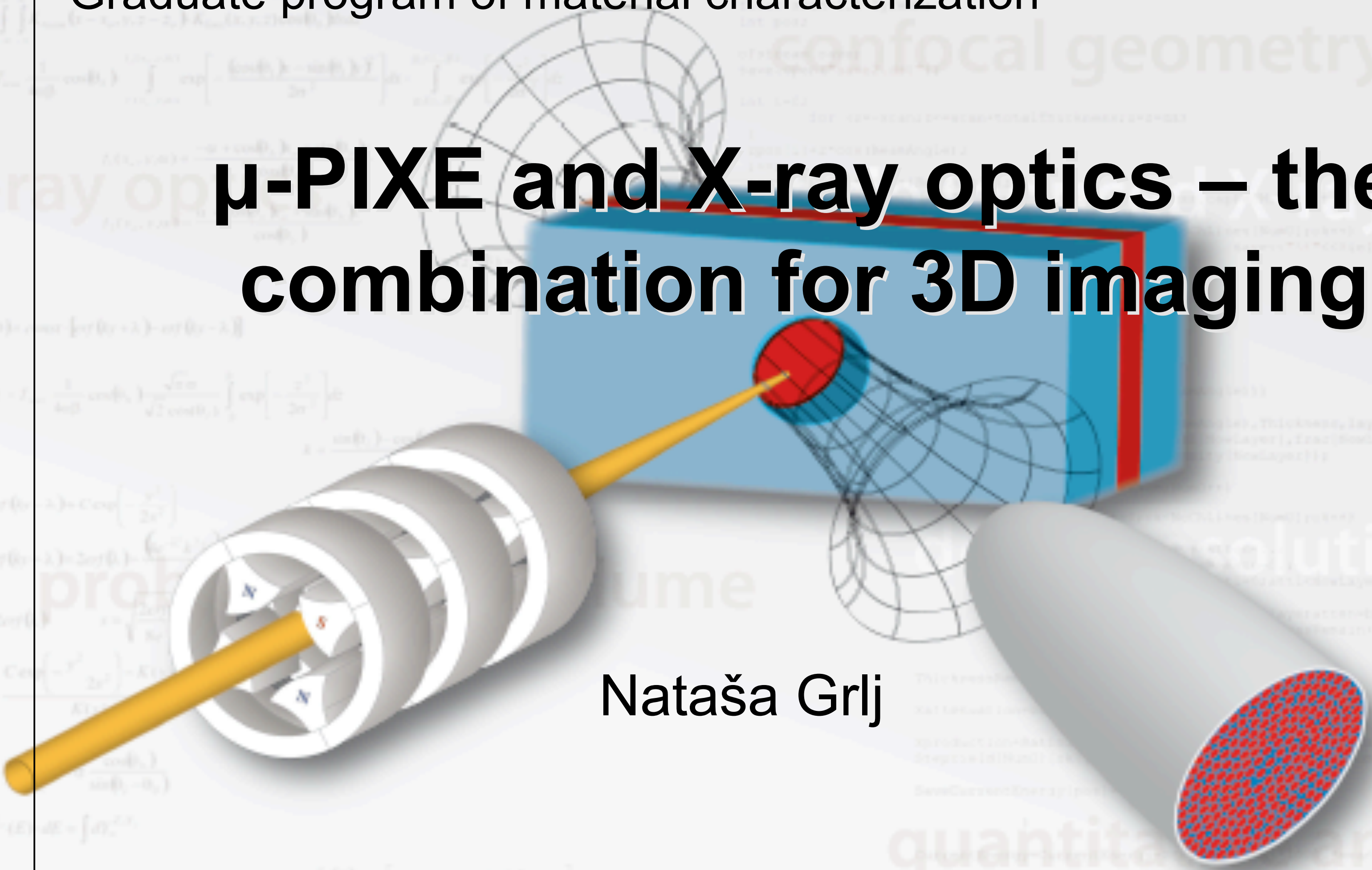
University of Nova Gorica

Graduate program of material characterization

µ-PIXE and X-ray optics – the combination for 3D imaging



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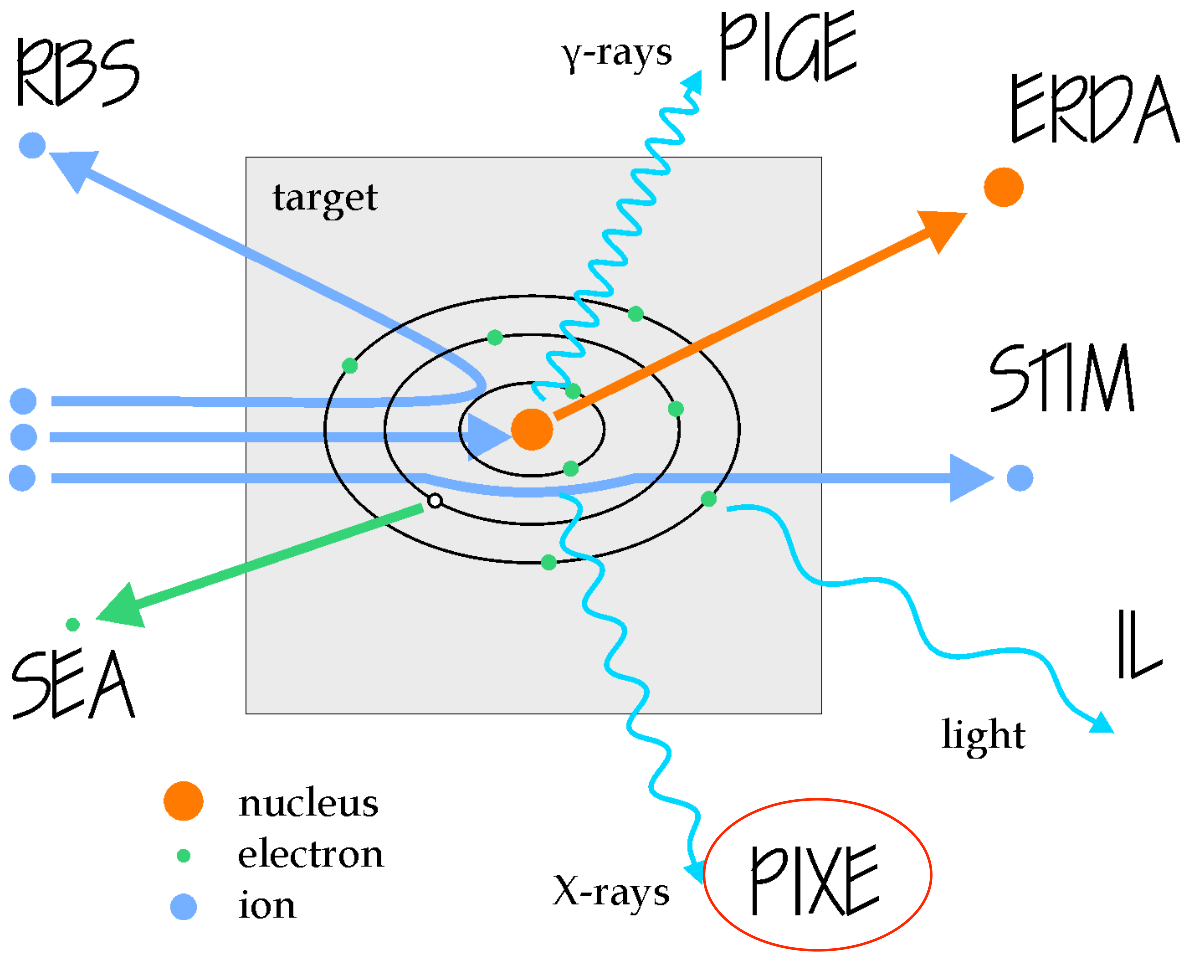
Thursday, 18.6.2009

