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# Tensile failure of unflawed polycrystalline $Al_2O_3$

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Scanning electron microscopy and acoustic emission are used to investigate the initial stages of tensile failure in unflawed polycrystalline alumina. It is found that deformation twinning plays an important role in crack initiation even at low homologous temperatures, and that the temperature-dependent strength behaviour between 23 to 410° C is controlled by twinning.

## 1. Introduction

Polycrystalline aluminum oxide is generally considered to be an archtype brittle ceramic. Yet for many years, it has been known that the tensile strength of $Al_2O_3$ is characterized by certain behaviour occurring at relatively low homologous temperatures whose basis has been laid to at least limited plasticity. Generally, this plasticity has been related to thermally-activated events at the tips of sub-critical cracks.

The kind of strength behaviour under discussion is shown in Fig. 1 [1], in which it can be seen that the bend strength first decreases with increasing temperature until around 400° C, above which point a dramatic increase in strength is observed. It has been suggested [2–4] that the strength increase might be caused by thermally-enhanced local crack tip plasticity, causing crack blunting or bifurcation. However, detailed TEM study by Weiderhorn, Hockey, and Roberts [5] has demonstrated conclusively the absence of dislocations at the tips of arrested tensile cracks in $Al_2O_3$. Manifestation of the strength minimum *in vacuo*, moreover, indicates that the effect is not environmental. It has been proposed [5] that some sort of thermally-activated complexing at crack tips might provide an explanation, but the possible structure and strength-enhancing mechanism of this "complex" has been discussed only sketchily.

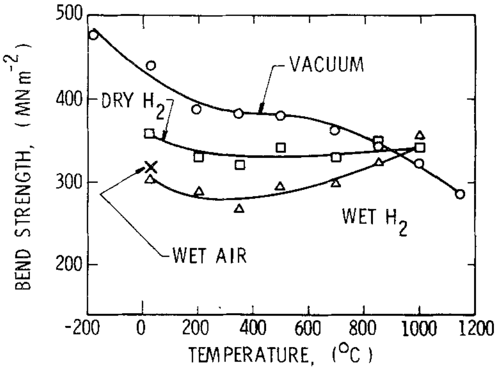

*Figure 1* Temperature–atmosphere effects on the bend strength of Lucalox [1].

Recently the writer reported the results of experiments in which the compressive strength of a polycrystalline aluminum oxide was determined as a function of strain rate and temperature [6]. The presence of a strength minimum at 200–300° C was reminiscent of the similar effect described above for the tensile case. This suggested that the same mechanism (twin-nucleated microcracking) responsible for the compressive strength minimum might explain the tensile strength behaviour as well. Indeed, cursory investigation of a few tensile fractures supported this idea. In this paper, the results of a more detailed look at tensile failure in Lucalox are reported, and are shown to support the suggestion that the strength minimum in unflawed $Al_2O_3$ is caused by thermally-activated deformation twinning, which in turn controls crack initiation.

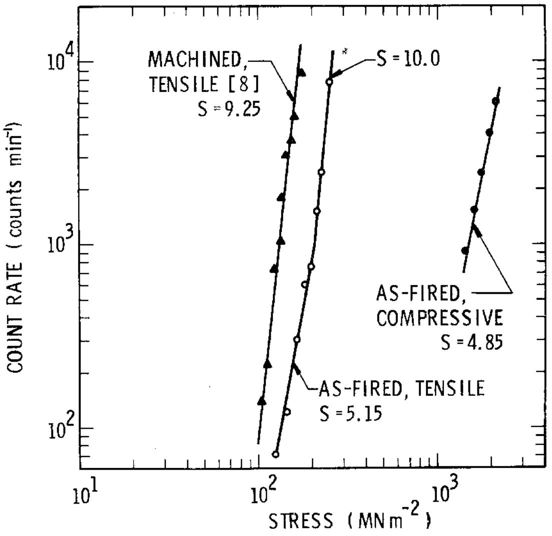

*Figure 2* Acoustic emission count rate versus applied stress for Lucalox.

