

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 measurement⁷⁾ by means of an a.c. mutual inductance bridge operated 230 Hz. 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 $1.0 \times 0.5 \times 0.03$ 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.

Acknowledgement

The authors express their gratitude to Professor S. Minomura for his encouragements and advices.

References

- J. W. Stewart: Modern Very High Pressure Techniques ed. R. H. Wentorf (Butterworths, 1962) p. 181.
- 2) J. E. Schirber: Phys. Rev. 140 (1965) 2061.
- 3) G. Fujii and H. Nagano: Cryogenics 11 (1971) 142.
- 4) P. F. Chester and G. O. Jones: Phil. Mag. 44 (1953) 1281.
- W. Buckel and J. Wittig: Phys. Letters 17 (1965) 187.
- 6) J. Wittig: Z. Phys. 195 (1966) 215.
- 7) J. C. Wheatly: Rev. sci. Instrum. 35 (1964) 444.
- 8) N.B.S. Symposium (1968) "Accurate Characterization of High Pressure Environment."
- C. A. Swenson: Metallurgy at High Pressure and High Temperature ed. K. A. Gschneidner, M. T. Hepworth and N. A. D. Parlee (Gordon and Breech, 1964) p. 190.
- 10) T. F. Smith, C. W. Chu and M. B. Maple: Cryogenics 9 (1969) 53.

- W deline beenceved with T mi

in desirat se ratification de constant in desirat se chânne face promotif e lie competition pressure a con refinantement i a lie competition acres acres formation

596