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A COMPACT CUBIC ANVIL HIGH PRESSURE APPARATUS

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The compact cubic anvil apparatus for generating to 100kb in a pyrophyllite cube was devised and the calibration curve of pressure was determined.

The external ram force applied to only one axis is converted to six equal components of force and the six anvils are forced to advance equally to the center of pyrophyllite cube by the functions of the tetragonal bipyramidal guide blocks and the trapezoid end blocks of the four anvils located horizontally and then throughout the pyrophyllite cube except its outer part is compressed homogeneously with increasing pressure.

The calibration curve of pressure was determined by the aid of phase transitions of Bi I-II, Tl II-III, Ba II-III and Bi V-VI. The hydrostatic pressure to 100kb can be generated in a pyrophyllite cube with good efficiency, that is, the load loss by gaskets of pyrophyllite is comparatively small.

Introduction

As the devices for generating static high pressures over 20 kb, the following three types are most important; piston-cylinder, opposed flat anvils and multiple anvil apparatus. In these devices, the multiple anvil apparatus is more suitable for hydrostatic compression. In this apparatus, it is necessary to form the space of the regular polyhedron by the flat faces of multiple anvils and to keep this geometrical condition for increasing pressure by equally advancing anvils toward the geometrical center of the polyhedron.

The tetrahedral anvil high pressure apparatus was first reported by H. T. Hall[1] in 1958. This apparatus consists of four independent hydraulic rams to apply force to the faces of a tetrahedron of pyrophyllite. Recently, the special anvil guide device[2] was installed on the head of ram in order to equalize the advance of each anvil.

Another type of the multiple anvil apparatus was developed by the researchers of the U. S. National Bureau of Standards[3]. This equipment employs an assembly in which external force is applied only to one of the four anvils and wedge reaction forces act on the remaining three anvils through the tetrahedron of pressure transmitting medium.

The present apparatus employs an assembly in which the external ram force is applied only to one vertical axis and is converted to act too along the other two recutangular axes of cubic pyrophyllite by means of the inner surfaces of the upper and lower pyramidal guide blocks installed on the head of press

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1) H. T. Hall, *Rev. Sci. Instr.*, **29**, 267 (1958)
2) H. T. Hall, *ibid.*, **33**, 1278 (1962)
3) E. C. Lloyd, U. O. Hutton and D. P. Johnson, *J. Res. Nat. Bur. Stand.*, **63C**, 59 (1959)

ram and the four trapezoid end blocks of anvils. Thus, the advance of all the anvils is automatically synchronized so that the cubic pyrophyllite is compressed hydrostatically. Moreover, this compact apparatus has the advantages of the rapid adjustment and the easy manipulation.

Construction and Operation

Photo. 1 shows the whole assembly of this cubic apparatus which consists of the lower pressure oil pump up to 125 kg/cm², the intensifier up to 2500 kg/cm² and the cubic anvil apparatus in place in the