## 2. Experimental procedure

As-received rods of polycrystalline alumina (type GW Lucalox*) were cut to length for three-point bending tests, and their ends ground flat for attachment of an acoustic emission transducer. Prior to testing, specimens were cleaned ultrasonically and rinsed in alcohol and distilled water.

Bend tests were carried out at a strain rate of $1 \times 10^{-5}$ sec$^{-1}$, at temperatures of 23 and 410° C. Acoustic emission within the frequency domain 100 kHz to 1 MHz was monitored during room temperature testing, using a PZT transducer resonant at 160 kHz (the acoustic emission apparatus and procedures are discussed in detail elsewhere [7]). The acoustic emission detection sensitivity level used in the tensile tests was identical to that used in similar compression tests [7]. The purpose of the three-point bend tests, as opposed to four-point, was to localize any tensile damage and thereby facilitate its detection visually. Similarly, the major goal of the acoustic emission work was to determine the stress level at which tensile damage commenced. In addition, it will be seen that the acoustic emission is helpful in actually assessing the failure micromechanism.

Most of the specimens were tested to failure and then studied in the SEM. A few were loaded to varying sub-failure stress levels and then unloaded for study of early damage. All specimens were coated with palladium for SEM observation.

## 3. Results

### 3.1. Acoustic emission

A particularly instructive format for utilizing the acoustic emission results is to plot the log of the count rate $dN/dt$ versus the log of applied stress $\sigma$, as shown in Fig. 2. Here, in addition, are shown earlier tensile results by Evans *et al* [8], for as-machined Lucalox, and recent compressive results [7] for the same as-fired Lucalox used in the present study. In all three cases shown, the strain rates and test temperatures are approximately the same. For the tensile-loaded as-machined material, the slope $S$ of log $dN/dt$ versus log $\sigma$ is essentially constant. On the other hand, the log $dN/dt$–log $\sigma$ curve for the as-fired material loaded in tension is divided into two regions of varying slope. The initial value of $S$ is considerably lower, by a factor of 50%, than for the as-machined Lucalox. At higher stresses, a transition occurs, with $S$ approximately equal to the as-machined value. It is interesting to observe that during early stages of damage, $S$ for the as-fired alumina seems to be independent of the mode of loading, i.e. the slope is approximately equal for both compressive and the first stage of tensile loading. However, both the threshold stress ($\sigma_{AE}$) for acoustic emission in tension, and also the initial count rate, are approximately an order of magnitude lower than the corresponding parameters in compression.

Bend strength ($\sigma_T$) and acoustic emission threshold stress results are summarized in Table I. The average strength level at 410° C is only slightly lower that that at 23° C, and according to the results of Fig. 1, should reflect the presence of whatever micromechanism is responsible for the general strength increase with rising temperature.

### 3.2. Scanning electron microscopy

Prefracture deformation is more difficult to locate at 23° C than at the higher temperature. However, a diligent scanning of regions adjoining the fracture surfaces of specimens failed at room temperature does reveal evidence of micro-

TABLE I Bend test results

| Temperature (° C) | $\sigma_T$ (MN m$^{-2}$) | $\sigma_{AE}$ (MN m$^{-2}$) |
|---|---|---|
| 23 | 215 ± 23 | 102 ± 17 |
| 410 | 177 ± 25 | – |

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* General Electric Lamp Glass Division, Cleveland, Ohio.

