

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 disk (1.5 mm o.d., 0.15 mm thick). The talc disk 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

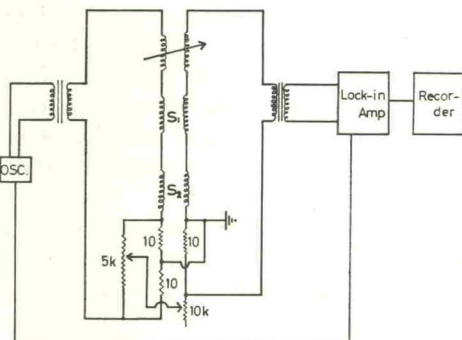


Fig. 4. Schematic diagram of a.c. mutual inductance measurement.

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<sup>7)</sup> 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 wound in 900 turns and 1500 turns (Cu wire, 0.14 mm in diameter), respectively.

The direction of the primary coil wound around  $S_1$  and  $S_2$  is the same one but the secondary coils are wound 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 Hz 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



be cancelled by the small variable inductor. A variable voltage driven from the resistive network is interjected directly into the secondary circuit and serves to cancel a resistive unbalance. The unbalance voltage is amplified by a lock-in amplifier and made available for detection.

In operation, the unbalance voltage is approximately nulled by the inductive and resistive network discussed above. The change of a.c. mutual inductance caused by the superconductive transition is then recorded by an X-Y recorder. The X axis records the temperature. The balance of the mutual inductance bridge is sensitive in a resistive change of the measuring coil. This lead wires for measuring the a.c. mutual inductance are passed through the vacuum tube.

#### § 4. Pressure Calibration

The pressure at room temperature 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. method. The pressure scale is based on N. B. S. Symposium Scale on 1968 (Table I).<sup>8)</sup>

The pressure is applied to a sample using a standard hydraulic press at room temperature. The resistive change is recorded directly by a d.c. method with slowly increasing the pressure of hydraulic press. Fig. 5 shows the load-resistance curve in Bi at room temperature. Figure 6 shows the pressure-load calibration curve at room tem-

Table I. N. B. S. Symposium Scale (1968).<sup>8)</sup>

	Bi I-II	Tl I-II	Bi III-V	Sn I-II
P. (kbar)	25.50	36.7±0.3	77±3	100±6

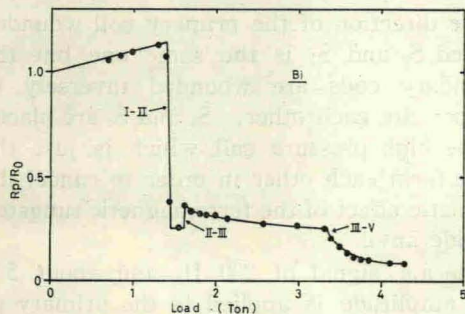


Fig. 5. Load-resistance curve in Bi at room temperature using a small Bridgman anvil (4.0 mm face) geometry.

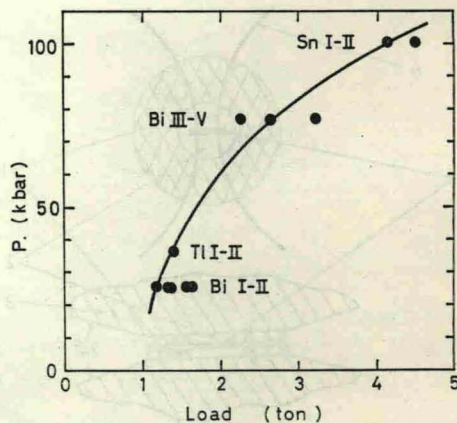


Fig. 6. Pressure-load calibration curve at room temperature using a small Bridgman anvil (4.0 mm face) geometry.

perature, using small Bridgman anvil (4.0 mm face) geometry.

The clamp is carried out in following ways. At first, the desired pressure has been applied to a sample by a hydraulic press and the pressure is monitored by means of recording the electrical resistance of the sample. This pressure is clamped by means of three bolts, and the clamped cell is detached from the hydraulic press. At this time, the pressure relaxation is often caused by the imperfect binding of clamping bolts. Therefore, the clamp is done usually at 5 percent extra pressure. But, the final check of clamped pressure is done with measuring the resistance of the sample.

A pressure manometer at low temperature is readily available in the form of a number of superconductors whose transition temperature ( $T_c$ ) are sufficiently sensitive to the change of pressure. Swenson<sup>9)</sup> proposed a particularly useful pressure scale up to 10 kbar using a tin manometer. The relationship is given in polynomial form by

$$\begin{aligned} \Delta T_c &= T_c(P) - T_c(0) \\ &= -4.7 \times 10^{-2} P + 3.6 \times 10^{-4} P^2 \end{aligned} \quad (1)$$

with pressure  $P$  in kbar.

Swenson's relationship between the tin superconductive transition temperature and pressure (eq. 1) is not valid at pressure greater than 10 kbar. However, Smith *et al.*<sup>10)</sup> avoided the limitations up to 100 kbar by adopting the relationship derived from the theory of Birch,

$$P = A(Z^7 - Z^5)[1 - B(Z^2 - 1)] \quad (2)$$