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Pressure Cell with Ten Electrical Leads for Liquid Hydrostatic Pressures to 60 Kilobars

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positive result followed exposure to several of the fatty acids, in particular, acetic, lauric, myristic, and palmitic acids. Since lauric acid is a solid at room temperature and has a vapor pressure suitable for this application, considerable testing was carried out using this acid as a conditioning agent. The results of a series of gain measurements are shown in Fig. 2. The test began with the acid source in the test chamber, and periodically thereafter the source was removed and replaced or the acid pressure was varied thermally. Throughout this test the multiplier was continuously excited by radiation from a xenon discharge at a counting rate of 5.5×10^3 pulses/sec. No measurable change in the photoemissive response of the channel was noted. In the upper portion of Fig. 2, the pressure in the test chamber, as monitored by the ion pump current, is indicated for certain times during the operating period. From the data of Fig. 2, it is clear that the gain of the channel multiplier responds to the presence of lauric acid n the environment. This gain recovery is not instantaneous but occurs over a period of several hours or days depending upon the amount of acid vapor present. Shortly after the acid is removed the gain begins to decay, implying the multiplier surfaces have undergone no permanent modification. For applications where the acid vapor is not objectionable on other grounds, this technique may be useful for extending the operating lifetime of certain multipliers.

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Pressure Cell with Ten Electrical Leads for Liquid Hydrostatic Pressures to 60 Kilobars

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A cylindrical two-part stainless steel pressure cell with 10 electrical leads that exit directly from a 2.33 cm^3 liquid working volume was designed for the generation of hydrostatic pressures to 60 kilobars in a liquid–solid hybrid system. The structural design, electrical lead system, high pressure liquid seals, and capability of the hydrostatic pressure system are described and discussed. The hydrostatic pressure cell has been used successfully for several years.

INTRODUCTION

To obtain high hydrostatic pressures, Barnett and Bosco[1] devised a liquid–solid hybrid system in which an equivolume mixture of normal and isopentane was contained in a stainless steel cylinder which in turn was situated within a pyrophyllite cube and pressurized in a hexahedral press. Extensive use of the cell, however, revealed difficulties that seriously limited the value of the technique. The major difficulty was that the electrical leads were usually broken by pyrophyllite flow and measurements could not be made. In addition, the cell walls characteristically folded and fissured causing some loss of high pressure liquid, small working volumes, and oftentimes crushed samples and manganin gauge. Also the method of mating the two-part cell was not reproducible or conducive to retaining the initially sealed liquid during assembly. One of the present authors (RJZ) was responsible for using pressure contacts to improve the continuity of the four side-anvil leads and for adding the two extra leads to the top and bottom anvils, but neither these nor other minor modifications of the original cell were successful in overcoming the major problems. Consequently a completely new structural design, electrical lead system, high pressure liquid seals, and method of assembly were developed while retaining the principle of pre-pressurizing the liquid to overcome differential compressibility in a liquid–solid hybrid system. The present hydrostatic pressure system is reliably capable of 60 kilobars liquid pressure with 10 electrical leads exiting directly from a hydrostatic chamber with a usable 2.33 cm^3 working volume described by 1.08 cm diameter and 2.54 cm length.

Hydrostatic pressure techniques are obviously desired for research on the preparation and properties of materials under high pressure, and hexahedral presses currently are

in existence elsewhere and are also commercially available. The development of a liquid–solid hydrostatic system is a tedious and expensive task, since the interdependence of the many variables involved makes it necessary in the final analysis to rely on empirical trial-and-error methods even though one is guided by scientific principles. The purpose of the present paper is to sufficiently describe and discuss a reliable hydrostatic pressure system that can be adapted to hexahedral presses in other laboratories, while certain aspects of the technique should also find use in other high pressure systems.

The physical details of the 1814 ton (metric) hexahedral press, anvils, and pyrophyllite cube have been described previously.[2] Six electrical leads from the cell are terminated at the cube faces and four electrical leads are withdrawn through the side gaskets. Electrical contact to the benchtop is made via insulated and shielded 0.25 cm diam copper wires, six of which are bolted to the anvil retaining rings while the other four are positioned in the side gasket areas of the press. Figure 1 shows the assembled cube with electrical leads and cell before final mating in the press. The volume of this assembly is reduced about 50% by compression to 30 kilobars, which sets a serious constraint on the continuity of the electrical leads and on the reproducibility of the working volume within the stainless steel cell.

