EFFECT OF INTERFACE ROUGHNESS ON CRACKING OF ANODIC OXIDE FILM ON ALUMINUM

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INTRODUCTION

The character of the interface between two components of a composite has an important bearing on the overall behaviour of the composite. One factor is its roughness, which would control the degree of mechanical gripping and keying between the two components; even after the failure of the bond, keying will permit load transfer between them. Tylecote [1], for example, observed that a rough interface between NiO and Ni led to enhanced adherence between the two. Hill et al [2] studied the mechanical bonding in Al/W. The interface roughness was obtained by etching the tungsten fibres at places. Composites containing unetched W fibres, with a graphite coating to prevent any chemical bonding between Al and W, showed 35% of the theoretically expected strength while composites containing W fibres etched at places to give a rough interface, and coated with graphite, showed 91% of the theoretical strength.

We have examined the effect of the interface roughness on cracking of anodic oxide film ("reinforcing element") on aluminum ("matrix element"). When such a simple composite is deformed, Al deforms plastically and transfers load to Al_2O_3. The latter deforms in conformity with the former until it cracks in one of several ways [3, 4, 5, 6]. In thick anodic oxide films it was found [5, 6] that cracks ran normal to the tensile axis and ignored grain boundaries, indicating an extensive detachment of the film. After a certain deformation, the coupling between the components vanished, additional strain in the matrix was not transferred to the reinforcing phase and the existing cracks widened. Edeleanu and Law [5] explained that 'interphase slip' between the two components must take place for such widening. Interphase slip may be sensitive to interface roughness; with a rough interface there may be restraints on the relative movement and efficient load transfer may occur up to larger deformations and more cracks may form.

EXPERIMENTAL PROCEDURE

Polycrystalline specimens of 1 x 6 mm section were prepared by machining cold rolled 99.999% aluminum. The specimens were given an annealing pretreatment - at 423K for 6 hr - to obtain a uniform grain size, viz. 0.045 mm diameter.

The specimens were glued to steel ball grips, 38 mm apart, in an alignment jig, using epoxy cement. The ball grips were stopped off for surface treatment. Electropolishing was next carried out in a perchloric acid-ethanol-ether bath. The electropolishing film was stripped in a phosphoric acid-chromic acid bath at 368K and the stripped specimens anodized in 3%

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tartaric acid solution. The pH of the solution was adjusted to 5.5 with ammonium hydroxide. The thickness of the anodized film formed at 90V was estimated to be ~ 120 nm (about 1.37 nm/V). The voltage was increased to the final value slowly, so that the current density did not exceed ≈ 100 A/m^2.

Interface roughness was obtained by chemical etching and mechanical indentation of Al before stripping and anodizing. In either case, bright electropolished specimens were prepared with discrete pits or indentations so that the cracks would be readily visible optically on bright smooth areas. A few (relatively large) isolated, gently sloping pits were obtained by swabbing the specimen for 3-4 s with an etchant consisting, in percent by weight, of HF: 5.3%, HCl: 44.5%, HNO_3: 12.6% and H_2O: 37.6%. Small uniformly distributed pits were obtained by immersing the specimens in 29% HF for 5-7 s at room temperature.

Isolated pits were also obtained by mechanical indentation with a Vickers indentor under a load of 1g in a Tukon tester. This was done after polishing, and the specimen was annealed at 373K for 3 hr to recrystallize the indentation. In all cases, the specimen was subsequently stripped and anodized at 90V to yield a relatively thick film in which cracks were readily visible optically.

The specimens were strained in a small rigid jig placed in an Ultraphot metallograph. The force was applied through a large micrometer head and the strain sensitivity was 0.01%. The specimens were viewed during straining and several regions were photographed at various stages of elongation in each case.

RESULTS

(a) *Small pits with Steep Walls and Sharp Edges.* Figure 1 shows three photomicrographs from a series taken with a uniformly pitted interface. Figure 1a shows the initial stage of cracking: a few cracks which are short and run from one pit to another have formed. Figure 1b shows the film at 8.7% elongation: more cracks which are also short and usually run between two pits can be seen. The cracks which had formed earlier do not lengthen but they widen noticeably, as for example, crack A. Figure 1c shows the film at a much higher strain - 17.3%: a few fine cracks have formed. These are short, like the ones formed at smaller strains. Crack A has widened further.

It is clear from these observations that the pits act as crack arrestors but their effect on crack initiation could not be specified. Although the specimen was under constant observation during straining, growth of individual cracks was not observed. They formed very suddenly to full length. Therefore the point of origin of each could not be specified, i.e., whether at a pit or a smooth part of the surface.

In the case of a smooth interface, where the crack ran across the width of the specimen, the crack density was the same for any longitudinal sampling line on the specimen. The crack density of short cracks in the pitted interface was determined from the average number of cracks per mm intersecting six longitudinal lines drawn $1^{1/2}$ crack lengths apart on the photomicrograph of a representative field. The results of these measurements are given in Figure 2 which shows plots of number of cracks per mm vs .% elongation.

