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Compressive strength and microplasticity in polycrystalline alumina

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The compressive strength of polycrystalline alumina at 23° C is found to be strain-rate sensitive, but insensitive to environment. Scanning electron microscopy of specimens loaded to near failure indicates the origin of the strength–strain-rate dependence to be localized plasticity in the form of twinning and, possibly, slip. The interaction of these deformation bands with grain boundaries causes the initiation of microcracks. Higher stresses produce still more twin/slip-nucleated microcracks, which finally coalesce at failure. It is suggested that twinning also may be related to the tensile failure of alumina.

1. Introduction

Certain ceramic failure processes of practical interest, such as those relating to impact and erosion, may be related to the microfracture behaviour of brittle materials subject to compressive loadings. Factors which might be expected to be important in such situations are strain-rate, environment, and temperature. Earlier work on alumina has shown that tensile failure is strain-rate and temperature dependent, and environment sensitive [1–9]. It has been argued that plastic flow both can [1, 6, 7, 10–13] and cannot [14–17] explain this behaviour. Almost no corresponding alumina data are available for compression, although Rice [18] has presented a rather convincing case for the view that room temperature plasticity may play an important role in the compressive failure of even the most brittle ceramics. In the present study, it is found that compressive failure of alumina is strain-rate sensitive, but is unaffected by environment. Failure at room temperature is shown to be controlled by microplasticity, i.e. twinning and possibly slip, which nucleate grain-boundary cracks. The implications of the results with respect to tensile failure and temperature effects are discussed.

2. Experimental procedure

From as-received rods of polycrystalline alumina (type GW Lucalox*), right circular cylindrical specimens (1.25 cm long x 0.625 cm diameter) were cut. Special alignment jigs were prepared for grinding the faces parallel within a tolerance of 1×10^{-4} cm. These faces were then polished metallurgically through 1 μm diamond grit, using a special lapping wheel fixture to maintain parallelism. The aim of this polishing procedure was to eliminate large flaws at the specimen platen interface, and thereby minimize scatter in the failure data caused by activation of anomalous flaws introduced during specimen preparation. Prior to testing, specimens were cleaned ultrasonically, rinsed in alcohol and distilled water, baked out under vacuum at 150° C for 12 h, and not touched thereafter. The average grain size was 25 μm.

For the compression tests, alumina platens were fabricated from a high purity alumina considerably stronger than the specimen material, i.e. AD 999.† As in the specimen preparation, the platens (1.875 cm long x 1.25 cm diameter) were ground parallel, and then polished metallurgically on both faces. In order to prevent yielding of the faces of the loading rams, they were isolated from the

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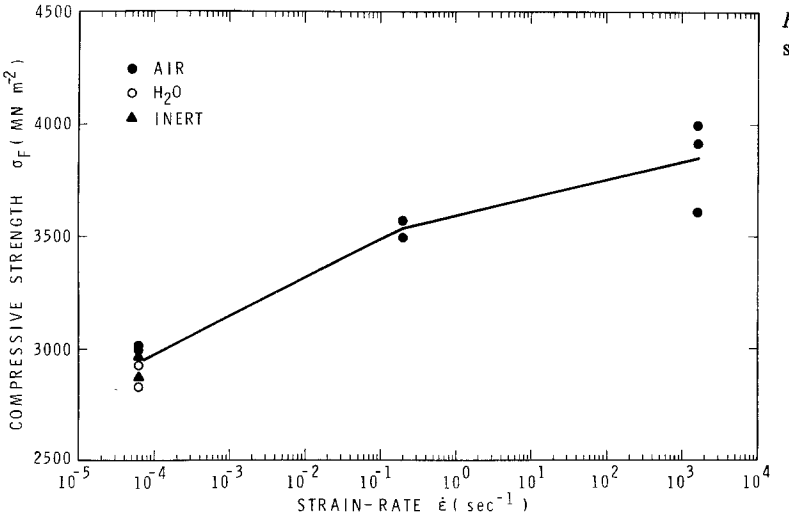


Figure 1 Compressive strength versus strain-rate.

ceramic platens by thin discs of ultra-high strength steel, precision ground to the same tolerance as the platens. Because of surface damage introduced during testing, these discs were either reground or discarded after each test. The platens always suffered at least some damage during a test, and were therefore also used only once; they were never repolished, since cracks introduced during initial testing might cause premature failure in subsequent tests.

