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High-pressure apparatus for the measurement of thermal and transport properties at multi-extreme conditions

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Abstract

A high-pressure apparatus which we can use at low temperature and high magnetic field has been designed for making measurements of thermal and transport properties of condensed matter. The details of the apparatus and some examples of the measurements are described and briefly discussed. It is proved that the apparatus is suitable for use up to hydrostatic pressure of ∼3.5 GPa, down to ∼2.0 K and up to ∼11 T.

1. Introduction

The measurements of physical properties under multi-extreme conditions, i.e., high pressure, low temperature and high magnetic field, attract much attention in various fields of science and engineering. Although there are lots of technical difficulties in such measurements, we could get more information about the electronic and crystalline structure of materials than that obtained when we change only one external force. In addition, such conditions sometimes induce an exotic electronic transition or crossover, which gives much important and helpful information helpful for understanding the electronic properties of solids [1]. In the present work, we report on a new type high-pressure apparatus in which we can apply hydrostatic pressure up to 3.5 GPa and magnetic field up to 11 T. Several examples of measurements of thermal and transport properties using this apparatus are described.

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2. Experimental details

2.1. High-pressure apparatus

Figure 1 shows a schematic cross-section of the present high-pressure apparatus. Here, (1) is the pressure intensifier which generates loads up to 30 ton with automatic control by a hydraulic pump; (2) is a piston made of SUS304; the He chamber is sealed by an O-ring (3); the load is transmitted through the compression member (4) to the high-pressure cell; (4) is an alternating pile of fibre-reinforced plastic (FRP) discs; in the present case, a piston–cylinder type apparatus is shown, in which the tungsten carbide piston (8) and Cu–Be or Ni–Co–Cr–Mo (MP35N) alloy cylinders (9) are used; (5) is the tension member made from SUS304; a superconducting magnet (10) is made of a NbTi superconducting coil located around (9), (6) is the thermal radiation shield and (7) is a cryostat. The load is always kept constant automatically within less than $\pm 1\%$ error. (4) has a role not only as a compression member but also as a thermal insulator, which is a very important role when we use liquid He. The maximum magnetic field, in the present work, of 9 T at 4.2 K (11 T at 2.2 K) is easily obtained by an Oxford superconducting magnet, (10). The sample was placed inside the Teflon capsule, whose sample space is 4.5 mm in diameter and 20 mm in height. In the present study, we used a mixture of Fluorinert FC70 and FC77 or Daphne7373 as the pressure-transmitting medium [2].

Next we briefly describe the cylinder, which is made of Ni–Co–Cr–Mo alloy having a hardness of HRC (Rockwell hardness in C-scale) ~50 after the appropriate heat treatment. The outer and inner diameters are about 17.5 and 6 mm, respectively. The cylinder is tightened by insertion in a CuBe jacket with the thickness of 5 mm subjected to a force of about 10 ton, in order to increase the strength.

2.2. Pressure calibration

Pressure in the cell was calibrated by observing the phase transitions of NH_4F, KCl and Bi at room temperature [3, 4]. The structural phase transitions of NH_4F I–II at 0.364 GPa, II–III at 1.159 GPa and of KCl I–II at 1.9 GPa have been observed in the load dependence of the volume of the sample filled in the cell, which can be detected by using a dial gauge. Figure 2 shows the result, in which we clearly see two discontinuous changes in displacement of the piston for NH_4F and a small change for KCl. The structural transitions of Bi I–II at 2.55 GPa and II–III at 2.7 GPa have been observed by measuring the electrical resistance R. Figure 3 shows the load versus the electrical resistance of Bi. Two clear jumps of R are detected at 10.7 ton and at 11.9 ton due to two structural transitions. We also measured the electrical resistance of manganin wire in order to estimate the pressure inside the Teflon capsule at low temperature.

On the basis of these results, a pressure calibration curve has been obtained, as shown in figure 4. With increasing load, pressure in the cell tends to saturate slightly. The curve has been fitted by the following equation:

$$P\ (\mathrm{GPa}) = 0.25L - 0.0018L^2$$

where L (ton) is the load. The pressure of 3 GPa is reached at a load of 13.2 ton.

3. Results and discussion

Here we show several examples of measurements obtained by using the present high-pressure apparatus described in section 2.

