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Dual-Level High-Pressure Clamp-Anvil Apparatus at Low Temperatures

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High pressure work at low temperatures has been conducted in the field of solid state physics, especially in studies of superconductivity.[1] Of many methods, the clamped cell method is most convenient for obtaining high pressure at low temperatures, thus avoiding excessive helium consumption.

In order to study the properties of solids at very high pressures and low temperatures, we have devised a dual-level clampted-type high-pressure anvil apparatus which accepts three Bridgman-anvils. This apparatus, shown in Fig. 1, has the following unique features:

(I) Any two different levels of pressure can be administered simultaneously with only one clamping procedure. In the conventional apparatus,[2] two anvils having the same anvil face diameter are accepted, and only one level of pressure can be obtained with one clamping procedure. Therefore, many experiments are needed to obtain results for different levels of pressure. This new apparatus has reduced the number of experimental runs by one-half. As a result, this new apparatus is much more economical in liquid helium consumption.

(II) A flange-type cell has been developed

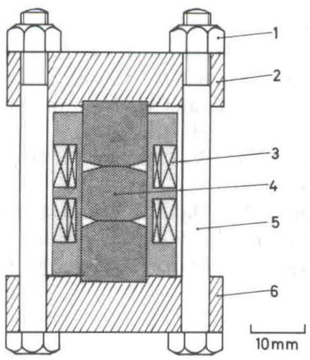

Fig. 1. Dual-level high-pressure clamp-anvil apparatus.

1. Fixing nut
2. Upper flange
3. Measuring coil
4. Anvil
5. Fixing bolt
6. Lower flange

Table I. Dimensions of anvils.

		I	II	III
Outer diameter		12^{ϕ}	12^{ϕ}	12^{ϕ}
Height		11	10	11
Anvil face diameter	No. 1	4^{ϕ}	4^{ϕ} and 6^{ϕ}	6^{ϕ}
	No. 2	3^{ϕ}	3^{ϕ} and 5^{ϕ}	5^{ϕ}

Dimension: mm

in order to clamp the specimens. This is more convenient for changing the sample and for avoiding breakage of the lead wires, because the lead wires are not twisted during the clamping process. Moreover, its structural design makes it possible to place the coils around the anvils, and thus one can adopt an a.c. mutual inductance method for measuring the superconducting transition temperature. This no-lead wire method avoids the trouble of breaking lead wires.

The clamp cell is constructed from three different sizes of anvils; I, II and III, which are shown in Table I. The three anvils are made from tungsten carbide. It is possible to assemble these anvils in many combinations of anvil face diameter, but we have so far constructed an apparatus with anvil face diameters of 6 mm$^{\phi}$ and 4 mm$^{\phi}$, and 5 mm$^{\phi}$ and 3 mm$^{\phi}$, respectively. With this assemblage, two different levels of pressure derived from the differing anvil face areas can be obtained at the same time with one clamping procedure.

The clamp procedure is carried out in the following ways. At first, a sample is placed between each of the two sets of anvils, and the desired pressure is applied to each sample with a hydraulic press. This pressure is maintained by means of three bolts tightened with a torque wrench.

The sample assembly has been described previously.[2] The entire weight of the clamp cell, including the three anvils, was 250 g, and the consumption of liquid helium was 0.3 l/h. As an example, the superconducting transition of Sn at 27 kg-cm of torque is shown in Fig. 2. This is measured by the mutual inductance method, in which two jumps, ⓐ and ⓑ, arise from the two different pressures generated between each set of anvils. The difference in the change of susceptibility depends on the sample volume. The transitions are fairly sharp. There-

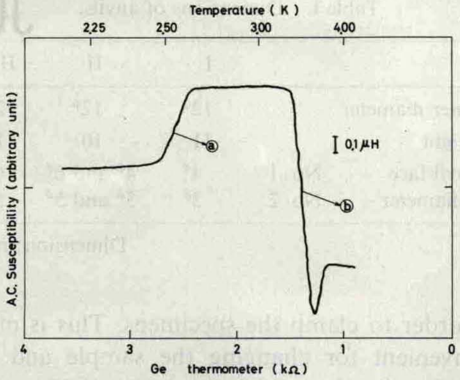

Fig. 2. Superconducting transition curve of tin at 27 kg-cm torque with 4^{ϕ} and 6^{ϕ} anvil. ⓐ and ⓑ correspond to the signal from 4^{ϕ} anvil and 6^{ϕ} anvil, respectively.

fore, the homogeneity of the talc-pressure-transmitting medium seems to be enough. Thus, the use of this apparatus results in the reduction of experimental runs by one-half. Figure 3 shows the pressure calibration curve of the new high-pressure clamp-anvil apparatus at low temperatures, which are determined from the tin manometer.[3)]

Using this apparatus, we have measured the electrical resistance of one-dimensional Pt-complex, bis (dimethylglyoximato-) platinum (II): Pt $(DMG)_2$ at pressures up to 130 kbar and temperatures down to 77 K.[4)] The electrical

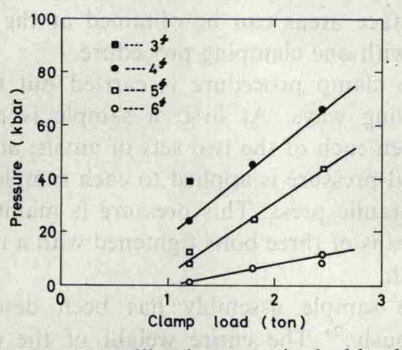

Fig. 3. Pressure calibration curve in dual-level high-pressure clamp-anvil apparatus.
○ . . . . . 6^{ϕ} Anvil □ . . . . . 5^{ϕ} Anvil
● . . . . . 4^{ϕ} Anvil ■ . . . . . 3^{ϕ} Anvil

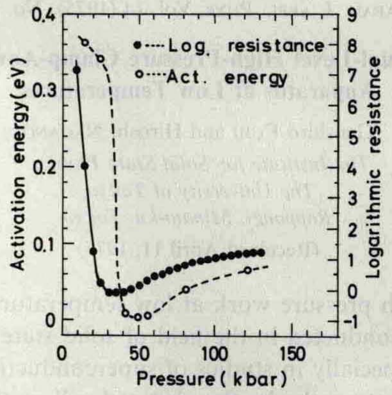

Fig. 4. Pressure effect on electrical resistance (at 293 K) and the activation energy (ΔE) of Pt$(DMG)_2$.
● . . . . . Logarithmic resistance
○ . . . . . Activation energy

resistance of Pt $(DMG)_2$ was drastically decreased by a factor of 10^{-16}, with increasing pressure up to 40 kbar, above which a resistance minimum appeared at room temperature. The estimated resistivity at the minimum was 0.5 Ω-cm. The activation energy was determined from the assumption of the following intrinsic semi-conductor behavior: $\rho=\rho_0 \exp(\Delta E/kT)$. The least activation energy of Pt $(DMG)_2$ was 0.01 eV at near 40 kbar. An anomalous resistance change against pressure may arise from an insulator-metal transition.

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