Compact wavelength-dispersive spectrometer for micro-fluorescence analysis at ID21 beamline.

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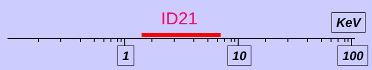
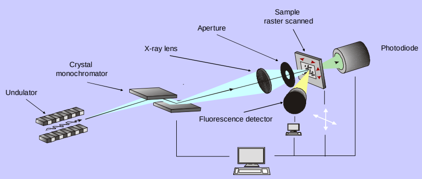
ESRF

Since several years, the polycapillary x-ray optics has become a very important tool in x-ray fluorescence applications. Polycapillary x-ray lenses can collect the radiation emitted from a small source into a large solid angle and therefore they have a strong impact on the development of x-ray fluorescence analysis by improving the detection limits. For this reason, polycapillary lenses are used in x-ray microanalysis, in particular in the Particle Induced X-ray Emission (PIXE), Electron Probe Micro-Analysis (EPMA) and Microbeam X-ray Fluorescence (MXRF). Increasing demands for developing new and complementary x-ray techniques to be used in different applications (e.g. semiconductor nano-technology, biology, geology, archaeology) are presently focused on high-resolution and sensitivity x-ray fluorescence techniques combined with a narrow, down to the sub-micrometer range, x-ray beam excitation. For this reason, we intend to implement at the ESRF beam line ID21 a new x-ray wavelength dispersive spectrometer (WDS) for the micro-fluorescence analysis.

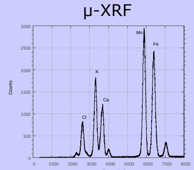
ID21 X-ray Microscopy Beamline



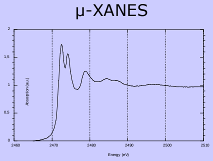


- 2 tunable mirrors Si, Ni, Rh
- Si 111, Si 220, NiB_4C multilayers
- Fresnel zone plate, 1x1 – 0.2x0.6 μm², ~ 10^9 ph/s
- 2D high spatial resolution scanning microscope
  - μ-XRF, μ-XANES, phase contrast

μ-XRF

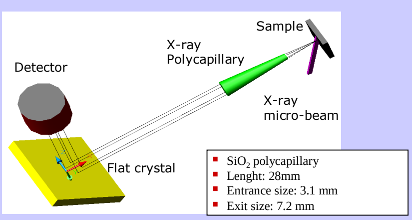


μ-XANES

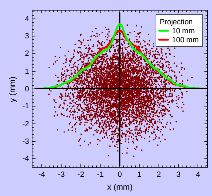


Monte – Carlo simulations

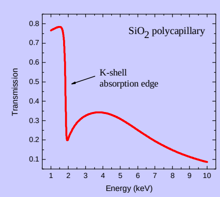
- Random generation of x-ray direction
- Total external reflection condition tested using Atomic Scattering Factors
- XOP crystal rocking curve



2D map of x-ray beam distribution at the output of polycapillary



Polycapillary transmission versus the photon energy

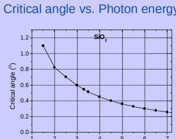


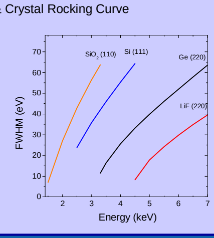
Spectrometer resolution

Γ_{exp}= Beam Divergence & Crystal Rocking Curve

$$\Gamma_{exp}=\Theta_{critical}\,\Theta_{crystal}{*}\mathrm{Cot}(\Theta_{Bragg})$$

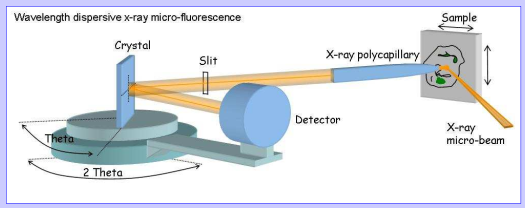
Critical angle vs. Photon energy

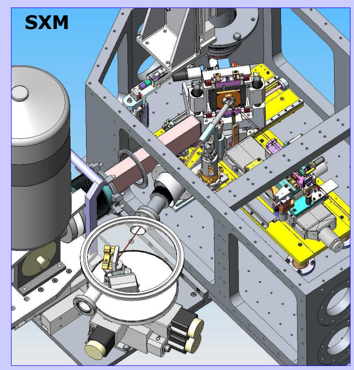




Spectrometer design

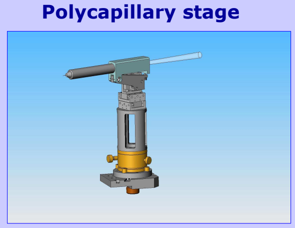
The wavelength dispersive x-ray mini-spectrometer will consist of an x-ray polycapillary optics, a theta two-theta rotation table, a flat crystal and an x-ray detector. The polycapillary optics will be installed in the Scanning X-ray Microscopy chamber of ID21 at a distance of few millimeters from the target. With this setup, a relatively large solid angle of about 20 degrees can be covered. At the exit of the polycapillary a quasi-parallel beam is formed which makes an angle theta with the flat crystal. The x-ray detector, placed at an angle 2 theta with respect to the incident x-rays, collects the photons which fulfill the Bragg condition. The rotation stage, crystal and detector will be enclosed in a separate chamber, installed next to the Scanning X-ray Microscope.



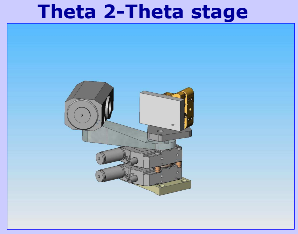


Mechanical design by E. Gagliardini, ESRF Drafting Office

Polycapillary stage



Theta 2-Theta stage



Polycapillary parameters:

- Input focal distance: 10mm
- Optic output diameter: 7.2mm
- Input capture angle: 20.04˚
- Input field of view: 50 – 100 μm
- Output divergence: 3.4 mrad @ 8keV
- Transmission efficiency:
  @ 1.5 keV – 33%
  @ 8.0 keV – 14%

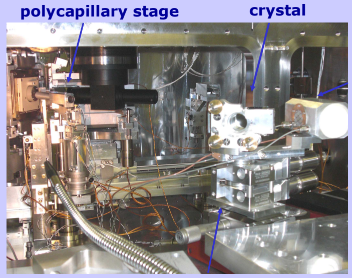
- High precision θ - 2θ rotation table
- Stepping resolution 0.002°

- Flat Ge(220) crystal ~ 5x5 cm
- Crystal energy resolution (ΔE/E = 5E-4)
- Bragg angles 20˚ - 70˚

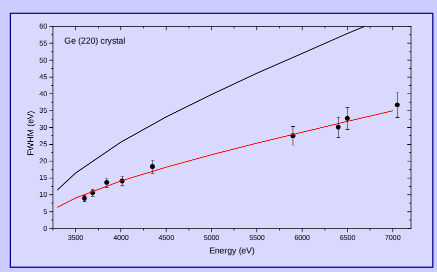
- Flow-gas detector (FP100 from Paralax)
- P10 gas at flow rate of 25 sccm
- Active area ~380 mm²
- Resolution 20%

Preliminary results

Experiment



Spectrometer resolution



X-ray fluorescence spectra