Low ($\dot{\epsilon} = 7 \times 10^{-5} sec^{-1}$) and intermediate ($\dot{\epsilon} = 2 \times 10^{-1} sec^{-1}$) strain-rate tests were carried out in an electrohydraulic, servocontrolled testing machine. The high strain-rate tests ($\dot{\epsilon} \sim 2 \times 10^{3}$ sec^{-1}) were performed in a Hopkinson pressure bar, as described elsewhere [19]. In all of the tests, special jigs were employed to centre and align specimen, platens, discs, and load rams.

Low strain-rate tests were carried out in three different environments, the first being the ambient conditions characteristic of the laboratory, i.e. 50% relative humidity. A wet environment was provided by immersing the specimen and platens in distilled water. Finally, an inert environment was generated by flowing dry nitrogen (< 1 ppm water vapour) through a plastic cell sealed about the specimen and platens.

A few specimens were loaded to successively higher stresses, with the specimens being replicated in the unloaded state following each load excursion. Metallic replicas were secured by coating the plastic replicas with palladium, plating the latter with nickel, and then stripping away the plastic [20]. The resulting positive replicas were studied in the scanning electron microscope (SEM).

3. Results

3.1. Failure tests

Tests of compressive strength (σ_F) at $\dot{\epsilon} = 7 \times 10^{-5}$ sec^{-1} in each of the three environments produced almost identical results, as shown in Fig. 1. Since environmental effects would be most likely to manifest themselves at low strain-rates, only ambient tests were run at higher strain-rates. It is interesting to note, in contrast, that measurements by Charles [9], of the bend strength of Lucalox at 23° C, $\dot{\epsilon} = 5 \times 10^{-6} sec^{-1}$, show the tensile strength in vacuum to be approximately 1.5 times that in wet H_2.

The results of compressive strength tests in the ambient laboratory environment at other, higher rates are plotted on the same graph (Fig. 1).

It can be seen that there is a marked strengthening in compression caused by increasing deformation rate. Similar constant strain-rate tensile tests of Lucalox [9] and other strong brittle materials [1–3] have shown that tensile strength increases with strain-rate, at least for $\dot{\epsilon} \gtrsim 10^{-3} sec^{-1}$, in both aggressive and inert environments. At higher strain-rates ($\dot{\epsilon} \gtrsim 10^{-3} sec^{-1}$), the stress level is sufficiently high that the critical stress intensity for failure is attained at the tips of growing cracks, and tensile failure becomes insensitive to further strain-rate increases [21]. On the other hand, since microcracks nucleated under compression are stable even at very high stress levels, the strain rate-strengthening

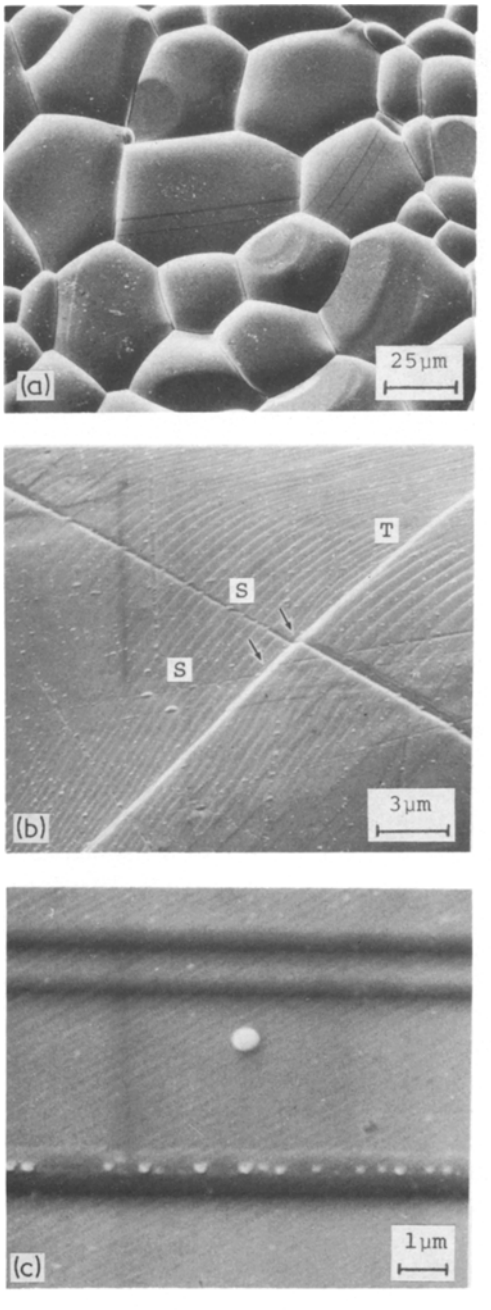


Figure 2 Twinning in compression, 85% σ_F. (a) Twins; (b) twin (T), showing reference scratch (S) offsets; (c) twins, showing thermal grooves on twin facets.

