

Investigation of Liquid Structure Using Aerodynamic Levitation Melting and Large-Area X-ray Detectors

R. Weber,¹ J.E. Rix,¹ J. Kim,¹ C.J. Benmore,² Q. Mei,² M.C. Wilding,³

¹Containerless Research, Inc., Evanston, IL, U.S.A., ²Argonne National Laboratory, Argonne, IL U.S.A.;

³The University of Wales, Aberystwyth, U.K.

Introduction

Investigation of structural evolution in liquids is of interest in connection with materials processing and development of value added products and to enhance the fundamental understanding of the liquid state and non-equilibrium materials. The use of high energy X-ray diffraction provides a means to achieve fast measurements over a large Q-range and probe the bulk liquid.

Methods and Materials

The sample environment was controlled using aerodynamic levitation in UHP argon and 240 W continuous-wave CO₂ laser beam heating. Similar techniques have been applied in prior work [1]. They access high purity liquids at high temperatures and in the superheated and undercooled states. The levitator was enclosed in a stainless steel chamber equipped with Kapton windows that transmit X-rays with virtually no attenuation or scattering. The instrument was integrated with beamline 11 ID-C at the Advanced Photon Source. The X-ray energy was 115 keV, scattered X-rays were detected using a MAR 345 detector.



Fig. 1. Photograph of the instrument installed at the beamline. The round plate is the X-ray detector, the X-ray beam enters the chamber through the port on the right. The system was aligned by adjusting the chamber with a CeO₂ test sample located in the levitation position. The inset shows a levitated liquid sample (at arrowhead) viewed through the front port of the chamber.

Samples were 0.2-0.3 cm diameter spheroids made from: (i) binary Al₂O₃-CaO compositions containing 50 and 64 molar % CaO, (ii) binary Al₂O₃-Y₂O₃ compositions containing 0 to 37.5 mole % Y₂O₃, (iii) binary MgO-SiO₂ compositions containing 33.3-50 mole % SiO₂, and (iv) Zr-based metallic glass forming alloys. Output from the detector was acquired by computer. Background, geometrical, and polarization corrections and a correction for oblique incidence of the beam on the image plate were applied. The resulting data were corrected for Compton scattering and form factors to obtain S(Q).

Results

Data were acquired over a temperature range from 1000°C for the metallic material to 2050°C for the YAG composition liquid. The time to acquire X-ray data was 5-30 seconds and it was determined empirically to achieve a high signal without saturating the detector. Readout of the detector took ~90 seconds. S(Q) data for two glasses made from 36/64 Al₂O₃-CaO and the liquid at 1900°C and liquid CaAl₂O₄ at 1900°C are shown in Fig. 2. We report apparent temperatures.

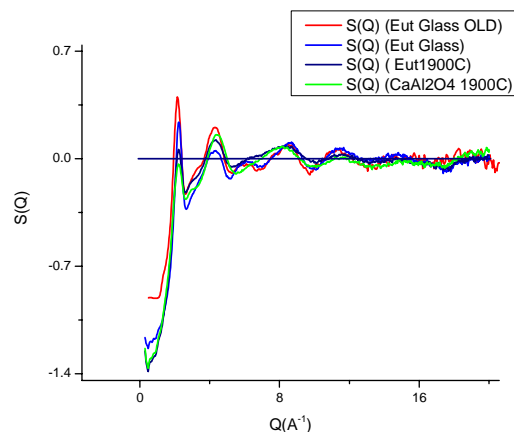


Fig. 2. S(Q) data for CaO-Al₂O₃ binaries.

Discussion

The preliminary experiments demonstrate that combining a containerless sample environment, high energy X-rays, and a large area detector achieves measurements of liquid structures in a period of a few seconds. The use of a direct readout X-ray detector would allow fast sequencing of measurements, enabling studies of structural evolution in stable and metastable liquids.

References

[1] S. Krishnan, *et al.*, Rev. Sci. Instrum. **68**, 3512-3518 (1997).

This work was supported in part by: NASA grant number NNM04AA23G and U.S. Department of Energy contract number W-31-109-ENG-38.