I. THE HYDROSTATIC PRESSURE CELL

A. Structural Design

A detailed drawing of the hydrostatic pressure cell is shown in Fig. 2. It is machined from No. 304 stainless steel and consists of two tapered cylindrical shells to allow sample building and initial compression of the liquid pressure medium. The two main problems associated with the structural stability of the cell were circumferential folding and splitting of the walls at the base of the liquid chamber and puckering of the bottom end of the cell. All dimensions of the cell are interrelated with regard to these problems. The combination of dimensions given in Fig. 2 was empirically determined so that high pressure liquid was not lost and there was little distortion of the cell in the area of the electrical leads. The top section of the cell initially protrudes 1.588 cm from the bottom section as machined, and is subsequently increased by the indium metal-to-metal seal discussed later.

A Teflon plug is seated into the 40° tapered section of the cell to position the electrical lead wires for passage from the

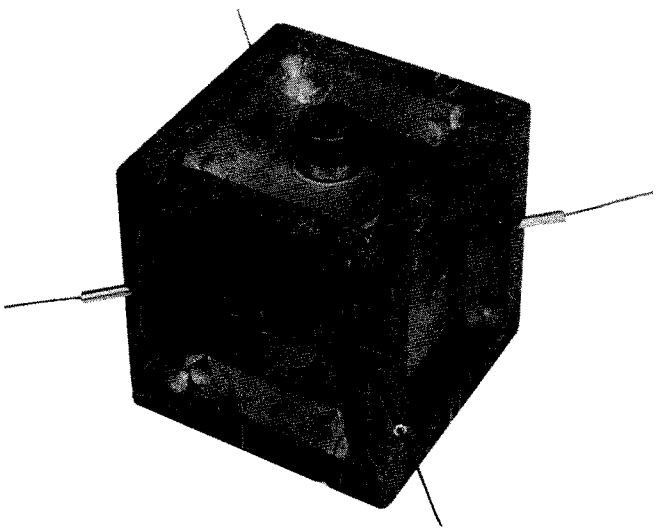

FIG. 1. View of the hydrostatic pressure cell and electrical leads assembled in the pyrophyllite cube before pressurization in the hexahedral press.

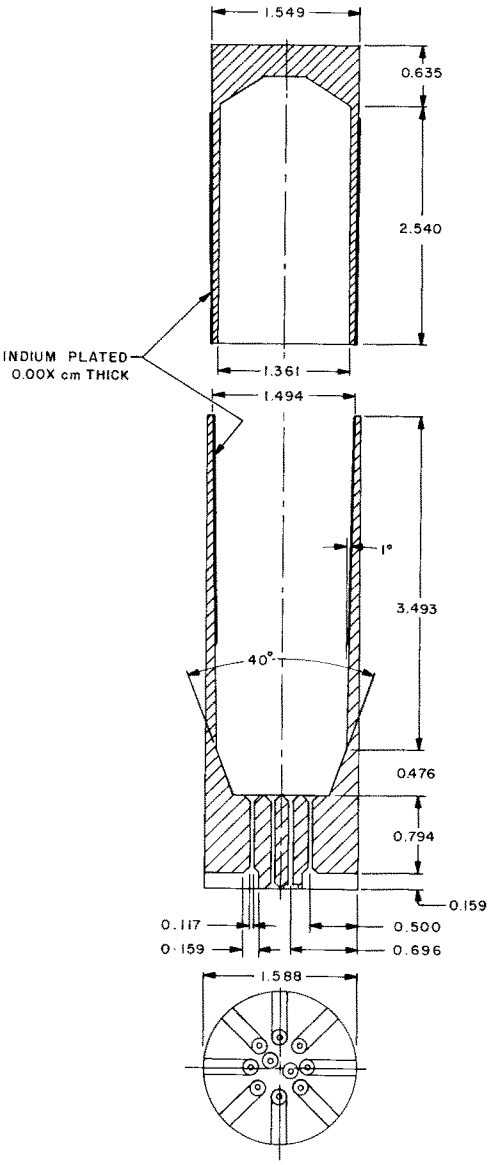

FIG. 2. Detailed drawing of the stainless steel hydrostatic pressure cell. Scale in centimeters.

