

measurement, heats up the whole cell and the lowest temperature attained by this cell is 80 millikelvin. An a.c. current of 70 Hz is applied to the primary coil. The critical current is 100  $\mu$ A at the temperature of 80 millikelvin which maintains the thermal equilibrium.

#### 6. Pressure dependence of superconducting transition temperature in Cd and Ag<sub>2</sub>F

The transition temperature of most superconductors are reduced by increasing the pressure and a linear extrapolation of the low pressure data on  $dT_c/dP$  might lead one to suppose that the superconducting transition temperature would become zero under high pressure. Brandt and Ginzburg(11), Smith and Chu(2), Seiden(12) and Boughton et al.(13) estimated the disappearance of superconductivity on cadmium at 120 kbar, 38 kbar, 700 kbar and 325 kbar, respectively. The experimental check of these disagreement is very interesting.

On the other hand, the physics of one dimensional and two dimensional materials become the topic at present. A silver subfluoride, Ag<sub>2</sub>F, which is a two dimensional material, is a superconductor with a transition temperature of 0.066 K(14). The pressure effect of the superconducting transition temperature on Ag<sub>2</sub>F might be a check of new superconducting mechanism.

The preliminary experiment of the superconducting transition temperature of Cd and Ag<sub>2</sub>F are done. The superconducting transition temperature of Cd is 0.515 K at normal pressure. The superconducting transition temperature under 8 kbar and 24 kbar are measured using the D-I type cell (4 mm Bridgman-anvil). The T<sub>c</sub> is 180 mK and 140 mK, respectively. Further experiment is still doing.

The pressure effect on the superconducting transition temperature of Ag<sub>2</sub>F is measured but is not completed yet. The main reason is the low resistivity of the sample ( $10^{-3}\Omega$  at 1 K). As the cooling power of the refrigerator decreases rapidly at lower temperatures, the maximum current in the case of the d.c. resistance measurement is limited. At a temperature of 66 mK, the critical current which maintains thermal equilibrium is 25  $\mu$ A.

#### 7. Conclusion

Two small high pressure clamp cell are devised. The highest pressure attained is 50 kbars. They are attached to the helium 3/helium 4 dilution refrigerator and are cooled down to the several tens millikelvin. One is used for the measurement of the d.c. electrical resistance and another is used for the measurement of the a.c. magnetic susceptibility. The former cell is cooled down to 30 mK and its temperature homogeneity is  $\pm 5$  mK, but the latter cell is cooled down to 80 mK owing to its structural reason, and the temperature distribution is not homogeneous. As the cooling power of the dilution refrigerator decreases rapidly at lower temperatures, the usual method for detecting the superconducting transition temperature is not applicable because of the small resistance change in the case of the d.c. method and the a.c. eddy current heating in the

case of the a.c. method. Therefore, it is necessary to develop further methods of detection.

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transmitting medium. The talc disc is made from pressed talc powder. The talc is much more plastic than the pyrophyllite and thus produces sufficient uniformity in the generated pressure.

The materials used in this apparatus are a copper-beryllium alloy and an austenitic stainless steel, as these materials have enough strength and ductility even at very low temperatures. The D-I cell shown in Fig. 1 is used for a d.c. electrical resistance measurement under high pressure. The dimensions of the flange are 30 mm in diameter and 10 mm in thickness. As for the guide of the anvil, a copper cylinder is used. The total weight of this cell is 420 gr.

The D-II cell shown in Fig. 2 is used for the a.c. magnetic susceptibility measurement. For the a.c. magnetic susceptibility measurement, the primary and the secondary coils are both divided in three parts. The coils are wound with an insulated copper wire (0.08 mm-diam). The inner coil is the primary which is totally 900 turns in the same direction. The outer coil is the secondary which is totally 2000 turns. The turn of the second (middle) part of the coil is reverse in its direction from the first (upper) and the third (lower) part and the induced voltages from each parts of the coil are compensated each others. Detection of the superconducting transition by the magnetic method does not need to use the lead wires which directly contact to the sample. The total weight of the clamp cell including the coils is 260 gr.

