GENERAL ELECTRIC
Research Laboratory

THE "BELT": ULTRA-HIGH-PRESSURE, HIGH-TEMPERATURE APPARATUS

by

H. Tracy Hall

Report No. RL-1064 March 1954

SCHENECTADY, NEW YORK

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ABSTRACT

An apparatus for subjecting materials to 80,000 atmospheres pressure at temperatures of 2400°C is described.

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THE "BELT": ULTRA-HIGH-PRESSURE, HIGH-TEMPERATURE APPARATUS

H. Tracy Hall

In the "belt," an ultra-high-pressure, high-temperature apparatus, materials can be subjected to a pressure of 80,000 atmospheres at a temperature of 2400°C.

Diagrams of the apparatus are shown in Figs. 1 and 2. A photograph is shown in Fig. 3. A photograph of the chamber parts is shown in Fig. 4.

The functions of the various parts are as follows (see Fig. 1): Two conical pistons (1) push into each side of a specially shaped chamber (2). Pressure is transmitted to the sample [contained in the metal tube (3)] by Wonderstone (4), a hydrous aluminum silicate. The Wonderstone serves also as thermal and electrical insulation. The sample is heated by passage of an electric current through the metal heating tube (3). Current enters the heating tube from the pistons via the steel conducting ring (5) and the metal end disks (6). The short Wonderstone cylinders (7) provide thermal insulation at the ends of the sample. A sandwich gasket composed of Wonderstone and steel parts (8), (9), and (10) maintains the pressure in the chamber. Hardened steel binding rings (11) and (12), which are strained almost to their elastic limits by forced-on tapered fits, greatly strengthen the chamber (2). Binding rings (13) and (14) do the same for the conical pistons. Soft steel safety rings (15) and (16) protect personnel from flying fragments when binding rings break.

The chamber (2) and rings (11) and (12) form a toroidal "belt" around the sample, hence the name of this apparatus. The equipment is water-cooled as shown.

The reasoning behind this rather unorthodox design can best be brought out by tracing its historical development.

In 1952, P.W. Bridgman(1) described an ingenious single-stage apparatus for producing pressures to 100,000 kg/cm^2 (1kg/cm^2 = 0.96777 atmosphere). The apparatus (see Fig. 5, I) consisted of two Carboloy "anvils" A and B held in compression by steel binding rings C and D. The anvil faces are one-half inch in diameter and slope away at a

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(1) P.W. Bridgman, Proc. Am. Acad. Arts and Sci. <u>81</u>, 165-251 (1952).

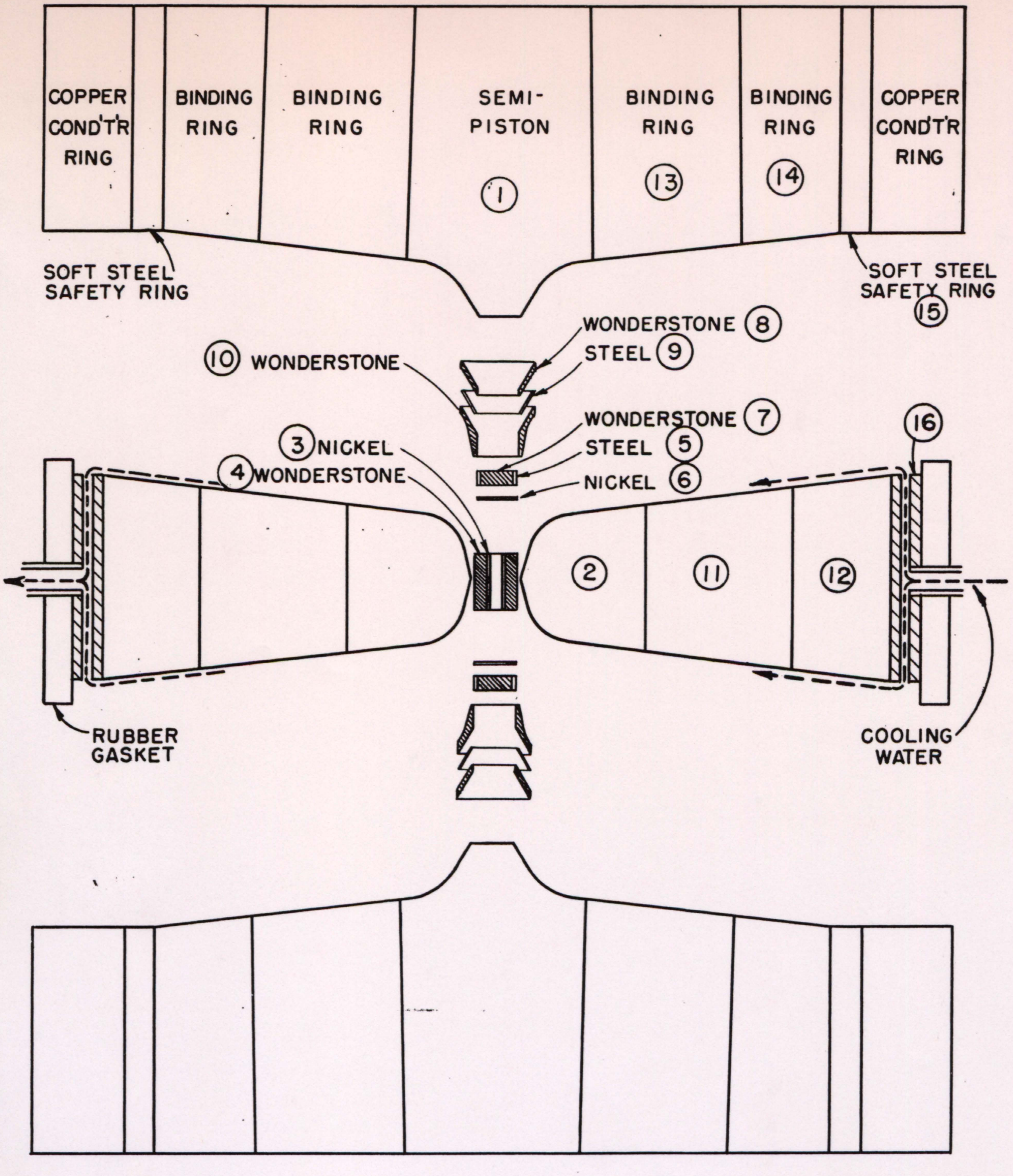

Fig. 1 The "belt": high-temperature, high-pressure apparatus. "Exploded" assembly.

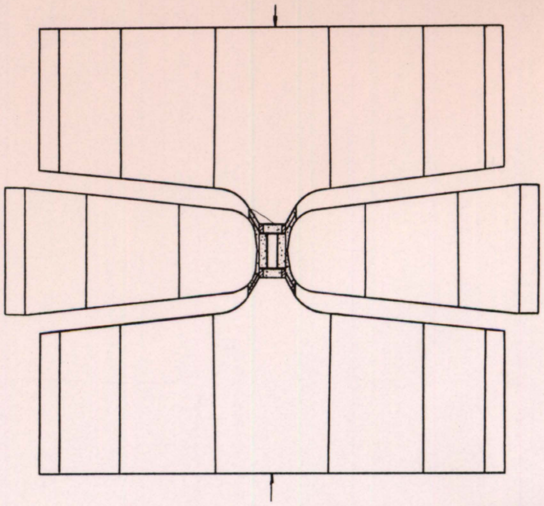

Fig. 2 The "belt," ultra-high-pressure, high-temperature assembly.

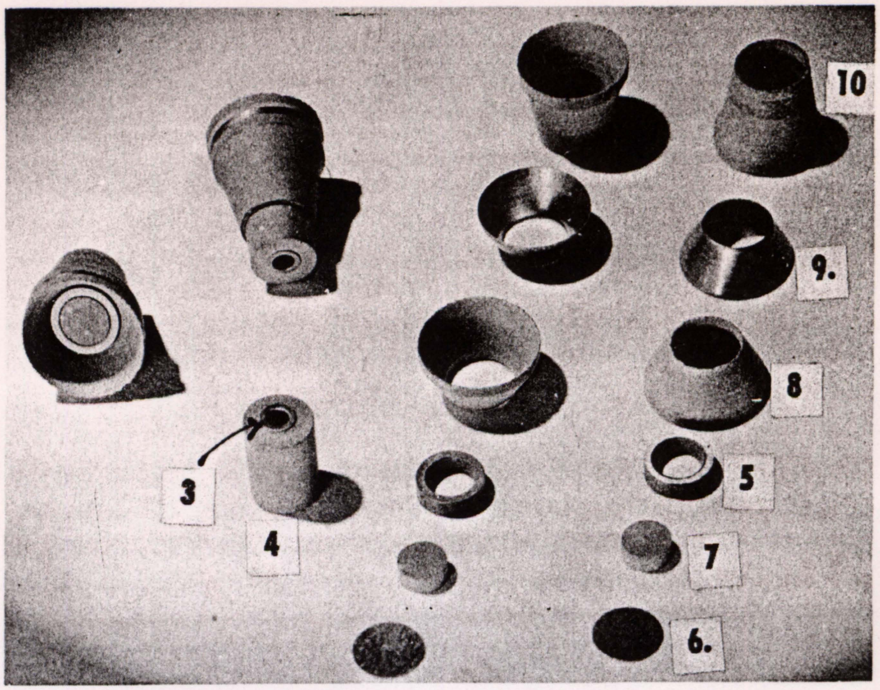

Fig. 3 Photograph of apparatus.