Outline

- PIXE and μPIXE
- X-ray optics – polycapillary
- Confocal X-ray microanalysis with PIXE
- Application
  - Capillary characterization
  - Depth profiling
  - 3D tomography
- Further approach

Material analysis with proton/particle beam



Proton induced X-ray emission (PIXE)

- Frequently used technique for elemental characterization
- A material is exposed to ion beam, atomic interactions occur that give off EM radiation in the X-ray spectral range – the energies are specific to a certain element
- Detailed knowledge of collision dynamics, energy losses, ionization cross sections, fluorescence yield etc. – calculation of elemental concentrations

$$N_K = N_p n_{at} \frac{\Delta\Omega}{4\pi} \eta \epsilon \omega \int_0^R \sigma_i(E(x)) e^{-\nu \frac{\cos\alpha}{\cos\beta} x} dx$$

photon yield (K shell)

solid angle seen by the detector

ionization cross section

geometry set-up

number of incoming protons

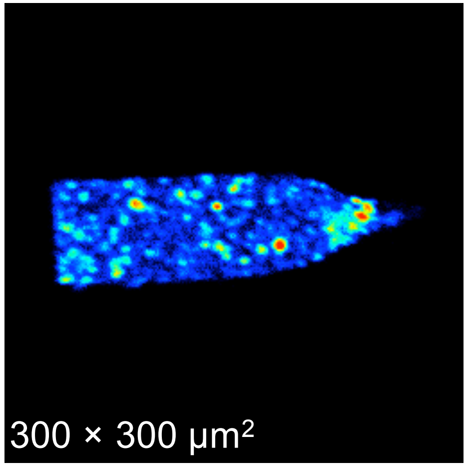
absorbers transmitivity and detector efficiency

absorption of emitted photons

What about μ-PIXE?

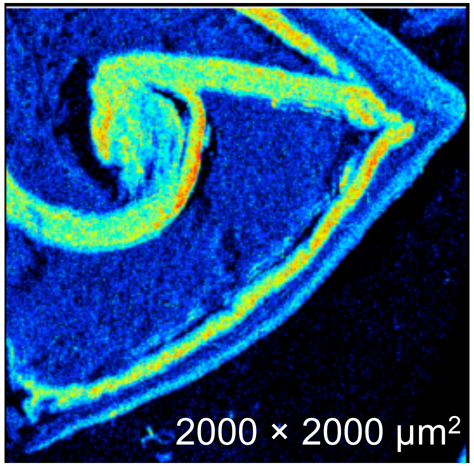
- a series of slits and quadrupole magnets in a nuclear microprobe allow us to focus proton/paricle beam to a micrometer level
- with the help of deflection magnet the beam can change its direction and we can scan the surface area
- at each point where the beam hits the target the spectra is collected
- in this way we can obtain 2D images and 2D elemental distribution of samples with micron lateral resolution

µPIXE – examples



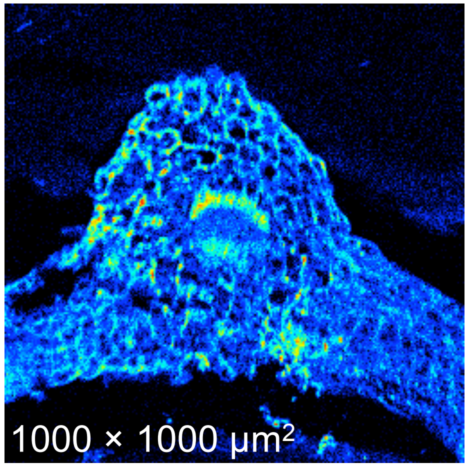
Tintinnopsis radix tr.

