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Brief Reports

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Yield strength of Al_2O_3 at high pressures

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Measurements in the diamond cell at 300 K show that the static yield strength of polycrystalline ruby (Al_2O_3:Cr) increases linearly from about 4 GPa at ambient conditions to 5.8(±0.3) GPa at an average pressure of 70 GPa. In all cases our experiments document a creep (or plastic) strength; no fracturing is observed. Our results are in general agreement with dynamic (shock-wave) measurements of yielding at high pressures, and demonstrate that the increase yield strength is proportional to the increase in the average shear modulus with pressure. Throughout the pressure range studied, the ratio of yield strength to shear modulus is 0.022(±0.002). which is within a factor of 2–5 of the theoretical yield strength. Extrapolating to higher pressures, the maximum difference between normal stresses is less than 10% (18 GPa) at 200 GPa. Thus, even for an infinitely weak sample contained in the diamond cell, the pressure obtained by the ruby-fluorescence technique should be within 10–20 GPa of the true sample pressure up to at least 200 GPa.

INTRODUCTION

There is considerable interest in the properties of ruby (Al_2O_3:Cr) under compression because of its use as a calibration standard in ultrahigh pressure static experiments.[1-9] The question often arises, however, as to what effect the presence of ruby might have on the state of stress existing in the sample. In particular, the relatively large strength of Al_2O_3 suggests that the sample pressure may on occasion be less than the mean stress of the ruby, especially if individual grains bridge the gap between the anvils of the high-pressure cell (e.g., Ref. 10).

The objective of our study is to experimentally determine the static yield strength of Al_2O_3 at high pressures. We have two purposes. First, little is known about the static yielding of materials under pressure (e.g., Ref. 11), so our experiments may provide insights into the fundamental processes that determine yield strengths.[12-14] Second, by obtaining bounds on the maximum differential stress that can be sustained by ruby, we are able to evaluate the possible bias that could enter into ruby-fluorescence measurements at high pressures. In this way, our results provide an estimate of the uncertainty for future nonhydrostatic calibrations of the ruby-fluorescence scale above 100 GPa. Interest in this second, more applied purpose is also heightened by the recent discovery[15-17] that hydrogen and deuterium undergo phase transitions at pressures at 150–250 GPa: In those experiments the samples consisted mainly of polycrystalline ruby, and it is important to determine whether the true sample pressure (as opposed to the ruby pressure) is well determined or not.

EXPERIMENTAL TECHNIQUE

Our method for evaluating the static yield strength of materials at elevated pressures is explained elsewhere in detail.[12,13,18] Briefly, we contain the polycrystalline sample inside a Mao-Bell-type diamond cell and measure the pressure distribution across our sample, as a function of its average pressure, by way of the ruby-fluorescence technique.[3] In the present case, fine-grained (<2 μm grain size) ruby powder is used as the sample material as well as the pressure calibrant. We use the same ruby (5000 ppm Cr) and spectrometer system as described in Ref. 5, and assume that the strength and elastic moduli of ruby are identical to those of pure alumina. The sample is contained between the diamonds by way of a spring-steel gasket, 150 μm in initial thickness. Prior to loading the sample, the gasket is preindented between the diamonds and then the indented region is removed; that is, the sample covers the entire culet and no gasket metal is present directly between the anvils.[12] All present experiments were carried out at room temperature with diamonds having 350-μm flat culets, and each pressure increment involved a strain rate less than $10^{-1}\,s^{-1}$ (the average strain rate throughout our experiments is less than $10^{-6}\,s^{-1}$ for each sample).[12]

Consideration of the momentum balance inside the diamond cell shows that the shear stress acting on the interface between the sample and the diamond anvil is closely approximated by

$$\sigma_{rz} = (h/2)\partial P/\partial r \, , \tag{1}$$

where h is the sample thickness (assumed to be constant across the sample) and P is the pressure at radial distance r from the center of the sample.[12,13,18,19] In these experiments, we assume that the ruby-fluorescence scale provides a measure of the mean normal stress.[3] Thus, measurements of the maximum pressure gradient across a sample are equivalent to determining the maximum shear stress supported by the sample, and hence the static yield strength at a given pressure. Note that the pressure gradient must vanish as $r \rightarrow 0$, so the yield strength is given by the pressure gradient outside the central region of the sample ($|r| > 20$ μm, in the present experiments).[12] The shear stress corresponds to $(\sigma_1 - \sigma_3)/2$, with σ_1 and σ_3 being the maximum and minimum normal stresses, respectively. The thickness of the sample is measured after decompression; by correcting this value for the effect of pressure, using the known isothermal equation of state of Al_2O_3,[20] we derive the sample thickness at high pressures.

