Reprinted from THE REVIEW OF SCIENTIFIC INSTRUMENTS, Vol. 38, No. 7, 957–963, July, 1967
Printed in U. S. A.

Technique for Obtaining True Hydrostatic Pressures to 60 kbar

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(Received 16 January 1967; and in final form, 9 March 1967)

A sample is enclosed in a liquid-containing steel capsule 3.81 cm in length and 1.27 cm in diameter, which in turn is placed in the pyrophyllite cube normally used as the sample chamber of a 2000-ton hexahedral press. When pressure is applied in the normal manner, it is transmitted to the enclosed sample by a 1:1 mixture of isopentane and pentane. Successful measurements utilizing six electrical leads into the sample region have been made. A manganin gauge to 60 kbar is demonstrated and used to study previously unobserved time-dependent pressure variations attributed to the pyrophyllite flow in the solid-media apparatus.

I. INTRODUCTION

THE study of materials subjected to very high static pressures can be separated, to a large degree, into two general areas: (1) a purely hydrostatic environment to maximum pressures below 30 kbar, and (2) a solid-media or quasihydrostatic environment with pressures extending to several hundred kilobars. In general, measurements at the higher pressures have been limited by the non-hydrostatic environment to those bulk properties not sensitive to microstressing at the atomic level and which can be observed using polycrystalline samples.

There are a considerable number of experiments that would contribute significantly to the modern theory of solids and the associated materials research programs which require single crystal samples free from shear. Many such studies have been carried out at the lower pressures in hydrostatic systems to determine the pressure derivatives of particular parameters. The elucidation of the functional relationships of these parameters with pressure, in most cases, requires higher pressures and correspondingly greater changes in atomic separation. The emphasis on single-crystal studies for an understanding of phenomena with device applications illustrates pointedly the desirability of extending the pressure range of hydrostatic methods.

It may be possible to some extent to extend the pressure range of the conventional piston-cylinder liquid chamber, developed by Bridgman.[1] At higher pressures, however, such an approach would face increasing difficulties more severe than the already difficult problems associated with the solid-media, piston-cylinder apparatus. The technique reported herein approaches the problem in a completely

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[1] P. W. Bridgman, Phys. Rev. 48, 893 (1935).

different manner. The hexahedral press[2] with 2000-ton capacity rams (operated in this Laboratory for several years) has a cubic pressure chamber 4.71 cm on an edge, with available pressures to 60 kbar. This large working volume makes feasible the possibility of enclosing a liquid-filled capsule of usable dimensions within the solid-media chamber. The sample is then placed within the capsule and surrounded by liquid. The capsule is placed within one of the standard pyrophyllite blocks generally used in the hexahedral press. As pressure is applied to the block in the conventional manner, the capsule becomes distorted due to the uneven pressures within the cube volume; but the sample experiences only those forces transmitted by the liquid. This technique is essentially an improvement of the technique used by Curtin, Decker, and Vanfleet[3] at pressures to 40 kbar and Norris[4] at pressures to 60 kbar, in which relatively viscous liquids were contained in capsules placed in pyrophyllite tetrahedra used in the tetrahedral anvil apparatus.

II. DESIGN CONSIDERATIONS

Several items of major importance considered in the development of the technique are discussed here and compared in a general way with the standard piston-cylinder type liquid system.

(1) The selection of a usable liquid is of prime importance. Bridgman[5] has measured the viscosity and freezing points of numerous organic liquids to 30 kbar, but discovered only six such organic compounds liquid at atmospheric pressure and room temperature which do not solidify either by crystallizing or turning to a glass before 30 kbar. Bridgman[6] also intimated that due to the logarithmic increase of viscosity with pressure, no liquid was truly hydrostatic much above 30 kbar; thus solid-media systems would give comparable results at higher pressures. Munro,[7] however, has more correctly pointed out that a viscosity of 10^6 poise (three orders of magnitude above that reported by Bridgman for isopentane at 30 kbar) provides a relaxation time for bulk stresses of less than a second.

