Novel applications in ion microbeam analysis

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Motivation

A nuclear microprobe opens up new research areas
at Demokritos Institute

Working Document on

THE RESEARCH INFRASTRUCTURES IN FP7

"…best research infrastructures existing in Europe.."
"…forefront of the advancement of research.."
"Centre of excellence…"
"Knowledge generation and, by implication, innovation…"
"…help industry … knowledge and technological know how"

What is your VISION?

Outline

Introduction

- HAS-ATOMKI Accelerator Centre /Ion Beam Laboratory
- Overview of the nuclear microprobe techniques

Research with microbeams in HAS-ATOMKI

- Environmental research
- Biomedical applications
- Archaeology
- Materials science
- Modification of materials

Nuclear microprobe laboratories around the world

HAS-ATOMKI

Institute of Nuclear Research
of the Hungarian Academy of Sciences
ATOMKI

- Staff: 198
- Main research fields:
  - Theoretical nuclear physics
  - Atomic physics
  - Materials science
  - Earth science
  - Environmental research
  - Nuclear techniques
- Associated member of the University of Debrecen

Accelerator Centre

- 5MV **Van de Graaff** accelerator
  $^{1}H^{+}$, $^{2}H^{+}$, He^{+}, C^{+}, N^{+} N_2^{+}
  Mainly used for IBA, Nuclear Physics & Astrophysics
- 100-1000kV **Van de Graaff** accelerator
  $^{1}H^{+}$, $^{2}H^{+}$, $^{4}He^{+}$, (C^{+})
  used for investigation of atomic collisions
- **ECR Ion Source**
  Electromagnetic wave frequency 14.5 GHz,
  Extraction (acceleration) voltage 2...30 kV;
  mainly used for plasma diagnostics and
  investigation of ion-surface interactions
- K=20MeV Compact **Cyclotron**
  $^{1}H^{+}$, $^{2}H^{+}$, $^{3}He^{2+}$, $^{4}He^{2+}$
  Mainly used for isotope production for medical applications & Nuclear Physics

Section of Ion Beam Physics

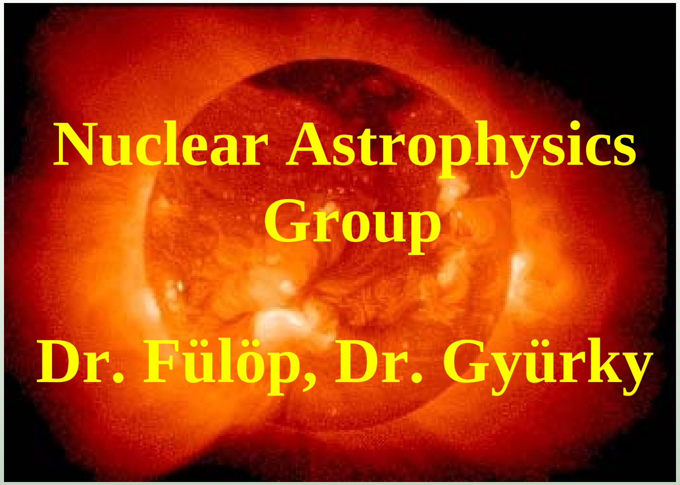
Beam lines on 5MV VdG

Millibeam lines:

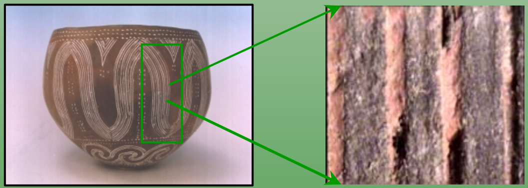
- Aerosol science (PIXE)
- Nuclear Physics & Astrophysics
- Electron spectrometer

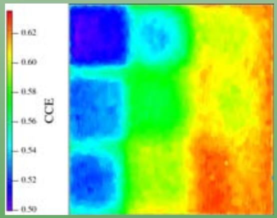
Nuclear microprobe (1995)

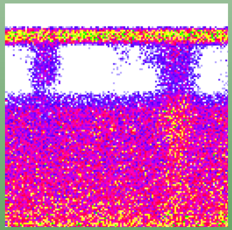
- RBS, PIXE, PIGE, DIGE, IBIC, STIM, PBM

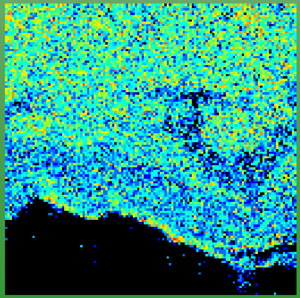


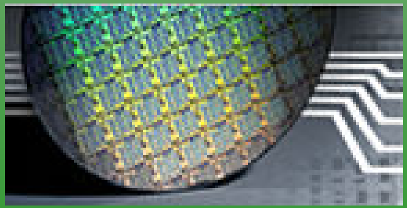
Ion Beam Analysis Group









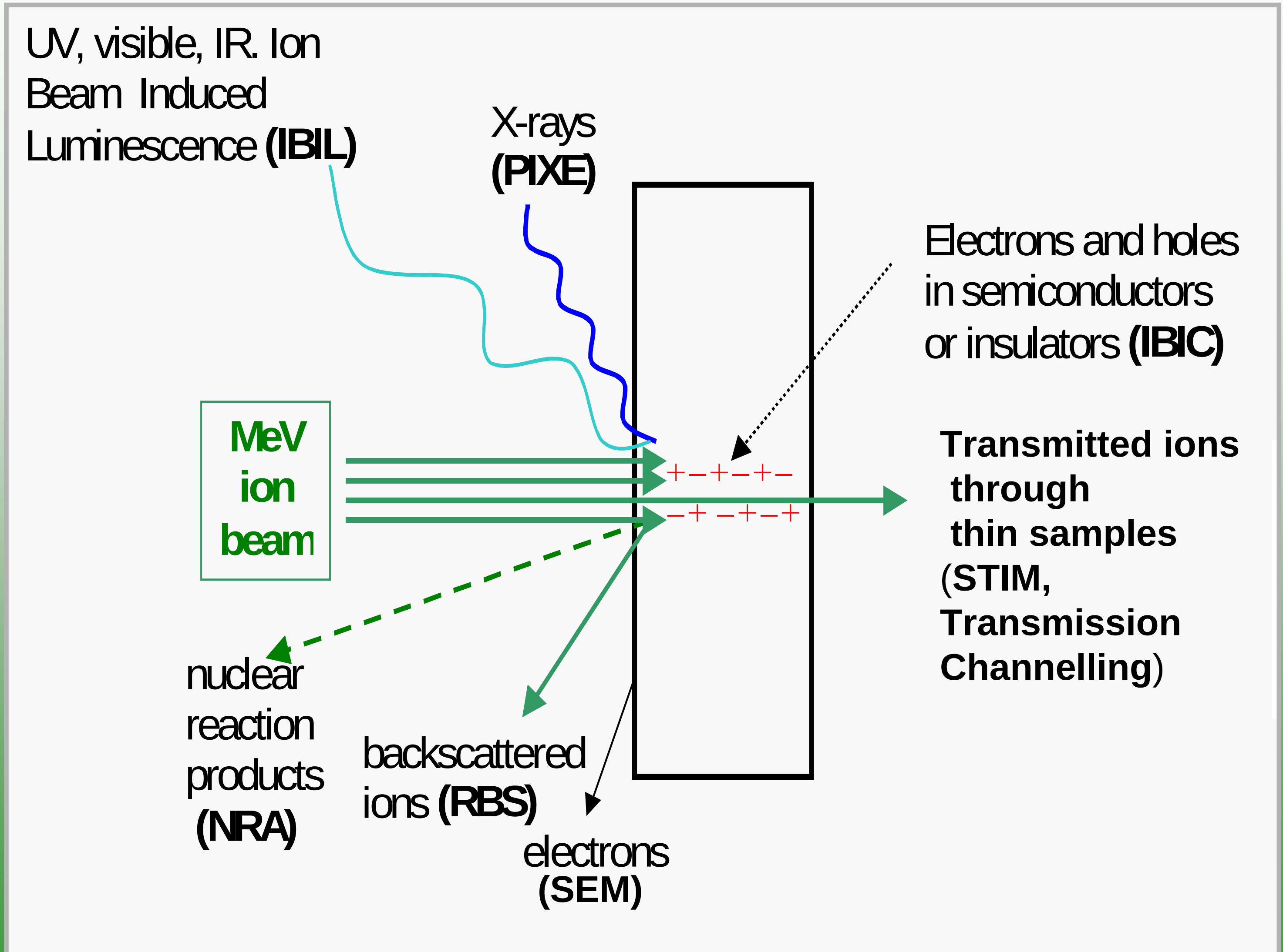


Definition

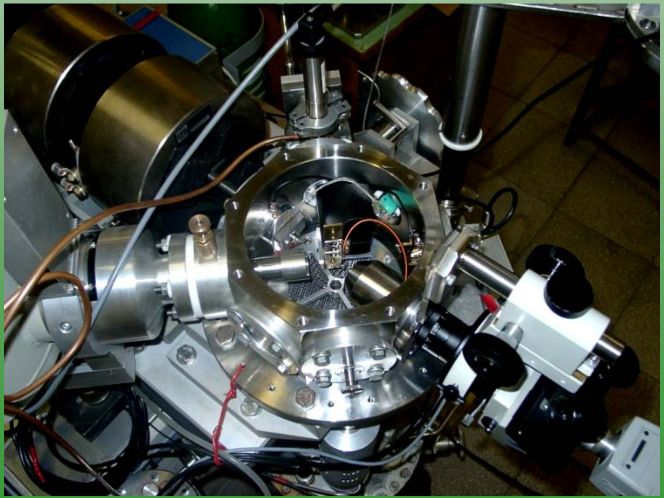
What is a nuclear microprobe?

