THE
REVIEW OF SCIENTIFIC
INSTRUMENTS

VOLUME 29, NUMBER 4 *APRIL, 1958*

Some High-Pressure, High-Temperature Apparatus Design Considerations: Equipment for Use at 100 000 Atmospheres and 3000°C*

H. TRACY HALL
Brigham Young University, Provo, Utah

(Received December 10, 1957; and in final form, December 30, 1957)

Three items of equipment are described in detail; (1) A simple-piston and cylinder device capable of generating pressures to 50 000 atmospheres simultaneously with temperatures of 5000°C, (2) a tetrahedral-anvil device that has been successfully tested at 100 000 atmospheres and 3000°C, and (3) a stepped-piston device capable of developing pressures to 200 000 atmospheres at room temperature.

In addition consideration is given to such problems as (a) geometrical elements of design, (b) higher strength materials, (c) use of mechanical anisotropy in design, (d) electrical leads, (e) thermal and electrical insulation, (f) solid pressure transmitting media, (g) binding rings, (h) internal generation of pressure, (i) multistaging, and (j) calibration.

CONVENTIONAL HIGH-PRESSURE APPARATUS DESIGN

THE ordinary form taken by a high-pressure apparatus is that of a cylinder provided with a closure on one end and a movable piston on the other. The specimen to be subjected to pressure is placed within the cylinder and is compressed by the moving piston, the latter usually being driven by the ram of a hydraulic press (see Fig. 1). In practice the fit between piston and cylinder must be very good and the mating surfaces must have a high polish. When the specimen is a liquid, sealing devices must be used to prevent leakage around the piston and closure. The Bridgman seal[1] is often used for this purpose. When the specimen is a solid, gaskets are generally not needed.

The maximum pressure that can be generated in such a device depends upon the strength of the materials of construction. At the present time cemented tungsten carbides have the highest compressive strength of any readily available materials (to 800 000 psi). Use of these materials for pistons and cylinders allows pressures in the neighborhood of 50 000 atmos to be obtained. The strength of carbides in tension is much less than in compression. Because of this it is necessary to provide lateral support to a tungsten carbide cylinder. This can be accomplished for a smaller apparatus ($\frac{1}{4}$-in. piston diameter) by use of a shrunk-on, hardened, alloy steel ring, or for larger equipment by multiple, tapered, interference-fit steel rings (to be described later). A series of hydraulic rams arranged circularly around the cylinder could also be used to provide lateral support. The work-heads of such rams would push against the external surface of the carbide cylinder.

Failure of a device such as that just described occurs in the movable piston—or pistons if the device is made "double-ended." (In a double-ended device a second moving-piston is substituted for the closure. When solids are compressed, double ending reduces internal and wall friction.) Sufficient lateral support can easily be given to the cylinder in order to prevent failure at 50 000 atmos. Pressures near 70 000 atmos (capable of being generated in the stepped-piston apparatus considered later) will, however, cause a cemented tungsten carbide cylinder (6% cobalt binder) to split in a plane perpendicular to the center line of the apparatus. This can be prevented by providing a clamping force to support the cylinder from top to bottom. When this clamping force has been provided, the cylinder is supported on all free surfaces and consequently is strongly restrained from breaking. That part of a movable piston that is within a close fitting cylinder will transmit, when under compression, part of its

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* Supported by grants from the National Science Foundation and the Carnegie Institution of Washington.

[1] P. W. Bridgman, *The Physics of High Pressure* (G. Bell & Sons, Ltd., London, 1949), p. 39.

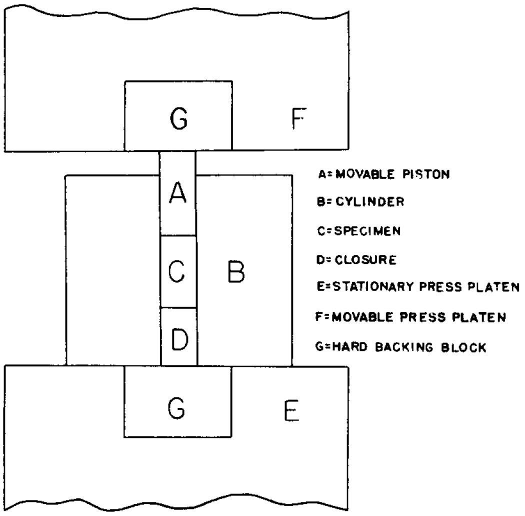

FIG. 1. Cross section of simple piston and cylinder high-pressure apparatus.

load to the cylinder as a consequence of its radial expansion. Under these circumstances this portion of the piston could support more axial load than would be indicated from its normal compressive strength. However, a certain length of the piston must protrude from the cylinder in order that motion may be obtained. This protruding portion of the piston has a cylindrical, unsupported surface, and it is here that the greatest stress is located. In practice, the highest pressures are obtained by making the protruding portion of the piston as short as is commensurate with the piston travel required.

Alignment of the various working components of the simple piston and cylinder device just described (including moving components of the press) must be very good. If this is not the case the carbide parts, particularly the pistons, may be loaded unevenly and caused to break at loads much smaller than those obtainable with good alignment. Small discrepancies in alignment can be remedied by placing a thin steel sheet of about 0.001 inch thickness between the piston and its backing block.