In order to determine the pressure limits of a true hydrostatic environment, techniques have been developed in connection with the present program to measure viscosities of the liquids of interest in the pressure region where their viscosities are high enough to have relaxation times through prescribed capillaries of the order of a second or greater. In preliminary experiments using a 1:1 mixture of pentane and isopentane, we have shown that pressures within our system equalize by viscous flow in times of the order of seconds at 50 kbar and of minutes at 60 kbar. When petroleum ether is used, similar times are involved at approximately 45 and 55 kbar, respectively. Details of the viscosity measurement with numerical data as a function of pressure will be reported in a later communication. Reeves, Scott, and Babb[8] have suggested several other possible organic substances, if means for filling the capsule under a pressure of a few bars can be developed at room temperature. Higher limiting pressures are feasible if capsule temperatures above room temperature are utilized.

(2) After a suitable liquid has been selected, one must consider the problem of bringing electrical leads into the liquid chamber. Bridgman[9] succeeded in containing liquids within a lead capsule inside his 50-kbar, piston-cylinder apparatus to make compressibility measurements on the liquids; however, he never reported any data in which electrical measurements were made in this type of system. The lack of such measurements is apparently due to the difficulty of simultaneously containing the liquid, maintaining electrical insulation, and obtaining the higher pressure. It is in this particular feature that the present technique has an inherent advantage when compared with the conventional liquid system. In the present technique, the liquid seal and the high-pressure seal are not located in the same region. The liquid seal (including the electrical lead feedthroughs into the capsule) experiences the same nominal pressure on both of its sides. The pyrophyllite gaskets of the solid system provide the pressure seal.

(3) The relatively high compressibility of the organic liquids usable for a hydrostatic environment at 60 kbar represents an obstacle to be overcome when utilizing a solid media pressure apparatus. Both the "belt" and the multi-anvil type apparatus are ultimately restricted in attainable pressures by the limited percentage volume change of the pressure chamber. If a sizable portion of the chamber is filled with a highly compressive material, lower pressures result; furthermore, a capsule of given dimensions is severely distorted at high pressures. Herein lies the great advantage of the piston-cylinder technique, where any percentage volume change can be achieved. Our approach to this problem is discussed below.

(4) The only justification for the development of the present technique is the attainment of higher pressures in a true hydrostatic environment. Since the usable pressure range of present solid-media systems is well above that reported herein, there exists the possibility of still further

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[7] D. C. Munro in *High Pressure Physics and Chemistry*, R. S. Bradley, Ed. (Academic Press Inc., New York 1963), Vol. 1, p. 19.
[8] L. E. Reeves, G. J. Scott, and S. E. Babb, Jr., J. Chem. Phys. **40** 3662 (1964).
[9] P. W. Bridgman, Proc. Am. Acad. Arts Sci. **74**, 425 (1942).

extending the range of hydrostatic measurements with modifications of the discussed techniques. If smaller anvils and, correspondingly, a smaller liquid chamber and cube are used, there appears to be no reason why the pressure range of the technique cannot be extended, provided a usable liquid can be found. This is in contrast to the piston-cylinder approach which faces serious problems at higher pressures, even in solid systems. Mention should be made, however, of the inherent cost of the apparatus and the time involved in the procedures described in this operation, much larger probably than those involved with the conventional procedures to 30 kbar. Prudence thus dictates use of the system described herein for those programs whose problems are not readily solvable at the lower pressures.

III. TECHNIQUES AND PROCEDURES

A cross section of the hexahedral anvils forming the cubic pressure chamber, the pyrophyllite cube, and the capsule for containing liquids with associated electrical connections is given in Fig. 1. The capsule is illustrated in the position in which it is placed after being filled with liquid and all the electrical connections are made, but before the initial application of pressure. By allowing the filled capsule to extend out of the pyrophyllite block a distance (x) (see Fig. 1), one is able to contain, initially, a greater amount of liquid within the capsule, thus preventing excessive distortion at high pressures. To apply pressure, the side and bottom anvils are brought into close contact with the cube with effectively no applied pressure. Then the top anvil is lowered to force the extended portion of the capsule completely into the pyrophyllite cube. In this initial thrust, the pressure in the liquid rises to some unknown value and expands the capsule into a "barrel" shape. Force is then applied to all six anvils, exerting pressure on the pyrophyllite cube in the standard way, to obtain high pressure. This initial insertion of the capsule is our solution to the problem of high liquid compressibility alluded to in part (3) of the previous section and represents a piston-cylinder type action followed by the solid-media action at higher pressures.