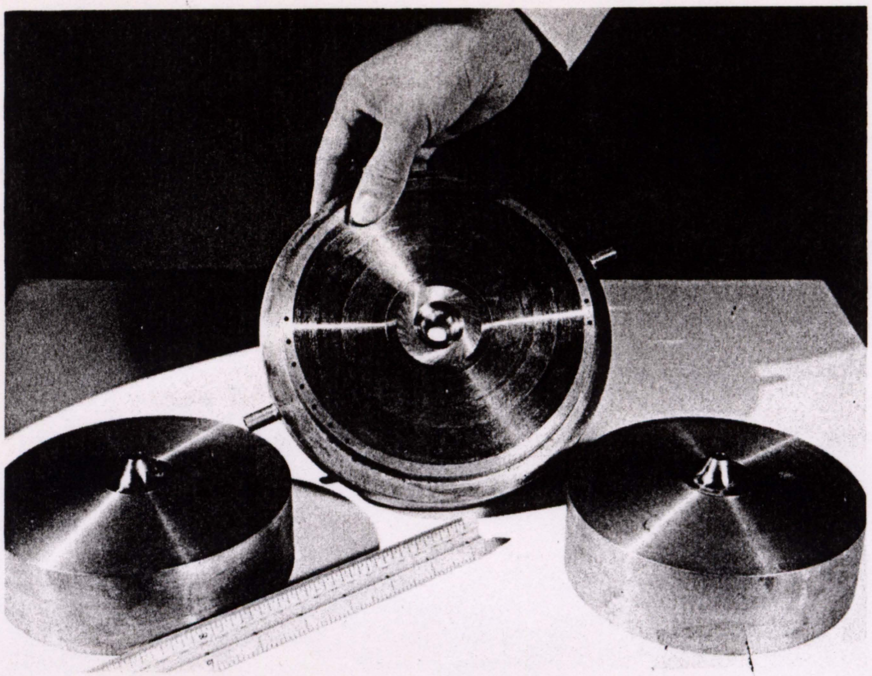

Fig. 4 Chamber parts.

four-degree angle. The sample to be compressed is a disk 0.407 inch in diameter and 0.010 inch thick. The sample is contained by a "pipestone" ring or gasket 0.500 inch OD, 0.407 inch ID, and 0.010 inch thick. "Pipestone" or Catlinite is a red, indurated clay from the upper Missouri region. It is found at Pipestone National Monument, a part of the Sioux Indian Reservation in Minnesota. As the name implies, the Indians carve smoking pipes from the material.

Pipestone, in thin sections, has the property of becoming very strong in a transverse direction to the direction of compression. Under compression, the submicron-size grit of the pipestone "bites" into the Carboloy, giving very high frictional resistance to relative Carboloy-pipestone motion.

Bridgman has measured the electrical resistance of most of the metals to 100,000 kg/cm^2 (taking pressure as force over area) by folding a ribbon of the metal between two silver chloride disks as shown in Fig. 5, Ib. Silver chloride has about the same compressibility as pipestone. In addition, it has an extremely low coefficient of friction and is the best solid pressure-transmitting "fluid" known at the present time. No liquids are known to remain liquid at pressures greater than 35,000 kg/cm^2 at room temperature.

The sample in Bridgman's equipment is very small, and of course cannot be heated to high temperature. A larger sample could be obtained at a sacrifice in ultimate pressure by making recesses in the anvils as shown in Fig. 5, II.

Francis P. Bundy, of this laboratory, designed a set of anvils along these lines [see Fig. 6 (A)]. The recess in the anvils is "saucer"-shaped.

The curved faces tend to force the gasket material (a) inward when the anvils come together helping to increase the pressure. Bundy provided recesses in the anvils filled with fired Al_2O_3 or MgO for thermal insulation. The sample to be compressed was placed inside a metal or graphite tube that could be heated by passage of an electric current. Bundy, following the suggestion of L. Navias of this laboratory, found that Wonderstone(2) (pyrophyllite), a hydrous aluminum silicate, could be used in place of pipestone for the gasket. It is more easily obtained, is less expensive, and is a more uniform material than pipestone. Bundy found about 0.030 inch to be the maximum gasket thickness that could be used at the anvil shoulders (the part of the anvils that touch if brought

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(2) Available as "Lava, Grade A" from American Lava Corporation, Chattanooga 5, Tennessee.

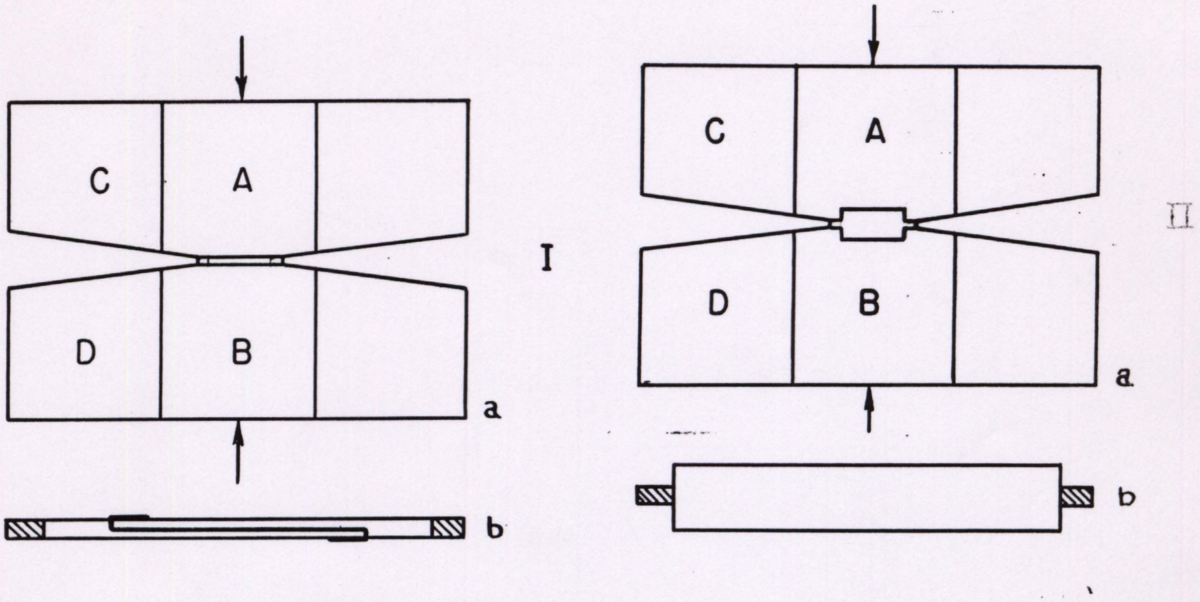

Fig. 5 I, P.W. Bridgman's "anvil" apparatus for producing pressures to 100,000 kg/cm^2; II, modification of Bridgman's apparatus.

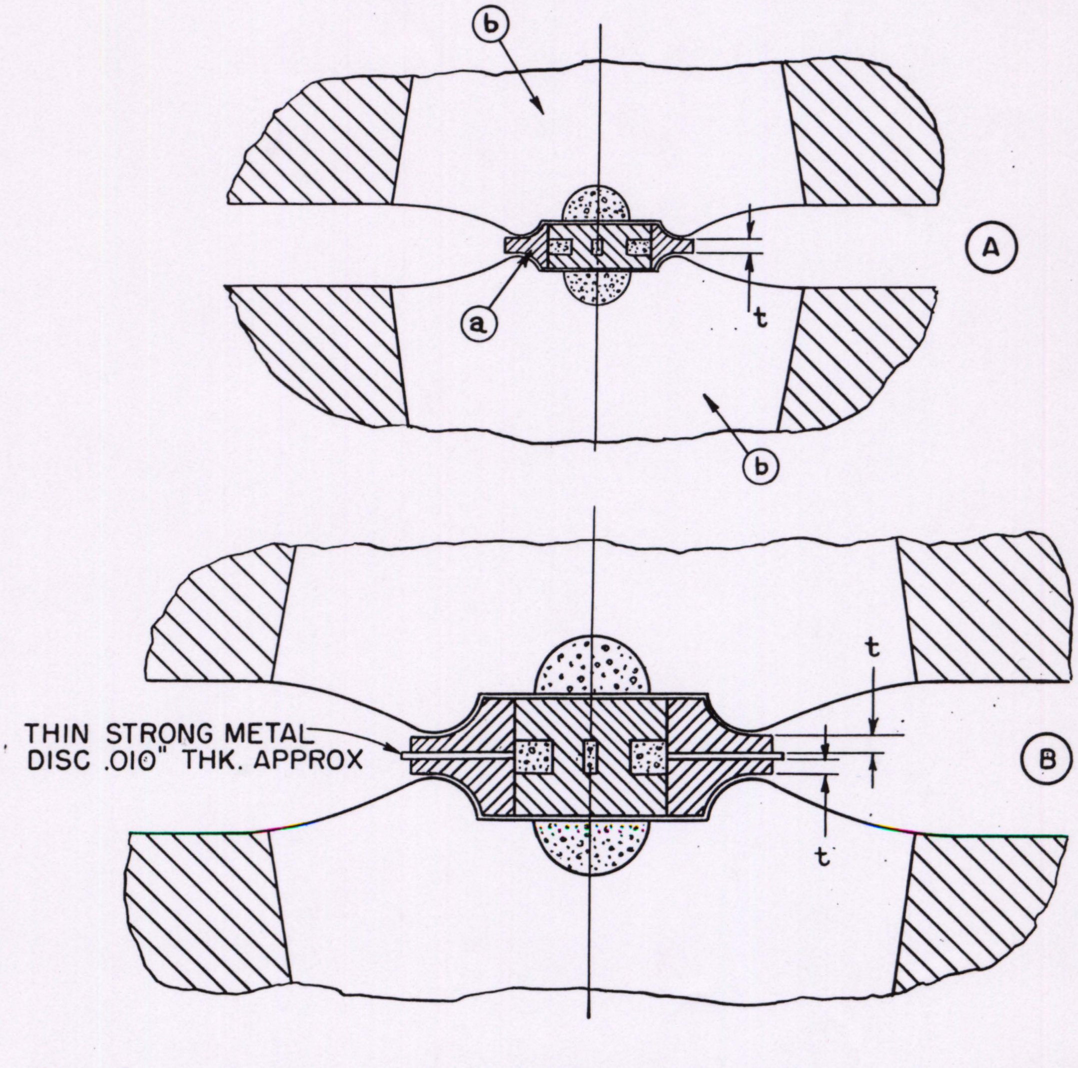

Fig. 6 A, Francis P. Bundy's "saucer" anvils; B, "sandwich" gasket.

together without gasket and sample). Thicker gaskets tend to crumble and break out, with resultant loss in pressure. Of course, the thickest gasket possible should be used, since the thicker the gasket, the larger the sample that can be accommodated. This device performed very satisfactorily to temperatures of about 2500°C and pressures somewhat over 35,000 atmospheres. At this pressure, the gasket thickness at the shoulders had decreased to a limiting value in the neighborhood of 0.002 to 0.004 inch. Further press load cannot bring the anvils closer together, and hence a limiting pressure is reached.

The success of Bundy's "saucer" anvils inspired a series of innovations by H.M. Strong, R.H. Wentorf, J.E. Cheney, H.P. Bovenkerk, and the author. These were aimed primarily at: (1) increasing the pressure and (2) increasing the sample size. The "belt," the author's own particular design, achieved these aims to a high degree.

Three ideas give the belt its high performance. They are listed below.

(1) The "sandwich" gasket (patent letter to C.G. Suits, May 22, 1953).

