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Precise Electrical Transport Measurements by Using Bridgman Type Pressure Cell at Low Temperature

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Abstract. We report a technique for the precise measurement of the electrical resisivity under high pressure at low temperature by using Bridgman anvils made of tungsten carbide. Quasi-hydrostatic pressure is generated up to ∼15 GPa in the relatively large working space which allows the use of large specimens and simple experimental procedures rather than using a standard diamond anvil cell. The application is demonstrated by the measurements of the electrical resistivity of lead in order to describe the effect of pressure on the superconducting transition.

1. Introduction

High pressure technique at low temperature has been proved to be one of powerful tools in studies of recent topics related to be fundamental problems in strongly correlated electron systems. Many interesting electronic properties are often found near the quantum critical point under high pressures at low temperatures[1].

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. 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[2]; it enables the measurements of the electrical resistance, the ac magnetic susceptibility, the thermal expansion and so on[3, 4, 5]. However, the generated pressure is limited at the most up to 3 GPa.

To generate hydrostatic pressure above 10 GPa, a diamond anvil cell (DAC) is widely used. However, the sample space must be extremely small, so that we need special technique to attach electrical leads to the sample. Instead, quasi-hydrostatic pressure has been developed by using a new high-pressure cell, which consists of Bridgman anvils made of tungsten carbide (WC). It is possible to have a large working volume at least one order of magnitude larger than that of DACs. In the present work, we describe the effect of pressure on the superconducting temperature of lead up to 15 GPa by using this high-pressure device. A preliminary results obtained at room temperature has been published elsewhere[6].

2. Experimental setup

Figure 1 shows the photograph of the high-pressure cell. Pressure is generated in a gasket (4) between anvils made of non-magnetic WC (2,5). The gasket is made of stainless steel (SUS304 or SUS310) and supported by a metal ring, which keeps the gasket from going away and enables the pressure in the pressure chamber to be more than 10 GPa. On the lower anvil (5), we prepared a pit, 1.0 mm in diameter and 0.1 mm deep. The sample is set on the lower anvil after we attached Cu or Au wires of $\phi 10 \sim \phi 20$ μm. The grooves are filled with almina powder and STYCAST2850 which insures electrical insulation as well as sealing off the pressure chamber[6]. Since the sample hole is 1.0 mm in diameter and 0.5 mm thickness, is the zero-pressure volume is nearly 0.47 mm^3, which is at least one order larger than that of typical DACs[7].
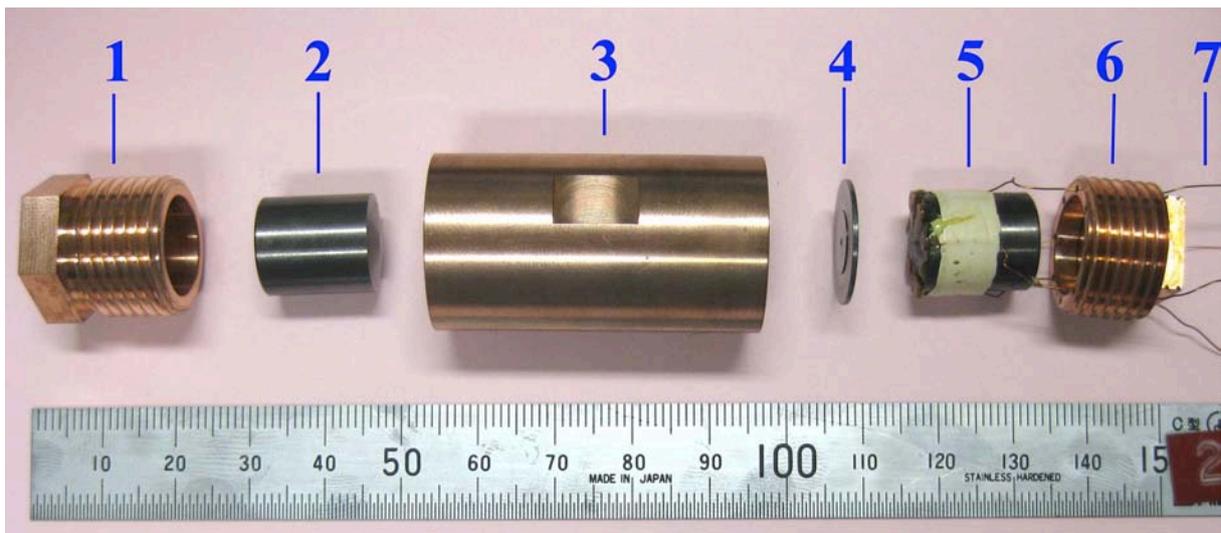

Figure 1. Photograph of the high-pressure cell: (1) upper screw, (2) upper anvil, (3) cylinder, (4) gasket, (5) lower anvil, (6) lower screw, (7) electrical leads. See the text for details.