The crack density, starting at a little over 1% elongation, became constant at 72 cracks per mm. The crack density for the film with a pitted interface was about the same initially as that with the smooth interface, i.e., the cracks first appeared at about the same strain and the crack density rose with strain at about the same rate. However, although the crack density with a smooth interface remained constant above 8% strain, it continued to increase in samples with a pitted interface, although at a slower rate.

(b) *Isolated Large Pits with Gently Sloping Walls.* Because the large gently sloping pits were isolated, observations relating to nucleation could be made. It was found that they did have some influence on crack nucleation. A number of cracks could be seen growing away from the pits. If the specimen was examined when cracking had just started, almost every crack was associated with a pit and there were very few in the pit-free region of the specimen, and Figure 3 shows two representative photomicrographs of a series. Figure 3a, which represents just the beginning of the cracking, 1.8% elongation, shows cracks which originated somewhere at the pits and started spreading in both directions. There is one crack, indicated by an arrow in the figure, not at a pit (perhaps at a grain boundary). However, with only a slight amount of further straining (i.e., to 1.9% elongation), the film was seen to crack at many places without any particular preference for the pits. Therefore, any contribution of these pits to crack nucleation was not an important or essential part of the cracking process. On further straining the transverse cracks continued to form regardless of pits. Figure 3b shows this stage. One notes that many cracks which intersect pits started away from the pits, some of these being arrested at the pits, while others pass through them (visible on focusing into the pit) and continue on the other side. From this, it can be concluded that these pits are less efficient crack arrestors than the small sharp pits.

DISCUSSION

From Figure 2, showing the kinetics of film cracking in specimens with smooth and pitted interfaces, one can see that the major difference between the two is that the crack density in the film with an interface with sharp pits goes on increasing continually with strain, long after it has attained a constant value in the film with a smooth interface. Thus, load transfer from Al to Al_2O_3 continues in a sample with a pitted interface long after it has stopped in one with a smooth interface.

A schematic diagram (Figure 4) of cracking in an interface with sharp pits, shows why a greater number of cracks is expected to form in this case. The film is considered to be completely detached at this stage, but is unable to slide over Al because it is hooked at the various pits in the interface, such as at A and B. With the elongation of the Al matrix, the portion of film between A and B is under tension T while in areas where the film has relaxed because it has cracked (e.g., C in Figure 4) the cracks open up. The cracks form in the stretched regions such as AB, where the load transfer from Al to Al_2O_3 can still occur. Such cracks lead to a higher crack density in the case of films with a sharply pitted interface. These cracks nucleate on a longitudinal line between two pits, but may have only limited lateral growth because the film is stretched less on either side of the line. The implication is that a rough interface in a real composite other things being equal, would lead to a more efficient load transfer from the matrix to the fiber.

As to the crack arresting property of these sharp pits it is supposed that the film becomes detached inside the pit but remains hooked at the sharp edges of the pit. Figure 4 shows the film detached inside the pit. It is possible that some load is transmitted part way down the film along the sides of the pit but mostly the stress is concentrated at the hooking points (B and D) and the film in the bottom part of the pit is essentially stress free. As the matrix elongates over the length of the pit it is supposed that the film in the pit accommodates this by bending as suggested in Figure 4. Now, if any crack runs into the pit it will meet an almost stress-free region of the film and hence its further growth is stopped.

According to this explanation the crack arresting property is essentially a consequence of the steep walls and sharp edges of these pits. In the case of the isolated (larger) pits, shown in Figure 2, and the Vickers indentations, both of which have gently sloping walls, the film may be flexible enough to follow the contours of the pit. The film then slides much as on a smooth interface; consequently, these pits would not exhibit such strong crack arresting properties as pits with sharp edges. This is in conformity with the observations.

CONCLUSIONS

Roughness of the interface between Al and Al_2O_3, in the form of sharp steep-sided pits, produced shorter cracks and higher crack density in the oxide films at larger strains as compared with a smooth interface. This is explained by the hooking of the film at the sharp pit edges so that if no crack lies between two pits, free interphase slip is prevented in this region and load transfer from Al to Al_2O_3 continues to occur with further strain. This accounts for both the higher crack density and the arresting of crack growth in the stress free part of the film within the pit.

