June 21, 1960 H. T. HALL 2,941,242

HIGH TEMPERATURE HIGH PRESSURE APPARATUS

Filed Feb. 14, 1955

Fig. 1.

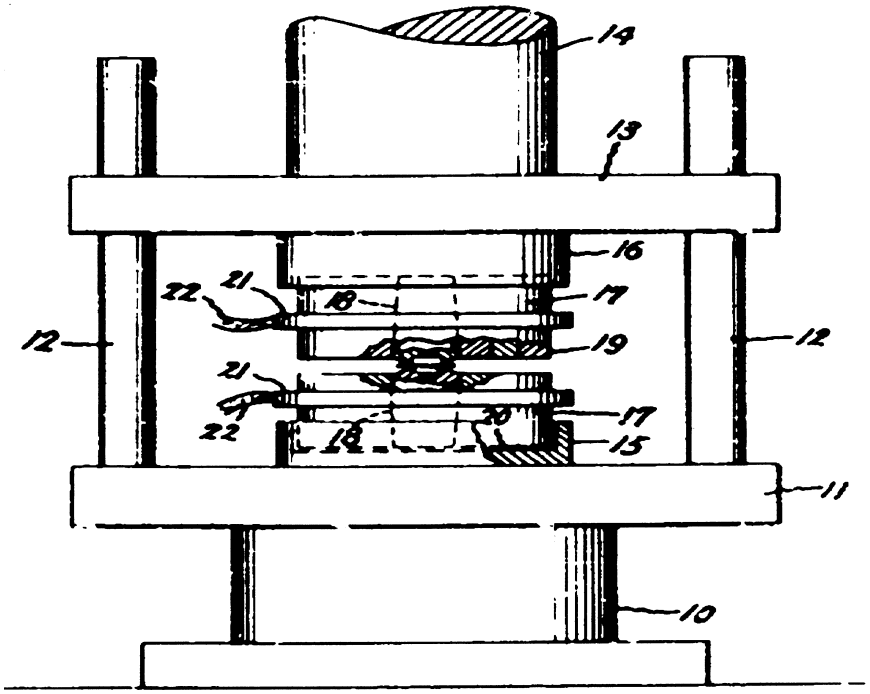
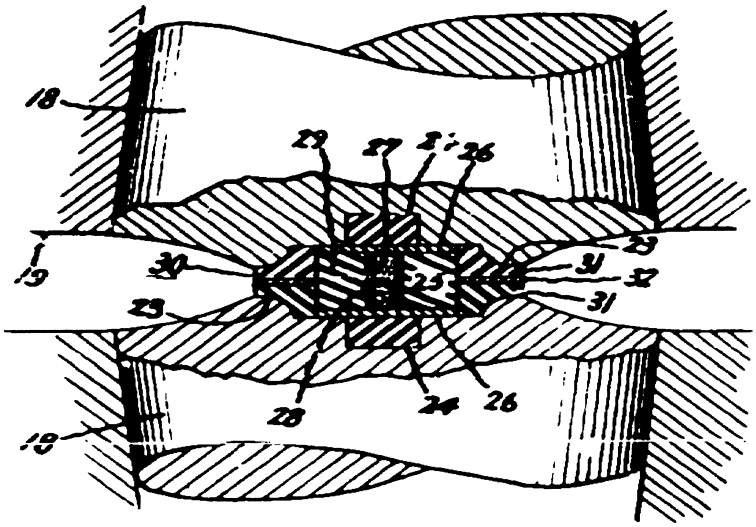


Fig. 2.



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HIGH TEMPERATURE HIGH PRESSURE APPARATUS

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My invention relates to high temperature high pressure apparatus and specifically to such apparatus in which a gasket assembly is provided to increase the volume of the reaction chamber and the pressure applied thereto.

In a copending application of Francis P. Bundy, Serial No. 448,042, filed concurrently herewith and assigned to the same assignee as the present application, an apparatus is described to effect and control reactions occurring at temperatures of the order of several thousand degrees centigrade and pressures of the order of 40,000 to 60,000 atmospheres for long time intervals. Such apparatus comprises a reaction vessel which is positioned between a pair of opposed, recessed dies to provide a specimen chamber to be subjected to temperatures and pressures of the order mentioned.

A high temperature high pressure apparatus which provides a reaction vessel of increased size is desirable to accommodate larger amounts of materials to be heated and pressed. In previous devices, the size of the reaction vessel was limited by thickness of the insulating and pressure resisting gasket. Such a gasket could be increased in size but application of high pressure caused lateral extrusion thereof from between opposed members with attendant loss of pressure in the reaction vessel The invention of the present application eliminates such gasket extrusion with a novel gasket assembly which permits an increase in both reaction vessel size and applied pressure.

Accordingly, it is an object of the invention to provide a new and improved high temperature high pressure apparatus.

It is another object of the invention to provide an improved high temperature high pressure apparatus in which both the size of the reaction vessel and applied pressure are increased.

