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New superconducting phase of Bi$_2$Te$_3$ under pressure above 11 GPa

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Abstract. We have investigated the pressure dependence of superconducting transition temperature $T_c$ of Bi$_2$Te$_3$ through electrical resistance measurement under high pressure up to 13 GPa. The value of zero-resistance critical-temperature $T_{c\text{zero}}$ is 2.7 K at 9.0 GPa. Pressurization up to 11 GPa reduces the $T_{c\text{zero}}$. However the pressurization at 13 GPa causes sudden enhancement of $T_{c\text{zero}}$, which is obtained to be 3.7 K. The onset temperature of superconducting transition $T_{c\text{onset}}$ shows the positive pressure dependence under pressure above 10 GPa. The value of $T_{c\text{onset}}$ is obtained to be 5.0 K at 13 GPa. The sharp increase of $T_{c\text{zero}}$ and the positive pressure dependence of $T_{c\text{onset}}$ suggest a presence of new superconducting phase.

1. Introduction
Bismuth telluride Bi$_2$Te$_3$ is a semiconductor with a band gap of 0.171 eV [1], containing a high density of state around the Fermi energy level. Therefore it shows a relatively low thermal conductivity. The fact of its being a thermoelectric material [2] is based on such transport properties.

On the other hand, it is expected that these characters cause its metallization and superconductivity under high pressure. Actually high-pressure transport-properties of Bi$_2$Te$_3$ had been investigated to find the superconductivity during the 1960’s and the 1970’s [1, 3-6]. The pressure dependence of superconducting transition temperature $T_c$ was reported by Il’ina et al. [5]. The highest $T_c$ was 4.3 K at 71 kbar, however, the zero resistances to characterize the superconducting transition were not observed in their experiments. We recently measured the temperature dependence of electrical resistance of Bi$_2$Te$_3$ under high pressure using a modified Bridgman anvil cell, which makes hydrostatic compression possible using the pressure medium [7, 8]. The value of $T_c$ showing the zero resistance is obtained to be 2.8 K at the pressure of 10 GPa.

We are interested in the crystal structure of Bi$_2$Te$_3$ under high pressure and its phase diagram because the crystal structure dominates the transport properties. Bi$_2$Te$_3$ has a hexagonal structure with a space group $R\overline{3}m$ at room temperature and atmospheric pressure; the lattice parameters are $a_0 = 4.395$ Å and $c_0 = 30.44$ Å [9]. In addition, the presences of two high-pressure phases are confirmed by the x-ray diffraction at room temperature [10]. The structural phase transition from phase I to II, is observed at 8 GPa, which is accompanied by a significant decrease in the electrical resistance.
Moreover, the structural phase transitions from phase II to III is observed at 14 GPa and room temperature.

In this study, the electrical resistance of Bi$_2$Te$_3$ has been measured at low temperature under pressure up to 13 GPa. We discuss the pressure dependence of $T_c$.

2. Experiments

Polycrystalline Bi$_2$Te$_3$ (99.99 % pure, Kojundo Chemical Lab. Co., Ltd.) was used as the sample. According to the powder x-ray diffraction measurement, the lattice parameters were $a_0 = 4.385$ Å and $c_0 = 30.51$ Å, respectively [10]. The sample cut into a small piece with a dimension of $1.0 \times 0.30 \times 0.14$ mm$^3$, was set in a Teflon capsule. The electrical resistance of sample set in the capsule was measured by four-probe method. The capsule was filled with a mixture of Fluorinert FC-77 and FC-70 (1:1 in volume), which is a pressure-transmitting medium to generate a quasi-hydrostatic condition. We used the modified Bridgman anvil cell for the electrical-resistance measurements under high pressure [7, 8]. The capsule was loaded into the Bridgman anvil cell. The pressure in the sample chamber was calibrated using the transition pressure of Bi at room temperature and the superconducting-transition temperature of Pb in the lower-temperature region. The cell loaded and clamped at room temperature, was cooled by $^3$He circulation-type Joule-Thomson 1 K cryogenic refrigerator with the Gifford-McMahon cycle cooler developed by IWATANI INDUSTRIAL GASES CORP. The temperature dependence of electrical resistance was measured in the temperature region between 0.9 and 300 K at each pressure.