Sliding surfaces subject to high loadings are best lubricated by a thin film of microsize molybdenum disulfide powder. This powder should be applied to the mating surfaces of the piston and cylinder and also to the external surface of any solid specimen being compressed.

The simple-piston and cylinder apparatus is quite adequate for making pressure-volume-temperature measurements at pressures to about 50 000 atmos and temperatures to about 300°. Temperatures above room temperature are obtained by immersing the entire apparatus in a suitable thermostatic bath. This method of heating is generally limited by the lowering of the strength of the materials of construction by temperatures above 300°. However, a modified form of P. W. Bridgman's flat-anvil apparatus has been described by D. T. Griggs and G. C. Kennedy[2] whereby small specimens may be subjected to pressures as high as 80 000 atmos at 500°. This apparatus uses external heating.

INTERNAL HEATING

Sir Charles Parsons in his work on diamond synthesis devised apparatus that utilized internal electrical resistance heating.[3] In his apparatus a refractory substance surrounded the specimen and both specimen and refractory were subjected to the force exerted by a piston.

A double-ended, simple piston and cylinder apparatus utilizing internal electrical heating has been used by Loring Coes, Jr.[4] In this apparatus the problem of providing electrical connections to the interior was solved by constructing the cylinder of hot-pressed alumina. Cemented tungsten carbide pistons operating in each end of this electrically insulating cylinder served to bring electrical power to the inside of the chamber. The materials to be heated and compressed were contained in an electrically conducting tube that served as resistance heating element and container.

The simple piston and cylinder apparatus of Fig. 1 may be easily adapted for use with internal resistance heating, even though piston and cylinder are both electrically conducting. This is accomplished by making the closure D undersize by 0.002 to 0.005 in. and wrapping it with a strip of paper of corresponding thickness. A thin strip of paper is also placed on the bottom of the cylinder. This paper provides insulation to make closure D electrically inde-

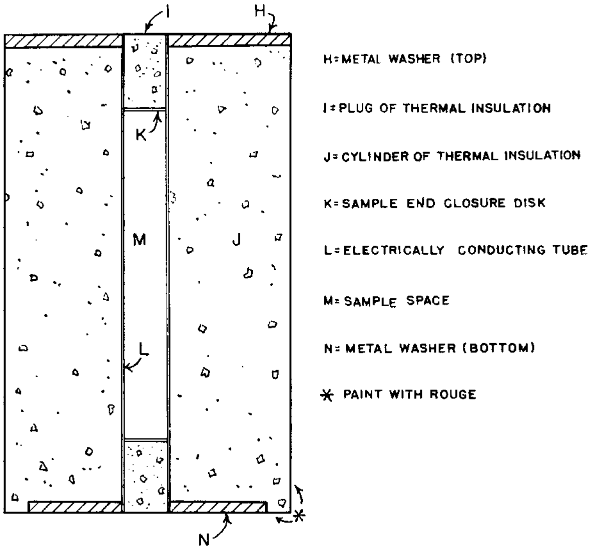

FIG. 2. Assembly for internal resistance heating.

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[2] D. T. Griggs and G. C. Kennedy, Am. J. Sci. **254**, 722 (1956).

[3] Sir Charles Parsons, Proc. Roy. Soc. (London) **44**, 320 (1888); Sir Charles Parsons, Trans. Roy. Soc. (London) **220A**, 67 (1919). See also Richard Threlfall, Engineering **87**, 425 (1909).

[4] Loring Coes, Jr., Norton Company, Worcester, Massachusetts (unpublished).

pendent from piston A. An assembly such as that shown in Fig. 2 is then made to occupy the specimen space C of Fig. 1.

In operation the piston compresses the entire assembly of Fig. 2. Heating current passes from the piston through metal washer H into electrically conducting tube L, thence through metal washer N to the cylinder closure and then to the metal backing block. The electrically conducting tube may be metal or graphite. The cylinder of thermal insulation should be a fine-grained, compact, electrically insulating substance with good pressure transmitting properties. Quite a number of compacted powders and fibers meet these requirements. An excellent, readily machinable material to use here is pyrophyllite, a naturally occurring hydrous aluminum silicate often called Tennessee Grade A Lava.[5]

If, during operation, the paper surrounding closure *D* of Fig. 1 tends to extrude, it should be painted with a suspension of rouge in a volatile organic solvent. This will greatly increase the wall friction. A little rouge painted in the region indicated by the asterisk in Fig. 2 will also be helpful. The remainder of the cylindrical surface of *J* should be coated with a thin layer of molybdenum disulfide powdered lubricant.