This idea was first tested in the "saucer" anvils. I found that the thickness of the gasket at the shoulders of the anvils could be more than doubled by inserting a thin metal disk between two gaskets as shown in Fig. 6 (B). This increases the sample thickness by 0.040 inch (0.010-inch-thick steel or nickel disk + 0.030 inch additional Wonderstone). In operation, each thickness of Wonderstone crushes down to its limiting value of 0.002 to 0.004 inch. The steel or nickel disk, like the stone, becomes extremely strong transverse to the direction of compression.

Pressures of 47,000 atmospheres, as measured by a manganin wire gage,(3) were obtained with this arrangement. By using still another metal and Wonderstone disk, sample thickness was increased an additional 0.040 inch, and a pressure of 51,000 atmospheres was obtained. At this writing, we do not know the limit to which this stacking may be carried nor the ultimate pressures that may be attained.

The "sandwich" gasket will increase the "stroke" and hence the sample size and ultimate pressure of any system using stone gaskets.

(2) The conical piston, gasket, and chamber (patent letter to C.G. Suits, February 3, 1953).

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(3) P.W. Bridgman, <u>The Physics of High Pressure</u>, G. Bell and Sons, London (1949), p. 72.

The author proposed that, for a gasket arranged at an angle less than 90° to the direction of compression [as is gasket (3) of Fig. 7], the thickness along the line of compression could be increased to $s = t/\cos\theta$. Here s is the distance between the arrows b-b (Fig. 7) and t is the usual thickness of 0.030 inch as in (A) of Fig. 6. The thickness perpendicular to the frictional surfaces (distance between arrows a-a in Fig. 7) is still t. The force acting perpendicular to the contact area between the gasket and the metal is reduced by $\cos\theta$ over the case where the gasket is perpendicular to the direction of compression. Therefore, the friction between the gasket and the metal will be reduced by the factor $\cos\theta$. Now, the smaller the angle θ is made, the larger s becomes. The limit on the smallness of θ is set by the frictional forces along the gasket-metal interface. When they become too small, the gasket will be blown out by the pressure. The design of Fig. 7 with $\theta = 45°$ gave pressures over 35,000 atmospheres in samples twice as thick as were possible in the "saucer" anvils, without utilizing the sandwich gasket.

The conical piston gives very much greater strength than the usual cylindrical pistons of high-pressure equipment. In addition, accurate alignment of tapered piston and chamber is not necessary, and a slight "cocking" will not break the piston.

(3) Double ending (patent letter to C. G. Suits, February 5, 1953).

If the part of the apparatus below line S of Fig. 7 is done away with and the part of the apparatus above S is rotated 180° around the line S, a "double-ended" apparatus which you now recognize as the "belt" has evolved.

Double ending has increased the symmetry, thus eliminating stress-concentration points.

Also, the sample size has been more than doubled. Removal of contents after a run is easily accomplished by ramming the material out of the chamber. In a design such as that of Fig. 7, a tedious digging process in the highly compressed Wonderstone is required to recover the contents of the sample tube.

Detailed plans for the "belt" are given at the end of this paper. The available stroke is 0.230 inch, compared with 0.026 inch for the "saucer." The chamber size is large enough to permit using thermal insulation as part of each sample instead of recessing it in the anvils as was done in the apparatus of Figs. 6 and 7. The recessed insulation is undesirable on two counts: (1) the recess weakens the anvils, thus lowering the ultimate pressure attainable and shortening apparatus life, and (2) the recessed insulation spalls and must be frequently replaced.

An unexpected bonus was obtained with the "belt." It was designed with the expectation that the total press load would be more or less evenly distributed over the projected area enclosed by the gasket of initial diameter 0.600 inch. Calculation of the average area over which the press load is applied to obtain known pressures (from known transition points) within the cell gives a circle of diameter about 0.400 inch. It is obvious that the center of the assembly, where the sample is located, sustains more load than the outer regions. Another indication of this is given in the picture of Fig. 8. This shows a gasket after a run at high pressure. The Wonderstone gasket is darkest where it has been compressed the most. The "belt," then, requires about half the force originally anticipated to obtain a given pressure within the sample. The gradual drop of pressure along the gasket probably occurs in the following idealized manner: The outermost shell of the gasket has only the atmosphere for outside support, but has some support from above and below due to friction. The outermost shell then gives some support to the back of the next shell, which can give even greater support to the next inner shell because of increased support at its back, and so on. The 30-degree "conical gasket" allows some slippage between gasket and metal; the outer shells slip more than the inner shells, so that the inner shells become more highly compressed. The gradual drop of pressure along the gasket provides a gradually decreasing support to the conical piston. This is a kind of multistaging effect. (Multistaging is building one piece of pressure apparatus inside another. Theoretically, tremendous pressures could be developed in this manner if the very difficult technical problems could be solved.)

The chamber [(2) of Fig. 1] also obtains a multistaging effect because the thrust of the conical pistons develops a compression component in the chamber parallel to the centerline of the system.

Temperature Calibration of the "Belt"

Alternating current is used for heating. If the heating tube is nickel (see plans for dimensions), a voltage drop of about 1.12 volts across the tapered pistons and a current of about 250 amperes is required to give a temperature of 1000°C.

Chromel and alumel thermocouple wire of 0.005-inch diameter with Formex insulation was used in the temperature calibration. A very tiny junction was placed at the desired point and the leads brought out through holes and shallow grooves in the Wonderstone. Figure 9, a picture of the chamber assembly and lead wires after a run, gives an idea of how this was done.

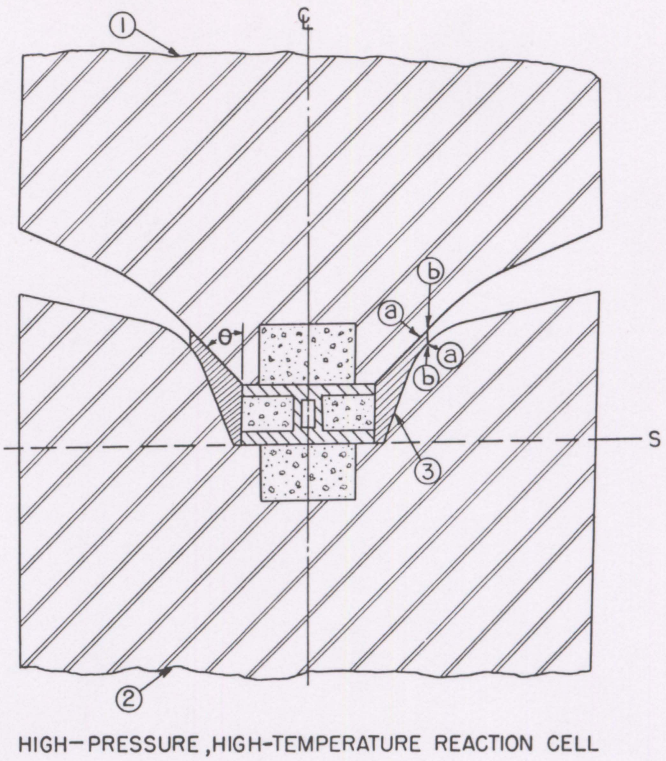

Fig. 7 High-pressure, high-temperature apparatus with conical piston, gasket, and chamber.

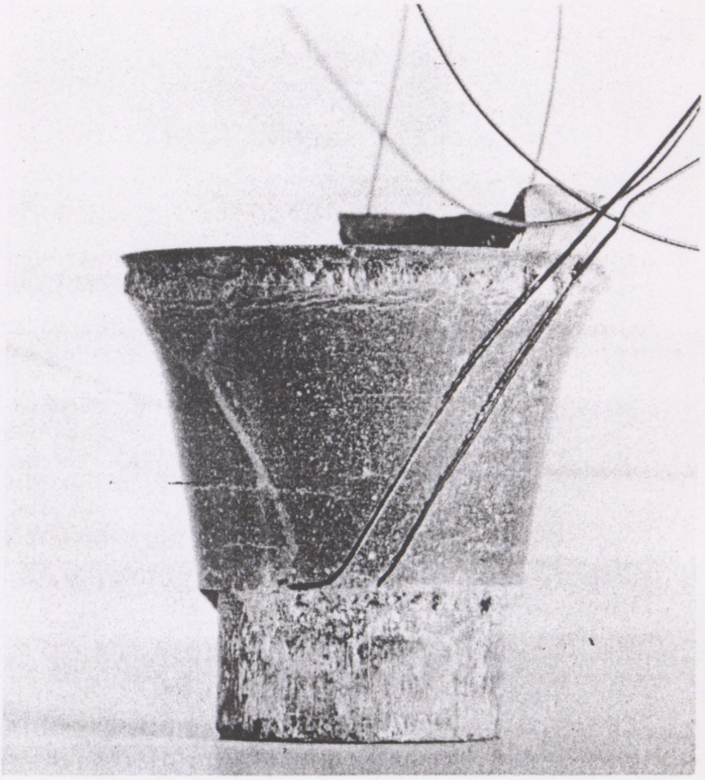

Fig. 9 Thermocouple wires emerging from gasket assembly.

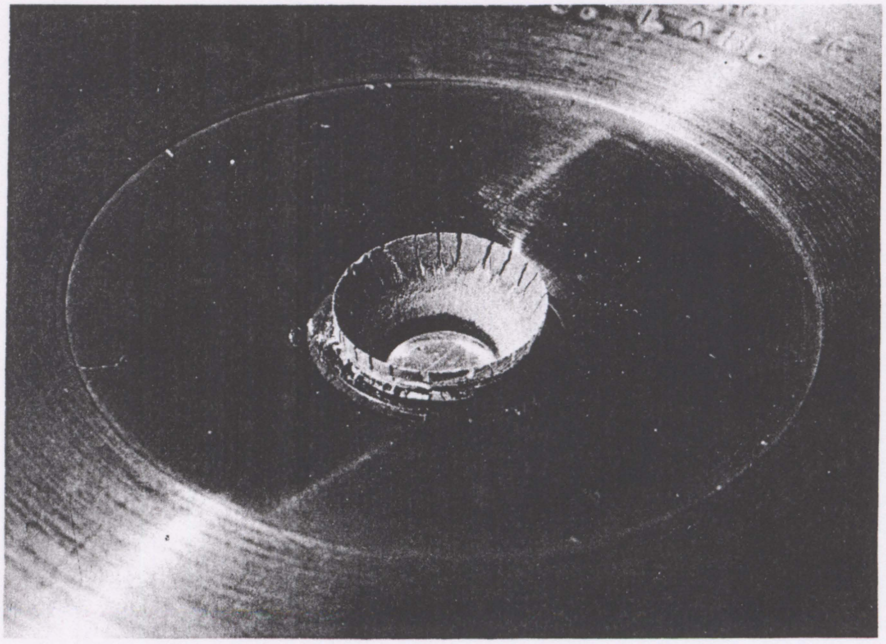

Fig. 8 Gasket after high-pressure run.