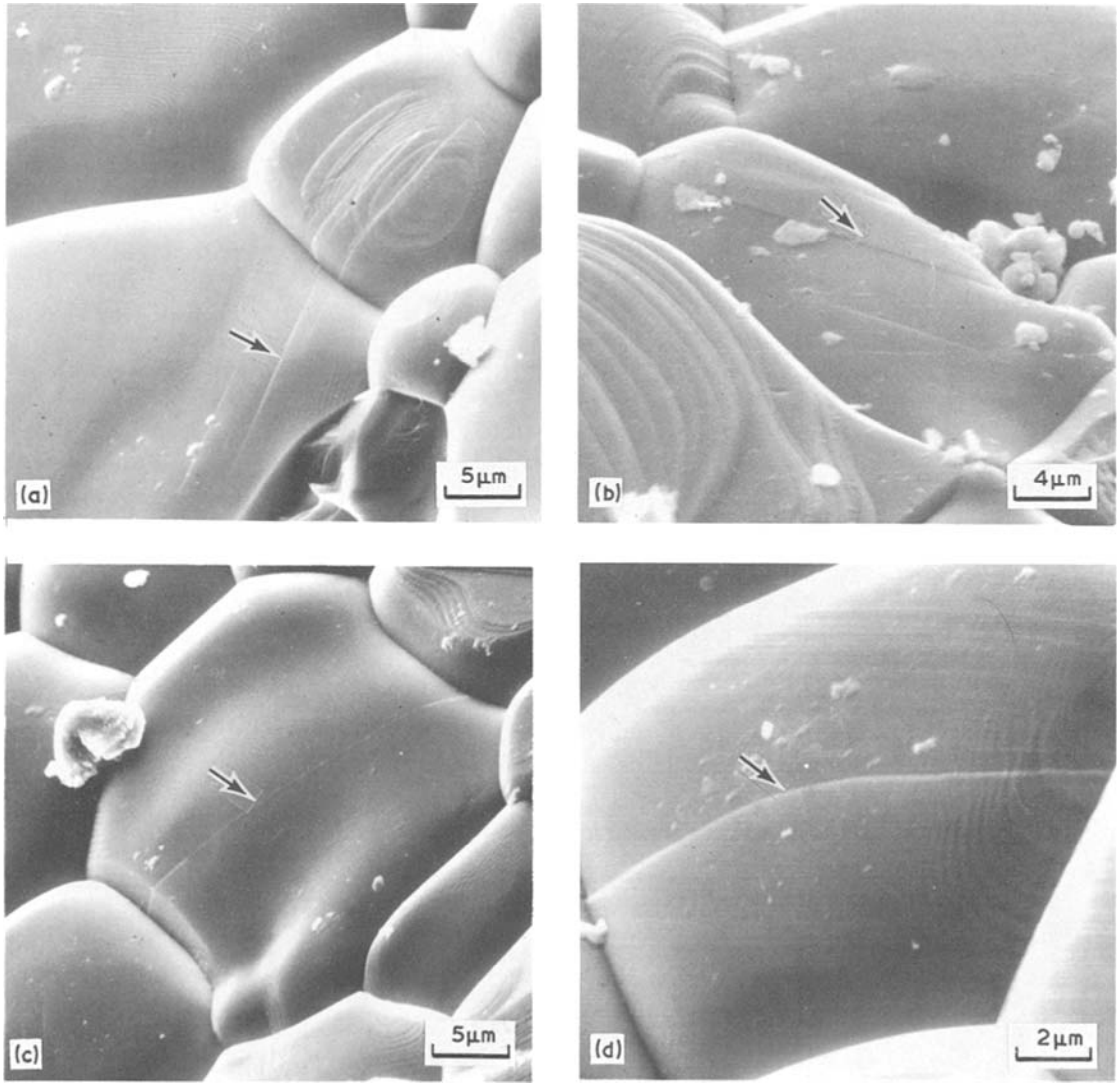

*Figure 3* Microplasticity (arrows) produced near tensile fracture surface at 23° C.

plasticity and indicates its role in the failure process as well.

In Fig. 3, several examples of microplasticity produced by uniaxial tensile (bending) stresses at 23° C can be seen. These areas all were located within a few grain diameters of the fracture surface (in each of the photomicrographs to be discussed, the stress axis is vertical). Whether these particular markings reflect twinning or slip cannot be determined with certainty, since they are so narrow. However, they do closely resemble surface deformation produced in the same material by compressive loading, and positively identified as twinning [6]. A comparison between such defects caused by tensile and compressive stresses is shown in Figs. 4a and b, respectively. Identification of the compressive flaws as twins was facilitated by the fact that they often are reasonably broad and permit observation of offsets in surface scratches. The tensile twins, particularly at room temperature, seem to be much thinner.

