ESRF	**Experiment title:** Experimental studies in x-ray phase retrieval	**Experiment number:** MI-522
Beamline: BM05	**Date of experiment:** from: 5.09.01 to: 9.09.01	**Date of report:** 18.09.01
Shifts: 12	**Local contact(s):** Dr T. Bigault	*Received at ESRF:*

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Report:

A novel experimental technique for the comprehensive characterisation of non-crystalline structures has been further investigated. The technique, phase retrieval x-ray diffractometry (PRXRD), involves the retrieval of the scattered x-ray phase via a logarithmic dispersion relation [1]. This experiment was a further development of the first successful demonstration of this technique for amorphous materials carried out in previous experiments at the ESRF (MI-387, May 2000) [2] and SPring-8, Japan [3].

This experiment successfully measured refraction/small angle scattering phenomena from an amorphous sample of low molecular weight, with x-ray properties approaching those of a "biological" material. The primary sample investigated was a polyimide film (Dupont "Kapton") with a 20 μm period depth variation across the surface created by excimer laser ablation at the Industrial Research Institute Swinburne (IRIS). In particular, this experiment measured the scattered x-rays at two incident energies to provide an unambiguous solution to the phase retrieval problem. Also investigated

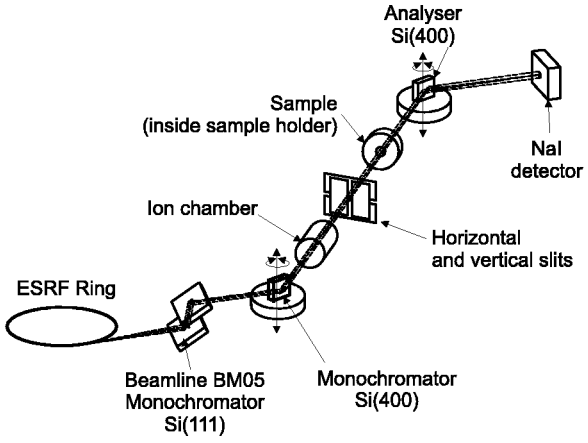

Figure 1: Experimental setup

were the x-ray scattering from an optical fibre (125 μm silica cladding diameter with 8.2 μm Ge doped core), and human hair (nom. 70 μm diameter).

The experiment was performed on BM05 at the ESRF. The experimental setup is shown in figure 1. A primary, tunable double-crystal Si(111) monochromator selected synchrotron radiation energies of 7.5 keV and 15.9 keV from the bending-

magnet source. Highly asymmetric reflections (Si(400), b = 0.023 at 7.5 keV, and Si(511), b = 0.038 at 15.9 keV respectively) 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 1.5 mm wide and 0.5 mm high. The scattered x-ray intensities were collected as a function of deviation from the exact Bragg position of the analyser crystal. The incident beam intensity was monitored using an ion chamber. Lead shielding was used to minimise the background count rate at the detector.

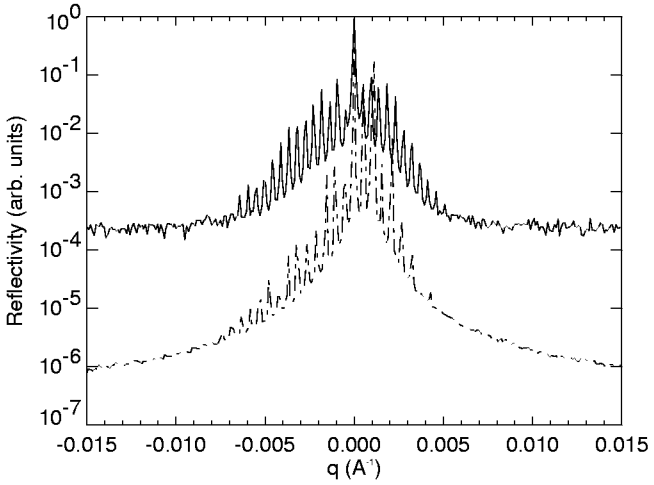

Figure 2: Analyser reflectivity profiles for kapton sample at 15.9 keV (solid line) and 7.5 keV (dot-dashed line)

Figure 2 shows the data collected from the Kapton sample at 7.5 keV (solid line) and 15.9 keV (dashed line). The reflectivity profiles indicate that the x-ray scattering from this low molecular weight material, even at the relatively hard energy, is profound. The amplitude contrast between the two energy profiles is clearly noticeable. Data at two incident energies is required to resolve the problem of finding the roots of the complex polynomial necessary to uniquely retrieve the x-ray phase. This data should thus allow the unambigous retrieval of the x-ray phase and hence comprehensive characterisation of the sample.

