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Strength of Solid Pressure Media and Implications for High Pressure Apparatus

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Abstract. The stress-strain properties of talc, pyrophyllite, silver chloride, sodium chloride, boron nitride and graphite have been measured under confining pressures up to 8 or 10 kb at room temperature, and, in the case of talc, also at temperatures up to 900° C. The extrapolation and application of these results to solid medium high pressure apparatus of piston-cylinder type is discussed and a calculation made of the correction to nominal pressure ("friction correction"), taking into account the stress gradients in the medium and the shearing between the medium and the cylinder wall. Correction to the nominal differential stress measured in solid medium stress-strain apparatus is also discussed.

Introduction

Solid media such as talc and pyrophyllite are widely used in experiments at high pressure, especially above 10 kb. These media are chosen, in part, because of relatively low or negligible strength. However, there is little published information on their stress-strain properties, although measurements on shearing discs give some indications (Bridgman, 1935, 1937; Vereshchagin and Zubova, 1961; Bundy, 1962; Towle and Riecker, 1969). Graf and Hulse (1964) and Hulse and Graf (1965) tested pyrophyllite and talc in compression at atmospheric pressure and elevated temperatures but their results are of doubtful relevance to high pressure conditions since they do not take into account either the effect of pressure itself or, at high temperatures, the effect of any water of decomposition on the strength.

The need for further information on the strength of solid pressure media is evident from discussions in the phase equilibrium field of the magnitude of the "friction corrections" to the nominal pressure in piston-cylinder apparatus, which probably involve in large part the strength of the medium. The corrections, obtained empirically from hysteresis measurements or from calibration against a known phase transition, vary widely between laboratories. For example, Boyd and England (1960a and 1960b) initially used a correction of — 13 percent at room temperature and — 8 percent at high temperature, while Green, Ringwood and Major (1966) arrived at a correction of — 10 percent for use over a wide pressure and temperature range. Klement, Jayaraman and Kennedy (1963) found a correction that increased more slowly with pressure and Pistorius (1967) uses a correction that is independent of pressure. Different effects of temperature on the correction have also been found. Thus Boyd and England (1963) concluded that correction could be neglected in high temperature work whereas some workers

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find relatively little decrease with increase in temperature (a number of the observations have been reviewed recently by Boettcher and Wyllie, 1968). Whether the final pressure is approached from below or from above also affects the correction.

We have therefore measured the stress-strain properties over a range of confining pressure up to 10 kb at room temperature of a number of materials used in solid media apparatus. We have also extended the measurements on talc at 4 kb up to 900° C. Whilst these do not represent normal operating conditions, extrapolation of the measurements permits more realistic estimates than hitherto of the properties of interest at high pressures.

Apparatus and Experimental Methods

The room temperature experiments were done in an apparatus previously described by Paterson (1964) in which the pressure medium was kerosene. The high temperature experiments were done in another apparatus (Paterson, 1970) which has an internal furnace and in which the pressure medium was argon. The temperature gradient along the specimens at high temperature is unlikely to have exceeded 10° C.

In both cases a small correction is applied for apparatus distortion and in the room temperature experiments there is a correction for piston friction; in the high temperature apparatus no friction corrections were necessary since an internal load cell was used. Correction has also been made for the small load borne by the copper jacket on the specimen. These various corrections and the procedures for deriving the stress-strain curves are discussed in more detail in the above references. Experimental errors are believed to be small compared to the scatter in results between individual specimens, except at strains less than about 1 percent. The results given are the means from two or three experiments in all cases except where individual measurements are indicated (as in Fig. 4).

The stress quoted is the "differential stress", that is, the difference between the total axial stress and the confining pressure. It has been calculated on "actual" cross-sectional area, obtained by assuming that the specimen has undergone uniform strain with no volume change.

The specimens were 10 mm in diameter and 20 mm long with ends ground true and flat within 0.02 mm. They had been air dried for at least several days before testing. They were then sealed in annealed copper jackets of 0.25 mm wall thickness. The strain rate was approximately 4×10^{-4} sec^{-1}. In the high temperature experiments on talc, after applying the pressure, the specimens were held at the test temperature for about 30 min before testing.

Materials Tested

1. Talc

(a) *Three Springs Talc (West Australia).* This consisted of fine-grained talc, threaded by veins and irregular patches of coarser-grained talc. X-ray texture goniometer measurements revealed no obvious preferred orientation and stress-strain measurements on orthogonally drilled specimens showed no anisotropy in strength. The density (2.71 gm cm^{-3}) was approximately 97 percent theoretical.

The results quoted are from one block; measurements on specimens from another block gave results approximately 20 percent higher.

(b) *Other Talc.* C and E refer to small blocks of talc supplied by Professor D. Griggs from different batches of commercially obtained supplies used in his laboratory. Two pairs of mutually perpendicular specimens were cored from each block. Some preferred orientation of grains was evident in thin sections and measurements on block E with the X-ray texture goniometer showed the preferred orientation to be quite strong in it.

2. Pyrophyllite

These specimens were cored from a block of "Grade A Lava" supplied by the American Lava Corporation. No obvious preferred orientation was seen in thin section and any anisotropy of strength was barely detectable in measurements on orthogonal specimens. The density (2.69 gm cm^{-3}) was approximately 95 percent theoretical.

3. Silver Chloride

Specimens were cast and machined from a commercially pure grade supplied by Townson and Mercer Pty. Ltd. (Australia). After deformation, the specimens were melted, re-cast, remachined and used again. Their density was approximately 97 percent theoretical.

4. Sodium Chloride

Specimens were formed in a piston and cylinder device from Analar analytical reagent grade material. They were not annealed before testing. The density was approximately 99.5 percent theoretical.

5. Boron Nitride

Specimens were machined to shape, all in the same orientation, from a block supplied by Union Carbide (Australia) Ltd. X-ray texture goniometer measurements showed moderate preferred orientation, about twice as many basal planes being parallel to the specimen axes as perpendicular to it. The density was approximately 91 percent theoretical.

6. Graphite

Specimens were machined from rods of "Electrographite Grade EY 9" supplied by Morganite Carbon Ltd. (Australia). X-ray texture goniometer measurements showed a strong preferred orientation of the graphite crystals, there being about three times as many with basal planes parallel to the specimen axis as perpendicular to it. The density was approximately 77 percent theoretical.