PRESSURE CALIBRATION

When an assembly such as that of Fig. 2 is compressed, there is a certain "frictional holdup"; i.e., a certain percentage of the pressure at the piston tip is not transmitted to the sample space M. This frictional holdup is primarily due to the internal friction of the cylinder of insulation J. With pyrophyllite cylinders the frictional holdup is usually 10 to 20%. This can be measured by substituting a pressure sensing element for the sample normally occupying space M. This element can take the form of a silver chloride rod which contains a metal wire along its axis (see Fig. 3). In Fig. 3, the assembly of Fig. 2 has been maintained intact except for a slight split in the sample tube which is necessary in order that the resistance of the wire Q may be measured. Among electrically insulating solids, silver chloride is the best pressure transmitting material known. For all practical purposes it transmits hydrostatically the pressure existing at the walls of the sample tube to the pressure sensing wire. Measurement of the electrical resistance of the pressure sensing wire as a function of load applied to the piston can establish, by means of known transitions in certain metals such as Bi or Tl,[6] a room temperature pressure calibration for the apparatus.[7]

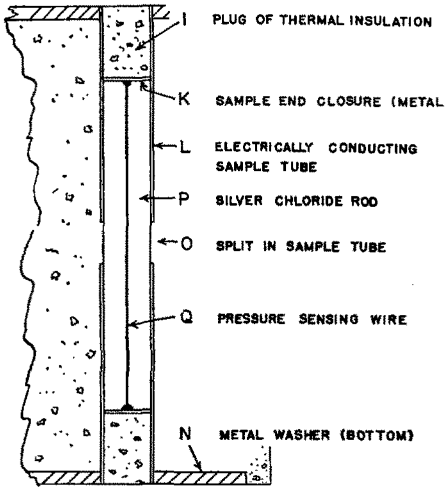

FIG. 3. Pressure sensing element located inside sample tube.

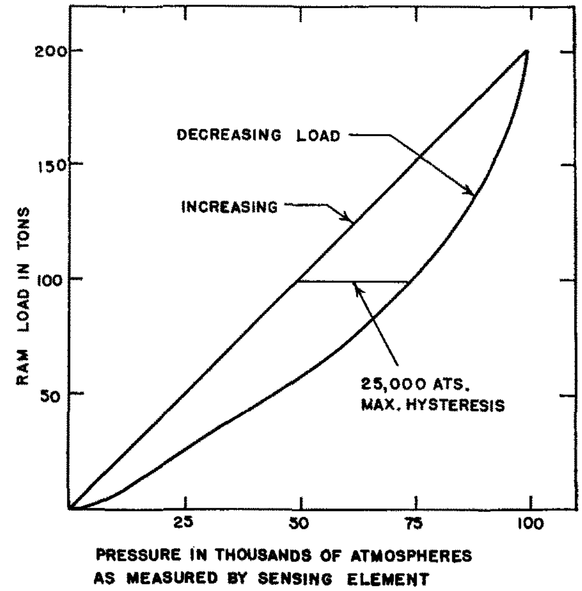

FIG. 4. Hysteresis phenomenon in pyrophylite.

This calibration is expected to be reasonably valid under high temperature operation. The pressure for a given piston load will tend to be higher at high temperature than at room temperature. This is because of incomplete transmission of the pressure generated by thermal expansion to the piston.

HYSTERETIC PHENOMENON

When solid substances are used as pressure transmitting media there usually exists a hysteresis in which, with increasing pressure external to the solid, the pressure at the core is less than that outside. However, on reduction of external pressure, the pressure at the core is higher than that on the outside. A complete hysteretic loop is shown in Fig. 4 where pyrophyllite is used in the role of J of Fig. 2. (Incidentally, it is possible to utilize the decreasing portion of the hysteresis loop to reduce the loading on pistons

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[5] Available from American Lava Company, Chattanooga 5, Tennessee.

[6] P. W. Bridgman, Proc. Am. Acad. Arts Sci. **81**, 165 (1952).

[7] H. T. Hall, J. Phys. Chem. **59**, 1144 (1955).

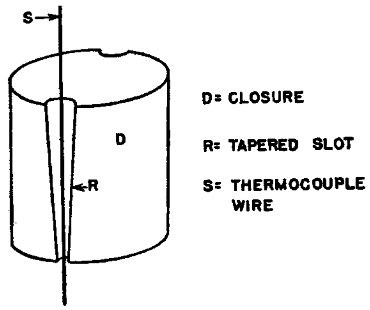

FIG. 5. Method of entrance for thermocouple wires.

or other components of a high-pressure apparatus and thereby increase their useful life. For example, Fig. 4 shows that press load could be increased to 200 tons whereupon a pressure of 100 000 atmos would be obtained at the surface of the sample. Load could then be reduced to 100 tons whereupon a pressure of 75 000 atmos would be maintained. Hysteresis would be maintaining a differential of 25 000 atmos! In practice, apparatus calibration is usually made on a basis of the ascending portion of the loop since this is linear.)

In the past, considerable suspicion has been cast on the use of solid materials to transmit pressure. This need not be. Pressure transmitted to the sample space by pyrophyllite is measurable and reproducible to within 10%. The use of solid pressure-transmitting media provides, at the present time, the only practical means for operating a system simultaneously at very high temperature and very high pressure. It seems reasonable to accept a possible 10% inaccuracy in the determination of pressure in order that the gross features of a pressure-temperature field hitherto unavailable might be explored.

The device just described, when constructed of cemented carbide components and when provided with suitable lateral cylinder support, operates quite satisfactorily at temperatures to 5000° (with carbon heater) at pressures to 50 000 atmos.