General purpose, multidisciplinary instrument,
where high energy (MeV) ions are focussed down
to a micron size beam and scanned over the sample
for getting spectroscopic and imaging data.

Ion-solid interactions



Debrecen Scanning Nuclear Microprobe



- Oxford Microbeams Ltd.
- Built on the 0^{o} beamline of the 5 MV Van de Graaff accelerator
- Beams: H^+, D^+, $^4He^+$
- Energy range: 0.6-3.5 MeV
- Beam size: 1 μm x 1 μm
- IBA techniques: PIXE, PIGE, DIGE, RBS, STIM, IBIC
- Applications: archaeology, geology, biology, environmental science materials science, modification of materials, PBM

Applications of a Nuclear Microprobe

- **Imaging of elemental distribution on micro-scale**
  Elemental maps of minute samples as for e.g. minerals, biological cells, aerosol particles, etc.

- **Micro-characterisation**
  Determination of elemental concentrations in micro-particles, composition and thicknesses of thin metallic films, etc.

- **Micro-charge deposition** (Ion Beam Induced Current -IBIC, single event upset)

- **Micro-irradiation** (e.g. single cell irradiation),

- **Micro-damage** (testing reliability of electronic devices),

- **Micro-machining** (producing miniature devices), etc.

Analytical techniques

"Traditional" techniques (~pA): PIXE, RBS, NRA, PIGE

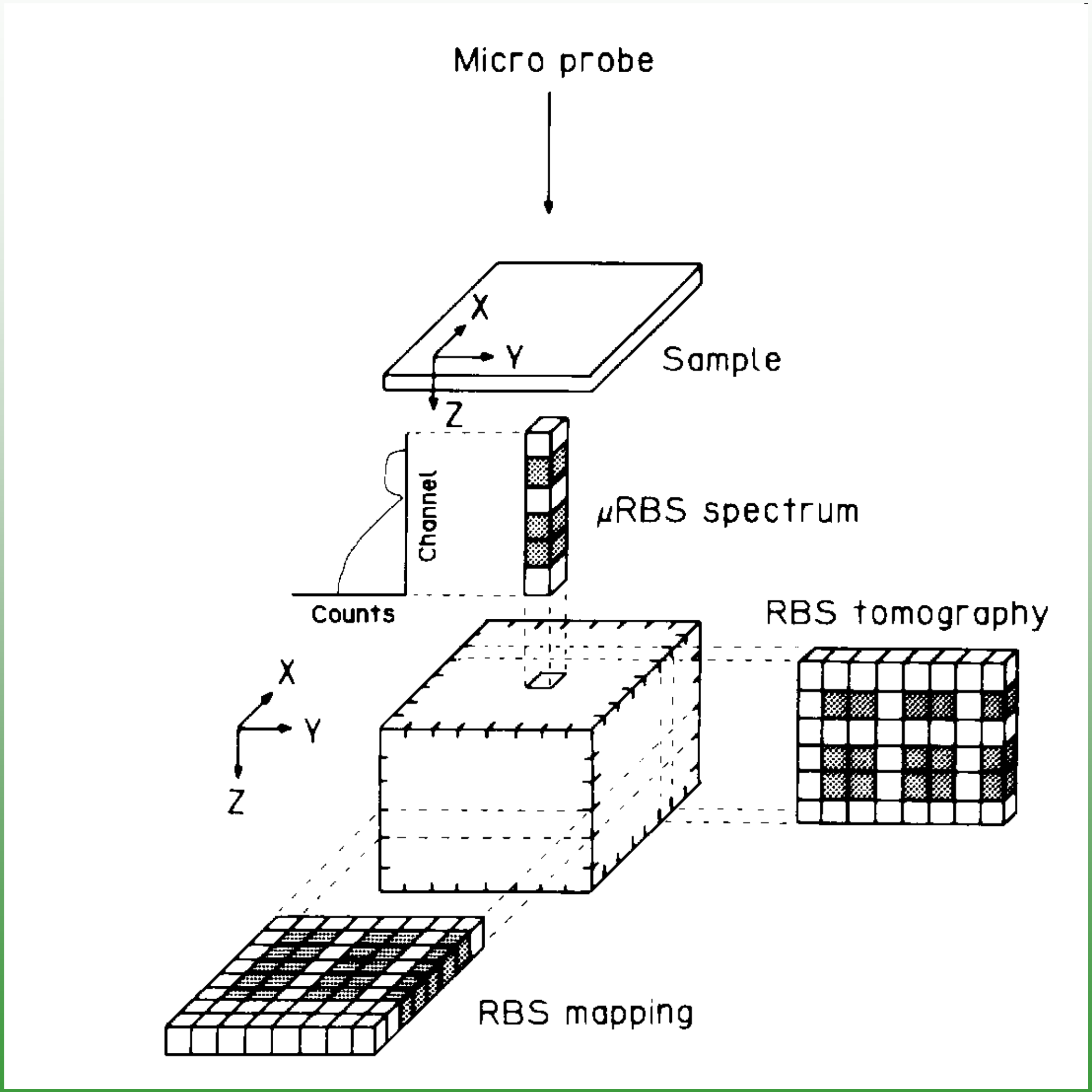
Low-current techniques (~fA): IBIC, IBIL, STIM

Data acquisition

- Spectra
- Imaging
  - Elemental maps
  - Tomographic images
  - Pulse height maps

