Oct. 4, 1949. P. W. BRIDGMAN ET AL 2,483,803

HIGH-PRESSURE AND HIGH-TEMPERATURE TEST APPARATUS

Filed Nov. 22, 1946

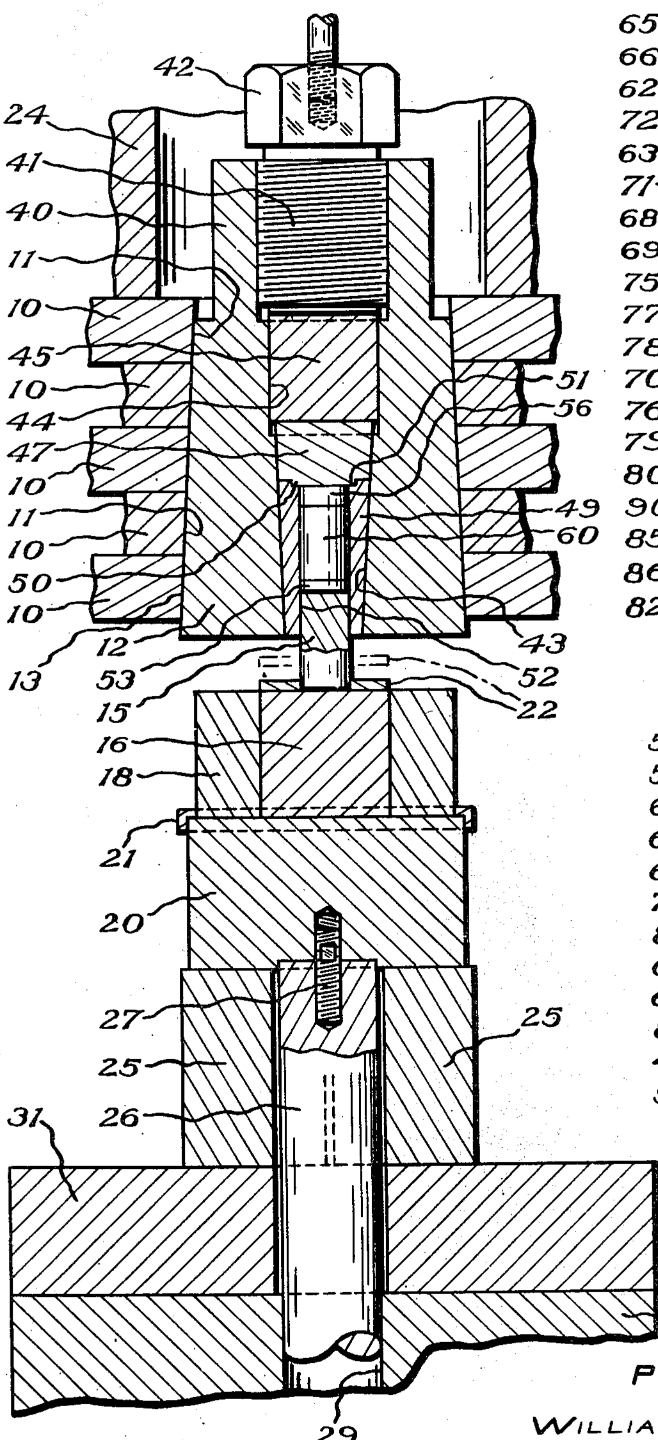

FIG. 1

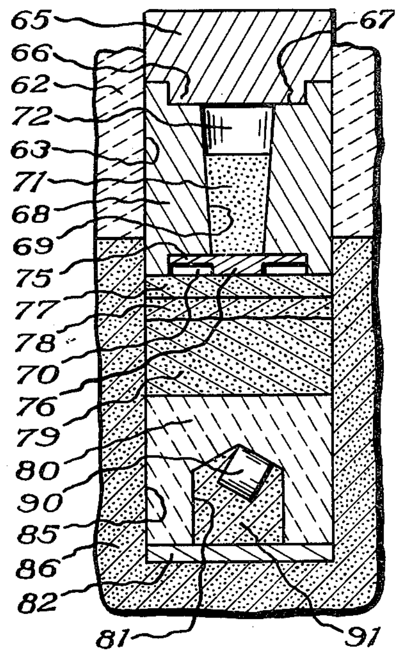

FIG. 3

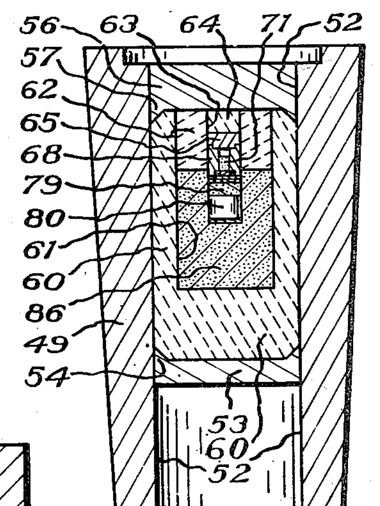

FIG. 2

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Patented Oct. 4, 1949 2,483,803

UNITED STATES PATENT OFFICE

2,483,803

HIGH-PRESSURE AND HIGH-TEMPERATURE TEST APPARATUS

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Application November 22, 1946, Serial No. 711,572

6 Claims. (Cl. 73—15.4)

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The invention relates to methods and apparatus for heating and pressing.

One object of the invention is to provide a method for subjecting material to a great pressure while heated. Another object of the invention is to provide an apparatus for heating and pressing simultaneously. Another object is to perform a series of chemical reactions producing intense heat in a closed chamber under great pressure. Another object is to provide an automatic detonating apparatus to detonate a charge responsive to subjecting the apparatus to great pressure. Another object is to provide a charge which will react to produce high temperatures under pressure of the order of many thousands of atmospheres. Another object is rapidly to produce high temperature under great pressure. Another object is to provide reaction charges and a mechanism for starting the reactions under enormous pressure without the use of any external connections so that the reactions can take place in a closed mold. Another object of the invention is to provide a satisfactory detonator to set off a charge under high pressure. Another object of the invention is to provide a satisfactory igniter charge or starter charge which will be ignited by the detonator and will set off the main charge under high pressure. Another object of the invention is to provide a main charge which will be set off by the igniter charge under conditions of great pressure. Another object is