TEMPERATURE MEASUREMENTS

The temperature of the sample in the internally heated piston and cylinder apparatus is measured by a thermocouple. Thermocouple leads gain entrance to the interior of the device as shown in Fig. 5. The closure D of Fig. 1 has two tapered slots formed in it as shown in Fig. 5. The thermocouple wire, which has a thin coat of electrical insulation, is placed in the slot and rouge is packed around it with a dental spatula. Tiny slots and holes made in the pyrophyllite cylinder located above the closure accommodate the thermocouple leads. The thermocouple junction is spot-welded to the sample tube. The closure is wrapped with the usual strip of paper and the closure plus assembly of Fig. 2 are inserted in the cylinder. During an experiment, the friction of the rouge is sufficient to prevent expulsion of the wires.

For many purposes a temperature calibration curve obtained by plotting thermocouple temperature *vs* power input in watts is useful. This plot is a straight line and may have additional calibration points placed on it by observing the wattage required to melt fine wires of tungsten or other high melting metals. These wires are inserted in the same manner as the thermocouple leads. Melting is detected by the occurrence of an open circuit.

Available evidence[7] indicates that the pressure effect on the reading of a platinum-platinum-10% rhodium or on a Chromel-Alumel thermocouple is less than ±3° at 1000° and a pressure of 100 000 atmos.

Pyrophyllite melts to a glassy substance at temperatures near 1500° at pressures of a few thousand atmospheres. Its melting point is increased considerably at very high pressures. Even when it is "melted," at high pressure, molecular holes have been "squeezed-out" and the material is very viscous and does not flow. At very high pressures the upper limit of temperature usefulness for pyrophyllite has not been established. When confined the material has proven to be a useful container at temperatures to 10 000° at pressures no greater than 15 000 atmos.[8]

IMPROVEMENT OF CONSTRUCTION MATERIALS

The upper limit of pressure usefulness of the simple piston and cylinder device is determined by the strength of the carbide components. Materials with twice the compressive strength of those now available would make this device operable to 100 000 atmos. A direct approach to the problem of obtaining higher pressures in any device, then, is to be found in the field of materials. Diamond has the highest compressive strength of any known substance. Its extremely high cost and unavailability in large sections precludes its general use in high-pressure apparatus although at least one device utilizing diamond has been described.[9] Diamond powder, however, is available in sufficient quantity and at such a price that a cemented diamond composition analogous to the cemented tungsten carbides could be used if such were obtainable. Such a material would be expected to have a higher compressive strength than the cemented carbides. Apparently attempts to produce a satisfactory diamond composition have not yet proved successful. The problem arises because diamond is unstable with respect to graphite at ordinary pressures. Possible binding materials of suitable strength have sintering temperatures above 1000°. At these temperatures, finely divided diamond readily graphitizes. Even momentary heating will produce a thin graphite film at interfaces with resultant weakening of the composition. One solution to this problem would be to carry out the sintering operation at a pressure where diamond would be stable over

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[8] Hall, Brown, Nelson, and Compton, "Apparatus for use with condensed phases at 10 000°C" (to be published).

[9] J. C. Jamieson, J. Geol. **65**, 334 (1957).

graphite. Some recent work at Brigham Young University indicates that the sintering might be carried out at pressures considerably below those required for diamond stability. The rate process for transformation of diamond to graphite is appreciably slowed by pressure. In attempting to make a cemented diamond composition, therefore, it would only be necessary to utilize such a pressure as would be required to reduce graphitization to a negligible amount at the temperature of operation and length of time involved. This pressure is generally considerably less than the diamond-graphite equilibrium pressure. It is quite possible that future work in the area of very high temperatures and pressures will uncover materials with properties superior to those now known that would be suitable for use in high-pressure equipment. One substance, cubic boron nitride, the equal of diamond in scratch hardness, has already been reported.[10] Because of the reputedly superior heat resistance of this material over that of diamond (and because of its different chemical constitution) it might be much easier to prepare a satisfactory sintered cubic boron nitride composition than the analogous diamond composition.

MULTISTAGING

In theory, it should be possible to attain any desired pressure by a cascading or multistaging process whereby one pressure apparatus is placed in the working volume of a larger apparatus and so on. The unsupported portion of a moving element such as a piston would, in such an apparatus, be subjected to a differential pressure no greater than its compressive strength. The inner piston of a two-stage apparatus should generate a 100 000-atmos pressure within an inner vessel if the inner apparatus is surrounded by a fluid at 50 000 atmos generated by the piston of the outer apparatus.

In practice multistaging is very difficult and has not been carried beyond two stages. P. W. Bridgman[11] has succeeded in making *P-V* measurements at room temperature in such a device. The difficulties encountered in introducing electrical leads into the inner chamber as well as the mechanical problems of assembly and disassembly have deterred the development of a two-stage apparatus which utilizes internal heating.