The possible role of the twins in causing tensile failure is suggested by Fig. 5. For the bend specimen shown in Fig. 5a, the apparent fracture origin was the thumbnail region indicated by the arrow. This region was immediately opposite the centre support in the three-point bend rig. Near the centre of the thumbnail, the grain shown in Fig 5b was located. This grain exhibits the same

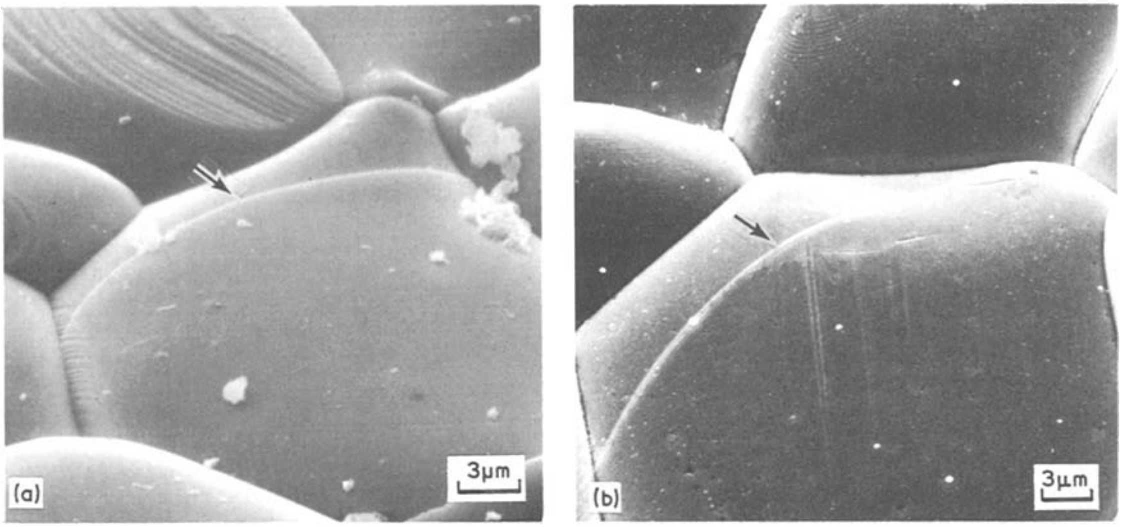

*Figure 4* Comparison of surface microplasticity (arrows) produced in tension and compression at 23° C (a) tensile (b) compressive.