Here we filled a 1:1 mix of Fluorinert FC70 and FC77[8] in the pressure chamber as pressure transmitting medium. Although all of pressure transmitting medium solidify at high pressure, the measurements in a liquid pressure medium are indispensable to obtain reliable values. Indeed, the different behaviors of the temperature dependence of the electrical resistivity are observed between the measurement by using the solid medium and that by using the liquid medium[9, 10].

The cell body is made of hardened beryllium copper (BeCu), 50 mm in length and 22 mm in diameter (3). In order to introduce the electrical leads into the cell body, the lower screw has several holes with a diameter of ϕ 1 mm (6). The load is applied to the anvils by the WC piston and is clamped by tightening the upper screw (1). On the other hand, this device can also be attached to the high-pressure apparatus based on a Swenson type one[2], in which a forth applied to the piston is controlled by a hydraulic press in order to keep the load constant during the cooling and warming processes.

3. Performance

To demonstrate the measurement using this pressure cell, we measured the temperature dependence the electrical resistance of lead. The superconducting temperature T_C was determined from the midpoint of the resistance drop associated with the superconducting transition. Figure 3 shows one of the results by using a clamp cell, in which the load is applied
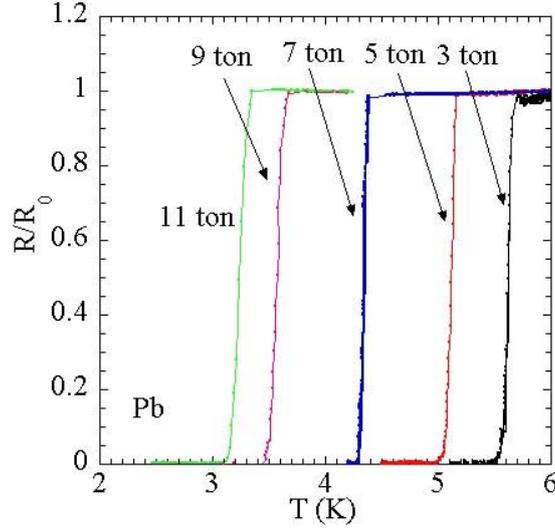
Figure 2. Temperature dependence of the relative electrical resistance of lead at various loads by using a clamp cell. R_0 is the resistance in the normal conducting state.

at loom temperature. It is noted that a transition width ΔT_C under high pressure still remains 0.2 K which is almost same as that at ambient pressure. This result clearly means that the generated pressure in this pressure cell are close to the hydrostatic one although the pressure transmitting mediums is solidified in this temperature region. As the applied load is increased by the pressure cell, T_C shifts to lower temperature. At 11 ton, T_C is obtained to be 3.2 K, indicating that the pressure in the pressure chamber comes to 15 GPa[11].

Figure 3 shows the temperature dependence of the electrical resistivity $\rho(T)$ of lead at low temperature, in which the load is applied by using a direct compression method. The $\rho(T)$ curves are similar to that of , where the pressure is applied by using clamp cell; ΔT_C remains 0.2 K under high pressure. Figure 4 shows the $\rho(T)$ curve in the wide temperature range up to room temperature. Above 50 K, the resistivity is described as $\rho = A + BT$, where A and B are -0.67 $\mu\Omega$cm and 0.045 $\mu\Omega$cm/K at 2.5 ton, -0.57 $\mu\Omega$cm and 0.036 $\mu\Omega$cm/K at 4 ton and -0.52 $\mu\Omega$cm and 0.031 $\mu\Omega$cm/K at 5 ton, respectively. The value of A increases as increasing the applying load, which is consistent with the fact that the residual resistivity also increases as seen in figure 3, indicating that some of uniaxial stress is applied to the pressure chamber.

On the basis of these results a pressure calibration curve has been obtained. Figure 5 shows the true pressure in the pressure chamber against the load. At low temperature, the true pressure is determined from T_C of lead by using the data of Tomasson *et. al*[11]. The pressure is increased in proportion to the applied force but saturated slightly above 14 GPa. High pressure of 15 GPa was successfully produced at the load of 11.0 ton.

Several fixed points of Ce, Bi and Fe are also plotted at room temperature from the recent results[6]. The the pressure calibration curve at low temperature is in good agreement with that at room temperature at the load of 2.5∼3.0 ton, which corresponds to 3∼4 GPa. However, the reduction in pressure is observed as the press force is increased; the pressure efficiency reaches about 70 % above the load of 4.5 ton.

