Moissanite-anvil cells for the electrical transport measurements at low temperatures

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Abstract. We have successfully measured the Hall effect of a single crystal of a high temperature superconductor La$_{2-x}$Sr$_x$CuO$_4$ in moissanite-anvil high pressure cells. A pressure cell with new Zylon-gasket and wiring arrangement survived under pressure up to at least 5 GPa. Pressure which was clamped at room temperature increased with lowering the temperature down to below 60 K by a factor of 1.3-1.4.

1. Introduction

High-pressure effect on the oxide high-temperature superconductors (HTS) is important to clarify the origin of the phenomena [1]. The Hall coefficient ($R_H$) measurement is useful in studying the carrier concentration, which is one of the key quantities for HTS. There have been several challenging high pressure works on the electrical transport properties using diamond-anvil cells [2-7]. Difficulties when used under the low temperature and high magnetic field conditions are in (1) the electrical feedthroughs, (2) the contacts of the leads with the sample, (3) the pressure medium and its hydrostaticity and (4) the electrical and magnetic characteristics of the gasket materials as well as (5) the cell size. Some of these methods and materials selection are mutually affected. We have studied the use of Moissanite-anvil cells to explore the Hall effect measurements under pressure, especially of the single crystals of high temperature superconductors.

2. Experimental

Hall effect measurements of HTS require high magnetic field and low temperature. Therefore, the sample cell should have smaller volume, non-magnetic materials, smaller mass/heat capacity, and low heat generation during the rotation. A diamond-anvil type setting was used in our study but with synthetic moissanite as an anvil material. The moissanite has Mohs scale hardness of 9.25 similar to the value 10 for diamond and was chosen because of its reasonable price. The size of an anvil was 4 mm in table diameter, 2.5 mm in height and 1.2 mm in culet diameter. The outer size of the cell made of CuBe was 11.4$^\text{H}$×13$^\text{H}$ mm. We used a stack of Zylon fiber coils embedded in Stycast 1266 as an outer gasket and diamond paste (1/4 $\mu$m powder with Stycast 1266) for an inner gasket, instead of a metal plate, as in figure 1. This composite gasket is insulating and avoids electrical shorts between the...
leads. Pressure medium was Phomblin oil that is non-reactive to the paste for the electrical contacts described below and also non-reactive to organic materials.

Figure 1. Zylon fiber gasket and anvil assembly. The outer gasket with diameters 3.0 and 1.1 is the Zylon gasket and the inner one with 1.1 and 0.45 is diamond paste. The seizes are in unit of mm.

Figure 2. Wiring arrangement with additional two dummy wires to balance the forces during the setting.

Figure 3. Pressure cell set in a rotor which is rotated by a fishing line [10]. The two glass fibers are inserted, one for the pressure cell and one for the ambient pressure reference.

Figure 4. Pressure at room temperature as a function of applied load.

A high quality single crystal of La$_2$-xSr$_x$CuO$_4$ ($x = 0.090$) [8] was used with a dimension of 280×50×25 μm and placed in the sample space of 450×860 μm. The crystal was placed in the sample chamber so that the a-b plane is parallel to the anvil surfaces. Four electrical contact tabs were made by Au-sputtering with thickness of 200 nm on the sides of the crystal and annealing at 600 °C for 90 min. Each tab was connected to a gold thin foil of 1-2 μm thickness and 10-20 μm width as a lead wire, as described in [2]. In this study we used for the contact graphite paste (Dotite XC-12 from Fujikura Kasei Co., Ltd) with solvent (2-Butoxyethyl acetate : n-Octyl acetate = 10 : 1), instead of
Since the crystal of HTS is a layered compound with high anisotropy, it was crucial to attach the wires to the sides of the crystal, especially the current wires for the a-b plane transport measurements, to obtain a uniform current flow through the stack of a-b planes in the crystal. The Hall voltage was measured by the AC resistance bridge LR-700 with an excitation current of 100 $\mu$A.

3. Results and discussion

We found that the geometrical arrangement of electrical leads on the gasket surface for current and voltage and the good electrical contact at the sample surface were important for the successful setting and measurements. During the course of trial and error of wire setting, the minimum number of wires four was not enough in terms of mechanical balance of wires and a sample. After trying three possible arrangements with four wires, we finally achieved a successful setting with six wires which were placed symmetrically in the sample region as in figure 2. The two of the four current lead wires were dummy and used for balancing the forces on the sample during the setting process. The good electrical contact was achieved by the Au-sputtering and the graphite paste as described above. The annealing at 600 °C after the Au-sputtering was found to be important.

The gasket with Zylon fibers and diamond paste survived at least to 5 GPa at room temperature, which was the highest one in the present series of experiments. The pressure was measured by a ruby manometer. The pressure efficiency was 18.4 GPa/tonf at room temperature, as in figure 4. The temperature effect on the ruby manometer was eliminated by a reference ruby under ambient pressure which was attached to a second glass fiber and placed close to the periphery of the high pressure cell, as in figure 3. The pressure clamped at room temperature was found to increase with lowering the temperature as in figure 5. The factor of the increase was 1.3-1.4, and tended to saturate below 60 K. In comparison, Thomasson et al. [4] observed a pressure decrease with lowering the temperature by a factor of 0.7. The difference will be due to the differences in the pressure cell design and the pressure gasket and medium. They used BeCu as a non-magnetic cell material, similar to ours, but also they used Belleville spring washers, which was intended to minimize pressure changes during temperature variation. They have observed below 125 K a stronger decrease in pressure which was ascribed to the solid-liquid phase transition of the pressure medium $^4$He.

The Hall voltage at 45 K, for example, was 1 $\mu$V or less in the 20 T region of magnetic field with voltage error of the order of 10 nV. Thus the $R_{H}$ in the a-b plane was successfully measured with good accuracy as in ambient pressure experiments. The pressure dependence $\frac{\text{d} \ln R_{H}}{\text{d} P}$ was about -2.3 %/GPa and -1.5 %/GPa for 296 K and 77.4 K, respectively [9]. In the La-Sr-Cu-O HTS system, the

![Figure 5. Change of pressure at low temperatures. There are two runs for the 1.9 GPa experiment.](image)
$R_H$ has been believed to be constant under pressure up to 1.4 GPa from the polycrystalline sample data [11, 12]. Although our study needs more experiments over a wide range of Sr-composition to compare with the previous results, it suggests that the single crystal study will be required to elucidate the pressure effect of La-Sr-Cu-O system.

In summary, we have described the diamond-anvil technique for the Hall effect measurements at low temperatures, using new materials for the anvil and gasket. We believe that the present experimental technique will be promising in the development of high pressure study of the electrical transport properties.

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