to provide a detonator and charges which will operate under great pressures and in which the charges will react substantially to completion under these pressures. Another object is to provide an apparatus and method for geophysical research. Other objects will be in part obvious or in part pointed out hereinafter.

The invention accordingly consists in the features of construction, combinations of elements, arrangements of parts, and in the several steps and relation and order of each of said steps, to one or more of the others thereof, all as will be illustratively described herein, and the scope of the application of which will be indicated in the following claims.

In the accompanying drawings illustrating one of many possible embodiments of the mechanical features of this invention,

Figure 1 is a cross sectional view taken along the axis of a mold constructed in accordance with the invention and showing the retaining plates of a press which support the mold.

Figure 2 is another cross sectional view of the mold of Figure 1 on a larger scale.

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Figure 3 is a third axial sectional view of the interior of the mold on a further enlarged scale to show the construction in better detail.

The apparatus of the invention is used in an hydraulic press. The press should be capable of exerting a thrust of five hundred tons. Referring to Figure 1 the press, not shown, holds a plurality of superimposed steel plates 10 having a continuous conical bore 11 extending therethrough, whereby the plates encircle and reinforce a central cavity block 12 which constitutes a mold. The thrust against the mold 12 is upward and as will be seen the mold 12 has a conical surface 13 which fits the conical bore 11, the lower end of the mold 12 and of the bore 11 being of larger diameter than the upper end of the mold and the bore. Thus upward pressure on the mold 12 tends to drive it into the bore 11 so that the reaction of the plates 10 exerts a compressive force radially inward of all parts of the mold 12.

