Strength of Synthetic Single Crystal Sapphire and Ruby as a Function of Temperature and Orientation

by J. B. WACHTMAN, JR., and L. H. MAXWELL

National Bureau of Standards, Washington, D. C.

The modulus of rupture of sapphire single crystals was determined as a function of temperature for specimens with orientations favoring plastic deformation and for specimens with unfavorable orientations. From 600°C. to 1000°C., the strength of both types increased with increasing temperature, but the increase was more pronounced for the former. Ruby specimens oriented favorably for plastic deformation also showed a large increase in strength. It is conjectured that the increase in strength results from stress relief by microscopic plastic deformation.

I. Introduction

The strength of brittle materials depends on many factors such as loading rate, temperature, and surface condition. For single crystals, it may also depend on orientation. This paper considers the unusual increase in the strength of single crystal sapphire with increasing temperature in the range 600° to 1000°C. under conditions of constant loading rate, constant surface condition, and known orientation.

The modulus of rupture of both polycrystalline aluminum oxide and single crystal aluminum oxide (sapphire) has been measured as a function of temperature by Jackman and Roberts.[1] A striking contrast was found: The polycrystalline specimens showed little change in strength with increasing temperature up to 700°C., and at higher temperatures the strength decreased markedly. Results for single crystal specimens showed great scatter but indicated that the strength had a minimum value in the range 300° to 600°C. and that at 1000°C. the modulus of rupture is approximately equal to its room temperature value. The decrease in the strength of polycrystalline aluminum oxide has been attributed to stress concentrations resulting from grain-boundary slip.[2] In this paper some data and observations are presented which support the hypothesis that the increase in strength of sapphire above 600°C. is related to plastic deformation at stress concentrations. This possibility was suggested by Jackman and Roberts[1] and recent knowledge of the mechanism of plastic deformation in sapphire[3] suggests an experimental test. Creep in sapphire occurs by slip on the (0001) plane for temperatures below 1600°C.[3(b)] Thus, if θ is defined as the angle between the direction of applied tensile stress (the direction of the specimen axis) and the [0001] direction, a specimen with θ equal to 45° is most favorably oriented for creep, but an ideal specimen with θ equal to 90° or 0° should not undergo creep. Actual specimens, however, are not ideal. The specimens used by Jackman and Roberts had θ values in the range 33° to 64°[4] and so were favorably oriented for creep. If stress relief by creep at a stress concentration is responsible for the increase in strength above 600°C., specimens with θ equal to 0° should show a smaller increase. The increase may not be entirely absent in such specimens because the angle between the local direction of the maximum tensile stress and the [0001] direction may not be zero near a stress concentration such as those that might be caused by microcracks.

II. Materials and Results

The specimens were synthetic flame-polished rods 0.100 in. in diameter and 1.5 in. long, obtained from the Linde Air Products Company. Modulus of rupture was determined for sapphire with θ equal to 45°, sapphire with θ equal to 0°, and ruby with θ equal to 45°. The modulus of rupture was determined with a bend-test apparatus described by Burdick and Parker.[5] The specimens with θ equal to 45° were tested with the [0001] direction in the plane of bending. An initial load corresponding to a stress of 1000 kg. per cm.^{2} was applied and then increased at a rate corresponding to 1200 kg. per cm.^{2} per minute until failure occurred. The stress was calculated from

$$\sigma = \frac{2Pa}{\pi r^3} \qquad (1)$$

P = load.
a = moment arm.
r = radius of specimen.

The modulus of rupture was taken to be the stress calculated from equation (1) using the load at failure.

It should be noted that the use of modulus of rupture, calculated from equation (1), as a measure of strength is open to

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At the time this work was done, the writers were, respectively, physicist and ceramic engineer, National Bureau of Standards. L. H. Maxwell is now with the Diamond Ordnance Fuze Laboratory, Washington, D. C.

[1] Elizabeth A. Jackman and J. P. Roberts, "Strength of Single-Crystal and Polycrystalline Corundum," *Phil. Mag.*, **46**, 809–11 (1955); "Strength of Polycrystalline and Single-Crystal Corundum," *Trans. Brit. Ceram. Soc.*, **54** [7] 389–98 (1955). The former paper summarizes results and conclusions; the latter gives details of the experimental procedure.

[2] J. B. Wachtman, Jr., and D. G. Lam, Jr., "Young's Modulus of Various Refractory Materials as a Function of Temperature," *J. Am. Ceram. Soc.*, **42** [5] 254–60 (1959).

[3] (*a*) J. B. Wachtman, Jr., and L. H. Maxwell, "Plastic Deformation of Ceramic-Oxide Single Crystals," *J. Am. Ceram. Soc.*, **37** [7] 291–99 (1954).

(*b*) M. L. Kronberg, "Plastic Deformation of Single Crystals of Sapphire: Basal Slip and Twinning," *Acta Met.*, **5**, 507–24 (1957).

(*c*) J. B. Wachtman, Jr., and L. H. Maxwell, "Plastic Deformation of Ceramic-Oxide Single Crystals, II," *J. Am. Ceram. Soc.*, **40** [11] 377–85 (1957).

