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Deformation Substructure in Polycrystalline Alumina

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Transmission electron microscopy techniques were employed in a study of substructure in two sintered, 20 μm alumina ($\geqslant 99\% Al_2O_3$) bodies, each being examined in the as-annealed condition and after achieving 2% permanent strains in compression at 1420 and 1700° C. The as-sintered microstructures were found to contain dislocation networks which were often associated with intragranular porosity. Twinned structures of rhombohedral and basal types were also observed but were relatively infrequent. Amorphous second phases commonly located at triple points were characteristic of the less pure (99% Al_2O_3) material, and were also observed in the 99.9% purity material, but only in very limited amounts. The plastic deformation behaviour of these bodies at elevated temperatures ($T \leqslant 1420°$ C) illustrates that dislocation motion is an important mode of deformation in alumina. The observation of $\langle 11\bar{2}0\rangle$ and $\langle \bar{1}011\rangle$ glide dislocations further suggests that alumina can exhibit ductile behaviour at elevated temperatures. Grain boundary shearing was also observed to contribute to plastic deformation, being associated with either grain-boundary dislocations or interfacial impurities which can alter deformation behaviour.

1. Introduction

Recently, ion-beam thinning techniques in conjunction with transmission electron microscopy have proved to be advantageous in studying the substructure in polycrystalline ceramics. Tighe and Hyman [1] and Tighe [2] have demonstrated that these direct imaging techniques are extremely effective in examining the microstructure of as-sintered alumina bodies. Furthermore, their general applicability has been illustrated in studies of deformation substructure induced in α-alumina [2-5] and other oxides [2].

The mechanical behaviour of polycrystalline alumina at elevated temperatures has been seen to be strongly influenced by plastic flow mechanisms. The works of Tighe [2] and Heuer *et al* [3] have shown that mechanical twinning and grain-boundary shearing were involved in the plastic deformation occurring during dynamic bend-testing of fine-grained alumina at elevated temperatures. Little, if any, evidence for plastic flow directly related to dislocation glide was detected in these experiments [2-3]. However, direct observation of dislocation glide as involved in high-temperature creep of sapphire single crystals has been reported [6]. In addition, Hockey has demonstrated that dislocations and twins are produced as a result of surface abrasion [4] and indentation [5] of sapphire crystals and polycrystalline α-alumina at room temperature. These direct observations indicate that dislocation glide and climb mechanisms, as well as twinning and grain-boundary sliding, can be activated during the deformation of polycrystalline α-Al_2O_3.

2. Experimental Procedure

This study was made on two sintered α-alumina bodies, each with an average grain size of 20 μm and consisting of:
(i) 99.9% Al_2O_3 (SiO_2, MgO, ZrO_2 and CaO, each at < 400 ppm levels, as major impurities), 99 + % dense, identified as body A†; and
(ii) 99% Al_2O_3 (SiO_2, CaO and MgO each $\leqslant$ 5000 ppm; and $Fe_2O_3 < 400$ ppm as major impurities), $> 98\%$ dense, identified as body B†.

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†Products of Coors Porcelain Co, Golden, Colorado.

Foils of the materials to be examined were obtained from both as-annealed stock and specimens deformed at elevated temperatures, from which thin disk-shaped sections ($\sim$ 50 μm thick) were prepared by standard sectioning and polishing procedures. Subsequent thinning to produce foils sufficiently thin to permit transmission of electrons was done by means of an ion-beam thinning device.‡ After application of a carbon film, these foils were then examined in a transmission electron microscope§ operating at 120 kV and analysed by standard transmission electron microscopy and diffraction techniques.||

The specimens to be deformed were cut from as-received stock and ground to obtain rectangular parallelepiped configurations having aspect ratios of 2:1. Subsequently, they were annealed at 1700° C *in vacuo* for a period of 2 h prior to testing. The testing scheme consisted of compressive loading *in vacuo* ($< 5 \times 10^{-5}$ torr) at temperatures of 1420 or 1700° C employing a constant strain rate of 5×10^{-3} min^{-1}. All test samples to be used for further microstructural analysis were deformed in the above manner to achieve permanent strains of $\sim 2\%$. During the testing schedules selected samples were deformed to failure to determine the degree of permanent strain accommodation in these bodies.

