Thermal Pressure in the Laser-Heated Diamond Cell

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Introduction

The laser-heated diamond anvil cell (DAC) is used to study equations of state, crystal structures, and phase boundaries at conditions of deep planetary interiors. Successful experiments require a detailed understanding of the thermal pressure effects generated within the heated sample [1]. For example, two recent DAC studies of the post-spinel phase boundary in Mg_2SiO_4, which is believed to be responsible for the 660-km seismic discontinuity in the Earth's mantle, disagree strongly regarding the magnitude of the thermal pressure effect within the sample chamber [2, 3]. These two studies used different methods to determine pressures at high temperatures. When combined with synchrotron x-ray diffraction (e.g. [2]), in situ measurements of pressure (and stress) changes during heating can be made using well-characterized standard materials. However, many studies that use the laser heated DAC (e.g. [3]) do not determine the sample pressure in situ, instead they rely on the use of the ruby fluorescence pressure scale [4] before and after heating (since ruby fluorescence measurements cannot be carried out directly at high temperatures). In this study, we examine pressure variation in the sample before, during, and after heating at pressures of 20-25 GPa and temperatures up to 2000 K. Using pressure standard materials of MgO, Pt, and ruby, we directly compare pressure measurements made by ruby fluorescence scale with those made by x-ray diffraction under in situ conditions. The results will serve as a direct test of the reliability of the widely used ruby fluorescence technique to estimate pressures in the sample achieved during and after heating. The pressure range encompasses the 660-km seismic discontinuity which is where there are large discrepancies between studies using the laser heated DAC and the large-volume press [2, 3, 5-7].

Methods and Materials

A powder mixture of MgO and Pt with weight ratio of 9:1 was compressed to form a foil of ~ 15 μm thickness. The foil was cut into a piece of ~50 by ~40 μm^2, and loaded into a hole of a pre-indented stainless gasket mounted in a symmetric DAC. Ruby grains were then distributed in the gasket hole outside the sample region, at the edge of the sample, and at selected positions within the sample. A YAG crystal was also placed at the edge of the hole to facilitate alignment of the x-ray and heating spots. Finally, Ar was loaded in the gasket hole cryogenically. In this cell assembly, it is expected that ruby records pressure in Ar pressure medium, while MgO and Pt is used to directly determine pressure in the sample. In situ x-ray diffraction experiments were carried out at 13-ID-D of the GSECARS sector of the Advanced Photon Source. Angle-dispersive diffraction method was employed together with a charge coupled device detector. Double-sided laser heating was performed using Nd:YLF laser heating system. Temperatures were measured by spectroradiometry. Pressures were determined based on equations of state of MgO [8] and Pt [9], and ruby fluorescence measurement at room temperature [4].

Two heating cycles were performed at pressures of ~20 GPa and 24.5 GPa. These pressures were determined by ruby grain(s) placed at the edge of gasket hole before heating. Prior to heating, diffraction patterns at various positions on the sample were collected with an interval of 10 μm. The sample was heated by combination of TEM01 and TEM 00 mode lasers up to ~2000 K for 8 minutes in the first heating cycle, and only by TEM 01 mode laser to 1000-1400 K for 7 minutes in the second heating cycle. During heating, we also collected x-ray diffraction patterns of the sample at the heated spot. After temperature quench, pressure distribution across the entire sample chamber was examined based on both x-ray diffraction and ruby fluorescence measurement. The diameter of laser beam was measured to be ~25 μm based on optical observation of both laser emission on the sample during heating and the surface texture of the quenched sample.

Results and discussion

A representative diffraction pattern of the sample before and after the first heating is shown in Fig. 1. The averaged pressures in the sample before, during, and after heating determined from individual hkl reflections of Pt and MgO are shown in Fig. 2. The results indicate that deviatoric stress existed in the sample before heating drastically decreased by heating. The pressures after heating calculated from MgO and Pt scales agreed well, yielding averaged values of 17.8 (4) after the first heating and 21.4 (5) GPa after the second heating. There was no difference between the pressures determined inside and outside the heated spot within the error of pressure calculation. By comparing the pressures determined by Pt and MgO scales during and after the second heating, it is estimated that the thermal pressure in the sample heated to 1000-1400 K was ~2GPa at ~23 GPa (Fig. 2).

On the other hand, the averaged pressures determined by ruby grains after the first and second heating were 19.8 (0.1) GPa and 24.7 (0.3) GPa, respectively. Small pressure variations indicate that pressures in Ar medium were quite uniform after heating. There was essentially no difference between the pressures in Ar determined before and after heating. By comparing the pressures determined by ruby scale and x-ray diffraction, it is clear that the pressure in the sample is ~2 and ~3 GPa lower than that in Ar after the first and second heating, respectively. Assuming ruby scale is reasonably consistent with MgO and Pt scales at ~23 GPa, it is concluded that the pressure outside the sample could be 2-3 GPa higher than the pressure inside the sample. The results demonstrate the importance of in situ measurement of pressure in the sample to precisely determine equations of state and phase boundaries.