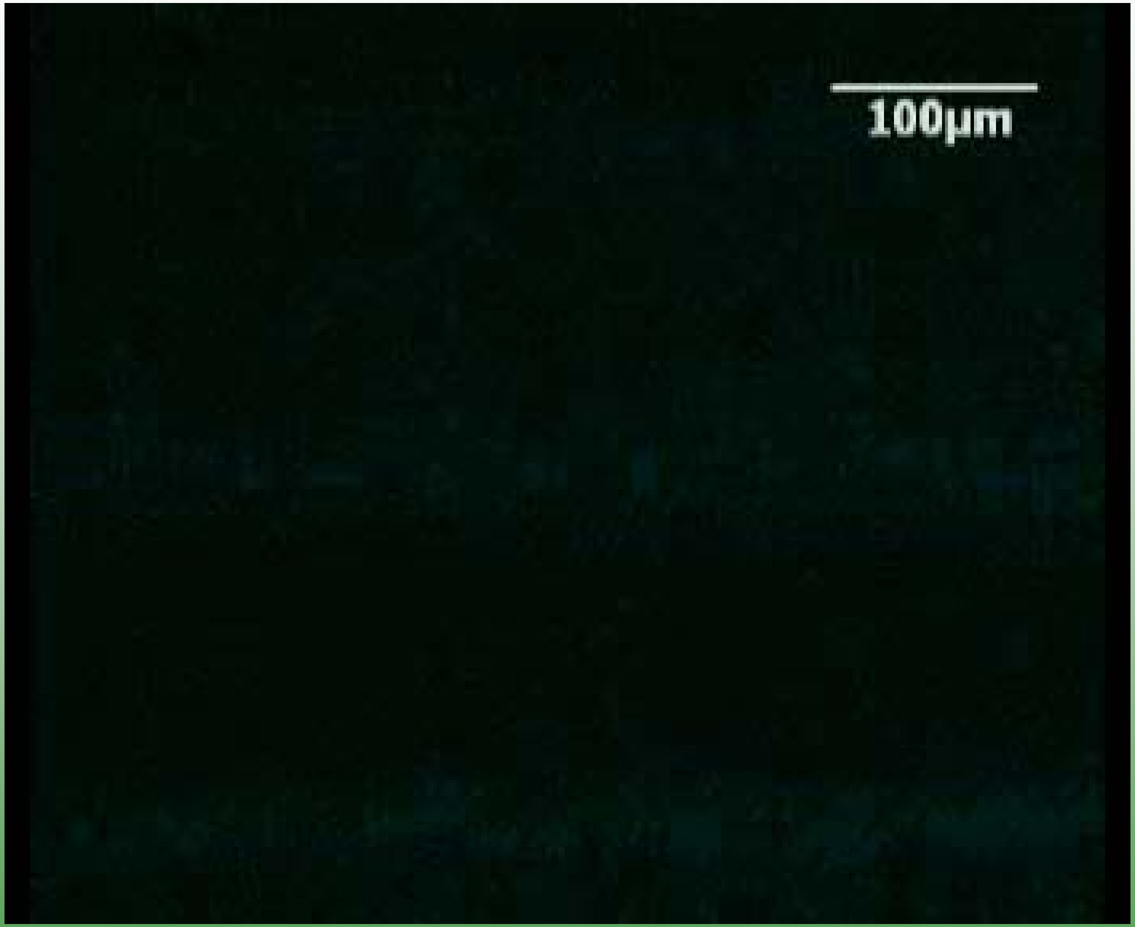
LISTMODE

Schematic diagram of data block for RBS mapping and tomography



M. Takai, et al., Nucl. Instr. and Meth. in Phys. Res. B 64 (1992) 277.

HAS-ATOMKI LOGO

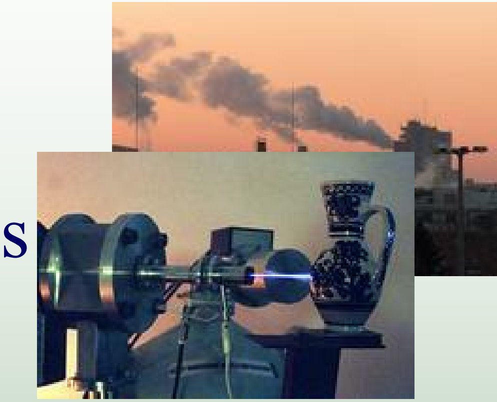


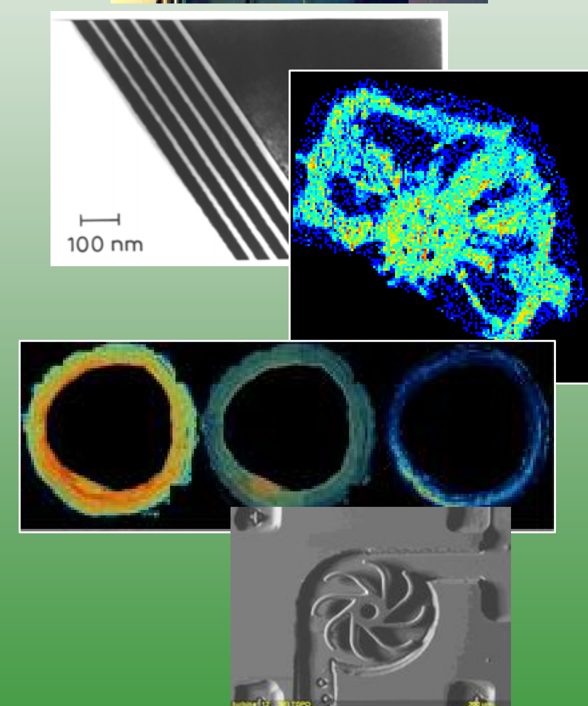
The beam is scanned on a quartz.
The subsequent images are integrated.

Courtesy to I. Rajta& M. Novak, HAS-ATOMKI

IBA application areas

- Environmental: atmospheric aerosols
- Cultural heritage: archaeological samples
- Materials Science: thin films, multilayers, detector performance
- Biological: roots of plants
- Medical: blood vessels, skin
- Proton Beam Micromachining





Environmental-Atmospheric aerosols

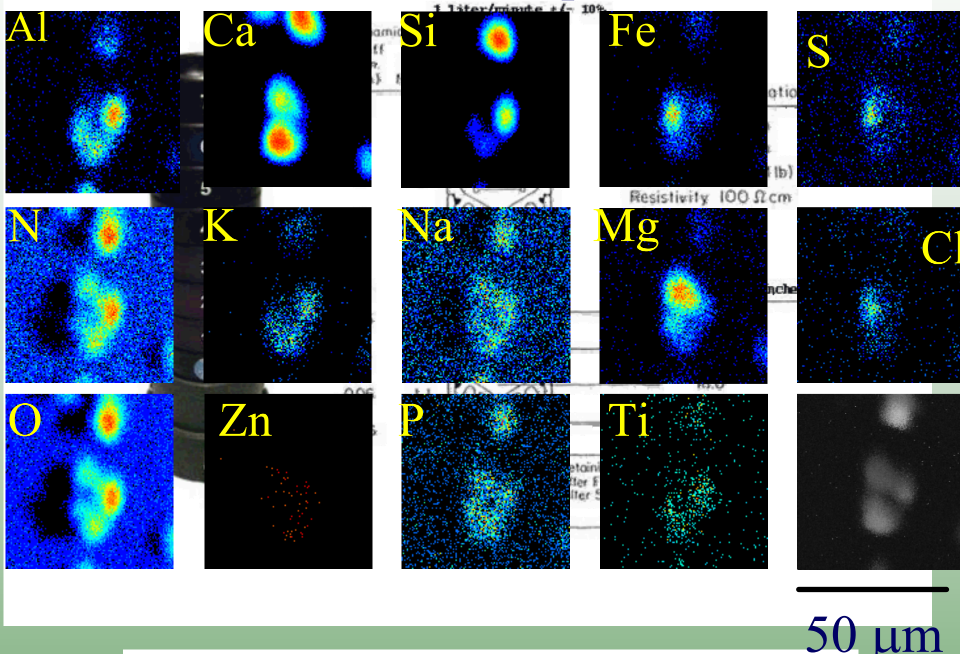
Rural sampling site for background values in Hortobágy

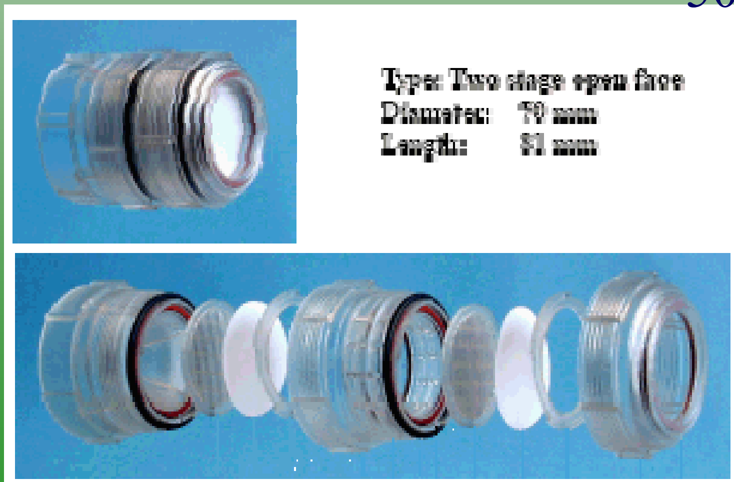
- Urban and rural aerosol in Eastern Hungary
- EU regulation
- Origin of aerosols
- Stochastic lung models (deposition of different sized aerosol particles in healthy and diseased bronchial)