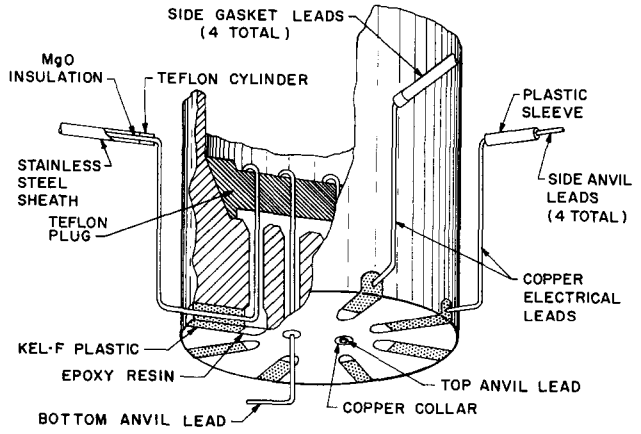

FIG. 3. Cutaway view of the electrical lead assembly of the hydrostatic pressure cell.

liquid and to facilitate sample building. Using the bottom section of the cell as a reference, 10 smaller concentric holes are drilled through the Teflon plug to assure alignment of the electrical lead wires in the feedthrough holes. The electrical lead wires are force fitted into the Teflon plug and are cut and bent 90° against the top side. Any experimental setup is built and connected with thinner wires above the plug. A 10-holed Teflon jig is employed temporarily against the bottom of the Teflon plug to prevent bending of the electrical lead wires during sample building. The Teflon plug makes an interference fit along the 1° taper of the cell so that when the plug is seated, the electrical lead wires are fixed in position through the stainless steel holes and the sample assembly is vertically centered. In order to avoid scraping the indium coating and to minimize rotation of the Teflon plug and wires during seating, the plug is machined to make contact with the walls of the cell when it is about 0.64 cm from its final position. A countersink 0.079 cm deep and 0.867 cm diameter is made at the bottom of the plug to accommodate the electrical lead and liquid high pressure seals which are described later. Another countersink at the top of the plug is made merely to allow a larger dimension of the hydrostatic working volume.

The electrical leads are withdrawn directly from the liquid chamber by 10 countersunk holes through the bottom end of the cell. This structural feedthrough design was made specifically to permit the use of continuous heavy gauge electrical lead wires from the liquid chamber to the contact points outside the pyrophyllite cube. It is this factor that made the electrical lead system reliable because it eliminated the use of fine-diameter lead wires from the liquid with solder or pressure contacts to larger-diameter wires in the pyrophyllite volume of the cube. This method also allows the cell to be filled and mated as the very last step prior to pressurization in the press, thereby avoiding the loss of liquid during handling and assembly operations and conveniently contributing to a reproducibly large hydrostatic volume.

B. Electrical Lead System

Annealed copper leads, 0.051 cm diameter and double coated with Formvar insulation, have proven to be sufficiently thick and ductile to maintain electrical continuity during the drastic compression, drawing, and bending to which the wires are subjected by pyrophyllite during the course of an experiment, particularly at the two right-angle bends. Annealed Chromel and Alumel wires, 0.051 cm diameter with an oxide coating, have also been found to be mechanically and electrically stable. Conap RN 1000 epoxy resin electrically insulates the wires in the feedthrough holes and simultaneously serves as the liquid high pressure seal described later. The electrical lead assembly is illustrated in Fig. 3.

The side-anvil and gasket leads exit from the pyrophyllite cube in the central horizontal plane. These leads are electrically insulated from the cell by Kel-F plastic cylinders across the base and by a pyrophyllite sleeve along the length. The lead wires are recessed into grooves in the surface of the sleeve. The four holes to the center of the side faces of the cube are drilled 0.15 cm diameter to facilitate assembly, and afterwards the void volume is conveniently filled by feeding a plastic sleeve over each lead wire. For the gasket leads the stainless steel rod and the regular right cylinder of Teflon are 0.318 cm diameter. The positioning of these parts against the pyrophyllite sleeve is precisely measured to assure that the lead wires are protected. The stainless steel sheath is obtained by removing the conductor from sheathed single-conductor thermocouple extension wire and serves to protect the lead wire from the extensive flow of pyrophyllite in the gasket area. The Teflon cylinder allows deformation of the lead while affording electrical and mechanical protection at the horizontal–vertical bend. To restrict the rods from slipping during initial compression and breakup of the pyrophyllite cube, the gasket holes are made to allow a slip-fit and during assembly the surface of each rod is coated with a viscous liquid mixture of acetone and cellulose nitrate glue which also fills void spaces.

