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HIGH PRESSURE HIGH TEMPERATURE APPARATUS

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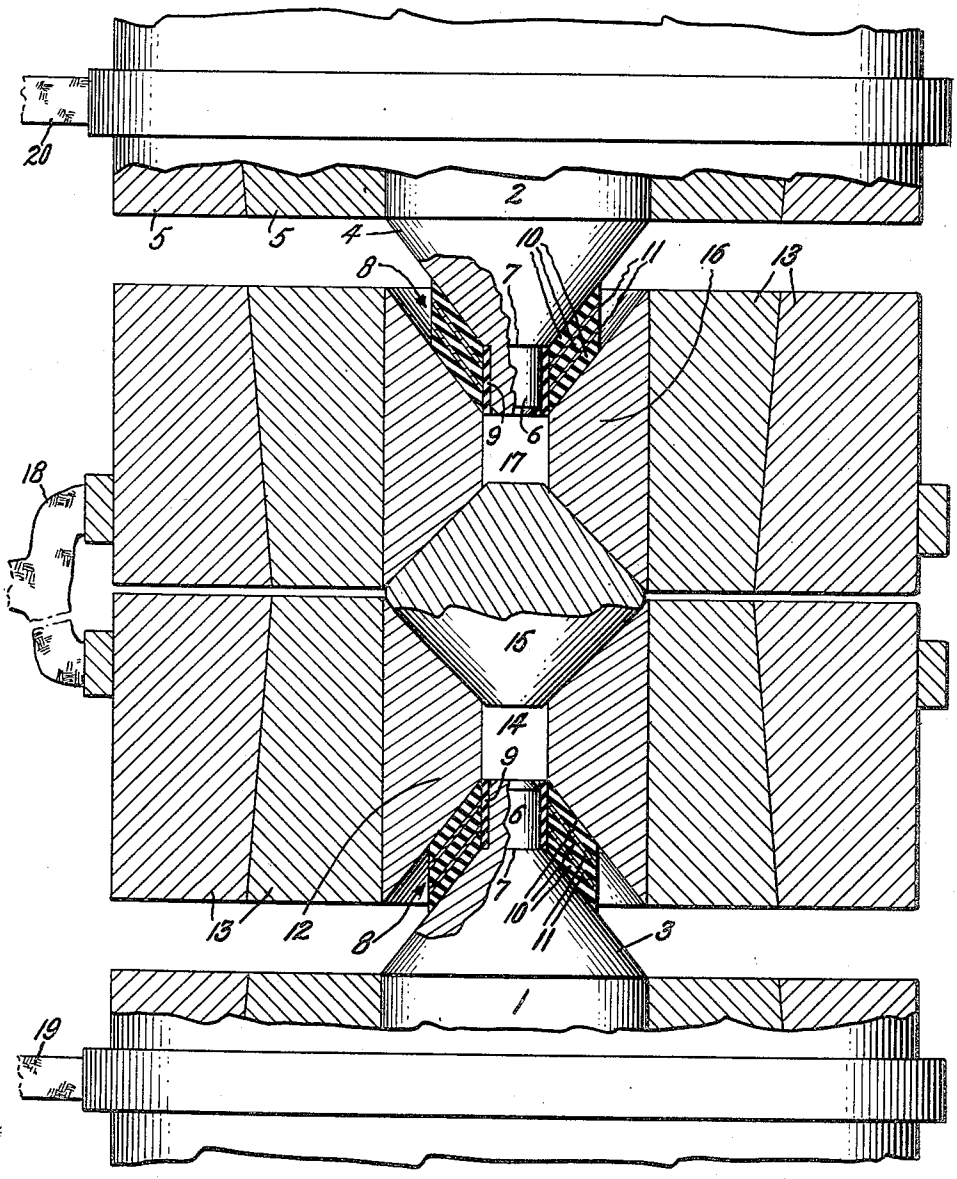

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HIGH PRESSURE HIGH TEMPERATURE APPARATUS

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This invention relates to high pressure high temperature apparatus and, more particularly, to a high pressure high temperature apparatus employing reaction cells in series.

High pressure high temperature apparatus has been heretofore employed to study the reactions of various materials when subjected to high pressures and high temperatures, or to produce physical or chemical changes of materials which affect their inherent characteristics. One change in one particular field which may be included among the innumerable examples is that of graphite to diamond where the changes of density and other characteristics are well known to those skilled in the art.

