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Twin sample chamber for simultaneous comparative transport measurements in a diamond anvil cell

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In static high pressure experiments, performed within a diamond anvil cell (DAC), several different methods of thermometry may be employed to determine the temperature of the sample. Due to different DAC designs or particular experimental designs or goals, uncertainties in the determination of the temperature of a given sample exist. To overcome the inaccuracy in comparing the temperature dependence of transport properties of different materials at high pressure, we have used a novel design of resistivity measurement in a twin sample chamber built on an insulated gasket in a DAC. In this design, the transport properties of two samples will be measured simultaneously and therefore the two samples will always be in the same relative temperatures. The uncertainties in the temperatures of the two samples will be exactly the same and therefore their relative phase diagram will be compared precisely. The pressures of the chambers can be slightly different and is easily determined by the ruby pieces placed in each chamber. To demonstrate the feasibility of this method we have compared the superconducting properties of two YBa2Cu3O7−x (0 ≤ x ≤ 0.65) samples with slightly different superconducting transition temperatures at ambient pressure as a function of pressures up to 11 GPa. The upper limit of the pressure achieved using this design would be lower than single chamber gaskets. The highest achievable pressure, as in a conventional single hole setup, depends upon the thickness of the gasket, the culet size, the size, and symmetry of the sample chamber. For the twin chamber, it also depends upon the separation of the holes from each other as well as from the edge of the culet. © 2013 AIP Publishing LLC. [http://dx.doi.org/10.1063/1.4821080]

INTRODUCTION

Static high pressures of several million atmospheres can be achieved in a diamond anvil cell (DAC). In a DAC variety of remote and in situ measurements, such as transport, optical, and magnetic measurements, can be carried out at extreme conditions.1 Comparing the physical properties of different materials, sometimes with slight differences, as a function of pressure is done in many studies (e.g., Refs. 2–6). However, uncertainties in reproducing the exact experimental conditions often challenge proper comparative studies on materials studied in different high pressure experiments. One such problem is the comparing P-T phase diagram or critical temperatures for phase transitions of different samples as a function of pressure. While temperature measurements can be carried out with high precision at relatively close distances to the samples in a DAC, the relation between the measured temperature and the actual value of temperature in the location of the sample depends on many factors. Slight differences in the design of high pressure cells, methods of thermometry, or even slight different temperature gradients which are present in the same setup but in two different high pressure runs can lead to differences of the orders of few Kelvin between different experiments. These differences generally define the boundaries of the phase diagram of a material. For many cases of comparative high pressure experiments, the study of the small differences between the temperature dependence of various physical properties of different materials is the desired result. These small differences can easily fall within the boundary of the uncertainties in temperature. In this work, to overcome the uncertainties in temperature differences between the two samples we have used a new method of simultaneous resistivity measurements with a twin chamber gasket. In a twin chamber gasket, two identical holes, serving as pressure chambers, are symmetrically drilled into a gasket (Figure 1, inset). A twin chamber gasket for optical studies has been recently used by Shukla et al.,7 in which the one sample was loaded in one chamber and the ruby was loaded in the other chamber to prevent contamination from the ruby, a source of oxygen, to the sample to be synthesized at high temperature and relatively high pressure. In this work, we have modified a gasket with a twin chamber design for transport measurements in a diamond anvil cell.

EXPERIMENTAL SETUP

In series of experiments, we have used various designs of the chambers in a gasket. While in these experiments we have achieved pressures as high as 33 GPa, there is no reason to assume that 33 GPa is the limit of highest pressure that could be reached in this design. The placement of the holes plays a very important role for the eventual drift in pressures of the twin chamber cell. The symmetry of the holes has been shown to be the most important factor for avoiding an early failure of the gasket. The next important parameter is the initial distance between the holes. The region of the highest pressure in the cell is the middle of the culet, in between the two gasket holes.8 This region will always deform, making the...
spacing between the two holes larger. On the other hand, very small separation between the two holes provides a thin barrier between the two samples which may fail under pressure, allowing electrical contact between the two samples. Proper distance between the holes is crucial in successful simultaneous transport measurements in twin chamber design. We have tried several arrangements of chambers with initial radii varying between 80 μm < r < 140 μm and the nearest distance between the inner sides of holes varying from 10 μm < d < 100 μm on 500 μm single beveled diamond anvils. The minimum drift of the pressure chambers from each other, which can warrant achieving maximum pressures, was achieved in the design that we describe here. In this study we experimentally verified the feasibility of twin chamber design up to pressures as high as 33 GPa. Quantitative analysis of the optimum gasket design is beyond the scope of this work. However, theoretical models by finite element analysis or similar methods that have been used for single sample chamber gasket design (e.g., Refs. 9–11) are very useful in understanding the criteria for the stability of the gasket in twin chamber design.