GEOMETRICAL ADVANTAGE—USE OF COMPRESSIBLE GASKET TO OBTAIN MOTION

By definition the compressive strength of a substance is that obtained by measurement of the load required to cause a right circular cylinder of the material to fail under axial load. In this test the load is distributed across the entire face of each end of the cylinder. The ratio breaking-force/area gives the average pressure on a face at failure. If the specimen subjected to axial load is a truncated cone rather than a right circular cylinder, a "geometrical-advantage" is obtained and the truncated section will withstand greater pressure at failure than a right circular cylinder of the same cross sectional area. This is so because atoms on the truncated section have mechanical ties fanning out in regularly increasing area towards the base of the cone whereas the mechanical ties to the end of a right circular cylinder reach back into an area that remains constant with distance. The greater the included angle of the truncated cone, the more pronounced will be the geometrical-advantage effect. P. W. Bridgman[12] was the first to make use of this principle in the design of high-pressure equipment. In addition to this principle, he used the idea of a compressible gasket to obtain motion. His device, the so-called flat anvil apparatus, is shown in Fig. 6. Two hard-metal anvils A and B, each surrounded by a shrunk-on binding ring C and D (to provide lateral support), oppose each other. The opposing portions of the anvils are broad, truncated cones. Thin circular disks of solid materials placed between the truncated areas can be subjected to average pressures of 100 000 atmos and more. (Bridgman obtained 450 000 atmos average pressure between opposing faces of the anvils by surrounding the entire assembly with 30 000 atmos hydrostatic pressure.)

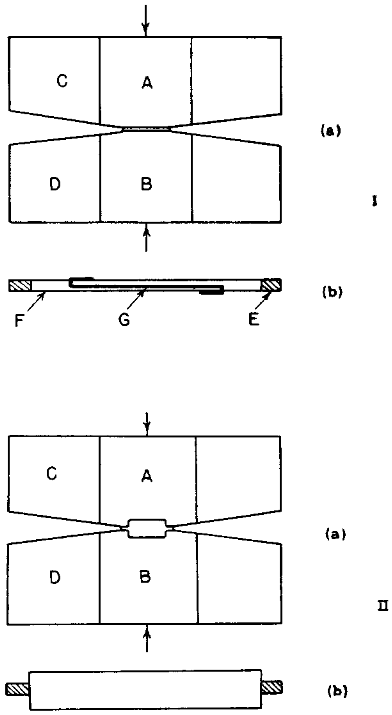

FIG. 6. Bridgman "anvil" apparatus.

By use of an assembly shown in enlarged cross section at I(b) in Fig. 6, Bridgman measured the electrical resistance of many conductors at room temperature to 100 000 atmos. This assembly was approximately $\frac{1}{2}$ in. diameter (same as

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[10] R. H. Wentorf, Jr., J. Chem Phys. **26**, 956 (1957).

[11] Reference 1, p. 401.

[12] P. W. Bridgman, Proc. Roy. Soc. (London) **A203**, 1 (1950).

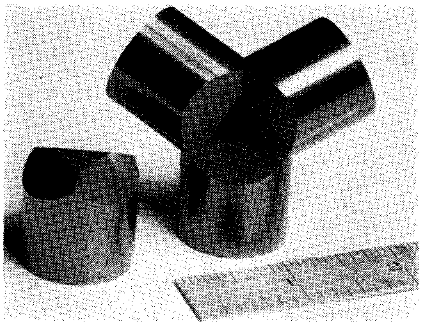


FIG. 7. Detail of "tetrahedral anvils."

anvil face diameter) and 0.010 in. thick. A pipestone (catlinite) ring E surrounds a circular wafer of silver chloride F in which is imbedded a ribbon of metal G, the resistance of which is to be measured. This assembly is compressed between the anvils. The pipestone ring E serves as a gasket to confine the silver chloride which is highly fluid at high pressures. Pipestone is an iron-containing, hydrous aluminum silicate very similar in constitution and properties to pyrophyllite. Its surface and internal friction are greater than those of pyrophyllite, it is much more difficult to obtain, is less uniform and is more difficult to machine. In general pyrophyllite can be substituted for pipestone in high-pressure gasketing applications. When the surface friction of pyrophyllite proves to be too low in a given application (as evidenced by undue extrusion or blowing of gaskets) the situation can be remedied by application of a coat of red iron oxide powder (rouge) to the surface as previously described.

TETRAHEDRAL ANVIL DESIGN

Bridgman's anvil apparatus is primarily a two-dimensional device in which the sample to be compressed has appreciable breadth and width but very small depth and consequently a small volume. It would be very desirable to somehow preserve in a three-dimensional device the features that make this two-dimensional device operable. In a practical three-dimensional device it would be desirable to have the breadth, width and depth of the sample of comparable dimensions. One means to this end would be to provide cylindrical recesses in the anvil faces as shown in Fig. 6-II(a). The sample could then be thickened as shown in 6-II(b). Bridgman (unpublished) experimented with such a device and reported that pressures obtainable were much reduced and breakage was severe. Lower pressures would be expected because of the great reduction in relative motion obtained. Relative motion, here defined, is anvil motion/sample thickness. Relative motion can not be increased by increasing the thickness of the pipestone gasket because greater thickness (other dimensions constant) blows the gasket.

