, Si (major component of smectite) and P (major component of apatite), were distributed from the surface to deeper position. This indicates that Eu was not only sorbed on the surface of the mixture film, but also penetrated to deeper areas from the surface. The surface distribution of europium has not good correlation with Si but with P. This indicates that most of europium sorbed on the mixture was associated with apatite. Almost all of europium was sorbed by smectite, when a 1 mM Eu of 30 ml was contacted with 0.1 g smectite for 1 day indicating that smectite has high performance on the uptake of europium from groundwater. These suggest the uptake mechanism as follows: Europium sorbed by both apatite and smectite at the initial stage of uptake by the mixture, followed by the movement of the sorbed Eu from smectite to be associated with apatite. Since apatite works as sink of the sorbed Eu by the mixure, the mixture of apatite and smectite is an effective material to remove REEs from contaminated groundwater.

EB-P10

HEAVY METAL LOCALIZATION IN *Suillus luteus* MYCORRHIZAS – COMPARISON OF PREPARATION METHODS AND MICROANALYTICAL TOOLS

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Mycorrhizas of *Suillus luteus/Pinus sylvestris* collected from zinc wastes in Southern Poland were selected as potential biofilters. The fungus was not only extremely resistant to heavy metals but in addition it accumulated high level of heavy metals within outer part of the hyphal mantle. This kind of detoxification mechanism has been so far reported only in case of *Rhizopogon roseolus*. Mycorrhizas of *Suillus luteus* were prepared by freeze drying technique or using conventional chemical fixation protocol, followed by dehydration and embedding in epoxy resin. The material was analysed by SEM, TEM and proton microprobe using EDS, EELS and PIXE techniques. Each of these methods has its own advantages. SEM accompanied by EDS is the best for selection of the material and analysis of large number of mycorrhizas. Unfortunately data obtained cannot be treated as quantitative due to lack of proper standards. Much more reliable data can be obtained using PIXE. Both techniques can hardly be used for element mapping on the ultrastructural level. TEM accompanied by EDS and EELS is more useful in such cases. However, the data obtained on chemically fixed material are always controversial, as there is a possibility of redistribution of elements during material preparation. For that reason the PIXE elemental maps obtained on freeze-dried and chemically fixed material were compared. Although the possibility of elemental movement at the ultrastructural level could not be completely eliminated, there were no visible differences in localization of heavy metals observed under 1000x magnification. On the other hand, the removal of Cl or S from the mycorrhizas into the resin blocks was an obvious phenomenon observed in chemically fixed material. Regardless of these artefacts, the results of heavy metals accumulation within outer fungal mantle were confirmed by all methods used.

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EB-P11

HEAVY METAL UPTAKE OF GEOSIPHON PYRIFORME

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Geosiphon pyriforme, is a symbiotic fungus belonging to the arbuscular mycorrhizal (AM) fungi. More then 80% of the land plant species form an AM, showing clearly the importanceof this symbiosis for the terrestrial ecosystem. *Geosiphon pyriforme* is the only known endo-symbiosis of a fungus with blue green algae (cyanobacteria). 18s-gene analyses have shown doubtless, that the fungal partner of this symbiosis belongs to the arbuscular mycorrhiza (AM) fungi. The *Geosiphon* symbiosis is characterised by the unicellular symbiotic "bladders", which are 1-2 mm in size and thus an easy to investigate system. Therefore we use *Geosiphon* as a model system for the AM, allowing us to study many topics difficult, or not possible at all, to investigate in the AM symbiosis. For heavy metal polluted soils, phytoremediation (use of plants for remediation) can include the use of hyperaccumulative plants, tolerant plants and additional treatments and combinations of plants and mycorrhizal fungi.
For a long time, plants on heavy metal soils were believed to be non-mycorrhizal, but recently it was shown, that that some plants are strongly colonised by AM-fungi, including a species which confers heavy-metal tolerance also to crop plants.We now try to answer some basic questions regarding heavy metal uptake or resistance of AM-fungi. These are e.g. 1) which heavy metals are taken up to what extent? 2) where are heavy metals deposited, in the vacuoles, cell wall, the symbiotic compartment? 3) confers the symbiosis heavy metal resistance to the photosynthetic symbiosis partner? We present quantitative micro-PIXE measurements of a set of heavy metals (Cu, Cd, Tl, Pb,) taken up by *Geosiphon*. The uptake is studied as a function of the metal concentration in the culture medium and on the time *Geosiphon* spent in the heavy metal enriched medium. Also the influence of the heavy metal uptake on the nutrition transfer of other elements will be discussed. The measured heavy metal concentrations are in a range of several ppm to some hundred ppm.

