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Nature of Pressure Transmitting Media at Low Temperature

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temperature of tin. The pressure gradient in the sample reflects on the width directly, because higher the pressure, lower the superconducting transition temperature.[5] Increasing the load, the width increased. The pressure gradient along the side of the sample was about 2 kbar in talc medium and about 3 kbar in teflon medium at 8.5 ton load. The load versus pressure curve is shown in Fig. 5, in which the broken line is a value calculated from the ratio of load to piston area. The pressure loss, which was defined by the difference between the broken line and the solid (experimental) line, is about 30% in both pressure transmitting media, because of the friction between piston and cylinder, and also the lack of the plasticity of pressure transmitting media. In the previous work[6] without a graphite lubricant in the inner surface of cylinder, the pressure loss was 45%; thus this lubricant is presumed to be fairly useful in order to reduce a friction.

§ 3. Conclusion

The direct piston displacement apparatus was built and as the pressure transmitting media, talc and teflon were used. It was found that both media is very similar to their nature as pressure transmitting media. The pressure gradient was about 2 kbar and 3 kbar at 8.5 ton load, respectively and the pressure loss was about 30% in both media compared with the value calculated simply from the ratio of load to piston area which is mainly caused by the frictional effect between piston and cylinder and also by the lack of plasticity of talc and teflon used as pressure transmitting media.

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Clamp Type High Pressure Apparatus Using Small Bridgman Anvil at Low Temperature

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technique of the measurement of a.c. magnetic susceptibility. The pressure in this high pressure cell is calibrated by the phase transition of Bi I-II, III-V, Tl I-II and Sn I-II at room temperature, and by the pressure dependence of superconductive transition temperature of tin at low temperature. In this paper, the design of the pressure apparatus, its pressure calibration and the methods in measuring of the resistance and susceptibility are discussed.

§ 2. Clamp Type High Pressure Apparatus

Clamp type high pressure apparatus which accepts a small Bridgman anvil is most convenient to obtain the extreme conditions of lower temperature and higher pressure. Furthermore, this apparatus can avoid excessive helium consumption.

Thus we have built a clamp type cell used a small Bridgman anvil (4.0 mm face) geometry which is made from tungsten carbide. This has mainly two advantages as follows; the first is to have used the flange type for clamping mechanism and the second is to have developed an a.c. mutual inductance method for measuring the superconductive transition temperature using weakly ferromagnetic tungsten carbide anvil.

Wittig[6] had used a mechanism which clamped a Bridgman anvil tightly each other with an attached screw nut.

At first, we had used Wittig's mechanism. But in that way, we had often troubled to break of lead wires during the clamping process because the lead wires are twisted when the screw nut is tightened to clamp. Therefore, we have improved Wittig's cell in several points. The high pressure cryostat is shown in Fig. 1. As shown in Figure 2, to clamp a sample, two flanges and three bolts are used. This type is convenient to exchange the sample and is free from the twist of lead wires. Moreover, using this cell, one can adopt an a.c. method of a measurement which means a no-lead wire method so it is convenient to avoid the trouble of the lead wire discussed above. The material of the high pressure apparatus at low temperature is one of serious problem. Most steels become brittle at low temperature. In general, steels with low carbon and high nickel content (austenitic stainless steel) are sufficiently ductile at low temper-

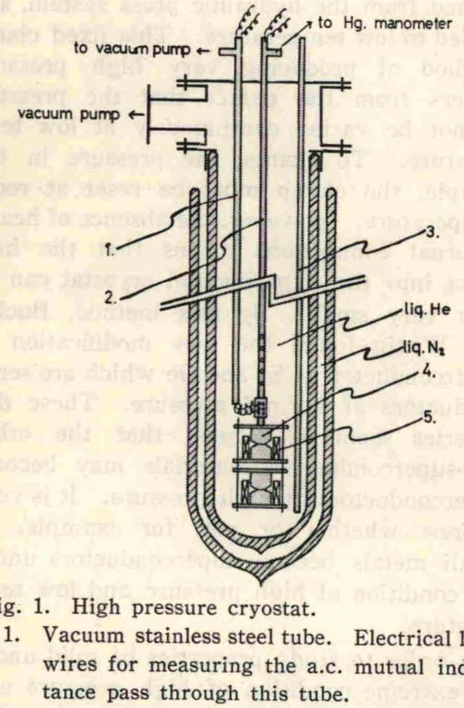

Fig. 1. High pressure cryostat.

1. Vacuum stainless steel tube. Electrical lead wires for measuring the a.c. mutual inductance pass through this tube.
2. Stainless steel tube which supports the high pressure clamp apparatus. Electrical lead wires for the d.c. measurement pass through this tube.
3. Inlet for the liquid helium.
4. Tungsten carbide anvil.
5. Measuring coil.

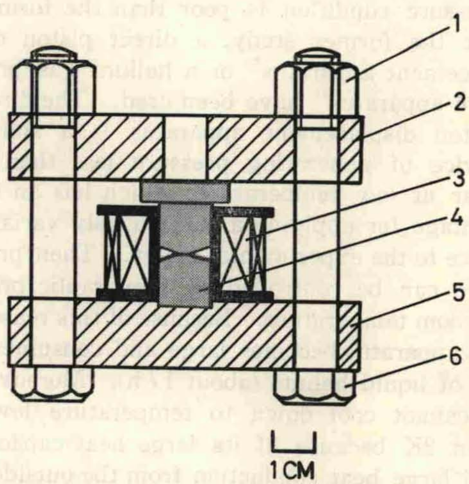

Fig. 2. High pressure clamp apparatus.

