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Precompressed Pyrophyllite for Ultrahigh Pressure Research*

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Pyrophyllite, a mineral used for high pressure gasketing, exhibits wide sample-to-sample variations in density and in mechanical properties. Samples of varied initial densities, after compressions to uniform density, are found to have uniform mechanical properties in subsequent use. Large improvements in work hardening response of tungsten carbide anvils are obtained through use of precompressed pyrophyllite gaskets.

INTRODUCTION

PYROPHYLLITE is widely used both as the anvil-supporting material and as the pressure transmitting medium for Drickamer type[1] "supported taper" pressure apparatus. For pressures above 300 kilobars the inhomogeneity in density in pyrophyllite causes uncertainties in producing experimental results. In this laboratory, we have found that by precompressing the inhomogeneous pyrophyllite obtained from the suppliers, a nearly homogeneous pyrophyllite can be produced. In addition to having a uniform density the precompressed material has uniform mechanical properties. Improvements in the work hardening characteristics of tungsten carbide anvils are obtained through the use of precompressed pyrophyllite gaskets.

EXPERIMENTAL

Grade A pyrophyllite blocks of 30×30×15 cm were purchased from American Lava Company (two, blocks, labeled as A and B) and Maryland Lava Company (two blocks, labeled as M and X). About 700 disks of 2.223 ±0.0005 cm diameter and 0.466 cm±0.002 cm thickness were machined from each of the four pyrophyllite blocks. The variation in thickness of each individual disk was less than 0.001 cm. The densities of the disks were calculated from their weight and their dimensions; the average values were 2.670, 2.705, 2.675, and 2.662 g/cm³, ±0.005 g/cm³, for disks made from A, B, M, and X pyrophyllite, respectively.

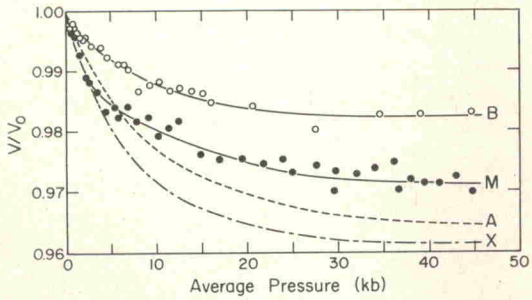


FIG. 1. Relation between V/V_0 and the average applied pressure for the A (– – –), B (–○–), M (–●–), and X (–·–) pyrophyllite disks.

Compression of a pyrophyllite disk was achieved by placing the individual disk in the cell of K68 Kennametal of 2.225 cm i.d. and compressing it between two flat pistons of GE Carboloy 999 of 2.223 cm diameter. MoS_2 powder was used as a lubricant on the lateral surface of the disk and the inner wall surface of the cell. The applied load was varied from 0.9 to 181 metric tons. Each disk was compressed at one predetermined load for 2 to 5 min. The disk must be forced out of the cell after compression. No damage on the surface of the disk was observed when lubrication was provided.

As a result of the compression the thickness and volume of the disks decreased and their diameter increased to a degree dependent upon the maximum load applied. The ratio V/V_0, where V and V_0 are, respectively, the volume of the disk after and before compression, is plotted against the average pressure P for each group of disks as shown in Fig. 1. At high pressures the density of all the disks appears to be approaching a uniform value of about 2.75 g/cm³. This is to be compared with the specific gravity of 2.80 g/cm³ given by Strong.[2] It was also observed that, at a given loading, V/V_0 increased almost linearly with V_0. This is shown in Fig. 2. This indicates that the thinner the disk, the smaller the load needed to compress the disk to a higher density.

The compressed disks were observed to become darker than the original ones. The degree of darkness increased

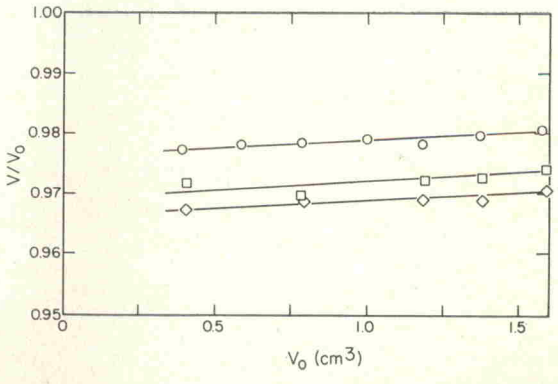


FIG. 2. Relation between V/V_0 and V_0 for M pyrophyllite disks at constant average pressures of 12 (○), 23, (□), and 45 kilobars (◇). V_0 is the volume of an uncompressed disk and V is the volume of the disk after compression.

