ESRF	**Experiment title:** **Experiments in x-ray phase retrieval for refraction/small angle scattering studies**	**Experiment number**: MI-387
Beamline: BM5	**Date of experiment**: from: 03 May 2000 to: 08 May 2000	**Date of report**: 12 January 2001
Shifts: 15	**Local contact(s)**: Dr Eric Ziegler	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*** Dr. Andrei Y. Nikulin**, Department of Physics, Monash University, Clayton, 3800, Australia.

*** Dr. John R. Davis**, Department of Physics, Monash University, Clayton, 3800, Australia.

*** Ms. Karen Siu**, Department of Physics, Monash University, Clayton, 3800, Australia.

Report:

The experiment was performed on the European Synchrotron Radiation Facility (ESRF), BM5 (BL-10) beamline, Grenoble, France. A primary, tunable double-crystal Si(111) monochromator selected synchrotron radiation energy in the vicinity of 17.48 keV (λ = 0.71 Å) from the bending-magnet source. Highly asymmetric (333) reflections (b=0.04) from the Si(111) 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 50 μm wide and 1 mm high. The incident beam intensity was monitored using an ion chamber. Lead shielding was used to minimise the background count rate at the detector. The experimental setup is shown in figure 1.

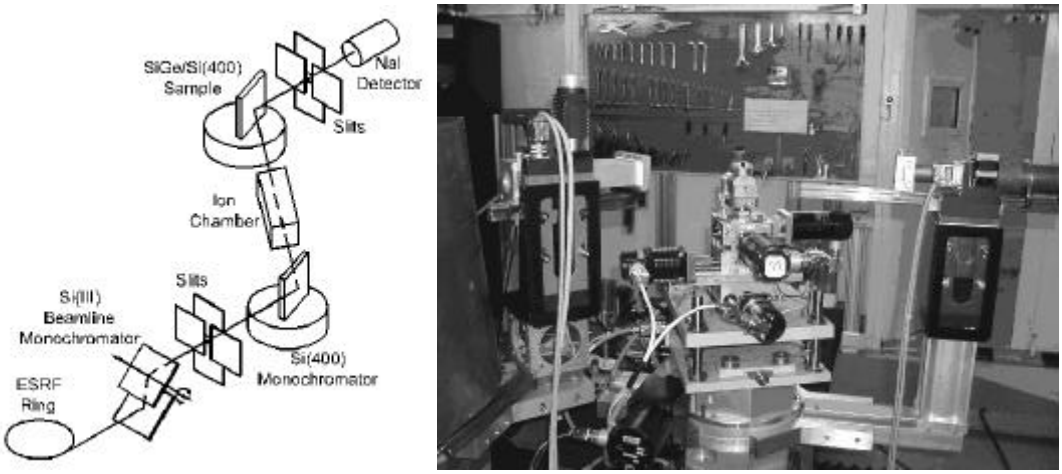

Figure 1: Experimental setup shown schematically (left) and as built at BM05 (right)

