Internal Resistive Heating in a Diamond Anvil Cell

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The phase diagram and PVT equation of state are the fundamental parameters for mapping the structure of Earth and planet interiors, and they also can be used for guiding the synthesis of novel materials. From this point of view, it is very important to obtain precise structural and thermodynamic information for materials at simultaneous high pressure and high temperature conditions. The generation and measurement of simultaneous high pressures and temperatures in the diamond anvil cell (DAC) in conjunction with a synchrotron radiation source has undergone rapid development in recent years. Laser heating in the DAC is one of the most widely used methods for this purpose. However, it suffers from large temperature gradients and instabilities [1]. Reliable measurements at these conditions are quite challenging.

A modified internal resistive heating method, which offers a very homogeneous temperature profile and excellent time stability for studying both metallic and non-metallic materials with in situ x-ray diffraction and Raman spectroscopy has been developed at CHESS, Cornell University [2]. As shown in figure 1, the key element of this method is a miniature metallic heater made of a rhenium ribbon with a small hole as the sample chamber, well isolated from the diamond anvil surface by surrounding pressure medium. It can be heated up to several thousand degrees K resistively under tens of gigapascals of pressure. Pressure measurements can be made by measuring the lattice parameter of a standard calibrator with known PVT equation of state accompanying the sample. Incandescent light radiation has been used for in situ temperature measurement by an optical spectroscopic system. Fig 1 shows photomicrographs of the assembly before and during heating of a gold sample. Figure 2 shows the homogeneous temperature profile in the heater.

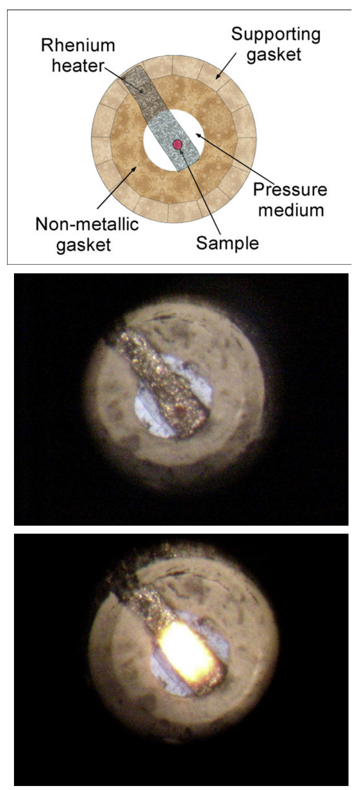

Fig. 1 Photomicrographs taken before and during heating. The heating element is rhenium ribbon with 80 microns width and 20 microns thickness. Gold powder was packed into a 25 micron hole at the center portion of the heater, seen as a red dot in the top picture. The heater is surrounded by transparent pressure medium of SiO_2 glass. The temperatures in the glowing area range from ~1600 K to ~1700 K.

Preliminary results of gold melting measurements have been obtained. Gold powder was packed into the small hole at the center of the rhenium heater as a sample, and SiO_2 glass was used as the pressure medium. Synchrotron radiation x-ray diffraction was used for both detecting the melting point and determining pressure. The disappearance of x-ray diffraction signals at high temperature is a useful criterion for melting, and gold itself is one of the most often used pressure standards for high PT conditions [3]. Figure 3 shows the diffraction peaks of gold changing with temperature and disappearing on melting.

During the past decades, engineering interests in rhenium and its alloys as important construction materials for parts subjected to severe conditions have dramatically increased. A materials PVT equation of state under static pressure is fundamentally important for understanding its scientific and engineering applications. Figure 4 shows the PVT equation of state for rhenium obtained by using this heating method. Thermal expansion at extreme conditions and many other useful higher-order thermoelastic parameters can be estimated from this measurement.

Raman spectroscopy is a useful and direct probe for the changes in structure and bonding at high pressures and temperatures, including phase transitions likely to occur at extreme conditions for many materials.

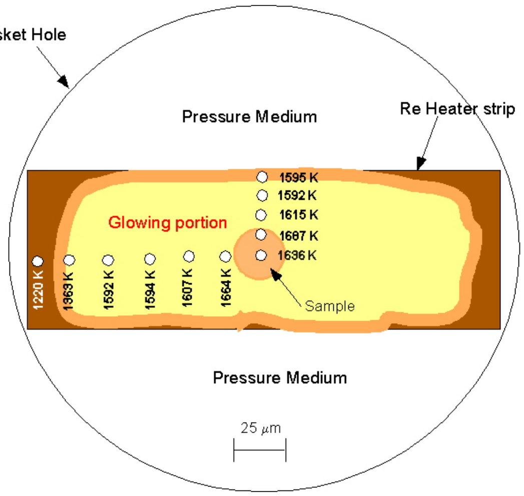

Fig. 2 Temperature profile in the heater's glowing area. The glowing area is about 80 x 200 microns.