Fe K_α



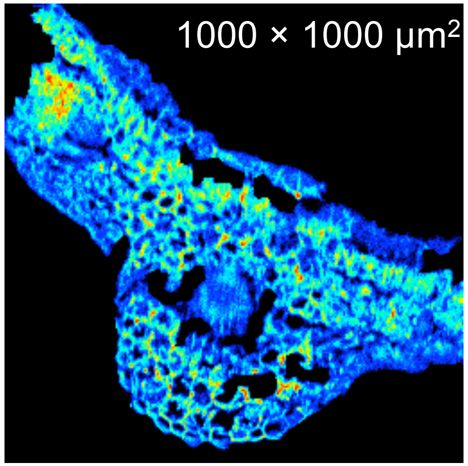
Buckwheat grain

S K_α



Arabidopsis leaf

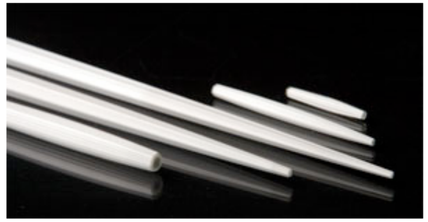
K K_α



Arabidopsis leaf

Ca K_α

X-ray optics – polycapillaries

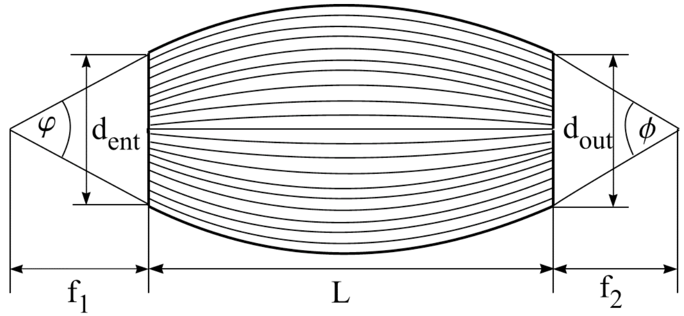


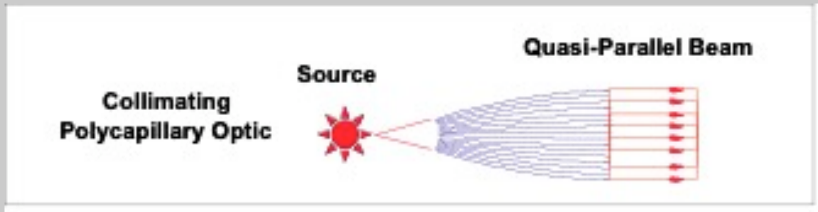
Special optical systems used to collect, collimate or focus X-rays

Made out of packed hollow glass tubes

The beam is transferred through tubes by multiple total reflections at the air-glass surface

Different applications in X-ray methods

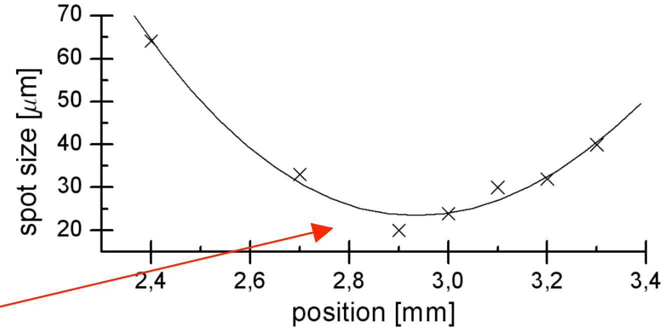




Model of semi-lens. It can be used in collecting (divergent beam from the source is collimated) or in focusing (collimated beam is concentrated on a small spot) mode.

Focus

- The envelope of collected rays reaches its narrowest point at some distance from the end of the polycapillary and the transverse dimension of the beam's profile reaches its minimum there – in this way, focus is defined



- The intensity profile can be described ~ with a 2D Gaussian function → FWHM a size representing quantity

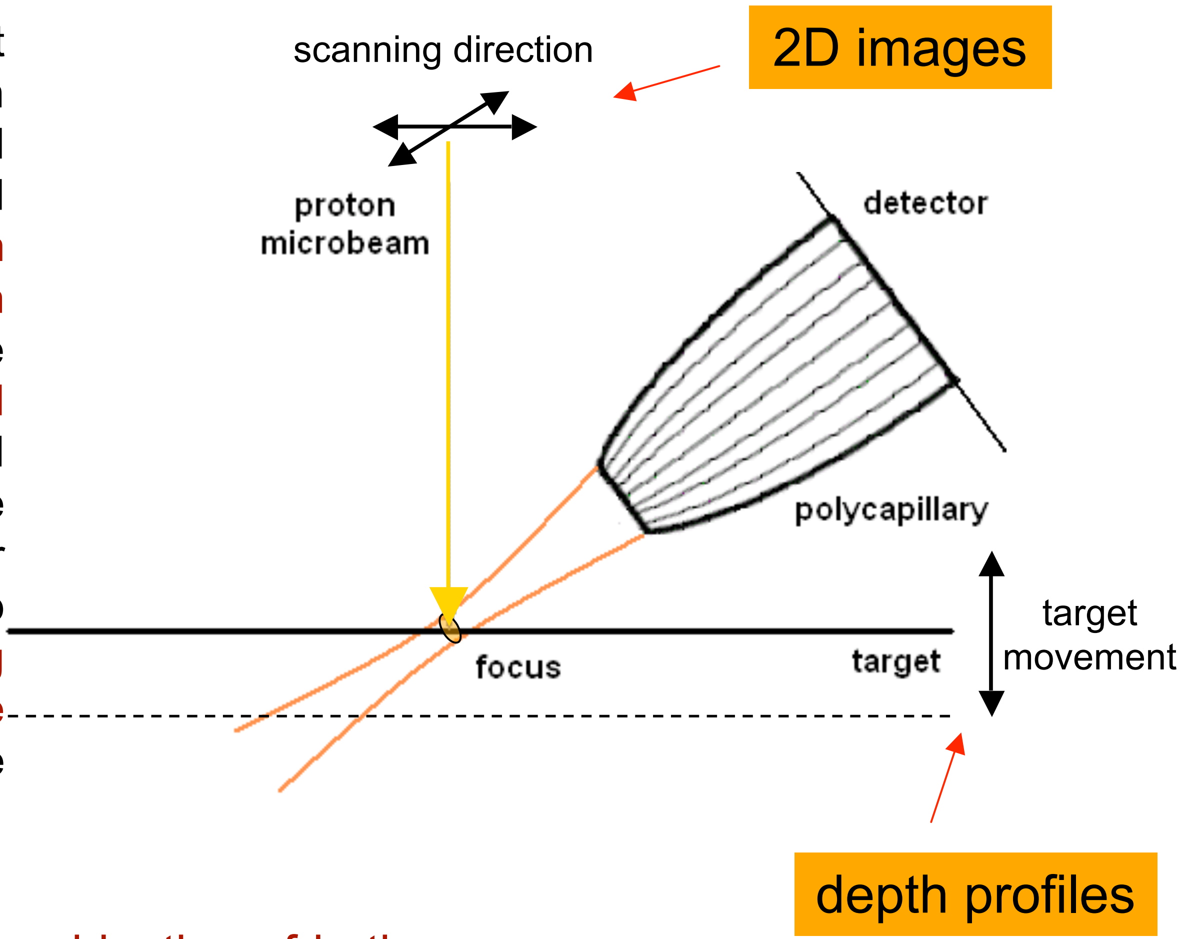
- FWHM strongly energy dependent �the transverse size of focal point as well