3. Results

The microstructures of the A and B bodies revealed several structural similarities after annealing to achieve stable grain size. Generally, dislocations were found in networks rather than as singularities and were often associated with intragranular pores (fig. 1a). These types of microstructure are consistent with those previously observed in sintered and hot-pressed alumina materials [1, 2]. Further, some intergranular porosity was observed in these two bodies, together with occasional rhombohedral and basal twins (fig. 1b). However, the B body was distinguished by an appreciable concentration of amorphous interfacial second phase and a higher pore content as compared to the A body.

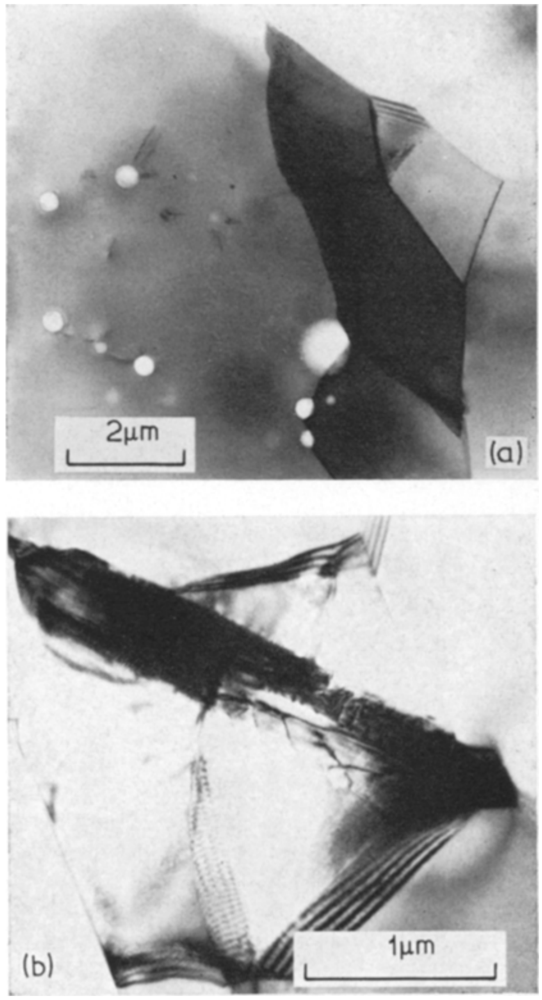

Figure 1 Microstructural features characteristic of the as-annealed 20 μm grain sized alumina bodies. (a) Dislocation arrays and intragranular porosity. (b) Typical basal twin structure.

During mechanical testing (fig. 2), the B body was found to be consistently less resistant to applied stress than the A body, which accommodated 2% plastic strain or more. At 1420° C, the B body was subject to rather severe structural degradation, a factor which prevented subsequent thin foil preparation. Microstructural analysis was therefore accomplished, using optical and scanning electron microscopy, on the B body tested at 1420° C.

The A body was found to contain $\langle 11\bar{2}0 \rangle$ dislocations, coupled with some evidence for dislocations with $\langle \bar{1}011 \rangle$ Burgers vectors when tested at 1420° C (fig. 3). These glide dislocations are generally in irregular networks, with some tendency for tangling, although parallel dislocation bands were detected. These observations,

‡Ion Micro Milling Instrument, Model IMMI-2, Commonwealth Scientific Corp., Alexandria, Virginia.
§Model JEM-120, Japan Electron Optics Laboratory Co, Ltd, Tokyo, Japan.
||Based on a structural unit cell having c/a ratio of 2.73:1.