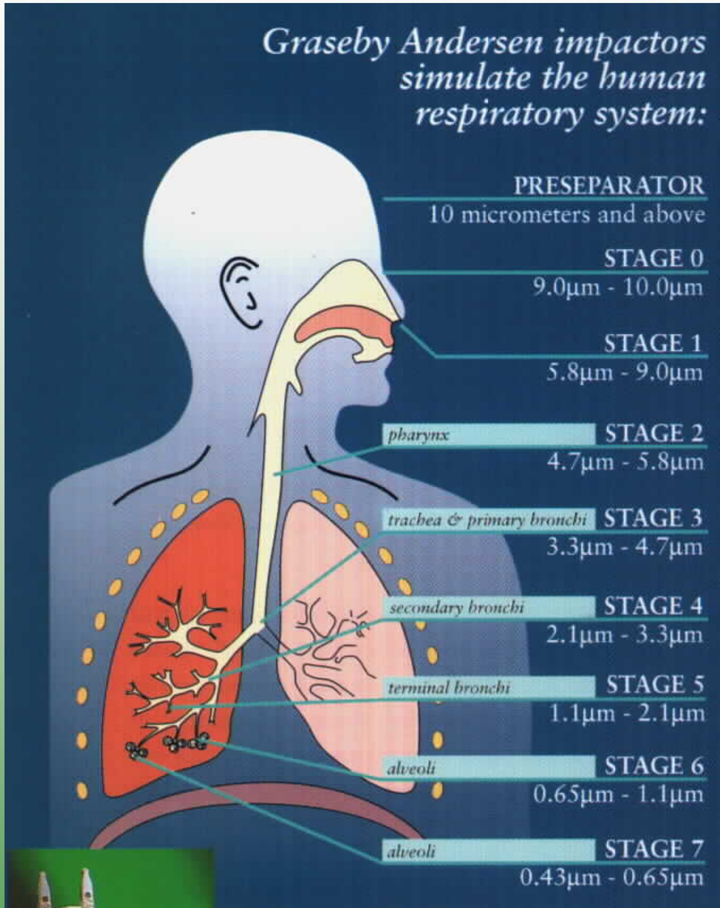
Environmental- Individual aerosols

µPIXE, STIM, µRBS

Individual aerosol particles



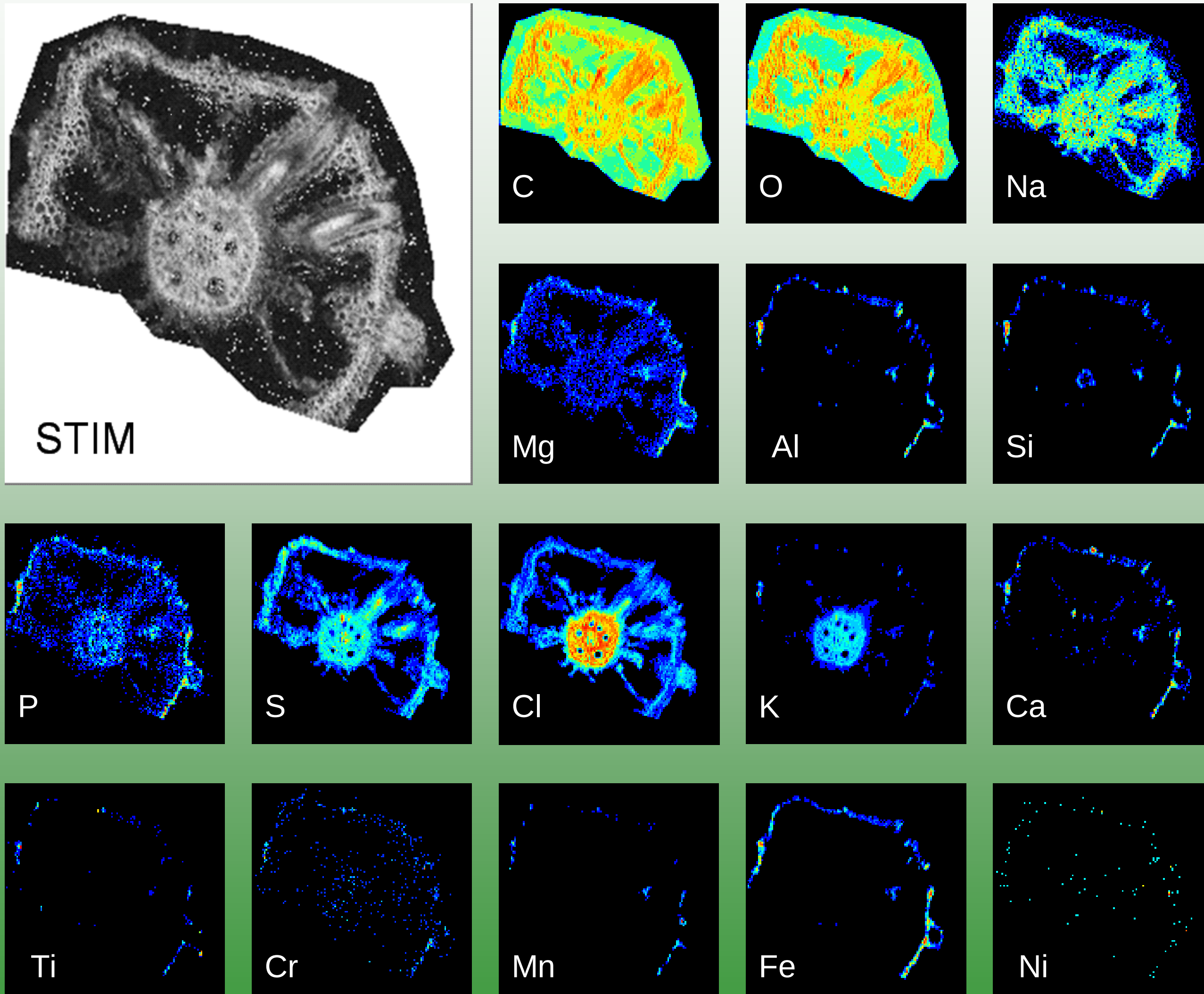




The health effects of the inhaled particles may strongly depend on the location of deposition within the lung.

Environment- Metal uptake of plants

μPIXE, STIM



Identified tissues:

- Rhisodermis
- Parenchyma
- Vascular cilinder

The uptake of toxic element is associated with the presence of iron plaques.

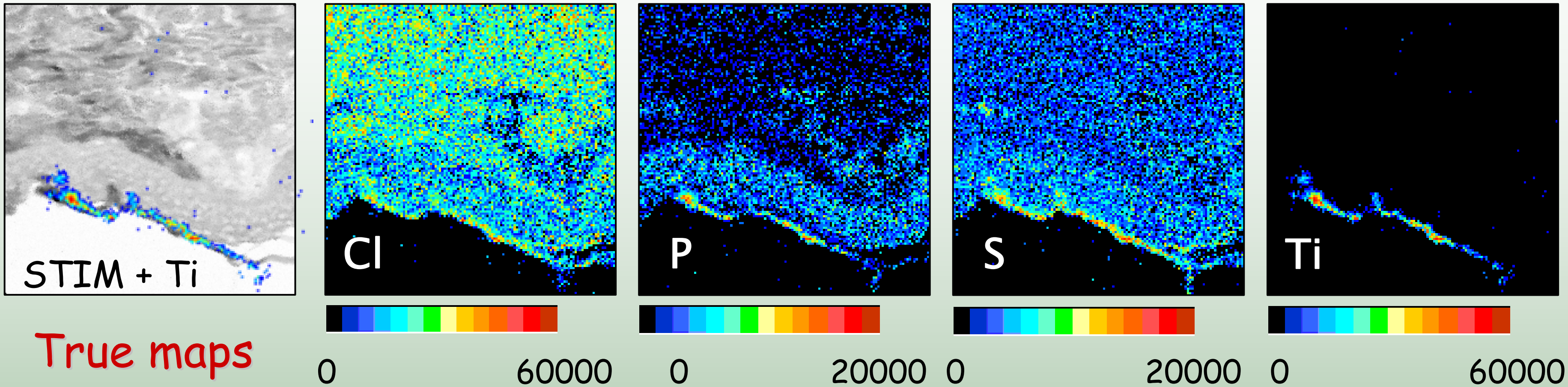
These elements remain trapped in the root.

These plants can be used for monitoring.

Biomedical applications

μPIXE, STIM, μRBS

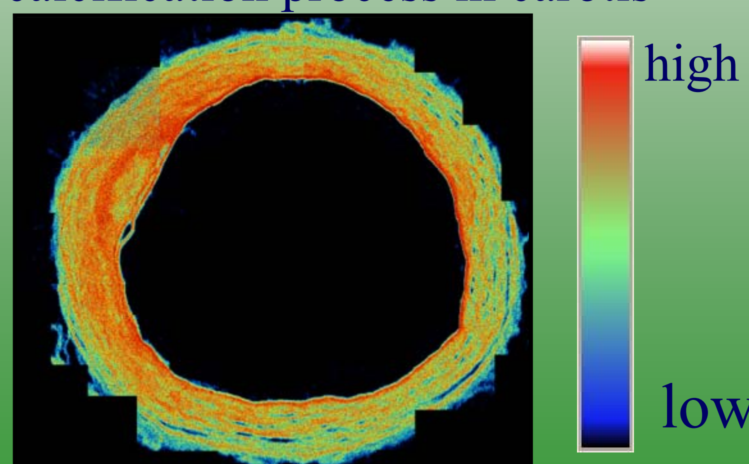
TiO_2 nanoparticle penetration in the epidermis of human skin xenografts



True maps

Study of calcification process in carotis

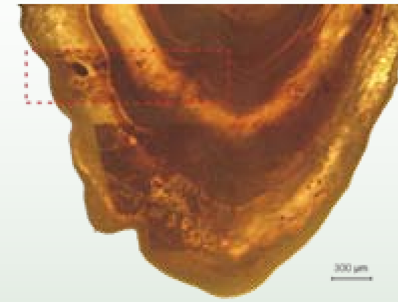
Cross sectional distribution of Ca concentration.

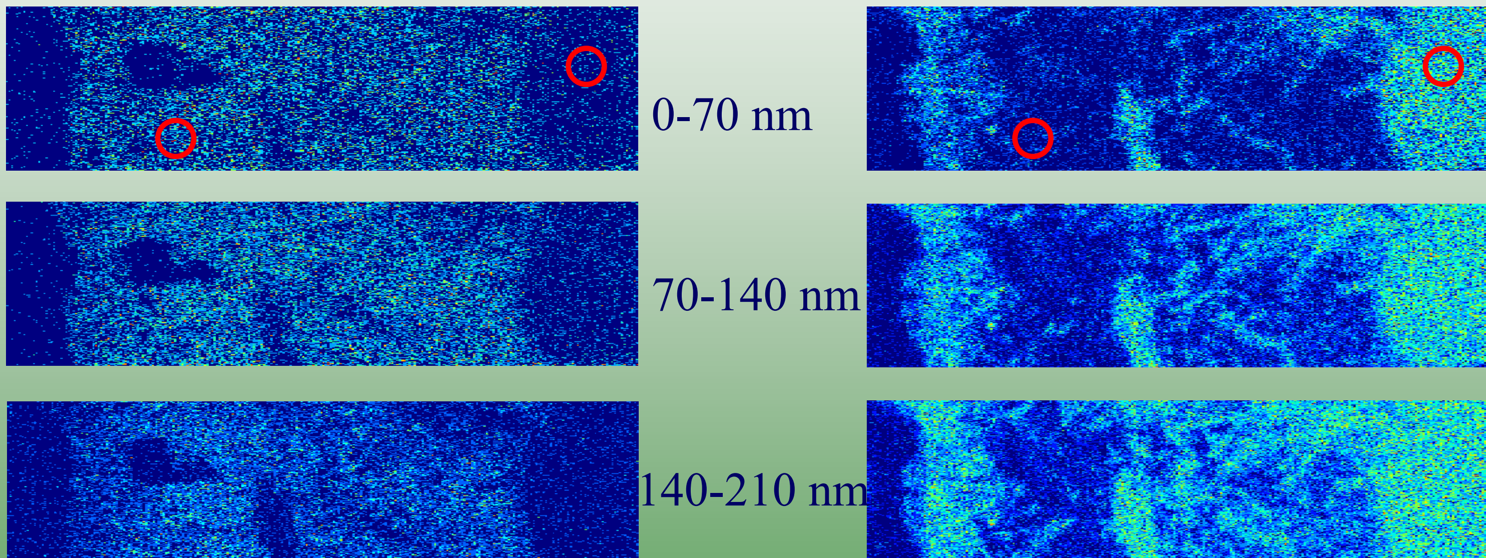


Number of concatenated bitmaps: 21.
∅ ~ 7 mm.