RESULTS AND DISCUSSION

Typical profiles of pressure as a function of distance across samples of polycrystalline ruby are shown in Fig. 1. The gradients are close to linear outside the central region, as expected, and therefore provide good estimates of the shear stresses in the samples.[12] The results for all of our experiments are listed in Table I, and are summarized as a function of average sample pressure (see Ref. 14) in Fig. 2. What limits our pressure range in this study is the cupping of the diamonds, because the analysis of the stresses inside the diamond cell depends critically on the assumption that the culets remain flat and parallel.[12]

Our static values of yield strength are in general agreement with dynamic measurements.[21,22] A direct comparison is complicated by the fact that for dynamic loading the strain rates are high (of order 10^8 s^{-1}) and the temperature increases with pressure.[21] These effects would be expected to increase and decrease the observed yield strength, respectively,[23] and may to a degree cancel out. Thus, the factor of 2 agreement shown in Fig. 2 seems acceptable.

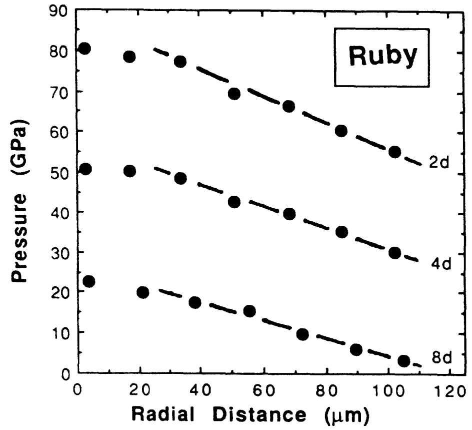

FIG. 1. Representative profiles of pressure as a function of radial distance for polycrystalline ruby in the diamond cell at 300 K. The pressure gradients are observed to be symmetric about the $r=0$ axis, and linear outside the central region of the sample. Run numbers are indicated for comparison with Table I.

Similarly, compressive yield strengths measured on slightly porous aggregates of alumina at ambient conditions are broadly compatible with our high-pressure data: scaling by the Knoop hardness to full density yields an estimated value of 2.8(±0.2) GPa for the maximum supportable shear stress, as we have defined it here.[24,25] It is unclear what role fracturing has played in these compressive strength measurements, however.[24,25] Notably, our present values of yield strength are somewhat higher, if anything, than those previously measured for polycrystalline alumina. That is, an extrapolation of our data to zero pressure yields a value of 4.0(±0.1) GPa for the yield strength at ambient conditions (Fig. 2), suggesting that the earlier values are indeed slightly low due to fracturing. In our own experiments the ruby is observed to flow homogeneously, with a radially symmetric strain and no evidence of fracturing or acoustic emissions (cf. Ref. 26).

Because the conventional theories of yielding predict that the strength of a solid is proportional to its shear modulus, whether the strength is limited by elastic instability or by dislocation processes,[11,27] we compare our measurements of the maximum shear stress supported by ruby, σ_m, with the average (isotropic) shear modulus, μ, as a function of pressure. The isotropic shear modulus of

TABLE I. Static yield strength of ruby at 300 K.

Run number	Average pressure P_{av} (GPa)	Pressure gradient $\partial P/\partial r$ (GPa/μm)	Thickness h (μm)	Max. shear stress σ_m (GPa)	σ_m/μ
1*c*[a]	70.9(±1.3)	0.313(0.009)	37.5(±1.2)	5.87(±0.26)	0.022(±0.001)
2*d*	61.6(±3.0)	0.306(±0.022)	37.7(±1.2)	5.77(±0.46)	0.023(±0.002)
3*d*	42.5(±0.8)	0.257(±0.006)	38.2(±1.2)	4.92(±0.20)	0.021(±0.001)
4*d*	34.7(±1.6)	0.255(±0.011)	38.5(±1.2)	4.91(±0.27)	0.023(±0.001)
5*d*	24.3(±3.2)	0.242(±0.024)	38.8(±1.2)	4.71(±0.49)	0.023(±0.002)
6*c*	63.1(±2.5)	0.252(±0.017)	42.4(±1.2)	5.34(±0.39)	0.021(±0.002)
7*c*	42.9(±2.6)	0.257(±0.018)	41.2(±1.2)	5.30(±0.40)	0.023(±0.002)
8*d*	7.3(±1.1)	0.206(±0.007)	41.6(±1.2)	4.28(±0.19)	0.024(±0.001)

