ESRF	**Experiment title:** Development of high Energy Focusing Optics for Materials Diffraction (longterm proposal)	**Experiment number**: MI-225
Beamline: ID15a/BM5	**Date of experiment**: from: 2-7-99/7-7-99 to: 4-7-99/9-7-99	**Date of report**: 16 July 99
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Report:

(1) ID15a: Characterisation of $Si_{1-x}Ge_x$ gradient crystals

Aim: The experiment was aiming to characterise a SiGe gradient crystal obtained from the IKZ (Berlin). The high potential of SiGe gradient crystals as optical elements at high energies has been demonstrated at HASYLAB where five crystals were investigated but only a [1 1 1] oriented crystal had the required perfection.

Experiment:

Three 2 mm thick crystals were cut along the [1 1 1] growth direction as to provide symmetric [1 1 1] Laue reflection. The lateral dimension of the crystals were about 70 x 32 mm. [1 1 1] rocking curves were recorded at 80 keV as function of the lateral position on the crystals. A bandwidth of 0.12% and a reflectivity of about 90% was observed in a central area of about 10 x 20 mm^2. The absolute lattice parameter and therefore the Ge concentration were measured by mounting a perfect Si reference crystal to the gradient crystal. Gradient-gradient crystal rocking curves were recorded at selected positions for parallel and anti-parallel gradient directions. A reflectivity of about 86% was measured independent of the gradient orientation. [3 3 3] rocking curves were recorded at 200 keV reproducing sharp peaks at the flanks of the rocking curves which were also detected at HASYLAB. Most rocking curves were taken with a beam size of 1x1 mm^2 but selected scans were repeated with smaller beam size of 0.1x0.1 mm^2. Reflectivities were higher with the small beam reaching up to 96%.

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Conclusion:

(1) The SiGe crystals show dynamical diffraction behavior in a central region

(2) The crystals are therefore suitable components for a source size conserving monochromator for beam sizes of a few mm^2.

(3) The size of the dynamically behaving region was smaller than on a crystal grown earlier for HASYLAB. The growth of gradient crystals is therefore not yet reproducible but both [1 1 1] oriented crystals have perfect inner regions.

(4) Peaks in the rocking curves of high orders were observed at independent samples and set-ups. They are not yet explained by dynamical theory and might be due to strain relaxation at the crystal surfaces.

BM5: Combination of a SiGe double crystal monochromator and micro-focussing multilayer.

Aim: We were aiming to proof that the large polychromatic divergence upon a single Laue gradient crystal reflection is compensated in a double crystal setting and that micro-focussing is still possible in the diffraction plane.

Experiment:

All components diffracted in the vertical plane to profit from the narrow vertical source size. Two SiGe crystals in non dispersive [1 1 1] geometry were set-up as fixed exit monochromator. The energy could be varied between 40 and 80 keV limited by the range of the longitudinal translation of the second crystal. A meridionally graded multilayer was mounted behind the monochromator and focussing achieved by elliptical bending. The horizontal beamsize was 1 mm throughout whereas the vertical beam size was decreased from 1 to 0.7 mm with increasing energy to retain a footprint of about 180 mm on the multilayer. The focus size decreased from 6.3 µm (40 keV) to 4.5 µm (80 keV). The minimum focus size was limited by the multilayer slope error of 1 µrad (RMS) resulting in 4 µm focus broadening at the focal length of 0.8 m. The energy bandwidth was measured with a perfect horizontal analyzer crystal and increased from $\Delta E/E = 0.061$ % to 0.12 % at 40 and 80 keV, respectively.

Conclusion:

It was demonstrated that SiGe gradient crystals can provide a broad band, fixed exit monochromator at high energies which allows for micro-focussing. The potential of micro-focussed high energy x-rays within materials research is being demonstrated with the commissioning of the ID11 extension hutch. A fixed exit monochromator would open the following possibilities: (1) energy scanning techniques, (2) use of glass substrates for the multilayers (3) substantially reduced shielding allowing shorter focal length and therefore reduced focal spot size and (4) fast change of experimental set-ups. Future work will therefore concentrate on the optimization and availability of depth graded multilayers. This optics is, to our knowledge, the only route to a fixed exit, micro focusing monochromator.