H. Tracy Hall

soldered connection the foil has been given the shape of a cup which is placed over the end of the body. The soldered joint is made along the edge of the cup. The foil is supported by the net 3 made of 0.25 mm tungsten wire with 4.8 mm pitch. The wires of the net are secured in the ring 4 by means of the pins 5, which has already been described in detail above. In order to provide a more uniform support for the foil, thus increasing the strength of the thin wall, the net was placed in front of the foil with a clearance of 0.1-0.2 mm. Such walls resist a working gas pressure of 4 atm.

These walls were assembled and prepared for the test by A. A. Shubin.

LITERATURE CITED

[1] G. F. Bogdanov, N. A. Vlasov, S. P. Kalinin, B. V. Rybakov, and V. A. Sidorov, Proceedings of International Conference on the Neutron Interactions with the Nucleus, Columbia University, 236 (New York, 1957).

[2] L. F. Kondrashev, A. A. Kurashov, A. F. Linev, V. A. Sidorov, N. I. Sokolov, and N. N. Khaldin, Pribor. i Tekh. Éksp. 1, 17 (1958).*

[3] G. F. Bogdanov, N. A. Vlasov, S. P. Kalinin, B. V. Rybakov, L. N. Samoilov, and V. A. Sidorov, Zhur. Éksp. i Teor. Fiz. 36, 633 (1959).

[4] N. A. Vlasov and V. P. Rudakov, Zhur. Éksp. i Teor. Fiz. 36, 24 (1959).

[5] G. F. Bogdanov. N. A. Vlasov, S. P. Kalinin, B. V. Rybakov, and V. A. Sidorov, Zhur. Éksp. i Teor. Fiz. 30, 185 (1956)

SOURCE Instruments & Experimental Techniques

VOLUME	NUMBER	DATE	PAGES (Inclusive)
4		7-8/60	628-631

HOLL'S TETRAHEDRAL PRESS FOR OBTAINING PRESSURES UP TO 10^5 ATM AT TEMPERATURES UP TO 2000°C

L. F. Vereshchagin, V. A. Galaktionov, and V. V. Popov

Institute of High Pressure Physics of the Academy of Sciences, USSR
Translated from Pribory i Tekhnika Éksperimenta, No. 4, pp. 106-109, July-August, 1960
Original article submitted December 15, 1959

A press with four hydraulic cylinders was constructed according to Holl's design for investigating a method of obtaining pressures up to 10^5 atm at 2000°C. It was found that a tetrahedral press retains pressure in the substance up to 70 katm at 2000°C during several hours. The defects of such a method of obtaining pressure were established: the difficulty in synchronizing the movement of the four cylinders and the impossibility of reinforcing the components subjected to the greatest load, thus making it impossible to raise the pressure any higher.

At present high pressures of $\sim 10^5$ atm are of considerable interest not only to a narrow circle of specialists who are investigating the properties of matter in a condensed condition. They have become of considerable practical interest in connection with new materials which have outstanding physical properties such as, for instance, artificial diamonds and borazon (boron nitride with a crystalline structure of a diamond), which are made at high pressures and temperatures.

Such pressures in conjunction with high temperatures make it possible to carry out in laboratories various geophysical and geochemical experiments in that range. In the near future metallurgical research at very high

* See English translation.

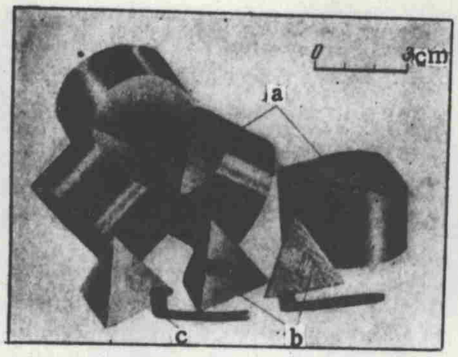

Fig. 1. The most essential details of the equipment. a) Anvils; b) the basic tetrahedron in the assembly of the container; c) container for the substance under test; the container also serves as a heating element.

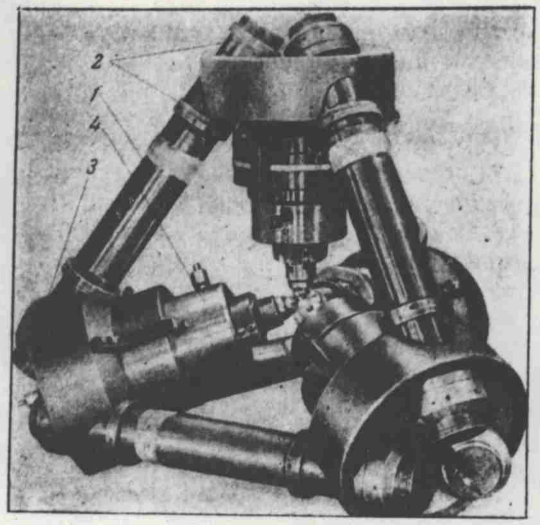

Fig. 2. Appearance of the tetrahedral press. 1) Columns; 2) adjusting nut; 3) flanges; 4) hydraulic presses.