$$FWHM \propto \frac{1}{E}$$

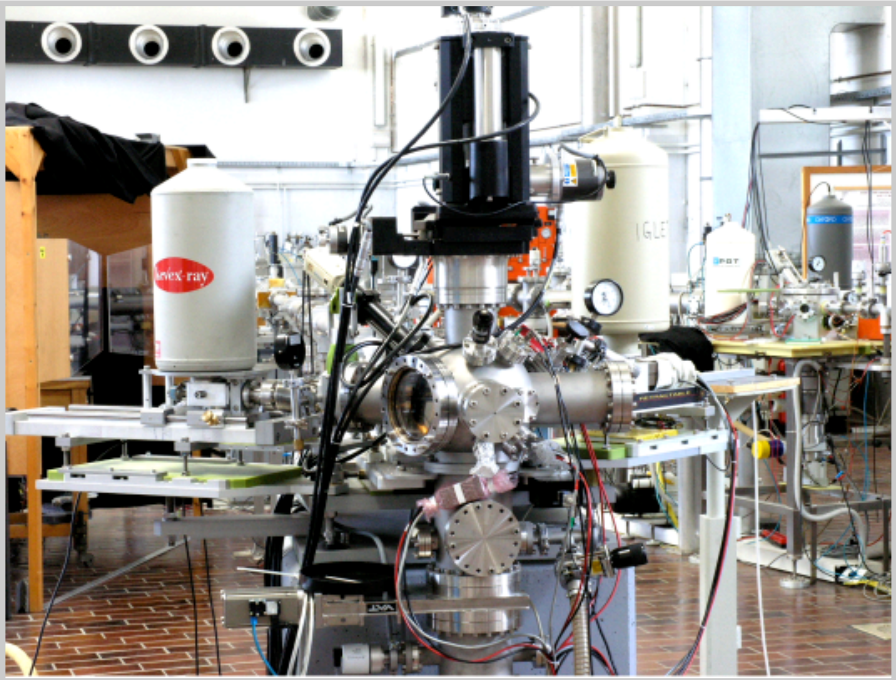
$$S \propto \frac{1}{E}$$

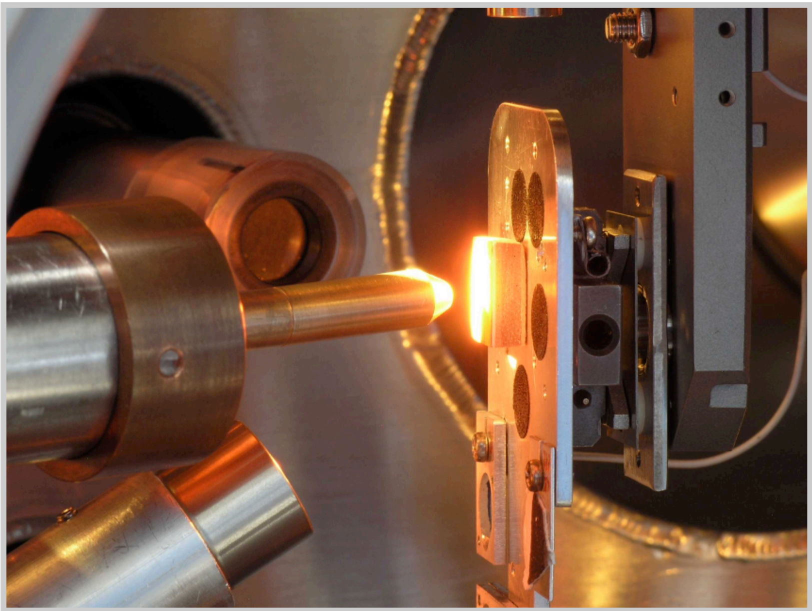
Combining PIXE with polycapillary lens

Two measurement possibilities – depth profiles extracted from signal obtained when the beam stays stationary in one point, while the target is moved forward and backwards from the incoming beam; or we can use superb lateral scanning capabilities of the microbeam on the stationary target.

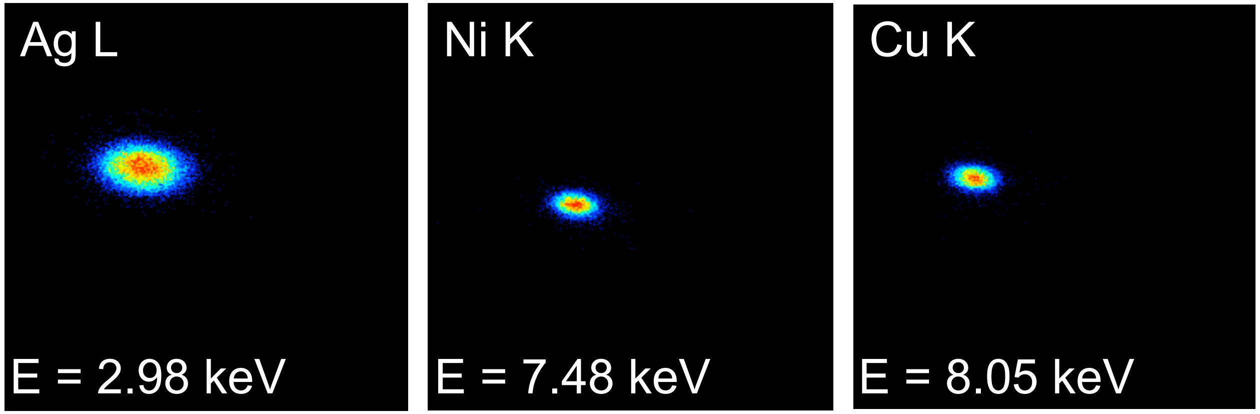


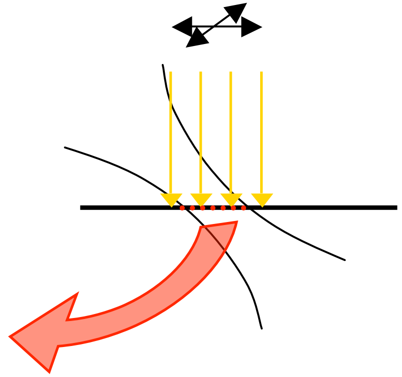
With the combination of both ways, we can analyse three-dimensional objects.