We provide a specimen piston or ram 15 of small diameter, which exerts the pressure in the mold 12. This piston or ram 15 may rest upon a block 16 of some hard and strong material such as cemented tungsten carbide. An annular piece of steel 18 constitutes a retainer for the block 16 and prevents it from splitting. The retainer 18 is centered on the top of a ram head 20 by means of a centering ring 21. We may also provide a centering ring 22 for visually centering the piston or ram 15 on the block 16.

As shown, the ram head 20 is supported by a pair of semi-annular steel blocks 25 surrounding an auxiliary ram piston 26 to which the head 20 is removably secured by means of a bolt 27. The auxiliary ram 26 is movable in a cylinder 29 in the main hydraulic ram 30. Upon the top of the main hydraulic ram 30 is a removable ram head 31 having a hole therethrough to receive the auxiliary ram 26.

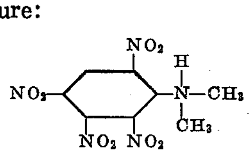
The foregoing construction is not per se our invention and may be varied widely. The use of the auxiliary ram 26 and the semi-annular blocks 25, which are removable, makes it possible to move the specimen piston or ram 15 through a comparatively long distance at low pressure and under accurate control, and also rapidly. In other words, this is an expedient for readily setting up the apparatus for operation at high pressures and for quickly lowering the piston or ram 15 and clearing a space below it for removing the mold 12. However, so far as the main features of this invention are concerned, any device or apparatus for exerting a powerful thrust upon a small piston or ram, such as the piston or ram 15, may be employed.

Still referring to Fig. 1, the cavity block or mold 12 is a massive piece of steel having a cylindrical extension 40 above the portion with the conical surface 13, which extension 40 has massive screw threads on the inside to receive a large screw threaded plug 41 with a hexagonal head 42. The cavity block or mold 12 has a conical bore 43, the small end of which is at the under end of the mold 12, and the large end of which opens into a cylindrical chamber 44 in which is located a cylindrical steel plug 45 abutting the screw plug 41.

We provide a tapered anvil member 47 which fits the upper part of the conical bore 43 and extends part way into the cylindrical cavity 44. We further provide a tapered mold liner 49, which fits the conical bore 43 just below the anvil member 47. The anvil member 47 preferably has a cylindrical projection 50 on the bottom thereof, which fits a counterbore 51 at the top of the liner 49, thus to provide a good seal between these parts. The anvil member 47 and the mold liner 49 may be made of steel, such as a good grade of tool steel. The liner 49 is in a real sense itself a mold.

Referring to Figures 1 and 2, the liner 49 has a cylindrical bore 52 extending through it from end to end. The ram or piston 15 fits in this bore 52, and on the upper end of the ram or piston 15, when it is in the bore 52, is a soft steel sealing plate 53 having on its upper side an interiorly tapered annular projection 54. Similarly, under the cylindrical projection 50 of the anvil member 47 is a soft steel sealing plate 56 having an interiorly tapered annular projection 57. The heating charge, the insulation and the specimen are placed in the chamber formed by the bore 52 and the sealing plates 53 and 56, and are therein subjected to pressure. The pressure is transmitted from the ram 30 via the head 31, the removable blocks 25, the head 20, the block 16 and the piston 15 to the sealing plate 53, then through the charge, insulation and specimen to the sealing plate 56, thence to the anvil member 47, the plug 45, the screw plug 41 to the cavity block 12, which transmits the thrust to the plates 10 which are held down by a massive iron cap 24 which is a permanent part of the hydraulic press.

In accordance with the invention we provide a suitable heating charge, which will heat the specimen while it is under pressure, and we provide suitable means to ignite this charge. Desirably, we provide a detonator, an igniter and a main charge. We will describe the best combination now known to use, but it should be understood that other chemicals may be employed so far as the broad aspects of the invention are concerned. A suitable detonator is tetracene. This is understood to have the following structure:



This detonator reacts upon impact and produces an intense heat which ignites the igniter. As will hereinafter be described, we provide means to set off the detonator upon the attainment of a certain pressure.