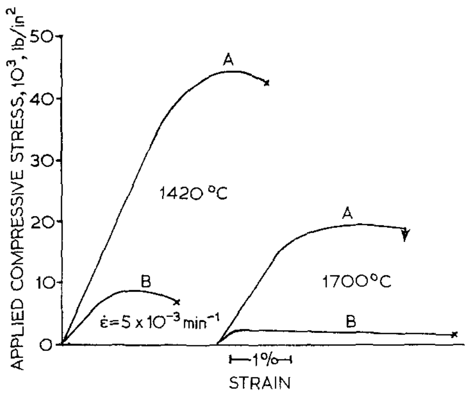

Figure 2 Hot Mechanical behaviour of 20 μm grain-size alumina ceramics of 99 and 99.9% purities.

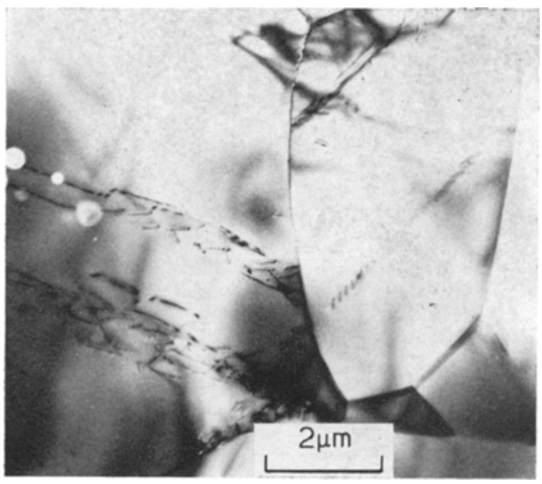

Figure 3 Dislocations generated as a result of plastic deformation induced at 1420° C.

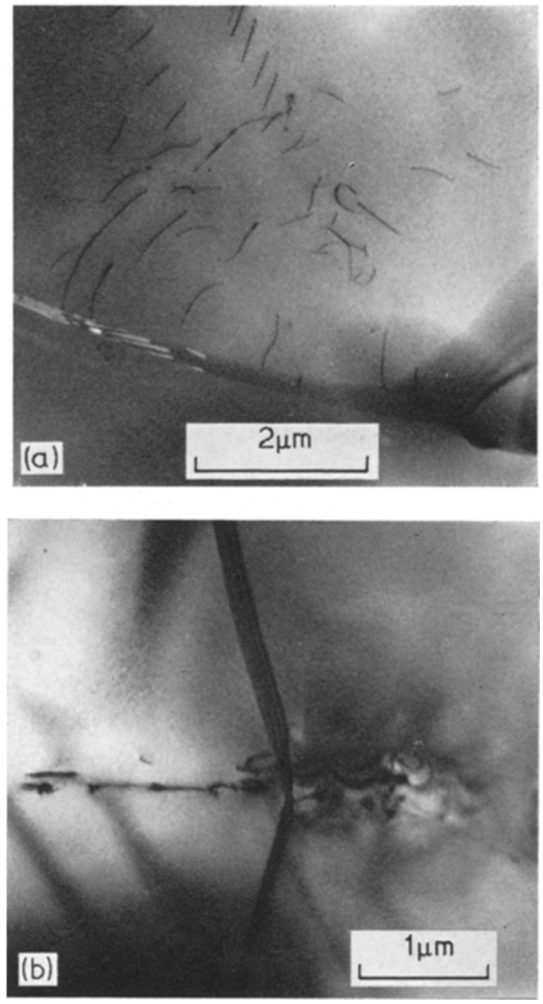

Figure 4 Substructure induced by boundary shearing in A body. (a) Interfacial porosity. (b) Dislocations incurred by shearing in adjacent triple point.

