

DIA-DAC: A Triple-stage High-pressure Setup for High-pressure Research Using Synchrotron X-rays

B. Li

Mineral Physics Institute (MPI), State University of New York at Stony Brook (SUNY),
Stony Brook, NY, U.S.A.

Introduction

Double-stage (MA-8, MA-6), large-volume, high-pressure apparatus and diamond anvil cells (DACs) have been the tools commonly used for high-pressure research. Large-volume, high-pressure apparatus has the advantages of being able to accommodate a large sample volume and provide a uniform sample temperature distribution, but it is limited to working under pressure (P) ranges of <30 GPa. DACs have the super power of being able to withstand pressures of >150 GPa, but they have the disadvantages of being able to accommodate only small sample volumes and producing a large temperature gradient in the sample region because of laser heating. The determination of pressure at high temperatures is still a major task. When different pressure standards are used, the pressure can differ as much as 20% at high temperatures. This study explores the possibility of combining the DAC and large-volume techniques by using a third-stage diamond anvil in double-stage, large-volume, high-pressure apparatus. The objective is to reach a pressure of 50 GPa while working with a moderate sample volume and providing a stable/homogeneous temperature.

Methods and Materials

Figure 1 is a schematic diagram of the cell assembly used in this study. In previous attempts, it was claimed on the basis of resistance measurements [1] that pressures of >100 GPa were reached. In this study, two polycrystalline diamond inserts with slightly tapered tips were inserted into the graphite furnace. The sample and pressure stand material was sandwiched between the diamond inserts. The sample was 1.5 mm in diameter and 1.5 mm thick. A W5%Re-W26%Re thermocouple was fed into the center of the cell from the gap between the two horizontal anvils. Between the polycrystalline diamond and the WC anvil, different materials were tested, including alumina, tungsten carbide, and diamond. Enclosing the polycrystalline diamond in the cell assembly has the advantages of preserving the diamond's strength and preventing the diamond from back-transforming to graphite. In principle, by using this combination of techniques, temperatures as high as 2000K can be reached.

Results

Initial experiments were conducted with NaCl as the only sample material. Before the current experiment, a test

experiment was performed at the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory by using olivine plus NaCl as the sample. However, the diffraction of the pressure standard became very weak after pressures of >26 GPa were reached. It is assumed that the phase transition of NaCl to B2 occurred. In this experiment, the purpose is simply to maximize the NaCl volume in order to observe the B1-B2 phase transition of NaCl.

Figure 2 shows the x-ray diffraction patterns at room pressure (top) and at 12 GPa (65 tons, bottom). In this experiment, the x-ray diffraction pattern shows a higher intensity than did previous runs. However, the pressure generation efficiency is lower than observed as shown in Fig. 3. A large sample volume and a broken diamond insert were attributed to this, as discovered after the experiment.

The second experiment was conducted with WC end-plugs instead of Al_2O_3 . Although the sample showed high efficiency at the beginning, the sample volume became very small at high pressures, and the identification of NaCl became difficult. Modification of the cell seems to be needed in order to keep a larger sample volume

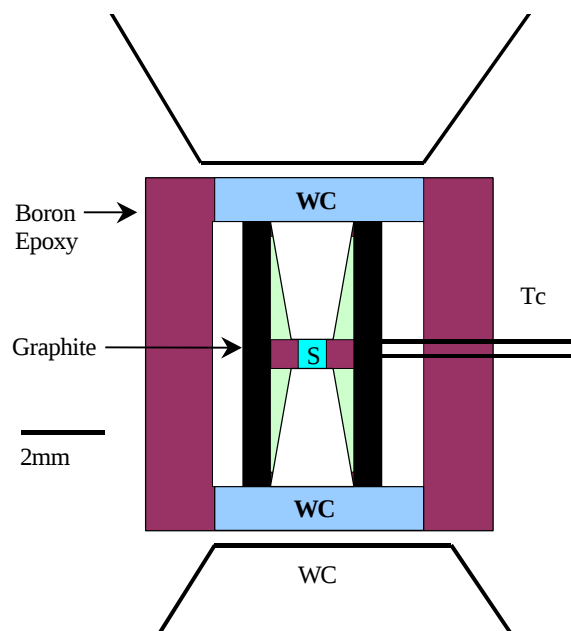


FIG. 1. Cross section of the cell assembly.

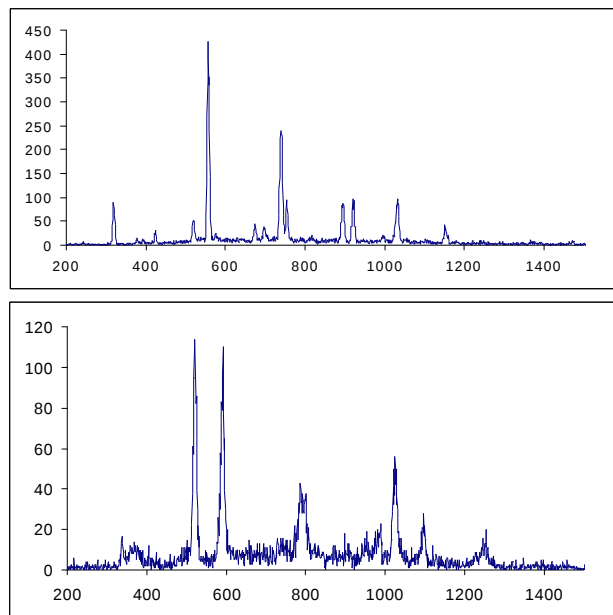


FIG. 2. X-ray diffraction pattern at 0 ton and 63 tons.

between the diamond inserts. This was done in later experiments, yielding good x-ray diffraction patterns at pressures of >40 GPa.

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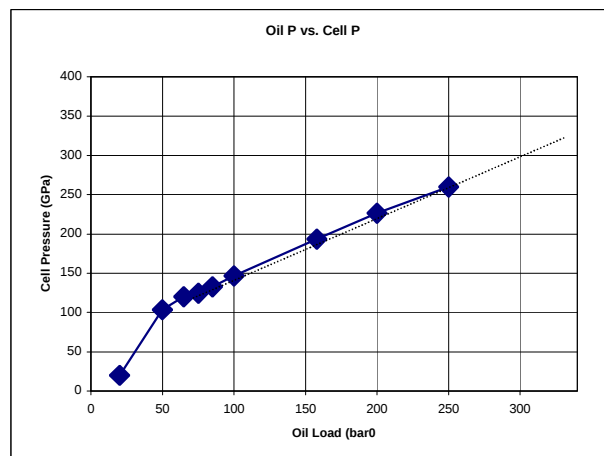


FIG. 3. Pressure vs. oil load.

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- [1] S. Endo and K. Ito, *Triple-stage High Pressure Apparatus Using Sintered Diamond Anvils*, in *High Pressure Research in Geophysics*, edited by S. Akimoto and M. H. Manghnani (Center for Academic Publication, Tokyo, Japan, 1982).