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Toward comprehensive studies of liquids at high pressures and high temperatures: Combined structure, elastic wave velocity, and viscosity measurements in the Paris–Edinburgh cell

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ABSTRACT

Techniques for measuring liquid structure, elastic wave velocity, and viscosity under high pressure have been integrated using a Paris–Edinburgh cell at Beamline 16-BM-B, HPCAT of the Advanced Photon Source. The Paris–Edinburgh press allows for compressing large volume samples (up to 2 mm in both diameter and length) up to ~7 GPa and 2000 °C. Multi-angle energy dispersive X-ray diffraction provides structure factors of liquid to a large Q of ~19 Å. Ultrasonic techniques have been developed to investigate elastic wave velocity of liquids combined with the X-ray imaging. Falling sphere viscometry, using high-speed X-ray radiography (>1000 frames/s), enables us to investigate a wide range of viscosity, from those of high viscosity silicates or oxides melts to low viscosity (<1 mPa s) liquids and fluids such as liquid metals or salts. The integration of these multiple techniques has promoted comprehensive studies of structure and physical properties of liquids as well as amorphous materials at high pressures and high temperatures, making it possible to investigate correlations between structure and physical properties of liquids *in situ*.

1. Introduction

Knowledge on the structure and physical properties of liquids at high pressures and high temperatures is important in understanding dynamics and evolution of the interiors of the Earth and other planets. In materials science, liquid materials possess many properties that are distinct from solids, with unique applications to industrial and engineering. However, understanding the physics of liquid materials remains a challenge, especially under high pressure. Structure and physical properties of liquids have been much less studied than those of crystalline materials due to experimental difficulties. Some efforts have been made to investigate structure of liquids (e.g., Tsuji et al., 1989; Mezouar et al., 2002; Shen et al., 2004; Yamada et al., 2011), physical properties such as density (e.g., Katayama et al., 1998; Shen et al., 2002; Ohtani et al., 2005), viscosity (e.g., Kushiro et al., 1976; Kanzaki et al., 1987; Dobson et al., 2000; Terasaki et al., 2001; Perrillat et al., 2010), and elastic wave velocity (e.g., Krisch et al., 2002; Decremps et al., 2009; Nishida et al., 2013). However, these results were often based on individual techniques, and the discussions were made by comparisons with results obtained by other researchers using in different apparatus using different techniques. Integration of these techniques should promote a more comprehensive understanding of the behavior of liquids at high pressures and high temperatures. In this paper, we report a new experimental setup which integrates liquid structure, elastic wave velocity, and viscosity measurements in a Paris–Edinburgh cell at high pressures and high temperatures at Beamline 16-BM-B, HPCAT, the Advanced Photon Source (APS).

2. Beamline overview

Fig. 1 shows the experimental setup in the 16-BM-B experimental station, which is capable of white beam X-ray diffraction and radiography experiments using a Paris–Edinburgh (PE) cell. The PE cell allows compression of large sample volumes (up to 2 mm in both diameter and length) at high pressures and high temperatures. Liquid structure studies are conducted by the multi-angle energy-dispersive X-ray diffraction (EDXD) technique. A large Huber stage holding a Ge solid state detector (Ge-SSD) allows precise control 2θ angle from 2° to 39.5°. Elastic wave velocities can be determined *in situ* by measuring both elastic wave travel times using ultrasonic techniques and sample length using white X-ray radiography. For sample length measurement, a CCD camera (Prosilica GC1380) was used. The camera can be moved in and out of the beam vertically to avoid interference with the collimator for X-ray diffraction measurement. Another camera, a high-speed Photron SA3, is located at the downstream side of the Ge-SSD. The SA3

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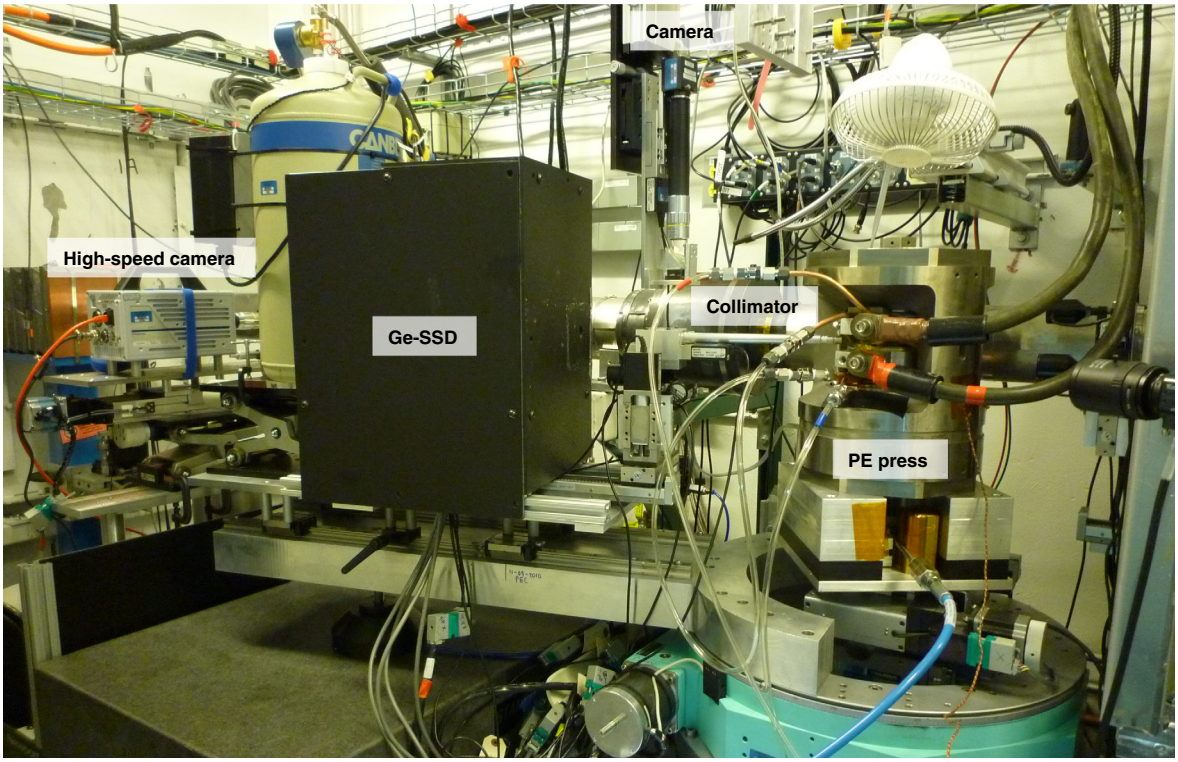