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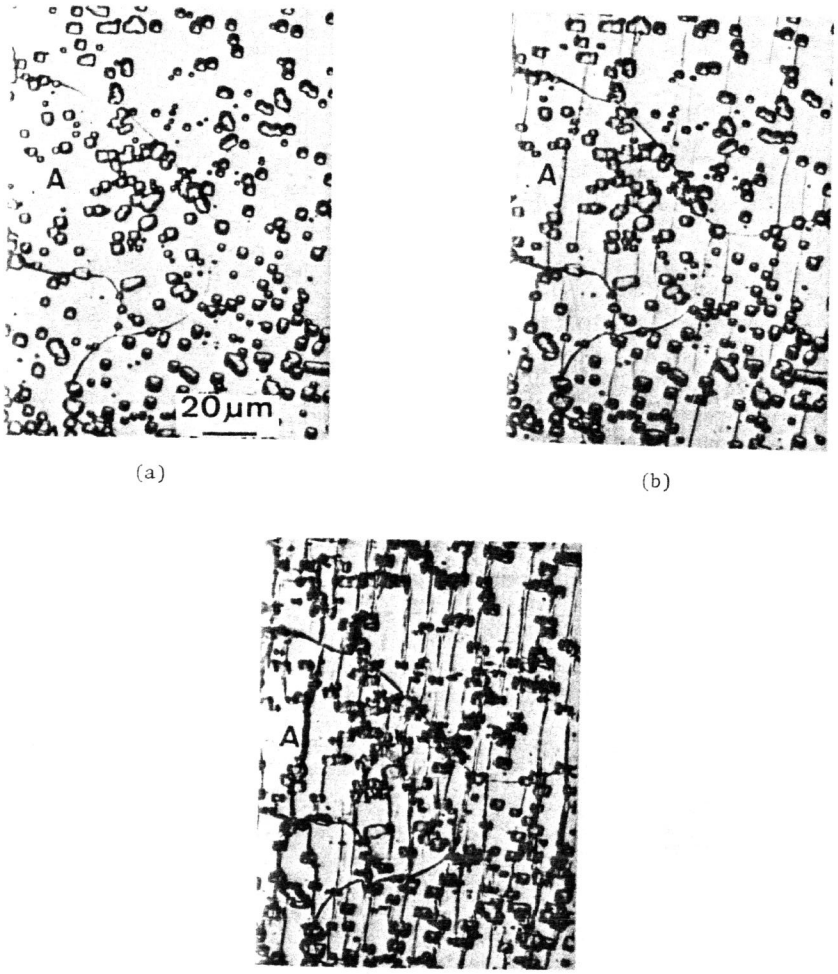


(a) (b)

(c)

Figure 1 Cracking in Al_2O_3 on Al with a Sharp Pitted Interface. Specimen Axis Parallel to Magnification Marker. (a) e = 2.3%, (b) e = 8.7%, (c) e = 17.3%

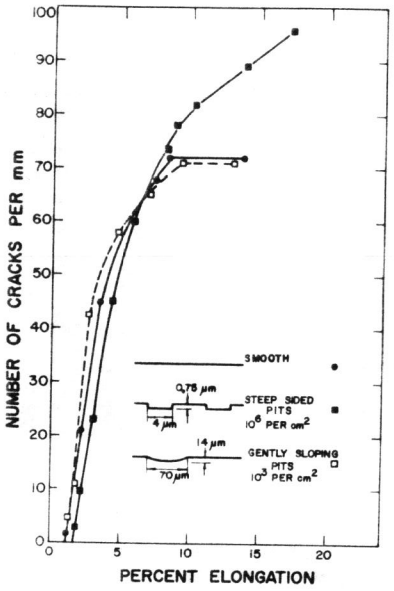
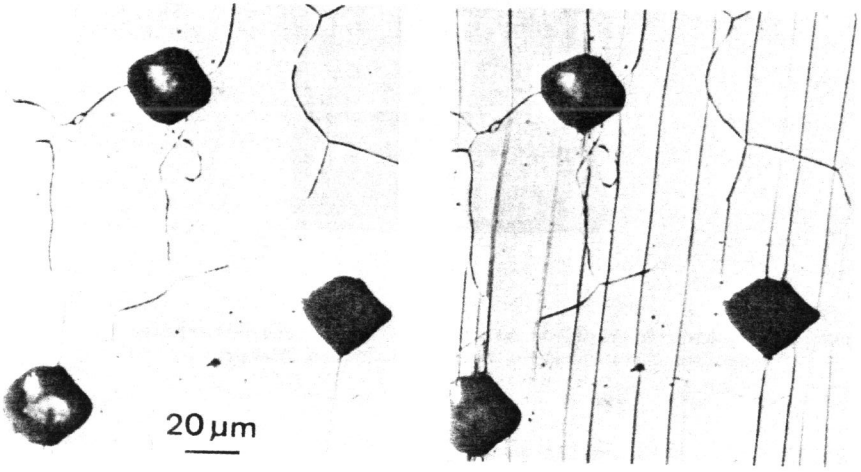


Figure 2 Kinetics of Cracking of Al_2O_3 on Al with Smooth and Pitted Interfaces



(a) (b)

Figure 3 Cracking of Al_2O_3 when the Interface had a few Isolated Gently Sloping Pits. Specimen Axis Parallel to Magnification Marker. The Grain Boundaries are Visible Through Small Differences in Level Produced by the Pre-Etch. (a) e = 1.8%, (b) e = 7%

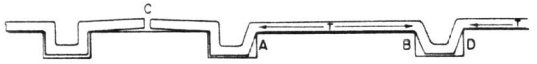


Figure 4 Schematic of Cracking in Al_2O_3 on Al when the Interface Contains Sharp Pits