One of the center leads goes to the bottom anvil that is electrically insulated from the cell by a pyrophyllite plate that forms the bottom face of the pyrophyllite cube. The remaining electrical lead is short circuited to the cell via a copper collar that is recessed into a square-well countersink in the bottom surface, and electrical contact is made with the top anvil that bears directly on the top of the pressure cell. Electrical contact with the other anvils is made by passing the electrical lead through the center of a square copper foil and cutting and bending the lead into a 0.05 cm groove in the pyrophyllite faceplate of the cube. The four

gasket leads are soldered directly to the heavy gauge copper wires that extend to the benchtop.

C. High Pressure Liquid Seals

There are two high pressure liquid seals, one for the exit of the electrical leads through the bottom of the cell and another for the mate of the two sections of the cell. For the former, a 9:1 weight ratio of epoxy–resin:hardener is applied as a fluid after the leads are positioned in the Kel-F cylinders, and is allowed to harden *in situ* overnight. All the feedthrough holes in the cell are accessible to the countersink in the Teflon plug. With the cell inverted, epoxy is slowly introduced into only a few holes and eventually all holes become filled without entrapped air pockets by equalization of the liquid level. The interference fit of the Teflon plug with the stainless steel wall serves both to hold the plug in place when it is inverted and to prevent the fluid epoxy from leaking past the plug onto the inside walls of the chamber. A thin film of silicone grease applied at the bottom of the Teflon plug is an additional preventative. Under high pressure the chamber walls deform away from the Teflon plug and leave it freestanding while the epoxy effects the high pressure liquid seal around the electrical leads.

The two sections of the hydrostatic pressure cell are mated with an interference fit along the 1° taper of the top section into the bottom section, and the liquid pressure medium is contained by an indium metal-to-metal seal. Each bearing surface is electroplated equally with indium by the Selectron method. A good mechanical coating is obtained by first sandblasting the stainless steel parts and applying a flash coating of nickel. Since the high pressure working volume depends on the pressure generated during pre-pressurization of the liquid by the top anvil, it is important to control the height of the cap at the stage when the cell is filled and mated. Either crushed or split chamber walls occur if the as-machined interference between the two mating parts is too small or too large, respectively. Pre-pressurization is also a function of the relative volumes of liquid and solid materials (samples, maganin coil, wires, etc.) in the cell since the chamber is essentially constant volume when the top anvil is advanced to the cube. Desirable working volumes are routinely obtained with a standard as-machined cap height for all experiments while using electroplated indium to reliably regulate the cap height and volume of sealed liquid. For ambient temperature experiments the volume of solids in the chamber is generally between 0.5 and 0.7 cm^3. In these cases the optimum working volume results when the cap protrudes 2.25 cm after electroplating, and slight variations are not critical. For temperature experiments a resistance tube furnace is incorporated into the liquid chamber, and it is necessary to use a smaller thickness of indium to compensate for the larger solid volume. To date, the largest volume of solids that were run satisfactorily in an experiment was 1.2 cm^3, and in this case the cap height was 2.00 cm before filling with liquid.

D. Assembly

The electrical leads, 30–35 cm long initially, are positioned in the Teflon plug and soldered to the smaller diameter wires from the manganin coil pressure gauge and samples. The other ends of the wires are then inserted one at a time through the feedthrough holes in the cell and the plug is lowered to the top of the chamber. The wires are visually aligned vertically, and the plug is manually drawn into contact with the walls of the cell. The plug is then firmly seated by means of a hollow cylindrical plunger and a lever press. The Kel-F rods are used to bend and position the leads in the channels across the base of the cell. The length of the rods assures that the lead wires do not make contact with the cell at the right angle bend. Fluid epoxy resin is applied as described earlier, and after hardening overnight the excess epoxy and Kel-F is filed away. The copper collar is fixed in place making certain of good electrical contact. The pyrophyllite sleeve is then positioned flush with the end of the cell to bend the lead wires into position in the grooves. With the cell and sleeve held above the center hole in the pyrophyllite cube, the leads are fed out to the exterior of the side faces and gaskets. The sleeve is removed and the cell is drawn into position by simultaneously pulling the eight lead wires. The sleeve is then inserted into place from the opposite face of the cube. In each face of the assembled cube there is a stainless steel and pyrophyllite faceplate, each 0.159 cm thick. The cell and sleeve rest on the bottom pyrophyllite faceplate, and the sleeve extends through the plates at the top. A Teflon washer with 0.159 cm wall fits in the steel plate around the lead wires. The sleeve fits snugly between the cell and a 1.905 cm diam hole through the cube. The steel side-plates are notched to prevent pinch-off of the gasket rods, and the plastic sleeves around the side-anvil lead wires extend through the plates. The cube is 5.72 cm face to face after assembly.