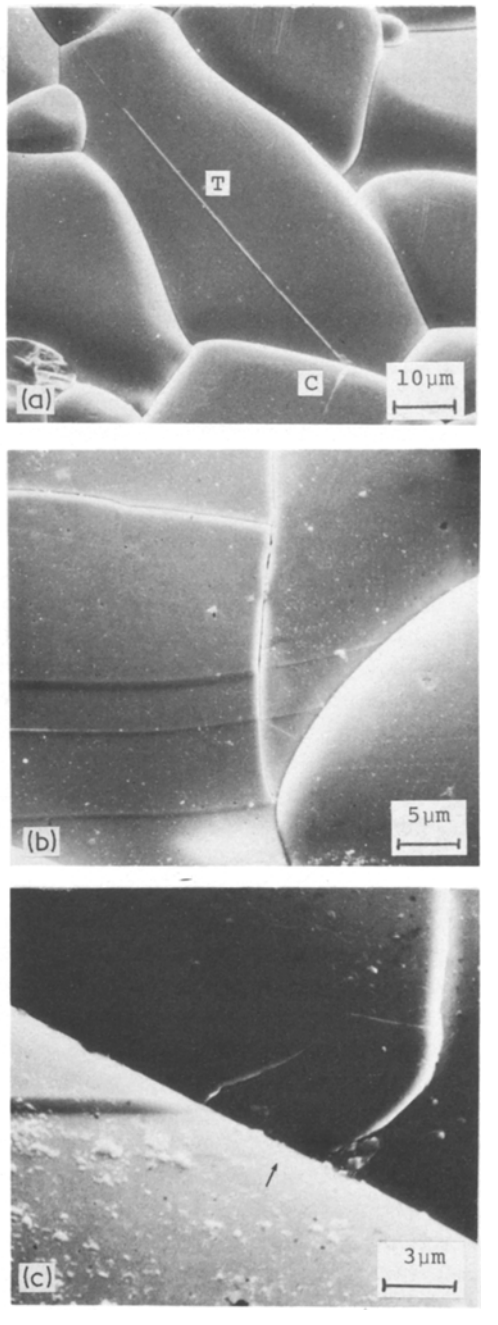


Figure 3 Twin-nucleated cracking at grain boundary (GB), 85% σ_F. (a) Twin (T), crack (C); (b) twin-nucleated cracking; (c) twin-nucleated cracks.

effect for compressive failure persists to much higher strain-rates.

3.2. Microscopic analysis

The increase in compressive failure strength with loading rate can be understood on the basis of SEM observations carried out on prestressed but intact specimens. The photomicrographs discussed below were taken from specimens compressed to approximately 85% of their failure strength, at $\dot{\epsilon} = 7 \times 10^{-5} \text{sec}^{-1}$. Specimen tilt in the SEM was 45°.

Typical deformation features induced by compressive loading are shown in Fig. 2. The features shown in Fig. 2a are twins formed in neighbouring

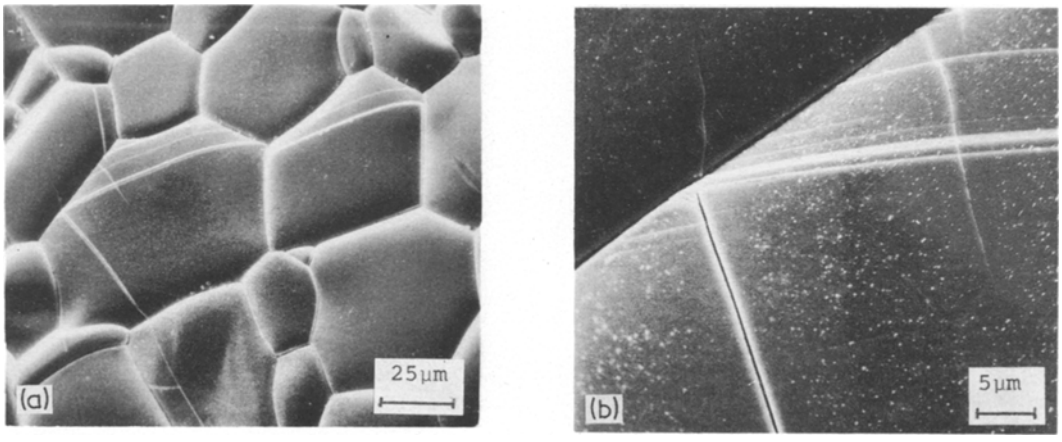


Figure 4 (a) Twin-nucleated axial cracking, 85% σ_F. (b) Higher magnification of (a).