For the temperature calibration shown, all stone components were Wonderstone except the 0.250-inch-diameter by 0.100-inch-thick cylinder providing thermal insulation at the ends. This was pipestone. The heating tube was nickel, as were also the end disks. The heating tube was filled with compressed Wonderstone powder. The thermal conductivity of the material inside the heating tube will not affect the measured temperature very much, since the volume of sample inside the tube is only 1/25 of the volume of insulating material surrounding it.

In Fig. 10, Curve (1) gives the temperature measured by thermocouples located at the middle of the heating tube as a function of watts input. Curve (2) gives the temperature at $r = 0.200$ inch, $z = 0$ inch (using cylindrical coordinates for the system with the center of symmetry as origin). Moderate pressures of 2,000 to 10,000 atmospheres were used in making the temperature calibrations. Prior to the temperature calibration by thermocouple, the watts input required to melt lithium and potassium carbonates had been used to obtain an idea of the temperature. These points fell within the bounds of the thermocouple curve. More recently, "Thermocolor" paint,(4) which changes color at fixed temperatures, has been used to check the thermocouple calibration. The agreement is good. Curve (1) of Fig. 9 probably gives the temperature at the center of the tube to 5 per cent at the higher temperatures. Accuracy will be better at the lower temperatures. Thermocolor paint indicates that the temperature at the ends of the heating tube is near 900°C when the temperature at the center is near 1000°C. Homogeneity of temperature could probably be obtained by slightly increasing the thickness of the end insulation. Since our current interest is in crystal growing, we prefer to have a temperature gradient present.

Pressure Calibration of "Belt"

The press load required to produce a given pressure in the sample chamber of the "belt" has been determined with the aid of phase transitions. Unfortunately, there are not many of these suitable for making calibrations. Five were used: the sharp changes in electrical resistance of bismuth, thallium, cesium, and barium(5) at 24,800, 43,500, 53,200, and 77,400 atmospheres, respectively, and the growth of a new dense silica at 35,000 atmospheres(6). The calibration is shown in Fig. 11. Each of the transitions is discussed below.

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(4) Made by Badische Anilin and Soda Fabrik, Ludwigshafen A. Rheim. Available from Bryson Oil Co., Harriman, Tennessee.

(5) P. W. Bridgman, Proc. Am. Acad. Arts and Sci., 81 (4), 165-251 (1952).

(6) L. Coes, Science, 118, 131-2 (1953).

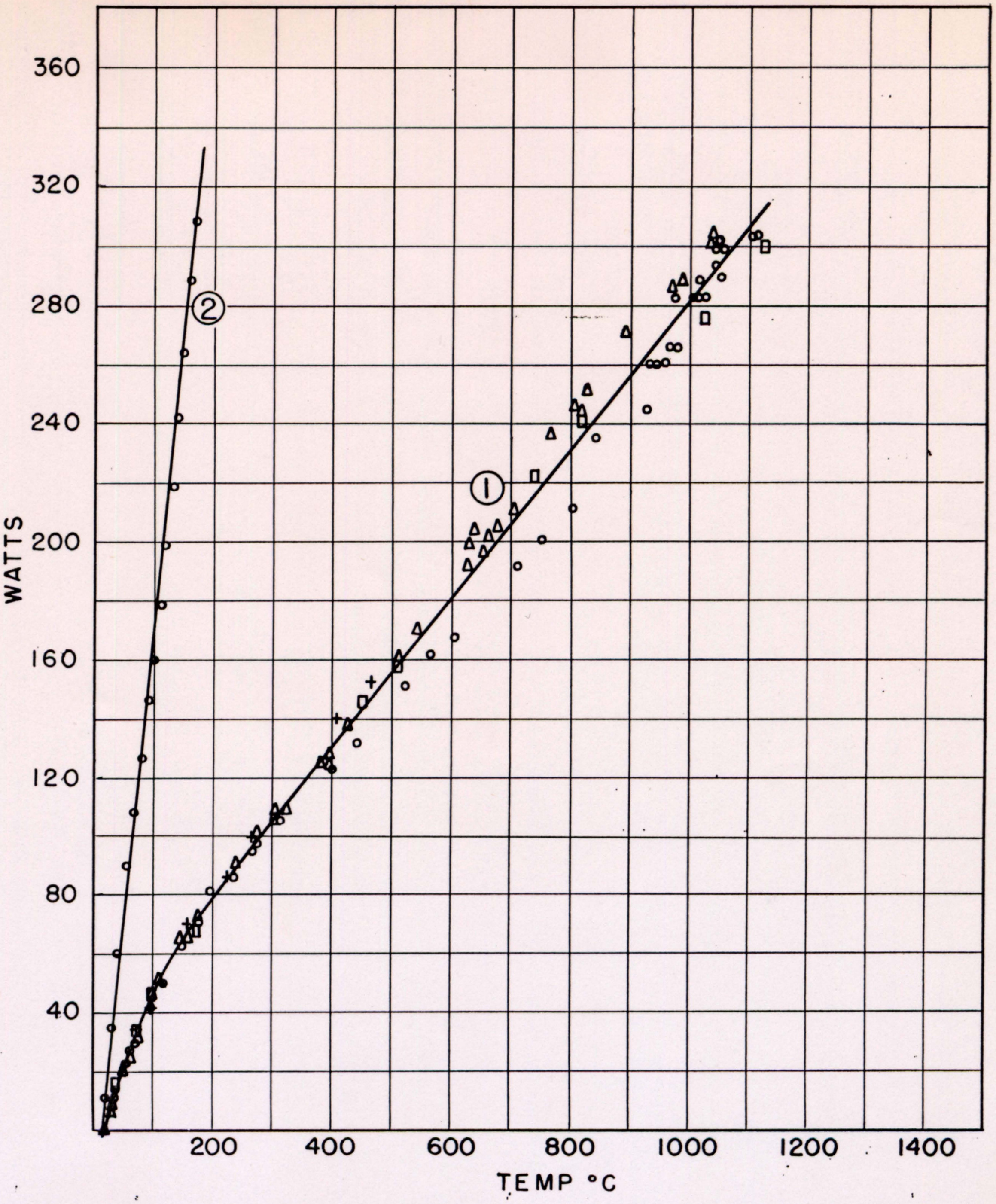

Fig. 10 Temperature vs watts input for "belt." (1) Center of heating tube; (2) temperature at r = 0.200 inch, z = 0 inch (center of symmetry = origin).

Bismuth Transition

A cylinder of silver chloride the same diameter and length as the heating tube was put in the Wonderstone in place of the heating tube. A bismuth wire about 0.025 inch in diameter was placed in a 0.030-inch-diameter hole drilled down the axis of the silver chloride. The bismuth wire made contact at each end with the nickel end disks. Resistance was measured with a Kelvin double bridge.

Figure 12 shows the resistance change with loading (1), unloading, and reloading (2).

The sharp decrease in resistance is due to the Bi I$\longrightarrow$Bi II transition occurring at 24,800 atmospheres. The less spectacular rise in resistance following the large drop is due to Bi II$\longrightarrow$Bi III at 26,200 atmospheres. A large hysteresis is noted on reducing the press load. Bridgman observed this same type of phenomenon in his "anvil" work.(1) When the press load is increased a second time, the Bi I$\longrightarrow$Bi II transition occurs at about 10 per cent lower press loading than on the initial loading. The initial loading apparently compacts the sample and allows the Wonderstone to adjust itself, so that less press load will cause the transition to occur on the second loading.

Dense Silica Formation

The author has studied the formation of a new, dense, crystalline form of silica(6) from solutions of waterglass. Near 650°C there is a sharply defined press load (71,000 pounds for the "belt") below which quartz crystals grow from the waterglass. Above this sharply defined press load, only dense silica grows. A change of 3 per cent in press load will shift the equilibrium one way or the other. Changing the temperature between 550° and 750°C does not appreciably affect the transition pressure. Five minutes of growing time is sufficient to produce myriads of dense silica crystals up to 500μ across. This is interesting in view of Coes'(6) reported 15 hours required to grow 40-μ crystals from sodium silicate in diammonium hydrogen phosphate. A photomicrograph of a dense silica crystal is shown in Fig. 13.

Coes gives the dense silica transition pressure as 35,000 ± 2000 atmospheres. This pressure is based on an extrapolation of the Bi I$\longrightarrow$Bi II transition pressure.

We noted how the press load required to produce the Bi I$\longrightarrow$Bi II transition was about 10 per cent lower on the second application of pressure than on the first. Such "preloading" of a waterglass sample does not

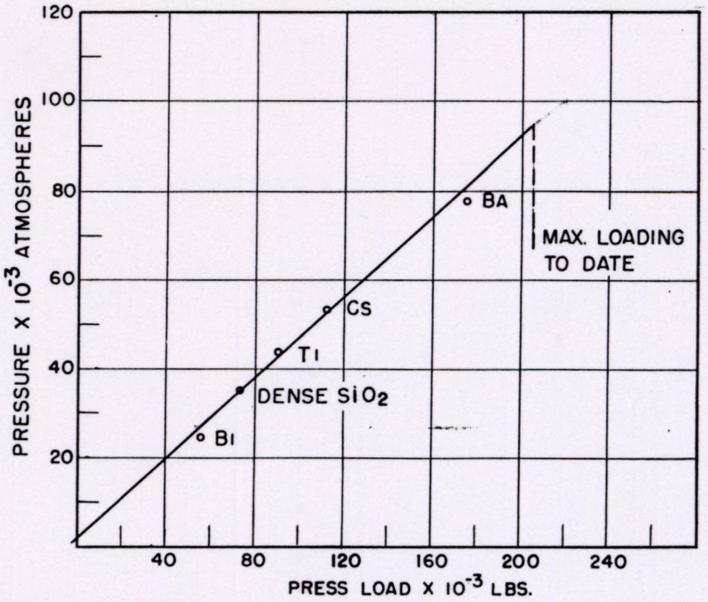

Fig. 11 Pressure calibration of the "belt."