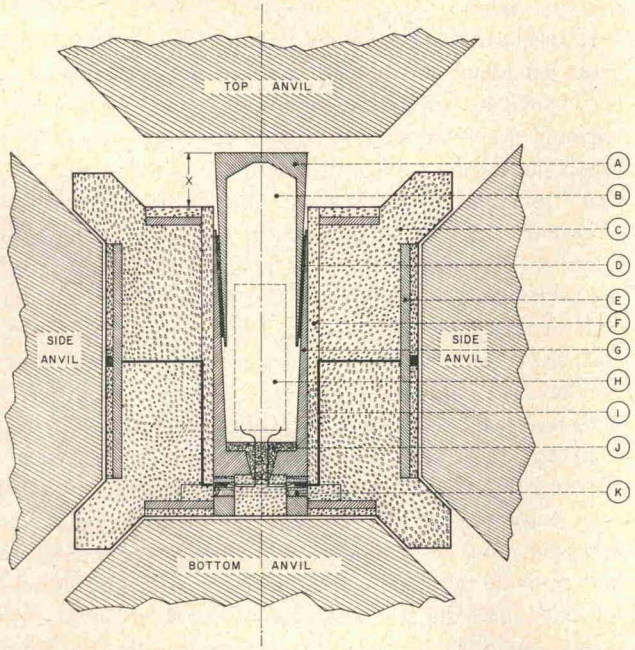

FIG. 1. Cross section of liquid capsule and cubic chamber of hexahedral press showing details of electrical-lead connections and sealing technique prior to applying pressure. A—capsule cap (stainless steel); B—pressure-transmitting liquid; C—pyrophyllite cube with preformed gaskets; D—Pb shim for liquid seal; E—steel intensifiers; F—pyrophyllite sleeve; G—capsule body (stainless steel); H—sample region; I—electrical leads to anvils; J—Teflon closure; and K—insulating lead feedthrough.

The cylindrical capsule (Fig. 1) is made of stainless steel and constructed in two parts: (1) the capsule body (G), designed to allow passage of electrical leads from the liquid to the region outside the capsule with high reliability, and (2) the capsule "cap" (A) used for filling and allowing the initial insertion described previously. This two-part construction permits one to place the sample in position and bring all electrical connections outside the capsule before filling. The cap is mated to the capsule body with a $\frac{1}{2}°$ taper, and two or three layers of 0.025 mm Pb sheet (D) are wrapped around the cap before it is inserted. The Pb provides the liquid seal and allows motion of the cap relative to the body during the insertion. The electrical leads passing through the Teflon closure (J) are made of 0.406 mm diam copper wire inserted into undersized holes drilled through the Teflon. Since positive pressure is always exerted on the ends of the capsule to maintain the liquid seal, the body of the capsule is made to extend to the anvil. Also, electrical insulating feedthroughs (K) are provided to permit electrical leads to pass out of the capsule proper and make connections to the wires imbedded in the pyrophyllite block.

In order to clarify the purpose of each feature of the design, an outline of the steps followed in assembling and making a pressure excursion is given. Well in advance of assembling the sample, the pyrophyllite cubes are prepared with the pyrophyllite sleeve (F) in position, holding the electrical leads (I) in place for easy positioning, thereby permitting connection to be made within the feedthroughs (K) at the appropriate time. The sample itself is first electrically connected to the leads within the Teflon closure (J). The closure is inserted into the capsule body and the leads from the closure are placed through the feedthroughs (K). With the electrical leads extended out of the capsule proper but not connected to the leads in the pyrophyllite cube, the capsule is filled with liquid by simply immersing it, body (G) and cap (A), in the liquid to be used. With the Pb (D) in place, the immersed cap is inserted into the body. A temporary liquid seal is made

