

Coes (6) has used a simple piston-cylinder apparatus with a high-density sintered alumina inner cylinder. This instrument is capable of 3.5×10^4 atmospheres at temperatures up to 1200K. High temperatures are achieved with internal resistance heaters operated with low voltage currents. Electrical insulation of the tungsten carbide pistons is complete because of the alumina inner cylinder which contains the reaction charge. External support is given to the alumina by a shrunk-on hard alloy steel cylinder. The latter is surrounded by a water jacket to facilitate sufficient heat dissipation. The limiting pressure of this apparatus has been the compressive strength of the unsupported pistons.

Hall (7) has developed two apparatus designs which are somewhat different from those described above.

The first consists of a stepped piston-tapered cylinder assembly provided with independent clamping mechanisms for both the piston and cylinder. In principle, the stepped piston is mated with the stepped bore of the cylinder. The annular cavity formed between the large diameter of the stepped tungsten carbide piston and the small diameter of the inner tungsten carbide cylinder bore serves as the sample space. This device is reported capable of 2×10^5 atmospheres at room temperature. In order to achieve this limit, however, it is necessary to provide auxiliary support to both the stepped piston and the bore cylinder at their respective end faces (lateral support to the bore cylinder is provided by the surrounding tapered ring assembly). If this is not done, lateral failures occur at the step plane due to the sharp stress gradient at this position. Vertical cylinder support is provided by strong steel end plates drawn together by high strength bolts. Axial piston support can be achieved in cooperation with the required piston working force by means of two independent coaxial hydraulic ram systems.

Hall's second design can be described as a system of four converging anvils of the Bridgman type aligned in a regular tetrahedral arrangement. The working face of the anvils are provided with a triangular face rather than the normal circular one. The pistons and their respective hydraulic rams are oriented and held by a tetrahedral network of steel tie rods and corner forgings. The solid pressure transmitting medium and sample holder consists of pyrophyllite (Tennessee Grade A Lava) machined in the shape of a regular tetrahedron 25 per cent oversize on edge with respect to the edge dimension of the triangular face of the anvil. The 25 per cent oversize is allowed in order

to provide for piston movement, a compressible gasket seal along the otherwise open edges, electrical insulation between anvils and to permit easy access to the sample area for sensing devices such as thermocouples. The pyrophyllite is bored diametrically across a pair of edges to provide for a tubular resistance heater and sample space. With this tetrahedral design, Hall incorporates all the advantages of the Bridgman anvil and at the same time, eliminates the disadvantages of small sample size and heating difficulties. The apparatus is reported to be capable of pressures of the order of 1×10^5 atmospheres and temperatures of 3×10^3 K.

Two types of pressure apparatus have been used by the authors thus far in studies conducted at USASRD. The first consists of an enclosed anvil-tapered cylinder assembly, and the second is a supported step piston-tapered cylinder assembly. Schematics of these two instruments are given in Fig. 2.

Tungsten carbide (6 per cent cobalt) is used for all compression members in both designs. This grade of carbide possesses the greatest compressive strength and appears to be preferred by most workers. Inner bore cylinders of both alloy steel and carbide have been successfully used; however, for higher pressures and temperatures the carbide is superior. Both lateral and axial support is provided to the cylinder assembly of each apparatus.

The enclosed anvil apparatus has been successfully operated thus far to pressures and temperatures of approximately 6×10^4 atmospheres and 2×10^3 K, respectively. Anvils of approximately 5 cm diam, 1.25 cm flat working face and tapers of 25° , 30° and 35° have been used.

The most critical variables with respect to work efficiency are found to be the height of the sample volume, and the solid pressure-transmitting material used. Sample heights of 0.25 to 0.75 cm produced a pressure "gain" (gain being defined as the ratio of the average pressure across the 1.25 cm working face of the tapered piston to the average pressure applied across the flat back face of the piston) of 3 to 2, respectively. NaCl, hot pressed BN and pyrophyllite have been tried as pressure transmitting materials. Of the three, pyrophyllite has been found to be best with respect to ease of fabrication, cost, chemical stability and pressure performance.