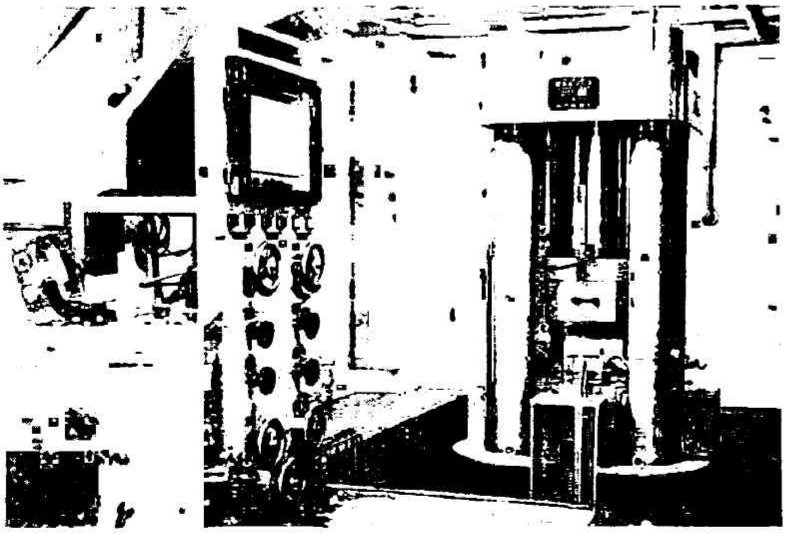

Photo. 1 High pressure assembly of compact cubic anvil

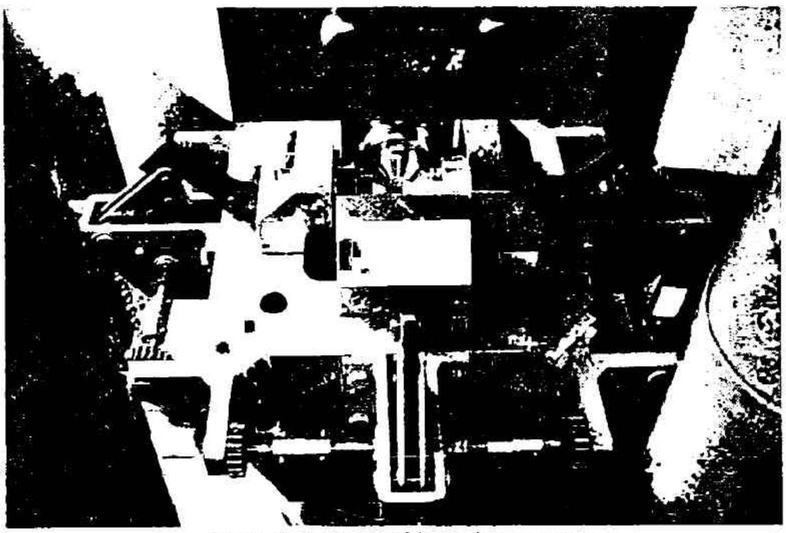

Photo. 2 Nest of cubic anvil apparatus

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1200 tons press rams. Photo. 2 shows the cubic nest of tungsten carbide anvils. The edge-length of the flat face of the anvil is 10 mm, or 15 mm. Figs. 1 and 2 illustrate the general principle and the details of the construction of this apparatus. The upper and lower guide blocks of SNCM 8 steel in JIS are attached to the heads of the press rams, the diameter of which is 250 mm. The inner surface of these guide blocks is just as the tetragonal bipyramid with $\theta=45^{\circ}$ as shown in Fig. 1. The two of the six anvils are along the center line of this bipyramid and are fixed opposite to each other on the bottom of each guide block. The other four anvils are horizontally located on the mid points of the square edges of bipyramid. This results in the formation of a cubic nest by the flat faces of six anvils. As shown in Fig. 2, the vertical