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with the load applied. The compressed disks were easier to machine than the uncompressed disks. No fracture was observed on any of the disks studied up to the maximum load of 181 metric tons.

When Drickamer type high pressure apparatus is used, the supporting pellets are most essential. The degree of the work hardening of the pistons and the reproducibility of the calibration points of the apparatus are dependent upon both the geometry and the density of the pellets.

Compressed disks of about the same density were machined into pellets of proper geometry[1] and the pellets were then used for work hardening of pistons and for specimen investigations. We have used pistons with a taper of 18° (relative to the anvil flat) and supporting taper pellets with a taper of 17.3°. It was found that the degree of work hardening and hence the volume of the work hardened region in pistons varied with the density of the pyrophyllite. Pellets made from pyrophyllite of density of about 2.65 g/cm³ could only be used to work harden pistons (GE Carboloy 999) at a maximum applied load of 18 metric tons; at higher loads the pistons fractured after load was removed. A maximum load as high as 99.8 metric tons could be used to work harden the pistons when using the precompressed pyrophyllite pellets with density of about 2.75 g/cm³. Figure 3 shows the extent of the work hardened region in the pistons for two such cases.

The relative deformation δ of the pistons (the tapered region of the piston is exactly the same as in Ref. 1), defined as the depth at the center of the flat with respect to the edge of the flat after work hardening, has been measured by using the optical microscope. The maximum value obtained is 0.05 mm$\pm$0.005 mm. It is observed that the tapered portions of work hardened pistons may break into several small pieces, when δ is large enough, and a sudden release of the stored energy of cold work occurs. The chance of a piston (with the geometry specified as in Ref. 1) breaking is very small if $\delta \leqslant 0.04$ mm. Generally, in order to work harden a pair of tapered pistons effectively, an optimum δ must be reached. This depends upon the material of the pistons, the geometry of the pistons, the geometry of the supporting pellets, and the density of the pyrophyllite from which the pellets are made. The task of optimizing δ is greatly simplified if the latter variable is removed by the precompression process.

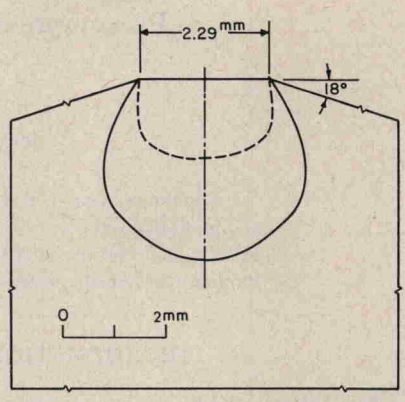


FIG. 3. Extent of plastic zone attained during work hardening. (This is the first clearly visible zone nearest to the flat of the piston.) —— for pistons work hardened at 25 kilobars average pressure, – – – for pistons work hardened at 6.5 kilobars average pressure.

Another important factor for using compressed pyrophyllite is that once a right combination of the density and geometry of the pellets is discovered through trial and error, reproducible results are constantly obtainable. Using pellets of density of about 2.75 g/cm³ and pistons of GE Carboloy 999, work hardened at between 81 and 90.7 metric tons of applied load, the Pb transition was constantly obtained at 22.7–26.31 metric tons of applied load, the Fe transition at 20.87–23.59 metric tons, and the 19.9 wt% Co–Fe transition[3] at 36.3–39.01 metric tons.

ACKNOWLEDGMENT

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[1] H. G. Drickamer and A. S. Balchan, in *Modern Very High Pressure Techniques*, R. H. Wentorf, Ed. (Butterworths and Co., Ltd., 1962), p. 37.

[2] H. M. Strong, in Ref. 1, p. 95.

[3] F. P. Bundy, J. Appl. Phys. **38**, 2446 (1968).