Fig. 1. A photograph of the experimental setup at the sector 16-BM-B, HPCAT.

has a high frame rate of >1000 frames/s (fps) and is used for high-speed falling sphere viscosity measurement.

3. High-pressure and high-temperature experiments in the Paris–Edinburgh cell

High-pressure experiments are conducted with a VX-3 Paris–Edinburgh press (Besson et al., 1992; Klotz et al., 2004). Cup-shaped WC anvils with the cup diameter of 12 mm and the bottom diameter of 3 mm are used to generate high pressures (Yamada et al., 2011). Fig. 2 shows a standard cell assembly design for liquid structure measurement and/or viscosity measurement. We use a different design of cell assembly for ultrasonic measurement (see Section 5 below). The cell assembly mainly consists of boron–epoxy (BE) gaskets, an MgO ring, ZrO_2 caps, a graphite heater, and a BN capsule. Capsule material differs depending on sample. For example, graphite inner capsule was used for silicate melt experiments inside BN sleeve (Yamada et al., 2011; Sakamaki et al., 2012). Large volume samples of up to 2 mm in both diameter and length are available in this cell assembly. However, the height of X-ray window is limited by the anvil gap at high pressures. At around 7 GPa and 1700 °C, a typical anvil gap is ~0.7 mm.

A ring-shaped BE (boron:epoxy = 4:1 in weight ratio) is used as gasket with a supporting outer polycarbonate plastic (Lexan) ring. The BE gasket and ZrO_2 caps in the assembly provide good thermal insulation for high temperature experiments. An MgO ring is placed between BE gasket and graphite heater to increase stability of the cell assembly and maintain anvil gap. High temperature is generated by the graphite heater. Fig. 3 shows temperature calibration curves up to 2000 °C as a function of applied load, which was determined in a separate no-X-ray experiment with W5%Re–

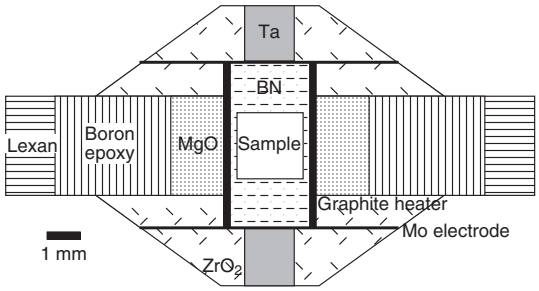

Fig. 2. A schematic illustration of a standard cell assembly for liquid structure and/or viscosity measurements.

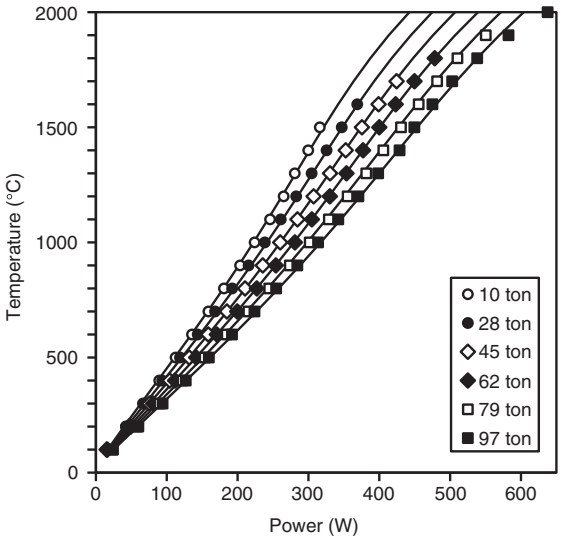

Fig. 3. Temperature-power relation measured up to 2000 °C and 97 ton hydraulic load (~7 GPa in sample pressure). The curves represent the results of fitting for the experimental results to two dimensional (power and load) polynomial equation.