Figure 3 shows the reflectivity profile for the optical fibre, examined at 15.9 keV. The core structure is clearly evident in the satellite fringes away from the Bragg peak. Although such refraction effects have previously been observed in studies of this sample (eg. [2]), the broader extent of the structure in the reflectivity profile indicates a higher real space resolution in this instance. This improvement is a direct result of the lower angular divergence offered by the improved optics coupled with the unique collimation in both the parallel and perpendicular directions to the diffraction plane of this beamline. Figure 4 shows the reflectivity profile for a human

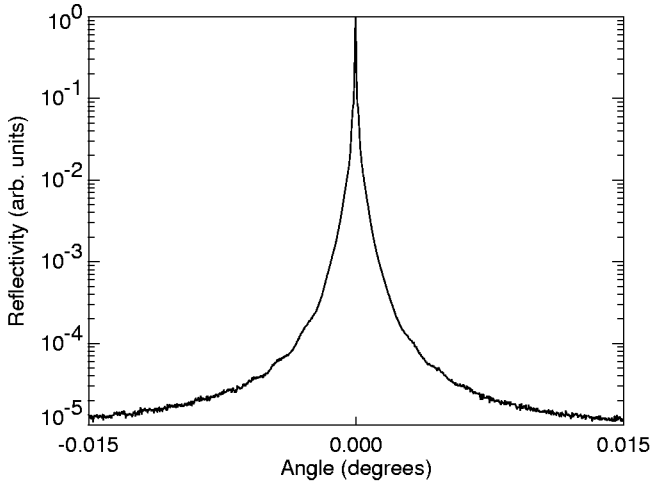

Figure 3: Analyser reflectivity profile from 125 μm diameter optical fibre at 15.9 keV

hair of nominally 70 μm diameter, also at 15.9 keV. Again, strong refraction phenomena are clearly visible in the reflectivity profile, which were not observed in previous attempts to characterise human hair.

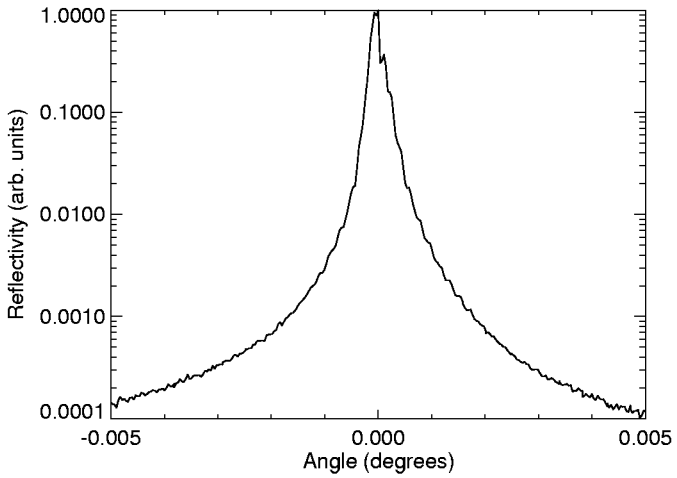

Figure 4: Analyser reflectivity profile for human hair (nom. 70 mm diameter) at 15.9 keV

The successful observation of refraction effects from the kapton and the human hair samples, particularly at relatively hard x-ray energies, indicate that the phase retrieval technique could sucessfully be used to characterise genuine biological structures. Such hard energies are advantageous to minimise the dosage when characterising samples of medical interest. PRXRD would be particularly useful for such amorphous samples of low molecular weight as the amplitude contrast is poor, whilst the phase contrast is relatively marked.

References

1. A. Y. Nikulin, "Phase retrieval x-ray diffractometry: A tool for unambiguous characterisation of crystalline materials", in *"Recent Research Developments in Applied Physics,"* eds. M. Kawasaki, N. Ashgriz, R. Anthony, Research Signpost, 1998, p. 1.
2. A. Y. Nikulin, J. R. Davis, K. Siu, E. Zeigler, "Experiments in x-ray phase retrieval for refraction/small angle scattering studies," *ESRF Annual Report 2000*, (Users' Reports) *submitted.*
3. K. Siu, A. Y. Nikulin, K. Tamasaku and T. Ishikawa, "An application of phase retrieval x-ray diffractometry to refraction/small-angle scattering data, " *J. Phys. D: Appl. Phys.* **34**, 2912-2917.