[a] Values collected on compression and decompression are labeled *c* and *d*, respectively. Three samples were studied, corresponding to runs 1–5, 6, and 7 and 8.

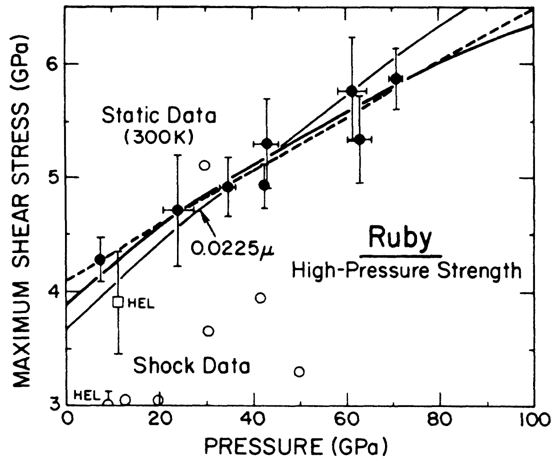

FIG. 2. Static yield strength, corresponding to the maximum shear stress supported by polycrystalline ruby, is shown as a function of average sample pressure (solid symbols). The data are fitted with a linear dependence of either σ_m/μ (bold solid curve) or σ_m (dashed curve) on pressure. For comparison, dynamic measurements are shown by open symbols [square with error bar (Ref. 21) and circles (Ref. 22)]. The thin solid curve indicates 0.0225 of the shear modulus of ruby as a function of pressure, as calculated from the elastic constants and finite strain theory (Refs. 28–33).

alumina is calculated from the ultrasonically measured elastic constants[28–31] as the average of the Hashin-Shtrikman bounds.[32] The pressure dependence of the shear modulus is then obtained from Eulerian finite-strain theory,[33] which is known to reproduce the isothermal equation of state from the ultrasonic moduli.[20] It is thus evident from Table I and Fig. 2 that our measurements result in a ratio $\sigma_m/\mu = 0.022(\pm 0.002)$. Thus, the yield stress of ruby scales with the shear modulus as a function of pressure, in accord with what is expected from the elastic-continuum theory of dislocations.[27]

The yielding of ruby contrasts with that previously found for MgO and NaCl, which both exhibit a significant increase in σ_m/μ with pressure (cf. Refs. 12 and 13). One likely reason for this contrast is that the observed yield strengths of MgO and NaCl are initially far below the theoretical limits ($\sigma_m/\mu < 0.005$ observed in both cases at ambient conditions versus 0.035 and 0.018 predicted,[11] respectively, for plastic yielding), whereas the observed strength of ruby is within a factor of 2.5 of the theoretical limit. Indeed, the theoretically calculated yield strength is probably uncertain to within a factor of 2–4 in any case.[11] Therefore, our results for ruby support the elastic-continuum theory and we expect that the high-pressure yield strengths of MgO and NaCl saturate at the theoretical limit, with a diminished pressure dependence upon compression beyond the range investigated to date.

As a specific application of our data, we consider the maximum difference between the pressure in ruby and the pressure in a coexisting sample in an ultrahigh pressure static experiment. As the pressure is given by the average of the principal stresses (e.g., Ref. 34), the maximum difference between the pressure in an infinitely weak sample and that given by the ruby-fluorescence scale is $2\sigma_m = \sigma_1 - \sigma_3$. Extrapolating our measurements to 200 GPa shows that the maximum pressure difference is 12–18 GPa, or less than 10% of the pressure (this range of values corresponds to extrapolations based on σ_m/μ being constant or on σ_m increasing linearly with pressure, respectively; see Fig. 2). Therefore, pressures reported to date from diamond-cell experiments in the 200 GPa range are not significantly biased by the strength of ruby.

ACKNOWLEDGMENTS

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