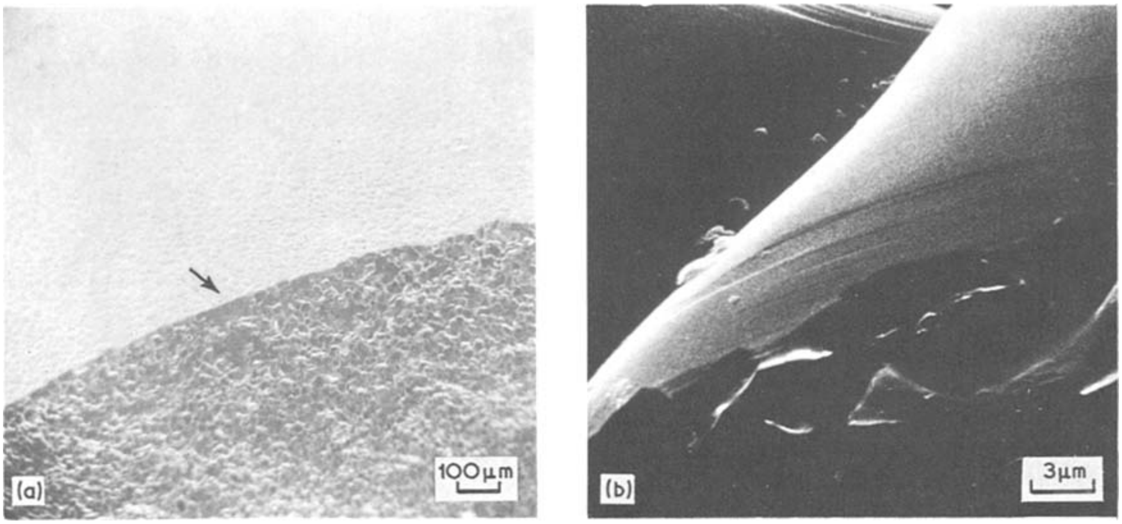

*Figure 5* Relationship between twinning, microfracture, and tensile failure. (a) Fracture surface; arrow indicates origin of failure, (b) grain associated with initial flaw, showing apparent twin-induced microfracture.

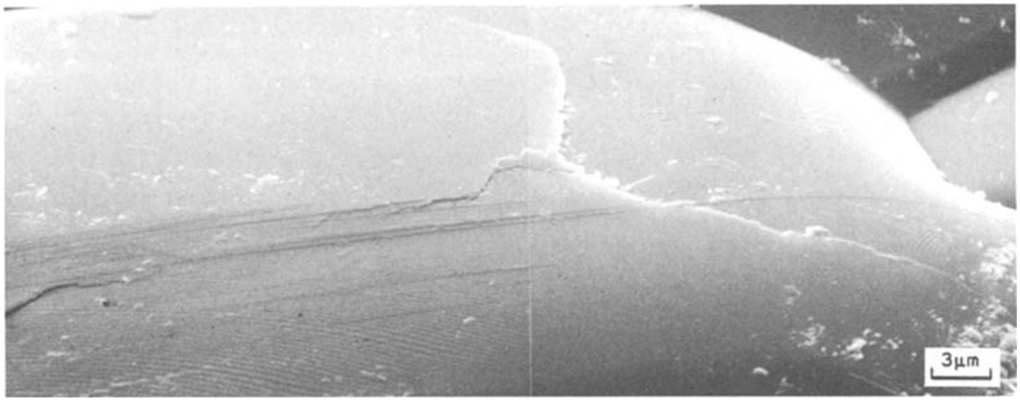

*Figure 6* Twinning under tensile loading at 410° C.

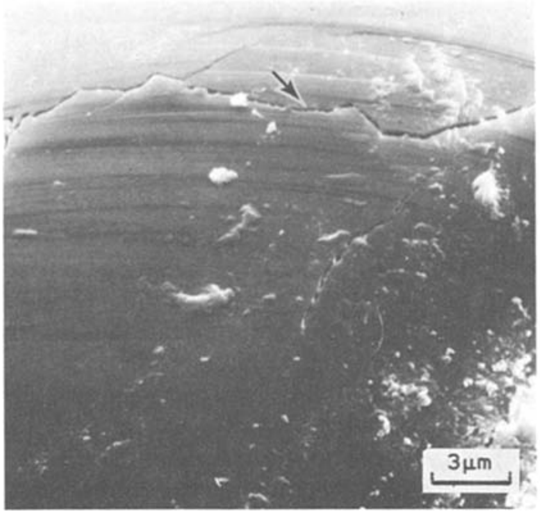

*Figure 7* Twinning and twin-plane microcracking (arrow) under tension at 410° C.

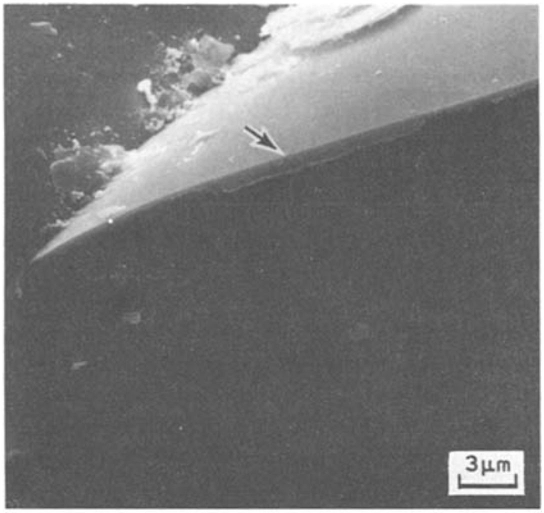

*Figure 9* Possible twin facet (arrow) near tensile fracture origin ($T = 410°$ C).

twin-like features noted previously, with the fracture plane appearing to wander along several twin planes. Such behaviour has been observed during compressive microfracture of Lucalox, and is typical of twin-nucleated cracking [6] in this material.