together with the mechanical behaviour at 1420° C, strongly suggested that slip was insufficient to allow grains to conform freely to neighbouring grain deformation. This retention of grain rigidity results in stress concentrations at grain interfaces which can induce grain-boundary shear. Boundary sliding was in fact, detected and was primarily associated with porosity which propagated along the deforming interface (fig. 4a). The observed grain boundary behaviour was also associated with offsets in triple points, triple point voids and shear in adjacent grains favourably oriented for slip (fig. 4b). Occasionally, boundary shear resulted in localised plastic deformation in boundary pore ledges and serrations. In a few instances, grain boundaries devoid of porosity sheared at 1420° C by movement of dislocations in the interfaces. These grain-boundary sliding dislocations (fig. 5) were similar to those reported by Gleiter *et al* [7] in that they were identified by their analogous, unique image contrasts. To date, only dislocations having $\langle 11\bar{2}0\rangle$ Burgers vectors have been observed to be associated with interfacial shear.

Mechanical twinning was also found to be occurring at 1420° C. Although twins were not prevalent, they occasionally induced shear in the adjacent grains. Generally, crack initiation by twin-grain boundary or twin-twin interactions was limited, while crack nucleation by dislocation pile-ups was not detected.

The increased plastic flow in the A body at

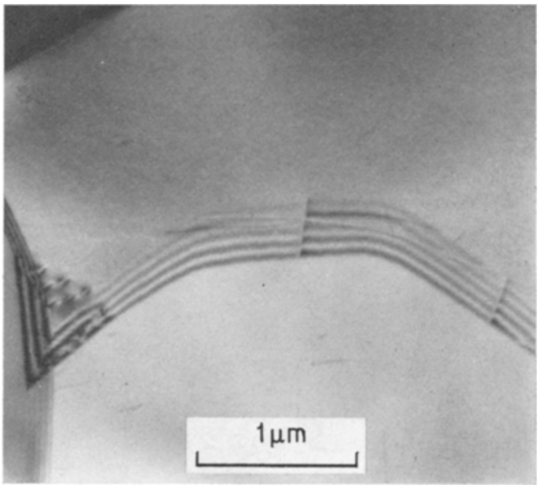

Figure 5 Grain-boundary sliding dislocations detected in polycrystalline α-alumina.

1700° C was related to a substantial gain in dislocation activity involving both $\langle 11\bar{2}0\rangle$ and $\langle\bar{1}011\rangle$ Burgers vectors (fig. 6). In conjunction with the increased role of dislocations at 1700° C the contribution of boundary shear was significantly reduced. However, in contrast to the behaviour at 1420° C, diffusional processes which resulted in extensive polygonisation of dislocations were very important at 1700° C. Finally, the A body did not appear to deform by mechanical twinning, and crack initiation by twinning or dislocation mechanisms was not observed at this test temperature.

As noted previously, the mechanical properties of the B body are very much altered relative to those of the A body. At 1420° C, the stress-strain results (especially the low stresses obtained) suggested that dislocation glide was of less importance, and microstructural analysis illustrated that grain-boundary shear was the predominant deformation mode. Similar behaviour was found after testing at 1700° C (fig. 7), although there was evidence of some possible increase in the activity in $\langle 11\bar{2}0\rangle$ and $\langle\bar{1}011\rangle$ dislocations. The contribution of dislocations to plastic deformation at 1700° C occurring in the B body was, however, much less than that on the A body tested at either 1420 or 1700° C.

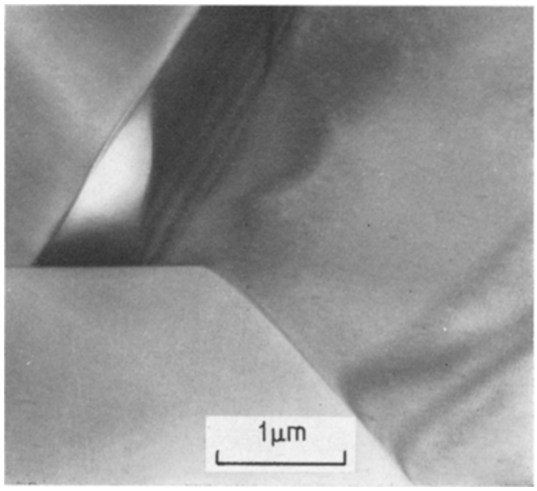

Figure 7 Amorphous second phase at boundary, associated with boundary shear.