EB-P12

AIR POLLUTION SOURCE IDENTIFICATION BY COMBINING THE MICRO-PIXE SPECTRUM OF A SINGLE AEROSOL PARTICLE WITH THE PATTERN RECOGNITION TECHNIQUE

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In order to find the pollution sources of individual aerosol particles, we developed an identification system by combining their micro-PIXE spectra with the pattern recognition technique (PR). Each individual particle was characterized by its PIXE spectrum, which was treated as a fingerprint. A fingerprint library, which collected the characteristic spectra of aerosol particles from different pollution emitters, was established. The PR technique was then used to identify any unknown particle by comparing its spectrum with those in the library. This approach essentially emulated the human visual inspection of the spectral feature, but on a more quantitative and accurate basis. Both the Leipzig Nanoprobe and the Shanghai microprobe were used for the micro-PIXE measurement. More than one thousand spectra of

the single particles, collected from the Shanghai atmosphere, were applied to test the PR system. Most of the particles were smaller than 3 μm and many of them even less than the beam spot size (1 μm). The influence of limited particle size upon the spectrum feature was considered theoretically and experimentally. The application of the microprobe technique to small objects (< 1 μm) measurement was discussed. The result of the PR test showed that the cement factory is the primary source of the aerosol particles in this region and that lead contaminated particles from automobile exhaust should not be neglected even though leaded gasoline had been prohibited since 1997 in the city. About 14% of unidentified particles were clustered and some new pollution sources were found.

Session AA : Applications in Art and Archeology

AA-P1

THE STUDY OF THICK CORROSION LAYERS ON ARCHAEOLOGICAL METALS USING CONTROLLED LASER ABLATION IN CONJUNCTION WITH AN EXTERNAL BEAM MICROPROBE

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The variation with depth of the composition of corrosion layers on buried metal objects can provide the archaeologist with valuable information relating to the burial conditions of the object. In some cases these layers can be very thick (up to 1mm) and so normally, destructive methods such as sectioning are used to characterise the layers. This paper describes the use of a microfocused high power pulsed Nd:YAG laser to ablate the corrosion layer in a series of controlled steps while monitoring the composition of the exposed surface using PIXE and RBS in the external beam facility of the Oxford Scanning Proton Microprobe. The region of the surface removed by the laser beam is typically less than 500μm diameter and so by comparison with other sampling techniques, the effect on the appearance of an object is minimal.
The modifications to the facility to allow the precise registration of the laser beam and the ion beam are described and preliminary results on corroded bronze artefacts are presented.

AA-P2

ANALYSIS OF GLAZES WITH MICRO-BEAMS OF CHARGED PARTICLES: EXAMPLES FROM THE STUDY OF SOME DELLA ROBBIA TERRACOTTA SCULPTURES

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The Italian renaissance terracotta sculptures are characterised by the use of bright, opacified, coloured glazes which appear for the first time, on large artefacts, with the production of the

Della Robbia bottega in Florence. A glaze is a vitreous coating, about 200 μm thick, composed of silica that forms the glaze infrastructure, different fluxes that lower the melting point of the mixture, metallic oxides as colours and opacifying agents. Compared to glass, glazes are often heterogeneous on a micro scale, due to the presence of non melted constituent grains, 5 to 10 μm in diameter, and secondary minerals. The structure and texture of a glaze, as well as the occurrence of minor and trace elements, depend on the recipe followed in the preparation of the mixture and on the sources of raw materials and should help characterising each pottery and different periods of production. We have applied both Scanning Electron Microscopy (SEM) and μ-PIXE to several artefacts of the Italian renaissance period to differentiate the original production of Della Robbia bottega from contemporary or late imitations, specially in the French collections.

