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A tungsten external heater for BX90 Diamond Anvil Cells with a range up to 1700 K

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Abstract: Resistive heating of a sample in a diamond anvil cell (DAC) can generate a homogeneous temperature field across the sample chamber with reliable temperatures measured by a thermocouple. It is of importance in experiments aiming at exploring phase diagrams and quantifying thermoelastic properties of materials. Here, we present a ring-heater design developed for BX90 diamond anvil cells (DAC). It is made of a ring-shaped aluminum oxide holder hosting a tungsten wire coil inside and coupled with $Ar + 2\%$ H_2 gas to prevent oxidation during experiment. This modular plug-and-play design enables *in situ* studies of samples via x-ray diffraction up to the temperature of 1700 K. Temperature in the BX90 sample volume as measured through a thermocouple was calibrated using the melting point of gold. As an application of this design we report the thermal expansion coefficient of MgO at 9.5(1) GPa.

Keywords: Tungsten ring heater, diamond anvil cell, high pressure-high temperature, reversible heating/cooling

1. Introduction

Physical properties at elevated pressures and temperatures are of fundamental importance for geoscience and material science. During the past few decades, diamond anvil cell techniques have become the most successful method of pressure generation capable of working in the multi-megabar pressure range, while high temperature is achieved by either laser heating, external resistive heating of the sample area within the cell, or internal resistive wire heating. Due to low emissivities at low temperatures, quantitative laser heating can only be deployed in the temperature range above 1200 K. Even with the aid of a beam expander to reduce lateral temperature gradients**,** [1,2] or using laser-heating in combination with resistive heating [3,4], the laser heating technique introduces temperature gradients across the sample, which are

often of the order of 100 K/μm. Such gradients can pose severe challenges for a variety of high-pressure experiments, including single crystal diffraction experiments and high-temperature rheologic measurements.

Resistive heating is broadly used for the temperature range up to 1300 K; such heating typically has a less than 10 K temperature gradient across the sample chamber [5,6] relative to laser heating, and temperatures can be stably maintained for several hours or longer [7]. There are two types of electric heaters: internal heaters, in which the heating element is placed inside the pressure chamber, and involve heating of resistive wires; and external heaters, in which the entire area around the sample chamber is heated using a furnace surrounding the sample area. The design and performance of internal heaters have been investigated extensively [8-12]. The simplest internal heater consists of a metal wire that passes through the sample, with its smallest cross-section (and hence most localized heating) being within the sample itself [8-9]. This principle has been varied into more complex heated composite gaskets [11,12]. Internal resistive heaters have been reported to reach temperatures in excess of 1900 K at pressure in the diamond cell, and have often been deployed to probe the properties of the heated metallic wire itself in the diamond anvil apparatus, with temperatures measured spectro-radiometrically [8,11]. Alternatively, heated gaskets or metallic samples embedded within wire-heaters have been used with composite gaskets. However, the fabrication of heatable composite gaskets can be complex and time-consuming and is thus difficult to deploy at a user facility which has to accommodate users with only minimal training and a large range of technical proficiency.

In external resistive heaters, the sample is heated through resistive wires surrounding the entire diamond-gasket assembly. Temperature is measured with a thermocouple attached to the external diamond facets, as long as careful calibrations of the temperature difference between thermocouples mounted on the diamonds and the sample inside the DAC chamber is produced. The external heating method can be combined with a wide variety of high pressure probes for in situ studies, including x-ray diffraction, Raman and Mossbauer spectroscopy, and x-ray absorption measurements [13]. Furthermore, to ensure more uniform heating environments, external heaters can be used in different geometries, such as two separate heaters above and below the gasket, or inter-nested heaters [14]. A range of different external heater materials have been used. Graphite has been used as a resistive heater over the widest temperature range [3]; it has a large range of thermal stability, with a sublimation temperature of 3900 K. Nevertheless, it requires a highly reducing environment, and its softness and fragility have limited its usage. Platinum, [15],

Molybdenum [16-17], Platinum/Rhodium [12], and tungsten wires have all been used (at times in conjunction with vacuum chambers) [18-19], but these have seldom been deployed above 1300 K [14].

Here we present a modular, user-friendly plug-and-play resistive heater that is durable and able to reach temperatures corresponding to the Earth's upper mantle and transition zone pressures (1700 K) at corresponding pressures (~ 25 GPa), and hence help quantify properties of materials at these conditions. In this paper, we provide a technical description of our design, and report an example study where we measure the pressure dependence of the thermal expansion coefficient in MgO.