It is a further object of the invention to provide an improved high pressure apparatus which is subjected to a high temperature range.

In carrying out my invention in one form, a gasket around a casing within a pair of opposed, recessed dies to provide an enlarged reaction vessel which is subjected to high temperature and high pressure.

These and various other projects, features and advantages of the invention will be better understood from the following description taken in connection with the accompanying drawing in which:

Fig. 1 is a front elevational view of a hydraulic press with a high temperature high pressure apparatus which embodies my invention; and

Fig. 2 is an enlarged sectional view of the high temperature high pressure apparatus which is shown in Fig. 1.

In Fig 1 of the drawing, a hydraulic press comprises a base 10 with a press bed 11 on which is mounted a plurality of vertical shafts 12 to support a carriage 13 with a hydraulic shaft 14. A pair of opposed pistons 15 and 16 on bed 11 and carriage 13 are recessed to partially position members 17 therein, each of which members 17 comprises a central die 18 with surrounding binding rings 19. A layer of electrical insulation 20 is provided between at least one member 17 and its associated piston 15 to prevent conduction of electric current through the press. An electrical connection to each member 17 is provided by a conducting ring 21 with a connector 22 which ring is positioned around the periphery of each outer binding ring 19 to supply electric current from a source of power (not shown) through rings 19 and dies 15 to the high temperature high pressure reaction vessel which is described below.

As is best shown in Fig. 2, the outer face of each die 18 has a raised annular lip 23 which defines a recess. The lip and recess thus formed

are spaced from the periphery of the face of the die. A block 24 of electrically insulating material is positioned in the central portion of each recess to prevent burning out dies 18 through the application of current thereto. A reaction vessel 25, which is located within the recesses of opposing dies 18, comprises a pair of spaced conductive discs 26 with a hollow conductive cylinder 27 therebetween adapted to contain a specimen 28 to be subjected to high temperature high pressure conditions. An annular washer 29 of electrically insulating material is positioned around cylinder 27 between discs 26 to complete the assembly of reaction vessel 25. If it is desired, reaction vessel 25 may be in the form of a hollow casing which is in electrical contact with opposing dies 18.

An annular gasket assembly 30 surrounds vessel 25 and tapers laterally to have its outer edge fit between and be engaged by lips 23 of dies 18. Gasket assembly 30 is shown as comprising a pair of annular thermal and electrical insulating and pressure resistant washers 31 with a metallic washer 32 between adjacent washers 31. While only a pair of washers 31 with a separating washer 32 are illustrated in Fig. 2, 1 have found that a plurality of alternate washers further increases the size of reaction vessel 25 to which greater pressures may be applied without lateral extrusion of gasket assembly 30 with attendant loss of pressure. Such higher pressures are attained with gasket assembly 30 because the non-extruding material increases the length of travel of die 18 along its center line to develop increased pressures. Examples of suitable materials from which blocks 24, and washers 29 and 31 may be made are pyrophyllite and catlinite.

In the operation of the high temperature high pressure apparatus shown in Figs. 1 and 2, each member 17 with associated conducting ring 21 and connector 22 is positioned partially within the recess of its respective piston in the press. Block 24 is then set in the central portion of the recess of each die 18 to provide electrical insulation. Specimen 28 which is to be subjected to a high temperature high pressure environment is placed in cylinder 27 within washer 29 between discs 26 to complete reaction vessel 25. Gasket assembly 30 is positioned around vessel 23 after which this vessel and its associated gasket assembly 30 are positioned within the recesses of opposing dies 18 between blocks 24. The outer edge of gasket assembly 30 fits

between and is engaged by opposed lips 23 to prevent lateral extrusion of this assembly with attendant loss of pressure to vessel 23 when pressure is applied by the press.

Pressure is applied to substance 28 in cylinder 27 by shaft 14 of the press. At the same time, electric current is supplied from one electrical connector, such as upper connector 22, to upper conducting ring 21, binding rings 29, die 18 and upper disc 26 to generate heat in cylinder 27 of vessel 25. The current path continues from cylinder 27 through lower disc 26, die 18, binding rings 19, conducting ring 21, and connector 22 to the electrical source. Pressures in excess of 50,000 atmospheres at temperatures higher than 2500*C. have been maintained in such apparatus for periods of hours. If it is desired, reaction vessel 25 may also be subjected to high pressures alone

As will be apparent to those skilled in the art, the objects of my invention are attained by the use of a gasket assembly which is positioned between a pair of opposed, recessed dies to provide an enlarged reaction vessel to be subjected to high temperature and high pressure conditions.

