Apparatus

Reprinted from THE REVIEW OF SCIENTIFIC INSTRUMENTS, Vol. 34, No. 1, 45–47, Januar
Printed in U. S. A.

Pressure Distribution and Hysteresis Effects in a Tetrahedral Anvil Device

B. C. DEATON AND R. B. GRAF
Applied Research, General Dynamics/Fort Worth, Fort Worth, Texas
(Received 24 October 1962)

Measurements have been made on the distribution of pressure and hysteresis effects in a tetrahedral anvil apparatus by simultaneously monitoring the resistances of calibration wires placed at various positions in the pyrophyllite tetrahedrons. A pressure gradient of about 1% is observed, and it is therefore suggested that pressure calibration wires may be placed on the tetrahedron face to determine the pressure throughout the tetrahedron volume. This technique will allow pressure calibration when sample porosity is not known and could possibly permit pressure calibration at elevated temperatures.

I. INTRODUCTION

THREE kinds of high pressure devices are in common use for generating static pressures in excess of 20 kilobars. These are the piston-cylinder devices patterned after Bridgman's early work,[1] opposed piston assemblies employing the principle of massive support[2] (from which the belt[3] was developed), and multiple anvil apparatus such as that first used by Hall.[4] This paper presents measurements made with a tetrahedral anvil device similar to the one described by Hall.[4] Measurements were made concerning the distribution of pressure in the sample tetrahedrons and also hysteresis effects observed because of the pressure transmitting media and the pressure configuration used. It has recently been reported that very large pressure gradients exist in the wafer-type samples used in opposed-anvil devices,[5] and it was thus felt that an investigation of the pressure distribution in the tetrahedral anvil equipment would be profitable.

The present apparatus consists of four 7-in. hydraulic rams having 0.78-in. triangular tungsten carbide anvils which are driven together simultaneously on a pyrophyllite sample holder having an edge length of 1 in. The high pressure equipment has been calibrated using the resistance phase transitions which occur in bismuth, cerium, thallium, cesium, and barium. The highest pressure obtainable is near 70 kilobars on the new pressure scale.[6] The solid pressure transmitting medium, pyrophyllite, is a hydrous aluminum silicate obtained from the American Lava Company.

II. EXPERIMENTAL TECHNIQUE

The distribution of pressure in the pyrophyllite sample holder was investigated by placing a calibration wire of bismuth, thallium, or barium at various places throughout the tetrahedron while the resistance of another wire at the center of the tetrahedron was monitored simultaneously. The resistance discontinuities corresponding to the Bi I–II, Ba II–III, and Tl II–III phase transitions served to indicate pressure values at the two positions in the tetrahedron. Sample wires with 0.025-in. diameter and about 0.25-in. length were placed at various depths in the face or edge of the tetrahedron and monitored along with the wire through the center of the tetrahedron. Figure 1 is a cross section of a tetrahedron showing several of the sample positions used. The bismuth, barium, and thallium wires were placed in $\frac{3}{32}$-in. silver chloride sleeves so that a nearly hydrostatic environment could be achieved. The tetrahedrons were coated heavily with iron oxide after assembly and were then dried for several hours at 90°C in a vacuum oven.[7] About ten runs were made with each configuration studied.

III. RESULTS

Figure 2 illustrates a typical run showing the resistances of two bismuth calibration wires plotted against the oil pressure on a 7-in. ram in psi. One of the bismuth wires was placed in a silver chloride sleeve through the center of the tetrahedron and the other on the tetrahedron face in a silver chloride sleeve which was in contact with the tungsten carbide anvil. For increasing pressure it is seen that the Bi I–II and Bi II–III transitions occur at the same value of ram pressure for both wires within roughly 1%. This indicates that the gradient in pressure is very small between the center and face of the tetrahedron. Twelve runs with wires at various depths in the face

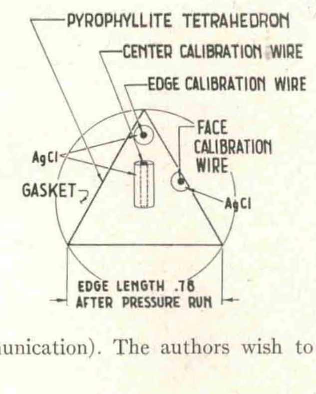

FIG. 1. Cross section of a pyrophyllite tetrahedron showing several of the sample positions used.