Results

1. Talc

(a) *Three Springs Talc.* The stress-strain curves at room temperature are shown in Fig. 1. The change from falling to rising curves corresponded with the transition

from brittle to ductile behaviour. It is seen that the level of the stress-strain curves rises with increasing pressure but at a reducing rate. This effect is illustrated by plotting the stress at 10 percent strain against confining pressure (Fig. 2). The gradient, tan ψ, of this plot can be taken as a measure of the pressure sensitivity of the stress-strain curve at the given strain and confining pressure (Paterson, 1967) and used in extrapolating to higher pressures (Table, p. 152).

The weakening of the talc at higher temperatures is illustrated in Fig. 3, in which the maximum stress in tests at 4 kb confining pressure is plotted against temperature; also a selection of the stress-strain curves from which Fig. 3 is derived are plotted in Fig. 4 to show the effect of temperature on their shape. Using X-ray powder diffractometry, decomposition could only be detected after the 900° C experiments; in these, some talc remained but a large part had been converted to quartz, enstatite and anthophyllite[1]. No detectable decomposition had occurred in the $^1/_2$ hour heating period at 800° C, possibly because of sluggishness of reaction since the dehydration temperature at 4 kb pressure appears to be slightly below 800° C (Kitahara, Takanouchi and Kennedy, 1966).

Unexpectedly, sealed specimens of talc did not show an abrupt weakening and embrittlement at 800–900° C associated with dehydration, such as was observed in serpentinite (Raleigh and Paterson, 1965). Instead, there is a more nearly steady rate of fall of strength with increase in temperature. Also in the tests at 400 to 800° C, the talc behaved in a fairly brittle manner; there was a sharp drop in the stress-strain curve after a few percent strain and the specimens showed single shear fractures. Another discrepancy with the observations on serpentinite is the absence, above the dehydration temperature, of any marked effect on the strength resulting from venting one end of the specimen to atmosphere. The venting was done by using a hollow piston of 1.5 mm bore but, in order to avoid extrusion of the talc, a 1.5 mm thick molybdenum alloy disk with four 0.4 mm diameter holes was interposed between the specimen and the hollow piston. Probably the venting has not been fully effective in eliminating pore pressure, either because of low initial permeability or because of reduction of permeability due to collapse of pores in the weak talc matrix as water is removed Two other factors that may be involved in the discrepancy in behaviour between talc and serpentinite are, first, that talc is much weaker than serpentinite and, second, that the amount of water involved in dehydration of talc is much less than for serpentinite (namely, 4.8 percent by weight for the reaction talc $\rightarrow$ enstatite + quartz + water compared with 11.7 percent for serpentine $\rightarrow$ forsterite + talc + water).

Further tests suggest that adsorbed water remaining after airdrying may also be important in the behaviour of the talc (the serpentinite specimens of Raleigh and Paterson had been oven-dried). Heating for an hour or so above 100° C leads to about 0.4 percent weight loss in the air-dried specimens. Tests at 600° C after this showed significantly higher strength (Fig. 3) although the specimens still developed a shear fracture after a few percent strain. This increase in strength is found even though both sets of specimens were vented using the arrangement

1 The presence of anthophyllite is inferred from clearly-defined diffractometer peaks additional to those of talc, quartz and enstatite, for spacings of 8.30, 3.66, 3.06, 2.75, 1.73 and 1.62 Å, all of which are important reflections for anthophyllite.

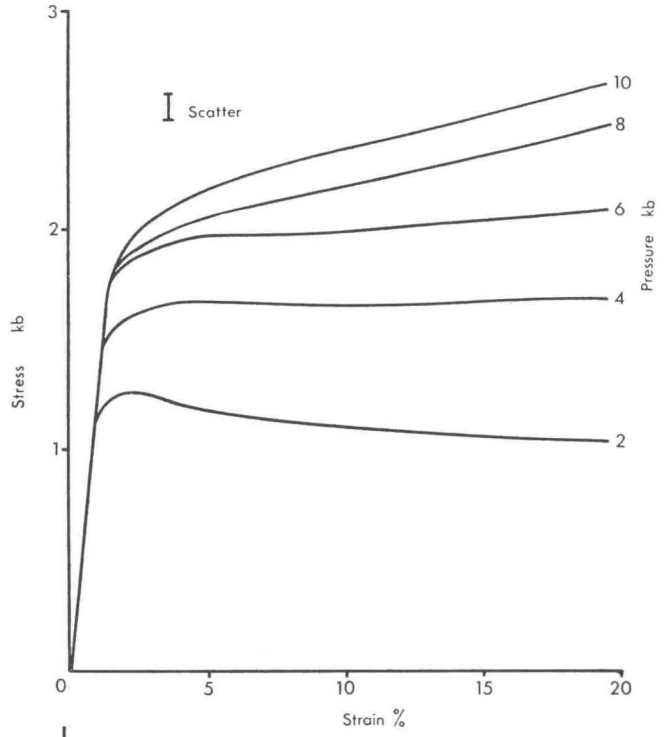

Fig. 1. Stress-strain curves for Three Springs talc at room temperature and confining pressures shown. The "scatter" band indicates the range of scatter among repeat experiments which applies approximately to all the curves shown

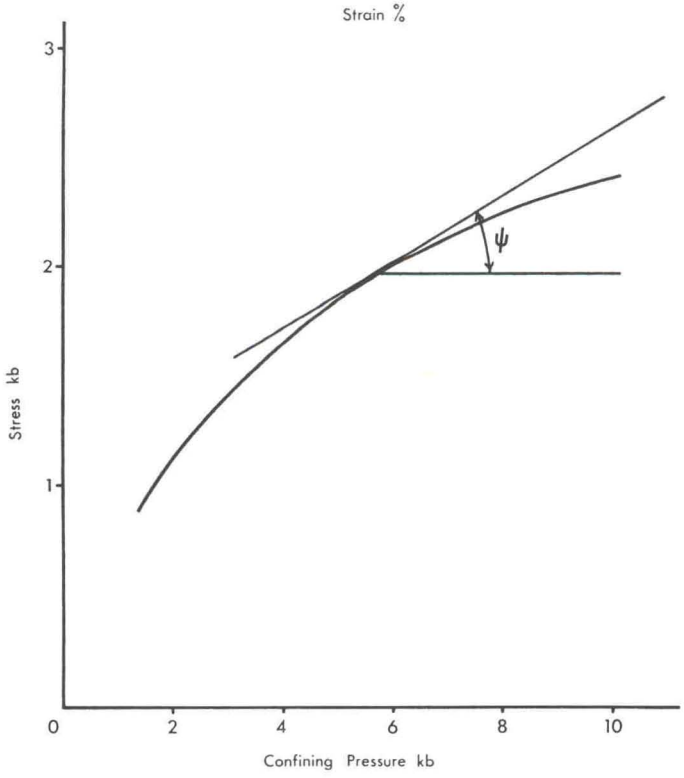

Fig. 2. Stress at 10 percent strain for Three Springs talc (Fig. 1), plotted as a function of confining pressure