Similar twinning and twin-related cracking are found for tensile tests carried out at higher temperatures. In Figs. 6 and 7, extensive fine-scale twinning has occurred in grains adjacent to the major fracture surface, with microcracks having initiated within some of the twins (note the wandering of the crack (c) in Fig. 6b from one twin plane to another). Also at higher temperatures, it is observed that multiple twin systems begin to be activated, as was the case during compressive testing [7]. This is shown in Fig. 8, where intersecting twins (T) have initiated cracks within (C1) and normal to (C2) twin traces. Some of the twins nucleated at 410° C achieve a significantly greater thickness than those found at room temperature. These twin facets were often noted near failure nucleation sites; such a location is shown in Fig. 9, in which the local fracture has wandered along a possible twin facet (arrow).

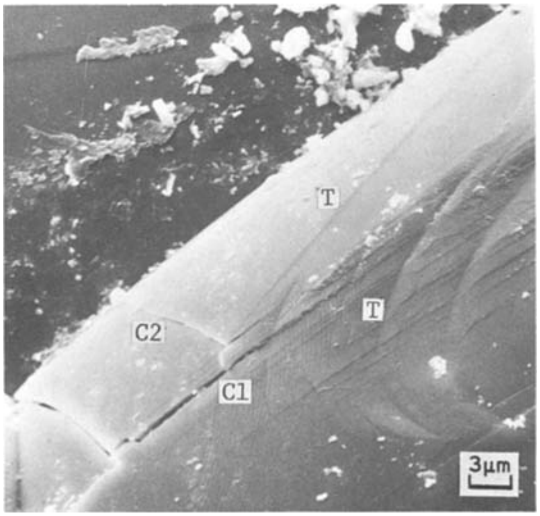

*Figure 8* Multiple twin systems activated in tension at 410° C. Intersecting twins produce cracks within (C1) and normal to (C2) twin traces.

## 4. Discussion

The first indication that some mechanism other than microcracking alone is responsible for the observed thermally-activated tensile strength behaviour can be deduced from the acoustic emission results. Earlier, Evans *et al.* [8] had developed an analytical model for acoustic emission due solely to microcracking in bulk specimens. Based on this model, the slope of ln ($dN/dt$) versus ln ($\sigma$) should be approximately 10. This was found to be the case for their as-machined Lucalox specimens, which contained a large population of prior flaws, i.e. machining cracks, and which gave a slope of 9.25 (Fig. 1). In the present case, the much lower initial slope and the markedly reduced count rate for equivalent stresses point to the operation of some mechanism other than microcracking to account for the early stages of acoustic emission in unflawed $Al_2O_3$. As noted by Evans *et al.* [8], the only other obvious candidates are dislocation motion and twinning. The fact that the compressive and early stages of tensile stressing of unflawed Lucalox produce essentially the same $\ln(dN/dt)$ versus $\ln(\sigma)$ slope implies that the source of acoustic emission is identical in both cases. SEM work previously has proven twinning to be the earliest source during compression [6], and the present electron optical observations are compatible with the same conclusions for tensile failure. However, it is more difficult to separate twinning from possible slip traces in the latter case, since the apparent tensile twins are not as thick as those generated during compression.