In general, prior high temperature or high pressure apparatus utilized a single or double acting cell, the single acting cell being limited in length due to the frictional force created between the specimen in the cell and the walls of the vessel by the imposed compression. This frictional force reduces the pressure in the closed end of a single acting cell and results not only in a lower pressure in the closed end but also a pressure less than desirable to perform the required transformations. Alternatively, a disproportional high pressure must be exerted by the punch or other compressing means at the upper end to raise the pressure at the lower or closed end to the desired degree. One method of overcoming this difference is to use a double acting reaction vessel upon which high pressure is exerted at both ends of the vessel. While the double acting vessel may be longer than the single acting vessel, it is subject to the same length problem, and requires additional gaskets for sealing purposes.

Accordingly, it is an object of this invention to increase the length of a single acting vessel by providing multiple chambers.

Another object of this invention is to facilitate inserting and withdrawing the specimen from a single acting vessel.

A further object of this invention is to combine single acting cells in series relationship.

In carrying out this invention in one form, a pair of rings or belts whose inner surfaces taper to present a frustroconical cross-sectional configuration are concentrically mounted between a pair of opposing punches. A relatively loose fitting block or plug having frustroconical projections diametrically opposed in base-to-base relationship in positioned between the rings to reside in the space defined by the two adjacent rings of frustroconical configuration. There is thus defined by the punches, the rings, and the block, a pair of single acting cells, each sharing the same base. These cells are easily filled and emptied by means of the removable block.

Further objects, features, and advantages of this invention will be apparent from the following description and drawings, and its scope will be pointed out in the appended claims.

The figure illustrates a preferred form of this invention in the showing of a pair of single acting cells in series relationship.

Referring now to the figure, a suitable high pressure apparatus in the form of a press having a pair of opposed cylindrical punches **1** and **2** with frustroconical or tapered surfaces **3** and **4** formed on their opposed surfaces. Either or both of the punches **1** or **2** may be made movable toward the other to compress or apply pressure to a reaction vessel or specimen of a material therebetween. Since the pressure under consideration may approach 100,000 and more atmospheres, the punches are encircled by one or more strengthening rings **5** of very hard steel or other material to resist the high lateral forces developed by the high pressures involved.

Each tapered surface **3** and **4** has a cylindrical projection or nib **6** extending from the frustrum **7** of the frustroconical surfaces **3** and **4**. Mounted on the frustroconical surface **3** is a gasket assembly **8** which comprises a hollow cylindrical gasket **9** positioned around the nib **6** followed by a plurality of frustroconical gaskets **10** and **11** which are positioned on the frustroconical surface **3** of the punch **1** in stacked or interfitting relationship. Cylindrical gasket **9** and the frustroconical gaskets **10** are formed of thermally and electrically insulating material such as catlinite, pyrophyllite, or other such material of low compressibility and low spalling characteristics, and the gasket **11** is preferably of high strength material such as metal. While the specific number of gaskets **10** and **11** is not particularly critical, one preferred form of this invention discloses three insulating gaskets **10** spaced by two metal gaskets **11** therebetween. The gasket assembly **8** as described prevents undue failure of the assembly, first in that it maintains pressure within the chamber after considerable deformation, and, secondly, the spalling characteristics are such that the gasket remains in place with electrical insulating qualities.

Positioned concentrically with the punch **1** and adjacent the gasket assembly is a belt or ring **12**. Ring **12** is a high strength metal such as cemented tungsten carbide and is in turn encircled by one or more strengthening rings **13** in order to provide increased resistance to fracture from the high lateral forces imposed. The inner cross-sectional configuration of ring **12** is frustroconical and thus when positioned upon the gasket assembly **8** presents a tapered wall complementary to the tapered surface **3** of punch **1**, and separated therefrom by the gasket assembly **8**. The ring **12** and the punch **1** define a first chamber **14**, into which may be placed a reaction vessel of the type described and claimed in the copending application, Serial No. 448,050, Hall, and assigned to the same assignee as the present invention, for the purposes of the study of reactions of various materials under high pressure and/or high temperature conditions or to obtain chemical or physical changes which give added characteristics to existing materials. As heretofore stated, an example in one field, precious stones, is the transformation of graphite to diamond. Other changes and fields are too numerous to describe for the specific objects of this invention.

In order to completely enclose the chamber **14**, a block **15** of cemented tungsten carbide or other very hard material comprising a pair of frustroconical surfaces in base-to-base relationship is placed on the ring **12** such that one of the frustroconical surfaces of the block fits into the frustroconical space formed by the tapered surface of the ring **12**.