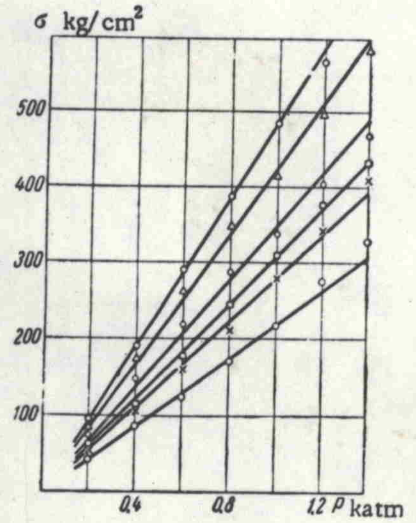

Fig. 3. Relation between tension in columns and pressure in hydraulic presses.

pressures and high temperatures will be developed, since there are reasons to expect considerable effects of pressure on the displacement of balancing curves in the diagrams showing the condition of various alloys.

Obtaining static pressures of ~ 10^5 atm meets with considerable difficulties since the details which surround the space where such pressures are obtained must experience stresses of the same magnitude. It is known that normal constructional steels have a tensile strength an order lower than the above figure. The fact that certain alloy steels have a slightly higher temporary tensile strength (~ 20 katm) does not alter the situation since these values are still only a fifth of what is required. Hard alloys consisting of tungsten carbides with a cobalt binding have a temporary compression strength up to 60-80 katm [1]. Thus the problem of obtaining pressures up to 10^5 atm with the existing materials would have been impossible if the strength of materials did not rise with pressure and the loaded portions were not supported by those with lesser loads. When these two effects are being used the problem is reduced to finding constructional forms and materials which would provide the required pressure gradients.

The construction of high-pressure equipment becomes considerably more difficult if the tests have to be carried out at temperatures above 1000°C.

It is therefore advisable to separate the zone of high temperatures from the details which have to sustain high pressures by means of various heat insulating materials, establishing in these details temperature gradients which would ensure their protection from excessive plasticity during the time of the experiment. In addition to the normal requirements of refractory materials consisting of a low heat conductivity and a high heat resistance, in this case a sufficiently high plasticity is also required over a large temperature range in order to transmit pressure from the hard details to the sample. Since electrical resistance methods are normally used for heating, the thermal insulator must also possess electrical insulating properties. Materials with all the above properties include pencilstone, catlinite, etc.

Thus the necessity of protecting the most highly stressed details from the effect of high temperature leads to a design where the pressure is transmitted to the sample by means of hard plastic bodies. Under these conditions the introduction of an additional element in the form of a liquid phase for establishing hydrostatic pressure does not appear to be advisable. The absence of an element with a liquid substance considerably simplifies the sealing of the equipment.

The tetrahedral press suggested by Holl [2] includes most of the above features. The increased size of the high-pressure chamber was another innovation as compared with the design developed by Bridgman [3]. It was considered useful to find the peculiarities in obtaining pressure by means of this method and the obstacles to a further increase of pressure and temperature in equipment of this type.

For this purpose the authors of this article have designed and constructed a device with four hydraulic presses which were placed at the apexes of the tetrahedron and provided by the converging movement of their pistons, which ended in details shown in Fig. 1, the required compression of the hard plastic body in the shape of a tetrahedron with a side of ~10 mm. Figure 1 shows the anvils by means of which the hard plastic body in the shape of a tetrahedron is compressed when the anvils are placed in their correct position. The same figure shows a pencilstone tetrahedron at various stages in assembling the container. The container is intended to carry the substance under test and to serve as a low-resistance electric heater. The electrical heating circuit of the container consists of anvils which are insulated from the body of the instrument and connected to the required voltage and the container itself which has the shape of a metallic tube with lids. The lids are welded to metal strips which are taken out of the pencilstone tetrahedron to make contact with the anvil. The high current density at a voltage of a few volts is obtained by double transformation. The temperature was estimated by means of the melting point of metals placed in the high-pressure zone.

The appearance of the equipment is shown in Fig. 2. The hydraulic cylinders can be interconnected by various means; in the construction shown in Fig. 2 the cylinders are connected by means of columns which are under tension when the sample is being compressed. The large diameter of the columns is due to the aim decreasing the stresses in them in order not to change the direction of the cylinder axes under compression.

The adjustment of cylinders is most essential in this type of equipment. In order to ensure a coincidence of the cylinder axes in the center of the tetrahedron at the beginning of the test an adjustment of the lengths of columns 1 by means of nuts 2 is provided, the nuts being placed on both sides of flanges 3 to which the cylinders 4 of the hydraulic press are fixed. In order to be able to observe any deviation of the cylinder axes from the correct position, the anvils, which are attached to the hydraulic press cylinders, are replaced by pointed rods. The pointed ends of the rods must converge at one point and the angles between the rods must all be equal. Despite the careful initial adjustment of the cylinders and anvils, it was often found during testing at high pressures (over 50 katm) that short circuits arise in the electrical heating system, thus indicating that the anvils deviate at high pressures from their original positions.