It is found that the reduction of pressure at low temperature can be seen by using both clamp cell and and a direct compression method. This reduction can be explained by temperature-dependent thermal expansions of the pressure devices. The load may contribute not only to the pressure chamber but also to the gasket made of hard metal in the case of the Bridgman anvil
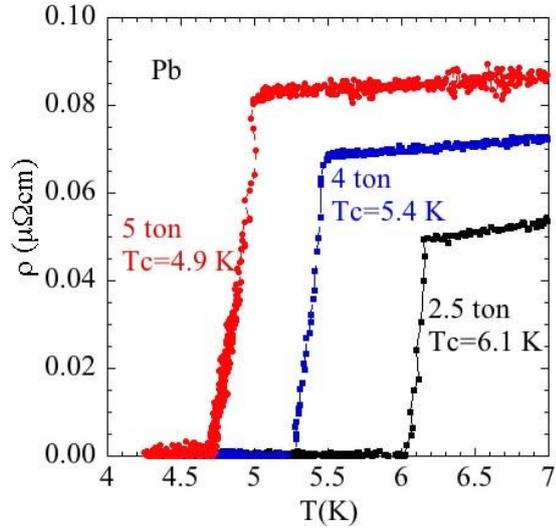
Figure 3. The electrical resistivity of lead at low temperature for several pressure loads by using a present pressure device with direct compression method.
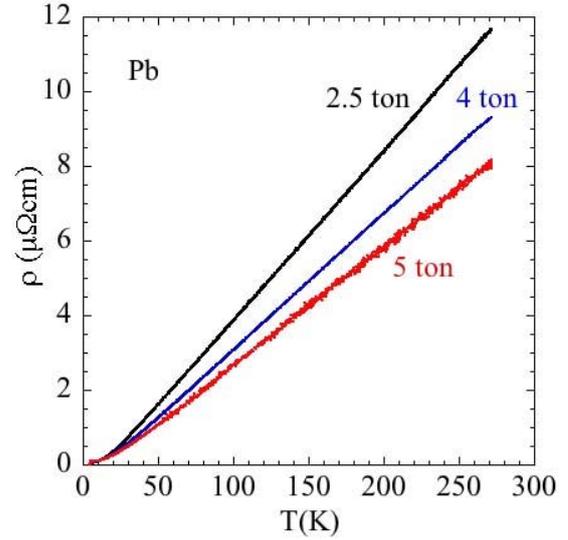

Figure 4. The electrical resistivity of lead as a function of temperature up to room temperature for several pressure loads.
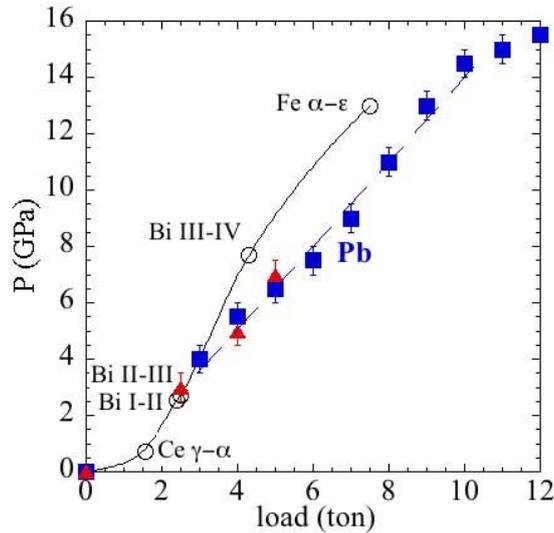

Figure 5. Pressure in the pressure chamber as a function of press force at room temperature (open circles), at low temperature by using a present pressure device with direct compression method (triangles) and at low temperature by using that with a clamp cell (squares).

cell, so the thermal expansion in the pressure chamber cannot be compensated even in a direct compression method. The data of temperature dependence of the decrease of pressure is needed in detail in order to determine the pressure more accurately.

4. Summary

We have demonstrated the precise measurement of the electrical resistivity under quasi-hydrostatic pressure at low temperature by using Bridgman anvil cell, which has relatively

large working space compared with a typical diamond anvil cell. For the demonstration of our pressure devices at low temperature, we measured the electrical resistivity of lead as a function of temperature. We observed sharp transitions for the superconducting transition temperature of lead under the high press force. The present result means that a high pressure up to 15 GPa is successfully produced in the pressure chamber at low temperature down to ∼1.5 K.

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