The two sections of the cell are filled and mated with the aid of a plastic funnel. The funnel fits flush against the end of the cell in the pyrophyllite sleeve and is contoured along the sleeve and top face of the cube, flaring outward at a 45° angle at the gaskets. The wall thickness of the funnel stem is 0.02 cm. The cap of the cell is laid horizontally in the funnel and is covered completely by the *n*- and *i*-pentane liquid. The cap is tilted to eliminate bubbles, is quickly placed through the funnel into the bottom section of the cell, and the two sections are rapidly mated by means of a small lever press in which the cube is positioned during this whole procedure. The excess liquid is dis-

charged, the funnel is slipped over the top of the pressure cell, and the cube is immediately placed in the press. Figure 1 shows the cube at this stage. In the press the side anvils are stopped mechanically at the faces of the cube, the liquid in the cell is pressurized as the top anvil is similarly positioned, and finally all rams are simultaneously activated for three-dimensional compression of the total cube. When the top anvil is advanced to the face of the pyrophyllite cube, about 1 kilobar is generated in the liquid pressure medium.

II. CHARACTERISTICS OF THE SYSTEM

The reliability of the present hydrostatic pressure cell is best related by the results of its actual use. In the last 17 experiments, not one anvil lead was lost and the maximum number of gasket leads lost in any experiment was 2. After each of the two experiments when this occurred, the anvils were realigned, and in the next experiment all ten electrical leads were maintained. In the 13 experiments conducted since the pressure cell was standardized, a blowout occurred on pressure release from about 50 kilobars in one experiment, and blowouts occurred through the indium seal during increasing pressure in two other experiments. The former was satisfactory in all other aspects, while the latter were due to the added large solid volume of a boron nitride tube furnace in the chamber and were remedied by lowering the cap height as described earlier. For the dimensions of the working volume in the 10 other experiments, the average circular diameter was 1.14 cm with an average length of 2.79 cm. The smallest dimensions of the set (1.08 and 2.54 cm, respectively) are used for the hydrostatic working volume in planning experiments.

Experimentally, pressures are obtained from a calibrated manganin coil immersed in the liquid and are thus independent of "load" or apparatus hysteresis. A Leeds & Northrup G-2 Mueller bridge is used to measure the manganin resistance to 0.1 mΩ which, for the 80 Ω coils employed, is equivalent to pressure sensitivity less than 0.001 kilobar. In one experiment pressure was cycled at the 27, 37, and 56 kilobar levels to measure the bismuth II⇌III, thallium II⇌III, and barium I⇌II equilibrium pressures according to the resistance change of a calibrated manganin pressure gauge.[3] As many as 25 pressure cycles were made in an experiment that lasted one month to examine the transformation characteristics of bismuth I−II.[4] Pressure was maintained constant within ±0.004 kilobar for 5 h to compare bismuth I−II initiation as a function of kinetic time and the nature of the sample.[5] Unencapsulated and encapsulated bismuth samples were simultaneously examined with concurrent pressure measurements to determine the effect of solid pressure media on the initiation and equilibrium pressures.[6] Pressure was altered in steps of about 0.007 kilobar and maintained constant within ±0.001 kilobar for periods of 30 min to measure the reproducibility of the region of indifference and equilibrium pressure of bismuth I⇌II as a function of the nature of the sample and the method of measurement.[7] The experimental setup for the latter work contained a manganin coil pressure gauge, four different samples of bismuth, and a column of mercury in a glass capillary tube sealed at one end. In addition, the large number of electrical leads allows an internally heated temperature capability to 500–600°C. Experiments have been conducted with 500°C at 40 kilobars by incorporating a Nichrome-wound tube furnace and Chromel–Alumel thermocouple directly into the hydrostatic pressure medium. The main limitation of an experiment with the hydrostatic pressure cell is the recovery of samples from experiments above about 50 kilobars due to blowouts on pressure release. Because of the 10^5–10^6 P viscosity of the pressure medium between 50 and 60 kilobars,[8] pressure is altered in small increments and appropriate stabilization times are allowed for pressure equalization between pressure changes to maintain hydrostatic conditions in this pressure range.

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