In our analyses we have given emphasis to micro-samples since for most of the artefacts, that cannot be removed from the museum, this is the only kind of samples made available. Compatibility of SEM and μ-PIXE on thin polished sections has been extensively checked and micro-analyses at a 100 μm scale have been integrated with the information given by direct analysis on potsherds or whole artefact on large (0.5 x 0.5 mm^2) areas. The results on a few objects from the Louvre museum are presented.

AA-P3

SURFACE MICRO-DISTRIBUTIONS OF PIGMENT AND THE RELATION BETWEEN SMEARING AND LOCAL MASS DISTRIBUTION

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In previous work by the Lund Nuclear Microprobe Group, the possibility to study the ink-paper interaction on newsprint has been demonstrated. In this work the process of smearing has been examined and especially the time evolution of the process. When smearing occurs, the print does not stay in place on the printed paper but colours other parts of the paper or the printing press and destroys the final product.
To study the re-distribution of ink, cyan ink with Cu in the colour pigment has been used. Non-printed paper has been pressed against paper, 1 and 5 seconds after the printing. The micro-distribution of ink on the non-printed papers as well as on the printed paper has then been examined. Basis weight was measured with the off-axis STIM technique and this data was correlated with the data from the print.
In this paper the effect of time on the smearing process and the non-uniformity of smearing, coupled to the paper structure, will be discussed.

AA-P4

METALLURGICAL ASPECTS OF GOLD AND SILVER COINS STUDIED USING MICROPIXE

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Ancient materials are often inhomogeneous on the scale of 10 microns or less : remains of imperfect smelting, segregated phases in alloys, inclusions. These inhomogeneities often

make it difficult to interpret the compositional results but, manytimes, could furnish solid arguments for provenance studies. So, the presence of platinum elements (Pt, Ir, Rh, Os, Pd) constitutes a fingerprint for each gold mine. Due to the primitive gold metallurgy, Pt elements are presented as inclusions (microns up to tens of microns diameter) in ancient gold objects. Using the Rossendorf Nuclear Microprobe installed at the 3 MV Tandetron, in the frame of the Fifth Framework Programme for Scientific Co-operation of the European Union, action "Access to Large Scale Facilities", an experiment for investigation of the distribution and the homogeneity of these inclusions in ancient gold matrices is in progress. As concerning silver coins, using the same Nuclear Microprobe facility, another experiment are also in progress : a completion of an investigation using a normal 3 MeV proton beam started in the frame of COST G1 Action on archaeometry at LARN Namur, Belgium on the minor elements (Cu, Pb, Au) in-depth profiling in ancient coins (wearing and corrosion effects) in relation to the relevance of surface informtion for real bulk composition. We use the microbeam to analyze the coins on surface and in bulk (after polishing) to investigate the effects of the different behavior at corrosion of each element and their microstructural affinities in the alloy's structure. The analytical results can provide a possible classification of the coins taking into account the relative concentrations of Cu – Pb – Au and the absolute concentration of Au.

AA-P5

IRIDESCENT ART NOUVEAU GLASS – USING ION BEAM ANALYSIS FOR THE CHARACTERISATION OF THE IRIDESCENT LAYERS

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Proton Induced X-ray Emission (PIXE), Proton Induced γ-ray Emission (PIGE) and Rutherford Backscattering Spectroscopy (RBS) were used for the characterisation of the thin iridescent layers on Art Nouveau glass artifacts. The investigations were carried out on glass fragments of the manufactures Tiffany/USA, Loetz/Austria and some modern glass samples made by Jack Ink/USA. The external proton beam of the Research Centre Rossendorf enables such measurements in a non-destructive and simultaneous way. An energy of 4 MeV was used therefore. Previous investigations carried out by SEM/EDX on cross-sectioned samples have shown that the iridescent layer has been directly applied to the bulk glass (lead containing glass) of Tiffany objects, whereas an additional layer of lead glass was found on the bulk material (potash–lime–silica–glass) of Loetz and modern artefacts.

Using the ratio of the Si-K/Si-γ - radiation it could be proofed that a thin layer containing Sn is present on the glass surface of all glass fragments. The elemental composition of the surface layer was obtained by PIXE and compared to the results determined at the cleavage. The results of the PIXE measurements (elemental composition) were used for simulation of the RBS-spectra. A thickness of the iridescent layer between 20 and 200-300 nm was obtained by means of RUMP®. Additionally, an interface (transition layer) could be determined by the RBS simulations. From the total amount of Sn obtained from the PIXE measurements approximately 80% Sn is presented in this transition layer and only 10-20% Sn as Sn-oxide is in the outermost surface layer.