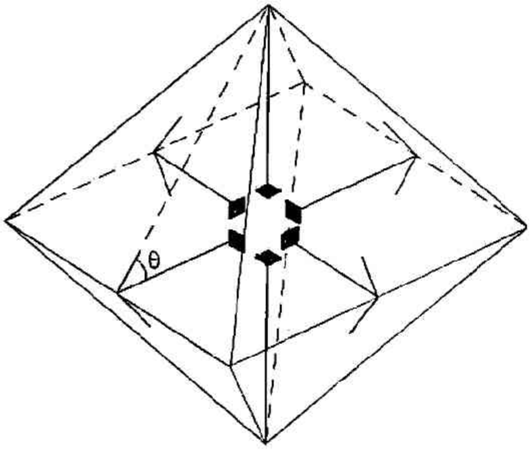

Fig. 1 Principle of compact cubic anvil apparatus

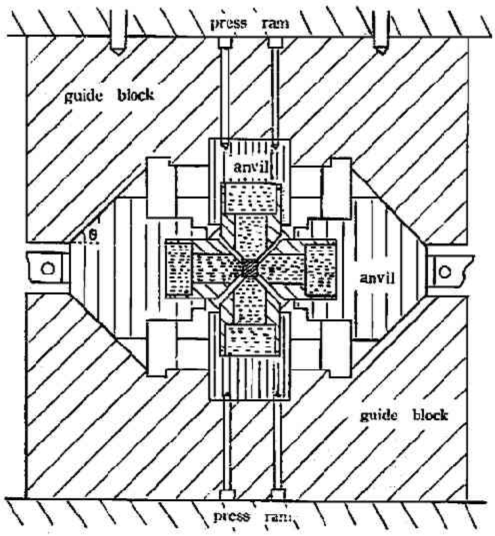

Fig. 2 Vertical cross section of compact cubic anvil apparatus

cross section of the end block of these four anvils is in the shape of trapezoid. These two oblique planes ($\theta=45^{\circ}$) of the end block of the four anvils are in contact with the inner surface of the bipyramidal guide blocks with teflon sheets inserted. In this way, the external ram force, which is applied to only one vertical axis, is converted to the six equivalent components of force which act on the cubic pyrophyllite along the three recutangular axes and the flat faces of the six anvils are then forced to advance equally toward the center of cube and always to form the cube with increasing pressure. This is illustrated in Fig. 3. The movement of the anvils compressing a copper cube (12.5 mm edge-length) was measured by the dial gauges. The advances of all the anvils are automatically equalized with increasing pressure.

The distribution of pressure in the pyrophyllite cube was tested by two methods. The pyrophyllite cube was divided into two equal parts and the fine wire mesh of copper was inserted. The assembly, after being coated with iron oxide, was compressed to a load of 90 tons corresponded to 62.5 kb (calibration pressure). Photo. 3 shows the patterns of mesh before loading and after unloarding, which illustrate the homogeneity of compression throughout the cube except in its outer part. The other was the simultaneous recording of the phase transition of three bismuth chips which were inserted in the pyrophyllite

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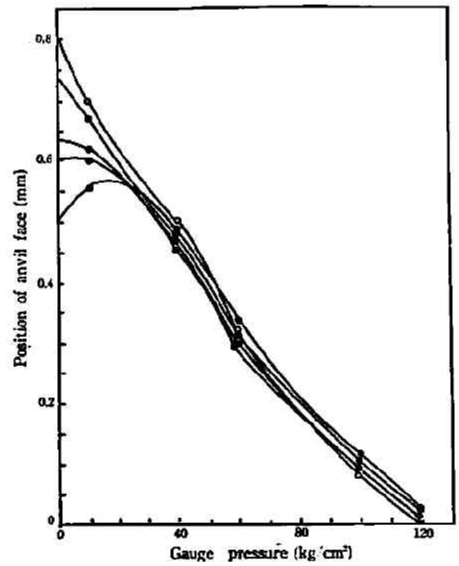

Fig. 3 Movements of anvils with increasing pressure
Origin of ordinate is at the final position of an anvil face.

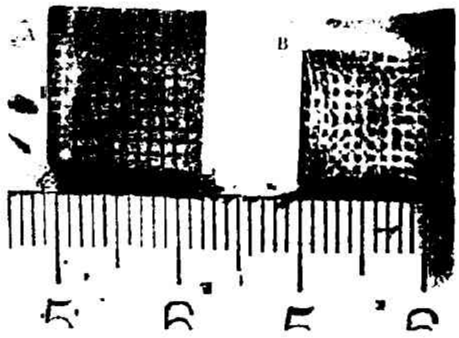

Photo. 3 Patterns of a fine wire mesh of copper before loading (A) and after unloading (B)

cube along the three recutangular axes. These results are shown in Figs. 4 and 5. The differences of transition pressure and in the time axis of the chart among the three chips are little in both cases of loading and unloading. Therefore, it is certain that the pyrophyllite cube is compressed homogeneously in this apparatus.