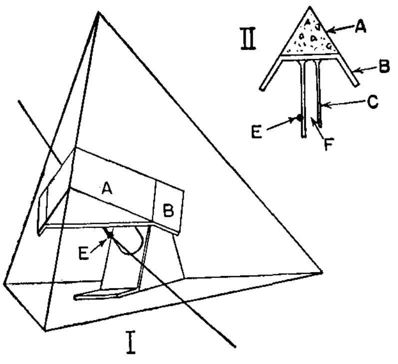


FIG. 8. (I) Detail of pyrophyllite tetrahedron with diagonal edge-to-edge heater-sample tube. (II) Detail of heater sample tube.

I have utilized the salient features of Bridgman's anvil apparatus in the construction of a three-dimensional device called the tetrahedral anvil apparatus. At the present time this apparatus has been successfully used at pressures of 100 000 atmos and temperatures of 3000°. Instead of two opposing anvils with circular faces, I have used four with triangular faces. The anvils are driven together by hydraulic rams along lines normal to the triangular faces, said lines intersecting at tetrahedral angles in the center of a regular tetrahedral volume enclosed by the anvils themselves (see Figs. 7, 8, and 9). During use, the anvils are each surrounded by a steel binding ring. In this device a regular, pyrophyllite tetrahedron [Fig. 8(I)], the legs of which are 25% longer than the corresponding legs on the triangular anvil faces, serves as pressure transmitting medium, thermal and electrical insulation, and provides the necessary compressible gasket. The sample container, a tube which also serves as electrical resistance heater, is located within the pyrophyllite tetrahedron and runs diagonally from opposite edges as is also shown in Fig. 8(I). Electrical connections are made to the sample tube [C of Fig. 8(II)] through metal tabs shaped as shown in cross section at B of Fig. 8(II). These tabs are spot welded to the ends of the metal tube C thus sealing in the contents.

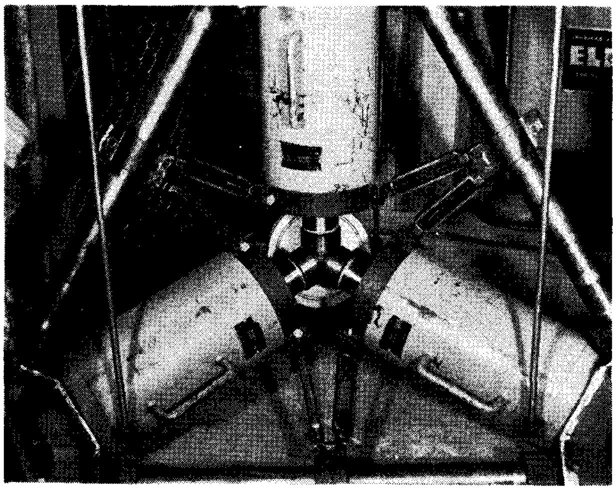


FIG. 9. Photograph of tetrahedral anvil apparatus.

The metal tabs from each end of the sample tube make electrical contact with the faces of a pair of anvils which bring in the heating current. Prisms of pyrophyllite A provide thermal insulation at the ends of the sample container. A thermocouple E is spot welded to the sample container, and the leads are brought out through edges of the tetrahedron as shown. Friction of the pyrophyllite on the fine wires is sufficient to hold them in place during high pressure operation. Several electrical leads may be inserted into the sample area without difficulty.

In assembling a system such as that described, it is usually necessary to drill several holes and make several saw cuts to effect assembly. Any large holes left in the pyrophyllite should be filled with snug fitting plugs of the same material. Small slots and holes can be filled with powdered lava, kaolin, or iron oxide worked in with a dental spatula.

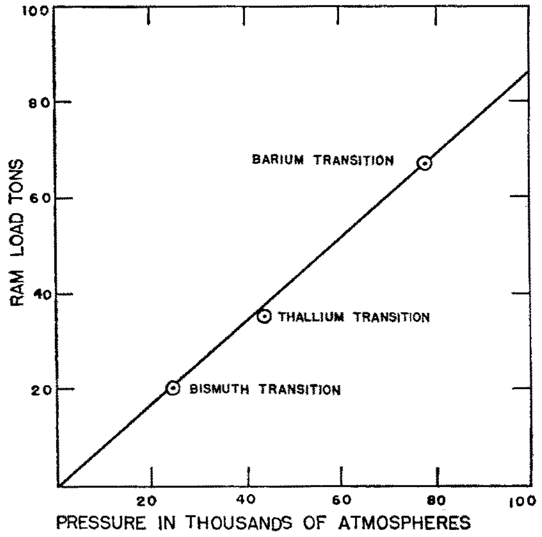


FIG. 10. Calibration plot for tetrahedral anvil apparatus.

In operation, the pyrophyllite tetrahedron is centered on the anvil faces which have been painted with rouge to increase friction. Then the anvils are simultaneously forced together. Since the triangular faces of the pyrophyllite tetrahedron are larger than the triangular anvil faces, some pyrophyllite is forced between the sloping sides of the anvils and a gasket is automatically formed. Continued motion of the anvils compresses gasket and tetrahedron and consequently the sample.

Pressure calibration of the sample volume F of Fig. 8(II) is made by means of silver chloride and pressure-sensing wires in the manner previously described for the simple piston-cylinder apparatus. A typical calibration plot for a tetrahedral anvil apparatus is given in Fig. 10.