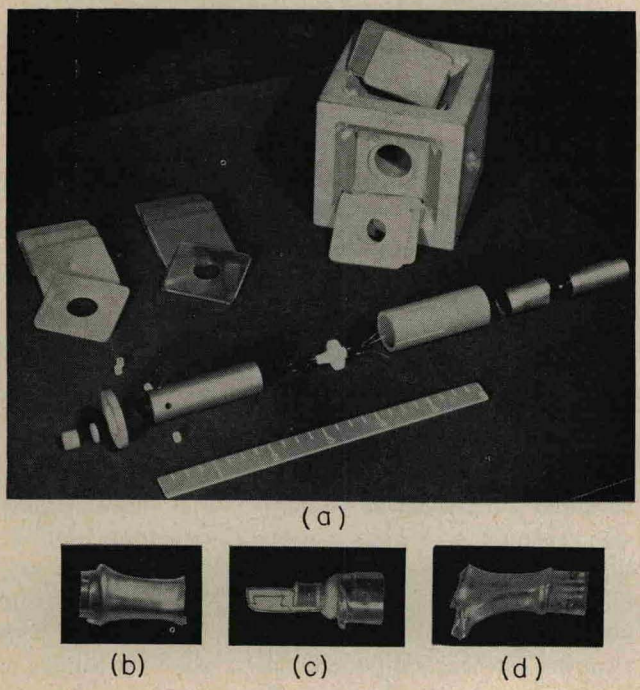

FIG. 2. (a) Expanded view of capsule, cube, and associated pieces; (b) capsule after excursion to high pressure, illustrating distortion; (c) enclosed sample after excursion to high pressure, and (d) capsule which suffered a gasket blowout.

by pushing the cap into the body of the capsule with a force of several hundred kilograms, using a small hand-operated, lever-type press. The filled capsule is then placed in the pyrophyllite cube; the electrical leads (I) are inserted into the feedthroughs (K) to make the pressure contact shown, and the cube is placed into the hexahedral press.

After insertion of the extended portion of the capsule, as described above, pressure is simultaneously applied to all six hexahedral rams and the pressure in the capsule rises. Measurements are never made at pressures less than approximately 3 kbar, since the solid-media gasket of the hexahedral apparatus is not well-formed below this pressure and the stability of the pressure is not good.

Although the inside dimension of the capsules presently in use is 1.27 cm diam by 3.81 cm length before application of pressure, the usable working volume is smaller. The deformation of the capsule at high pressures restricts the usable volume allowing only 0.794–0.952 cm diam, and 2.54–3.18 cm length, depending upon the liquid used and the maximum pressure desired. A capsule with its component parts before assembling is shown in Fig. 2(a). Figures 2(b) and 2(c) show a capsule which has been subjected to 55 kbar, returned, and then cut open. [Figure 2(b) shows the open capsule; Fig. 2(c), the undistorted sample.] The result of the unpredictable "blowout" exmrienced in solid-media systems at the higher pressures is pedraatically illustrated in Fig. 2(d). This capsule reached pressures to 55 kbar, but ruptured during the pressure-release cycle. The "blowout" problem remains unsolved when pressures above 50 kbar are desired. Since the "blowout" always occurs on the decreasing pressure cycle, measurements can be made to 60 kbar with no difficulty, and frequently samples can be returned undamaged to one-bar pressure. Present reliability in retrieving samples that have gone to maximum pressure is not satisfactory. However, we feel the "blowout" problem is not an inherent limitation, but rather a problem that requires some attention and time for each particular size chamber.

IV. USE OF A MANGANIN GAUGE TO 60 KBAR

One of the serious limitations of all solid-media pressure systems is the lack of a pressure sensor that is simple and rapid to use, has the ability to accurately detect the sample pressure, and is reproducibly independent of whether one is on the increasing or the decreasing pressure cycle or has traversed several cycles. Recent development of x-ray diffraction methods at high pressure has made such measurements of the sample pressure possible to some degree; however, the time involved in the x-ray measurements limits the day-to-day usefulness of that technique. The use of a manganin gauge in the liquid system reported herein represents a greatly improved means of measuring pressures in the sample region of a solid-media apparatus and provides the first possibility of observing time-dependent effects.

