High Pressure
GeniX Mo Small Spot

Application Note n° AN-G3

Xenocs
Reflecting Future Technology

Performing High Pressure diffraction experiments using Xenocs' GeniX Mo Small Spot beam delivery system

Data courtesy of Federico Gorelli, LENS (European Laboratory for Non-Linear Spectroscopy), Florence, Italy

Abstract

Xenocs' GeniX Mo Small Spot delivery system is composed of a microfocus sealed tube coupled to a single reflection multilayer focusing optic. High pressure (HP) experiments were performed by Dr. Federico Gorelli at LENS in Florence, Italy. The diffraction pattern of a fine quartz powder was measured successively with a traditional sealed tube/monocapillary combination and with the GeniX Mo Small Spot. A second measurement was performed on a sample of polyethylene (PET) and the data compared to a PET diffraction pattern measured at the European Synchrotron Radiation Facility (ESRF).

The properties of the monochromatic 17,4 keV beam delivered by the GeniX Mo Small Spot micro-beam system are well suited for performing X-ray diffraction (XRD) experiments on samples under high pressure placed inside a Diamond Anvil Cell (DAC). The small focal spot size and the high brilliance of the beam enable high resolution and a very good S/N ratio, both of which are particularly useful for performing successful in house HP XRD experiments.

LENS

Fig. 1 : The GeniX mounted in the High Pressure Diffractometer present at the High Pressure Facility at LENS

Introduction

The GeniX Mo Small Spot delivery system is composed of a microfocus sealed tube coupled to a single reflection multilayer focusing optic. The system delivers a beam with a spot size at focus of 100 x 100 µm² FWHM. The beam divergence is 0.23 degrees and the flux is 2×10^6 photons/s. The X-ray beam delivered by this source is ideal for performing X-ray diffraction experiments on samples under high pressure placed inside a Diamond Anvil Cell (DAC). The sample dimensions depend on pressure, but generally are about 100 µm in diameter and 50 µm in thickness. The X-ray beam should then be the best compromise among beam size at focus, beam divergence, and flux. The objective of these tests is to determine whether the GeniX system provides the right level of performances for in-house HP XRD experiments.

Experiment

For all experiments the GeniX Mo Small Spot system was operated at 50 Watts (50 kV, 1 mA). The sample in the DAC was positioned at the focal plane of the X-ray beam, at 250 mm from the centre of the mirror, using motorized translation stages to allow precise alignment. The detector consisted of a Princeton Instruments CCD camera (PI SCX 4300) cooled down to -50°C and positioned at 40 mm from the sample.

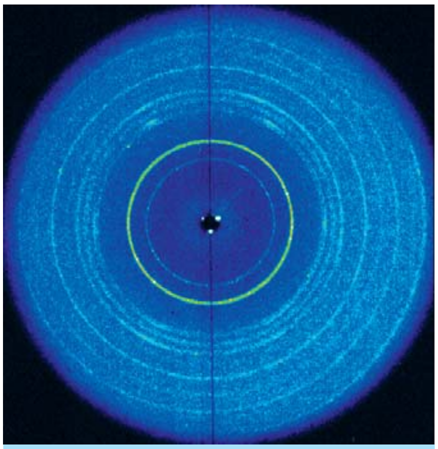

Fig. 2 : Diffraction rings (obtained using the GeniX) from quartz powder (SiO_2) placed inside the DAC. The vertical black line is the shadow of the beam stop holder.

The first sample loaded into the DAC consisted of a fine quartz powder that was embedded in a Rhenium gasket. The sample dimensions were 130 µm in diameter and 60 µm in thickness. In order to compare data quality successive measurements were performed; first with a traditional sealed tube/monocapillary system, then with the GeniX Mo Small Spot. The sealed tube was operated in fine focus mode (0.4 x 0.6 mm² source size) with a 50 µm Zr filter and a monocapillary optic.

The Debye–Scherrer rings obtained using the GeniX and the PI CCD are reported in Fig. 2. The integration time is 1800 seconds (3 accumulations of 600 seconds each). The diffraction pattern, obtained by 2D integration of the Debye–Scherrer rings, is reported in Fig. 3. The upper curve corresponds to the sealed tube and the lower curve to the GeniX.

Comparing the two data sets reveals that although the flux intensity for the measurement acquired using the GeniX Mo Small Spot is lower, the signal-to-noise ratio is much improved and a better resolution is obtained. This is not surprising since the brilliance of the GeniX Mo Small Spot is over 40 times that of the traditional sealed tube/monocapillary system, in part due to the fact that the Genix with its multilayer mirror delivers a monochromatic beam, while the sealed tube/monocapillary system delivers a polychromatic beam.