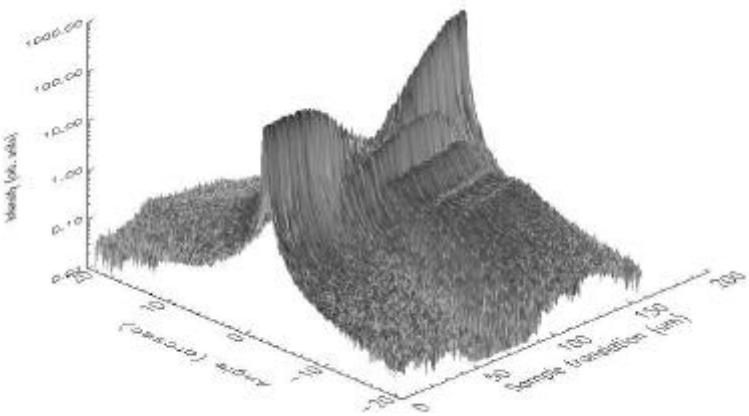

Figure 1: Copper filament scan, 50 μm diameter, 17.48 keV

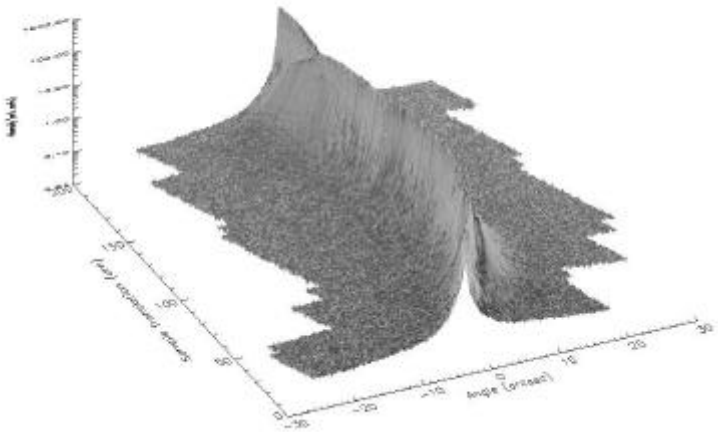

Figure 3: Optical fibre scan, 125 mm silica with 8 mm Ge doped core, 17.48 keV

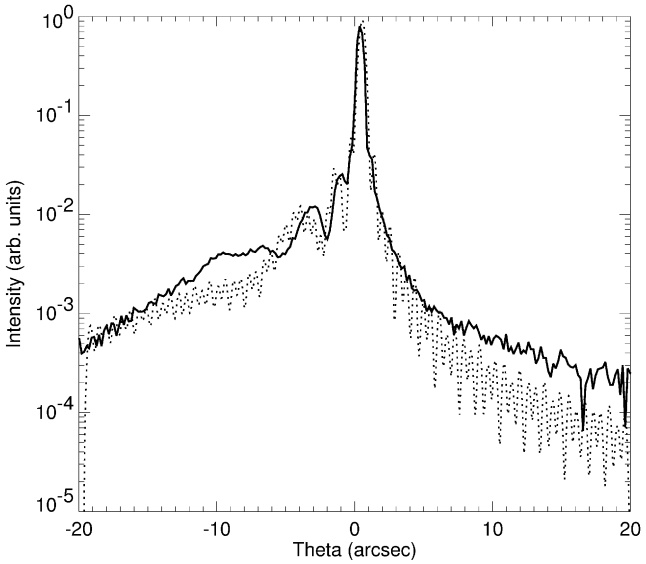

Figure 4: Data (solid line) for the Cu sample, approx. half occluded by the slit, compared to the theoretical simulation (dashed line).

This experiment collected data as a function of both angular deviation from the Bragg peak and sample translation perpendicular to the beam direction. "Two dimensional" scans were completed for both a 50μm diameter copper filament and a 125μm optical fibre (silica cladding, 8.2μm Ge doped silica core) samples. The data resolution corresponds to 0.5μm increments in real space. The resulting two dimensional scans clearly demonstrated the refraction effects in the samples.

The rocking curve scans from the copper sample show strong satellite peaks as well as broader features either side of the Bragg peak, which change position relative to the Bragg peak as a function of sample translation (figure 2). The effect of the sample attenuation is markedly evident at the centre of sample translation, which corresponds to the sample being centred with the slit centre. There is evidence of periodic features in the direction of the sample translation for a given angle also. The rocking curve scans from the optical fibre sample also shows satellite peaks either side of the main Bragg peak (figure 3). This main peak, appears to "shift" in the angular direction. Although some mechanical drift was in evidence during data collection, at least some of this shift is attributable to refraction effects. This is evident at the centre region of the translation, where the higher density core material, coincident with the centre of the slit, reverses the shift from the positive to the negative direction.

The improved source characteristics and crystal optics have led to much closer agreement with theoretical simulations than observed in previous experiments, as shown in figure 4. In particular, the possibility of source collimation in directions both parallel and perpendicular to the diffraction plane, and mono/analyser crystals with higher asymmetry provided an incident beam of extremely low divergence.

This experiment has provided data suitable for phase retrieval by both the PRXRD and Gerchberg-Saxton algorithms. Comparison of these two techniques will form an important test of the applicability of the PRXRD technique to refraction/small-angle scattering data.

1. Nikulin A., "Phase retrieval x-ray diffractometry: A tool for unambiguous characterization of crystalline materials." In: "Recent Research Developments in Applied Physics," eds. M. Kawasaki, N. Ashgriz, R. Anthony, *Research Signpost*, 1998, p. 1.
2. R.W. Gerchberg and W.O. Saxton, "A practical algorithm for the determination of phase from image and diffraction plane pictures", Optik 35, 237-246 (1972).