strength of the nonhydrostatic effects in the media was evaluated by the ruby (Al_2O_3: Cr^{3+}) fluorescence method using a clamp-type diamond anvil cell originally designed by Dunstan and Spain [16–18]. Figure 1 shows a schematic illustration of the DAC setup and the fluorescence spectra of the ruby R lines for the representative media of NaCl, glycerin, 4:1 M-E mixture and argon at 77 K and around 10 GPa. The culet of the diamond was 800 μm. A sample chamber of 400 μm in diameter was prepared in a 304 stainless steel gasket. Small ruby chips, containing 0.5 wt% of Cr_2O_3, were uniformly placed in the chamber filled with a pressure-transmitting medium. The diameter of the chips was less than 10 μm. It was confirmed by X-ray diffraction analysis that the chips were single crystals.

The ruby fluorescence was measured using a charged coupled device (CCD) spectrometer. More details on the optical system are given in Reference 19. A diode pumped solid-state (DPSS) green laser light (532 nm), introduced into the sample chamber through fiber optics, excited the ruby chips in the chamber. The diameter of the beam line was 600 μm, larger than that of the sample chamber. The present work reveals spatially averaged information on the nonhydrostatic effects in the whole sample chamber.

When the pressure becomes nonhydrostatic, the two R_1 and R_2 lines broaden. The pressure shift of the ruby R_1 line is sensitive to the uniaxial stress [20–22]. In the present study, a number of small ruby single crystals were uniformly placed inside the chamber with the directions of the single crystals being random. The broadening of the ruby R_1 line reflects both the inhomogeneous pressure distribution and uniaxial stress pressure.

The peak positions and line widths of the ruby R_1 and R_2 fluorescence lines were determined by deconvoluting measured R-line spectra into a pair of pseudo-Voigt functions that represent the contribution of the ruby R_1 and R_2 lines with a linear background. The pressure was determined using the hydrostatic ruby pressure scale by Zha, Mao and Hemley [23, 24].

The pressure-transmitting medium was loaded into the sample chamber at room temperature except for nitrogen, argon and helium done using a purpose built cryogenic device at 77 or 1.4 K [17, 18]. The pressure was applied and changed at room temperature. The ruby fluorescence spectra were measured after the pressure and the width of the ruby R_1 line had been stabilized. It took several hours for the quantities to be stabilized when a medium was in a solid state under high-pressure. The DAC was slowly cooled down to 77 K and 4.2 K using liquid nitrogen and helium, respectively. It should be noted here that the pressure did not significantly change during the cooling process with the present DAC where load was maintained using springs. The difference in pressure between at 4.2 K and 300 K was in the order of a few % when organic and argon media were used. The pressure-change with the nitrogen and helium media will be discussed later.

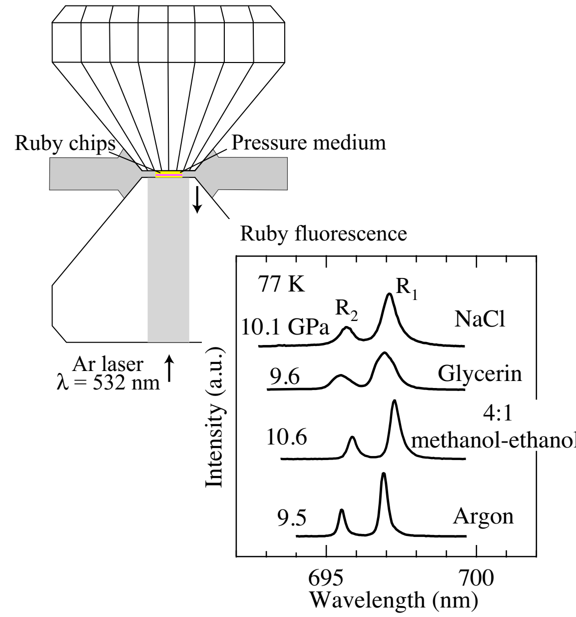

FIG. 1: (Color online) Schematic illustration of the DAC set up and the fluorescence spectra of the ruby R lines with representative media of NaCl, glycerin, 4:1 M-E mixture and argon at 77 K and around 10 GPa

Here we note differences between the present work and the previous studies on pressure-media from a technical point of view. The references 10 and 13 reported the inhomogeneous pressure distribution inside the sample chamber at room temperature [10, 13]. The previous studies in references 11, 12, 14 and 15 revealed spatially local information on the nonhydrostatic effects around the center of the chamber as the size of the "sensors" used to detect the effects, for example the ruby chips, Cu_2O or NaCl, was very small compared with the sample space [11, 12, 14, 15]. This work shows spatially averaged information on the nonhydrostatic effects inside the sample chamber. The observed broadening effect of the ruby R_1 line reflects both the inhomogeneous pressure distribution and uniaxial stress pressure. The purpose of the present study is to clarify the relative strength of the nonhydrostatic effects of the media in the low temperature region.

As shown in Figure 1, the broadening effect on the R_1 line depends on the media at 77 K and around 10 GPa. A clear broadening effect was observed in the ruby R_1 lines for NaCl and glycerin. However, the lines for the 4:1 M-E mixture and argon are comparably sharper. Therefore, it is possible to discuss the relative strength of the nonhydrostatic effects of the media from the width of the ruby R_1 line.

III. RESULTS

In this section, we show the pressure dependence of the full-width at half maximum (FWHM) of the ruby R_1 line