| ESRF | **Experiment title:** A partial structure factor determination of Ge02 and P3Se4 glass using combined x-ray anomalous scattering and neutron diffraction data. | **Experiment number:** HS033 |
|---|---|---|
| **Beamline:** ID 1 | **Date of experiment:** from: 15 Sept 1997 to: 21 Sept 1997 | **Date of report:** 12-August-1998 |
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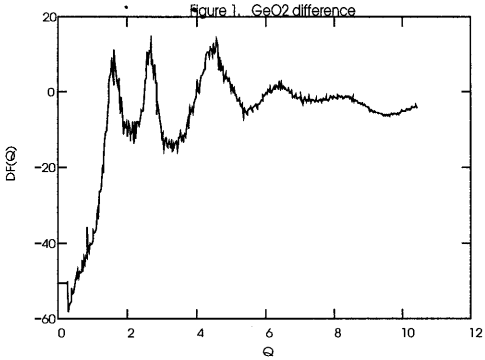
**Report:** This experiment was originally submitted in 1996 but was delayed due to the late commissioning of ID01 . After initially being scheduled for May 1997 it finally took place in September 1997.

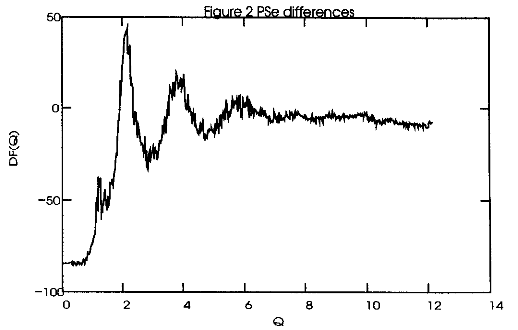
The aim of the experiment was to investigate the new opportunities for exploiting the X-ray anomalous scattering technique as method for determining partial structure information on amorphous materials. The development of the ID01 diffractometer at the ESRF gives considerable advantages over previous methods. These are:

- very high incident intensity with high energy resolution ($\Delta E/E<10^{-4}$).
- High energy resolution on the diffracted beam using perfect crystal analysers.
- Incident energies up to 40 keV with this high resolution.

Two samples were measured in the diffraction experiment:

- Glassy Ge02 at 10 eV and 200 eV away from the Ge K edge at 11.103 keV. This corresponds to a wavelength of 1.1 Å with a maximum Q of ~ 10.8 $Å^{-1}$.
- Glassy Pse at 10 eV and 200 eV away from the Se K edge at 12.658 keV. This corresponds to a wavelength of 0.98 A with a maximum Q of -12 $Å^{-1}$.



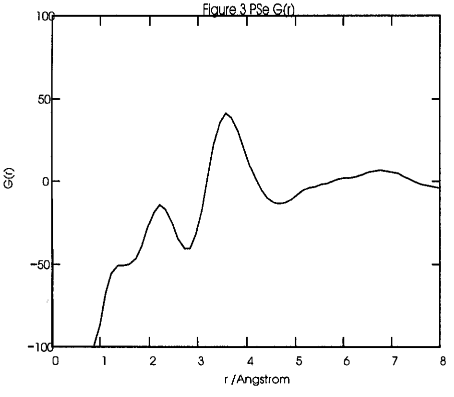
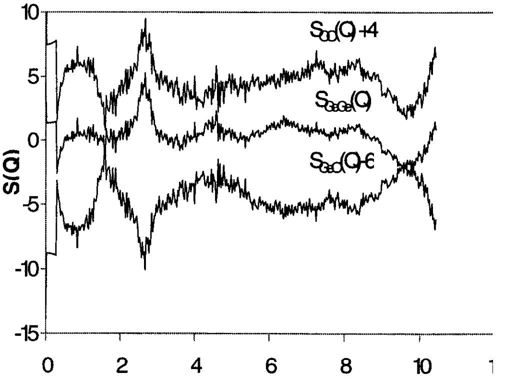




Figure 4 GeO2 partials from X-ray/neutron data




The data have been normalised and corrected for the self attenuation of the sample. No corrections have been made for Compton scattering or multiple scattering which are effectively removed by the analyser crystal. It is this analyser, with it's high energy resolution, which is the key advantage of IDl. Figure 1 shows the difference spectrum $\Delta F(Q)$ obtained from the $GeO_2$ sample. Figure 2 shows the difference spectrum obtained from the PSe sample. The absence of the first sharp diffraction peak in this function is immediately noticeable. The statistical precision obtained in the 6 hour scans can clearly be observed. Figure 3 shows the Fourier transform $\Delta G(r)$ of the PSe difference function. The peak at -2.2 Å corresponds to the P-Se distance and gives a coordination number of 2 consistent with the expected coordination of Se according to the S-N rule. Figure 4 shows the partial structure factors obtained by combining the $Ge0_2$ data with the neutron diffraction data of Wright et. al. taken using the D4 diffractometer at the ILL. The small Q range still needs to be corrected for the differing resolutions of the neutron and X-ray diffractometers. The results are in agreement with similar experiments we have carried out using X-ray/neutron data from the NSLS/IPNS in the U.S.A. The data are currently being fully corrected and interpreted and will be published soon. The results of this experiment encourage us to pursue the development of the anomalous scattering technique and its combination with neutron scattering data.