AA-P6

IN SITU RBS STUDY OF THE KINETICS OF GALENA THERMAL OXIDATION BY MEANS OF ^{4}He EXTERNAL MICRO-BEAM

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Galena (lead sulphide PbS) is a component of the eye shadows (kohl) used since ancient Egypt. Numerous traditional recipes in North Africa still deal with the processing of this mineral before applying it around the eyes. In particular, the Tunisian tradition stresses the usefulness of a heat treatment of the mineral lump wrapped in a piece of textile: « have the piece of kohl dried directly on live charcoal, after having wrapped it in a dark blue rag saturated with olive oil ». Experiments performed on cleaved cm-sized crystals of galena (PbS) show that heating in air during a few hours at temperatures varying from 400 to 600°C induces iridescence (1). This feature seems to be sought in North Africa to produce coloured eye make-up.

We have built a furnace with small dimensions to study in situ the oxidation of a mm-sized galena crystal. Experiments can be performed up to 600°C and the thickness of the oxidised layer is measured every 2 minutes by means of the lead signal in the RBS spectrum, using a 3MeV $^4He^{2+}$ external focused beam (about 100µm). We infer the diffusion mechanism from the growth of a phase, which is more or less stable when increasing the temperature. This probing beam allows one to characterise layers of thickness extending from about 100 nm to 2000 nm.

This technique can be used to investigate the kinetics of transformation in air which involves complex diffusion mechanisms associated with the formation of reactive phases at the interface between the modified layer and the pristine material. The archaeological interest of this work is to show a likely thermal treatment of material in Antiquity for aesthetic purpose and to precise the understanding of ancient metallurgical processes.

(1) B. Ponsot, J. Salomon, P. Walter, NIM, B136-138, pp.1074-1079.

AA-P7

PIXE ELEMENTAL MICRO-MAPPING OF AN ANCIENT EGYPTIAN PAPYRUS.

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This paper reports a study of colours and inks of an ancient Egyptian papyrus using PIXE and external proton micro-beam. Representing the Book of the Dead, this papyrus is dated from the 19th-20th dynasties, New Kingdom (c.1295-1069 B.C.), and belongs to the Kulturen museum in Lund, Sweden. Elemental maps were obtained by moving the papyrus under a fixed focused external beam using a motorised support. The maps were compared to photographic pictures taken in visible and U.V. light. Inks used in the hieroglyph text appeared to be based on carbon (black) and ochre (red). Coloured drawings illustrating the text showed a wider palette: iron oxide (hematite), ochre, orpiment, Egyptian blue, verdigris. Most intriguing was the observation in several parts of the drawing of a whitish pigment containing strontium. This could possibly be crushed Celestine, a pale blue mineral with $SrSO_4$ formula occurring in Egypt. Finally, fine powder and coarse grains of arsenic oxide were observed, probably used after excavation as an early preservation treatment against insect attacks.

AA-P8

CHARACTERIZATION OF GLAZE COLORANTS USED AT THE PALACE OF RAMSES III AT MEDINET HABU, IN MIDDLE EGYPT

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In the mid-12th century B.C. the pharaoh Ramses III added to the already extensive architecture of the area around Thebes (modern Luxor) a remarkable complex of palaces and Temples, the walls and pylons of which were decorated with a variety of carvings and inlays. Among the most unusual of the decorative features were a group of large plaques made of glazed inlays and tiles, some of them depicting foreign peoples who were under Egyptian rule at the time. We have studied the recipes for the coloration of these plaques and have identified an extensive, but previously unreported use of lead glazes that would give certain surfaces a high luster. These findings will be discussed both in technical terms (with particular reference to the leaching of lead from the glaze's surface), and with cross-reference to our previous work on Egyptian glasses and glazes which date to the preceding two centuries.