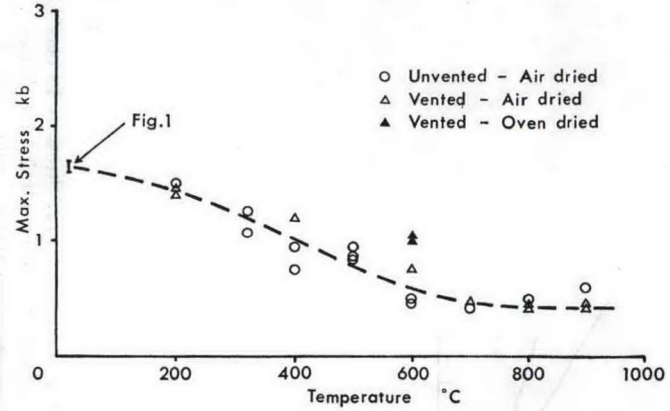

Fig. 3

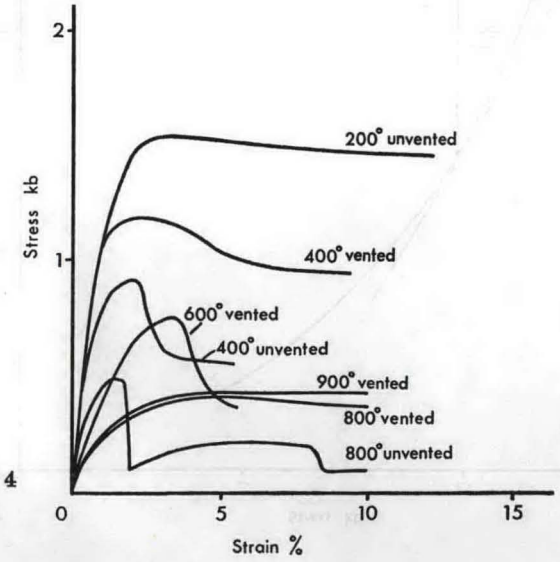

Fig. 4

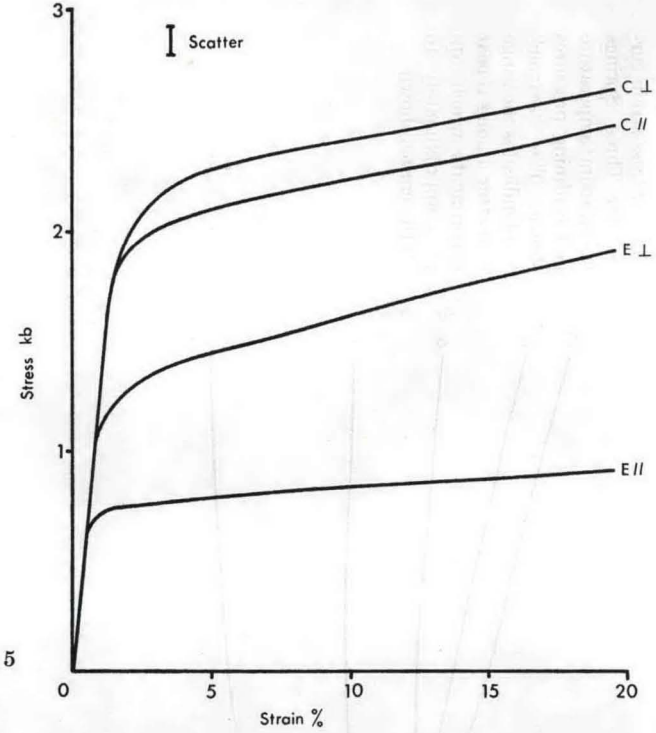

Fig. 5

Fig. 3. Maximum stress at 4 kb confining pressure for Three Springs talc at various temperatures

Fig. 4. A selection of individual stress-strain curves for Three Springs talc at 4 kb and temperatures shown. See text for details and discussion of "venting"

Fig. 5. Stress-strain curves at room temperature and 6 kb confining pressure for other talc samples (details in text)

described above (however, as in the tests above the talc dehydration temperature, it again appears that the venting is relatively ineffective since also at these lower temperatures airdried specimens gave substantially the same strengths when vented as when sealed, although venting does seem to reduce the tendency to fail on a single shear plane). If we assume that the venting is ineffective and that there is still 1.5 percent pore volume under the test conditions (as measured at 4 kb and room temperature, Edmond and Paterson, 1971a), 0.4 percent water would give rise to pore pressures of 0.1, 1.2, 2.4 and 3.5 kb at temperatures of 300, 400, 500 and 600° C, respectively, thus beginning to reduce significantly the effective pressure and hence the strength at temperatures around 400–500° C. However, these figures are very sensitive to the exact pore volume assumed and can be taken only to indicate qualitatively that the adsorbed water may be a significant factor in the observed behaviour of the talc.

(b) *Other Talc.* The stress-strain curves in Fig. 5 are from duplicate tests at 6 kb confining pressure and room temperature on cores from blocks C and E. These, together with the 6 kb curve in Fig. 1, indicate the wide range of strength that even fairly pure talc may have, depending on its origin and any preferred orientation of its crystals. The curves for block E also show how important the specimen orientation may be when there is a marked preferred orientation of grains. In this case, the grains in the stronger specimen ($E_{\perp}$) were preferentially oriented with their basal planes perpendicular to the axis of loading, while in the other specimen ($E_{\parallel}$) more of the grains had their basal planes oriented parallel to the axis of loading.