3. Results and discussion

We have been succeeded to pressurize up to 13 GPa using the Bridgman anvil cell. Figure 1 shows the pressure dependence of electrical resistivity $\rho$ obtained under pressure up to 13 GPa and at room temperature. The value of $\rho$ decreases with increasing the pressure. The $\rho$ vs. $P$ curve has two inflection points at around 2 and 8 GPa, respectively. The change in slope observed at around 2 GPa is probably associated with the electrical topological transition (ETT) [10, 11]. The rapid decrease of value of $\rho$, which is occurred at around 8 GPa, indicates the structural phase transition from phase I to II [10].

The temperature dependences of $\rho$ were obtained in the pressure range from 9.0 to 13 GPa as shown in Fig. 2. The $T$ vs. $\rho$ curves in the main figure and insert are displayed in the following regions: 0.9 K $< T < 10$ K for the main and 0.9 K $< T < 300$ K for the insert, respectively. The insert shows that each $\rho$ vs. $T$ curve exhibits metallic behavior. The main figure indicates that pressurization from 9 to 13 GPa causes the zero resistance state, which is one of the proofs of superconductivity. We determine the zero-resistance critical-temperature $T_c^{zero}$ and the onset temperature of superconducting transition $T_c^{onset}$ here. At 9.0 or 10 GPa, there is little difference between $T_c^{zero}$ and $T_c^{onset}$. The difference is less than 0.3 K. Pressurization beyond 11 GPa however broadens the difference, $T_c^{onset} - T_c^{zero}$. In particular, the transition process observed at 13 GPa takes place at higher $T_c^{onset}$ and $T_c^{zero}$.

Figure 3 shows the pressure dependence of $T_c^{onset}$ and $T_c^{zero}$. The pressure dependence of $T_c^{onset}$ is different from that of $T_c^{zero}$ at the pressures above 10 GPa. A continuous increase of $T_c^{onset}$ is observed in the region between 10 and 13 GPa. The value...
of $T_c^{\text{onset}} = 5.0$ K observed at 13 GPa is the highest temperature in all of the reports on $T_c$ of Bi$_2$Te$_3$ [5, 10]. The value of $T_c^{\text{zero}}$, decreases with increasing pressure in the region between 9 and 11 GPa, which turn to the increase at 12 GPa. In addition the pressurization at 13 GPa causes sudden enhancement of $T_c^{\text{zero}}$, which is 3.7 K.

The high-pressure x-ray diffraction study indicates that Bi$_2$Te$_3$ shows the phase II under pressures above 8 GPa and at room temperature [10]. Taking the previous study into account, it is reasonable to suppose that the negative pressure dependence in the value of $T_c^{\text{zero}}$ observed under pressure up to 11 GPa is caused by phase II. The sharp increase of $T_c^{\text{zero}}$ and the positive pressure dependence of $T_c^{\text{onset}}$ suggest a presence of new superconducting phase. It seems that the mixture of phase II and the new phase is present in the pressure region between 10 and 13 GPa. We suppose that the new phase corresponds to the phase III, which is present at the pressures above 14 GPa and at room temperature. In conclusion, we found the new superconducting phase of Bi$_2$Te$_3$ with $T_c^{\text{onset}} = 5.0$ K at 13 GPa. In addition it was clarified that the value of $T_c$ observed in phase II decrease with increasing the pressure.

**Figure 2.** Temperature dependence of electrical resistivity at the pressures from 9.0 to 13 GPa. The main figure and insert show the resistivity in the following temperature regions: 0.9 K $< T < 10$ K for the main and 0.9 K $< T < 300$ K for the insert, respectively.

**Figure 3.** Pressure dependences of $T_c^{\text{onset}}$ and $T_c^{\text{zero}}$ of Bi$_2$Te$_3$. Closed and open circles indicate $T_c^{\text{onset}}$ and $T_c^{\text{zero}}$, respectively.

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