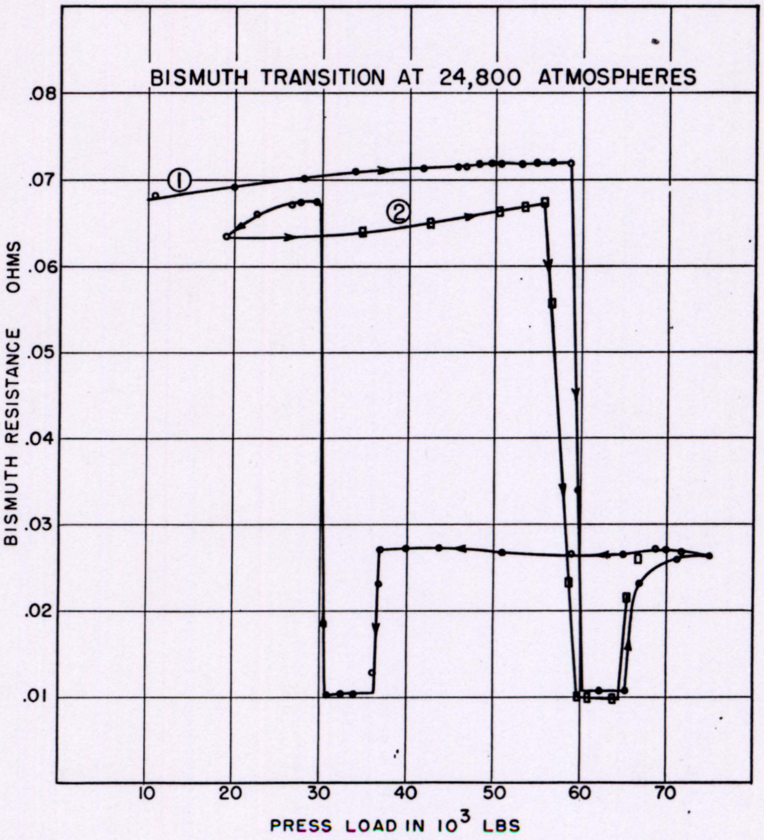

Fig. 12 Bismuth transition at 24, 800 atmospheres.

affect the load at which dense silica is formed on a second application of the load (first application cold; second application of load followed by heating). Apparently, the high temperature increases the Wonderstone "fluidity," so that the sample readily conforms to conditions imposed by the press load on the first application of pressure.

Thallium Transition

The thallium transition at 43,500 atmospheres was detected by the change in electrical resistance in the same fashion as the bismuth transition. A plot of resistance vs press load is shown in Fig. 14. Hysteresis is present as with bismuth. Observe also that the transition occurs at about 10 per cent lower press load on the second loading.

Cesium Transition

This transition gives a well-defined "pip" at 53,200 atmospheres (see Fig. 15). Curve (1) is the first cycle (compaction). Curve (2) is the second cycle and gives the usual lower press loading to obtain the transition.

The difficulties encountered in handling the extremely reactive metal cesium were overcome in the following way: A thin-walled glass vessel containing cesium in vacuum was broken under mineral oil. The mineral oil was at a temperature near 40°C, so that the cesium, which melts at 28.5°C, would be liquid. Thin-walled glass capillaries, 0.050 inch OD and about 10 inches long, were prepared. A little mineral oil was drawn into a capillary tube with a suction bulb and then, immediately behind the oil, the cesium was drawn in. When the tube was almost filled, a little mineral oil was drawn in behind the cesium. In this manner, the cesium was prevented from coming in contact with the atmosphere. A 0.400-inch-long section was then cut from the capillary, after daubing petroleum jelly where the cuts were to be made. The cut was made with side-cutting pliers.

The capillary containing the cesium was then inserted in a hole in the AgCl in the same manner as was the bismuth wire. The ends of the capillary were kept covered with petroleum jelly. Tiny copper "thumb-tacks" were then inserted in each end of the capillary to make contact with the cesium. The heads of the tacks make contact with the metal end disks.

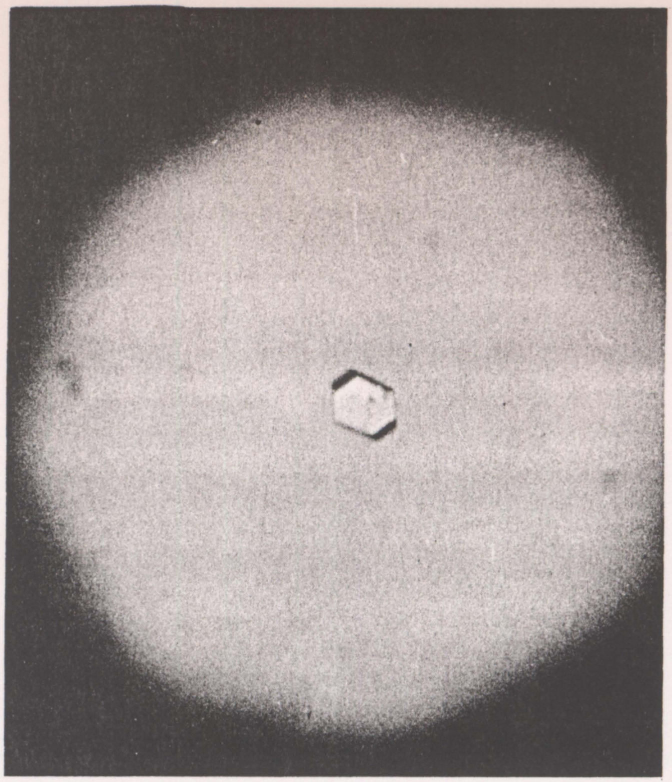

Fig. 13 Dense silica crystal.

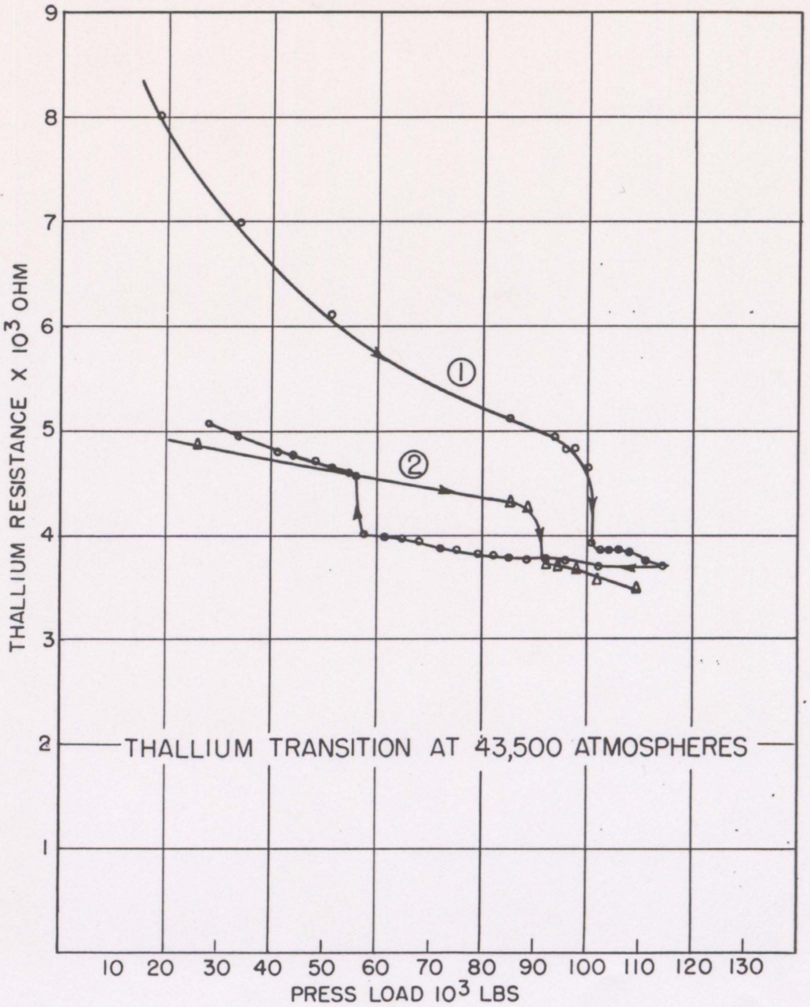

Fig. 14 Thallium transition at 43, 500 atmospheres.

Barium Transition

A barium wire was used in the same manner as was the bismuth wire. The barium must be protected from oxidation by a coat of petroleum jelly. The transition is shown in Fig. 16.

Life of Equipment

The tapered pistons and chamber of the "belt" are subjected to the highest stresses. The life of these parts will depend on the pressures used and the number of loadings and unloadings. Experience to date shows that conical, tool-steel pistons (Rockwell hardness C-60) begin to crack after about 100 runs at 45,000 atmospheres and 1000°C. Some of the runs lasted 5 to 6 hours, but the average run was 1/2 hour. The pistons can be used for about 25 additional runs after the cracks appear. At 45,000 atmospheres and 1000°C, the chamber when made of tool steel (Rockwell hardness C-60) begins to crack after about 40 runs. Again, some of the runs were 5 to 6 hours, but most of them average 1/2 hour. After the first cracks appear, an additional 10 to 20 runs can be made.

At 50,000 atmospheres and 1000°C, a Carboloy chamber shows its first fine crack after about 40 runs. It maintains pressure for approximately 35 additional runs. Enough runs have not been made with Carboloy tapered pistons at this pressure to determine their lifetime. At 100 runs, they seem as good as new.

Considering other workers' experience (10 to 20 runs) with equipment operating at these pressures and only at room temperature,(7) the "belt's" lifetime is excellent. At this writing, experience at 80,000 atmospheres in the "belt" is limited. Runs have been made at 80,000 atmospheres with temperatures to about 2400°C (temperature based on a linear extrapolation of the graph of Fig. 10). The lifetime under these conditions is materially shortened.

Mode of Cracking in Conical Pistons and Chamber

Cracks first appear on the surface of the cone of the steel pistons lying in a plane through the centerline. Later, the crack may spiral to the base of the cone, ending in a crack lying in a plane perpendicular to the centerline.

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(7) Reference 3, p. 396.

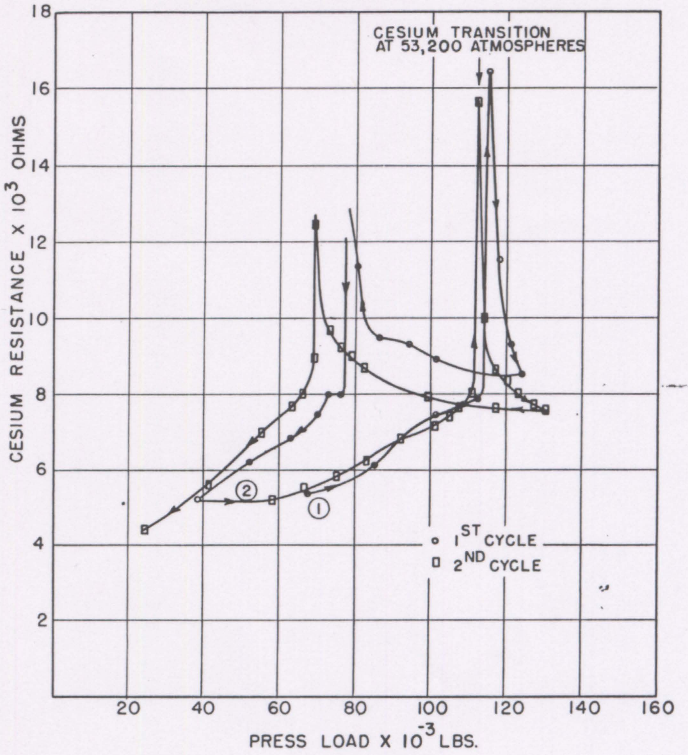


Fig. 15 Cesium transition at 53, 200 atmospheres.