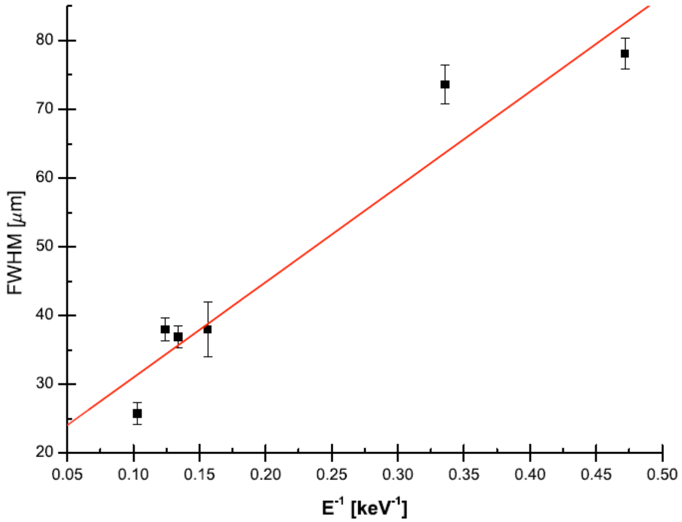


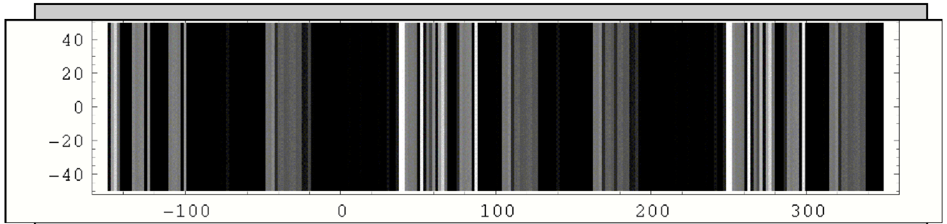
Measurements (1)
lens characterization





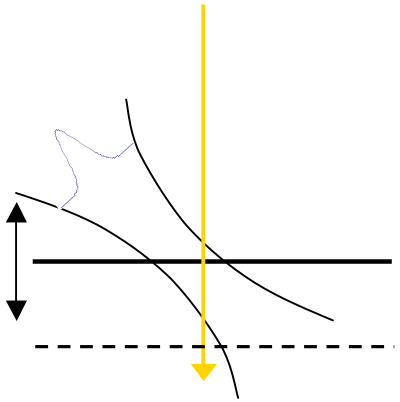
1 × 1 mm



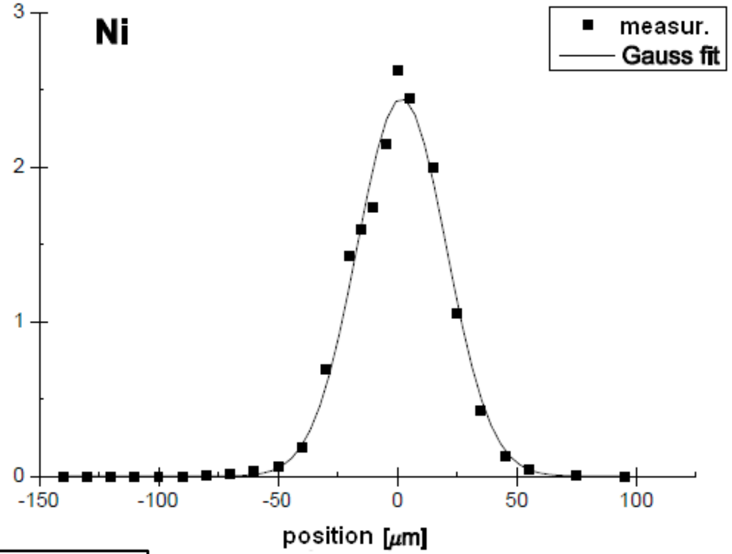


Superimposed 100 × 500 μm^2 X-ray images of 2 μm thick Fe foil, driven across the focus of the polycapillary lens.