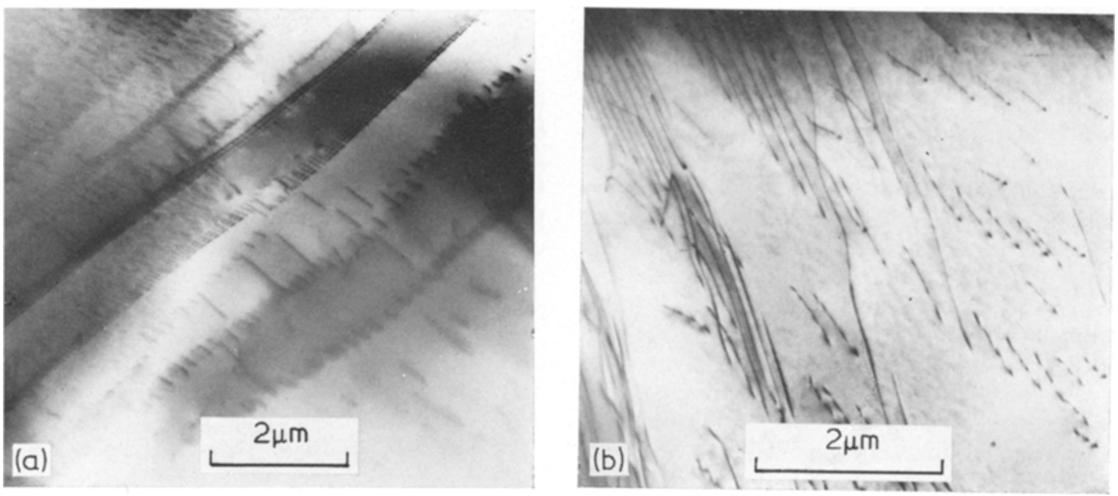

Figure 6 Dislocation structures developed during tests at 1700° C. (a) Dislocations with $\langle 11\bar{2}0\rangle$ Burgers vector; (b) $\langle\bar{1}011\rangle$ type glide dislocations.

In contrast to the A body, grain-boundary shear in the B body was a primary means of deformation and was generally associated with interfacial phases rather than the previously described porosity or GBS dislocations. Electron diffraction analysis has shown these interfacial second phases to be amorphous and are apparently calcia-alumina-silica glasses based on impurity contents.

4. Discussion

The results presented for these polycrystalline alumina ceramics and those previously reported for sapphire bicrystals [8a,b, 9] clearly demonstrate a pronounced influence of impurities on the hot mechanical behaviour of α-Al_2O_3. The effect here is related to the accentuation of grain-boundary shear over competing deformation processes particularly when impurities are segregated to grain interfaces. The observation of grain-boundary impurity segregation in very pure polycrystalline oxide ceramics [10] illustrates the importance which this type of grain-boundary shear associated with impurities can have on the properties of ceramics at elevated temperatures. As noted in connection with the behaviour of the B body, amorphous interfacial second phases clearly result in lowering the resistance to boundary shear, to the extent of generally eliminating slip and mechanical twinning. The behaviour of similar silica-rich grain boundaries in sapphire bicrystals has suggested lowering of the boundary viscosity as being important in the observed boundary sliding [8a, b]; similar behaviour is proposed for the less pure (99% Al_2O_3) B body. In addition, similarity of the microstructure of grain boundary shear in the A body with that reported for boundary sliding in Mg^{+2} doped α-Al_2O_3 bicrystal interfaces [8b, 9], although not conclusive, is indicative of a grain-boundary diffusion mechanism accentuated by impurities.