grains. (In this photo, and in all others as well, the compressive stress axis is vertical.) Higher magnification views of other twins can be seen to have rotated the scratches (arrows) as they cross the twin. The fact that the ledges in Fig. 2c are twins and not dislocation deformation bands is demonstrated by the presence of thermal grooves which cover each facet, i.e. there is no "slipped" facet on which the grooves are absent. Grain-boundary triple points were observed to be frequent twin nucleation sites.

The reason that such twins are important is illustrated in Fig. 3; they nucleate the transgranular microcracks whose eventual coalescence is responsible for compressive failure. All of the cracks shown are situated at twin/grain-boundary intersections (an arrow indicates a faintly visible microtwin lying within the grain-boundary "glow" in Fig. 3c). Whereas twins were often observed in the absence of microcracking, as in Fig. 2a, crack nucleation was associated almost exclusively with twin/grain-boundary intersection.

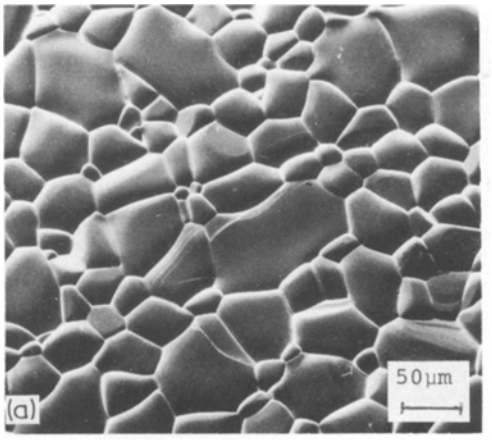
A particularly good example of the relationship between twinning and microcrack initiation is depicted in Fig. 4. Here it can be seen that each microcrack is associated with a twin/grain-boundary intersection. In addition, many very fine microtwins, or possibly slip bands, have nucleated at the grain boundary shown in Fig. 4b. If these are microtwins, they apparently are exceedingly fine, with a thickness of the order of 0.05 μm.

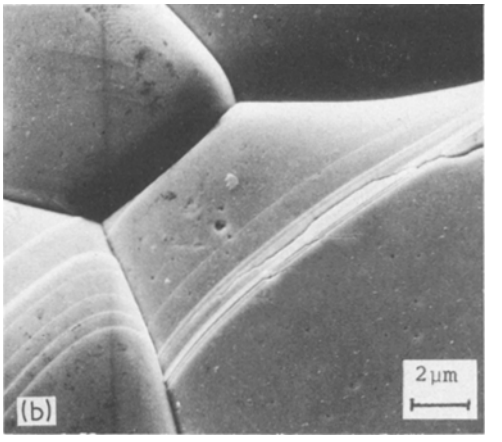
The influence of the twins upon microcrack formation is not limited to their interaction with grain boundaries. Microcracks which have nucleated within twin bands also were observed frequently, as shown in Fig. 5. Again, the presence at grain boundaries of very fine microdeformation bands is evident (Fig. 5c, arrows).

From the sequential replication studies, it was determined that twinning (and/or microslip) occurs at stress levels well below the failure strength. Twinning and associated microcracking were observed at stresses equal to $\sigma_F/2$, and a few twin-like features were observed at $\sigma_F/4$. However, at the latter stress levels the twinning frequency is sufficiently low that microscopic search for the twins becomes extremely tedious. Therefore, an acoustic emission apparatus is being set up to determine the minimum stress required for twinning.

It was noted that from 50% σ_F to 85% σ_F, microcrack extension was generally insignificant following nucleation. Rather, the major defect development associated with increased stress consisted of increasing amounts of twinning, which caused stable microcrack formation. Substantial crack growth apparently occurs only near failure. The failure event was interesting in that the specimen essentially exploded, reducing itself to a pile of exceedingly fine powder and many tiny (< 1 mm) chips.

Specimens tested at the intermediate strain-rate also exhibited twinning and related microcracking at stresses below σ_F. However, microscopic observation alone was insufficient to determine the relative stress levels and frequency at which twinning occurs for the different strain-rates. Acoustic emission again must be used to make this determination. For the present, it is important to know that twinning does occur to at least some extent over a widely varying strain-rate range.