Complex investigation of fish otoliths

μPIXE, μRBS, μERDA





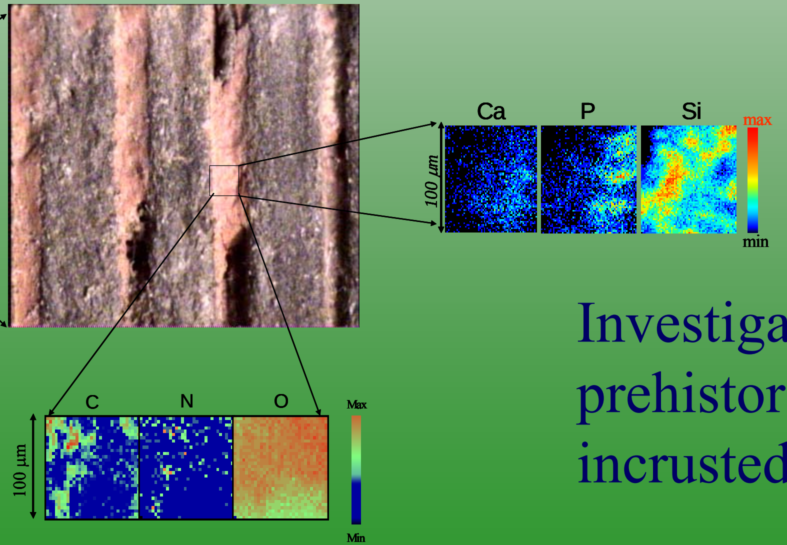
H maps by micro-ERDA

Ca maps analysed by micro-RBS

Cultural Heritage

Analysis of classical ring-stones from the 17-18th century and their imitations

Characterisation of Dyrrhachium silver coins from 68-43 years BC
→ degradation of the of Ag content
→ chronological classification



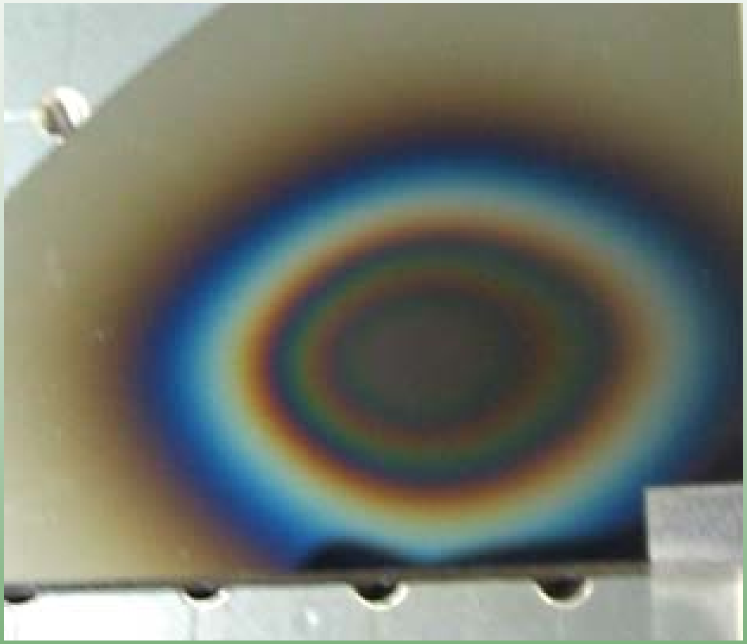
Ornamenting white substance does contain bone grit?

Investigation of prehistoric incrusted pottery

High lateral resolution mapping of the B/C ratio in a boron carbide film formed by femtosecond pulsed laser deposition

Boron carbide, B_4C

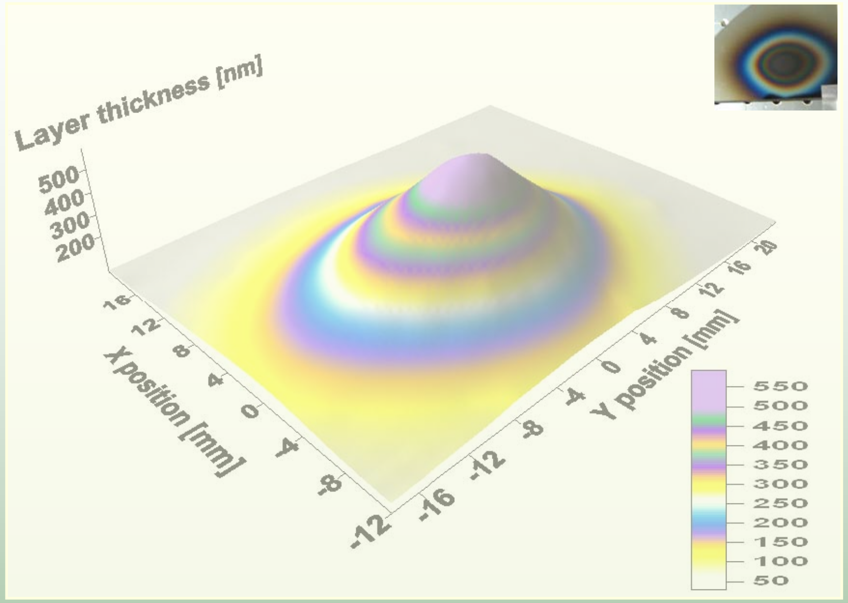
- Micro-drives used in consumer electronics require an increased areal storage density.
- Hard disk industry is aiming at 1Tbit/inch^2.
- Protective overcoat for the head and the disk requires smooth, wear resistant, pinhole-free, thermally stable material for wear and corrosion protection at 1nm thickness.
- Currently used $CN_x \Rightarrow B_4C$ (and/or SiN_x ,$B_xC_yN_z$)



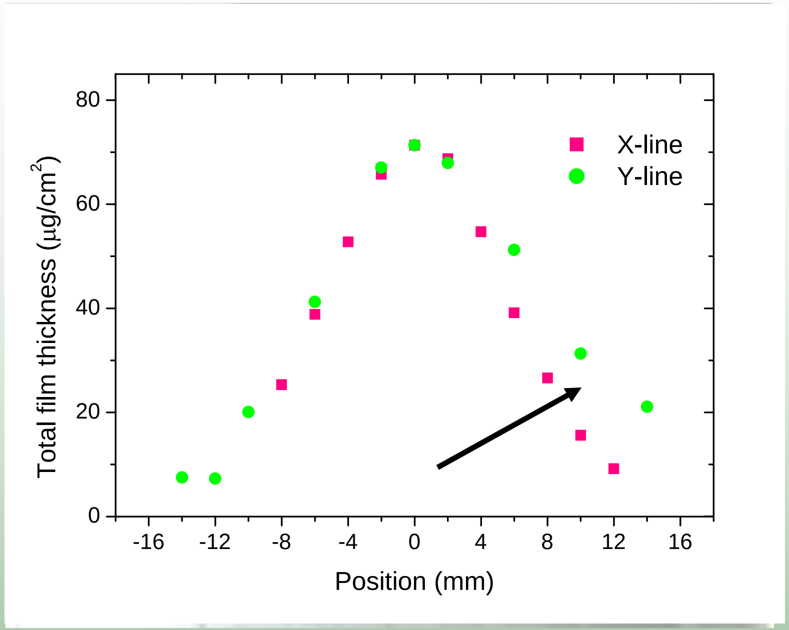
Deposit size: approx. 3 cm x 4cm
Please note the non-uniform film thickness

A. Simon et al. NIMB 249 (2006) 454.

Layer thickness distribution



Thickness distribution by **spectroscopic ellipsometry**

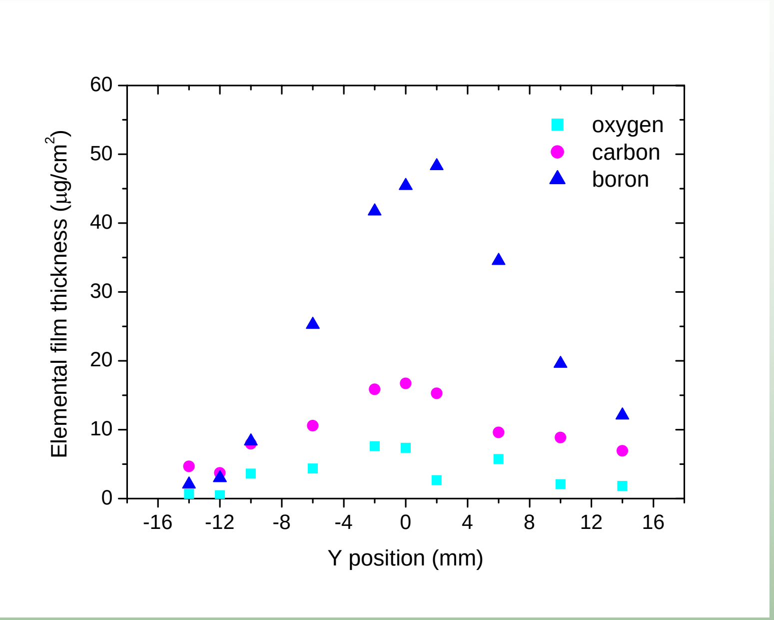


Thickness distribution by **RBS microbeam (2.5 MeV He^+)**

The results of the RBS analyses:

- the film thickness is not uniform;
- it has a maximum in the centre: d=71 μg/cm^2 (3736x10^{15}at/cm^2);
- a steep decrease is observed toward to the edges;
- minimum film thickness of d~9 μg/cm^2 (484x10^{15} at/cm^2).

