ESRF	**Experiment title:** Experimental studies in x-ray phase retrieval	**Experiment number**: MI-599
Beamline: BM05	**Date of experiment**: from: 13.02.03 to: 18.02.03	**Date of report**: 20.08.03
Shifts: 12	**Local contact(s)**: Dr T. Bigault	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

***Dr Andrei Y Nikulin**, School of Physics & Materials Engineering, Monash University, Clayton, 3800, Australia.

***Dr Imants Svalbe**, School of Physics & Materials Engineering, Monash University, Clayton, 3800, Australia.

***Mr Russell Horney**, School of Physics & Materials Engineering, Monash University, Clayton, 3800, Australia.

Report:

A novel experimental technique [1] for the high-resolution mapping of materials has been expanded into a 2D diffraction imaging method. This, MI-599, experiment was a significant new development of the previous successful demonstration of this technique for amorphous materials carried out in previous experiments at the ESRF (MI-387 and MI-522) [2] and SPring-8, Japan [3].

We have successfully measured x-ray Fraunhofer diffraction data from non-Bragg diffracting samples for multiple angular rotations of the samples around their axis. The primary sample investigated was a square-shaped acetate filament of approx. 100 microns cross-section. We also collected Fraunhofer diffraction data from intertwined copper and constantan filaments. The diameter of the copper filament was 30 microns and the diameter of the constantan filament was 50 microns.

The measurements were performed on BM05 at the ESRF. The double-crystal Si(111) monochromator selected synchrotron radiation energies of 18.2 keV from the bending-magnet source. Highly asymmetric reflections Si(511), b = 0.03, from the Si monochromator and analyser pair were used in a non-dispersive set-up, to ensure a pseudo-plane wave was incident upon the sample. The beam incident on the sample was spatially collimated with a slit nominally 0.2 mm wide and 0.5 mm high. The diffracted x-ray intensities were collected as a function of deviation from the exact Bragg position of the analyser crystal and the angular rotation of the sample around its axis. The incident beam intensity was monitored using an ion chamber. Lead shielding was used to minimise the background count rate at the detector.

Figures 1 and 2 show the experimental data collected from the acetate and copper-constantan samples, respectively. The upper scan in Fig. 1 was recorded for the free propagating beam, i.e. there was no any sample in the beam path.

The PRXRD method was applied to each recorded scan to reconstruct 1D projections of the complex refractive index profile with spatial resolution of 1 micron (Fig. 3). Then a full 2D tomographic reconstruction was performed using these 1D projections. The centres of projections were located and padded with zeros to make them identical in width as required for tomographic reconstruction. Some artefacts of the PRXRD were removed and the positions of the thickness baselines estimated using the projection integrals (since these integrals should be identical). Each projection comprised between 30

and 57 data points. They were cubic-spline interpolated to increase the number of ray-sums to a uniform 151 for reconstruction. Interpolation between projections was also required to increase the number to 180 to enable smooth reconstruction. It was necessary to assume some approximate symmetry properties of the sample (some first quadrant projections were used to supplement missing projections in the second quadrant). The sparse data and the nature of the sinogram of a square object cause various artefacts in the reconstruction, therefore, special procedures for tomographic reconstruction of sparse data sets are currently being assessed in an effort minimise their effects. Images were reconstructed using the 2D Fourier inversion procedure over 512 Fourier points [4].

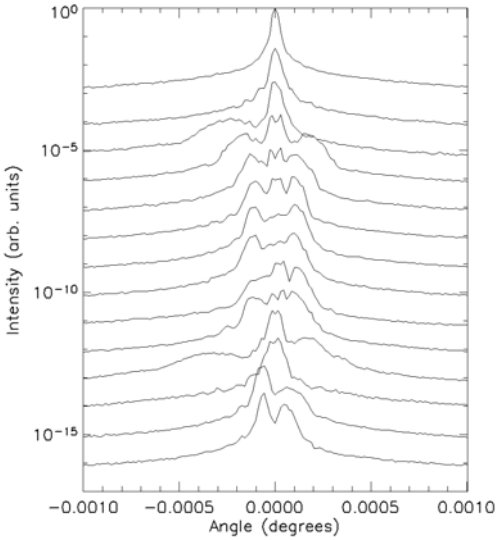

Figure 1: Experimental scans collected from the acetate sample for different angular rotation position of the filament. The scans are shifted by one order of magnitude for better visibility