A gasket with twin sample chambers was prepared with some modification after the model by Shukla et al. A gasket was pre-indented with 500 μm culet diamonds to <50 μm thickness, gaskets with a greater than 50 μm thickness were found to be unstable and prone to collapse. Two 120 μm diameter holes were drilled symmetrically with an electric discharge machine (EDM) less than 20 μm apart from each other. The holes are drilled with micron precision by a modified EDM from Hylozoic Products. Micrometer precision for drilling is achieved by adding a centering microscope to the EDM stage. Here we find it useful to outline the design in detail. Two identical magnetic kinematic mounts are secured under the EDM wire and under a centering microscope, respectively. The gasket was held in a cup that could be moved between the magnetic mounts, returning to exact position in each location. Initially, a centering mark is drilled on the edge of the gasket, well removed from the pre-indentation area, then the gasket is moved under the microscope. In the next step, microscope, which itself is mounted on an XY stage, is centered on the mark. Then the gasket was centered with respect to the microscope and shifted on one axial line away from the center for half the desired distance between the holes. The gasket is then moved under the EDM wire and the first hole drilled. The gasket is moved back under the centering microscope, which should be centered with the drilled hole if the alignment is maintained. Next the gasket is moved along the same axial line, proper distance away from the first hole, and the next hole is drilled by the EDM.

For transport measurements, the gasket must be electrically insulated. Thermally conducting STYCAST 2850 KT epoxy by Emerson and Cuming was used to insulate the gasket from the DAC, also to insulate the top of the gasket. A thin layer of epoxy was painted onto the entire top surface, saved for a small region to attach an electrical lead to the gasket, and roughly one third into the pre-indentation. At least three spherical rubies were loaded into each gasket hole before packing each hole and the pre-indentation with a combination of fine lithium fluoride and alumina powder which serves as a pressure medium as well as electrical insulation. The powder is pressurized until the gasket holes slightly deform and the powder becomes clear. Depending on the material studied in a particular experiment, a range of different insulating solid pressure transmitting materials, such as NaCl or a mixture of epoxy and alumina, can be used instead of the particular combination we used here. The resulting layer is then secured on its outer edges to the pre-indentation area with Cyanoacrylate adhesive. Smaller holes are poked into the gasket holes with a 70 μm tipped acupuncture needle.

Two quasi-four probes are built onto the insulating gasket using leads cut from a 25 μm thick platinum foil. The Pt leads are secured to the top of the gasket with Cyanoacrylate adhesive and the ends tied to 35 AWG copper leads. The joints between the Pt and Cu leads are secondarily secured with conducting silver epoxy over the mechanical connection. To prevent the failure of the entire pressure run, two additional leads were added to each quasi-four probe as back-up leads (Figure 2, inset).

To test the system we used two small pieces of two different samples of $\text{YBa}_2\text{Cu}_3\text{O}_{x-\delta}$ ($0 \leq x \leq 0.65$), with slightly different superconducting transition temperatures at ambient pressure (Figure 3). The samples were labeled A and B with ambient pressure superconducting transition temperatures of 91.2 K and 89.2 K, respectively. The samples were cut and loaded into each gasket hole making electrical contact with the respective quasi-four probes. Electrical contact between the samples as well as electrical contact of either sample with the gasket or the body of the DAC was checked and ruled out before beginning the pressure run and each data point.

The AC resistivity of both samples was recorded with two separate SR3830 lock-in-amplifiers. The reading from both lock-in amplifiers as function of temperature was recorded simultaneously using a LabView program. Since in this design samples and the electrical leads are very close to each other, it is necessary to pay close attention in designing the
measurement circuit. To avoid electrical interference, which would be detected by both lock-in-amplifiers if the measurements are carried out at the same frequency, the transport properties of the samples were measured in two different frequencies. The frequency for sample A was 7.061 Hz and for sample B 13.023 Hz (Figure 2). The DAC was cooled to liquid nitrogen temperature inside a Janis SVT-200 cryostat with a calibrated diode thermometer in thermal contact with the DAC. In this calibration test we compared the pressure dependence of the superconducting transition temperature of the two samples up to 11 GPa. Above 11 GPa, the $T_c$ of sample A dropped below the minimum temperature that could be measured in our liquid nitrogen cryostat. The pressure in the two chambers started to deviate noticeably above 5 GPa. The deviations of the pressures in sample chambers do not create a problem as long as the difference is known. Three rubies in each sample chamber were spread and their fluorescence was measured for each different pressure. The correlations between pressures of the sample chambers are plotted in Figure 1. From 1 GPa to 3 GPa, the pressures in each sample chamber are very close to being equal to each other, with the pressure in sample B being slightly higher than sample A. Above 5 GPa, the pressure began to diverge more significantly. While the gasket holes continued to deform with increasing pressure, their deviation from the central radial line of the culet did remain symmetric up to the highest pressure (Figure 4). The pressures, as divergent as they became, remained within the pressure gradient of each other, the highest pressure in chamber A being close or higher than the lowest pressure in chamber B. The rubies were not completely symmetric inside each sample chamber, the rubies for chamber B were placed closer to the center of the culet than the rubies for chamber A. The initial drift of the sample chambers slowed down as the pressure increased and there was no sign of approaching the maximum possible pressure.

**CONCLUSION**

This experiment has shown the feasibility of conducting a comparison of two samples within the same diamond anvil cell. The ability to measure two samples simultaneously allows for temperature dependent properties to be precisely recorded even if the method of thermometry is not as absolutely accurate. The simultaneous measurements of transport properties as a function of temperature have been shown to be precise and capable of determining differences of 1 K or less.
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