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SUPERCONDUCTING PRESSURE GAUGE AT HIGH PRESSURE AND LOW TEMPERATURE

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Le manomètre supraconducteur à basse température

RÉSUMÉ : *L'influence sur la pression de la température de transition de métaux supraconducteurs tels que le bismuth et le plomb est mesurée jusqu'à* 60 kbar.

En utilisant de l'hydrogène solide comme transmetteur de pression, on a mesuré l'influence de la pression sur une température de transition supraconductrice dans plusieurs métaux jusqu'à 10 kbar.

Les AA. ont bloqué la cellule à haute pression à la température ambiante à plusieurs niveaux de pression et l'ont refroidie à la température de l'hélium liquide. Dans le cas de la cellule à haute pression du type cylindre à piston ainsi que dans le cas de la cellule à pression du type à enclume, l'homogénéité de la pression hydrostatique n'est pas suffisante, mais les AA. les ont utilisées jusqu'à une pression supérieure à 10 kbar *et ont mesuré l'influence de la pression sur la transition supraconductrice de plusieurs métaux.*

1 — Introduction

Recent investigations at high pressure and low temperature have been made in the field of solid state physics. Although accurate pressure scales have been determined up to 100 kbar at room temperature and above, pressures at low temperature are not easily measured. The main reason is lack of a hydrostatic pressure transmitting medium at low temperature. Even liquid helium, the lowest boiling substance available, is a solid above $\sim$ 130 bar at 4 K.

Several methods for attaining nearly hydrostatic pressure at low temperature have been described by Swenson [1], Stewart [2], Brandt [3], and Itskevich [4]. In general their simple piston-cylinder type cells can be used to a maximum pressure of only 20 kbar because of limited cylinder strength. In order to attain higher pressures, Wittig [5], Buckel [6], Köhnline [7], and Brandt [8] have used anvil type cells, developed by Chester and Jones [9].

The purpose of this paper is to discuss in some detail the generation of pressure at low temperature and to examine the pressure dependence of the superconducting transition in tin as a possible pressure gauge.

2 — High pressure apparatus

We have made two types of high pressure apparatus for use at low temperature. One is a direct piston-displacement apparatus, the other is a clamped-cell apparatus.

A — Direct piston-displacement apparatus

This basic form consists of a piston-cylinder cell at cryogenic temperature actuated through poorly conducting compression and tension members by a high pressure oil system at room temperature.

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The tension component was made from a stainless steel (SUS. 27) tube 70 cm long. The compression member was a stainless steel rod operating inside the tube. For the high pressure piston-cylinder (30 × 8 × 30 mm), we used an alloy of copper and beryllium (1.82% Be) which is nonmagnetic. We were able to measure the magnetic properties of the sample up to 27 kOe with a superconducting magnet. The pressure attained with this piston-cylinder cell is lower than that with clamped anvils but the apparatus is suited for the measurement of fairly large samples.

B — Clamped-cell apparatus

In this method, pressure is applied at room temperature with a standard press, the anvils are clamped by means of three bolts, and the clamped cell is then cooled down to low temperature. The arrangement of the cell is shown in figure 1. A pyrophyllite ring ($4 \times 1.5 \times 0.15$ mm) was fixed with insulating cement to the face of the anvil. A thin strip of the sample was mounted between two discs of talc (1.5×0.15 mm) which served as a pressure transmitting medium. The talc surrounding the sample was much more plastic than the pyrophyllite ring and so generated a sufficiently uniform pressure.

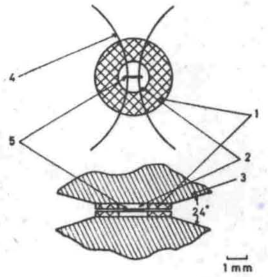

Fig. 1 — Sample assembly in the clamped cell: 1) pyrophyllite ring; 2) talc disc; 3) Bridgman anvil; 4) lead wire (Pt); 5) sample.

As shown in figure 1, the sample assembly was only 0.3-mm thick and extreme friction against the face of the anvil prevented the talc from squeezing out. The electrical resistance of the tin sample was measured by a DC method with Pt electrodes of 0.04-mm diameter. Temperature was measured by Allen-Bradley carbon resistors (91Ω, $\frac{1}{2}$ W) calibrated against a Honeywell germanium resistor.

3 — Results and discussion

A — Direct piston-displacement apparatus

Low-temperature pressure gauges can be devised from a number of superconductors whose transition temperature T_c is sufficiently sensitive to pressure change. Swenson [10] proposed a particularly useful pressure scale up to 10 kbar using a tin manometer. The relationship is given in polynomial form by

$$\Delta T_c = T_c(p) - T_c(o) = -4.7 \times 10^{-2} P + 3.6 \times 10^{-4} P^2 , \qquad (1)$$

with pressure P in kbar.