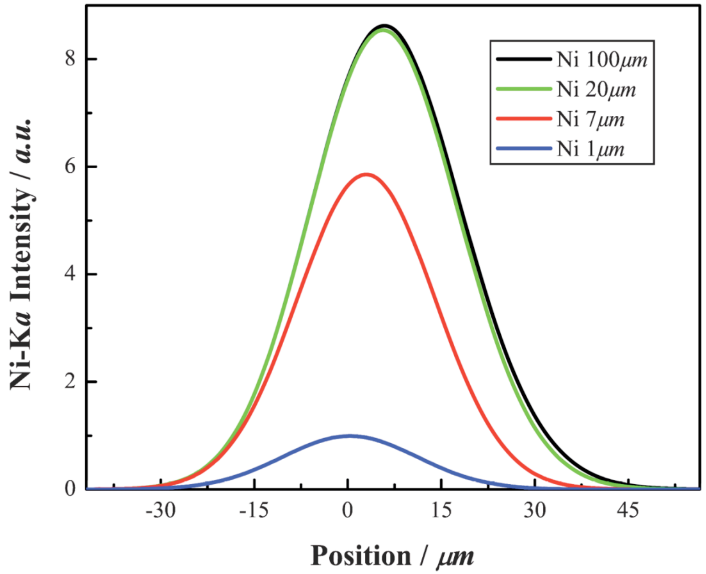
Measurements (2) – depth profiling



thin or thick target

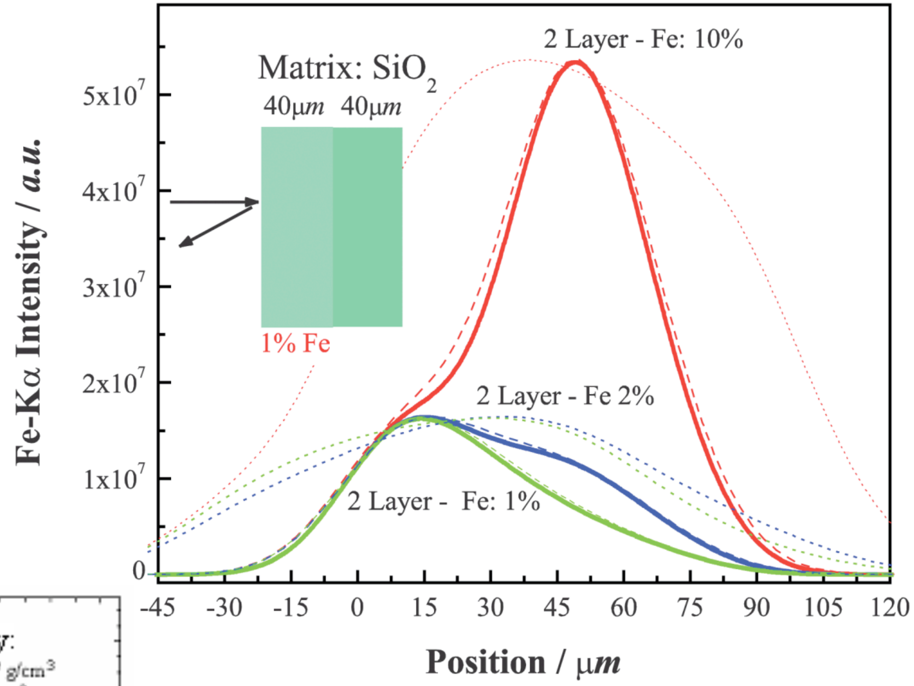


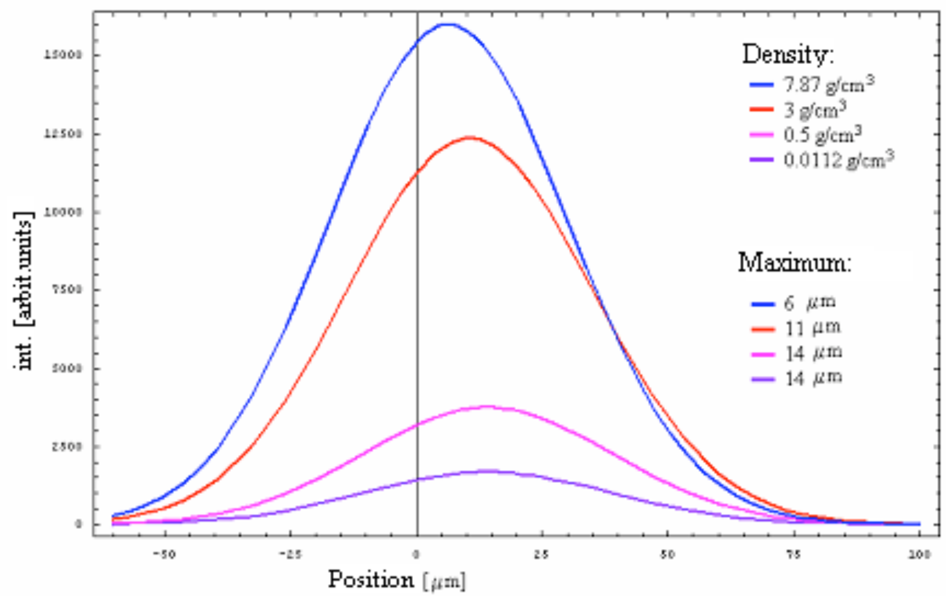
The signal from thick targets depends on its density – the energy loss and range of protons determine which part of target along the beam path radiate



The intensity (of every element in the target) at each target position is recorded and the recostruction gives again the polycapillary intensity profile.

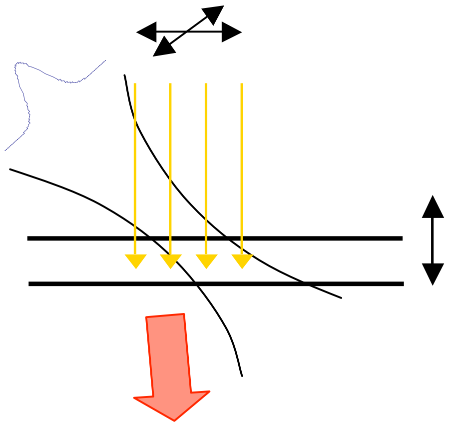
Signal from the spectrometer with mounted polycapillary is radically changed due to the samples thickness, density, layer position or elemental concentrations – in addition, all this can be extracted out of the signal.





In general – from signals collected in this scanning mode, some qualitative informations can be extracted – relative position of elements, density or layer thickness.

Measurements (3) – 3D imaging



The combination of lateral beam movement (surface scanning) and target movement in the third direction gives us three dimensional information about the sample.

We get a sequence of intensity maps for every element in the sample, collected at different target position.

To obtain a 3D image of the particle, it is necessary to write down the X-ray yield of a given element as a function of lateral displacement of the microbeam and of the displacement of the sample from the focal point. We get a set of equations for each pixel and for each target position.

Intensity yield of specific element →

$$I^f_{Z_i}(x,y) = N_p \frac{\Delta\Omega}{4\pi}\omega_f \int_{a(x,Z_i)}^{a+R\cos\alpha} dz \left[\sigma^f_i(E(x,y,z)) n^f(x,y,z) \times \epsilon^f(x,y,z) \exp\left(-\int_{z_1'}^{z_2'} \sum_e \sigma^e_a(E_f) n^e(x_1',y,z')\, dz'\right)\right]$$

The probability that an emitted photon is transmitted through the polycapillary lens

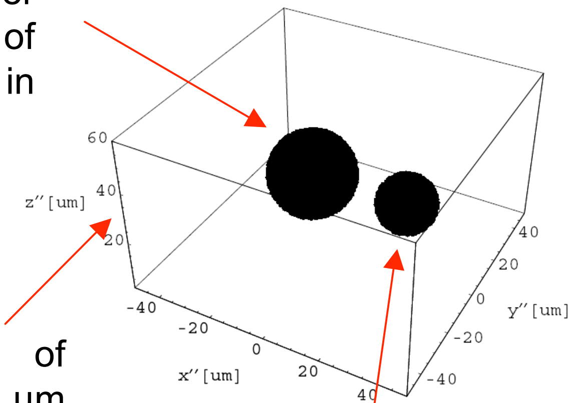
Path towards solution: the sample is divided into cells and the integrals are replaced with summation over them