[1] P. W. Bridgman, *The Physics of High Pressure* (G. Bell and Sons, Inc., London, 1949).
[2] P. W. Bridgman, J. Appl. Phys. **12**, 461 (1941).
[3] H. Tracy Hall, Rev. Sci. Instr. **31**, 125 (1960).
[4] H. Tracy Hall, Rev. Sci. Instr. **29**, 267 (1958).
[5] M. B. Myers, F. Dachille, and R. Roy, Am. Ceram. Soc. Bull. **41**, 225 (1962).
[6] G. C. Kennedy and P. N. LaMori, J. Geophys. Res. **67**, 851 (1962).

[7] H. Tracy Hall (private communication). The authors wish to thank Dr. Hall for this suggestion.

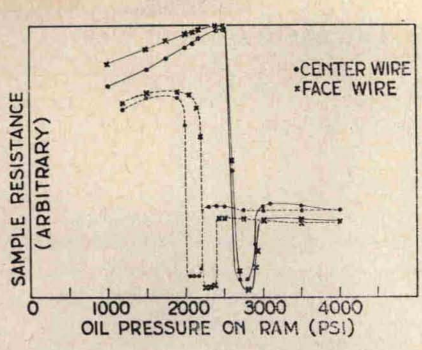

FIG. 2. The resistances of two bismuth calibration wires vs the oil pressure on a 7-in. ram in psi. One of the wires is through the center of the tetrahedron, while the other is on the tetrahedron face.

showed an average pressure gradient of less than 0.3 kilobars at the pressure of the Bi I–II transition (25 kilobars). When the pressure on the tetrahedron is decreased, it is seen that the center calibration wire exhibits about 30% hysteresis, as is generally observed for this configuration.[4] The calibration wire which is entirely outside the pyrophyllite shows considerably less hysteresis and provides a direct measure of the amount of pressure which is held by the pyrophyllite.

The behavior of the resistance of two barium wires placed simultaneously in a tetrahedron is shown in Fig. 3 as a function of ram pressure. One of the wires is through the center of the tetrahedron, the other is buried about $\frac{1}{16}$ of an inch in the tetrahedron face and is entirely surrounded by pyrophyllite. The wire in the face exhibits the Ba II–III transition at a slightly lower ram pressure than the center wire. The average pressure gradient for the barium runs was found to be 0.8 kilobars at the pressure of the Ba II–III transition (59 kilobars). No hysteresis difference is observed between the two wires in Fig. 3 because the barium face wire was completely surrounded by pyrophyllite.

Several runs were made with bismuth wires at various depths in the edge of the tetrahedron. Again very little pressure gradient was observed until the samples were placed near the edge of the gasket which is formed by the pyrophyllite between the anvils. At the gasket edge, the pressure was found to be about 70% of the value at either the center or face of the tetrahedron. As would be expected, the wire at the gasket edge showed very little hysteresis.

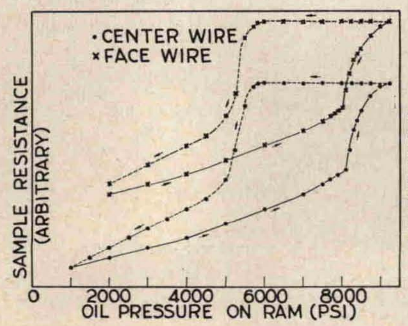

FIG. 3. The resistances of two barium calibration wires vs ram oil pressure in psi. One of the wires is through the center of the tetrahedron, while the other is buried in the tetrahedron face.