AA-P9

PIXE ANALYSIS OF BRONZE AGE WHITE PAINT ON MINOAN POTTERY

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In order to elementally analyse the white paint from Bronze Age Minoan pottery, sherds were sampled from four archaeological sites located on the central to eastern half of the island of Crete: Knossos, Kommos, Mochlos and Palaikastro. PIXE analysis was performed at the PIXE facility of the University of Delaware. Results show a higher amount of Mg in the white paint on the Kommos sherds. Kommos is located in the south central part of Crete and could have been involved in a larger southern center of pottery production. Knossos pottery with a high Mg content is typically southern in style and may have been an import to the north central site of Knossos. The Mg content of the white paint could be an indication of the pottery's origin and, therefore, would help in determining aspects of trade relations.

Session ALT : Alternative Techniques

ALT-P1

SYNCHROTRON INDUCED X-RAY MICROFLUORESCENCE ON SINGLE-CELLS

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Recent improvements in Synchrotron X-ray sources (third generation) and in X-ray focusing elements have been realized. This result in delivering highly collimated quasi-monochromatic

X-ray beam with tunable energy and highly focused beam with a (sub) micrometer diameter. Elemental mapping of single-cells was obtained for the first time by synchrotron induced X-ray fluorescence in the hard X-ray range with high spatial resolution. Pink beam and compound refractive lenses were used resulting in an incident flux of around 10^{12} photon/s and a micrometer beam size. Taking into account the properties of synchrotron radiation, experiment confirms that in our conditions, high energy, high intensity x-rays are well suited for microanalyses of sensitive biological specimen (freeze-dried cells). Results show that the synchrotron microprobe set-up at ESRF allows high accuracy in trace element measurements for cell treated with pharmacological doses of anticancer drug. Micro-SXRF on single-cells is at its starting point and is expected to become a powerful non-destructive method, highly complementary to Particle Induced X-ray Emission (PIXE) and other types of micro-analytical methods. Moreover, SXRF will be shortly improved toward fully quantitative analysis and will be used in conjunction with X-ray absorption spectroscopy (micro-XANES) and phase-contrast x-ray micro-imaging. Finally, microanalysis of living cells is an exciting perspective that could be reached using micro-SXRF.

ALT-P2

USE OF THE PHOTON AND NUCLEAR MICROPROBES TO THE CHARACTERISATION OF AUSTENITIC STEEL CORROSION PRODUCTS FORMED IN IGCC ENVIRONMENTS

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The study of the corrosion degradation of metallic alloys used to build Integrated Gasification Combined Cycle (IGCC) power plants is of major interest. The metallic materials are in contact with a gas stream containing CO, CO_2, H_2S, SO_2 and H_2O; thus corrosion degradation is likely to occur, especially since the gas also contains fly ashes. For instance, vanadium, sodium and sulphur which are present in oil residue gasification ashes can contribute to the enhanced degradation of the materials. To understand the complex corrosion mechanisms that take place in such systems, the precise identification of the corrosion scales products is necessary. We will present the analytical part of the study of several Fe-Ni-Cr austenitic alloys treated in an environment representative of the IGCC recovery boiler in association with synthetic ashes containing vanadium. To complement classical characterisation methods, we perform PIXE and RBS analysis. In addition, we attempted to determine the valence state of the vanadium contained in the corrosion scale by performing micro X-ray Absorption Near Edge Structure (XANES) analysis at the V K-edge.

ALT-P3

INVESTIGATION OF MANGANESE IN SALT- AND FRESH-WATER PEARLS

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In the past the origin of a pearl was a matter of speculation. Even to day we do not know the exact reason of formation of each and every individual pearl. However modern techniques enable us today to answer various questions regarding the formation of a pearl, an unique product of biomineralization. The concentration of certain chemical elements in pearls from

fresh-water mollusks are higher than those from salt-water. Particularly the manganese content varies from species to species even fresh-water and salt-water pearls. It is found that the manganese concentration is generally higher in fresh-water pearls compared to salt-water paerls. The content of manganese in shells and in soft parts, e.g. in the mantle of mollusks is influenced by environmental factors like temperature and salinity of water.
We present results from analyzing pearls from fresh- and salt-water environments with proton induced x-ray emission (PIXE), cathodoluminescence (CL)-spectroscopy/-microscopy and electron spin resonance (ESR)-spectroscopy. We found that the concentric structure of fresh-water pearls is often a mixture of different carbonates containing calcite, Mg-calcite and/or aragonite phases. Using the combination of PIXE and CL-spectroscopy we evaluate the Mn^{2+} concentration and the relative distribution coefficient in each mineral phase. The ESR-analyses were performed to investigate paramagnetic defects caused by radiation experiments and by the charge transformation of Mn^{2+} to Mn^{4+} in the calcite, Mg-calcite and aragonite phases.