[4] J. P. Roberts, private communication.

[5] M. D. Burdick and H. S. Parker, "Effect of Particle Size on Bulk Density and Strength Properties of Uranium Dioxide Specimens," *J. Am. Ceram. Soc.*, **39** [5] 181–87 (1956).

criticism because this equation is strictly correct only for tests in which there is no plastic deformation. Use of equation (1) may give values of stress 1.7 times the true value if plastic deformation several times as great as the elastic deformation occurs.[6] Examination of specimens on a flat surface after test, however, gave no evidence of macroscopic plastic deformation. It is thought, therefore, that the calculated modulus of rupture values are a true indication of relative strength.

Measurements were made in standard statistical patterns on sets of specimens cut from several long rods. For example, the first experiment on the sapphire rods with θ equal to 45° was a Youden square design[7] in which 28 specimens, cut from 7 long rods, were tested at 7 temperatures and the best estimate for the modulus of rupture at each temperature was calculated. The best estimates are shown in Fig. 1.

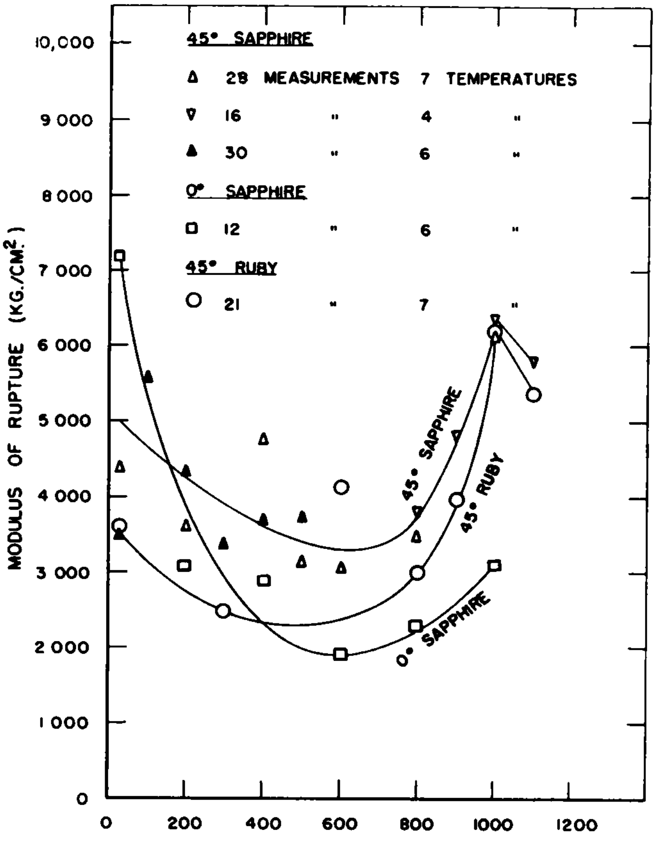

Fig. 1. Modulus of rupture of synthetic single-crystal sapphire and ruby.

III. Discussion

The data are consistent with the hypothesis that microscopic plastic deformation at stress concentrations is responsible for the rise in strength above 600°C. Three observations may be made to support this assertion:

First, there is a large increase in strength for both 45° sapphire and 45° ruby.

Second, the rise in strength from 600°C. to 1000°C. of 0° sapphire is, as expected, much smaller than the increase in strength of 45° ruby and sapphire.

Third, there is much greater scatter in strength values from measurements made at temperatures below 600°C. than from measurements made at higher temperatures. This difference in scatter is clearly evident in the individual values from which the averages shown in Fig. 1 were calculated. It seems reasonable that partial relief of stress concentrations should decrease the scatter in strength values.

In connection with the first point, it should be noted that the creep yield stress for synthetic ruby, although it may be twice the creep yield stress for synthetic sapphire (pure aluminum oxide)[3(b)] is still orders of magnitude less than the theoretical strength. When failure occurs, it presumably begins in a small region where the concentrated stress has reached the theoretical strength. Before this happens, the stress in this region must greatly exceed the creep yield stress of either sapphire or ruby and, therefore, it is reasonable to expect the same rise in strength for 45° ruby as for 45° sapphire despite the fact that a larger stress is required to cause macroscopic creep in ruby than in sapphire.

IV. Summary

The hypothesis that a limited amount of microscopic plastic deformation at stress concentrations is responsible for the observed increase in strength with increasing temperature for sapphire and ruby in the range 600° to 1000°C. has been tested by measuring strength in two different orientations—one favoring creep (plastic deformation) and one not favorable for creep. The data support the hypothesis.

Acknowledgment

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[6] J. B. Wachtman, Jr., and L. H. Maxwell, "Bend-Test Method of Determining Stress Required to Cause Creep in Tension," *ASTM Bull.*, No. **211**, 38–39 (1956); *Ceram. Abstr.*, **1956**, June, p. 121*i*.

[7] W. J. Youden, Statistical Methods for Chemists, p. 102. John Wiley & Sons, Inc., New York, 1951. 126 pp.; *Ceram. Abstr.*, **1952**, September, p. 173*e*.