High-Temperature Heater for Diamond-Anvil Cells

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High-pressure studies provide crucial information for many scientific and industrial applications. Volume changes can produce structural, electronic, magnetic and other phase transitions, initiate chemical reactions, and many other phenomena. During last decades diamond anvil cell (DAC) technique has become the most popular method of pressure generation capable for work in a multimegabar pressure range. However, there still are a number of problems related to high-temperature experiments with DACs.

There are two main methods of heating in DACs – laser and electrical. Laser heating techniques cover a wide P-T field: P>100 GPa, T=1300-5000 K. The sample preparation for laser-heating experiments is relatively easy and there is practically no risk for diamonds due to heating. However, temperature measurements in laser heated DACs is a complex problem which requires multiwavelength spectrometry and knowledge of a pressure and temperature dependence of emmisivity. Very localised laser heating creates thermal stress in the DAC complicating interpretation of the results. The temperature range below 1200 C is difficult to handle with laser heating systems. Contrary, external electrical heating allows generating temperature in a wide range (form 300 K to over 1800 K), relatively easy measuring it (with thermocouples at T<2000 K), maintaining it practically constant (±5 K at 1500 K) during hours. Moreover, in externally heated DAC at T>800 K stresses are practically absent and heating is quite homogeneous. In other words, electrical heating could be a perfect complementary to the laser heating method to study materials at extreme conditions.

We developed an external heating assembly which allows conducting experiments in DAC at pressures above 130 GPa and temperatures above 1200 K. It consists of external resistive heaters placed around a cell, temperature resistant lever system, and a miniature DAC of a cylindrical shape (diameter 23 mm, height 15 mm) made out of a special high-temperature Ti-based alloy. The new system allows fine (within 1 GPa) adjustment of pressure in the whole temperature range. It maintains constant pressure (within 1 GPa at megabar pressure range) and temperature (within 5 K at 1000 K) during several hours, allows measuring temperatures accurately with an external thermocouple, and does have measurable temperature gradient within the pressure chamber. The new heating assembly is easily coupled with experimental set up at synchrotron beam lines and have been used in pilot experiments on Fe-Ni alloys (their behaviour up to 1 Mbar was studied), magnetite Fe3O4 (phase transformations and the thermal equation of state), wustite FeO (phase relations to 60 GPa), and H_2O (melting curve to 30 GPa).

Fe-O System at Extreme Conditions

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Iron oxides, such as wüstite FeO, magnetite Fe_3O_4, and hematite Fe_2O_3, undergo a number of phase transitions (Mao et al., 1996; Dubrovinsky et al., 2000a; Badro et al., 1999; Pasternak et al., 1999; Fei et al., 1999; Haavik et al., 2000). The importance of phase transitions in iron oxides for lower mantle mineralogy can be illustrated by the behavior of magnesiowüstite at HPHT. On increasing pressure above 100 GPa at 300K wüstite transforms to the NiAs (B8), or the anti-NiAs (a-B8) structure, while periclase retains the NaCl (B1) structure to at least 200 GPa. The topological difference between the B8 and the B1 structures at high pressure could lead to an immisibility gap in a region of the MgO-FeO solid solution. We found that magnesiowustites with compositions $Mg_{0.5}Fe_{0.5}O$, $Mg_{0.6}Fe_{0.4}O$, $Mg_{0.8}Fe_{0.2}O$ decompose on iron- and magnesium-rich components at conditions of Earth's lower mantle. This result is important because the decomposition of magnesiowustite into a low-density and a high-density phase might cause dynamic effects that would lead to mantle heterogeneity.

Studies of the nature of the high-pressure polymorph of magnetite have a long and controversial history. Using electrically- and laser-heated diamond anvil cells at pressures above 40 GPa we synthesized pure high-pressure Fe_3O_4 phase (h-Fe_3O_4) and performed an *in situ* structural refinement. In good agreement with our *ab initio* calculations we found that h-Fe_3O_4 adopts the $CaTi_2O_4$-type structure (space group *Bbmm*). Electrical resistivity and Mössbauer spectroscopy measurements show that h-Fe_3O_4 is metallic and magnetic up to at least 70 GPa. The magnetic properties are associated with one of the structural positions presumably occupied by Fe^{2+} ions. High density and low compressibility of h-Fe3O4 polymorph could make it potentially important Earth' lower mantle phase.