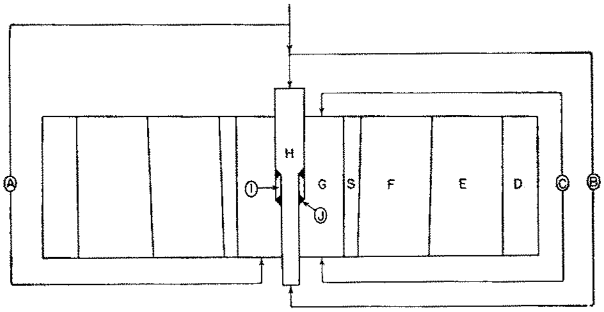


FIG. 11. Schematic diagram of stepped piston apparatus.

The volume of the pyrophyllite tetrahedron in the apparatus currently being used is about 16 times as large as the sample container volume. Because of this large ratio, differences in compressibility of various liquids and solids subjected to high pressure and temperature within the sample space do not appreciably affect the pressure calibration.

Although the tetrahedral-anvil device that has been built and tested provides only a very small working volume, there is no reason why the apparatus could not be scaled-up considerably to increase the working volume to a more convenient size.

STEPPED PISTON DESIGN

I have used the geometrical-advantage principle in the design of another device: the stepped-piston apparatus shown in Figs. 11 and 12. In this apparatus the working volume consists of an annular space I (Fig. 11) formed by the stepped piston H and stepped cylinder G. The annular area of the piston step is approximately $\frac{1}{5}$ of the area of the large diameter portion of the piston. This annular area has mechanical ties reaching back into areas of larger cross section and will, therefore, by the geometrical-advantage principle support a greater load on the annulus than would a right circular cylinder of the same area. This same principle applies to the annular step of the cylinder.

A force applied as shown at A of Fig. 11 will cause a

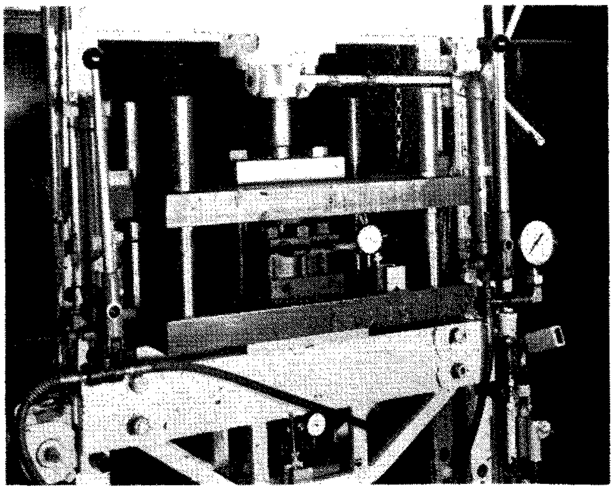


FIG. 12. Photograph of stepped piston apparatus.

specimen in the annulus I to be compressed. Experience with this device shows that pressures in the annulus near 45 000 atmos cause the cemented tungsten carbide piston (6% cobalt binder) to break at the step in a plane perpendicular to the center line of the piston. This is, no doubt, a manifestation of the pinch-off effect previously described by P. W. Bridgman.[13] A piston clamping force applied as shown at B of Fig. 11 will prevent the pinch-off effect. The sum of the forces A+B when divided by the area of the large diameter portion of the piston must yield a quantity no greater than the normal compressive strength of the material from which the piston is constructed. With forces applied as thus far described, the apparatus is operable to about 70 000 atmos. At this pressure a lateral break, beginning at the step, occurs in the carbide cylinder G. This can be prevented by a cylinder clamping force shown as C in Fig. 11. By utilization of these clamping forces it has been possible to compress solid materials in the stepped-piston apparatus to annular loadings corresponding to 200 000 atmos before piston failure occurs.

The letters D, E, F, and S in the diagram of Fig. 11 represent tapered, interference-press-fit binding rings which are designed to give lateral support to the cylinder G. Hardened steel rings of triangular cross section (Fig. 11, J) are used as gaskets when necessary. All sliding surfaces, including the external surfaces of any solid specimen to be compressed, are lubricated with molybdenum disulfide powder.

In this design the piston and cylinder should fit within ±0.0002 in. above and below the step. This requires a rather expert and expensive concentric grinding operation. Some preliminary experiments have indicated a possible solution to this problem. The stepped piston and cylinder (with associated binding rings) are each built in two pieces—one piece being that portion of piston or cylinder located above a horizontal plane through the step and the other piece being that portion below the plane. These pieces are clamped together by the clamping forces indicated in Fig. 11. Before the pieces are clamped together, however, the contacting surfaces are given a thin coat of rouge. The high friction of the rouge between the clamped surfaces prevents entry of the specimen being compressed. The advantage of building these parts in two pieces lies in the fact that each piece may be precision ground to its own diameter without regard to any problem of concentricity.

In the form just described, the stepped piston apparatus is useful for making compressibility measurements at room temperature. If increased considerably in size it could conceivably be adapted to internal heating along the lines already described for the simple piston-cylinder and tetrahedral-anvil designs.