The igniter which we have successfully used is a combination of two mixtures. The mixture nearest the detonator was sugar and potassium chlorate, $KClO_3$, the sugar was sucrose. The sugar and the potassium chlorate were mixed in stoichiometric proportions according to the reaction,

$$8KClO_3+C_{12}H_{22}O_{11}=11H_2O+12CO_2+8KCl$$

The second igniter mixture which was placed next to the first igniter mixture was magnesium and sodium peroxide. These were mixed in stoichiometric proportions according to the reaction

$$Mg+Na_2O_2=MgO+Na_2O$$

The proportion of the igniter mixtures was .080 part of the first and .350 part of the second. The main charge reacted according to the following formulae:

(1) $Mg+Na_2O_2=MgO+Na_2O$

(2) $3Mg+KClO_3=3MgO+KCl$

However, stoichiometric proportions were not used, rather, the following proportions were found to give good results:

	Per cent
Na_2O_2	19.1
$KClO_3$	42.3
Mg	38.6

The above mixture could be used, but produced a temperature higher than was desired. To obtain a calculated temperature of about 3660° K., it was found that 15.25 parts of the Na_2O_2, $KClO_3$, Mg mixture should be diluted with 9.75 parts of MgO. This mixture was successfully used for the main charge and fused the molybdenum disc 82. It is quite practical to use more than 39% of MgO, or less than 39% of MgO to obtain various other temperatures, it being understood that the addition of MgO lowers the temperature.

Referring now to Figures 2 and 3, extending between the sealing plates 53 and 56 was a cylindrical graphite casing 60 having a cylindrical chamber 61 opening to the upper side, the casing being bevelled at bottom and top to fit the annular projections 54 and 57. This graphite casing 60 was of course crushable under the high pressure employed, but would stay more or less in position and constituted refractory insulation. Inside and at the upper end of the graphite casing 60 was a graphite plug 62 having an axial bore 63. Plugging the upper end of axial bore 63 was a graphite plug 64. Under the graphite plug 64 was a steel plug 65 fitting the bore 63, the steel plug 65 having a cylindrical projection 66 fitting a counterbore 67 in a steel container 68, also fitting the bore 63. This steel container 68 had a tapered axial bore 69, the large end being up, and the small end opening into a wide cylindrical chamber 70 at the bottom of the container 68.

The tetracene in granular form is shown at 71 in the bore 69, not quite filling the bore, but leaving an air space 72 above it. The bottom of the bore 69 was closed by a hardened steel disc 75 of a diameter the same as that of the chamber 70, but of lesser thickness except for the central portion where an integral projection or button 76 fills the chamber 70. Under the container 68 and under the button 76 was a layer of powdered graphite 77 having the shape of a disc. Below the graphite layer 77 was the first igniter mixture 78, also in the form of a disc. Under the first igniter mixture

78 was the second igniter mixture 79, more or less in the form of a cylinder. The mixtures 78 and 79 were loose mixtures and the boundaries were not exact, and some intermixture occurred.

Under the second igniter mixture 79 was a graphite container 80 having an axial bore 81 opening at the bottom of the container 80. Under the container 80 and blocking the bore 81 was a molybdenum disc 82. The lower part of the steel container 68 plus the graphite 77 and the mixture 78, the mixture 79 and the graphite container 80 and the molybdenum disc 82, were all contained in a straight cylindrical bore 85 in the main charge 86, which filled the bore 61 under the plug 62, all as clearly shown in the drawings, Figures 2 and 3. The main charge was molded and pressed to the shape shown, and had enough strength to hold together while being filled with the materials and parts described. In the bore 81 the specimen 90 was surrounded by powdered graphite 91

The purpose of the apparatus is to subject the specimen 90 to great heat and pressure simultaneously. Once the parts and mixtures are assembled as described, the pressure is applied whenever the main ram 30 exerts the desired thrust, which is accomplished by manipulation of the controls of the press, not shown. As soon as the pressure reaches a certain value, the charges are ignited and the specimen 90 is heated. This occurs in the following manner.