In the absence of second phase accumulation at grain interfaces, the grain boundaries are apparently more resistant to shear. As was found in the A body, grain-boundary sliding then is more likely to be a result of the inability of dislocation glide or mechanical twinning to thoroughly accommodate the imposed strains. Thus interfacial sliding is an alternative means of plastic flow which relieves stress concentrations, although the porosity associated with the predominant mode of boundary shear would certainly tend to weaken the body. This is substantiated by the limited plastic deformation of the A body at 1420° C and an observed preponderance of intergranular fracture in this body. Furthermore, the fact that grain-boundary shear is most likely an alternative or secondary deformation mode in the purer alumina body, is corroborated by its decreased contribution when dislocation activity is substantially increased (i.e. at 1700° C).

The increased resistance to grain-boundary shear in the A body also has demonstrated that considerable strain can be accommodated by dislocation motion in a medium grain size alumina body. The $\langle\bar{1}011\rangle$ glide dislocations detected in these 20 μm bodies are indicative of either a reported $\{10\bar{1}2\}$ $\langle\bar{1}011\rangle$ slip system [8a, 11] or possibly the $\{\bar{1}2\bar{1}0\}$ $\langle\bar{1}011\rangle$ system inferred by Gulden [12a, b]. If rhombohedral slip ($\{10\bar{1}2\}\langle\bar{1}011\rangle$) is indeed activated, three independent slip systems result from the three $\langle\bar{1}011\rangle$ translations allowed in α-Al_2O_3. This slip system, together with basal slip, would yield five independent slip systems and result in ductile behaviour in α-alumina when these slip systems are free to interpenetrate. The increased plasticity with increasing temperature, coupled with the enhanced activity of $\langle\bar{1}011\rangle$ dislocations in the A body at 1700° C, is clearly indicative of probable ductility in polycrystalline α-Al_2O_3. In addition, the author has observed similar behaviour, initiated at 1420° C, in fine-grained alumina where plastic strains of at least 10% could be accommodated under similar test conditions [8b, 9].

The ease of activation of multiple slip systems at these temperatures (especially 1700° C) is also taken to be a positive factor in the reduction of mechanical twinning. This mode of deformation was much more prevalent at temperatures below 1420° C [8b, 9], a point which is consistent with a multiple slip type of deformation. Furthermore, although multiple slip is more prominent at 1700° C, diffusional processes were sufficient at this temperature to substantially reduce the rate of strain-hardening. This is apparent in the prevalence of polygonisation of dislocations at 1700° C, compared with that occurring at 1420 °C. In addition, there is a possible increase in the ease of interpenetration of slip systems aided by the increased thermal energy.

The behaviour of the purer A body is contrasted somewhat by that reported for fine-grained alumina deformed in bending [2, 3]. However, as noted by Heuer *et al* [3], their data indicated an increasing contribution of non-viscous grain-boundary shear with decreasing grain size (10 to 1 μm) similar to the behaviour observed in metals [13]. The present observation that boundary shear by dislocation mechanism plays a relatively minor role in 20 μm alumina tends to support an increasing contribution of this type of boundary behaviour with reduction of grain size in purer aluminas. In this case, decreasing grain size can also result in decreased impurity concentration at a grain boundary, reducing boundary shear phenomena related to impurities (overall, impurity levels and species being approximately equal). Therefore, in much purer or in fine-grained alumina of relatively high purity (99.9 %), an intrinsic, boundary shear, involving dislocation motion, could be much more prominent and, in conjunction with multiple slip within grains, foster the ductile behaviour of alumina. This should be tempered by the fact that only $\langle 11\bar{2}0\rangle$ type dislocations were observed in this study to participate in a non-viscous boundary shear behaviour, coupled with the occurrence of interfacial shear in non-doped (0001) bicrystal boundaries [14] and not in similar $\{11\bar{2}6\}$ interfaces [8a, b, 9]. This suggests that boundary-shear dislocations might have only those Burgers vectors characteristic of matrix glide dislocations, which would require various degrees of glide and climb in order to achieve non-viscous grain-boundary sliding; alternatively, such sliding may occur by a zonal shear process in α-alumina.

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