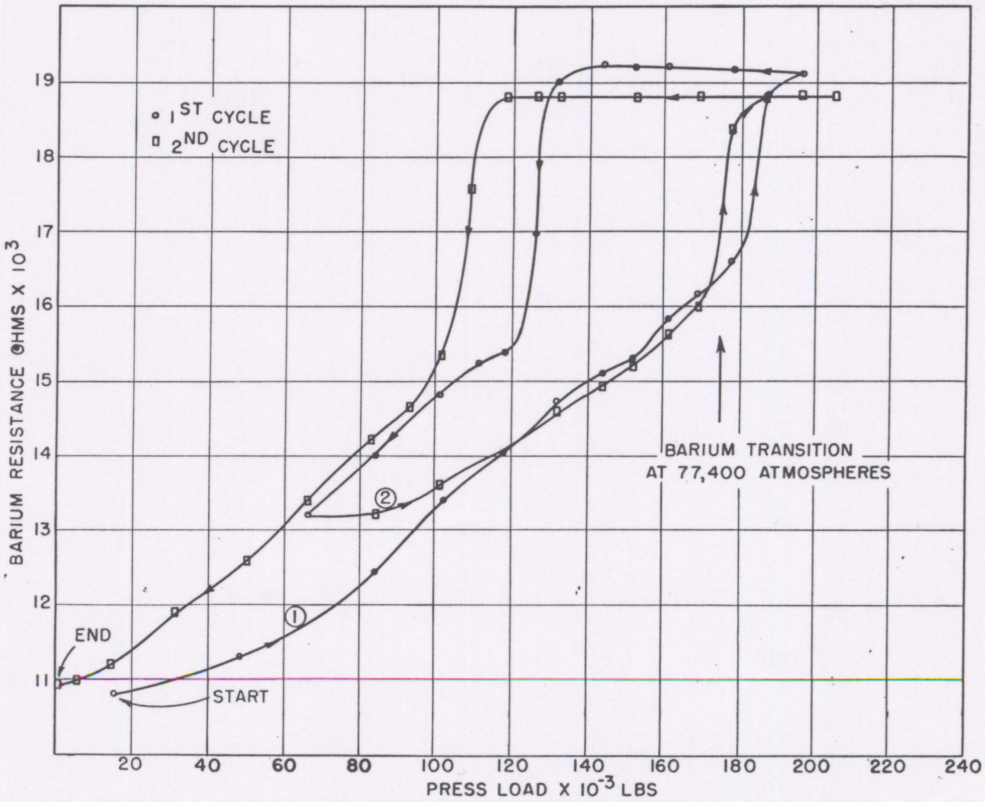


Fig. 16 Barium transition at 77, 400 atmospheres.

The chamber (of steel or Carboloy) cracks first on the inside surface in lines lying in planes through the centerline of the system. Later, a single crack perpendicular to these appears. This crack eventually splits the ring in two.

Construction Details

Figures 17 through 21 are self explanatory and give the detailed dimensions of the major components going into the "belt." The binding rings were constructed of AISI 4142 alloy steel of the following composition: C, 0.4 to 0.5; Mn, 0.75 to 1.00; P, 0.040 max; S, 0.040 max; Si, 0.2 to 0.35; Cr, 0.8 to 1.10; Mo, 0.15 to 0.25. The pistons and chamber were constructed of Carboloy, Grade 44A, or of tool steel of the following composition: C, 0.95 to 1.05; Mn, 0.3 to 0.8; P, 0.025 max; S, 0.025 max; Si, 0.15 to 0.40; Cr, 5 to 5.5; Mo, 0.95 to 1.25; V, 0.5.

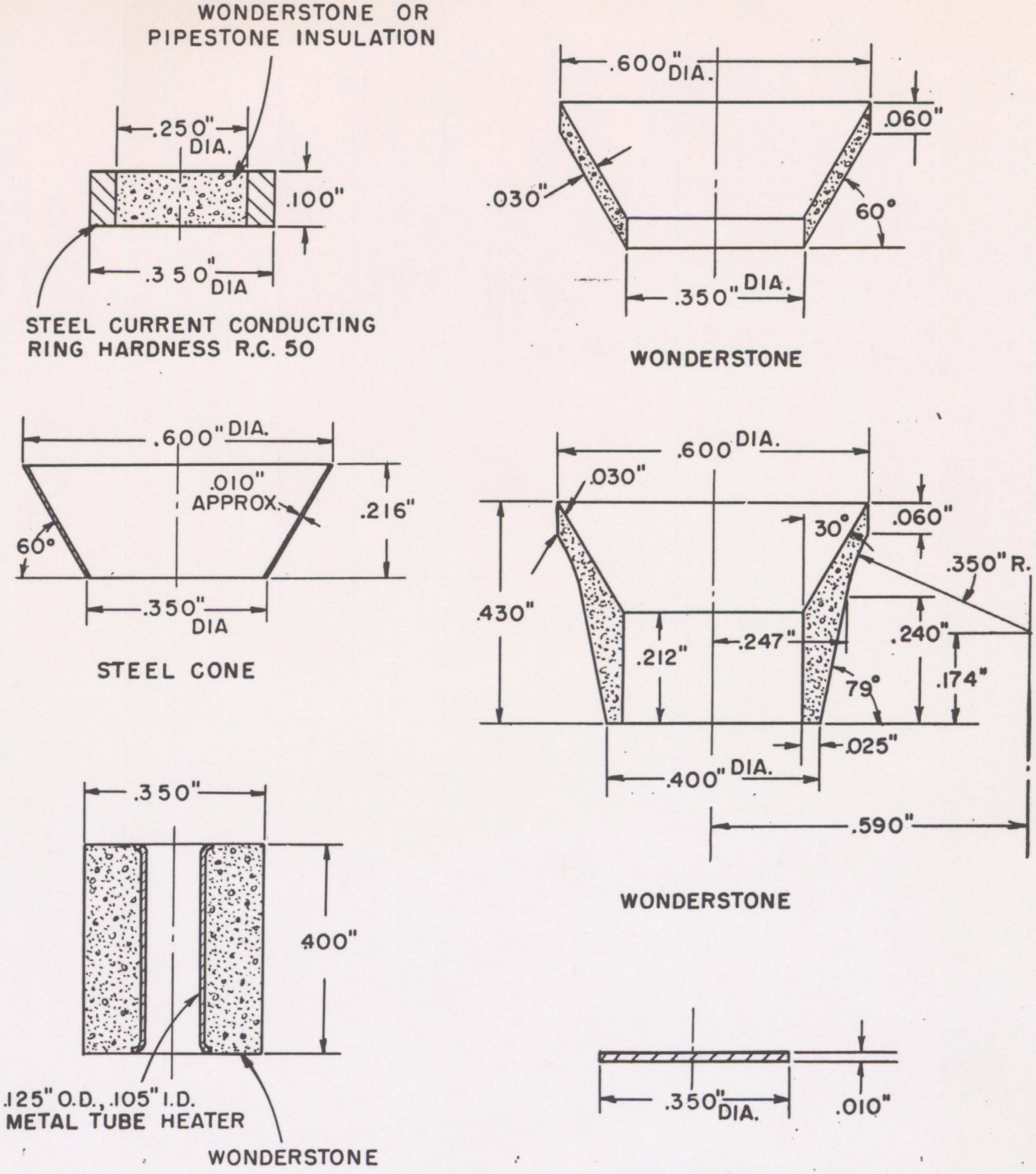


Fig. 17 Detail of chamber parts.

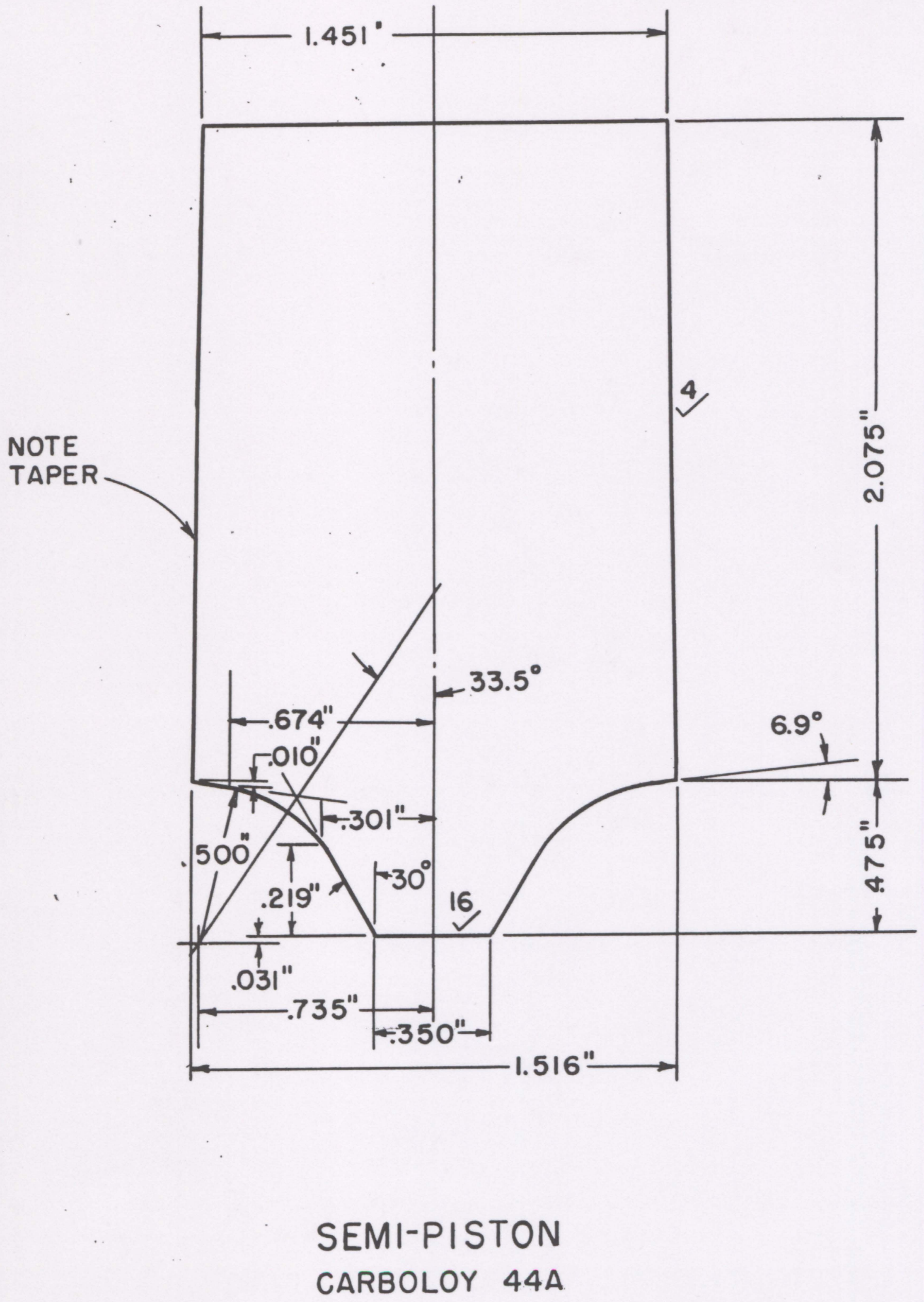


Fig. 18 Detail of conical piston (sometimes called a semi-piston or tapered piston).

