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High-Pressure Apparatus for the Measurement of Electrical Resistance at Low Temperatures

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High-pressure cells for use at liquid-helium temperatures have taken a variety of forms. The ice bomb of Lasarev and Kan (1)[1] was one of the first of these, followed by the clamping device of Chester and Jones (2). Stewart and Swenson (3) developed a cell which made use of the compressibility of solid hydrogen, thus permitting pressure to be varied during an experiment, and later Brandt (4) employed a cell in which a comparatively massive specimen was surrounded by a layer of graphite lubricant in a close-fitting pressure chamber. Another technique, usable at least to liquid nitrogen temperatures, is that of Souers (5), who placed his Bridgman anvils in thermal contact with copper blocks which were cooled directly with liquid nitrogen. A new concept in apparatus is described in the paper.

[1]Numbers in parentheses designate References at the end of the paper.

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The apparatus to be described in the paper resembles the Stewart-Swenson designs except that a silver chloride pressure medium is used instead of solid hydrogen. The major objection to this approach is the likelihood of high-pressure gradients and hysteresis in the medium, but these effects, while certainly present at low temperatures, have proven to be quite manageable. The equipment was designed for measurements of high-field magnetoresistance in bismuth, and its form was dictated largely by magnet and dewar geometries.

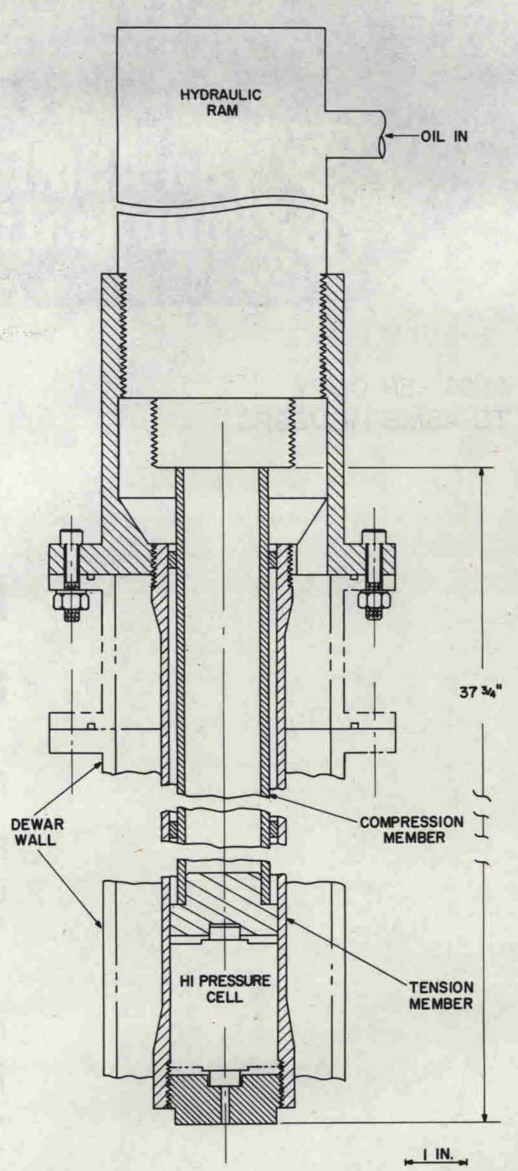

Fig. 1 Cross-section view of complete apparatus

DESCRIPTION

A cross-section of the apparatus is shown in Fig. 1.

The shaded members are all Type 304 stainless steel, chosen because of its low thermal conductivity at low temperatures and because it retains a limited ductility when cold. A hydraulic ram at the top of the apparatus acts on a compression member which consists of a steel tube of 1/8 in. wall thickness and 1 1/2 in. od. The bottom of this tube bears on the movable piston in the high-pressure cell. The cell is supported by a tension member, which is also a tube with 1/8-in. wall and 2 in. od. The overall length of the assembly, including the ram, is 49 in., and pressures of the order of 40 kilobars can be produced in the cell with ram pressures of 2000 psi. The pressure cell itself is shown in Fig. 2.