2. Pyrophyllite

The stress-strain curves are shown in Fig. 6. The change from falling to rising curves with increase in pressure again corresponded to the transition from brittle to ductile behaviour. However, the strength of the pyrophyllite was much higher and more pressure sensitive than talc (Table).

3. Silver Chloride

This was the weakest of the materials tested. The stress-strain curves (Fig. 7) showed a small but continual workhardening at all pressures and the specimens deformed uniformly except for some barrelling. There was a measurable increase in strength with confining pressure, which was quite appreciable relative to the actual stresses although in absolute terms the effect was very small (Table) and it may partly be associated with elimination of porosity during straining.

4. Sodium Chloride

At all pressures, the specimens were ductile and showed appreciable work hardening. However, within the scatter of results, no systematic effect of confining pressure on the stress-strain curve (Fig. 8) was detected; duplicate tests at 0.25, 0,5, 1, 2, 6 and 8 kb all gave curves falling within the scatter band shown.

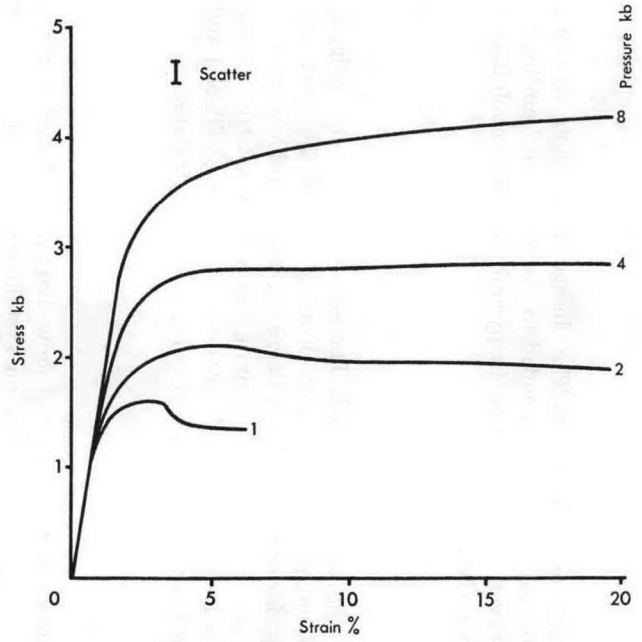

Fig. 6

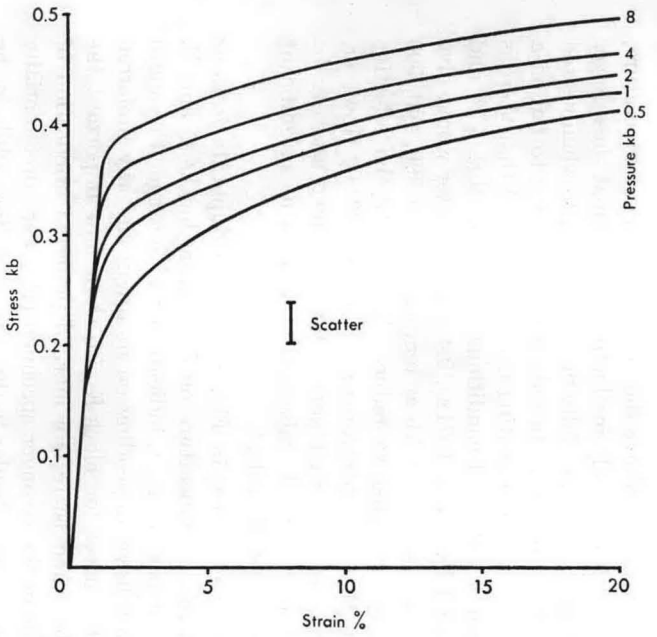

Fig. 7

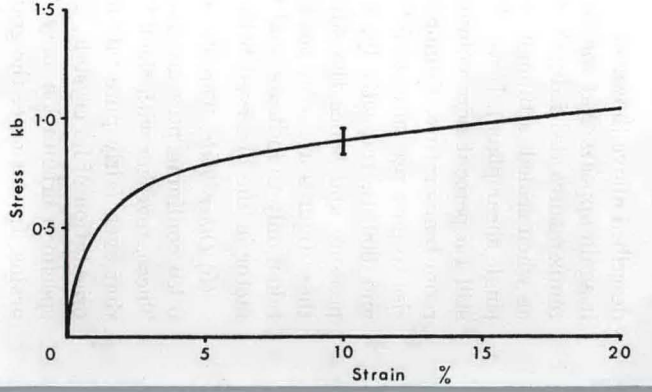

Fig. 8

Fig. 6. Stress-strain curves for pyrophyllite at room temperature and confining pressures shown

Fig. 7. Stress-strain curves for silver chloride at room temperature and confining pressures shown

Fig. 8. Stress-strain curves for sodium chloride at room temperature and confining pressures up to 8 kb (all curves fell within the scatter band indicated and no definite trend with pressure was established)

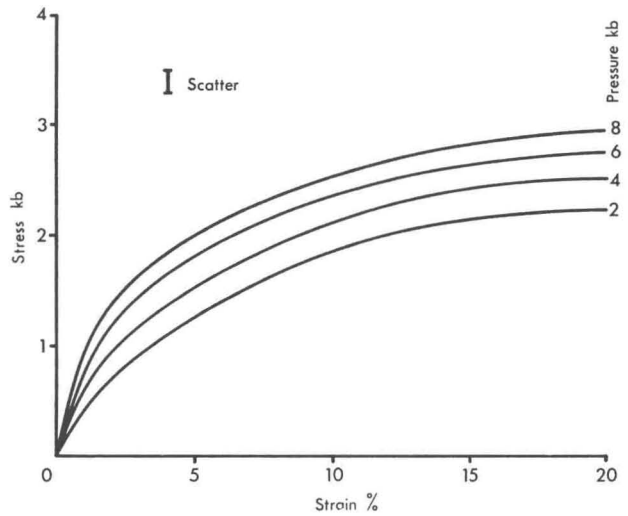

Fig. 9. Stress-strain curves for boron nitride at room temperature and confining pressures shown

5. Boron Nitride

Here also the specimens deformed uniformly and the stress-strain curves (Fig. 9) showed marked work hardening at all pressures from 2 to 8 kb. Compared to talc, the boron nitride is weaker at strains below 5 percent but of similar strength at larger strains. In other orientations, however, somewhat different strength may be expected because of the preferred orientation of the grains.