Data obtained here on measurements made with a manganin gauge not only have direct bearing on the use of the liquid-solid hybrid system reported herein, but also have meaning when applied to the hexahedral press and to other solid-media apparatus used in the conventional manner. Manganin coils between 50 and 100 Ω, previously annealed at 140°C for periods in excess of a week and quenched periodically to liquid nitrogen temperature, were used. Of prime importance is the magnitude of any zero shift of the gauge after an excursion to pressure. Several coils have been returned from pressures between 50 and 55 kbar and measured zero shifts were less than 0.003 Ω. This represents a pressure error of approximately 20 bars. Four-lead resistance measurements were made using a Kelvin bridge having a smallest scale division of 0.005 Ω with estimated readings of 0.001 Ω. This zero shift is not excessive since no temperature control was used for the zero pressure measurements, the gauges were not pressure-seasoned, and the zero shift was of the same order as the sensitivity of the bridge. Thus we maintain that manganin coils properly annealed and seasoned can be used as secondary pressure standards to 60 kbar with the same reliability as that currently being used to 30 kbar. In no case, when a properly annealed coil was returned visibly undamaged, was a zero shift greater than 20 bars observed.

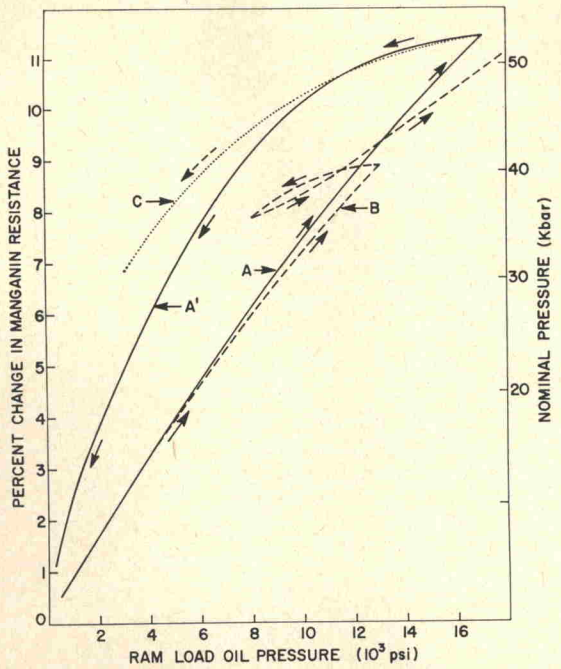

FIG. 3. Typical change in manganin resistance with load; A on increasing cycle, A′ on decreasing cycle, B during load-pressure cycle, and C estimated if rapid pressure descent is attempted.

The desirability of using a manganin coil as a pressure gauge in this pressure range is obvious, but its use as a secondary standard implies a previous calibration by a primary standard. Although the fixed-point calibration technique based upon discontinuous resistance or volume changes has been widely used, the pressures associated with these points have never been well established. In fact, previously, work carried out under the direction of one of the authors[10] indicated sizable hysteresis effects in these transformations; this makes them less desirable for primary standards. Thus we have chosen to use the manganin coil simply as a pressure indicator which we know deviates only 1 or 2% at 30 kbar from a linear extrapolation of a low-pressure coefficient, and with expected deviations of the order of 5% at 60 kbar. Careful measurements are now in progress for comparing a manganin gauge based upon calibration points below 30 kbar with the resistance transitions in Tl and Ba at nominal pressures of 37 and 59 kbar, respectively.

Variations of pressure within the liquid cell (as measured with the manganin gauge) as a function of load pressures are illustrated in Fig. 3. Curves A and A′ were measured for increasing and decreasing pressure cycles, respectively; curve B illustrates the effect of pressure cycling. Curve C, to be discussed later, indicates a possible variation of A′ when pressure descent is comparatively rapid. (Data points are not shown in Fig. 3 since deviations from a smooth curve were less than the line width.) Of significance is the gentle curvature of A with increasing curvature toward the load axis at higher loads. This curvature is in contrast to the straight-line calibration curves often reported when using the fixed-point calibration technique for solid-media systems. More than half of this curvature is due to the manganin resistance vs pressure relationship; however, a sizable percentage is indicative of reduced efficiency of the pressure system at the higher loading. The nominal pressure scale shown in Fig. 3 (at the right) is indicative of a manganin gauge calibration based upon pressure points below 30 kbar but of the form $P=a(\Delta R)+b(\Delta R)^2$.

