Beamtime report CH2807

Combined high resolution powder X-ray diffraction and XAS data collection on ITQ-7

Our beamtime was shortened due to difficulties with setting up the capillary in situ cell on site. Secondly we also experience flow gradients due to our samples being very compact powders. This was solved by preparing sieved fractions of our samples. We collected high resolution powder X-ray diffraction and transmission XAS data during thermal treatment of an ITQ-7 system. The XAS data were collected in the quickscan mode, but later EXAFS analysis found this to be a poorer quality than what we usually expect from SNBL.

The thermal stability of the sample was tested by calcination with a 21% oxygen in helium mixture, ramp rate 5 degrees per minute, using a flow of 5 ml/min through the cell. The sample was heated by a blower while collecting quick XAS scans. Powder diffraction data was collected at selected temperatures. The output was analysed by a Pfeiffer vacuum Omnistar mass spectrometer.

Results

High resolution X-ray powder diffractograms of ITQ-7 during calcination is shown in Fig. 1, confirm that the phase is contained during removal of the template. However, there are reduced relative intensities after template removal. The normalised XANES for as-synthesised ITQ-7 at selected temperature intervals is shown in Fig. 2 compared to the model compound, amorphous GeO_2. The absorption edge, E_0, of the ITQ-7 sample was set to 11108.8 eV which is shifted +1.2 eV compared to that of amorphous GeO_2. The absorption edge value of ITQ-7 is comparable to that reported for quartz type germanium oxide in which germanium(IV) is tetrahedrally coordinated. The as-synthesised ITQ-7 shows only one broad feature after the white line at +16.9 eV in the XANES. These high energy XANES features have been reported to orginate from scattering within the GeO_4 tetrahedron. The XANES of ITQ-7 show a reduction of the intensity of the white line upon calcination to 650°C. In addition a feature at +11.2 eV appears at 400°C which indicates disorder beyond the first shell due to a distribution of intertetrahedral angles. It appears that the removal of the template leads to a higher degree of disorder in the system, as the feature is also apparent in the XANES of amorphous GeO_2 but not in for the quartz type oxide.

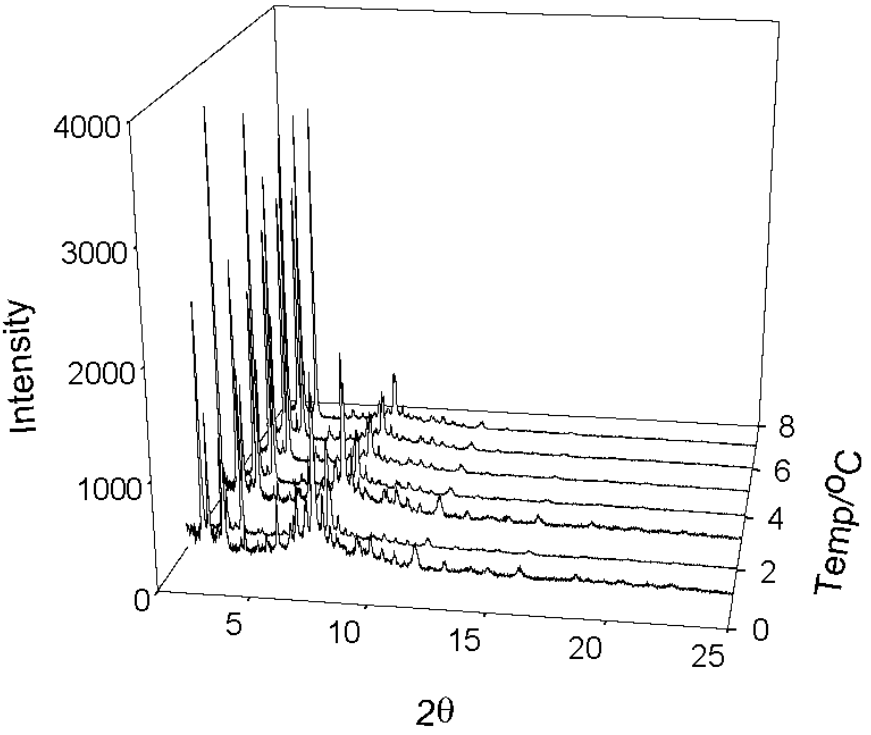

Fig. 2 High resolution X-ray powder diffractograms of ITQ-7 during calcination.

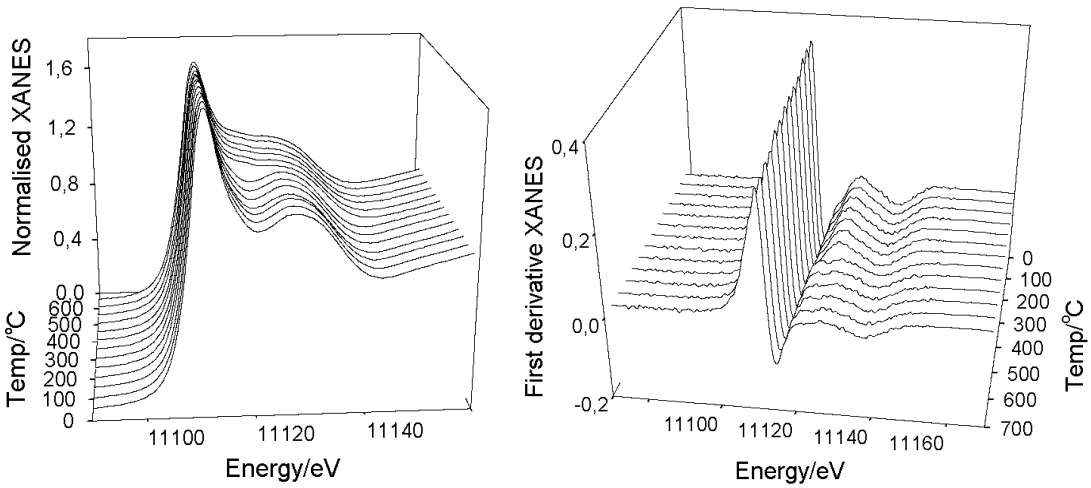

Fig. 2 Normalised (left) and first derivative (right) XANES for ITQ-7 during calcination in 5% oxygen.