6. Graphite

The stress-strain properties of graphite under pressure show a number of peculiarities (Fig. 10). There is no obvious elastic range or yield point in the stress-strain curves at any confining pressure from 1 to 8 kb. Also, an extraordinary degree of reversibility appears in the strain, the specimens returning approximately to their original dimensions upon release of both differential stress and confining pressure even though strains of 20 percent shortening had been reached at high pressure. Thus, length measurements using the technique of Paterson (1963) on a specimen of similar graphite showed the following sequence of length changes: applying the confining pressure of 4 kb caused an initial shortening of 3.5 percent; after 20 percent further shortening at 4 kb as a result of applying differential load, removal of the differential load was accompanied by a length recovery of 7.5 percent; finally, the length recovered a further 14 percent during release of the confining pressure, resulting in the final specimen length being only slightly less than the initial. Other specimens deformed 20 percent at 2 to 8 kb confining pressure gave similar recoveries on release of pressure. Similar changes occur in the volume; these and their interpretation are discussed elsewhere (Edmond and Paterson, 1971a and 1971b).

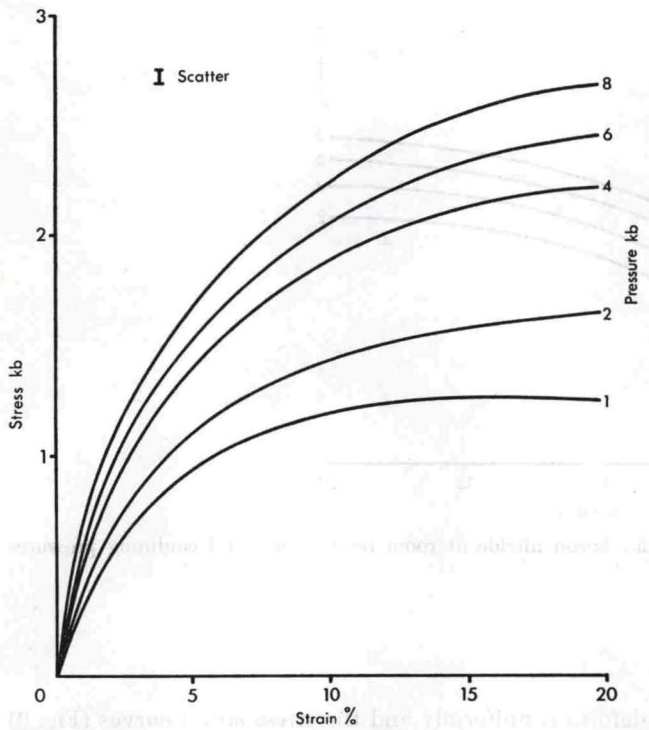

Fig. 10. Stress-strain curves for graphite at room temperature and confining pressures shown

Discussion

It is seen that, even at room temperature, all the materials tested can undergo substantial permanent deformation when subjected to differential stress under moderate confining pressures. However, there are considerable differences in the amount of stress needed and in the influence of confining pressure on it. Thus, at 8 kb confining pressure, the strongest material, pyrophyllite, will support differential stresses an order of magnitude higher than will silver chloride, and the effect of pressure on the flow stress is also an order of magnitude gigher.

The differences in pressure sensitivity of the flow stress probably reflect differences in deformation mechanism. For a polycrystalline body to deform uniformly by crystallographic slip in its grains, five independent slip systems are required (von Mises, 1928; Groves and Kelly, 1963; Paterson, 1969). This condition is satisfied in silver chloride which deforms by pencil glide in the [110] direction (Nye, 1949) and it is probably satisfied by sodium chloride through activity of (100) as well as the usual (110) slip planes under the constraint of the confining pressure. Then, the absence of a strong effect of pressure on the stress-strain curve in these materials can be attributed to their deforming entirely by slip in the grains, a process which is known normally to be only slightly affected by pressure.

On the other hand, talc, pyrophyllite, boron nitride and graphite have layer structures with strong bonding within the layers and slip can only be expected

to occur readily parallel to the layering. Such slip can, at most, only contribute two independent slip systems and so additional deformation mechanisms are needed for the polycrystalline bodies. Kinking can meet some of the additional requirement but probably not all (Paterson, 1969). However, crystallographic slip on non-basal planes may be difficult to achieve. In this case, in the absence of effective diffusion mechanisms at room temperature, it can be expected that cataclastic mechanisms, that is, fracturing or grain boundary parting and relative movement of grains, will also be involved in the deformation. Because of the friction and volume changes associated with cataclastic flow (c.f. soils) this will introduce substantial pressure sensitivity in the stress-strain curve. The observation of higher pressure sensitivity of flow stress in these materials can probably therefore be taken as evidence for some cataclasis being involved (Edmond and Paterson, 1971 a). However, in the case of talc at the higher pressures used here, the pressure sensitivity is only marginally higher than for fully plastic behaviour in materials such as copper, which suggests that nonbasal slip systems may then be contributing to the deformation.

Application to Solid Pressure Media

1. Strength of the Media

The capacity of the medium in a piston-cylinder apparatus to support a shear stress affects its practical application in several ways. Thus, error is introduced in calculating pressure from the piston load and cross-sectional area. Also, the specimen is subjected to non-hydrostatic stress. Further, the conditions tend to be affected by relaxation during the experiment and by variations in details of technique such as whether the pressure is approached from above or from below. We shall now discuss these factors in the light of the present experimental results.

Extrapolation to higher pressures of the stress differences supportable by the various media at room temperature involves uncertainties because of the non-linear dependence on pressure, but for moderate extrapolation, say to 25 kb, fairly firm limits can be estimated. A lower limit is set by assuming no further increase with pressure beyond 8 or 10 kb (actually, the flow stress is more likely to be raised at least in the same proportion as are the elastic constants). An upper limit is set by assuming a rate of increase with pressure equal to that at the upper end of the present range of observation. These two limits are given in the first and third columns of Table for a pressure of 25 kb and 10 percent strain.