Lateral elemental profiles of the boron carbide layer μRBS



- The elemental composition is indeed highly non-uniform.
- The B/C ratio has a maximum at the centre with elemental contents of
  - ▲ boron: 50±5 μg/cm^2) ;
  - ■ oxygen:8±3 μg/cm^2;
  - ● carbon: 16±2 μg/cm^2.
- The B/C~1 ratio measured at the edge of the deposit increases up to about 3 toward the centre (the target composition was B_4C).

Similar investigations of target uniformity with the Nuclear Astrophysics Group in order to increase the precision of their measurements.

Kiss G. Gy. [1 +] , Rauscher T. [4] , Gyürky Gy. [1 +] , Simon A. [1 +] , Fülöp Zs. [1 +] , Somorjai E. [1 +] : *Coulomb suppression of the stellar enhancement factor.* Physical Review Letters **101** (2008)19:1101(4)/

Rauscher T. [4] , Kiss G. Gy. [1 +] , Gyürky Gy. [1 +] , Simon A. [1 +] , Fülöp Zs. [1 +] , Somorjai E. [1 +] : *Suppression of the stellar enchancement factor and the reaction 85Rb(p,n)85Sr.* , Physical Review C **80** (2009)3:5801(12)

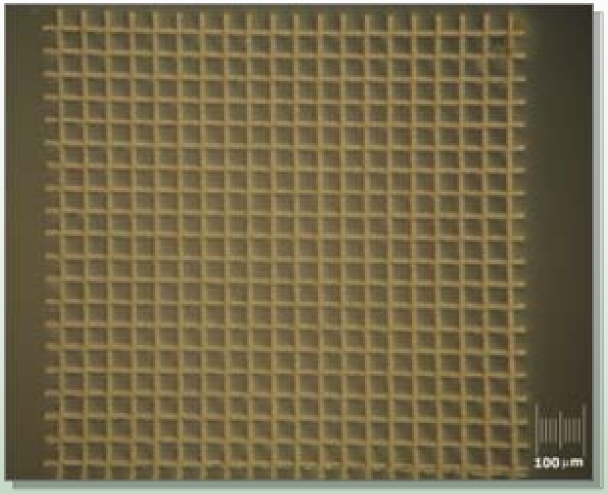
Yalcin C. [2 + 2] , Güray R. T. [4] , Özkan N. [4] , Kutlu S. [4] , Gyürky Gy. [1 +] , Farkas J. [1 +] , Kiss G. Gy. [1 +] , Fülöp Zs. [1 +] , Simon A. [1 +] , Somorjai E. [1 +] , Rauscher T. [4] : *Odd p isotope 113In: Measurement of alpha-induced reactions.* Physical Review C **79** (2009)5801(9)/

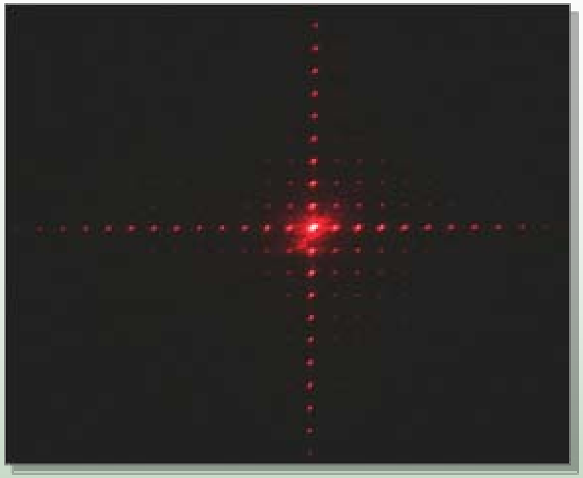
Proton Beam Micromachining

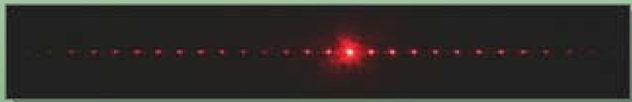
- Focused MeV energy ion beam is scanned over a suitable resist material and subsequently chemically developed
- Advantages of PBM:
  - Direct write 3D lithography method
  - Short irradiation time
  - Good depth of field: nearly 90° walls
  - Well defined depth due to ion ranges: buried microchannels
- Post lithography step: electroplating
  - Metal micro-molds and stamps

Microreactors, Micro-electrochemical cell, Microfluidic-reactor, micro-optical devices, Silicon: micro- turbine, pump, filters for medical research, etc.

PBM-Diffraction gratings





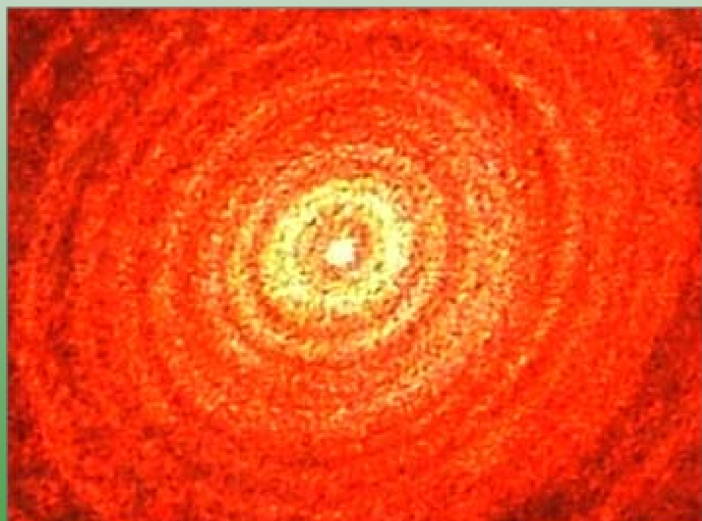


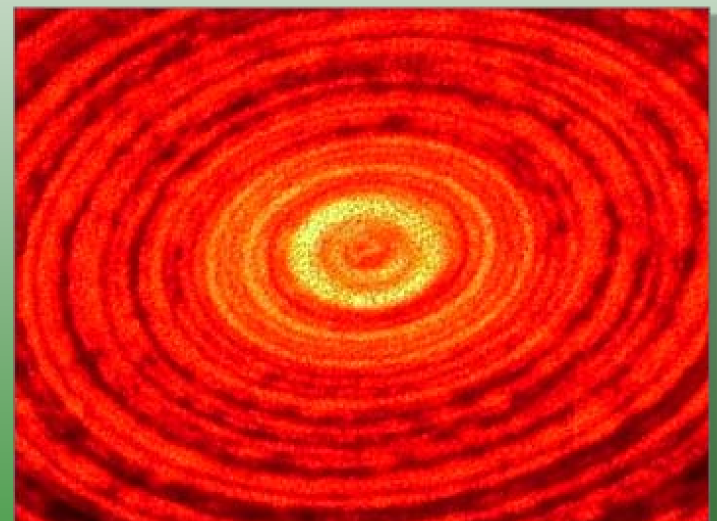
R. Huszank et al., Fabrication of optical devices in poly(dimethylsiloxane) by proton microbeam, Opt. Commun. (2009), doi:10.1016/j.optcom.2009.09.066

Top: 8 µm wide and 50 µm lattice constant two-dimensional
Bottom:15 µm wide 30 µm lattice constant one-dimensional grating
and their diffraction images

PBM-Fresnel zone plate

R. Huszank et al., Fabrication of optical devices in poly(dimethylsiloxane) by proton microbeam, Opt. Commun. (2009), doi:10.1016/j.optcom.2009.09.066



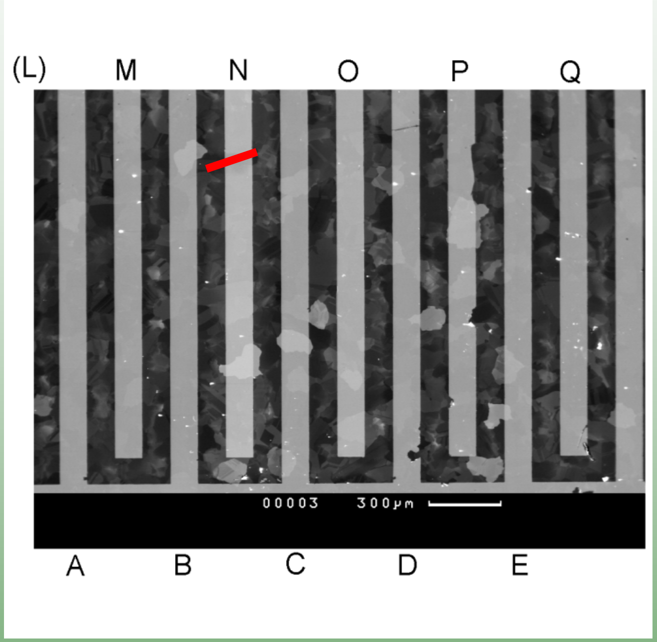


Top: Fresnel zone plate, 130 mm focal length with 2000 µm diameter convergent type
Bottom: diffraction images (left) at the focal point and (right) at 420 mm from the zone plate.