The invention is thereafter completed by a second ring **16** similar in all respects to ring **12**, encircled by additional strengthening rings **13**, and positioned concentrically between punch **2** and ring **12**. The ring **16** and block **15** define a second chamber **17** into which an additional reaction vessel as heretofore described may be

placed. A further gasket assembly 8 is placed on tapered surface 4 between punch 2 and the tapered surface of the ring 16.

It thus may be seen that the block performs a dual function of acting as an easily removable common base member for a pair of single acting chambers 14 and 17, while permitting a double end compression or, in one operation, compressing a combined chamber volume exceeding the heretofore single action or double action variety.

The material or specimen in the chambers 14 and 17 may be subjected to high temperatures by many well known means. In one form of this invention, resistance heating may be applied by conducting current from a conductor 18 through the strengthening rings 13 and rings 12 and 16 to the specimen or reaction vessel and out through the punches 1 and 2 by means of leads 19 and 20. For resistance heating, such a circuit is preferred over a circuit passing current through the chambers in series, since one cell may present a greater resistance than the other and thus absorb most of the current leaving the remaining cell at a reduced temperature.

While other modifications of this invention and variations of apparatus have not been described, the invention is intended to include all such as may be embraced within the following claims.

What I claim as new and desire to secure by Letters Patent of the United States is:

1. A double end single stage reaction apparatus comprising in combination, a pair of opposed punches, at least one of which is movable toward the other, a first ring positioned between and coaxially with one of said punches to define an open end chamber therewith into which the chamber defining punch may progress, a gasket between the said punch and said ring, a second ring positioned between and coaxially with said other punch to define a second open end chamber therewith into which said other punch may progress, a gasket between said other punch and said second ring, a loose fitting block positioned between and coaxial with said first and second rings to close said first and second chamber, and a binding ring on said punches and said rings.

2. A double end single stage reaction apparatus comprising in combination, a pair of opposed punches, at least one of which is movable toward the other, a first ring between and concentric with one of said punches to define an open chamber therewith, a gasket between said punch and said first ring, a second ring positioned between and concentric with said other punch to define a second open chamber therewith, a gasket between said other punch and said second ring, and a loose fitting block between and coaxial with said rings to close said chambers, means to conduct an electrical current to said chambers, and a binding ring mounted on each of said punches and said rings.

3. A double end single stage reaction apparatus comprising in combination, a pair of opposed tapered surface punches at least one of which is movable toward the other, a first ring having tapered inner surfaces positioned between and concentrically with one of said punches to define a chamber therewith, sealing means between the tapered surface of said punch and the tapered surface of said ring, a second ring having tapered inner surfaces positioned between and coaxially with the other of said punches to define a chamber therewith, sealing means between the tapered surface of said other punch and said second ring, a loose fitting block positioned coaxially with and between said rings with tapered surfaces thereof adjacent tapered surfaces of said rings to close each of said chambers, and a binding ring for said punches and said rings.

4. A double end single stage reaction apparatus comprising in combination, a pair of opposed punches having opposed frustroconical surfaces thereon, at least one of said punches being movable toward the other, at least one strengthening ring encircling said punches, a cylindrical nib projecting from the frustrum of each of said frustroconical surfaces, a first ring having an inner projecting frustroconical cross-sectional configuration positioned between and concentrically with one of said punches, sealing means between the frustroconical surface of said ring and the frustroconical surface of said punch, at least one strengthening ring about said first ring, a loose fitting block comprising a pair of projecting frustroconical surfaces in base to base relationship positioned with one of its frustroconical surfaces in the frustroconical space defined by said ring, a second ring having an inner projecting frustroconical cross-sectional configuration positioned between and coaxially with the other of said punches to define a second chamber therewith, sealing means between the tapered surface of said punch and the tapered surface of said ring, said block having its other projecting frustroconical surface in the frustroconical space defined by said second ring, at least one strengthening ring about said second ring, and means to conduct an electrical current to each of said chambers.

5. The invention as claimed in claim 4 wherein each of said sealing means includes a thermal and electrical insulating cylindrical gasket coaxially positioned on said nibs, a plurality of thermal and electrically insulating frustroconical gaskets positioned in interfitting stacked relationship on the frustroconical surface of said punches, and an electrically conducting metal gasket positioned between said frustroconical gaskets.

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