The pressure between the sealing plates 53 and 56 compresses everything between them, but the pressure on the button 76 is unsupported on the upper side due to the space 72. Finally, therefore, the disc 75 shears and the button 76 shoots like a bullet into the tetracene detonator 71. This sets off the detonator, which flashes back through the graphite 77 and into the first igniter charge 78. This charge 78 ignites and heats the main charge 86 and also sets off the second igniter charge 79. The two charges 78 and 79 together set off the main charge 86, which provides intense heat to the required temperature. This temperature is maintained for a considerable time, due to the fact that it is developed entirely inside of an envelope of graphite. For that reason it does not melt the steel parts. Gradually, of course, the heat is dissipated, but the pressure can be maintained from the moment the tetracene is ignited right through the heating cycle, and in fact for an indefinite period. In practice the pressure has been maintained for usually about 5 minutes, sometimes longer.

The detonator may be other than tetracene, the igniter may be different and the main charge may be different. For example, the detonator might be mercury fulminate, or sodium azide NaN_3 or tetra nitro guanidine. Many nitrated organic compounds might be used. A detonator should be sensitive and it should produce a powerful shock. We consider that picric acid is the borderline case with regard to sensitivity and our detonator should be at least as readily detonated as picric acid. Explosive chemists can measure the sensitivity of detonators.

With regard to the shock producing properties we define our detonator as one which produces gas. By gas we mean anything which is a gas at just above 100° C. or at 100° C. and somewhat less than 30″ of mercury. Water is a gas under these conditions.

In some cases the igniter charge might be omitted and the main charge be fired by the detonator. However for the most reliable results we prefer to have an igniter and it will be noted that in the illustrative embodiment of the invention a two stage igniter was provided.

It will be seen that the main charge comprises, and in fact in the illustrative embodiment mainly consists of, an elemental metal and a substance containing combined oxygen. In the illustrative embodiment magnesium is the element and sodium peroxide and potassium chlorate are the compounds containing oxygen. The elements magnesium, aluminum, lithium, calcium, strontium and barium have oxides which have a high heat of formation. The elemental metal which may be referred to as an element should be selected from this group. The oxygen containing compound should be an oxidizing agent and may be defined as a compound or compounds whose oxygen is as easily detached as the oxygen of iron oxide FeO. A mixture of aluminum and iron oxide when ignited gives off intense heat and is known as "Thermite." It will be noticed that in the illustrative embodiment the reaction of the main charge does not produce gas as above defined. It will be noticed on the other hand that one of the igniter mixtures reacts to produce gas. This is the mixture of potassium chlorate and sugar. The other or second igniter mixture is seen to be identical with one part of the main charge and from a chemical standpoint may be considered as part thereof.

It has been pointed out that the plates 10 act as a reinforcement of the cavity block 12 due to the tapered outer surface of the latter and the thrust against it from the large end of the taper. The reverse taper of the bore of the cavity block permits the mold liner 49 and anvil member 47 to be readily withdrawn from the cavity block 12 even after the ram 15 has exerted an enormous thrust equivalent to 30,000 atmospheres or more. We have found that a large screw plug 41 of the type shown and described can be tightened sufficiently to hold the mold parts in place under these conditions of high pressure. One feature which permits the cavity block 12 to withstand the high tensile force is the comparatively long dimension of the cavity block from the anvil member 47 to the plug 41.

The sealing plates 53 and 56 made of soft steel effectively confine the charges and the specimen between them since the annular projections 54 and 57 try to expand under the pressure. The taper of the bore 69 allows the button 76 to move like a bullet in the bore once the disc 75 has sheared off. This insures detonation. The molybdenum disc 82 was provided as a check on the temperature reached and in many of the experiments the molybdenum melted partially. Molybdenum has a melting point of 2620° C. at ordinary pressures and we believe that the melting point under these high pressures is not a great deal higher.

It will be seen from Figure 2 that the various charges and the specimen are all enclosed in graphite so that the heat persists for a reasonably long time. Probably if the temperature at the time of reaction is 2700° C. it does not drop below 2000° C. for a minute.

The apparatus and method of the invention can be used to study polymorphism and for experiments in geophysical research. It can also be used to change the state of matter where that can be done with heat and pressure of the order indicated.