In the apparatus pictured in Fig. 12, forces A and B of Fig. 11 are provided by hydraulic rams while force C is provided by a mechanical clamp. This clamp consists of two heavy hardened plates which are drawn together by six heavy tension bolts.

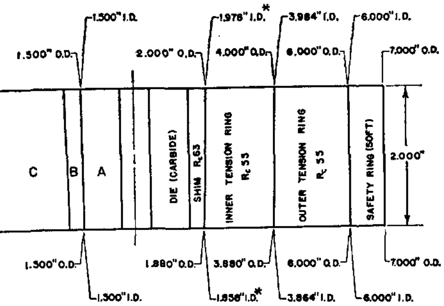
BINDING RINGS

For several years I have experimented with compound binding ring assemblies for use in providing lateral support to cemented tungsten carbide cylinders such as are used in the simple-piston and cylinder and stepped-piston designs. The initial designs were based on theoretical calculations. These have been modified by experience. Figure 13 gives dimensions and other pertinent data for a typical set employing two tension rings. Note that the rings are tapered and are in interference; i.e., the outside diameter of the inner ring is greater than the inside diameter of the outer ring. Rings are assembled from the "outside-in" by means of a hydraulic press. The most practical material available at the present time to use in construction of the tension rings is aircraft quality, mandrel forged AlSl 4340 steel. This material is much used in the construction of aircraft landing gear.

In the assembly of Fig. 13, the shim serves an important function. If the shim were not used, the carbide die would be built with a tapered outside surface that would be in contact with the inside tapered surface of the inner binding ring. After continued usage, the die will eventually break. The broken die, which is under lateral compression provided by the tension rings, requires considerable force for its removal. During removal the jagged fragments of the carbide die invariably damage the inner tension ring beyond repair. If a shim is interposed between cylinder and tension ring, the shim plus broken cylinder can be simultaneously pushed out without causing any damage. The shim, which is composed of four segments, is then readily removed from the broken carbide.

MISCELLANEOUS METHODS FOR OBTAINING HIGH PRESSURE

If the simple-piston and cylinder apparatus were clamped shut with no portion of the piston protruding it



*NOTE — THIS DIMENSION IS GROUND WITH RING C IN D IN E. OTHER BINDING-RING AND SHIM DIAMETERS ARE GROUND WITH RINGS DISASSEMBLED. SHIM, AFTER HARDENING & GRINDING, IS SLIT LONGITUDINALLY INTO 4 EQUAL SECTIONS.

FIG. 13. Binding ring set.

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[13] Reference 1, pp. 33 and 91.

would probably withstand 500 000 atmos if the pressure were somehow generated from within. Pressure could be generated from within by materials that expand on freezing. Bismuth which can generate a maximum theoretical pressure near 18 000 atmos by this means has been used in some applications. Germanium offers the best prospects for obtaining very high pressures in this manner. Measurements of the melting point of Ge as a function of pressure have been made to 180 000 atmos[7] and disclose that even at this pressure the liquid is more dense than the solid. Therein lies the possibility of achieving maximum pressures of at least 180 000 atmos on the freezing of this substance. Chemical reactions, phase changes, and expansion due to heating also offer possibilities for obtaining high pressures within a confined volume.

Another possible method for obtaining high pressures depends on a mechanical anisotropic property. Many substances, when axially compressed, suffer a larger deflection per unit length in the direction of the applied force than in a direction transverse to the force. Should a transverse force be applied to remove the transverse deflection it would be discovered that the force per unit area required to do this would be greater than the original force per unit area in the axial direction. This phenomenon is a manifestation of the internal arrangement of atomic bonds . . . a complex system of levers. These levers give a mechanical advantage on the atomic scale which, because of the large numbers of atoms involved, gives a macroscopically observable effect.

One method of utilizing mechanical anisotropy in a high-pressure device would be to construct a cylinder of the anisotropic material with an axial hole. A solid specimen would then be placed inside, and plugs of high friction material would be used to seal each end. Cylinder plus specimen would be slid into the chamber of a simple-piston and cylinder apparatus (the cylinder being a snug fit to the chamber wall). The piston would then compress cylinder plus sample. If the solid specimen to be compressed were a material of sufficiently low compressibility, the pressure that would be exerted on it would be greater than the average pressure on the piston face. Of course the pressure on the central area of the piston face which covers the plug of high friction material would be greater than that on the remainder of the face. This would be dissipated however by the geometrical advantage effect.

The great pressures existing in the depths of the earth or the still greater pressures within the stars do not depend for their generation upon materials of great compressive strength (as compressive strength is ordinarily understood) but depend, of course, upon gravitational forces between the particles that comprise these bodies. It would seem that the attainment of comparable pressures in the laboratory must ultimately depend upon a similar process. Gravitational forces are, of course, too small to be used in this manner on a laboratory scale. However, the use of effectual electric and magnetic fields is not beyond the realm of possibility. Indeed, pulsed magnetic fields exhibiting tremendous forces have just recently been described.[14]

ACKNOWLEDGMENT

The author expresses his appreciation to Duane Dudley for his extensive testing of the tetrahedral-anvil apparatus.

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[14] Furth, Levine, and Waniek, Bull. Am. Phys. Soc. Ser. II, 2, 7 (1957).