Its design contains many elements of the optical cell of Drickamer et al (6), without, of course, the windows. Successful runs have been made using a cell with carboloy pistons and an insert of Solar steel, but to avoid the problems of differential thermal contraction on cooling, a carboloy insert should be used. No attempt has been made to use nonmagnetic materials because the cell is intended to be used in solenoid magnets which deliver fields far in excess of the saturation fields of the cell components.

The lower piston is stationary and is insulated from the body of the cell by a bakelite collar and a thin layer of silver chloride. From the top of this piston, a short gold wire is passed through a mica insulator and through a disk of AgCl to one end of the sample, Fig. 3.

The sample, a bar of bismuth, is placed horizontally in a preformed cavity in a second disk of AgCl; the disk is then located in the chamber so that the wire from below touches one end of the bar. A second wire is run from the other end of the bar through a third disk of AgCl to contact the face of the moving piston, which is electrically connected to the compression member of the press. In this way, when pressure is applied after assembly, tight packing, with good electrical contact, can be achieved without undue damage to the sample.

A. CEMENTED CARBIDE PISTON (MOVABLE)
B. SOLAR STEEL INSERT
C. CEMENTED CARBIDE PISTON (STATIONARY)
D. BRASS GUIDE
E. BAKELITE INSULATING RING
F. S. A. E. 6150 STEEL JACKET
G. SOLAR STEEL BINDING RINGS

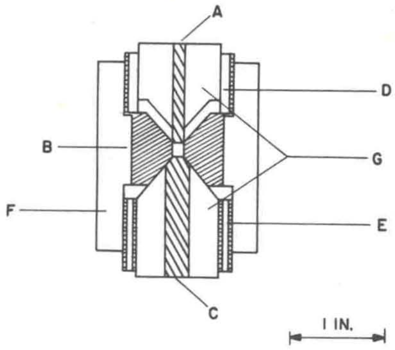

Fig. 2 Cross-section of high-pressure cell

A. AND B. SOLAR STEEL RINGS
C. MICA
D. Ag Cl
E. GOLD CONTACTS

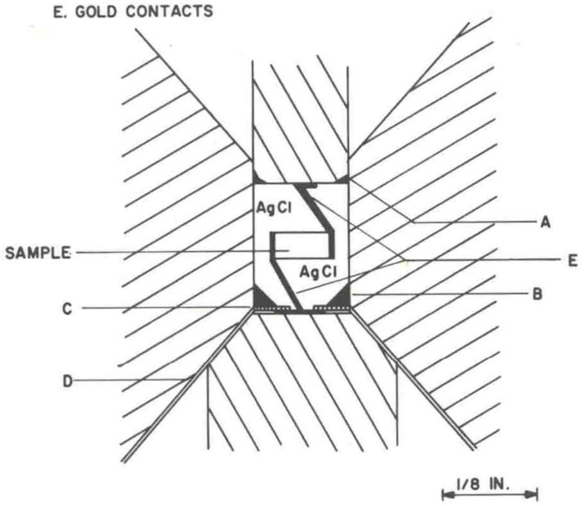

Fig. 3 Detailed drawing of sample chamber

The method of measurement is quite straightforward. An X-Y recorder is used, with pressure plotted on the X-axis and resistance on the Y-axis. The pressure is converted to an electrical signal by a strain-gage transducer mounted in the oil reservoir supplying the ram. In a typical run, the change of resistance at some fixed temperature is plotted as the pressure is first increased to a point above the bismuth I - II - III transitions, and then decreased until the transitions reappear in reverse order. The resulting curve is thus a loop, and the assumption is made that the transition pressures observed on the "up" and "down" curves may be averaged to yield the true transition pressure. At room temperature