Except in the case of talc, all the measurements have been done at room temperature but they should still give a useful guide to behaviour in a high-temperature apparatus because much of the pressure medium in such an apparatus is at a much lower temperature than the specimen. In a typical apparatus with specimen temperature around 1000° C, the temperature near the outer diameter, in the region making up the larger part of the cross-sectional area, is probably not above 200–400° C. In the case of talc and pyrophyllite, the measurements on talc (Fig. 3) indicate that at such temperatures, the stress difference supported by the medium will not be substantially below what it is at room temperature. The strengths of graphite and boron nitride are likely to be little affected even by the furnace

Table. *Extrapolated strength limits at 25 kb (10 percent strain)*

Material	Flow stress at 10% strain at 8 kb[a] = lower limit	Tan ψ at 10% strain at 8 kb[a]	Extrapolation to 25 kb using tan ψ at left = upper limit
Three Springs Talc	2.4 kb	0.08	3.6 kb
Pyrophyllite	4.0	0.21	7.5
Silver Chloride	0.5	0.01	0.7
Sodium Chloride	0.9	<0.01	1.0
Boron Nitride	2.6	0.10	4.3
Graphite	2.2	0.06	3.3

[a] 10 kb for talc.

temperature because of their extremely high melting points (at atmospheric pressure, graphite actually increases in short-term strength up to 2500° C; Riley, 1967). On the other hand, silver chloride and sodium chloride, having relatively low melting points, can be expected to show appreciable weakening at 200–400° C and so the room-temperature figures could give a substantial over-estimate of their strengths in a high-temperature apparatus.

2. Nominal Pressure Correction

As mentioned in the Introduction, most workers using piston and cylinder apparatus of the Boyd and England (1960a) type determine the pressure at the sample by applying a correction, often called the "friction correction", to the nominal pressure (load divided by piston area). It is generally agreed that several factors contribute to this correction and it is convenient for discussion to distinguish the following (c.f. Tamayama and Eyring, 1967):

(a) Stress Gradient in the Sample Region. This mainly concerns the sample itself and any other medium inside the graphite furnace. Since the dimensions of this region are generally small relative to the overall dimensions of the high pressure volume, stress gradients within it can probably be neglected for the nominal pressure correction, especially at high temperatures where the strengths are low.

(b) Stress Gradient in the Pressure Media. Here we consider the contents of the high pressure cylinder apart from the sample region, that is, in the simplest case, the graphite furnace and surrounding medium such as talc. The ability of these materials to support shear stress allows the axial stress component to differ from the radial stress component adjacent to the sample region. Moreover, the radial and, therefore, the axial stress can increase or decrease at larger radii, depending on the loading history and boundary conditions, analogously as in a thickwalled tube under external or internal pressure. Thus, averaging the axial stress gives a nominal pressure that differs from the pressure in the sample region, the latter being given by the radial stress at the boundary of the sample region. It is very difficult to calculate precisely the stress distribution in the pressure cell because of the non-linear stress-strain properties of the media, the volume changes that occur during their deformation, the difference in properties of the components

(such as talc and graphite), the complicated boundary conditions arising from shearing resistance at the outer diameter and the influence of loading history. However, if the shearing resistance at the outer boundary is ignored for the present (it is considered separately below), a rough estimate can be made in the case in which the final pressure is approached from below.

Consider the idealized situation of a sample region of negligible shear strength surrounded by concentric cylindrical sleeves of graphite and another medium such as talc. Since the strengths of graphite and talc may be comparable, we assume them to be equal, as a first approximation. Further, we assume that, in the final pressure increment, yielding occurs throughout the pressure cell but that changes in volume and strength of the media during this increment are negligible. Then we have a relatively simple model in which the stress distribution can be calculated with the aid of the theory of plasticity. For a variety of yield and boundary conditions, this can be expected to lead to a nominal pressure correction that is proportional to the strength of the media, that is,

$$p = p_n - C\sigma_0$$

where

p pressure in sample region,
p_n nominal pressure applied to pressure cell (neglecting piston-cylinder and cylinder-medium friction),
σ_0 stress difference that the medium supports at the appropriate pressure in the tests of the described above (c.f. uniaxial yield stress in plasticity theory),
C a constant depending on the geometry of the pressure cell and on details of the yield criterion and boundary conditions.

If the axial stress at all points outside the sample region is assumed to exceed the pressure in the sample region by an amount σ_0, C will have the value $1 - a^2/b^2$ where $2a$ is the diameter of the sample region and $2b$ the inside diameter of the pressure vessel. A more detailed calculation (Appendix), assuming a rigid pressure vessel and von Mises yield criterion for the media, gives an expression for C which is a more complicated function of a/b; this is plotted in Fig. 12, from which it is seen that C is nearly unity for the range of values of a/b likely to be important in practice. It is also shown in the Appendix that allowing for the elastic distortion of the pressure vessel would still give the same form for C as that plotted in Fig. 12 but the relevant value of C would correspond to a larger value of a/b than that for the actual pressure cell; that is, in general, the value of C may be somewhat smaller than suggested by Fig. 12 (the more so for a steel vessel than for a carbide one) but it will probably still be not much less than unity for relevant values of a/b.

Thus, in a pressure vessel consisting of talc and graphite in which the average stress difference supported by these media is approximately 1 kb (that is, shear strength about 0.5 kb) and in which the sample region is not larger than about two-thirds of the diameter of the cell, the correction to the nominal pressure for the strength of the media alone should be approximately 1 kb. This figure may be expected to apply very roughly to many practical situations using talc at pressures of 20–30 kb and not to increase very much with pressure in this range.

If, contrary to the assumption above, the graphite has negligible strength (for example, if it is weakened by the pressure of water from decomposing talc), it

can be regarded as part of the sample region. Even then, the ratio a/b for the talc alone may still be in the range (Fig. 12) for which C is only slightly less than unity and so a similar correction would apply. On the other hand, for silver chloride and sodium chloride media, it follows from the present strength measurements that, as would be expected, the correction will be small compared with that for talc, although some correction may still be needed for the graphite furnace. In any case, the considerations in this section only apply when the final pressure is approached from below ("piston in"); the irreversibility is discussed in the comments below. Also, the compressibility of the medium has been ignored, although this is probably not serious at the level of approximation concerned here for "piston in" conditions.