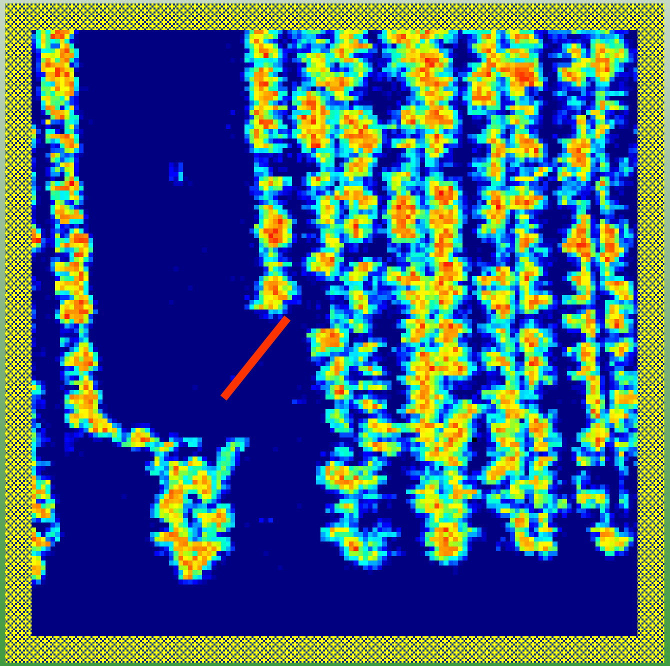
IBIC Microscopy Imaging of CVD Diamond

- Investigating radiation sensor performance
- Uniformity of charge collection efficiency
- Imaging intra-crystallite charge transport
- Correlating CCE with

morphology **electric field** **temperature**



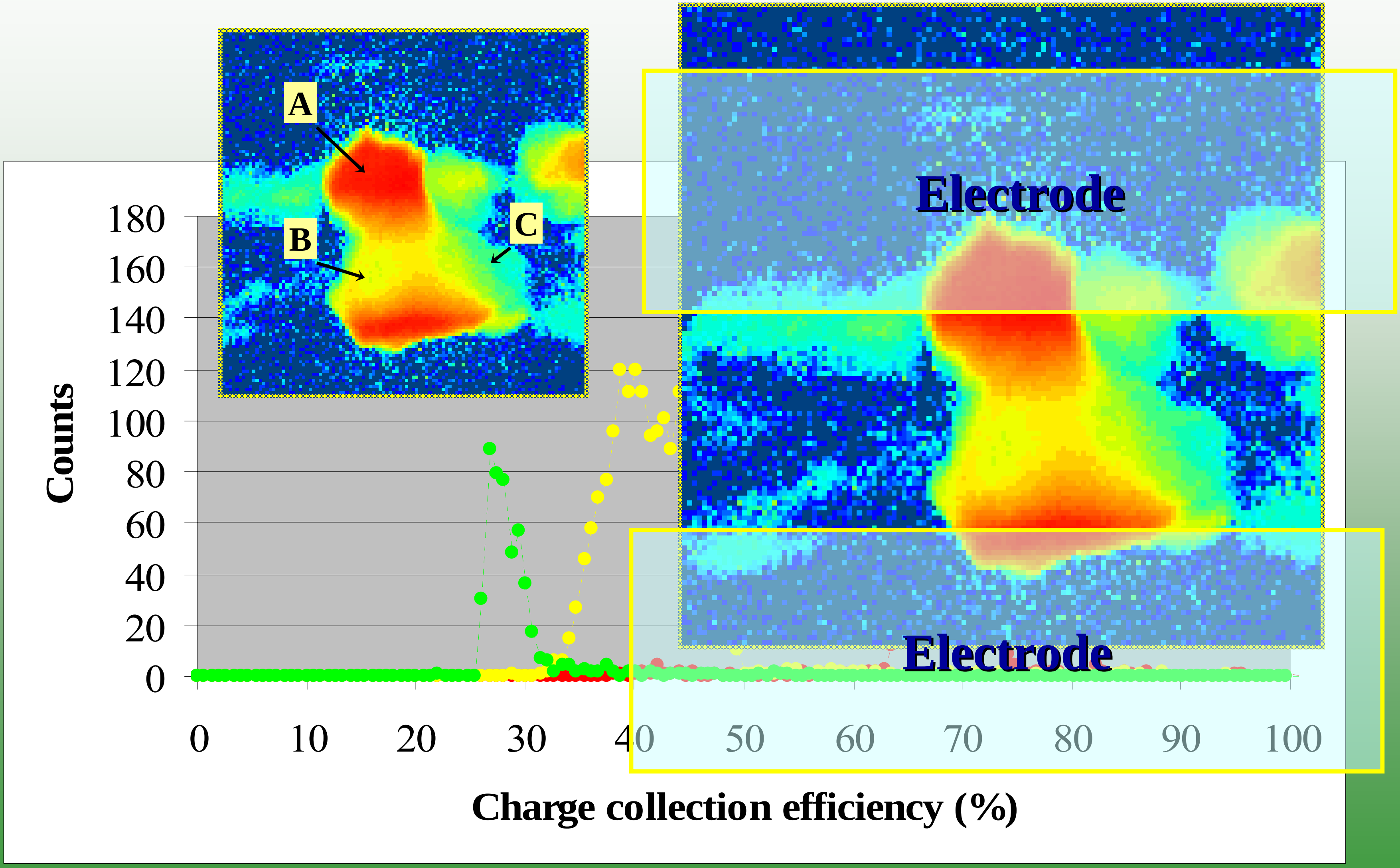
SEM Back-scattered electrons



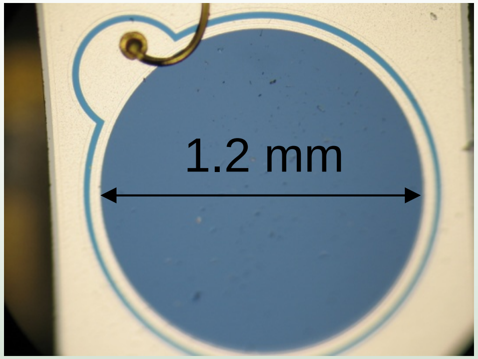
IBIC map

A. Simon et al. NIMB 219-20 (2004) 1022.

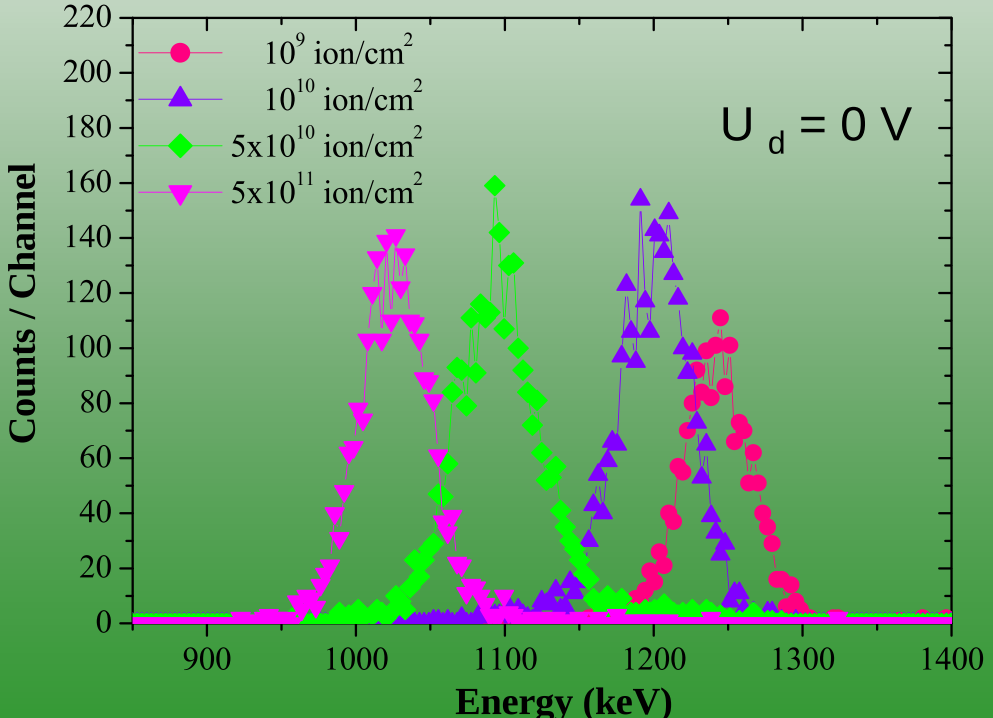
Pulse height spectra of selected regions within the crystallite