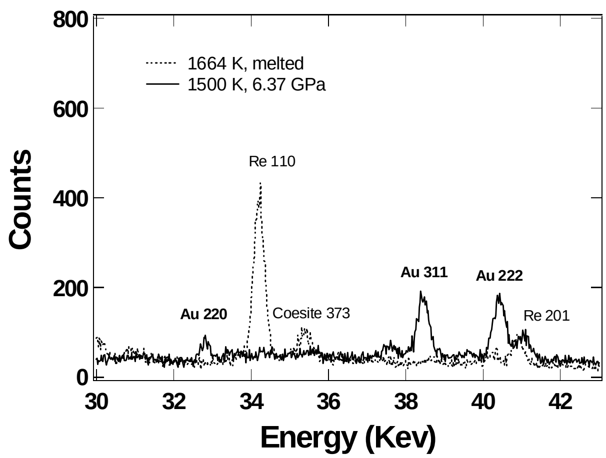

Fig. 3 Energy dispersive x-ray diffraction spectra at two different temperatures showing that the diffraction peaks shift with temperature and that all of gold lines disappear at the melting point.

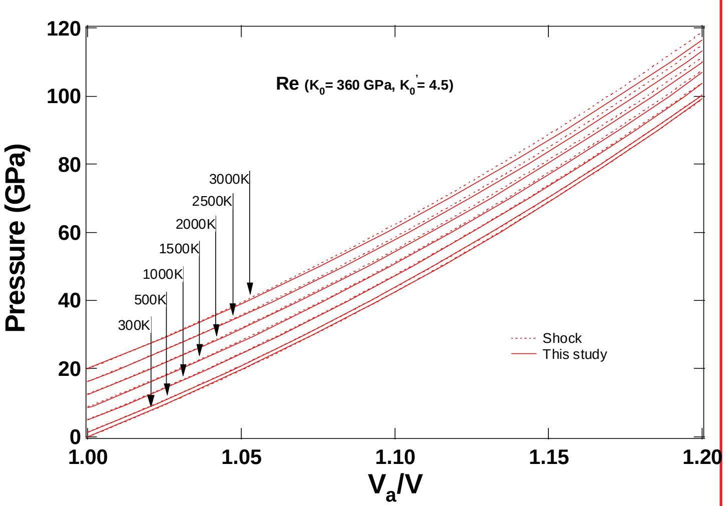

Fig. 4 PVT equation of state for rhenium measured with shock wave experiments previously [4] and static compression at high PT conditions of this study.

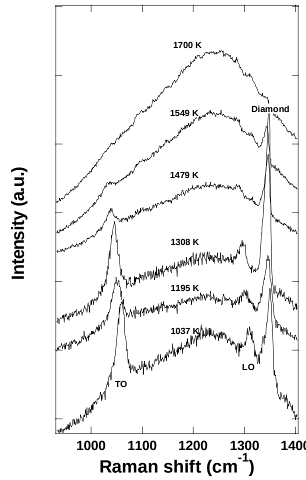

Fig. 5 Raman spectra of cubic boron nitride at high pressure and elevated temperatures. Internal resistive heating could play an important role in the pressure calibration of a second-order optical pressure sensor such as cubic boron nitride by using x-ray diffraction and optical measurement under simultaneous high PT conditions.

The internally resistive heated DAC looks very promising for use with simultaneous high PT Raman spectroscopy.

Figure 5 shows the anti-Stokes shift of both LO and TO Raman modes for cubic boron nitride under high pressure and different temperatures. Figure 6 shows the phase transition from SiO_2 glass to coesite during heating cycling.

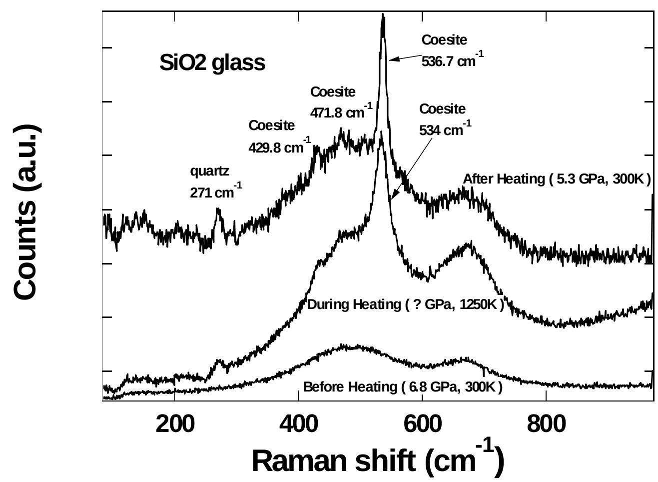

Fig. 6 Raman spectra of SiO_2 glass before, during, and after heating. The strong coesite peak around 534 cm^{-1} appeared during heating to 1250 K. The pressures noted for before and after heating were measured using the ruby fluorescence scale.

References

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