A technique for precise electrical-transport measurements under pressure above 10 GPa

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Abstract. We report a technique for the precise measurement of electrical resistivity at high pressures up to 15 GPa by using Bridgman anvils. The relatively large size of the pressure chamber (1.0 mm in diameter) allows the use of large specimens and simple experimental procedures rather than using a standard diamond anvil cell. A SUS310 gasket is pressed by two tungsten carbide anvils. A sample with typical dimensions of approximately $0.5 \times 0.2 \times 0.1 \text{mm}^3$ is placed in a small hole of the gasket. In order to obtain a quasi-hydrostatic pressure, the pressure chamber is filled with a 1:1 mixture of Fluorinert FC70 and FC77 as the pressure transmitting medium. Electrical leads are introduced through shallow grooves milled into the anvil. The grooves are filled with a mixture of alumina powder for insulation. Accurate data of the resistance values of Bi and Fe at room temperature are available. We observe sharp transitions for Bi at 2.55, 2.7 and 7.7 GPa. The electrical resistance of Fe shows a sudden increase due to a structural transition near 14 GPa.

1. Introduction
It is well known that the application of high pressure induces various types of electronic transitions in materials. One of the typical examples is the pressure-induced quantum phase transition[1]. Many interesting electronic properties are often found near the quantum critical point under high pressures at low temperatures. In view of these pressure-induced phenomena, the electrical resistance measurement under high pressures has been recognized as one of the powerful tools to investigate the electronic properties.

However, all pressure transmitting medium freeze upon cooling and subject the immersed sample to shear stress of varying strengths. In order to maintain the sample under hydrostatic pressures at low temperatures, a wide variety of techniques have been used[2]. In general, a large working space and a fluid pressure transmitting medium are needed to generate hydrostatic pressures. Piston-cylinder devices have been used so far for precise measurements because of their large working space in which quasi-hydrostatic pressure is generated; it enables the measurements of not only the electrical resistance and the ac magnetic susceptibility but also the thermal expansion by using a strain gage method[3]. However, the generated pressure is limited at the most up to 3 GPa.

A diamond anvil cell is widely used for measurements that require higher pressures. By using helium as the pressure transmitting medium, purely hydrostatic pressures can be generated[4]. However, it is inconvenient to study the electronic properties since the sample space is extremely small; therefore, it is difficult to attach electrical leads.
Recently, Mōri[5] and Nakanishi et al.[6] developed a Bridgman anvil apparatus that yielded nearly hydrostatic pressures in which a large working volume of $\phi 1.0 \times 0.9$ mm$^3$ and a fluid pressure transmitting medium were realized in a Teflon capsule between anvils made of tungsten carbide (WC). However, the generated pressure is limited at the most up to 8 GPa.

In the present study, we describe a new technique for the precise measurement of the electrical resistivity under high pressures up to 15 GPa by using Bridgman anvils. The relatively large size of the pressure chamber (1.0 mm in diameter) with a fluid pressure transmitting medium allows not only to maintain nearly hydrostatic pressures but also simplifies the experimental procedures as compared to those of the standard diamond anvil cell.

2. Experimental setup
Figure 1 shows the schematic cross section of the present high-pressure cell, which consists of two anvils made of (A) WC, (B) a metal gasket, and (C) a ring to tighten the gasket.

The anvils were grounded with a taper angle of $10^\circ$ and terminated at a flat tip with a diameter of 3.0 mm diameter. Four shallow grooves are milled into the lower anvil in order to introduce electrical leads into the pressure chamber. The grooves are filled with alumina powder, which ensures electrical insulation as well as sealing off the pressure chamber.

The metallic gasket is made of stainless steel (SUS304 or SUS 310) with a thickness of 0.5 mm and diameter of 8.0 mm. A sample hole with a diameter of 1.0 mm is prepared as the pressure chamber. A sample with typical dimensions of approximately $0.5 \times 0.2 \times 0.1$ mm$^3$ is placed in the hole. The gasket is tightened by using a metal ring – made of alloys such as SNCM8 alloy, NiCrAl alloy and so on – with a Rockwell hardness on the C-scale (HRC) of $\sim 50$; the ring prevents the movement of the gaskets when it is compressed by the anvils. After precompression, a 1:1 mix of Fluorinert FC70 and FC 77 is filled in the pressure chamber as the pressure transmitting medium. To compress the metal gasket, a force is generated and applied to the WC anvils. Resistivity measurements are carried out using the standard four-probe technique.

3. Pressure calibration
Pressure in the cell was calibrated according to the phase transitions of Bi and Fe at room temperature. Figure 2 shows the results of the resistance of Bi at room temperature by using our pressure apparatus. It is well-known that Bi has three phase transitions: Bi I-II at 2.55 GPa, Bi II-III at 2.7 GPa, and Bi III-IV at 7.7 GPa[7]. As shown in Fig. 2, all the three transitions are observed in our result for the electrical resistance. The sharpness of the transitions shows that a hydrostatic pressure up to 8 GPa is obtained in the pressure chamber.
Next, we show the results for the resistance of Fe at room temperature in Fig. 3. At first, the electrical resistance $R$ decreases as the pressure load is applied. After showing a minimum at 5.9 ton, $R$ shows a sudden increase near 7 ton. Although we stopped applying the pressure load at 8 ton and waited for several hours, $R$ continued to increase. Since the increase in $R$ reflects the change in the electron-phonon scattering, it arises from a first-order transition from the bcc $\alpha$-phase to the fcc $\epsilon$-phase near 13~14 GPa\cite{8, 9}. As shown in the results, the transition is sluggish and observed over a much broader pressure gradient. In order to exactly calibrate the pressures, the measurements according to other standard materials are needed.
4. Summary
We have succeeded in developing a new high-pressure apparatus for measuring the electrical resistivity under hydrostatic pressures up to $\sim 15$ GPa. Because of its compact size – nearly as large as a conventional piston-cylinder cell, it can be easily combined with a cryostat and a superconducting magnet. To verify the performance of our pressure apparatus, the electrical resistances of Bi and Fe have been measured at room temperature. The measurements according to other standard materials will be needed to calibrate the pressure at low temperatures.

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