

Reprinted from JOURNAL OF APPLIED PHYSICS, Vol. 35, No. 12, 3466-3468, December, 1964  
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## Effect of Temperature on the Mechanical Properties of Solid Pressure-Transmitting Media. I. Talc

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(Received 17 June 1964)

High-temperature compression tests have revealed unsuspected high-temperature strength in block talc. Analyses by x-ray diffraction indicate that the increase in strength is associated with the transformation of the talc to protoenstatite, silica, and water. The bulk modulus of elasticity also increases with increasing temperature, reaches a maximum at 1100°C and then decreases at higher temperatures. These findings suggest that block talc is not as suitable for a solid pressure-transmitting medium at elevated temperatures as is commonly supposed.

### INTRODUCTION

THE advent of utilizing solid pressure-transmitting media has made available new areas for high-pressure-high-temperature experimentation, even though the data obtained are not as accurate as those measured at lower pressures using other media. Many pressure data which have been reported are based on room-temperature calibration curves constructed by the standard techniques.<sup>1,2</sup> Such measurements are considered to have some validity at high temperatures based on the assumption that the solid pressure-transmitting media decrease in strength with increasing temperatures.<sup>1,3</sup> However, there are apparently few data available concerning the effect of temperature upon the mechanical properties of solid pressure-transmitting media.

The materials which are most widely used as solid pressure transmitting media are talc and pyrophyllite. Talc, which is used principally in piston-cylinder devices, has been preferred over pyrophyllite for this application because it transmits pressure more efficiently,<sup>1,2</sup> and therefore provides more pressure on the

sample for a given applied load. At elevated temperatures the situation is complicated by the transformation of the talc to protoenstatite, silica, and water. Moreover, because of the thermal gradients that exist in high-pressure devices, the extent of the transformation varies throughout the talc cylinder from a maximum at the surface near the heater to no reaction at the outer surface. The confined water vapor may also have some effect on the high-temperature strength of the components in the high-pressure cell.

The present study was undertaken in order to investigate the effect of temperature upon the strength and ductility of block talc, particularly at temperatures where the talc begins to transform to protoenstatite, silica, and water. In addition, the talc was characterized by room-temperature testing of fired samples, volume change, and weight loss measurements. X-ray diffraction, electron microscope, and differential thermal analyses were also utilized in this investigation.

### EXPERIMENTAL PROCEDURE

The block talc used in this investigation was obtained from the American Lava Corporation and has their designation of Lava Grade 1136. The test specimens were machined into the shape of cylinders 0.500 in.

<sup>1</sup> F. R. Boyd and J. L. England, *J. Geophys. Res.* **65**, 746 (1960).

<sup>2</sup> J. E. Tydings and A. A. Giardini, ASME Preprint 62-WA-307, presented at the Winter Annual Meeting, New York (1962).

<sup>3</sup> F. R. Boyd and J. L. England, *J. Geophys. Res.* **68**, 312 (1963).

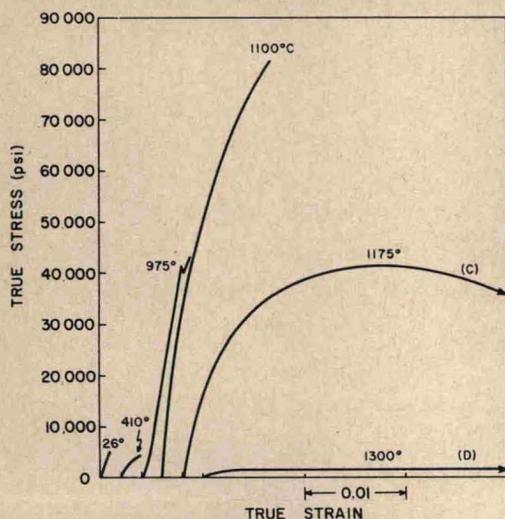


Fig. 1. Typical stress-strain curves of talc in compression at different temperatures.

(1.27 cm) in diameter by 1.125 in. (3.18 cm) long and their ends polished so as to be perpendicular to the axis of the cylinder. The compressive tests were performed using a Tinius-Olsen Universal Testing machine equipped with alumina rams having alumina pads inserted between the talc specimens and the rams in order to prevent damage to the rams from glassy phases formed in the pryophyllite upon firing. A furnace with silicon carbide heaters was used to heat the talc and rams at a rate of  $6^{\circ}$  to  $10^{\circ}\text{C}/\text{min}$ . The specimens were held at the test temperature for 30–50 min, and then tested in compression at a strain rate of  $1 \times 10^{-3} \text{ min}^{-1}$ . Temperatures were measured with a platinum-platinum 10% rhodium thermocouple whose exposed hot junction was next to the specimens. Typical true-stress versus true-strain<sup>4</sup> curves of talc in compression at different temperatures are shown in Fig. 1, while the ultimate stress and strain in compression versus temperature are shown in Fig. 2. The stresses were calculated using the original cross section of the specimen prior to firing. In the case of the fired specimens which were tested at room temperature (Fig. 4), the firing treatment was similar to that experienced by the specimens which were tested at high temperature.