Certain work by others is in general agreement with the idea that twinning controls tensile fracture in cases such as the present one. More than ten years ago, Heuer [9] showed that deformation twinning was associated with fracture initiation in corundum single crystals broken in bending at temperatures as low as $-196°$ C. Later, Congleton *et al.* [2] showed that there exists a minimum at 250° C in the tensile fracture stress of polycrystalline specimens containing drilled holes. Two possible explanations for this minimum were offered: (1) temperature-dependent crack tip plasticity, or (2) thermally-activated crack initiation involving slip or twinning. The TEM work by Wiederhorn *et al.* [5], discussed earlier argues against the former. Recently, Becher [10] has shown that the tensile failure of as-ground sapphire bars is controlled by grinding-induced deformation twins, along the habit planes of which the initial microfractures propagate. The implication here is that while grinding introduces several forms of surface damage, including microcracking, slip, and twinning, the propensity for the twin/parent interface to fail under tensile loading, combined with the size of the twins, can cause twinning to dominate as the principal failure mechamism. Finally, deformation markings similar to some of those seen in the present study have been observed by Coble and Parikh [11] on Lucalox deformed in tension. However, the markings at that time could not be unambiguously associated with failure, and their identification as twins was only tentative. The present results indicate the correctness of these earlier speculations [11].

It is interesting to consider the stress levels apparently required for twin nucleation in tension and in compression. Acoustic emission indicates that twinning during tensile loading ($T = 23°$ C, $\dot{\epsilon} \sim 10^{-5}$ sec$^{-1}$) commences at around 130 MN m$^{-2}$, while compressive loading under similar conditions requires a stress of 1400 MN m$^{-2}$ to initiate twins, as shown in Fig. 2. The reason for this ten-fold strength differential may be related to the inherent stability of flaws nucleated under compression as compared to those formed in tension.

It is possible that during compressive deformation, microtwins actually nucleate at stress levels significantly lower than those detected by the present acoustic emission system. However, it is already well known [6] that flaws such as microcracks, once nucleated in compression, are exceptionally stable and frequently require much higher stress excursions in order to extend. If the stress wave displacement due to initial compressive twinning were below the minimum displacement sensitivity of the acoustic emission system, as might be the case for very small microtwins, then the presence of the twins would be undetected. Detectable acoustic emission would then occur at higher stresses when the twins are driven completely across grains, which is generally accomplished without cracking the twin/parent boundary; rather, it is more common in compression [6] for cracks to nucleate at twin/grain boundary intersections. Usually the twins are stable enough to grow sideways to achieve a finite thickness. On the other hand, microtwins formed in tension may be less stable. They usually are very thin, tend to extend completely across and often are microcracked along the twin/parent boundary. A suggested scenario is one in which the first twins nucleated in tension zip across a grain as soon as they are formed, accompanied by stress wave emission. Upon reaching a critical size on the other of the grain size, the weak twin/parent interface frequently parts in tension, which effectively precludes further thickening of the twin. At higher stress levels, these cracked twins themselves begin to serve as active flaws, causing crack initiation and extension in adjacent grains. Thus, they can from this level on be considered as "microcracks," whose acoustic emission response undergoes a change from that characteristic of twinning to that corresponding to microcrack extension. This is manifested in the $S$ value for higher stresses being approximately equal to that found by Evans *et al.* [8], for initially microcracked (machined) specimens.

The fact that twinning begins to occur on multiple systems at higher temperatures explains the observed temperature dependent strengthening effect. Plastic accommodation at local stress concentrations through multiple twinning reduces the necessity for crack formation. Moreover, cracks which do occur in such multiple twin fields tend to interfere and interact with one another rather than running neatly across each twinned grain. This could reduce the propensity for further crack extension in such regions, thereby effectively raising the failure stress.

## 5. Conclusions

The temperature-dependent strength behaviour of unflawed polycrystalline alumina in the temperature range 23 to 410° C is caused by deformation twinning through its role in microcrack initiation. The twinning is responsible for acoustic emission, whose count rate is dependent upon stress according to the same relationship observed for twinning in compression. The strengthening effect at higher temperatures is caused by enhanced twinning generally, and multiple twin system activity in particular, which aids in accommodating local strain concentration.

## Acknowledgement

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