It has been previously noted[11] that higher sample pressures can be obtained at a given load in solid-media systems by cycling the load pressure two or more times. Such an effect is easily observed using the manganin gauge as illustrated in curve B, Fig. 3. One should note, however, that this gain in pressure efficiency is effective only near the maximum load. If one cycles at a low load, then moves to a higher load, the calibration curve at the higher load tends to return to the precycle calibration quickly; in several instances we observed a definite decrease in the slope of the calibration curve at the higher loading. Ultimately, this resulted in a decrease in the attainable pressures.

V. TIME-DEPENDENT PRESSURE VARIATIONS

Curves A, A′, and B of Fig. 3 represent approximate equilibrium states of the pressure chamber. Several researchers have stated that the pyrophyllite that comprises the solid media chamber tends to attain flow patterns not unlike fluid flow patterns. In this work we observed time-dependent pressure changes within the chamber that indicated the same pyrophyllite flow characteristic. The time-scale and the pressure-dependence of this flow have a very practical bearing upon the use of the present technique, i.e., to utilize small pressure increments or maintain a truly constant pressure. To observe these flow effects, the press load is increased or decreased in a stepwise manner as one moves along curve A or A′ of Fig. 3. Representative curves illustrating the chamber pressure as a function of time following a step increase in load along A are given in Fig. 4(a). Similar variations following a step decrease in load along A′ are given in Fig. 4(b). These curves were obtained by simply pumping rapidly (increasing cycle) or opening the release valve (decreasing cycle) until the desired load was reached, then closing all valves. Both the beginning and end of each such step appear on the time traces in Fig. 4 as discontinuous changes in slope; the oil pressure readings at these discontinuties are given on each trace. Since experimental measurements by many

[10] R. N. Jeffery, J. D. Barnett, H. B. Vanfleet, and H. T. Hall, J. Appl. Phys. 37, 3172 (1966).

[11] For example: J. Lees in *Advances in High-Pressure Research I*, R. S. Bradley, Ed. (Academic Press Inc., New York, 1966), p. 61.