Acknowledgements

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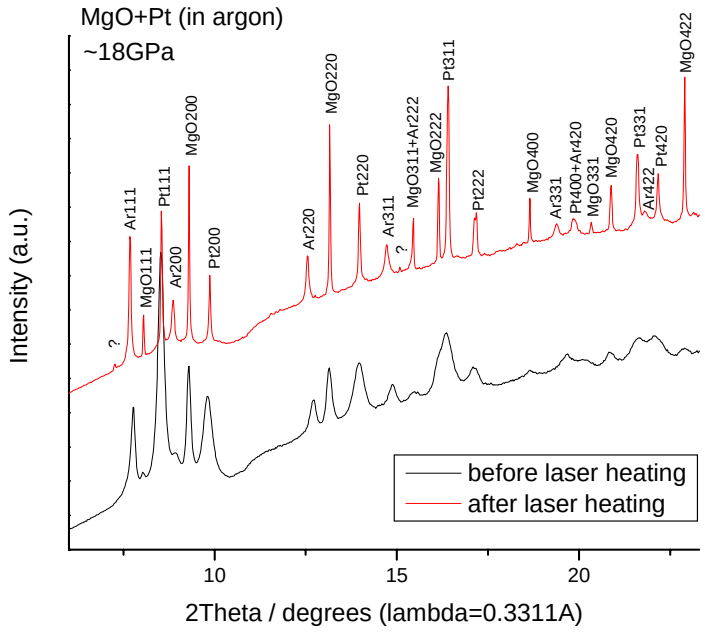

Fig. 1. A representative diffraction pattern of the sample before and after the first heating.

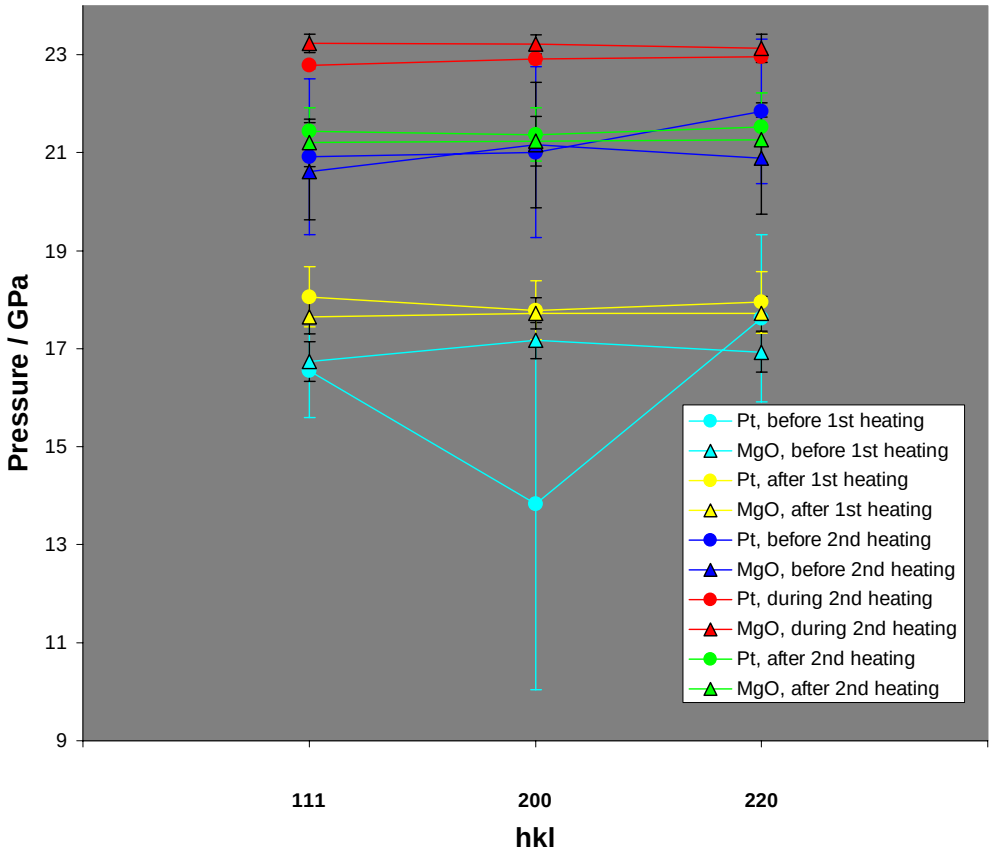

Fig. 2. . The averaged pressures in the sample before, during, and after heating determined from individual hkl reflections of Pt and MgO.