ALT-P4

SYNCHROTRON X-RAY MICRO-BEAM STUDIES OF ANCIENT EGYPTIAN MAKE-UP

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The extensive use of green, white and black make-up has been known since the earliest periods of Egyptian history for their aesthetic and therapeutic purposes. In previous works, we have studied the inorganic and organic contents of the cosmetics dated between 2000 and 1200 BC, kept in their original containers and preserved in the Louvre Museum. Two well-known natural lead-based compounds were identified: crushed ore of galena (PbS) and cerussite ($PbCO_3$). In addition, our analyses reveal two unexpected synthetic products: laurionite (PbOHCl) and phosgenite ($Pb_2Cl_2CO_3$).
The natural ores of galena and cerussite incorporated into the cosmetics originate from different mines, in particular from the Pb-Zn ores or from "the Punt country" (Yemen, Somalia or Ethiopia). From the identity and the respective abundance of the trace elements present (Fe, Ni, Cu, Zn, Ag, Sb), it is possible to classify the samples according to their extraction provenance. For synthetic compounds, the trace elements are linked to the minerals used during the preparation process (natron, sea or rock salt,...). We show that the natural galena and the synthesised phosgenite exhibit different impurity patterns.
The powdered cosmetics made of isolated grains are spread on kapton foils and are examined by optical microscopy to identify/locate the minerals prior to X-ray analysis. Then the focused X-ray beam (2x5 μm^2) is successively tuned at 11 keV, below the L_{III} absorption edge of Pb, and 31.8 keV, above the K absorption edge of Sb. The impurity signal integrated over each single grain is detected against the X-ray micro-diffraction pattern collected in transmission with a bi-dimensional detector. Furthermore, for galena grains rich in Zn, the XANES signal at the K absorption edge of Zn can show the immediate nearest-neighbour environment (ZnS nanocrystals or Zn^{2+} substitution).

In combining micro-diffraction, micro-fluorescence and micro-XANES techniques, we emphasise the capability of synchrotron micro-beam to track minerals of great archaeological interest and to identify the provenance of manufactured materials in Antiquity.

ALT-P5

X-RAY FLUORESCENCE MICRO-TOMOGRAPHY OF AN INDIVIDUAL FLUID INCLUSION USING A THIRD GENERATION SYNCHROTRON LIGHT SOURCE

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The aim of this study was to establish the feasibility of obtaining 3D elemental images of fluid inclusions, which consist in complex, irregular and non-homogeneous objects trapped in a host mineral. These experiments are based on the fluorescence micro-tomography technique, which combines fluorescence measurements and 2D tomographic reconstruction algorithms. They were performed on beamline μFID22 of the European Synchrotron Radiation Facility (Grenoble, France), using a 15 keV incident photon beam, tuned by means of a fixed exit monochromator (Si [111] plane) and focused by a set of 50 parabolic Al Compound Refractive Lenses to an incident flux of about 10^{10} ph/s. To monitor the incident and the transmitted beam, flux-integrating detectors (PIN-diodes) were located behind the lenses and the sample, respectively. The fluorescence measurements were carried out with a Si(Li) detector of 150 eV resolution at 5.9 keV, set at 90° to the incident beam. Multiphase aqueous fluid inclusions saturated with respect to NaCl, Zn, Fe, Cu, were synthesized in fractured quartz crystal within gold capsule at high temperature and pressure (700°C and 0.7 GPa). A single fluid inclusion was isolated in a tiny quartz parallelepiped, polished on all its faces. The quartz chip was mounted on a rigid pyrex micro-capillary and positioned on a goniohead that suspended the sample in air, in front of the focused beam. On the basis of the parallel collection technique, the sample was successively scanned sideways over an area of few hundred μm, by step of 3 μm and with acquisition time of 1 s/point, and rotated by an angle of 2°. The 2D projection images, which consist in relative elemental concentration regions, provide the size and the location of the fluid inclusion as well as the constituent phases (i.e., vapor, liquid and solids). By iterating the procedure, in the other dimension, along an area spanning the inclusion, full 3D elemental images can be obtained

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