

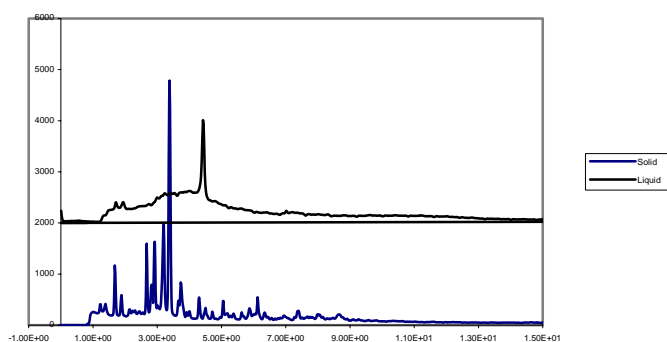
	Experiment title: The liquid-liquid transition in molten jadeite and its relation to sodium	Experiment number:
Beamline: ID30	Date of experiment: from: 31 October to: 4 November 2001	Date of report: 30/08/202
Shifts: 15	Local contact(s): Wilson Crichton	<i>Received at ESRF:</i>
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Report:

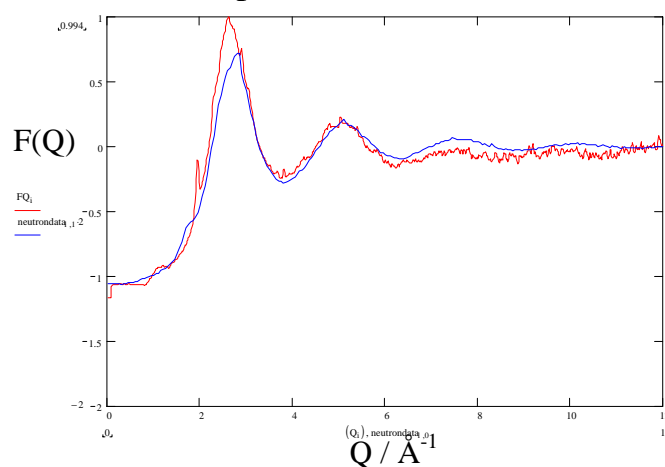
The aim of this experiment was to carry out high pressure/high temperature measurements of two molten silicates of geophysical interest. The target was to identify if there are any structural changes taking place in these liquids that corresponds to the sharp change in viscosity observed in the melts in the 1-1.5 GPa range at temperatures of 1300-1400°C. These conditions were particularly challenging for the ID30 pressure setup.

Experimental measurements on liquid Albite ($\text{NaAlSi}_3\text{O}_8$) and Jadeite ($\text{NaAlSi}_2\text{O}_6$) were carried out pressures of ~0.75 GPa and ~1.5 GPa and temperatures between ambient and 2500K. The experimental setup up proved to be extremely difficult as these low atomic mass samples showed very little contrast with that of the surrounding container, heating element and gasket of the Paris-Edinburgh cell in which the experiments were carried out. This made it particularly difficult to achieve accurate sample positioning through transmission measurements. The problem was partially resolved by the inclusion of a thin highly absorbing Pt foil in the cell as a positional marker. This also acted as a useful additional pressure and temperature marker in addition to that from the boron nitride sample container. Melting and recrystallization of the materials was successfully observed in the experiments. The figure below shows the diffraction pattern obtained from the sample at 0.75 GPa and 1.5 GPa at a temperature of 2250K. The clear change from the molten to the crystalline state is visible. At the current time a detailed analysis of the results is taking place and is aimed at careful corrections for the background scattering due to the gasket and heater materials.

Due to the difficult sample positioning problems we also decided to carry out experiments on both CuI and Ag₂Se samples that had known phase diagrams. Measurements on these were used to establish our ability to obtain good pressure and temperature calibrations for samples in the Paris-Edinburgh cell. The advantage of these materials was that they were high Z and therefore easy to locate in the transmission measurements. For the case of Ag₂Se we particularly keen to look at the liquid phase as very high quality neutron diffraction data exist for this liquid (at the partial structure factor level) so that we have a good indicator of the ability to carry out accurate data corrections for the X-ray diffraction data taken in the complex sample environment of the Paris-Edinburgh cell. The figure shows Faber-Ziman weighted X-ray and neutron total structure factors for high temperature liquid Ag₂Se at low pressures (~0.5 GPa) compared to the neutron diffraction data at ambient pressure. The agreement, given the slight difference in weighting from the scattering lengths and X-ray form factors, excellent. We are therefore encouraged that we can use this system to undertake accurate measurements on high Z materials with the current setup.



Diffraction pattern from Albite at 2250K. Top corresponds to a pressure of ~0.75 GPa and the bottom to recrystallization as the pressure was increased to 1.5 GPa. The strong peak in the liquid pattern is due to gasket scattering.



Comparison of the X-ray total structure factor of liquid Ag₂Se at 0.5 GPa with neutron total structure factor at ambient temperature.