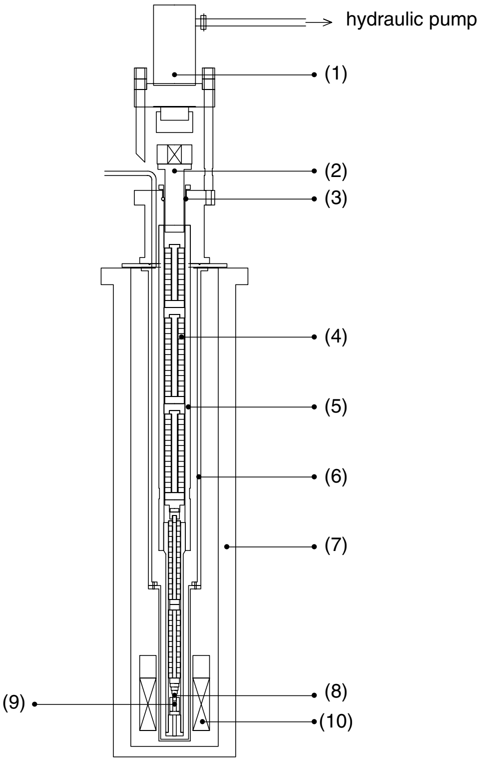

Figure 1. A schematic cross-section of the present high-pressure apparatus. See the text for the details.

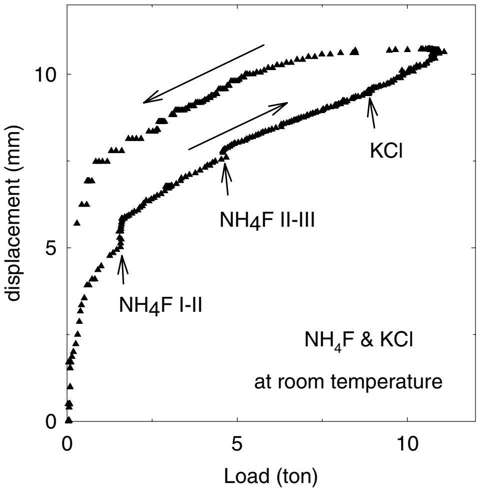

Figure 2. The load dependence of the displacement of the piston.

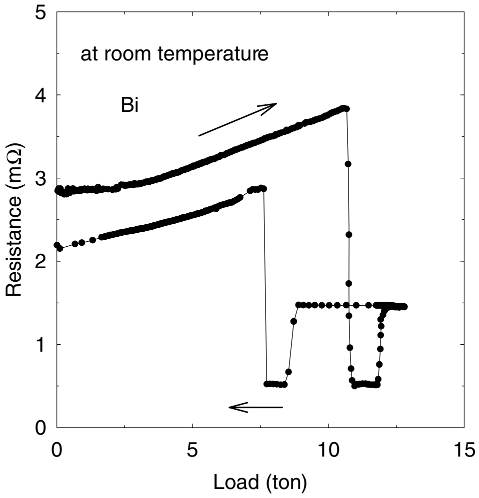

Figure 3. The load dependence of the resistance of Bi at room temperature.

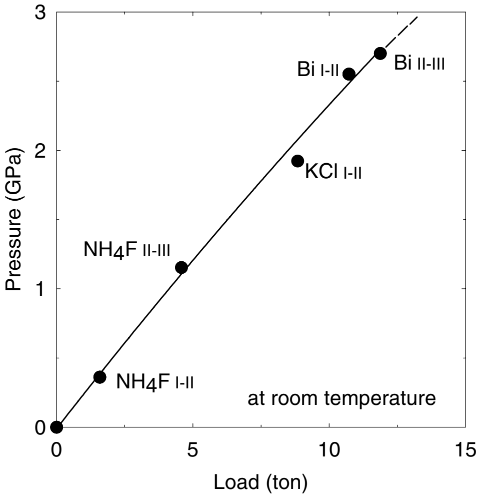

Figure 4. The pressure calibration curve.