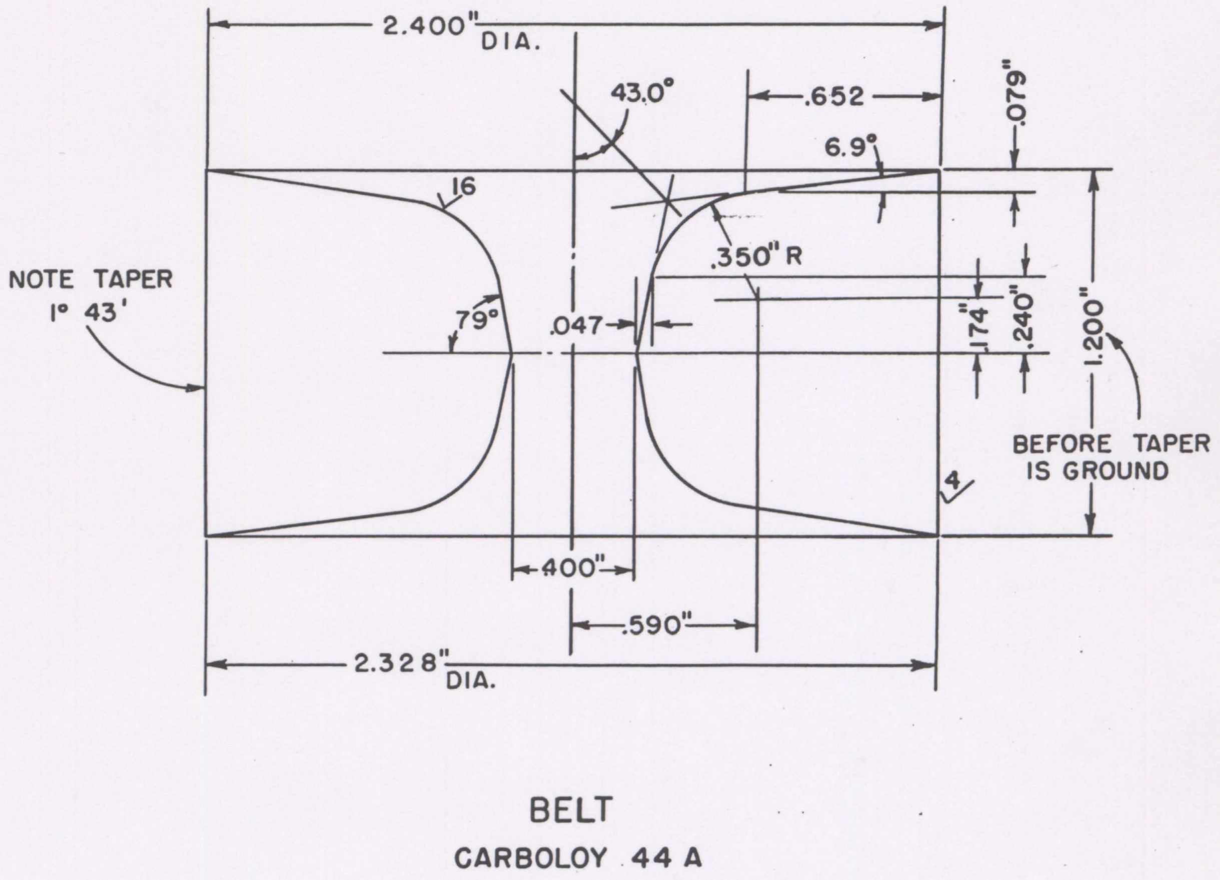
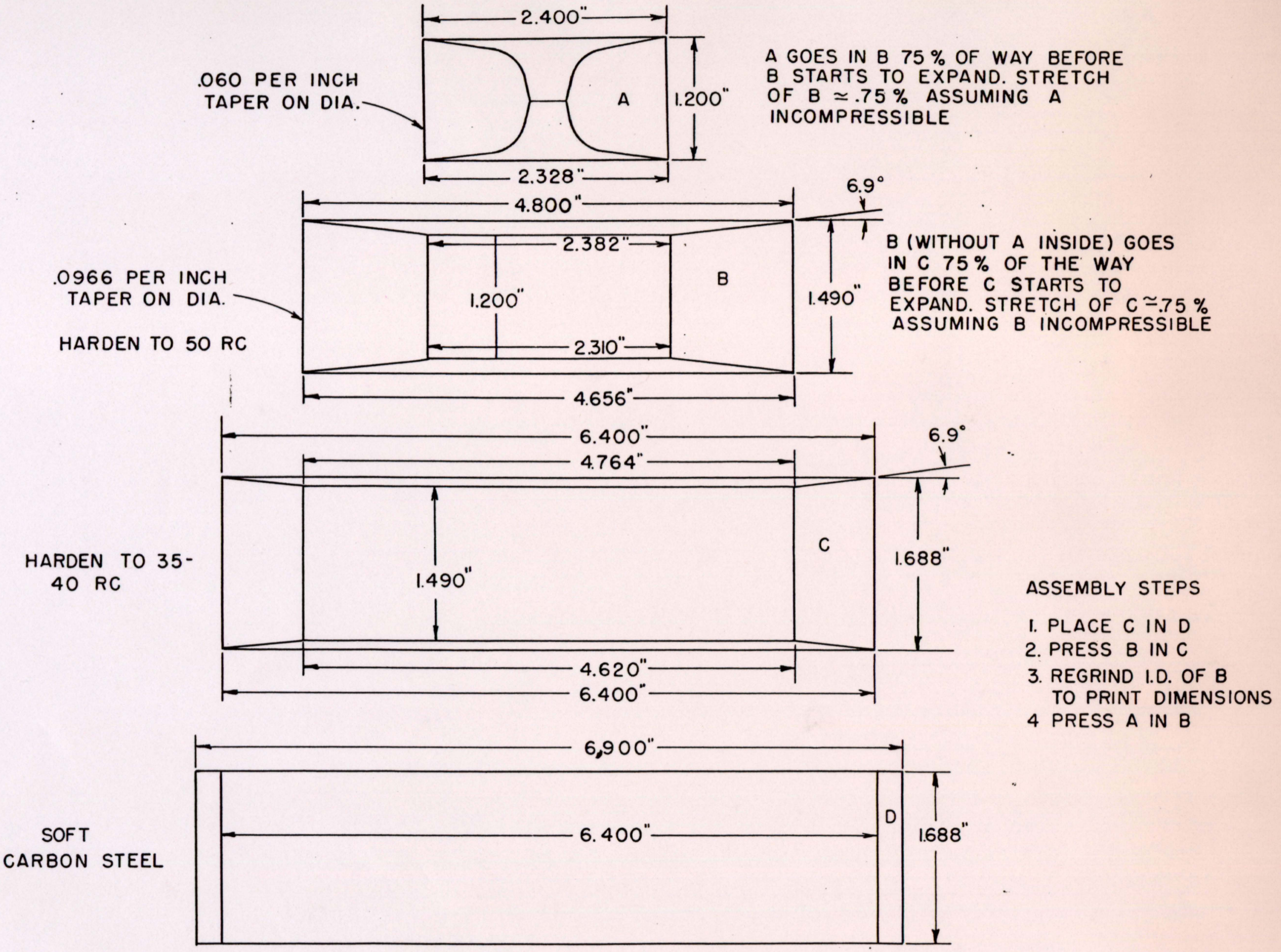


Fig. 19 Detail of innermost (chamber) ring of "belt" assembly.