#### RESULTS AND DISCUSSION

As shown in Figs. 1 and 2, the compressive strength of talc begins to increase at about  $800^{\circ}\text{C}$ , reaches a maximum at about  $1100^{\circ}\text{C}$ , and then decreases at higher temperatures. The increase in strength with increasing temperature approximately coincides with the development of protoenstatite<sup>5</sup> in the talc. For example, in Fig. 2, specimen A whose x-ray diffraction

pattern indicated the presence of only a small amount of protoenstatite had an ultimate strength of 20 000 psi ( $1400 \text{ kg}/\text{cm}^2$ ), more than three times the strength of talc at room temperature. Specimen B in Fig. 2, having a more intense pattern for protoenstatite, had a compressive strength of about 80 000 psi ( $5600 \text{ kg}/\text{cm}^2$ ) at  $1100^{\circ}\text{C}$ , which is approximately equal to that of sintered alumina at this temperature.<sup>6</sup> However, it should be noted that above  $1100^{\circ}\text{C}$  the strength decreases with increasing temperature even though the x-ray diffraction pattern of protoenstatite is still better defined. Neither specimens A, B, or C contained sufficient cristobalite to be detectable by x-ray diffraction, although a specimen fired at  $1175^{\circ}$  for 1 h contained a small amount.

The stress-strain curves in Fig. 1 indicate that the bulk modulus of elasticity increases with increasing temperature up to  $1100^{\circ}\text{C}$ . Specimen C at  $1175^{\circ}$  and specimen D at  $1300^{\circ}$  did not fracture but continuously deformed plastically under the compressive load. The scatter in the data in Fig. 2 at  $1100^{\circ}\text{C}$  is attributed to the lack of ductility in the samples.

The transformation of the talc at approximately  $800^{\circ}\text{C}$  to protoenstatite, silica, and water is also reflected by the weight loss and differential thermal analysis, and volume shrinkage curves as shown in Figs. 3 and 4, respectively. A comparison of the weight loss data with the strength measurements and the x-ray diffraction analyses indicates that it would not be possible to dehydrate talc without obtaining an appreciable increase in strength due to the formation of protoenstatite. The water expelled from the talc is a possible source of contamination if the samples in a high-pressure cell are not well sealed. This water may

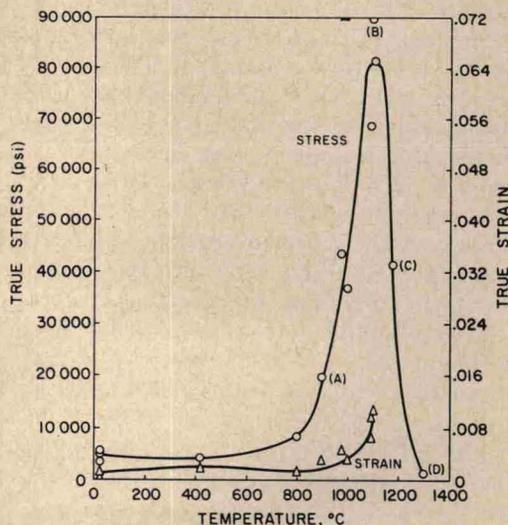


Fig. 2. Ultimate stress and strain for talc in compression at various temperatures.

<sup>4</sup> A. Nadai, *Theory of Flow and Fracture of Solids* (McGraw-Hill Book Company, Inc., New York, 1950), p. 70.

<sup>5</sup> R. B. Graf, F. M. Wahl, and R. E. Grim, *Am. Mineral.* 47, 1276 (1962).

<sup>6</sup> E. Ryskewitch, *Oxide Ceramics* (Academic Press Inc., New York and London, 1960), Chap. 2, p. 140.

either cause hydrous compounds to be formed<sup>7</sup> or it may act as a solvent to carry impurities into the sample container.<sup>8</sup>

A comparison of the room-temperature data on fired specimens (Fig. 4) and the elevated temperature data (Fig. 2) shows that the samples fired to 1000°C and over generally have higher ultimate compressive strengths. Samples fired to 1100°C were found to contain longitudinal cracks, which may explain why their strength was lower than that of the samples fired to 1000°C. No cracks were observed in those samples fired to 1300°C.