Example model

Two objects – spheres – immersed into the nonabsorbing matrix.

r = 15 μm

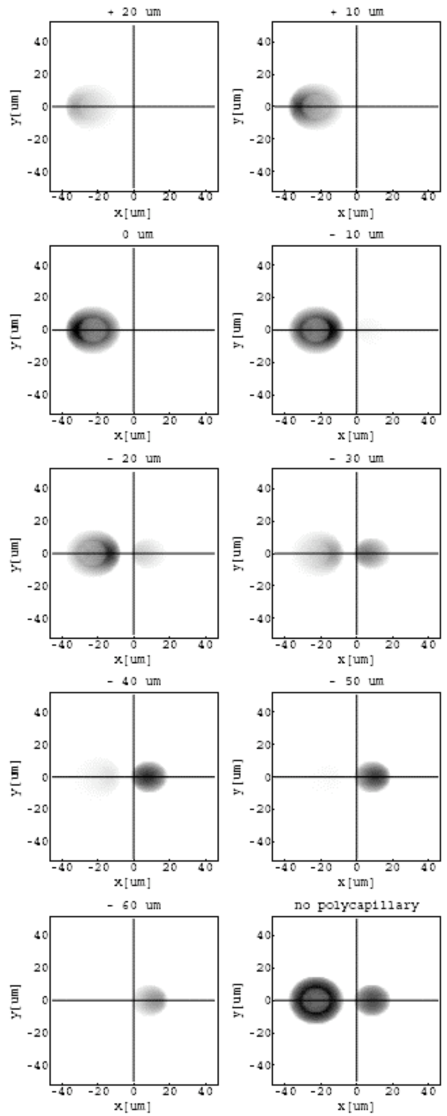
Nonhomogeneous – 5 times larger concentrations of certain element in the radial shell



The centres of both are 30 μm below the surface

r = 10 μm

Homogeneous



X-ray images at different Zi (20, 10, 0, -10, -20, -30, -40, -50, -60 μm) and the image of the detector without the polycapillary.

If only the last image would be recorded, one can assume two cylinder objects immersed in the matrix.

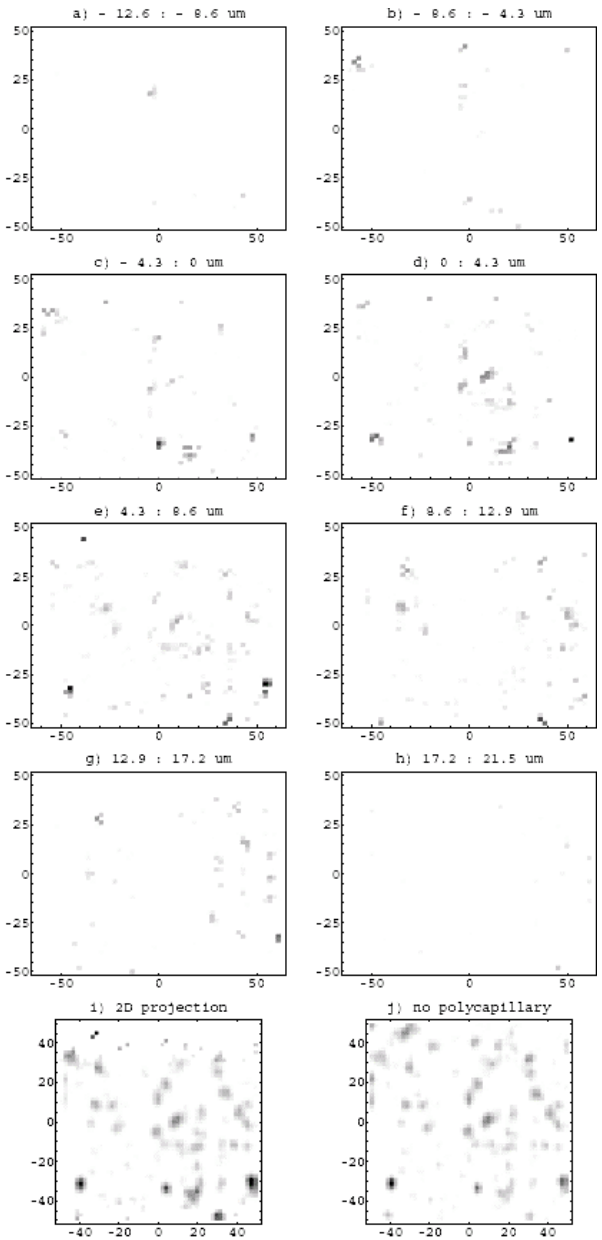
Example (1) – PM_{10} in a quartz filter

100 × 100 μm^2

A sequence of 17 iron images taken at different target position – Fe aerosol particles collected on quartz filter.

A diluted quartz matrix is quasi transparent for 3 MeV protons and for Fe K_α X-rays so the energy loss of protons and photon absorption can be neglected.

The matrix is very lightly doped – for each pixel only a single cell of the selected size is active (contains Fe). With this assumption, the Fe concentration distribution can be found.

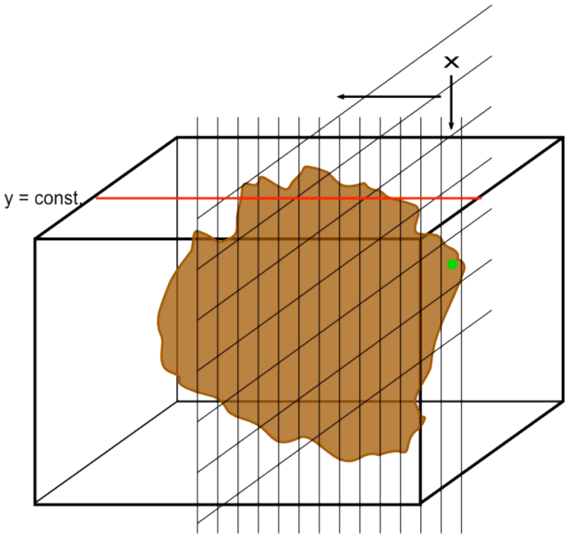


Fe concentration maps of 4.3 μm thick subsequent aerosol layers

The signal of all eight layers projected onto the lateral microbeam plane xy (left) and the μ-PIXE image of the same region taken with HPGe spectrometer without the lens (right).