2. Heater design and setup:

BX 90 diamond cells are widely used in conjunction with angle-dispersive X-ray diffraction because their wide symmetrical conical axial opening [20]. The BX 90 cell has several features that make it suitable to combine with an external resistive heater. First, the BX 90 cells have an annular space (24 mm outside diameter x 14 mm inside diameter) inside the piston that allows for the placement of a resistive heater. Second, the cells also have U-shaped cuts in their cylinder part that can serve as wire inlets for thermocouples and electrode wires.
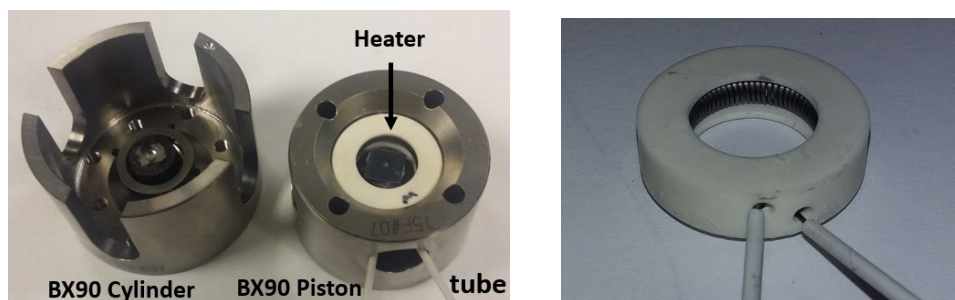

Figure 1. (Left) Tungsten ring heater sitting inside a BX90 piston. (Right) A real W heater made of a L-shaped alumina holder and cover, with W ring inside it. Heater and diamonds are approximately to scale. Height of individual diamond: ~ 2 mm.

The basic framework of the homemade heater is a ring-shaped aluminum oxide holder with a tungsten wire coil inside (Figure 1). The aluminum oxide holder is made of a ring of an "L" shaped cross-section and a cover. The wall thickness of the aluminum oxide holder is 1 mm. The tungsten wire of 0.5 mm in diameter is wound into a 4 mm in diameter coil with 60 turns in total. The electric leads are isolated from the frame by alumina tubing ((#8746K451, McMaster-Carr)). The tungsten wire is subject to oxidation in air at temperatures above 900 K, diamonds need to be protected from oxygen at T's > 970 K. Externally

heated DAC assemblies therefore require a protective can that keeps the DAC assembly in an inert gas mixture of argon + 2% H_2 to protect the tungsten wire, diamonds and DAC components from oxidation.

Both the internal heater [9] and the graphite heater [3] integrate the heater into the gasket, which make the sample loading more complicated and time consuming. For the tungsten heater in this paper, its internal diameter is larger than the outer diameter of BX90 cell seats, and therefore, the tungsten heater is independent of gaskets and sample loading, allowing for simpler assembly.

WC- or cBN- seats are commonly used as backing-plates to support the diamonds. If kept in air, WC-seats are unstable and oxidize at temperatures as low as 750 K [21]. The cBN-seats (from Almax.easyLab) have good oxidation resistance up to 1273 K [22]. At higher temperatures, cBN forms a thin protective layer of boric acid on its surface that prevents further oxidation, and is stable in reducing atmospheres up to 1773 K. [23] Our tests show that WC-seats oxidize at temperatures below 1200 K, while cBN-seats show no visible damage to at least 1700 K following a few heating cycles. Diamond is known to begin to oxidize in air when heated to 973 K, and starts graphitizing at 1073 K [24]. Within the reducing atmosphere of Ar+2.8 % H_2 used for this work, no noticable oxidation and/or graphitization of the diamonds was observed after 5 heating cycles at temperature ranging from 1200 K to 1700 K.

The W ring heater is powered by a ZUP36-24 model (TDK Lambda Corporation) power supply, with output voltage of 0-36 V and output current of 0- 24 A. Two type-S thermocouples are cemented onto the pavilions of the two diamonds with Resbond 904 HT fast cure Alumina Adhesive to measure temperature. They were located both within 0.5 mm from the culet. Thermocouple detacthment proved to be an occasional issue and the use of two thermocouples helped identify this problem with the detached thermocouple clearly to lower temperature. The experiment could still continue with one thermocouple.