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. High temperature high pressure apparatus comprising a pair of opposed, recessed pistons, means for exerting pressure against at least one of said pistons, a pair of members, one of said members being positioned partially within each of said recesses, a layer of insulation between one of said members and its associated piston, an electrical connection to each of said members, each member comprising a die, the outer face of said die having a raised annular lip defining a recess, a cylindrical reaction vessel located within the recesses of opposing die faces, said reaction vessel being in electrical contact with said dies, and an annular gasket assembly surrounding the side of said vessel, said gasket assembly comprising a pair of annular insulating and pressure resisting washers, an annular metallic washer positioned between said pair of insulating and pressure resisting washers, said gasket assembly having its outer edge positioned adjacent said annular lip, the outer edge of said

gasket assembly being arranged to fit between and be engaged by the lips of said dies.

2. High temperature high pressure apparatus comprising a pair of opposed, recessed pistons, means for exerting pressure against at least one of said pistons, a pair of members, one of said members being positioned partially within each of said recesses. a layer of insulation between one of said members and its associated piston, an electrical connection to each of said members, each member comprising a die, the outer face of said die having a raised annular lip defining a recess, a cylindrical reaction vessel located within the recesses of opposing die faces, said reaction vessel comprising a hollow conductivity cylinder adapted to contain a specimen to be subjected to high temperature high pressure conditions in electrical contact with said dies, and an annular gasket assembly comprising a plurality of annular insulating and pressure resisting washers, an annular metallic washer positioned between adjacent insulating and pressure resisting washers, said gasket assembly surrounding the side of said vessel and having its outer edge positioned adjacent said annular lip, the outer edge of said gasket assembly being arranged to fit between and be engaged by the lips of said dies.

3. High temperature high pressure apparatus comprising a pair of opposed, recessed pistons, means for exerting pressure against at least one of said pistons, a pair of members, one of said members being positioned partially within each of said recesses, a layer of insulation between one of said members and its associated piston, an electrical connection to each of said members, each member comprising a die, at least one binding ring around said die, the outer face of said die having a raised annular lip defining a recess, a block of electrically insulating material positioned in the central portion of said recess, a reaction vessel located within the recesses of opposing die faces between said blocks, said reaction vessel comprising a hollow conductive cylinder adapted to contain a specimen to be subjected to high temperature high pressure conditions in electrical contact with said dies, and an annular gasket assembly comprising a plurality of annular insulating and pressure resisting washers, an annular metallic washer positioned between adjacent insulating and pressure resisting washers, said gasket assembly surrounding the side of said vessel and having its outer edge positioned adjacent said annular lip,

the outer edge of said gasket assembly being arranged to fit between and be engaged by the lips of said dies.

4. High temperature high pressure apparatus comprising a pair of opposed, recessed pistons, means for exerting pressure against at least one of said pistons. a pair of members, one of said members being positioned partially within each of said recesses, a layer of insulation between one of said members and its associated piston, an electrical connection to each of said members, each member comprising a die, at least one binding ring around said die, the outer face of said die having a raised annular lip defining a recess, a block of electrically insulating material positioned in the central portion of said recess, a cylindrical reaction vessel located within the recesses of opposing die faces between said blocks, said reaction vessel comprising a pair of spaced conductive discs, a hollow conductive cylinder adapted to contain a specimen to be subjected to high temperature high pressure conditions positioned between said discs, an insulating washer around said cylinder between said discs, and an annular gasket assembly comprising a plurality of annular insulating and pressure resisting washers an annular metallic washer positioned between adjacent pressure resisting washers, said gasket assembly surrounding the side of said vessel and having its outer edge positioned adjacent said annular lip, the outer edge of said gasket assembly being arranged to fit between and be engaged by the lips of said dies.

5. A high pressure high temperature apparatus comprising in combination, a pair of opposed dies, means to move one of said dies toward the other, each of the opposed surfaces of said dies having a raised annular tapered lip spaced within the periphery of said surface, said lips defining a recess to contain an object to be subjected to high pressures and high temperatures, an annular thermal and electrically insulating gasket adjacent each die and surrounding and engaging said object, an annular metallic gasket between and in contact with said thermal and electrically insulating gaskets and surrounding said object. the said insulating gaskets being engaged by the lips of said die for deformation thereof upon movement of one of said dies toward the other, and means to conduct electrical current through said object for heating thereof.

6. A high pressure high temperature apparatus comprising in combination, a pair of opposed dies, means to move one of said dies toward the other, each of the opposed surfaces of said dies having a raised annular tapered lip spaced within the periphery of said surface, said lips defining a recess to contain an object to be subjected to high pressures and high temperatures, a block of thermal and electrically insulating material in said recess of each die, and a reaction vessel in said recess between said blocks, said reaction vessel comprising a hollow, thermal and electrically insulating cylinder to contain a specimen material therein, an electrically conductive disc closing each end of said hollow cylinder and in contact with each die, an annular thermal and electrically insulating gasket adjacent each die and surrounding and engaging said reaction vessel, an annular metallic gasket between and engaging said thermal and electrically insulating gaskets and surrounding said reaction vessel, the said gaskets being engaged by the lips of said dies for deformation thereof upon movement of one of said dies toward the other and means to conduct an electrical current through said reaction vessel.

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