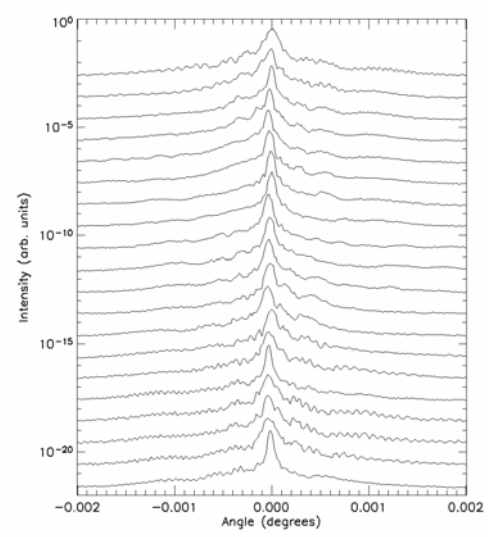

Figure 2: Experimental scans collected from the acetate sample for different angular rotation position of the filament. The scans are shifted by one order of magnitude for better visibility

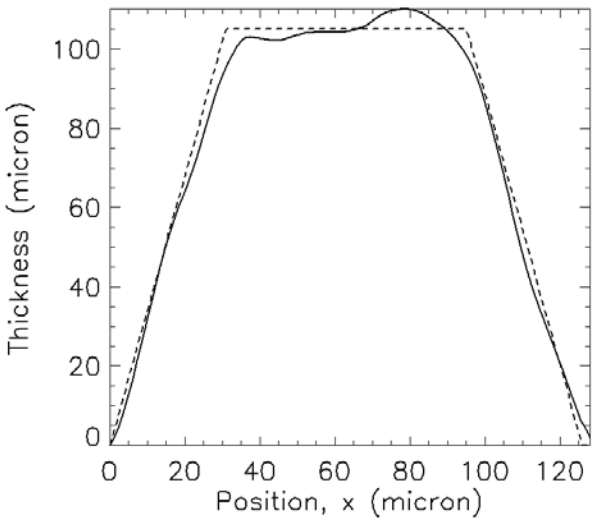

Figure 3: Reconstructed 1D thickness projection of the acetate sample for 20-degree position of the sample.

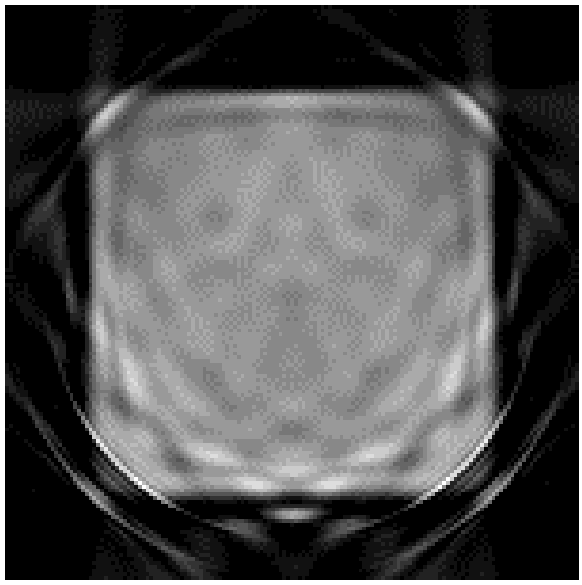

Figure 4: Reconstructed 2D thickness projection of the acetate sample (preliminary results).

References

1. A. Y. Nikulin, in *"Recent Research Developments in Applied Physics,"* eds. M. Kawasaki, N. Ashgriz, R. Anthony, Research Signpost, 1998, p. 1.
2. K. Siu, A. Y. Nikulin, K. Tamasaku and T. Ishikawa, *Appl. Phys. Lett.,* 2001, **79** 2112.
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4. A. Y. Nikulin, R. B. Horney, A. V. Darahanau, I. D. Svalbe, T. Bigault, E. Ziegler (in preparation).