ESRF	**Summary report on longterm proposal** Development of high Energy Focusing Optics for Materials Diffraction (longterm proposal)	**Experiment number**: MI-225
Beamline: BM5 / ID15a	**Date of experiment**: from: Sept-97 to: July-99	**Date of report**: 27-7-99
Shifts: 9 / 9	**Local contact(s)**: U. Lienert	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

U. Lienert*, A. Freund, H. Graafsma, O. Hignette, V. Honkimäki*, Å. Kvick, M. Lingham, Ch. Morawe, E. Ziegler (ESRF); S. Garbe, E.M. Lauridsen*, H.F. Poulsen* (Risø, Materials Research Department, DK-4000 Roskilde, Denmark),
C. Schulze* (Swiss Light Source, PSI, CH-5232 Villigen PSI, Switzerland),
S. Keitel*, W. Caliebe*
(HASYLAB at DESY, Notkestr. 85, D-22603 Hamburg, Germany)

1 Aim and context of the proposal

The aim was to develop optical elements that can micro-focus high energy radiation for local structural characterisation of polycrystalline bulk materials. The long term proposal provided beamtime for experimental tests during the design and construction period of a dedicated extension hutch at ID11.

2 Main results

(i) Stability of air cooled Laue monochromator

Cooling of bent crystals is a sophisticated technical problem in particular for vertical scattering geometry. Furthermore, strain scanning applications with micrometer spatial resolution require angular stability and shape errors on the micro-radian level. Cooling mechanisms are known to interact with the bending and therefore introduce shape errors and drifts. Relying on tests at ID15a we decided that water cooling is not required but to place the crystal in air. The stability of the Laue monochromators at ID11 turned out to be excellent. The tedious development of a water cooling scheme would have substantially complicated and slowed down the commissioning of the ID11 extension hutch optics.

(ii) Focusing analyzer multilayer optics

Local gauge volumes are conventionally defined by crossed-slit-techniques placing a narrow slit behind the sample. It was demonstrated at BM5 that replacing this slit by an imaging (focussing) element provides major advantages: intensity gain by larger aperture, better spatial resolution, remedy of systematic errors occurring in strain measurements and

simultaneous data acquisition.

(iii) Energy tunable, fixed exit, broad band monochromator

At the moment, there is no fixed exit monochromator scheme that provides a broad energy band and conserves the source size. Two schemes based on bent perfect Si crystals and SiGe gradient crystals were investigated. Micro-focussing was demonstrated behind monochromating SiGe crystals. The source size conservation was also demonstrated with the bent crystals but technical (not principle) problems prevented micro-focussing.

(iv) Scattering from narrow apertures

Despite their limitations, narrow apertures can not be replaced in all situations by focussing optics. Experience with commercial slits showed that often substantial scattering is produced at high energies preventing aperture openings below 10 μm. It was found that diffraction limited beams (down to 2 μm) can be prepared by rounded and polished WC blades. W single crystals were successfully prepared to prevent wide angle scattering.

3 Conclusion

The experimental results from the long term proposal substantially advanced the design and construction of the ID11 extension station. Beamtime was provided during the design phase on the versatile ID15a and BM5 stations. During this period manpower was extremely limited and experiments could only be performed as external scientists could be invited. Valuable contacts to the PSI and HASYLAB were established, the latter led to the finally successful test of the SiGe gradient crystals.

4 Outlook

Substantial impact is expected from the demonstration of the fixed exit monochromator and the focussing analyzer on the future development of the ID11 extension station and application of focussed high energy radiation to materials science in general. After an intensive instrumentation phase the scientific possibilities provided by the ID11 extension station should be exploited in parallel with further optics development.

5 Publications

Lienert U., Poulsen H. F., Honkimäki V., Schulze C. and Hignette O., 'X-ray optics for the local structural characterization of materials with synchrotron radiation', J. Synchrotron Rad. (1999) **6** in print

U. Lienert, R. Martins, S. Grigull, M. Pinkerton, H.F. Poulsen, Å. Kvick, 'Local strain scanning by a reconstruction crossed-beam-technique', to be published

U. Lienert, S. Keitel, W. Caliebe, 'An energy tunable, fixed exit, broad band monochromator for high energy micro-focussing', to be published