Fabrication of a barrel-type modified Bridgman anvil cell

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Abstract. We fabricated a small Bridgeman anvil cell with a barrel-like shape for measurements under high-pressure up to 8 GPa. A Teflon cell with an inner volume of 1.5 mm in diameter and 2 mm in length is inserted in the pyrophyllite gaskets and is fixed between upper and lower tungsten-carbide (WC) anvils. The anvils are fastened by a Cu-Be holder with an outer diameter of 40 mm and 43 mm in length. Both of the top and bottom edges of the holder are tapered. This barrel-like shape enables rotation in a superconducting magnet with an inner diameter of 54 mm. The pressure is calibrated by measuring the resistivity of Bi at room temperature. The resistivity of Bi changed under loads at 8.0, 8.45, and 14.7 tons, corresponding to the structural phase transitions.

1. Introduction
Pressure plays an important role in a study of the field of strongly correlated electron systems. It is a clean parameter unlike chemical substitution: Lattice parameters can be changed by applying pressure without introducing randomness. Under high pressure, some materials exhibit interesting phenomena. For example, a spin-ladder cuprate Sr_{14-x}Ca_xCu_{24}O_{41},[1] a series of β-vanadium bronzes β-A_{0.33}V_2O_5 (A=Li, Na, and Ag),[2] and Bechgaard salts exhibit pressure-induced superconductivity. However, some of the compounds have not been investigated precisely because of experimental difficulties under high pressure: β-vanadium bronzes exhibit superconductivity above 6.5 GPa,[3] which exceeds the upper pressure limit of a piston-cylinder-type pressure cell.[4] Therefore, improvements in high-pressure techniques are highly desired.

The upper pressure limit of a diamond anvil cell is more than 50 GPa, however, its application to various research is limited because of the small sample space with less than 0.5 mm in diameter.[5] It is not available for measurements which need large sample volume. A cubic anvil cell is a promising apparatus for various measurements with using a sample of a relatively large size under high pressure. The upper pressure limit of this cell is more than 10 GPa, and the sample space with 1.5 mm in diameter and 2.0 mm in length is much larger than that of the diamond anvil cell.[6, 7] However, a cubic anvil cell is not suitable for the measurements under high magnetic field because the size of the cell is too large compared with the bore of a conventional superconducting magnet.

We focused on a modified Bridgman anvil cell developed by Nakanishi et al. for measurements under high magnetic field.[8, 9] The upper pressure limit of this apparatus is more than 8
Figure 1. Cross-section view of the barrel-type modified Bridgman anvil cell.

GPa. The effective working volume is 1.0 mm in diameter and 0.9 mm in length, and is large enough for various measurements. Moreover, this apparatus has a compact body with 38 mm in diameter and 140 mm in length. This size allows measurements in a conventional superconducting magnet. For example, Nakanishi et al. studied the magnetic field effect on the pressure-induced superconducting state in \( \text{Sr}_2\text{Ca}_{12}\text{Cu}_{24}\text{O}_{41} \) up to 5.7 GPa.\[11\]

On the other hand, the field direction against the sample crystal is an important factor for measurements under magnetic field. The above mentioned modified Bridgman anvil cell is too long to turn in a conventional magnet, therefore, the magnetic field can be applied only for the axial direction. In this study, we fabricated a shorter modified Bridgman anvil cell with a barrel-like shape to fit in a conventional magnet. Hereafter, we call the barrel-type modified Bridgman anvil cell as the barrel-type cell and call the above mentioned modified Bridgman anvil cell as the standard cell. We calibrated the pressure in the barrel-type cell from the load dependence of the resistivity of Bi at room temperature.

2. Pressurizing apparatus

Figure 1 shows a cross-section view of the barrel-type cell. A Teflon cell with an inner volume of 1.5 mm in diameter and 2.0 mm in length is inserted in the pyrophyllite gaskets and is fixed between upper and lower tungsten-carbide (WC) anvils. The thickness of the anvil is 8.0 mm, and the diameter of the anvil face is 8.0 mm. As terminals of electrical lead, stainless plates with 0.3 mm in thickness are inserted in the gaskets. In order to press them straight, the anvils and gaskets are supported by a Cu-Be guide. The anvils are fastened by a Cu-Be holder. The outer diameter of the holder is 40 mm and the length is 43 mm. Both of the top and bottom edges of the holder are tapered. This barrel-like shape allows rotation in a superconducting magnet with an inner diameter of 54 mm. The effective working volume of the apparatus is large enough to measure a sample with the size of about 0.9×0.3×0.3 mm\(^3\).

3. Experimental results

For pressure calibration of the barrel-type cell, the resistivity of Bi at room temperature was measured by a conventional four-probe method. As a pressure mediation liquid, we used a mixture of fluorinated liquids, Fluorinert FC-70 and FC-77. For comparison, we also measured the resistivity of Bi with the standard cell. In both measurements we used a Teflon cell and gaskets of the same size. Figure 2 shows the load dependence of the resistivity of Bi. Open
Figure 2. Load dependence of the resistivity of Bi at room temperature. Open circles indicate the resistivity measured with the barrel-type Bridgman anvil cell and closed squares indicate the resistivity measured with the standard cell.

circles indicate the resistivity measured with the barrel-type cell, and closed squares indicate the resistivity measured with the standard cell.

It is well known that structural phase transitions of Bi, I-II, II-III, and III-V transitions, occur at 2.55, 2.77, and 7.68 GPa, respectively, accompanied by remarkable changes in resistivity. As shown in Fig. 2, the resistivity measured with the barrel-type cell showed the changes taking place at about 8.0, 8.45, and 14.7 tons, respectively. On the other hand, the resistivity measured with the standard cell showed the changes taking place at about 9.3, 9.7, and 19.0 tons, respectively.

Figure 3 shows the relationship between the load and the pressure in the Teflon cell obtained from the resistivity of Bi. Open circles indicate the results for the barrel-type cell, and closed squares indicate those for the standard cell. As shown in the figure, the curves tend to bend upward with increasing load. This trend is mainly attributed to the behavior of the pyrophyllite gaskets under the applying loads. Beginning to apply loads, the pyrophyllite gaskets are squeezed out considerably and such a deformation causes the loss of pressure in the cell. With increasing loads, however, the force at the anvil face makes the pyrophyllite keep not to flow outside so much, so that the pressure inside the Teflon cell increases efficiently. Therefore, the load efficiency is enhanced with increasing load. Moreover, it is found that the slope of the curve for the barrel-type cell was steeper than that for the standard cell. This suggests that the load efficiency of the barrel-type cell is better than that of the standard cell.

Finally we mention a problem of the barrel-type cell. In several experiments, cracks were observed in the upper WC anvil after releasing the load. The cracks are likely attributed to the thickness of the anvil. The thickness of the anvil of the barrel-type cell is about a half of that of the standard cell. Therefore, only a small part of the anvil is supported by the Cu-Be guide, and it is difficult to press the Teflon cell and the gaskets straight. When the load is not imposed straight, the anvil will be distorted. In order to use this apparatus at higher pressure more than 8 GPa, improvements of the WC anvil are needed.

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Figure 3. Pressure calibration curves at room temperature. The open circles and closed squares indicate the point of phase transitions, which are observed for the barrel-type modified Bridgman anvil cell and the standard cell, respectively.

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