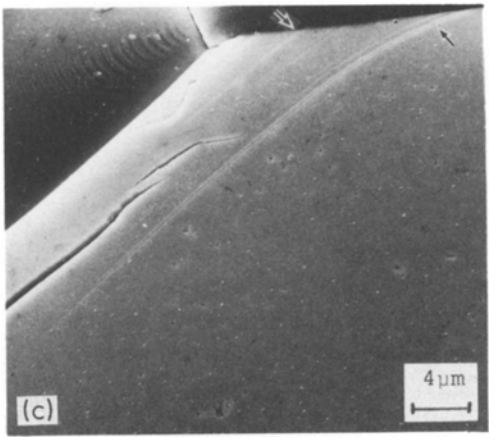


Figure 5 (a) Twinning and twin-plane cracking. (b) and (c) Higher magnifications of (a).

4. Discussion

The preceding experimental observations offer firm evidence of the presence of plasticity at room temperature in a strong, polycrystalline ceramic. Since it was further shown that this plasticity (twinning) is the principal cause of microcracking, it is reasonable to think that the observed strain-rate dependence of the compressive failure strength of Lucalox can be attributed to deformation twinning. The possibility that thermally activated subcritical crack growth might explain this strain-rate effect is unlikely, since the compressive strength is insensitive to environmental influences; the latter are generally believed [21, 22] to control strength through their effect on crack growth, rather than upon crack initiation. Moreover, as pointed out earlier, no significant crack growth appears to occur during the compression of Lucalox until close to failure.

Additional evidence of room temperature twinning under conditions in which the combined stress state includes a strong compressive component has been provided by the results of abrasion [23, 24] and indentation [25, 26] studies. Previously [27] it was shown that twinning could nucleate cracks at temperatures in excess of 1200° C. The present work, which to the author's knowledge documents for the first time room temperature plasticity in polycrystalline alumina subject to a "simple," uniaxial stress state, shows that twins can cause microcracking at lower temperatures as well. In view of these facts, it is suggested that room-temperature microcrack formation under sharp indenters and during non-spherical particle impact may be twin-nucleated.

It is further possible that twinning is related to low temperature tensile failure. The results of preliminary compression tests at temperatures between 23 and 450° C indicate that there exists a minimum in compressive strength at around 200° C. This is similar to the minima in tensile strength [4, 7, 9, 13] and fracture surface energy [6] which have been noted in the past during tensile testing of single crystal and polycrystalline aluminas, respectively. (Polycrystalline alumina shows a strength decline from 0 to 300° C, with strength remaining constant thereafter until declining again at around 1000° C [9, 28].) The compressive strength minimum probably is caused by twinning; it is reasonable, by analogy, to suppose that the tensile strength minima, which occur even in inert environments [9], may have a similar origin.

If twinning is related to brittle fracture in general, then it should be possible to produce twins under tensile conditions as well. Since twinning in

compression of Lucalox commences at stresses between 25% and 50% σ_F, shear stresses on the order of 500 to 1000 MN m^{-2} appear to be sufficient for twinning at 0° C. Under similar test conditions, the tensile strength of Lucalox having a grain size of 25 μm is approximately 350 MN m^{-2} [9]. In the presence of a stress concentration factor of 2.5, which may prevail near grain-boundary triple points, shear stresses produced in tension would reach 700 MN m^{-2}. This is within the estimated shear stress range for twinning, based on the compression experiments.

In studies of alumina single crystals at room temperature, Heuer [10] observed deformation microtwins near the tensile fracture surfaces of specimens broken in bending, while Becher [29] has been able to determine that thin basal twins introduced during grinding of single crystal alumina specimens frequently constitute the fracture nuclei during bending at 22° C. In the latter study, the twin plane serves as a plane of weakness within which an initial crack is formed. Recently, the author has observed twin-like features adjacent to the tensile fracture nuclei of Lucalox rods broken in bending.

5. Conclusions

Compressive failure at room temperature in polycrystalline alumina is shown to be strain-rate dependent, with the rate-dependent mechanism being deformation twinning. This twinning, which commences at between 25% and 50% of the failure stress level, initiates microcracks at twin/grain-boundary intersections. Failure is independent of environment, and crack growth apparently is not important until the latter stages of microfracture coalescence. Twinning probably is one of the mechanisms through which cracks are nucleated beneath sharp indenters and during impact of non-spherical particles. It is suggested that twinning may play a role in crack nucleation during tensile deformation at low temperatures.

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