For the piston-cylinder assembly we investigated two types of sandwich structure. One consisted of a pyrophyllite disc, silver chloride disc, the sample, silver chloride disc, pyrophyllite disc, and the Cu-Be piston stacked in sequence (A-type structure). Another was made up of a pyrophyllite disc, talc disc, silver chloride disc, the sample, silver chloride disc, talc disc, pyrophyllite disc, and the Cu-Be piston (B-type structure). Silver chloride was found to extrude at an 8-ton load in the A-type structure, whereas it was fairly well contained in the B-type structure.

The pressure dependence of T_c in tin was measured in the direct piston displacement apparatus. At 10 kbar the pressure calculated from equation (1) was as much as 45% lower than the pressure calculated from the ratio of load to area, as shown in figure 2. Although the pressure can be calculated satisfactorily from load and area in the piston-

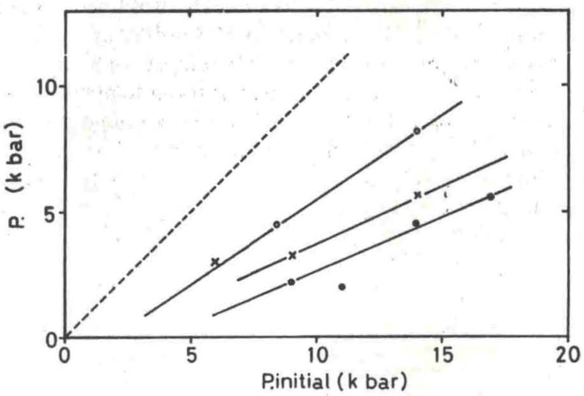

Fig. 2 — Pressure calibration of piston-cylinder apparatus. $P_{Initial}$ is the pressure calculated from the load and area. P is the actual pressure at liquid helium temperature as determined from the superconducting temperature of tin (eq. 1). ○, A-type sandwich; × and ●, B-type sandwich. The broken line shows ideal behavior.

cylinder apparatus at room temperature, the actual pressure at low temperature is considerably smaller because the transmitting medium becomes nonhydrostatic and a large friction develops between piston and cylinder.

B — Clamped-cell apparatus

For this apparatus the pressure standards were based on resistance changes associated with phase changes in various metals, for example, Bi I-II (25.50 kbar), Tl I-II (37.7 ± 0.3 kbar), Bi III-V (77 ± 3 kbar), and Sn I-II (100 ± 6 kbar) at room temperature [11]. Using the fixed points of Bi, Tl, and Sn, we obtained a pressure-versus-load calibration curve at room temperature.

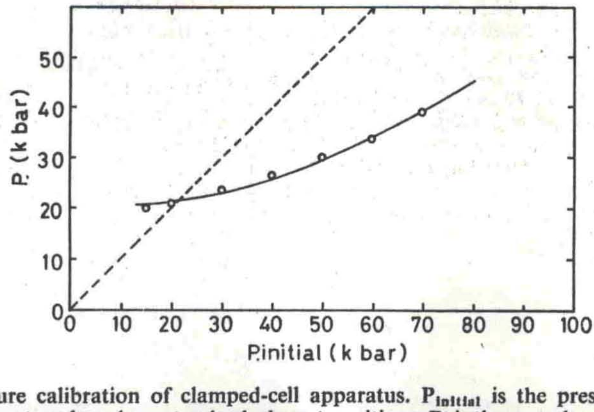

Fig. 3 — Pressure calibration of clamped-cell apparatus. $P_{Initial}$ is the pressure applied at room temperature based on standard phase transitions. Pt is the actual pressure at liquid helium temperature as determined from an extrapolation of the superconducting temperature of tin. The broken line shows ideal behavior.

Swenson's relationship between the tin superconducting transition temperature and pressure (eq. 1) is not valid at pressures greater than 10 kbar. However, Smith *et al.* [12] proposed an extrapolation formula up to 100 kbar in the form of a polynomial derived from the theory of Birch.

Again we used the superconducting temperature of tin as our pressure gauge. The tin sample of 99.9% purity was rolled to a thickness of 0.03 mm and was annealed at 150 °C for two hours. From the relationship of Smith *et al.* [12], we calculated the clamped-cell pressure at low temperature as shown in figure 3.

Although the pressure applied to the sample at room temperature was determined accurately, the corrections for relaxation during the clamping process and the effects of differential thermal contraction coupled with changes in elastic properties caused the pressure to drop appreciably with temperature. At low temperature the pressure relaxation was about 25%.

4 — Conclusion

We have discussed the superconducting transition temperature of tin as a possible pressure gauge at low temperatures. In the direct piston-displacement apparatus, the pressure loss was 45% at 10 kbar because of frictional effects in the piston and cylinder and in the pressure transmitting medium itself. In the clamped cell, the pressure loss was about 25% because of relaxation in the clamp and effects associated with the thermal and elastic properties of the apparatus.

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