Figure 4 is a plot of the data on hysteresis phenomena obtained from the two-wire runs. The hysteresis in kilobars is plotted as a function of sample pressure. One curve shows the hysteresis for samples of bismuth, thallium, and barium at the center of the tetrahedron, while the other curve gives the hysteresis for samples on the anvil face, outside the influence of the pyrophyllite. The difference between the two curves is just the amount of the hysteresis which is caused directly by the holding of pressure by the pyrophyllite in the tetrahedral configuration.

IV. INTERPRETATION

The most significant result of the present work is the determination that only a very small pressure gradient exists throughout the pyrophyllite tetrahedron that forms the sample holder. This gradient is found to be only about 1% over the range of pressures from 20 to 60 kilobars. This is in contrast to the gradient of the order of 50% observed by Myers, Dachille, and Roy[5] in an opposed piston device. The existence of such a small gradient in pressure in the tetrahedral apparatus leads to several important possibilities. In the first place, it is seen that sample placement and size are not extremely critical if the sample is kept away from the gasket edge. The slight sample distortion noted in these measurements always occurred at the ends of the sample near the gasket edge where there is a large movement of the pyrophyllite in forming the gasket. The other consequence of having a small pressure gradient is the important possibility of being able to place a calibration wire on the face of the tetrahedron to determine the pressure throughout the tetrahedron volume. This will allow pressure calibration runs in which effects of sample porosity are not known. A thorough study of the change in the pressure calibration of the apparatus due to sample porosity has not been made, but preliminary results indicate that easily compressible samples may cause a 5 to 15% error in the calibration curve determined in the usual manner. It is also possible that a modification of the present method of calibration could be used for pressure calibration at high temperatures. If the anvil on which the calibration

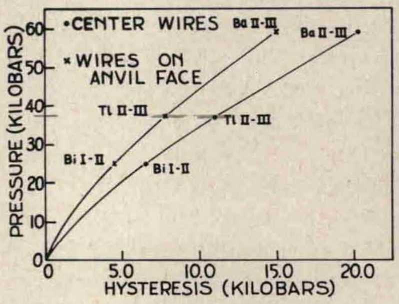

FIG. 4. Hysteresis in kilobars as a function of sample pressure in kilobars. One curve shows hysteresis for samples at the center of the tetrahedron, while the other is for samples on the anvil face, outside the influence of the pyrophyllite.

wire is placed could be kept near room temperature during a high pressure and temperature run, it might be possible to obtain such a calibration. At the present time no other technique for obtaining a high temperature pressure calibration is available.[8]

The experimental data taken in the present work also give considerable insight into the hysteresis phenomena observed in resistance versus pressure runs. We originally believed that the hysteresis observed was entirely due to the pyrophyllite holding the pressure in stages. For this reason, calibration runs were made with wires at various distances from the center wire to measure the staging effect. It was found, however, that any wire completely surrounded by pyrophyllite exhibited the same hysteresis as the center wire, no matter where it was placed in the tetrahedron. Therefore, it is found that the pyrophyllite holds the same pressure throughout the volume of the tetrahedron. This is to be expected when the results of the pressure gradient studies are taken into account.

In order to find the amount of the total observed hysteresis which is caused by the holding of pressure by the pyrophyllite, runs were made with calibration wires on the face of the carbide anvil, out of the influence of the pyrophyllite. Figure 4 is a plot of the results obtained and shows that only about 30% of the observed hysteresis is caused directly by the holding of pressure by the pyrophyllite. This amount of the hysteresis is associated with the permanent density increase of pyrophyllite when subjected to pressure.[9] The remainder is then due to the pressure configuration used and is believed to be a consequence of the elastic rebound of the compressed pyrophyllite tetrahedron.

[8] H. M. Strong, *Modern Very High Pressure Techniques*, edited by R. H. Wentorf, Jr. (Butterworths, Scientific Publications, Ltd., London, 1962), p. 113.

[9] E. C. Lloyd, U. O. Hutton, and D. P. Johnson, J. Research Natl. Bur. Standards **63C**, 59 (1959).