In order to find the reasons for this undesirable phenomenon pressure gauges were used to measure tensions along the columns during testing. Pressure gauges were glued to the columns and connected to a bridge circuit making up two of its arms (the remaining two arms form the input circuit of an electronic amplifier). In order to compensate for temperature errors one of the transducers was glued directly to the column and the other was placed near it on an unstrained surface. The strains in the columns during tests were compared with those of special beams placed on two rests and symmetrically loaded by means of two equal concentrated forces (such a loading provides a portion of the beam with calculable bending moment). On the basis of the equality between the strains in the column and the beam thus obtained the absolute value of tensions is calculated. The results of tests carried out in the six columns which comprise the equipment show the variation of tension versus the pressure in the cylinders and are given in Fig. 3.

The study of the relation between the tensile force and the hydraulic pressure points to the difference in the loading of columns and the lack of symmetry in the forces applied. The dispersion of the experimental points which should represent linear relations between tensile forces and pressures is due to a difference in the friction of various hydraulic cylinders and errors in estimating the pressure in the cylinders. It should be noted that the tensions in various columns differ by as much as a factor of two when the geometrical dimensions of the columns are practically equal, the general geometrical configuration of the equipment is symmetrical, and the pressure in the hydraulic cylinders cannot differ by more than 3-4% (the accuracy of the strain gauges is evaluated at 2-3%).

Thus the large difference in the tensions of columns cannot be due to anything else but the asymmetrical compression of the pencilstone tetrahedron. Calculations indicate that such tensions may arise if the compression efforts exerted on the tetrahedron deviate from the center by less than 0.2 mm. It is more probable that this phenomenon is due to the nonuniformity of efforts exerted on the side faces of the anvils when the thickness of the pencilstone layer is not uniform. In order to localize the moments due to the asymmetrical loading of the press it is possible to try and prevent the anvils from slipping away from their position, which, it would appear, was attempted in Holl's design.

The attempt to prevent the converging of anvils by placing mica sheets into the gaps between them leads to the pressure on the tetrahedral housing rising at a considerably lower rate than that of the pressure in the cylinders instead of being proportional to it. This is due to the good plastic properties of pencilstone which is forced into the gaps between the anvils. When mica, which has bad plastic properties, is placed into the gaps, a rising pressure in the cylinders does not lead to a similar rise in the pressure on the tetrahedron, since the greater part of the effort exerted in the cylinders is taken up by the side faces of the anvils.

In a press of a tetrahedral construction it is difficult to provide a mechanical support for the anvils. Due to the latter circumstance the use of anvils made of a hard alloy VK-6 limits the maximum pressure to 70-80 katm. Attempts to raise this pressure break the anvil. Under these conditions better results were obtained with anvils made of 45KhNMFA steel of a hardness of $R_c = 50$.

This is due to the fact that high pressures in the anvils are established in a relatively small area and the remaining volume being made of ductile steel supports efficiently the highly loaded parts of the anvil. In the case when the anvils were made of tungsten carbide, despite the great resistance to compression of this metal, the loaded parts remained unsupported by the brittle mass of the hard alloy. A similar result is obtained with anvils made of ball-bearing steel despite the fact that it possesses a high compressive strength.

In order to prevent short circuits and decrease the efforts developed by the press, the anvils had additional bevels added to them (Fig. 1).

The press was calibrated for pressure at room temperature by observing the variations in the electrical resistance of bismuth and thallium when they changed in phase and by the electrical resistance of zirconium. It was found that the pressure inside the pencilstone tetrahedron increased linearly with the rise in pressure in the hydraulic cylinders until the pencilstone film on the side faces of the anvils decreased to hundredths or tenths of a mm. After that any further increase in the effort exerted by the hydraulic presses does not lead to any substantial rise in the pressure on the sample, since the effort is taken up by the anvils without being passed to the pencilstone at their center. The pressure which it was possible to register in equipment of this type amounted to 70-80 katm.

Thus it was established that the principle employed by Holl for the design of the tetrahedral type equipment is correct. However, the practical application of this design encounters several unexpected difficulties which we have analyzed in this work.

It should be noted, however, that the problem of balancing the tetrahedral housing by very large forces directed normally to its faces, although feasible in practice, is very difficult to achieve experimentally and the destruction of even a single detail of such a press immediately leads to a collapse of the whole equipment since its balance of forces is destroyed.

LITERATURE CITED

[1] Yu. N. Ryabinin, Fiz. Met. i Metall. 6, 893 (1958).

[2] H. T. Holl, Rev. Scient. Instrum. 29, 267 (1958).

[3] P. W. Bridgman, Proc. Roy. Soc. A203, 1 (1958).