Example (2) – particle of hematite

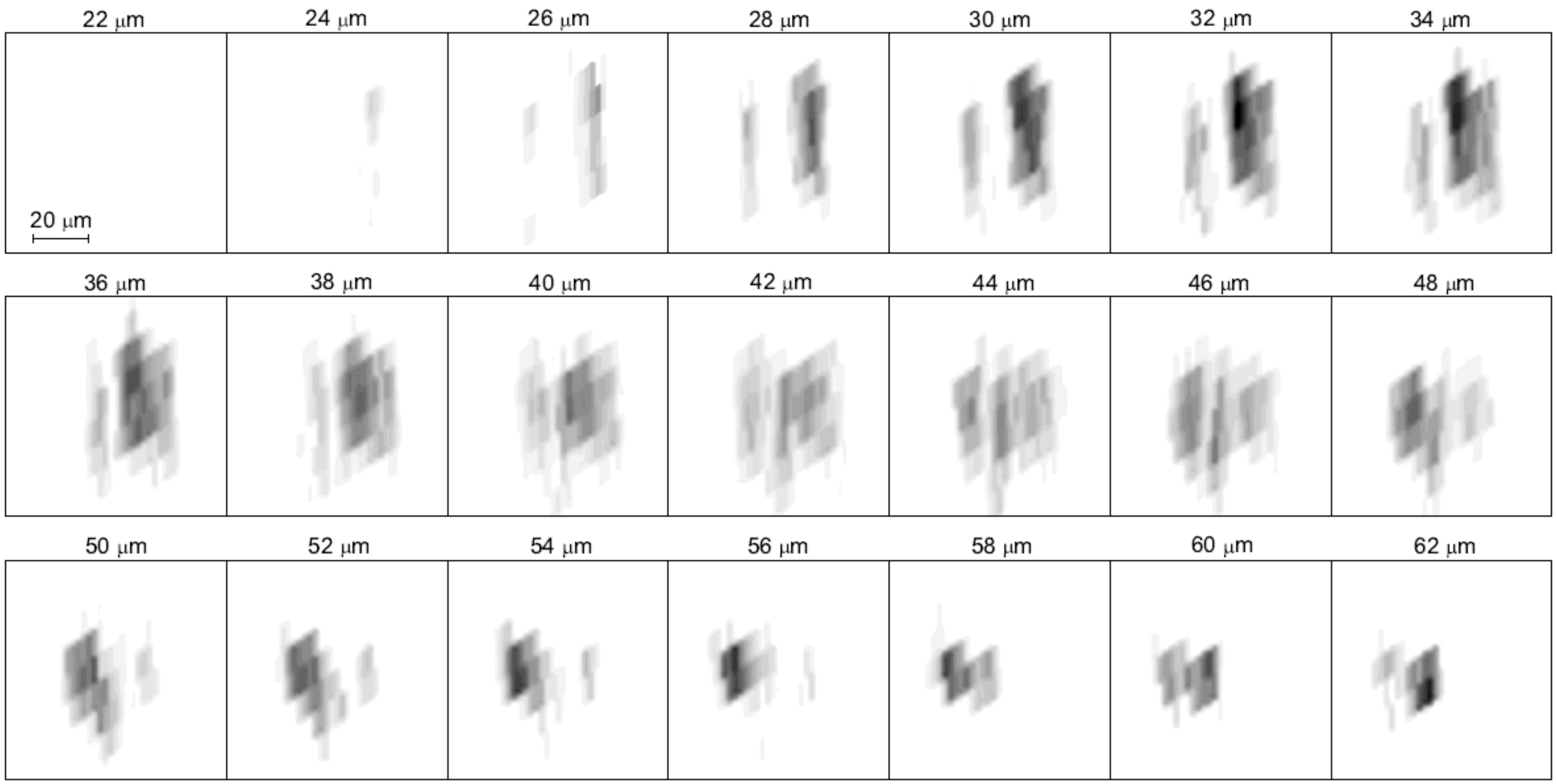
100 × 100 μm²



A sequence of 13 iron images taken at different target position – dust particle of iron ore (hematite) sitting on a nuclepore filter.

The effects of a filter can be neglected, however the energy loss (the range of 3 MeV protons is ~ 45 μm) and the absorption (the attenuation depth of Fe Kα X-rays is ~ 35 μm) can not.

In this case, each cell in a column (x = const., y = const.) contributes to the total yield in one pixel.



A sequence of the reconstructed slices xz in the particle region. Black colour correspond to cells fully occupied by hematite and white correspond to empty cells.

Summary

- PIXE is a common method for material characterization
- μ-PIXE allows us to achieve micron lateral resolution
- With the use of polycapillary optics in front of the spectrometer the confocal arrangement is obtained and we get a 3D μ-PIXE method
- This method can be used for capillary characterization, depth profiling, qualitative depth measurements or three dimensional imaging
- Great for the analysis of layered structures or for 3D concentration distribution measurements of heavier elements in light matrix
- In search for better lenses (or different X-ray optics) to achieve micron depth resolution

Papers:

1) Karydas et al.: 3D Micro-PIXE – a new technique for depth-resolved elemental analysis. *J. Anal. At. Spectrom*. 22 (2007) 1260

2) Žitnik et al.: Three-dimensional imaging of aerosol particles with scanning proton microprobe in a confocal arrangement. *Appl. Phys. Lett*. 93 (2008) 094104

3) Sokaras et al.: Quantitative analysis in confocal μ-PIXE – general concept and layered materials. *J. Anal. At. Spectrom*. 24 (2009) 611

4) Wolff et al.: Performance of a polycapillary halflens as focusing and collecting optics – a comparision. *J. Anal. At. Spectrom*. 24 (2009) 669

5) Žitnik et al.: Element-selective three-dimensional imaging of microparticles with a confocal micro-PIXE arrangement. *X-Ray spectrom*. (submitted)