Comparison of Gold and MgO Pressure Scales at 22-56 GPa and 300-1150K

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Introduction

Knowledge about the phase transitions of earth materials at high pressure and high temperature is important for understanding observations of the seismicity of deep earth structures. A seismic discontinuity at 660-km depth has been known for more than 60 years [1], and it is expected that information on the origin of the globally observed boundary will be critical for understanding the dynamics and structure of the mantle [2]. Earlier experiments for quench samples [3] showed that the phase transition of a dominant upper mantle phase, ringwoodite (Mg_2SiO_4), to $MgSiO_3$ perovskite + MgO periclase ("post-spinel" transition) is responsible for the discontinuity at the 660-km depth. This is significant in that the isochemical phase transition rules out the possibility of chemical layering at the 660-km depth and opens the possibility of mixing between the layers above and below the discontinuity.

In the first *in situ* measurements, however, it was found that the post-spinel transition occurs at a depth that is 60-km shallower (2-GPa lower) than the 660-km discontinuity [4]. Then, in contrast, a more recent experiment at the GSECARS sector at the APS revealed that the phase transition is indeed observed at the pressure expected for the 660-km discontinuity [5]. This result is also supported by an independent experiment [6] and recent high-resolution seismic observations [7]. It has been proposed that the discrepancy is mainly caused by the uncertainties in pressure and temperature scales [5, 8].

In most *in situ* x-ray diffraction measurements at high pressure and temperature, the pressure, volume, and temperature (P-V-T) equations of state of inert metals, such as gold and platinum, have been used to estimate the pressure for a given volume and temperature. In order to provide high-quality equations of state of gold, we designed several high P-T measurements for gold and MgO mixtures. MgO has been extensively studied, and its equation of state is relatively well-constrained (e.g., Ref. 9), whereas significant electronic contribution is expected for the equation of state of gold [8]. Intercalibration between these scales will allow us to refine these important *in situ* calibrants.

Methods and Materials

Ten weight percent (10-wt %) gold powder was mixed with MgO powder and loaded in indented rhenium gaskets. Neon was loaded as a pressure-transmitting medium. The samples were compressed to 56 GPa by using Mao-Bell type diamond cells. Both internal micro- and external jacket-heaters were used to heat samples externally to 1150K. This heating method provides very homogeneous heating. The temperature was monitored by placing a Pt-Pt0.9Rh0.1 thermocouple between the diamond anvil and rhenium gasket, directly against the surface of the diamond. X-ray diffraction patterns were measured at GSECARS beamline stations 13-BM-D and 13-ID-D at the APS. An angle-dispersive diffraction setup was used with 2-D detectors, such as imaging plates and a charge-coupled device (CCD). The measured diffraction peaks were fitted to pseudo-voigt profile shape functions to obtain peak positions and intensities. Unit-cell parameters of gold, MgO, and neon were calculated on the basis of the measured d-spacings.

Results

Gold and MgO mixtures were used in a neon pressure medium for a total of three heating cycles. We calculated pressure by using gold scales [8, 10-12] and MgO scales [9].

Since 300K isotherms serve as references to extract thermal parameters from high-temperature data, it is important to test the consistency of the scales at 300K. We used static compression results by Heinz et al. [11] and Takemura et al. [14] for gold and from Speziale et al. [9] for MgO (Fig. 1). Note that the experiments by Takemura et al. [14] and Speziale et al. [9] used a helium pressure medium with a ruby scale, whereas Heinz et al. [11] used a gold scale and no pressure medium.

Our result shows that the gold and MgO scales are in agreement within 1 GPa. However, Takemura's static compression slightly underestimates pressure. The magnitude of the discrepancy above 30 GPa is approximately 1 GPa (Fig. 1).

We also plotted the pressure difference between the scales as a function of pressure at high temperature (Fig. 2). Shim's gold scale underestimates pressure by 0.7 GPa at 20 GPa. The magnitude increases with pressure, and, at 50 Gpa, Shim's gold scale underestimates pressure by 2.5 GPa compared with Speziale's MgO scale. This difference is even greater than that between Shim's gold and Speziale's MgO scales at 300K (1 GPa). This indicates

that there is a systematic difference in the thermal pressures of Shim's gold and Speziale's MgO scales at high temperature: the MgO scale yields a higher thermal pressure than the gold scale. The difference is very sensitive to pressure. We also examined temperature dependence, but no clear trend was observed. Thermal pressure calculated from the gold scales is systematically smaller than that calculated from the MgO scale by 3 GPa at 40-50 GPa. Possible reasons for the discrepancies at high temperature are (1) nonharmonic effects (e.g., electronic and anharmonic contributions), (2) different equations of state used, and (3) differential stress in the samples.

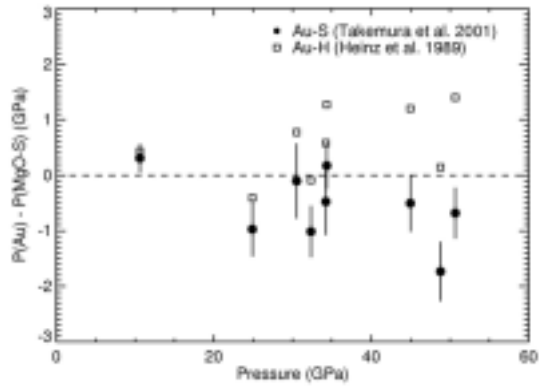

FIG. 1. Pressure difference between gold and MgO scales at 300K. For MgO, the scale by Speziale et al. [9] is used. For gold, the scale by Heinz et al. [11] (open circles) and Takemura et al. [14] (solid circles) are used.

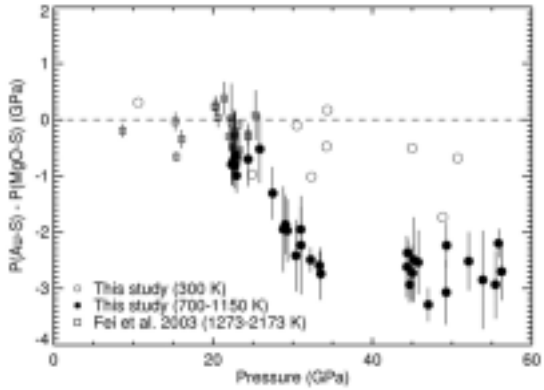

FIG. 2. Pressure difference between Shim's gold and Speziale's MgO scale at high temperature (solid circles). The difference at 300K is also shown for comparison (open circles). Multianvil data by Fei are shown by open squares.

Discussion

Because of their consistency and use of higher-quality data over a wide P-T range with a similar approach, it is legitimate to use Speziale's MgO and Shim's gold scales at pressures of 20-30 GPa and temperatures above 1000K. When these scales are used, the earlier *in situ* multianvil study for the post-spinel boundary becomes consistent (within 1 Gpa) with the expected P-T conditions of the 660-km discontinuity. Previously determined thermal equations of state of $(Mg,Fe)SiO_3$ perovskite are affected by this pressure-scale problem, since one of the most important studies (e.g., Funamori et al. [13]) is calibrated with Anderson's gold scale. The use of Shim's gold and Speziale's MgO scales tends to increase the Gruneisen parameter of the Mg-silicate perovskite in the lower mantle.

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