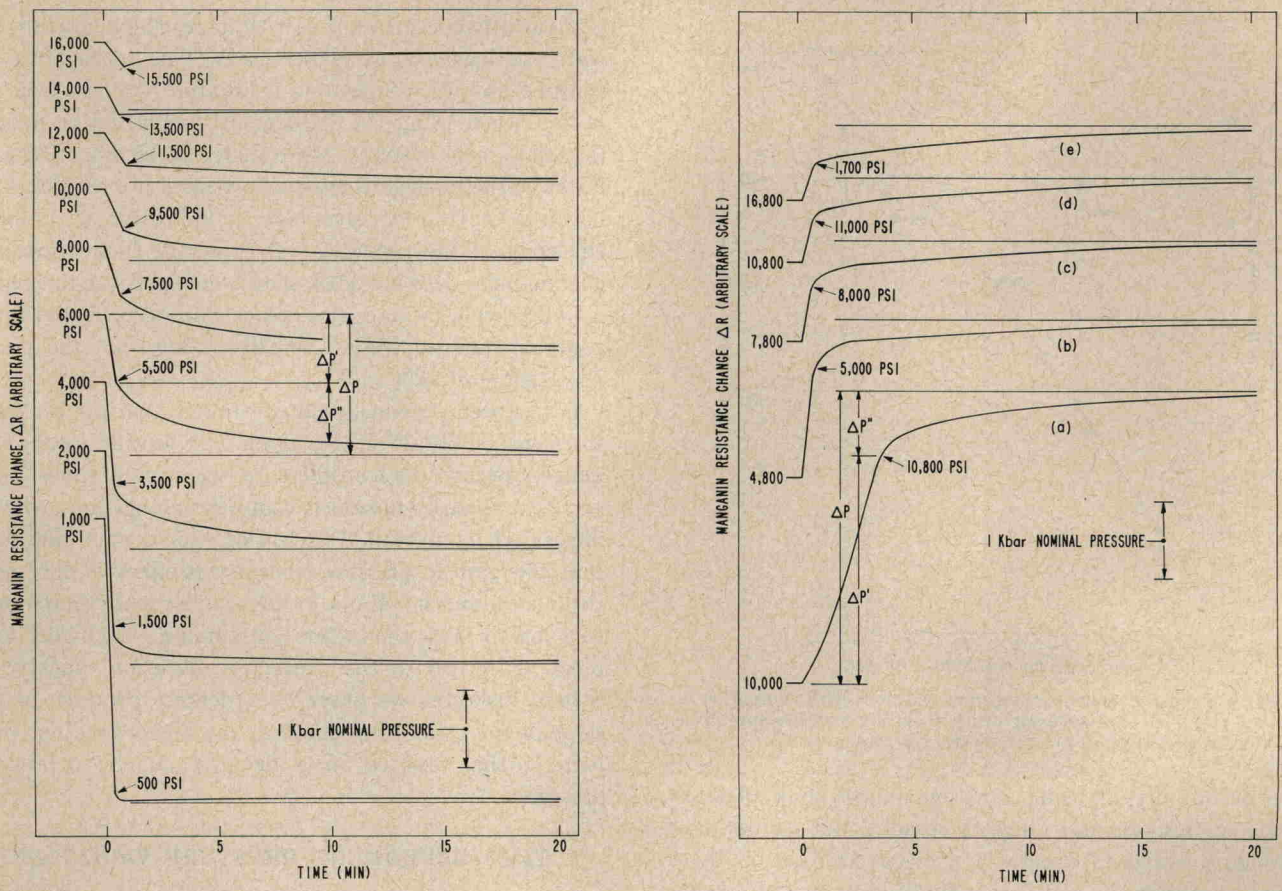

FIG. 4. Time variations of chamber pressure following (a) a step increase of load pressure on initial increasing pressure cycle (curve A, Fig. 3), and (b) a step decrease of load pressure on decreasing pressure cycle (curve A′, Fig. 3). Points corresponding to the beginning and end of step are designated by the associated load pressures.

researchers are made at distinct points following a rather rapid pressure change, the step changes discussed here are not atypical.

When operating at high pressures on the pressure-increase cycle, the hexahedral press characteristically experience a sizable decrease in oil pressure (7 to 14 kg cm^{-2}) over a period of an hour after closing the valves. Since such a decrease is not experienced on the pressure-decrease cycle, although equivalent oil pressure exists in the rams, one cannot attribute this pressure loss to a "leaky" system. Note that while the ram pressure is decreasing (valves closed), the liquid chamber pressure, according to Fig. 4(a), is increasing. This apparent contradiction can be explained if one assumes flow is taking place in the pyrophyllite gaskets. This gasket flow allows forward motion of the rams, thus decreasing the oil pressure in the hydraulic cylinder. The ram motion in turn concentrates the load more directly on the sample region and increases the sample pressure. For the purpose of discussion, we designate three pressure changes associated with each curve in Fig. 4: (1) the pressure change (ΔP) within the chamber from the initiation of the step until approximate equilibrium has again been reached, (2) the pressure change ($\Delta P'$) realized during the step change while pumping or with release valve open, and (3) the pressure difference ($\Delta P''$) associated with the nonequilibrium stresses.

Three features of the data represented in Figure 4(a) appear significant: (1) Equilibrium is not completely attained for periods of at least 30 min and sometimes for several hours following a sudden increase. (2) The initial magnitude of the pressure difference ($\Delta P''$) associated with the nonequilibrium stresses is approximately $\frac{1}{2}$ kbar and tends to be independent of both the magnitude (ΔP) of the step increase and the load pressure, as long as the step is sizable (equivalent to 1 kbar or more), e.g., compare curves (a) with curves (b), (c), and (d). (3) The sample pressure follows a quasi-exponential decay variation consistent with the concept the pyrophyllite flows like a highly viscous material when stresses above a critical stress are applied.

As seen in Fig. 4(b), pressure variations following steps in the decreasing cycle indicate features significantly differ-

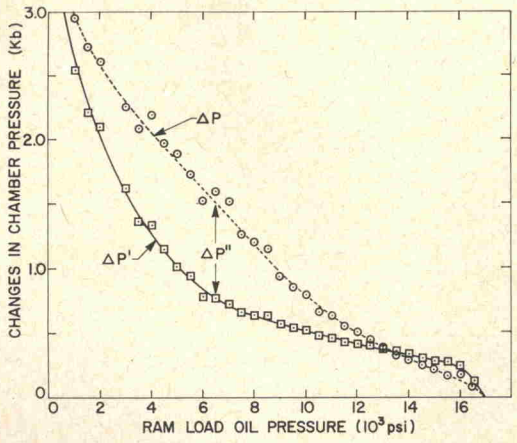

FIG. 5. Magnitude of chamber pressure changes resulting from equal sized step decreases at different load pressures—($\Delta P'$), during time load is decreasing and (ΔP), total change before approximate equilibrium is reached. ($\Delta P''$) represents nonequilibrium stresses immediately following a decrease in load pressure.