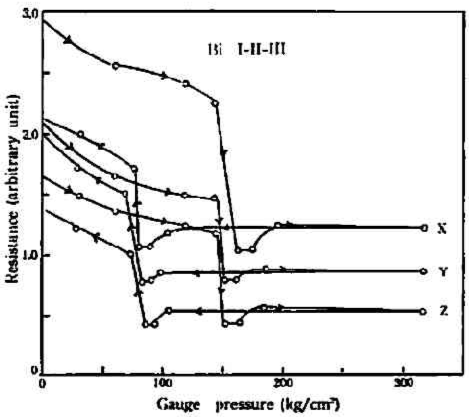

Fig. 4 Phase transition of three bismuth chips placed in pyrophyllite cube along three recutangular axes; X, Y and Z——Resistance *vs* Pressure

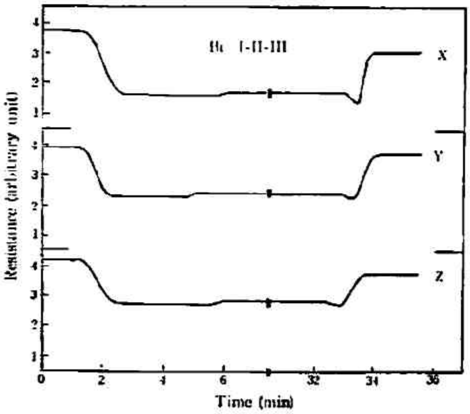

Fig. 5 Phase transition of three bismuth chips placed in pyrophyllite cube along three recutangular axes; X, Y and Z——Resistance *vs* Time

The guide blocks are insulated from the press rams with teflon. The anvils are mutually insulated with teflon and polycarbonate and each anvil is provided electric terminal for physical measurements.

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Pressure Calibration

The press load required to produce a given pressure in the pyrophyllite cube has been determined by the aid of phase transitions of Bi I–II (25.4 kb), Tl II–III (37 kb), Ba II–III (59 kb) and Bi V–VI (89 kb)[4] which are detected by the sharp changes in electrical resistance. Now, in this experiment, the anvil with edge-length of 10 mm was used. As the solid pressure transmitting medium, pyrophyllite (hydrous aluminum silicate) obtained from the American Lava Company was used. The initial edge-length of pyrophyllite cube was about 25 per cent larger than the edge-length of the anvil face. The sample foil with about 0.08 mm in thickness, 0.5 mm in width and 8 mm in length was placed, in parallel with