1. Fixing nut.
2. Upper flange.
3. Measuring coil.
4. Tungsten carbide anvil.
5. Lower flange.
6. Clamping nut.

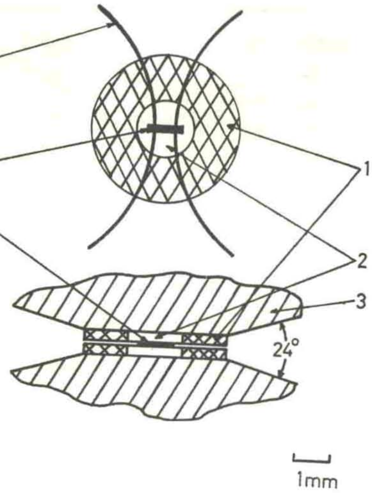

Fig. 3. Sample assembly for d.c. method.
1. Pyrophyllite ring.
2. Talc disk.
3. Tungsten carbide anvil.
4. Lead wire.
5. Sample.

ature. Then, two flanges and a clamping bolts are made from the 18-8 stainless steel (SUS 27). Two flanges are 80 mm in diameter and 15 mm in thickness. Three bolts are 10 mm in diameter and 120 mm in length.

The sample assembly is shown in Fig. 3. The pyrophyllite ring (4.0 mm i.d., 1.5 mm i.d., 0.15 mm thick) is heated at 650°C, for 30 minutes in order to increase the hardness, which is fixed with an insulating cement to a face of the anvil. A specimen is placed in the talc disc (1.5 mm o.d., 0.15 mm thick). The talc disc is made from pressed powder of talc. The talc is much more plastic than pyrophyllite and thus produces sufficient uniformity in the generated pressure. The consumption of liquid helium in this clamp type cell is only 0.3 *l*/h. When the high pressure apparatus is cooled to low temperature, we may expect the pressure in the sample to remain homogeneous throughout, if the sample and pressure transmitting medium shrink isotropically.

§ 3. Measurement of Electrical Resistance and Magnetic Susceptibility

The electrical resistance is measured by a conventional d.c. four leads method. In a resistance measurement, however, the most serious problem is the break of a lead wire because of the extrusion of the talc and

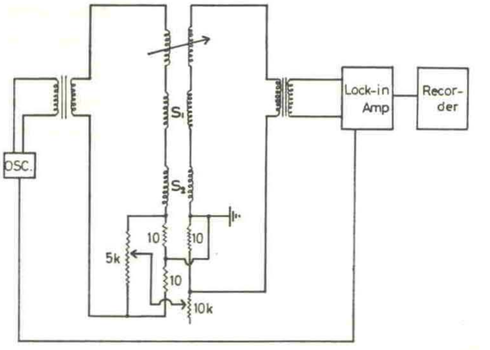

Fig. 4. Schematic diagram of a.c. mutual inductance measurement.

pyrophyllite under pressure. Therefore, a no-lead wire method is convenient for the experiment. We have developed a method of an a.c. magnetic susceptibility measurement[7] by means of an a.c. mutual inductance bridge operated 230 H_z. Usually, in the experiment of a magnetic measurement under pressure, the alumina anvil had been used instead of the tungsten carbide anvil because it is weakly ferromagnetic. But we have used the tungsten carbide anvil (11 mm in height, 12 mm in diameter, 4 mm in face) because of obtaining very high pressure. Although it may make less sensitive than using the alumina anvil, we could gain enough sensitivity to detect the superconductive transition of the sample as small as $1.0\times0.5\times0.03$ in its size.

Figure 4 shows the schematic diagram of the a.c. mutual inductance measurement. The sample is represented by S_1 and S_2 and usually, S_1 is the non-compressed sample and S_2 is under compression. The primary and secondary coils are wounded in 900 turns and 1500 turns (Cu wire, 0.14 mm in diameter), respectively.

The direction of the primary coil wounded around S_1 and S_2 is the same one but the secondary coils are wounded inversely to compensate each other. S_1 and S_2 are placed in the high pressure cell which is just the same form each other in order to cancel the magnetic effect of the ferromagnetic tungsten carbide anvil.

The a.c. signal of 230 H_z and about 5 V rms amplitude is applied to the primary of the measuring coil and rms primary current is typically about 15 mA. The inductive unbalance voltage in the secondary circuit can

§ 5. Conclusion

The high pressure apparatus of clamp type was built. This apparatus was convenient to the high pressure experiment at low temperature because the experimental procedure is very simple and the consumption of liquid helium is very little because of its small heat capacity, that is 0.3 l/h. We have experimented the d.c. electrical resistance and a.c. mutual inductance measurement which are enough to detect the pressure dependence of the superconductive transition temperature of tin using the ferromagnetic tungsten carbide anvil (4 mm face) geometry. At room temperature, the pressure was generated up to 100 kbar using the small Bridgman anvil (4 mm face) geometry.

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