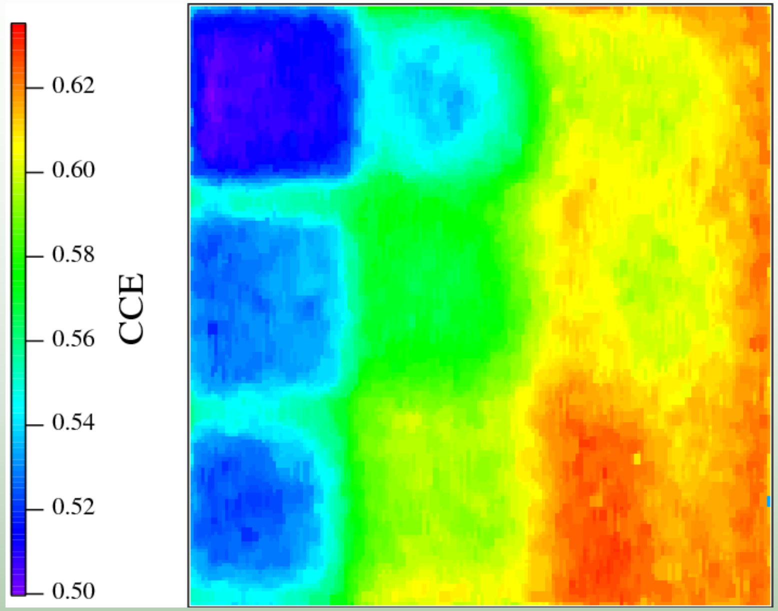


Si PIN photodiode



Hamamatsu S5821
PIN photodiode

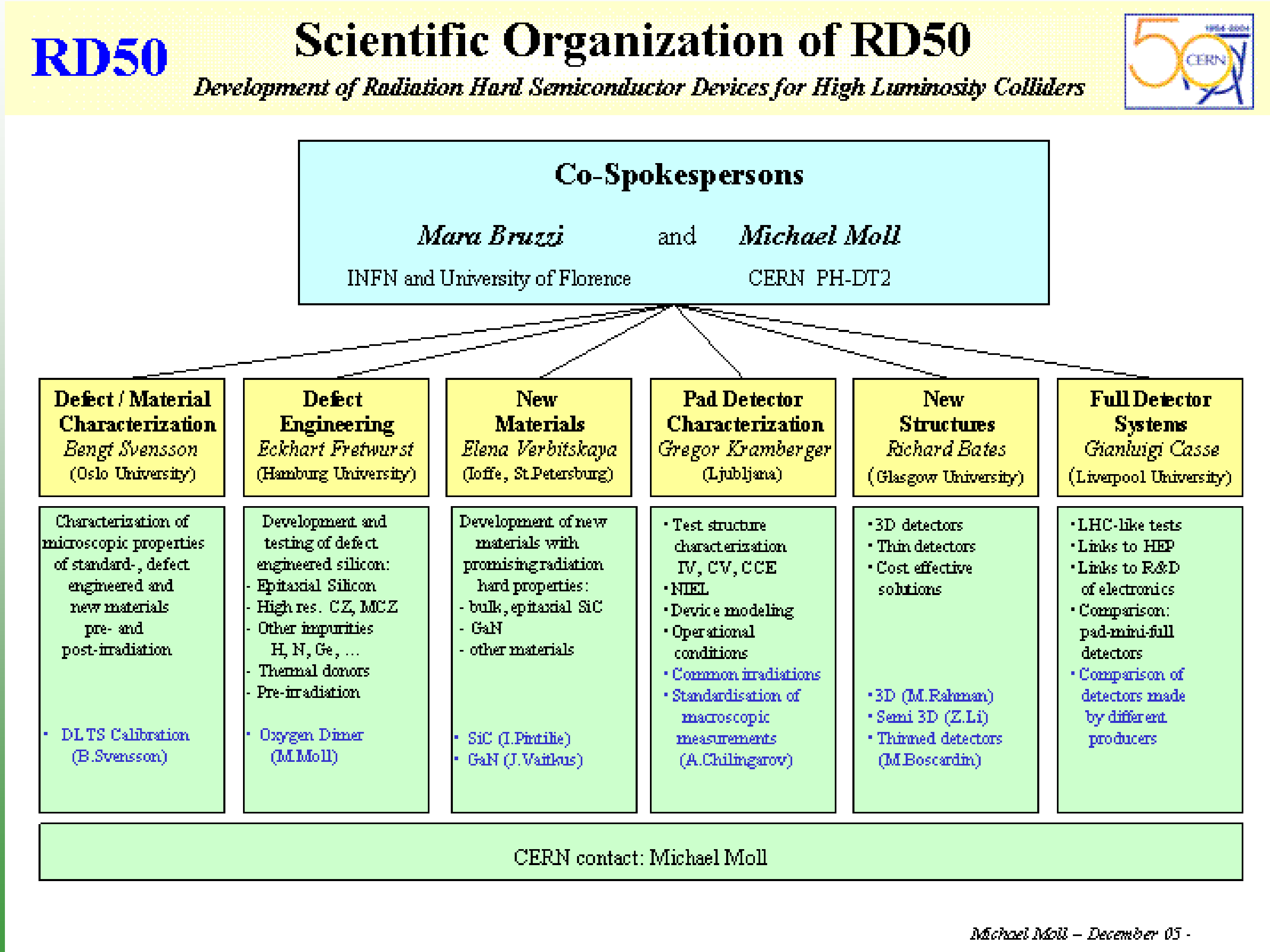




$U_d = 0$ V

IBIC map: 0V bias left. The full irradiated area (340x340 μm^2) is shown including the individually irradiated 100x100 μm^2 squares by fluences from bottom to top and right to left: 0.01, 0.02, 0.05, 0.1, 0.2, 0.5, 1, 2 and 5×10^{11} ion/cm^2, respectively.

CERN-RD50 collaborations



http://rd50.web.cern.ch/RD50/

SPIRIT

http://www.spirit-ion.eu/

Support of **P**ublic and **I**ndustrial **R**esearch using **I**on beam **T**echnology

SPIRIT represents an Integrated Infrastructure Initiative (I3) funded by the European Commission. The SPIRIT consortium integrates leading european ion beam facilities and R&D providers, which provide ions in an energy range from ~10 keV to 100 MeV for the modification and analysis of solid surfaces, interfaces, thin films and nanostructured systems. The main application areas are materials, biomedical and environmental research and technology.

SPIRIT integrates 11 leading ion beam facilities from 6 European Member States and 2 Associated States.

7 partners provide TransNational Access.

TNA Services offered by SPIRIT

http://www.spirit-ion.eu/

SPIRIT TNA Areas of Activities	Materials																Biomedical					Environment / Cultural Heritage		
	Ion Beam Analysis								Irradiation								Ion Beam Analysis			Irradiation		Ion Beam Analysis		
	RBS, ERD, NRA incl. Channeling	High Depth Resolution	Hydrogen Profiling	3D Analysis, Tomography	External Beam	Nanobeam	High-Resolution PIXE	Real-time in-situ IBA	Implantation	Plasma Immersion Ion Implantation	Very high energy	Radioactive Implantation	Ion Beam Lithography	Focused Ion Beam Irradation	Multi-beam Irradiation	In-situ structural analysis	IBA Tomoraphy	External Beam	nBeam Mapping	Radioactive Implantation	Targeted Irradiation	IBA Tomography	External Beam	Nanobeam
FZD	■	■	■					■	■	■				■	■									
CNRS				■	■	■											■		■		■	■	■	■
KUL	■	■							■			■								■				
JSI			■	■	■		■						■						■				■	
UBW		■	■	■		■															■			
CEA											■				■	■								
SUR	■			■	■	■			■				■			■	■	■	■		■	■	■	■

FZD (coordinator of SPIRIT) Forschungszentrum Dresden-Rossendorf , CNRS Centre National de la Recherche Scientifique, Bordeaux, KUL Katholieke Universiteit Leuven , JSI Jozef Stefan Institute, Ljubljana, UBW Universität der Bundeswehr München, CEA Commissariat à L'Energie Atomique SUR University of Surrey, Guildford

CHARISMA-FP7 I3 project 2009-2013

charisma

Cultural Heritage Advanced Research Infrastructures: Synergy for a Multidisciplinary Approach to Conservation/Restoration

Coordinator:Prof. Bruno G. Brunetti
UNIVERSITA DEGLI STUDI DI PERUGIA, Italy
Dipartimento di Chimica, Centro SMAArt

21 participants : museums, research institutes, universities

Project started: 1st October, 2009, TNA available from 1st January, 2010

FOUNDATION FOR RESEARCH AND TECHNOLOGY - HELLAS Institute of Electronic Structure and Laser, Demetrios Anglos
IDRYMA "ORMYLIA, Art Diagnosis Centre, Hieromonk Serapion Simonopetritis

HAS- ATOMKI, For TNA service contact: Aliz Simon, a.simon@atomki.hu

Nuclear Microprobes all over the world

Albuquerque, NM, USA, Ion Beam Materials Research Laboratory, Sandia
Albany, NY, USA, Ion Beam Laboratory
Bhubaneswar, India, Institute of Phisics
Bordeaux, France, Centre d'Etudes Nucleaires de Bordeaux Gradignan
Bochum, Germany, Ruhr-Universität
Buenos Aires, Argentina, "Tandar" Laboratory
Budapest, Hungary, KFKI Reseasrch Institute for Particle and Nuclear Physics
Chiba, Japan, NIRS
Cracow, Poland, Institute of Nuclear Physics, Polish Academy of Sciences
Darmstadt, Germany, GSI
Debrecen, Hungary, ATOMKI, Institute of Nuclear Research of the HAS
Denton, TX, USA, University of North Texas
Dharan, Saudi Arabia, KFUPM
Dresden-Rossendorf, Germany, Institute of Ion Beam Physics and Materials Research
Eindhoven, The Netherlands, TU/e, Accelerator Laboratory
Eugene, OR, USA, University of Oregon
Faure, South Africa, iThemba LABS, The Materials Research Group
Florence, Italy, INFN LABEC
Guelph, Canada, University of Guelph, PIXE Group
Guildford, UK, University of Surrey Ion Beam Centre
Hyderabad, India, CCCM
Irvington, NY, USA, Columbia University, RARAF
Johannesburg, South Africa, Schonland Research Centre
Kiev, Ukraine, "SPECTR" Laboratory, T.M.M. Company

http://icnmta.atomki.hu/microprobefac.html

Nuclear Microprobes all over the world

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