### 3. Cooling process of clamp cell

A helium 3/helium 4 dilution refrigerator has been made to cool the high pressure clamp cells. Fig. 4 shows the main part of the helium 3/helium 4 dilution refrigerator. The refrigeration process of the helium 3/helium 4 dilution refrigerator is as follows. The concentrated helium 3 stream is cooled gradually by liquid He (4.2 K) and is further cooled by liquid He which vaporizes under reduced pressure (1.2 K). Then the helium 3 is liquefied and further cooled by the still and the heat exchangers. This cooled concentrated helium 3 mixes with liquid helium 4 in the mixing chamber and absorbs the heat of mixing (heat of dilution). This cooled helium 3/helium 4 mixture (dilute phase) returns to the heat exchangers and cools the incoming concentrated helium 3. As the vapor pressure of helium 3 is different from that of helium 4, helium 3 is selectively vaporized in the still. This vaporized helium 3 is pumped and recirculated. Our dilution refrigerator is able to attain about ten millikelvin. Its refrigeration power at 100 millikelvin is about 50  $\mu$ W and its circulation rate is  $5 \times 10^{-5}$  mol helium 3/sec. The clamped high pressure cell is attached to the underside of the mixing chamber by a screw and fine copper wires are used between the guide of the anvil and the mixing chamber for good thermal conduction.

### 4. Pressure calibration

The generated pressure at room temper-

atures is calibrated using several fixed points which are the phase transition of Bi I-II, III-V, Tl I-II and Sn I-II, by means of the d.c. electrical resistance method. Fig. 5 shows the pressure-load calibration curve for the small Bridgman anvil (4.0 mm diameter of face). The pressure is based on the N.B.S. Symposium Scale of 1968(8). When the high pressure clamp cell is cooled down to low temperatures, the pressure in the cell is reduced by the effect of the differential thermal contraction of the materials used. Therefore, the clamped pressure has to be calibrated at low temperatures.

A pressure manometer at low temperatures is readily available in the form of a number of superconductors whose transition temperatures ( $T_c$ ) are sufficiently sensitive to the change of pressure(9). The pressure calibration of the D-I cell and the D-II cell are done using the tin manometer(10). Fig. 6 shows the pressure-load calibration curve for the anvil with a 4 mm and a 5 mm diameter face. The tin sample of 99.999 % purity is rolled to a thickness of 0.03 mm and is annealed at 150°C, for 2 hours. The superconducting transition temperature is taken from the midpoint of the transition. As the transitions become sharply, the homogeneity of generated pressure is considered to be fairly good. From Figs. 5 and 6, a pressure loss of about 40 % is estimated.

### 5. Temperature distribution of cell

At temperatures lower than 100 millikelvin, the Kapitza's thermal boundary resistance increases and it is very difficult to cool the high pressure cell by heat conduction and thermal contacts.

The temperature distribution of the D-I apparatus is measured by the Speer carbon resistors. These are 220  $\Omega$ , Grade 1002, 1/2W which are calibrated in Cerium Magnesium Nitrate (CMN) magnetic temperature  $T^*$ .

The resistance thermometers are attached to several parts of the apparatus, the underside of the mixing chamber, the upper flange and the lower flange. These temperatures are compared with the carbon resistance thermometers which are placed inside the mixing chamber. Fig. 7 shows the results.

Because of the Kapitza's thermal resistance and the heat leakage from the lead wires, the temperature of the lower flange is the highest, and the temperature difference between the lower flange and the outside surface of the mixing chamber is about 10 millikelvin. The temperature of this D-I cell reaches 30 millikelvin, however, the sample is heated up by the platinum lead wires if there is excessive current running. For example, at a temperature of 50 millikelvin, the critical current which maintains thermal equilibrium is 15  $\mu$ A.

In the case of the D-II cell, the mutual inductance coils are put outside the anvils instead of the copper guide, so that it is difficult to cool by heat conduction, and the eddy current, which is induced by the a.c. magnetic susceptibility