3. Temperature calibration

A temperature versus power curve for a typical heater assembly described is shown in Figure 2 and shows a linear relationship up to 1450 K. Above 1450 K, the slight decrease in slope is indicative of increased thermal losses at the higher temperature. The uncertainty at 1450 K is about ±4 K for a typical S-type thermocouple. No PID thermal power control loop was employed. The temperatures reported in Figure 2 were recorded after 5-10 min thermal equilibration after changing the power settings.
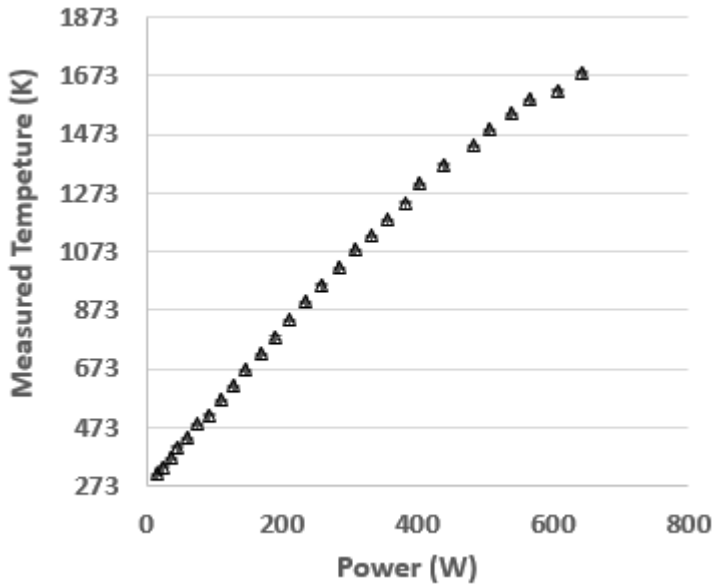
Figure 2. Temperature as a function of power at 5.1 GPa for a tungsten ring heater seated in a BX90 DAC. Temperatures are measured by a thermocouple cemented onto the pavilion of the diamond within 0.5 mm from the culet. For each datum, the error bars are within the size of the data symbols.

To calibrate the temperature, we conducted a melting point test of gold powder at ambient pressure. Gold has a melting temperature of 1337 K at ambient pressure. The gold powder was loaded loosely without a pressure medium into a rhenium gasket with a sample chamber of 80 micron in diameter, and held between the diamond anvils with 400 μm diameter culets. The gold was monitored by x-ray diffraction (XRD) conducted at beamline 12.2.2, Advanced Light Source, Lawrence Berkeley National Laboratory [25]. The XRD diffraction patterns were recorded by a MAR345 image plate at an energy of 25 keV with a 10×10 μm^2 spot size X-ray beam. It was observed that gold starts to melt as the diffraction peak intensity decreased at 1340 K and the peaks had essentially disappeared at 1348 K. We conclude the thermocouple measures the sample temperature with a slight 11 K (0.8%) offset, indicative of small thermal gradients with the hot B90 cell.
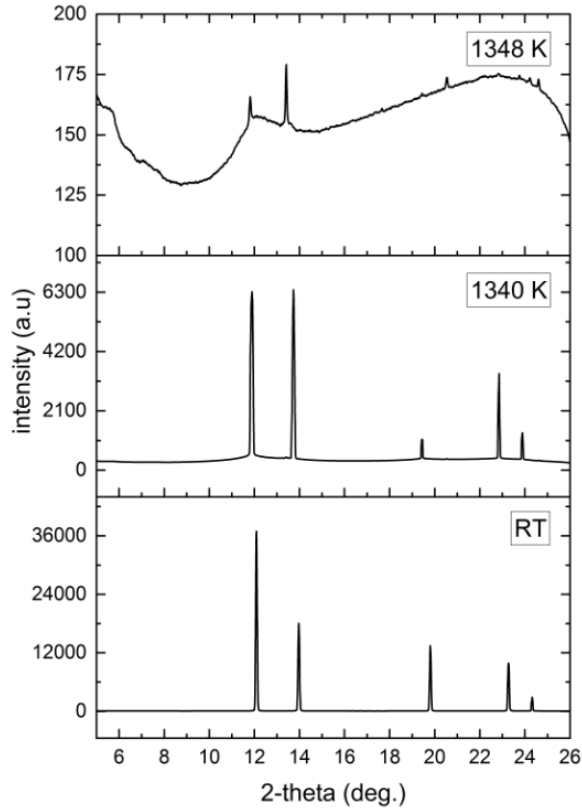
Figure 3. X-ray diffraction patterns of gold power in a BX90 cell at ambient pressure. The bottom one is at room temperature, the middle at 1340 K and the top one 1348 K.

4. Thermal expansion of MgO under pressure

The thermal expansion coefficient used here is defined as

$$\alpha = \frac{1}{V}\left(\frac{\Delta V}{\Delta T}\right)_P \tag{1}$$

where V, T and P are volume, temperature and pressure respectively. Its experimental determination requires sample heating at constant known pressure. We mount our resistively heated DAC with a gas membrane piston system that applies pressure to the DAC and is dynamically maintained constant using a "Druck PACE5000" control system. The DAC is free to thermally expand with temperature change but the pressure applied to the DAC and the pressure on the sample is intended to remain constant.