The microstructures of those samples showing the greatest variation in mechanical properties were examined with an electron microscope. The specimens were polished, etched with hydrofluoric acid, and replicated with a carbon-platinum mixture. The electron micrographs of specimen B taken with a magnification of 15 000 indicated that the specimen was composed of grains several microns in diameter, some of which contained a parallel arrangement of fibers while in others the most pronounced feature was parallel rows of etch pits. An electron micrograph of specimen C in Fig. 2 revealed two sets of fibers intersecting at 60° angles. Sample D was composed of grains with fewer etch pits and more heavily etched grain boundaries than in the other samples.

Although the mechanism responsible for the high-temperature strength is not known, it is apparently related to the bonding and interlocking of the fibers of protoenstatite. The general increase in strength obtained upon cooling of the cylinders is considered to be due to the solidification of a glassy siliceous phase, particularly in the case of specimens prefired at the higher temperatures. The silica is expelled from the talc structure as the reaction proceeds and gradually accumulates to form cristobalite as the temperature is increased. This accumulation of finely dispersed silica into cristobalite or a glassy siliceous phase may be responsible for the more heavily etched grain boundaries in the higher-temperature test specimens. This also

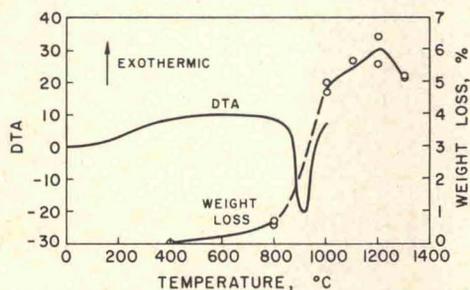


FIG. 3. Differential thermal analysis and weight loss after firing of talc.

<sup>7</sup> F. R. Boyd and J. L. England, *J. Geophys. Res.* **68**, 315 (1963).

<sup>8</sup> E. J. Bradbury *et al.*, Tech. Doc. Rept. No. ASD-TDR-62-73, Directorate of Materials and Processes, Wright-Patterson AFB (1962) (unpublished).

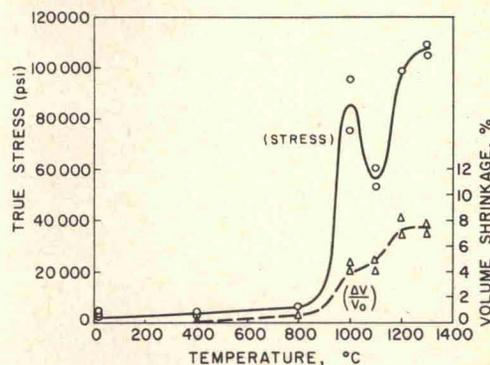


FIG. 4. Room-temperature compressive strengths and volume change in talc fired to the indicated temperatures.

suggests that at least part of the deformation may be due to grain boundary sliding.

The effect of firing time upon the high-temperature strength was not measured in this study. The probable effect of longer firing times would be to increase the strength at a given temperature, but it should not change the essential character of the ultimate strength curve in Fig. 2. No significant increase in the strength of talc could be expected below the transformation temperature, and the strength should still decrease at temperatures higher than 1100°C.

#### CONCLUDING REMARKS

The compressive strength of talc does not decrease with increasing temperature, but increases until it is as strong as sintered alumina at 1100°C. Above 1100°C, specimens of talc become weaker and can be deformed plastically. However, it should be noted that talc heated above 800° under conditions where a thermal gradient exists, such as in a high-pressure device, always would have a zone of strong, brittle material present. The effects of the increase in the bulk modulus of elasticity should also be emphasized. In an ideal high-pressure cell the pressure medium should deform easily to accommodate the volume changes due to compaction or phase changes. If the modulus increases, greater stress is necessary for the medium to adjust to these changes. The influence of temperature and stress upon deformation can be estimated from the curves in Fig. 1. The compressive tests in the present study were performed on unconfined cylinders, a situation in which fracture could occur by lateral movements of material. If the cylinders were confined, even higher stress levels could be expected.

It would be difficult to estimate the effect of this high-temperature strength upon the transmission of pressure in an actual high-pressure cell. In any event, the main effect would be expected at temperatures greater than 800°C, and any possible effect should become larger with increasing temperature and time. The pressure distribution, the effect of water vapor, and the temperature distribution in the cell are complicating factors which would need to be considered.