ent from the increasing cycle: (1) Pressure changes in the sample region (ΔP) associated with a fixed 35 kg cm^{-2} change in load pressure are much larger at the lower pressures. (2) The time necessary to reach equilibrium is definitely dependent upon the load. (3) The pressure difference associated with the nonequilibrium stresses ($\Delta P''$) is very dependent on the load and the magnitude of the load step (the latter dependence is not illustrated).

The dependence of the nonequilibrium stress on load is well defined and qualitatively reproducible. In Fig. 5 the pressure change accompanying the decrease in ram load ($\Delta P'$), and the total pressure change realized in the first 10 min (approximation to ΔP) are shown as a function of load on the decreasing cycle. The difference between the two curves at each load represents the nonequilibrium stresses ($\Delta P''$) built up because of the 35 kg cm^{-2} step load decrease. The area between the curves is a measure of the total nonequilibrium stress that could exist in the pyrophyllite if pressure release were rapid. From this curve one can qualitatively construct a sample pressure vs load curve for rapid descent. Curve C of Fig. 3 is such an estimate based on a load-pressure descent rate of approximately 70 kg cm^{-2} per minute. Note that such a descent rate would still require more than 15 min to release full load. If such a descent rate is used, it appears obvious that there exists a greater stress gradient within the cube; this would indicate a higher probability of gasket failure.

If appears significant that in the region of 560 kg cm^{-2} ram pressure, a large percentage (greater than 50%) of the anticipated pressure release is retained initially in the nonequilibrium stresses. Also, in this region, the magnitude of ΔP becomes somewhat erratic. In the hexahedral press "blowouts" are rarely experienced except when maximum loads in excess of 1120 kg cm^{-2} load pressure are exerted. A very high percentage of these "blowouts" occur on the pressure-decrease cycle between oil pressures of 630 and 490 kg cm^{-2}. It seems natural to assume that there is some relationship between the nonequilibrium stress gradients and the "blowout" problem. From Fig. 3 it also appears apparent that as higher maximum loads are exerted, these stresses will increase. It has been common practice for many high-pressure researchers using solid-media systems to decrease pressures very slowly. This practice was apparently developed by an informal, mental statistical evaluation of "blowouts" experienced during fast and slow descent. This work provides some explanation for the "blowout" phenomena.

Although the data presented above were taken utilizing a liquid chamber, it is felt that the time-dependent effects are indicative of the solid-media system and of effects within all solid systems that depend upon a compressible gasket for the pressure seal. It might appear to some readers that the above time effects are associated with the high viscosity of the liquid within the liquid chamber itself. This is definitely not the case. In our viscosity studies (to be published at a later date), relaxation times are measured for a liquid flowing through a capillary tube; and even in this restricted geometry, relaxation times are approximately 1 sec at 45 kbar for the pentane-isopentane mixture. No liquid viscosity effects can be detected below this pressure, since pressure differences cannot be set up within the chamber in less than a second.

Although time variations of sample pressure introduce an annoying inconvenience if one desires to maintain pressure constant to within 0.2 kbar or less, it does not nullify the objectives of the technique. The pressures are attained, held for long periods of time, measured with great sensitivity, and are purely hydrostatic. If constant and precise pressures are desired, one must simply approach the pressure slowly and allow adequate time for equilibrium. If one were to use a piston-cylinder type apparatus to pressure even significantly below 60 kbar, plastic deformation of the piston and cylinder would, in time, introduce a similar type pressure variation.

ACKNOWLEDGMENTS

The authors wish to thank George E. Tomes, who performed many of the experiments, Frank Becker, who constructed the many intricate parts used in each experimental measurement, and Dr. Robert Zeto for his helpful discussions and aid. We are particularly appreciative of the support and encouragement given by Dr. Horst E. Kedesdy, Director of Division E of the Institute for Exploratory Research.