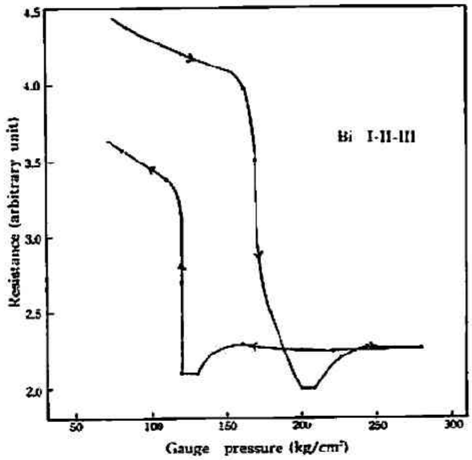

Fig. 6 Phase transition of Bi I-II and II-III

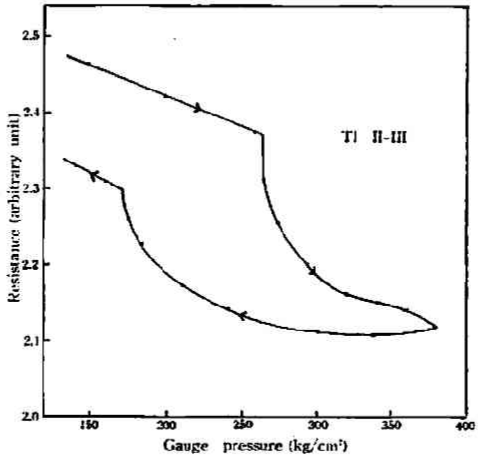

Fig. 7 Phase transition of Tl II-III

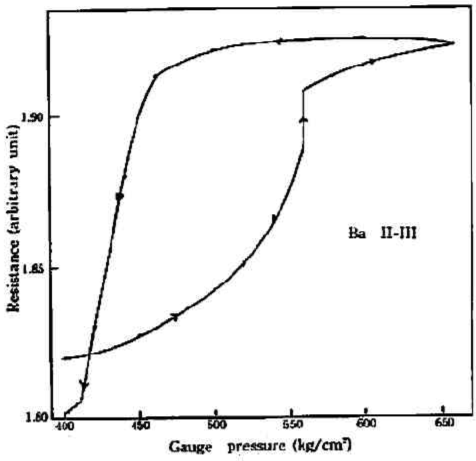

Fig. 8 Phase transition of Ba II-III

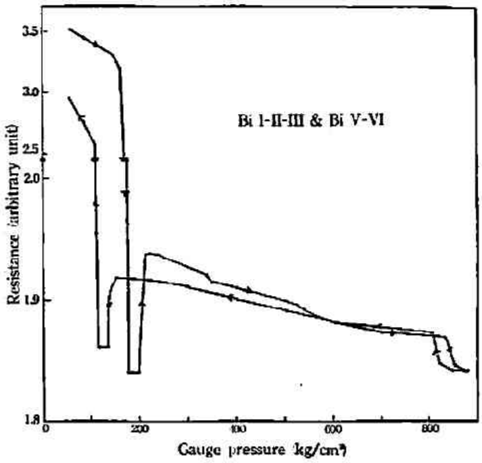

Fig. 9 Phase transition of Bi I-II, II-III and V-VI

4) F. P. Bundy, "*Modern Very High Pressure Techniques*" edited by R. H. Wentorf, Jr., p. 19 (1962), Butterworths, London

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the surface, on the semi cube of pyrophyllite. The surface of the pyrophyllite cube was coated with iron oxide powder after construction. As to the changes in electrical resistance of the sample, the potential drop, appling constant small current across the sample, was measured as a function of pressure by the potentiometer.

Fig. 6 shows the resistance change of bismuth (99.95%) with pressure. The sharp drops in resistance is due to the Bi I-II transition occurring at 25.4 kb. The rise in resistance following the sharp drop is due to Bi II-III transition occurring at 27 kb. Some hysteresis are observed on unloading.

The phase transition of thallium (99.99%) at 37 kb is shown in Fig. 7. Hysteresis is present as in the case of bismuth.

The making of barium foil sample is difficult because of the oxidation of barium metal. The preparation of the sample is as follows. The new surface of barium metal (99.9%) exposed by filing was

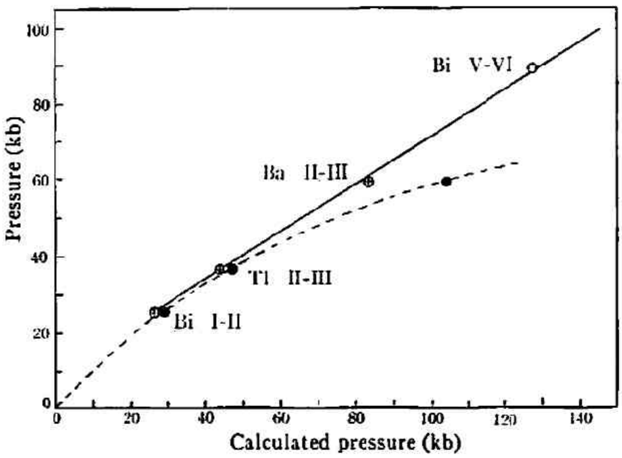

Fig. 10 Calibration curve of pressure

-O- : This experiment
-+- : Graf's value obtained by pyrophyllite dried at 90°C
-●- : Graf's value obtained by as-received pyrophyllite

covered with vacuum grease and pressed in thin foil. The greased sample of a thin foil was then cut into proper dimension. The transition is shown in Fig. 8.

The third transition of bismuth in electrical resistance is shown in Fig. 9.

The calibration curve based on these results is shown in Fig. 10. The real pressure in the pyrophyllite cube are plotted versus the calculated load pressure. The points can be connected by a straight line which does not pass through the origin. These results are in good agreement with the data obtained by R. B. Graf and B. C. Deaton[5] using tetrahedral pyrophyllite dried at 90°C and show that the load loss by gaskets of pyrophyllite is comparatively small.

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5) R. B. Graf and B. C. Deaton, *Nature*, 197, 678 (1963)