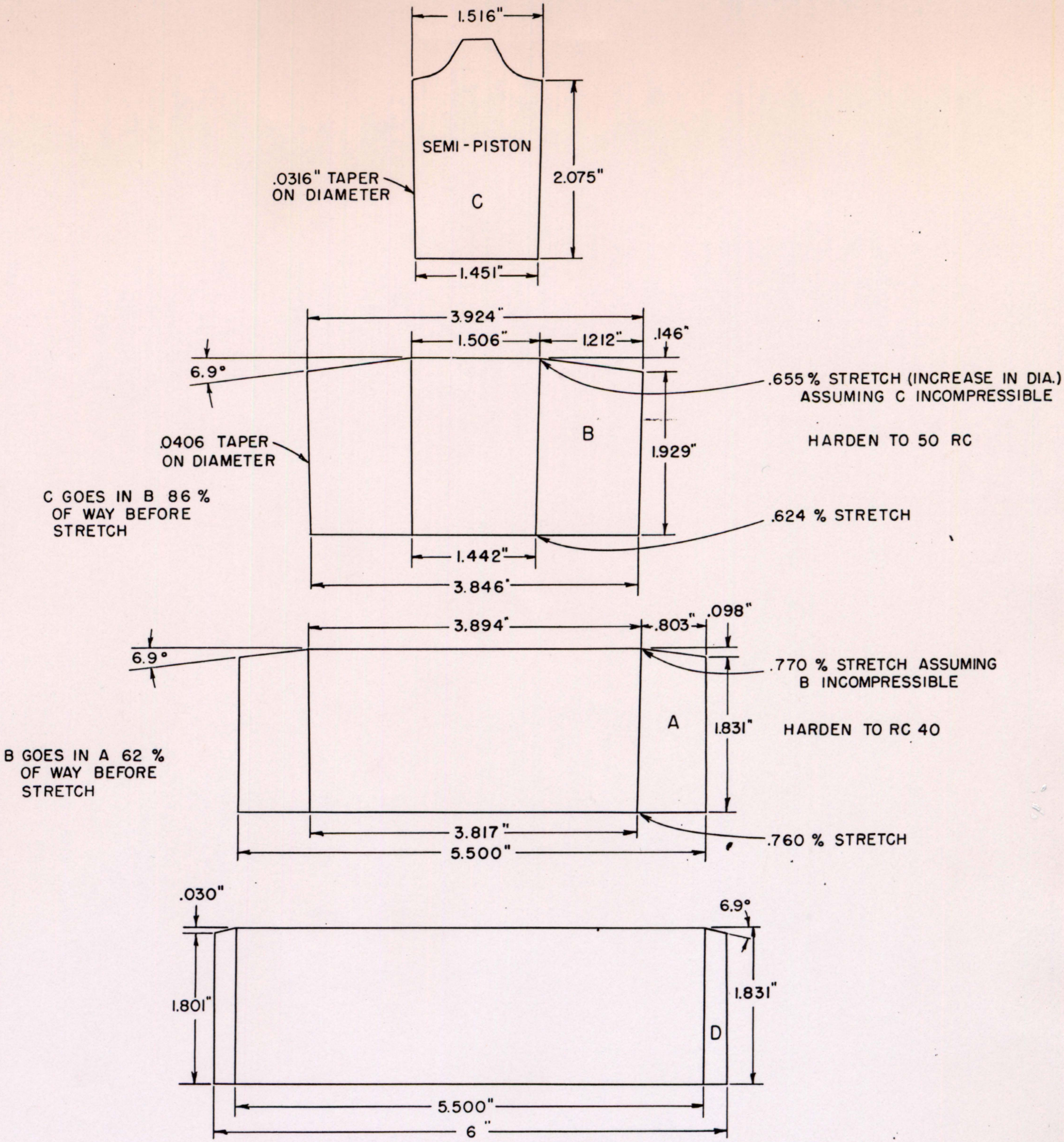
A GOES IN B 75 % OF WAY BEFORE B STARTS TO EXPAND. STRETCH OF B ≃ .75 % ASSUMING A INCOMPRESSIBLE

B (WITHOUT A INSIDE) GOES IN C 75 % OF THE WAY BEFORE C STARTS TO EXPAND. STRETCH OF C ≃ .75 % ASSUMING B INCOMPRESSIBLE

ASSEMBLY STEPS

1. PLACE C IN D
2. PRESS B IN C
3. REGRIND I.D. OF B TO PRINT DIMENSIONS
4. PRESS A IN B

Fig. 20 Binding ring detail of "belt."



ASSEMBLY STEPS

1. PLACE A IN D
2. PLACE B IN A
3. REGRIND INSIDE DIAMETER OF B TO PRINT DIMENSIONS
4. PRESS C IN B

Fig. 21 Binding ring detail for conical pistons.

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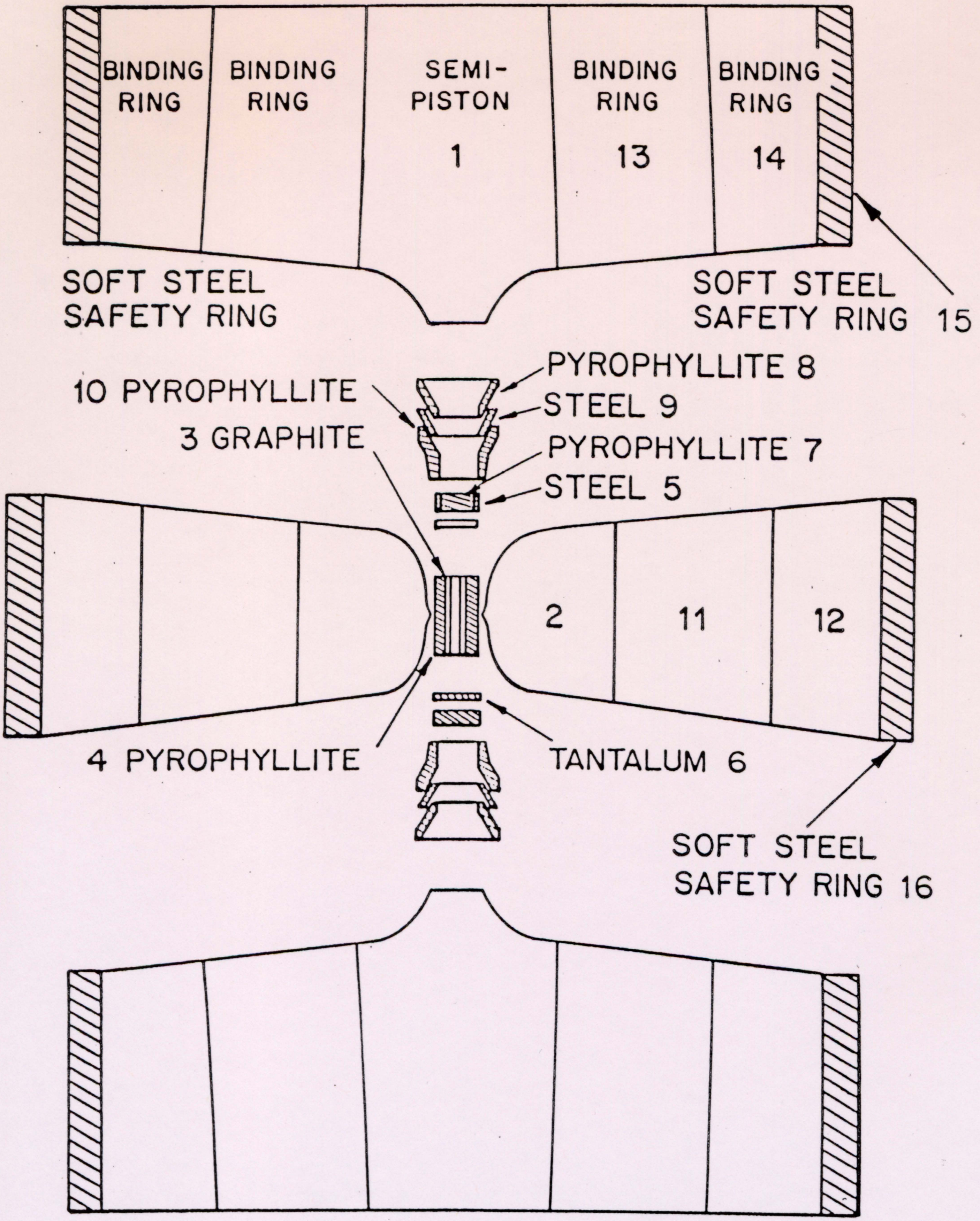

Fig.1 "Exploded" view of the Belt High temperature, High pressure apparatus.

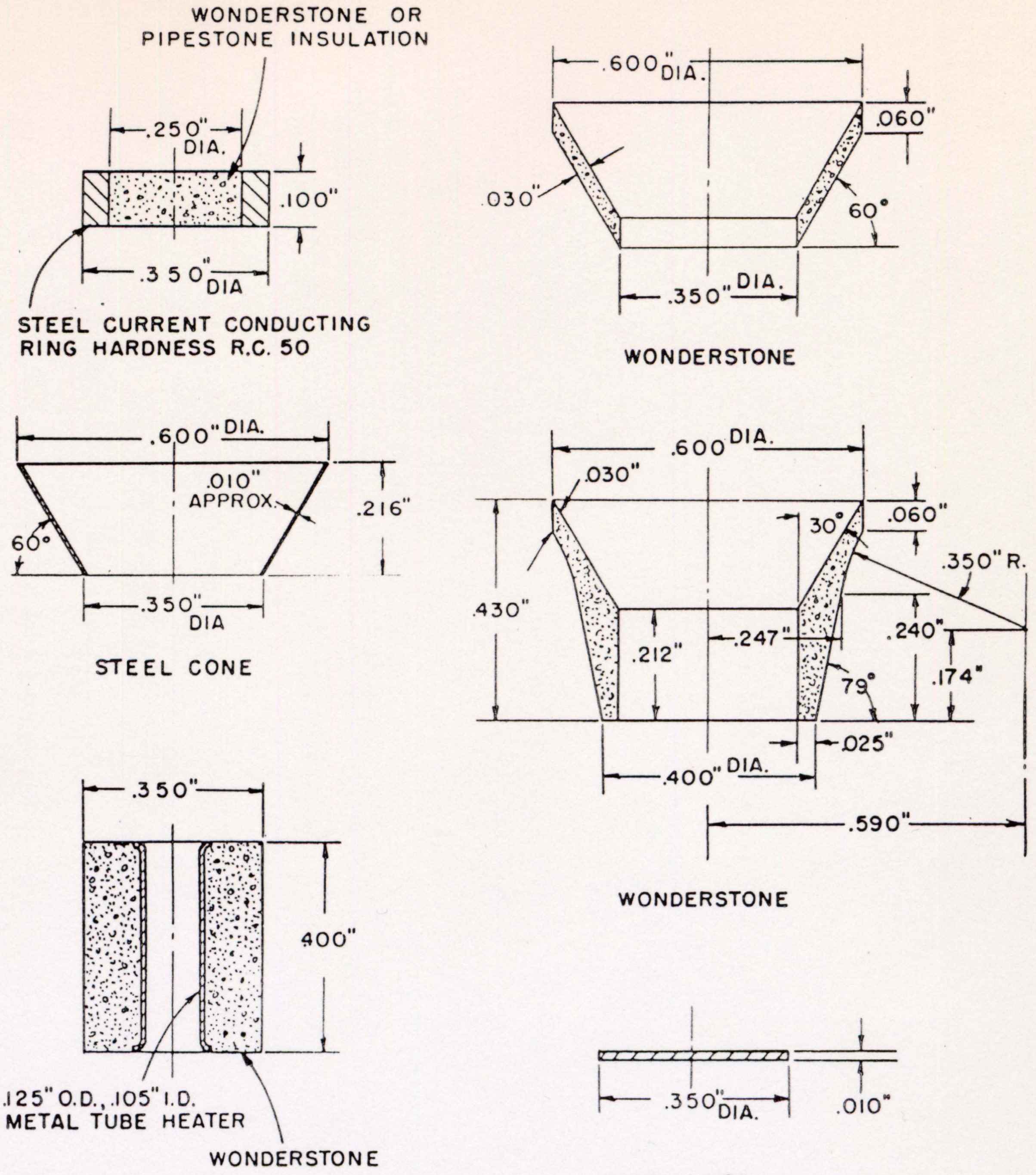

Fig. 8 Details of the parts that fit together within the carbide chamber.

BELT

U.S. PATENT NO.

2,941,248