

## ダイヤモンドと窒化ホウ素を用いた圧力測定 Pressure measurements using diamond and cubic boron nitride

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Experiments using diamond anvil cells have been important in many studies of crystals and liquids at high pressure and temperature because these cells permit not only optical observation of the sample but also in situ measurement of the sample's physical and chemical properties. The pressure in the sample chamber of such cells is often determined by the shift in the wavenumber of a fluorescence line of ruby inside the sample chamber. However, the intensity of the ruby fluorescence decreases rapidly with increasing temperature. It is known that the first-order Raman mode of diamond anvil has been considered as a strong candidate of pressure marker because its Raman signal is intense and the diamond is always used as the anvil material in the DAC experiments. Cubic boron nitride (*c*-BN) is also a candidate of pressure marker because of chemical inertness and high temperature stability. It is the purpose of this study to present the dependences of pressure and temperature in the Raman lines of diamond and *c*-BN, using techniques combining synchrotron X-ray diffraction measurement with the Raman spectroscopic measurement.

The first-order Raman line at the culet face of the diamond anvil was investigated because the high-frequency edge of the Raman mode was correlated with the normal stress at the culet face of the diamond anvil. Thus, the edge frequency is a function of pressure and temperature in the sample chamber of the DAC experiment. Raman spectrum of *c*-BN exhibits two intense lines at 1054 and 1305  $\text{cm}^{-1}$  under ambient conditions, corresponding to the Brillouin zone center transverse optical (*TO*) and longitudinal optical (*LO*) modes, respectively. The Raman line of the *LO* mode overlaps an intense Raman line of diamond at pressures higher than 3 GPa. Therefore, it is difficult to observe the *LO* line in high-pressure experiments using the diamond anvil cell. In contrast, the *TO* mode could be used as the pressure calibrant in diamond anvil cells under high pressure and temperature conditions.

High-pressure X-ray diffraction experiments were carried out in an external heated diamond anvil cell. The small sample sandwiched between pellets of NaCl powder was loaded into a hole that had predrilled into a rhenium gasket. The heating temperature was up to 1000 K, and was recorded using the *K*-type of thermocouples. The sample was probed using angle-dispersive X-ray diffraction, located on the synchrotron beam line, at BL10XU of the Spring-8. The angle-dispersive X-ray diffraction patterns were obtained on the X-ray CCD collection system. The pressure was calculated from the MgO unit cell volume using the equation of state (EOS) for MgO.

Experimental runs were carried out at pressures of up to 110 GPa. In each run, the sample was compressed to the desired pressure, and then heated to measure the shift of the Raman spectra of diamond [1] and *c*-BN [2] at high pressure and temperature. At each pressure increment, the cell was screwed to hold the sample pressure and the Raman spectra were acquired at 300-1000 K with decreasing temperature. Two peaks, which corresponded to the *TO* mode of *c*-BN and the *LTO* mode of diamond, were identified at high pressures and temperatures. At pressures higher than 90 GPa, the *TO* mode of *c*-BN overlapped with the intense Raman peak of diamond. This indicated that *c*-BN can be used as the pressure calibrant at pressures below 90 GPa in diamond anvil cell experiments [2].

[1] Ono et al., Raman spectra of culet face of diamond anvils and application as optical pressure sensor to high temperatures, *J. Appl. Phys.*, 116, 053517 (2014).

[2] Ono et al., *In situ* Raman spectroscopy of cubic boron nitride to 90 GPa and 800 K, *J. Phys. Chem. Solid*, 76, 120-124 (2014).

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