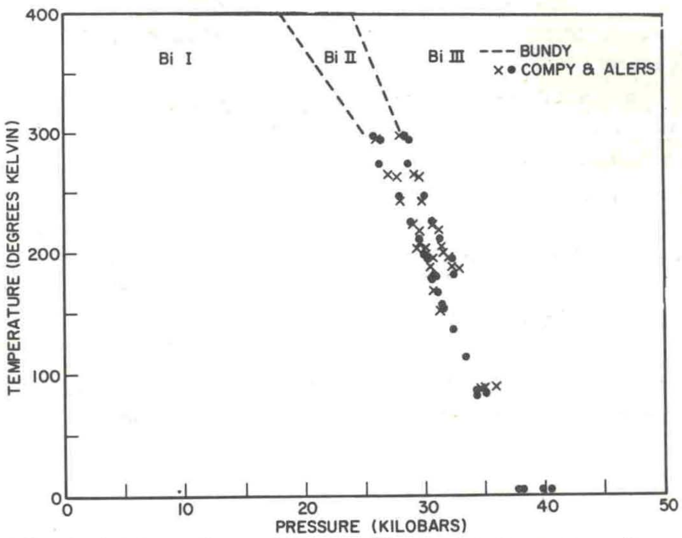

Fig. 4 Data on phase changes of bismuth taking place from zero to 40 kilobars in the 4.2 K to 300 K temperature range

such a procedure yields a Bi I - II transition which runs 2 - 5 percent high, and a Bi II - III transition about 6 - 8 percent high, when compared with the results of Kennedy and La Mori (7). A thermocouple, attached to the tension member as near as possible to the pressure cell, is used to monitor temperatures down to the boiling point of liquid nitrogen, and a carbon resistor is used to measure temperature in the liquid-helium range.

Because of the massive construction of the cell, some 15 liters of liquid helium are required to cool the apparatus from 77 to 4.2K, but the rate of helium boiloff is quite moderate: about 500 cc of liquid per hr.

PRELIMINARY RESULTS

To demonstrate the operation of this equipment, we have accumulated data on the bismuth transitions below room temperature. We are thus in a position to suggest the form of the phase diagram of bismuth from 300 to 4.2K and zero to 40 kilobars, complementing the work of Bundy (8) at higher temperatures and pressures. The data are shown in Fig. 4. The point at which Bi II disappears seems to be in the neighborhood of 180K, but our measurements are not refined enough to show the shape of the phase boundaries at this point.

REFERENCES

1 "Measurements at Low Temperatures and High Pressures," by B. G. Lasarev and L. S. Kan, Journal of Physics (USSR), vol. 8, 1944, p. 193.

2 "Superconductivity at Very High Pressures," by P. F. Chester and G. O. Jones, Philosophical Magazine, vol. 44, 1953, p. 1281.

3 Described in Modern High Pressure Techniques, R. H. Wentorf, editor, "High Pressure at Low Temperatures," by J. W. Stewart, Butterworths, Washington, D. C., 1962, p. 181.

4 Described in "Investigation of the Crystalline Modifications of Bismuth," by N. V. Brandt and N. I. Ginzburg, Soviet Physics-Solid State, vol. 3, 1961, p. 2510.

5 "Effect of High Pressure on the Electrical Resistance of Bi, Yb, and Dy," by P. C. Souers, Lawrence Radiation Laboratory Report No. UCRL 11220, University of California, 1964.

6 "Apparatus for Optical Studies to Very High Pressures," by R. A. Fitch, T. E. Slykhouse, and H. G. Drickamer, Journal of the Optical Society of America, vol. 47, 1957, p. 1015.

7 Progress in Very High Pressure Research, by F. P. Bundy, W. R. Hibbard, Jr., and H. M. Strong, editors, "Some Fixed Points on the High Pressure Scale," by G. C. Kennedy and P. N. La Mori, John Wiley and Sons, Inc., New York, 1961, p. 304.

8 "Phase Diagram of Bismuth to 130,000 kg/cm^2, 500°C," by F. P. Bundy, Physical Review, vol. 110, 1958, p. 314.