The diffraction pattern presented in Fig. 3 reveals a FWHM of 0.27 degrees for the curve obtained with GeniX Mo Small Spot, only slightly higher than the nominal X-ray beam divergence. The background profile is due to Compton scattering from the diamonds and from air. The two most intense Re gasket lines appear as shoulders on the SiO_2 lines (marked by an asterisk). At about 27 degrees the intensity rapidly decreases due to the maximum acceptance angle of the DAC.

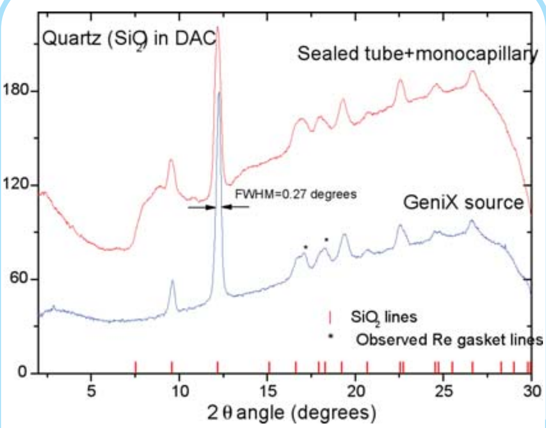

Fig. 3 : Diffraction pattern obtained by 2D integration of the Debye–Scherrer rings shown in figure 2.

A second test was performed on a sample of Polyethylene (PET). The PET powder was pressurized in the DAC to 0.02 GPa and then annealed at high temperature (250°C). The PET Debye–Scherrer rings are reported in Fig. 4. The experimental setup is the same as described above, but the integration time was 15 hours (90 accumulations of 600 seconds each). The diffraction pattern, obtained by 2D integration of the diffraction rings is reported in Fig. 5 as a function of d-spacing. In the same figure the diffraction pattern from a similar PET sample pressurized at 2.5 GPa and measured at the ID9 beamline of the European Synchrotron Radiation Facility (ESRF) is reported for comparison. In this last case the X-ray beam wavelength was 0.4108 Å, almost the half that of the Mo K-alpha wavelength (0.71069 Å). For a better comparison the two diffraction patterns are reported as a function of the d-spacing. As expected, the diffraction pattern measured with the GeniX source and the PI CCD detector is about three orders of magnitude less intense and the line width about 4 times larger than in the pattern measured at the ESRF with the MAR imaging plate. Nevertheless the most important features of the diffraction pattern are still readily apparent in the measurements performed at LENS, demonstrating that these types of experiments can be successfully performed with an in-house diffractometer using the GeniX Mo Small Spot micro-beam generator system.

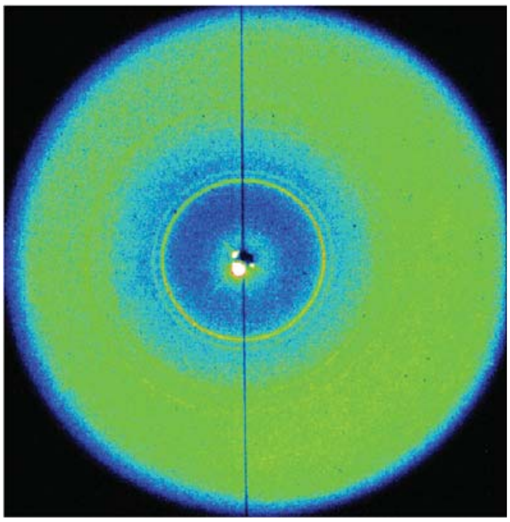

Fig. 4 : Diffraction rings (obtained using the GeniX) from polyethylene powder (PET) placed inside the DAC. The vertical black line is the shadow of the beam stop holder.

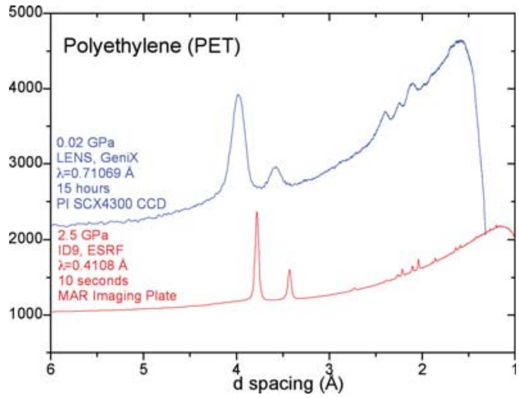

Fig. 5 : The diffraction pattern from PET obtained by 2D integration of the Debye–Scherrer rings shown in Fig. 4. The different d-spacing of the diffraction lines is due to the different pressure of the two measurements.

Conclusion

The GeniX Mo Small Spot micro-beam delivery system proves to be a high performance alternative to the traditional in-house X-ray installation. With its small focal spot and high beam brilliance, the GeniX results in improved resolution and signal-to-noise compared to the traditional sealed tube/monocapillary system, making the GeniX a valuable instrument for in-house HP XRD installations.

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