In practice, a solid piece of pyrophyllite, machined in the form of a double concave

taper with a centered axial hole to receive the sample (and heating element when desired), is placed between the opposed tapered pistons. Force is applied by means of a hydraulic press. Due to the increasing height of the peripheral pyrophyllite surrounding the sample, the relative compression experienced by outward lying material diminishes as the distance from the center of the material to its outer boundary on the cylinder wall increases. Ideally, the pyrophyllite can be thought of as a series of concentric thin walled cylinders of increasing height which surround the centrally located axial specimen. Each is relatively compressed in proportion to the reciprocal of its height for a given piston advance. Experimentally, however, the idealized radial pressure gradient is not realized because of radial flow. The latter can be reduced to some extent by saturating the pressure-transmitting material as much as possible with a high friction material such as Fe_2O_3 powder, or by using a material with a higher internal friction than pyrophyllite.

The supported stepped piston-tapered cylinder design shows promise for very high pressure-moderate temperature operation. Thus far, the apparatus has been used to pressures of approximately 6×10^4 atmospheres and temperatures of $1.8 \times 10^3\text{K}$ for reasonably sustained periods. Design philosophy is based on Bridgman's work with compressible gaskets and multistaging; the latter dealing with total immersion of a high pressure device within one of lower pressure in order to create an effective homogeneous support.

Essentially, the instrument consists of a pair of opposed multiple (stepped) tungsten carbide pistons fitted to a stepped carbide inner cylinder bore. The sample space is located between the opposing faces of the small diameter pistons. A compressible solid material is placed within the annular spaces created between the respective large piston tapers and the steps of the cylinder bore. The compressible material allows movement to the pistons and simultaneously provides support to the normally exposed back portion of the over-stressed small piston. A complete "immersion" of each small piston is accomplished by use of a thin shim of soft metal foil at the end face in contact with the anvil piston.

The ratio of support pressure to sample pressure is determined by the relative heights of the respective volumes, and the relative compressibilities of the materials placed in each space. True internal stresses generated within the sample and support

volumes are determined by observing reference electrical resistance changes due to pressure induced polymorphism in materials such as bismuth, cesium and thallium. (3) Electrical insulation is provided by allowing a tolerance between piston - cylinder fits and placing an insulating material within the resulting clearance. Low voltage currents minimize insulation requirements and are generally used. Since the large diameter pistons are operated within their normal strength limits, no lateral support is required. Support is given to the inner bore cylinder by a press-fit tapered steel ring assembly as previously described.

The application of either internal pressure, external pressure, or both to a hollow right circular cylinder, however, induces radial, circumferential and axial stresses. In vessels having a ratio of wall thickness to radius less than 1 to 10, the above stresses will not be uniform throughout the wall. A maximum will exist at the center and a minimum at the outer surface. A quantitative stress description is provided by the following equations; (8)

For conditions of uniform internal pressure

$$s_c = p \frac{a^2 (b^2 + r^2)}{r^2 (b^2 - a^2)} \quad (1)$$

$$s_r = p \frac{a^2 (b^2 - r^2)}{r^2 (b^2 - a^2)} \quad (2)$$

for uniform external pressure

$$s_c = p \frac{b^2 (a^2 + r^2)}{r^2 (b^2 - a^2)} \quad (3)$$

$$s_r = p \frac{b^2 (r^2 - a^2)}{r^2 (b^2 - a^2)} \quad (4)$$

where p = applied external or internal pressure

a = radius of cylinder bore

b = radius of outer wall

r = any intermediate radius at which stresses are to be calculated

s_c = circumferential stress (positive for tensile, minus for compressive)

s_r = radial compressive stress

Equation (1) shows that with $r = a$, $s = \max = p \frac{b^2 + a^2}{b^2 - a^2}$, the limiting value of $\frac{b^2 + a^2}{b^2 - a^2} = 1$ when $b \gg a$. As $b \rightarrow$ infinity, therefore, the smallest circumferential stress possible at the inner bore equals the applied pressure. It is clear that an increase in wall thickness is of limited value in adding to the ultimate strength of the assembly. External support, or pre-stressing must be employed to increase capabilities for very high pressure work. Although several methods are possible, the interference ring technique is considered best. Here, an initial state of compression is induced in the inner cylinder. As working pressure is applied to the inner