3.1. Thermal expansion of α-Mn

Figure 5 shows the fractional change of length $\Delta l/l$ for antiferromagnetic α-Mn at various pressures. $\Delta l/l$ decreases with decreasing temperature down to just above T_N (∼95 K at

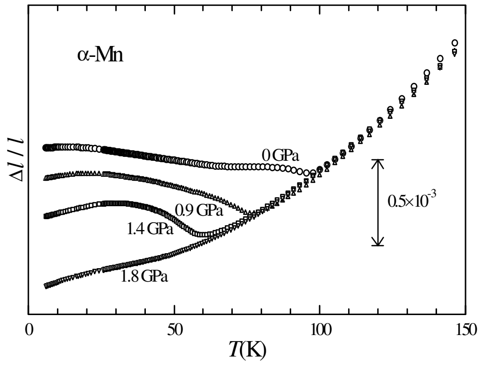

Figure 5. The linear thermal expansion of α-Mn at various pressures.

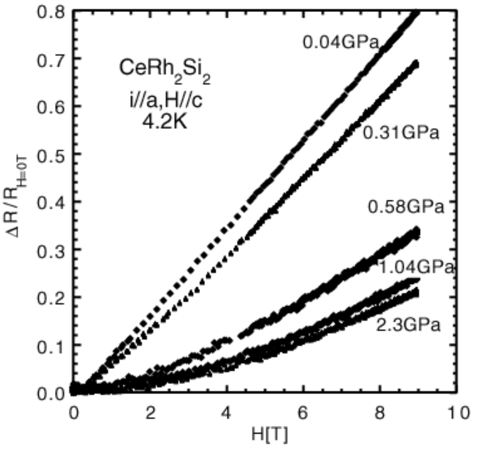

Figure 6. The magnetoresistance of $CeRh_2Si_2$ at 4.2 K.

ambient pressure), and it begins to increase on further cooling, which is well known as a magnetovolume effect caused by magnetic ordering. The temperature at which $\Delta l/l$ shows a minimum goes down with pressure. T_N defined from the thermal expansion coefficient decreases with pressure at a rate of -19 K GPa^{-1}.

3.2. Magnetoresistance of $CeRh_2Si_2$

Figure 6 shows the magnetoresistance $\Delta R/R$ of $CeRh_2Si_2$ single crystal, which is an antiferromagnetic heavy-fermion compound, at 4.2 K under various pressures. Below 0.31 GPa, $\Delta R/R$ increases linearly as magnetic field increases, while it exhibits a H^2-dependence above 0.58 GPa. This may arise from the pressure-induced magnetic phase transition at around 0.6 GPa, i.e., this change corresponds a crossover in the electronic state [5].

3.3. Electrical resistance of Co–Al–O granular film

Figure 7 shows the electrical resistance of a Co–Al–O insulating granular film as a function of temperature. The electron conduction in this material is well known to be dominated by the effect of tunnelling between nano-size Co granules. The resistance R increases with decreasing temperature, which is characteristic of semiconducting transport. However, on applying pressure, the increase in the magnitude of R is found to be suppressed. On applying 3.3 GPa, the value of R at around 80 K becomes about a half of that at ambient pressure. The result, shown in figure 7, implies an enhancement of the tunnelling conduction on applying pressure. Detailed analysis of these data was described in reference [6].

4. Conclusions

We have succeeded in constructing a new high-pressure apparatus by means of which we can apply pressure above 3 GPa or more by using the conventional piston–cylinder method. By combining this high-pressure apparatus with a cryostat and a superconducting magnet, multi-extreme conditions have been obtained readily in a large sample space. Some experimental

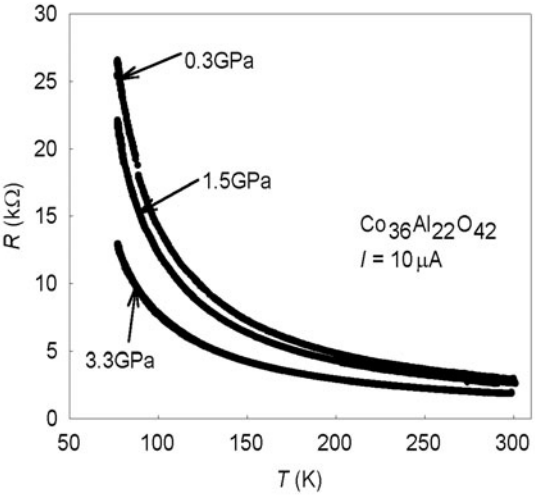

Figure 7. Electrical resistance versus temperature for $Co_{36}Al_{22}O_{42}$ at various pressures.

results (electrical resistance, thermal expansion and magnetoresistance) have been described as the examples. The results prove that the present apparatus has a wide range of usage in many research fields of physics.

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