(c) Shearing between Pressure Medium and Cylinder Wall. Since the coefficient of friction for sliding between the pressure medium and the wall of the pressure vessel is unlikely to be less than about 0.1, even with molybdenum disulphide lubrication, the shear stress parallel to the wall will generally tend to exceed the shear strength of the medium at pressures above 10–20 kb (and at much lower pressures in the case of silver chloride or sodium chloride). Therefore, we can usually take the shearing resistance at the wall to be equal to the shear strength of the medium, that is, about one-half of the stress difference σ_0 that the medium will support according to our tests, appropriately extrapolated. Thus the total shearing force required for shearing at the wall over a length l of the cylinder from the end of the piston to the level of the sample (usually l is about one-half of the total length of the pressure cell) is $2\pi b l \cdot {}^1/_2\sigma_0$. This corresponds to an increase in nominal pressure of $\frac{\pi b l \sigma_0}{\pi b^2} = \frac{l}{b}\,\sigma_0$, approximately equal to σ_0 in most practical cases. Thus, the nominal pressure correction for shearing at the cylinder wall should be approximately equal to the correction for stress gradient in the medium, namely, about 1 kb with talc at 20–30 kb pressure and not increasing much with pressure. If a thin layer of lead is used as a "lubricant", the shearing will occur in the lead, for which Bridgman (1935) gives a shear strength of 0.3 to 0.4 kb (that is, $\sigma_0 = 0.6$ to 0.8 kb) at pressures of 20–30 kb; the correction for shearing at the wall is then a little less than with talc alone but the difference is not appreciable.

(d) Friction between Piston and Cylinder. This will depend on the materials and clearances used in construction and on the use of anti-extrusion rings and lubricants. Our experience with 10 kb fluid medium apparatus, made from steel and using an anti-extrusion ring and O-ring packing lubricated with molybdenum disulphide grease, is that the friction is about ${}^1/_2$ to 1 percent (occasionally as high as 2 percent) of the load on the piston, provided that the clearances are large enough to avoid binding in the cylinder. This situation is rather similar to that in solid medium apparatus, suggesting a correction for piston friction of about 1 percent, although this could vary somewhat from apparatus to apparatus and it may not be strictly linear with pressure. Thus the correction to the nominal pressure for piston-cylinder friction would be 0.2 to 0.3 kb for 20 to 30 kb pressure.

Comments. These estimates are subject to considerable uncertainty and it may well be fortuitous that they add up to about 2.2 kb, roughly equal to the 10 percent correction suggested by Green *et al.* (1966), for pressures around 20 to 25 kb in talc. In particular, a gross approximation has been made in ignoring the inter-

action between the second and third factors since the shearing forces at the wall will perturb the stress distribution from what was considered under (b). Also, some relaxation of the stresses can be expected over long periods. Nevertheless, the results probably have some value in suggesting the relative importance of the above factors, that is, roughly equal contribution from stress gradients in the medium and from shearing at the wall, neither being strongly sensitive to pressure, while the piston friction is probably more nearly proportional to pressure and only predominant if the pressure media are much weaker than talc.

So far, we have only considered the case where the piston is advancing. If the final pressure is approached from above, it appears to be more difficult to relate the nominal pressure to the pressure at the specimen. The stress distribution in the medium can no longer be calculated from the theory of plasticity since the final approach to the working conditions will largely involve elastic changes and so the stress distribution will be sensitive to the elastic properties of the various materials in the pressure cell as well as of the cylinder. The situation will certainly not be the reverse of that of approach from below and so in neither case can the correction be obtained as half the difference in nominal pressures for a given transition determined by approach from either direction. The same remarks apply to a large extent to the effect of shearing at the wall. Even the piston friction probably cannot be obtained by reversal because our experience with fluid medium apparatus indicates that a considerable amount of movement, perhaps several tenths of a millimeter and in excess of the elastic range of the solid pressure medium, is needed before the friction settles down to its normal value.

3. Non-Hydrostatic Conditions

The presence of shear stresses in the medium implies a non-hydrostatic state of stress in the sample. This has been investigated experimentally by Lees and McCartney (1968) in a tetrahedral anvil type of apparatus. The magnitude of shear stress in the sample will be limited by its strength or by the strength of the immediately surrounding medium, whichever is the weaker, and when the temperature is high and duration of experiment long it will often be small compared with the hydrostatic component of the stress. On the other hand, it may sometimes be important to bear in mind that the specimen will have undergone some deformation in the course of reaching the operating conditions.

4. Effects in Stress-Strain Apparatus

Griggs (1967) has introduced the use of solid pressure media in apparatus for rock deformation. The confining pressure is generated in the same way as above and the differential load for the deformation of the specimen is produced by introducing an additional small piston concentrically with the confining pressure piston. To allow for friction the force needed to advance the differential load piston prior to contact with the specimen is recorded and increase in force beyond this is taken to be differential load applied to the specimen. However, during axial shortening of the specimen, pressure medium must be displaced radially. To achieve this, the radial component of stress in the medium immediately surrounding the specimen must be augmented to such an extent as to reverse the stress gradients initially

set up in the pressure medium during advance of the confining pressure piston; that is, during the deformation the confining pressure effective at the specimen will rise above the indicated confining pressure and the differential stress effective at the specimen will be less than that indicated above by an amount of the order of $4\sigma_0$ (instead of the confining pressure being about $2\sigma_0$ below the nominal confining pressure due to the combination of factors discussed above (2.), it will have risen to $2\sigma_0$ above). In the case of a talc medium at moderate temperatures and at pressures of the order of 20 kb, this therefore suggests an additional correction to the differential stress which will rise to a value of the order of 4 kb in the course of straining the specimen.

This estimate of the additional correction to the differential load for stress gradients in the medium and shearing at the wall is clearly a very rough one. Apart from the approximations involved in the plastic model, the assumed geometry and the extrapolated material properties, it will also be sensitive to experimental procedure. If the confining pressure is raised in such a way that the final increment is achieved by heating rather than by advance of the confining pressure piston, or if the nominal confining pressure is approached from above, the reversal of stress gradients considered above may already have occurred, at least to some extent, and so this correction will have been eliminated or reduced in amount. The influence of whether or not the graphite supports similar shear stresses as the talc and of the relative diameters of specimen region and cylinder bore should be determined by the geometrical factors discussed above under 2.

Carter, Christie and Griggs (1964) have noted extension fractures in quartz specimens after deformation in a solid medium apparatus which they attribute to stresses from expansion of the graphite furnace during pressure release. This explanation is supported by the present observation of unusually large strain recovery in graphite during pressure release.

