



Experiment title:

**Focusing optics for high energy bulk diffraction with
high spatial resolution**

Experiment

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MI 142

Beamline:

BM5

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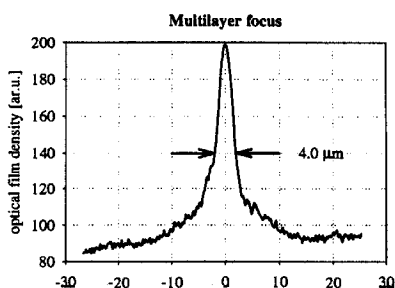
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Report: (1) Optics

Aim: At present, no routinely usable focusing, energy tuneable, fixed exit monochromator exists for high x-ray energies. A prototype version of such a monochromator, based on bent Laue and Bragg crystals, was installed at the ESRF beamline BM5 but no diffraction experiments were performed yet. This experiment was aiming at:

- (i) testing the performance of the monochromator in an actual diffraction experiment
- (ii) combining the monochromator with a vertically focusing multilayer
- (iii) demonstrate that a broadening of diffraction peaks by the focusing divergence can be avoided.

Laue-Bragg Monochromator: The intrinsic energy band path of the LBM is $\Delta E/E = 2 \cdot 10^{-4}$. 35 mm of the horizontal radiation fan was accepted. The focus size is limited to 1 mm by the penetration of the x-rays into the crystals. The focus was slit down to 100 - 300 μm to obtain sufficiently narrow diffraction peaks. By detuning the crystal bending such that the source went off-Rowland circle, an horizontal energy gradient was selected to match the dispersion of the Cu and Ni (3 11) reflections

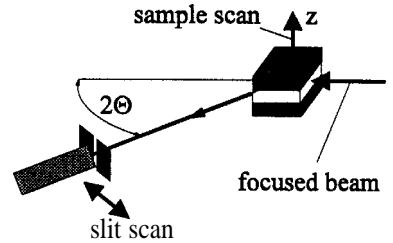


MultiLayer: The W/B_4C -ML (d-spacing of 25 Å, laterally graded) was grown by the ESRF ML group. The distance from the ML to the focus was 0.75 m and 220 mm of the ML were illuminated, equivalent to 800 μm beam height. Elliptical bending was achieved by a two moment bender which was calibrated by optical metrology. The x-ray energy was chosen to be just below the W K-edge.

Results: The vertical size of the line focus was measured to be $4\pm 1\ \mu\text{m}$ by high resolution film exposure, absorption knife-edge and slit scans. The focus width is limited by the remaining slope error of the ML substrate. A peak reflectivity of 80 % was measured at the centre of the ML decreasing down to 30% at the edges. The all over gain factor (as compared to a $4\ \mu\text{m}$ wide slit) is around 50. The proposed monochromator provides an intensity gain factor larger than 1000 as compared to existing non-focusing optics. The selection of an horizontal energy gradient by the LBM for dispersion compensation posed no problems.

(2) Diffraction

Aim: The aim of the experiment was to determine the bulk strain gradient in layered structures. This is only feasible in transmission geometry and requires high energies and (one-dimensional) focusing. Conventional techniques based on x-ray tubes are only sensitive to a surface region.



Experimental: Samples were Cu/Ni multilayers grown on Cu substrates by 'dual bath technique' (layer thickness 1 and $50\ \mu\text{m}$, total thickness $200\ \mu\text{m}$) and single $10\ \mu\text{m}$ thick CrN layers grown on Fe substrates. Due to the novel optics approach sufficient intensity and a spatial resolution of $4\ \mu\text{m}$ were obtained on a bending magnet source. Broadening of the diffraction peaks due to the horizontal focusing was avoided by dispersion compensation. The peak width was limited by the sample thickness, detector slit and incoming beam widths and by the intrinsic energy band of the monochromator ($\Delta E/E = 2 \cdot 10^{-4}$). Best resolution would be obtained by a large sample-detector distance which was, however, prevented by the hutch dimension. Still, the minimum peak width of a Fe calibration powder was obtained for the (2 1 1) reflection as expected by the dispersion contribution.

Results: The contour plot of the (3 1 1) diffraction peaks clearly shows that the peaks of the Ni layers are wider than for the Cu layers indicating sensitivity to micro structure (grain size). It is also clear that the spatial z -resolution is sufficient to obtain strain profiles within single layers. The quantitative evaluation of the strain gradient by peak fitting is under way but is also discernible by the slight tilt and waviness of the Cu ridge. The intensity variations are most likely due to texture. The detailed evaluation is in progress.