As an application example, the temperature dependent isobaric volume measurements for MgO were carried out at ambient pressure and 9.5(1) GPa. The ambient pressure x-ray powder diffraction

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measurements were conducted on samples in a quartz capillary using an infrared heated tube furnace [26] allowing sample temperatures up to 1250 K with temperature ramping rates up to 100°C/s. In separate experiments, we heated the sample under high pressure using our tungsten ring heater integrated into a BX90 DAC. For these experiments, MgO was loaded into a 140 μm diameter sample chamber within a rhenium gasket. Diamond anvils with 300 μm culets were used. MgO served both as sample, pressure medium and pressure marker. It was first compressed at room temperature to 9.5(1) GPa, followed by heating to the maximum temperature of 1250 K. In the heating and cooling process, the pressure was controlled, by a gas membrane as explained above. The membrane pressure was dynamically kept constant and generates constant pressure on sample in the gasket. In our case of this heating, 15 bar of membrane pressure yields 9.5(1) GPa on the MgO sample in the gasket as determined by the equation of state of MgO at room temperature. X-ray diffraction patterns were recorded on a MAR345 image plate with photon energies of 25 keV and exposure times of 60 s. Temperatures were averaged over the whole 60 seconds of x-ray diffraction collection time. During the averaging time, temperatures were stable within 4 degrees.

Figure 4 plots ln (V) versus temperature for MgO measured at ambient pressure and 9.5(1) GPa during heating and cooling along with the literature result from reference [29]. The observed unit cell volumes were reversible upon cooling. Linear fits of ln (V) vs temperature gave the thermal expansion coefficient of MgO as the slope of the fitted line,

$$\frac{d[\ln(V)]}{dT} = \frac{1}{V}\frac{dV}{dT} = \alpha \qquad\qquad (2)$$

The α_P derived this way are $4.13\pm0.04\times10^{-5}$ K^{-1} and $2.12\pm0.50\times10^{-5}$ K^{-1} ($2.74\pm0.42\times10^{-5}$ K^{-1} if 298 K point excluded) for ambient pressure and 9.5 GPa , respectively. The α_0 value obtained from ambient pressure data is in good agreement with results of Zhang [29] over a similar temperature range. The α_P at 9.5 GPa is lower than the literature value of ~3.10 (30)$\times10^{-5}$ K^{-1} reported for MgO at similar pressure see Fig. 3 in Ref [29]. The reason for the difference is unclear – we simply report our result and note that our high temperature data has a larger data point scatter compared to the other 4 data sets at lower pressures. A refining of the experimental technique is required to reduce the data scatter, but the concept of the isobaric DAC heating has been demonstrated.
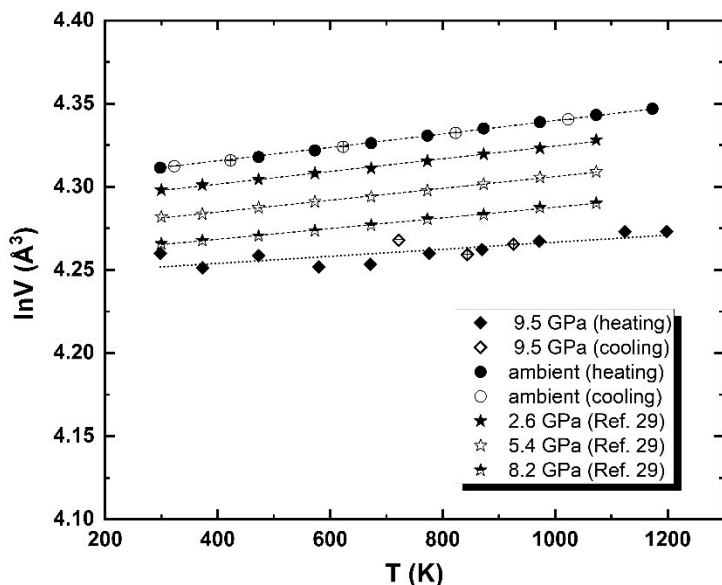
Figure 4. The pressure dependence of ln (V) over temperature for MgO at ambient pressure and 9.5(1) GPa, (top and bottom plots). The heating and cooling cycles were recorded and overlap.

In Fig. 4, the data points of 2.6 GPa, 5.4 GPa, and 8.2 GPa are from a large volume press [29], and the isobaric process was achieved by adjusting its pressure during heating [30, 31]. In this project, MgO was compressed up to 9.5 GPa at 300 K, and no pressure measurement in an reversible heating/cooling process, which could be an alternative of traditional isobaric heating for DAC [7,14,32-35].

5. Conclusion

A resistive heater, made of a ring-shaped aluminum oxide holder with a tungsten wire coil inside it, was developed for BX90 cells. Example measurements reported in this paper demonstrate the capability of the device for *in situ* XRD measurements up to 1700 K. This device has promising applications in studying the melting curves of terrestrial materials under high pressure and temperature conditions relevant to Earth's crust and mantle. An initial test of the system involving over a 300-1200 K temperature scan at an isobaric pressure of 9.5 GPa. indicated reasonable agreement with literature for the thermal expansion coefficient of MgO.

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The data that support the findings of this study are available from the corresponding author upon reasonable request.

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