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Appendix: Approximate Calculation of Nominal Pressure Correction in Piston-Cylinder Apparatus due to Stress Gradients in the Medium

We consider the simple model shown in Fig. 11 in which all correction factors other than the strength of the pressure medium are neglected and the pressure medium itself is assumed to fill uniformly all space outside the sample region and to have the properties of a homogeneous perfectly plastic material. In particular, we consider the part AB which is, in effect, a hollow cylinder that we assume to be undergoing uniform shortening axially, that is, to be undergoing generalized plane strain. Thus the boundary conditions are uniform relative displacement of the ends A and B, zero radial displacement at the outside diameter (pressure vessel assumed rigid), and radial stress component equal to pressure p at the inside diameter (note that we are not considering any effect from wall friction at the outside diameter in this calculation; see above).

A substantial amount of strain is supposed to have occurred throughout the medium while raising pressure because of the elimination of misfits and porosity

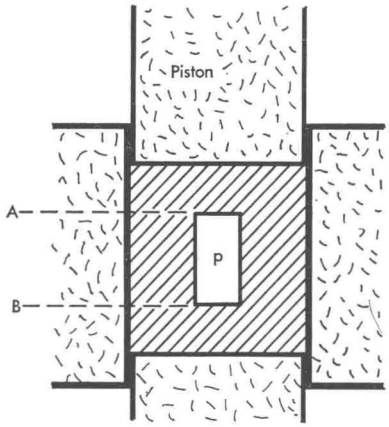

Fig. 11. Simplified model for piston-cylinder high pressure apparatus

in the assembly as well as the truly elastic compression of the parts. We therefore assume that all parts of the medium will have yielded and we consider the situation in the last pressure increment by which the final pressure is attained. During this increment, all parts of the medium are therefore assumed to be yielding plastically but without significant volume change, and we can then calculate the stresses in it using the theory of plasticity. The inside and outside diameters are taken to be 2a and 2b, respectively, and we use conventional cylindrical coordinates r, θ, ζ where ζ is directed along the apparatus axis. Following the same procedure as in Hoffman and Sachs (1953, p. 90–93) but using the above boundary conditions, it can be shown that the axial compressive stress component σ_ζ is given by

$$\sigma_\zeta = p + \frac{\sigma_0}{\sqrt{3}}\left(\frac{1+3r^2/b^2}{\sqrt{1+3r^4/b^4}} + \coth^{-1}\sqrt{1+3a^4/b^4} - \coth^{-1}\sqrt{1+3a^4/b^4}\right)$$

if one assumes that yielding is controlled by the von Mises criterion, σ_0 being the uniaxial yield stress. The Tresca yield criterion is difficult to apply in this situation but in general can be expected to give a result not more than about 15 percent different.

The total force to be applied by the piston at the face A of the pressure cell is then

$$F = \pi a^2 p + \int_a^b \sigma_\zeta \, 2\pi r \, dr$$

and so the nominal pressure p_n is

$$p_n = \frac{F}{\pi b^2} = \frac{a^2}{b^2} p + \frac{2}{b^2}\int_a^b \sigma_\zeta \, r \, dr.$$

Substituting the above expression for σ_ζ and carrying out the integration gives

$$p = p_n - C\sigma_0$$

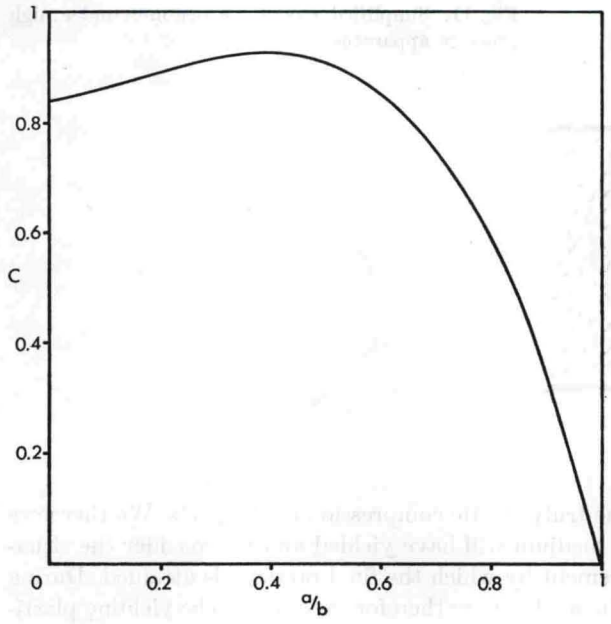

Fig. 12. Correction factor C as a function of diameter ratio a/b

where

$$C = \frac{1}{\sqrt{3}}\left[2 - \coth^{-1} 2 + \frac{a^2}{b^2}\left(\coth^{-1}\sqrt{1+3\frac{a^4}{b^4}} - \sqrt{1+3\frac{a^4}{b^4}}\right)\right].$$

Thus $C\sigma_0$ is the correction to be subtracted from the nominal pressure, where C is a geometrical factor, plotted as a function of a/b in Fig. 12, and σ_0 is the uniaxial compressive yield stress, which for a perfectly plastic material will be the same as the differential stress at yield in the high pressure tests reported in this paper. Only a slightly different result would be expected if the Tresca yield criterion were used.

This calculation can also be extended to take into account the change in bore diameter of the pressure vessel. Thus, if we take as boundary condition the radial displacement at the radius b to be $u_r = Kbp$ instead of zero (K is a constant involving the geometry of the pressure vessel and the compliance of its material), we now get

$$p = p_n - C'\sigma_0$$

where

$$C' = \frac{1}{\sqrt{3}}\left[2 - \coth^{-1} 2 + \frac{a^2}{\beta^2}\left(\coth^{-1}\sqrt{1+3\frac{a^4}{\beta^4}} - \sqrt{1+3\frac{a^4}{\beta^4}}\right)\right]$$

and

$\beta^2 = b^2\left(1 + 2K\frac{dp}{d\varepsilon_3}\right)$, ε_3 being the axial strain. That is, the expression for the correction is the same as before but with β substituted for b. Since K is positive and $\frac{dp}{d\varepsilon_3}$ negative, $\beta^2 < b^2$ always, and so the value of C' will correspond to a point to the right of the abscissa a/b in Fig. 12.

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