It will thus be seen that there has been provided by this invention a method and an apparatus in which the various objects hereinabove set forth together with many thoroughly practical advantages are successfully achieved. As many possible embodiments may be made of the above invention and as many changes might be made in the embodiment above set forth, it is to be understood that all matter hereinbefore set forth or shown in the accompanying drawings is to be interpreted as illustrative and not in a limiting sense.

We claim:

1. A mold for high pressure high temperature attainment comprising a cavity block with an exterior conical surface and a conical bore, the taper of the surface being opposite to that of the bore, means to plug the bore at the large end, a liner fitting the bore and having a cylindrical bore, a pair of sealing plates in said cylindrical bore, charges including a detonator and a main charge in said cylindrical bore between said sealing plates, and a shearable element adjacent the detonator which will fire it when great pressure is exerted between said sealing plates.

2. Apparatus of the class described comprising a cavity block having an exterior conical surface and a conical bore, the taper of the surface being opposite to that of the bore, there being a cylindrical bore in the cavity block beyond the large end of the conical bore, a cylindrical plug in said cylindrical bore, there being a cylindrical threaded bore in said cavity block beyond said cylindrical bore, a screw plug in said cylindrical threaded bore, a tapered mold liner in said conical bore, a ram fitted to said tapered mold liner in a cylindrical bore thereof, and an exothermic charge in the liner, whereby simultaneously to produce high heat and pressure when said charge is ignited and said ram exerts pressure.

3. In apparatus claimed in claim 2, the combination with the parts and features therein specified, of a detonating charge in the cavity block and a shearable element to set off the detonating charge responsive to pressure exerted by the ram.

4. In apparatus of the class described, a mold liner having a cylindrical bore, a pair of sealing plates in said bore, and in said bore between said sealing plates the following: a detonating charge, a shearable element adjacent the detonating charge and so situated that when it is sheared by pressure part of it will hit the detonating charge, and an exothermic charge, whereby pressure in said bore between the sealing plates shears the element which sets off the detonating charge which sets off the exothermic charge thus heating a specimen which may have been placed between the sealing plates.

5. In apparatus for coincidentally heating a specimen and subjecting it to high pressure, a mold having a bore, means blocking one end of the bore, a piston ram fitting the bore, a mass of refractory material in the bore said mass having a cavity, and in the cavity the following: a shearable element, a detonating charge adjacent the shearable element in such position that when the element is sheared part of it hits the detonating charge, an igniter charge adjacent the detonating charge, and a main exothermic charge adjacent the igniter charge, whereby when the piston ram exerts high pressure in the bore of the mold the element is sheared, the detonating charge is set off, the igniter charge is set off and the exothermic charge creates intense heat coincidentally with the pressure while the refractory material retards cooling.

6. In apparatus for coincidentally heating a specimen and subjecting it to high pressure, a mold having a bore, means blocking one end of the bore, a piston ram fitting the bore, and in the bore the following: a shearable element, a detonating charge adjacent the shearable element in such position that when the element is sheared part of it hits the detonating charge, an igniter charge adjacent the detonating charge, and a main exothermic charge adjacent the igniter charge, whereby when the piston ram exerts high pressure in the bore of the mold the element is sheared, the detonating charge is set off, the igniter charge is set off and the exothermic charge creates intense heat coincidentally with the pressure.

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REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
117,249	Bessemer	July 25, 1871
315,741	Henderson	Apr. 14, 1885
578,868	Goldschmidt	Mar. 16, 1897
1,277,311	Hansen	Aug. 27, 1918
1,311,740	Asbury	July 29, 1919
1,609,970	Schroeder	Dec. 7, 1926
1,801,449	Olsen	Apr. 21, 1931
2,375,032	Parke	May 1, 1945
2,398,287	Christie	Apr. 9, 1946
2,412,636	Short	Dec. 17, 1946

FOREIGN PATENTS

Number	Country	Date
523,064	Germany	Apr. 18, 1931
583,505	Germany	Sept. 5, 1933

OTHER REFERENCES

Jones: "Inorganic Chemistry," Blakiston Co., 1947, p. 259.