

The relationship between microstructure, fracture and abrasive wear in $\text{Al}_2\text{O}_3/\text{SiC}$ nanocomposites and microcomposites containing 5 and 10% SiC

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Received 12 February 2009; received in revised form 12 March 2009; accepted 13 March 2009

Available online 2 May 2009

Abstract

Alumina/SiC nanocomposites are much more resistant to severe wear than monolithic alumina. In order to clarify the mechanisms responsible for these improvements, alumina and alumina/SiC nanocomposites with 5 and 10 vol.% SiC and various alumina grain sizes were fabricated. For comparison, a 10 vol.% SiC “microcomposite” was also fabricated using $3\ \mu\text{m}$ SiC particles. The extent of cracking beneath hardness indentations was examined and the specimens were tested in abrasive wear. Quantitative surface fractography of the worn surfaces was carried out. The wear properties depended strongly on the grain size in pure alumina, but were independent of the alumina grain size in the nanocomposites. This is consistent with the idea that much of the improvement in wear resistance when SiC is added to alumina stems from a reduction in the size of the individual pullouts owing to the accompanying change in fracture mode. In addition, crack initiation by plastic deformation during abrasion and indentation was found to be strongly inhibited when 10 vol.% nanosized SiC was added to alumina. The addition of $3\ \mu\text{m}$ “micro-sized” SiC did not have the same effect. The ability of fine SiC particles to suppress cracking is attributed to the blocking of twins and dislocation pileups by intragranular SiC nanoparticles. This reduces the length of the twins or pileups and hence their ability to nucleate microcracks.

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Keywords: Nanocomposites; Fracture; Wear; $\text{Al}_2\text{O}_3/\text{SiC}$; Microstructure

1. Introduction

Alumina/SiC “nanocomposites” are produced by the incorporation of nanosized SiC particles in an alumina matrix with typical grain size $\sim 1\text{--}5\ \mu\text{m}$.¹ The most striking and reproducible improvement in properties of the nanocomposites is in their resistance to severe wear. The nanocomposites have better surface finish under the same grinding conditions.^{2,3} Erosive,^{4,5} sliding^{6,7} and abrasive wear tests^{8,9} also show that the nanocomposites are more wear resistant than monolithic alumina of a similar grain size. It is reasonable to suppose that the improved surface finish is at least partly responsible for the increased strength of the nanocomposites that has been reported.^{1,2,10}

Abrasive wear tests have previously been carried out on alumina and 2 vol.% SiC nanocomposites by Ortiz Merino and

Todd.⁸ Analysis of the results showed that the improvement of the wear resistance and surface finish of 2 vol.% SiC nanocomposites was mainly due to a reduction in the size of individual surface pullouts caused by brittle fracture. It was suggested that this is a geometrical consequence of the well known change in fracture mode from intergranular in alumina to transgranular in the nanocomposites. A key prediction of this explanation is that the wear behaviour of the nanocomposites should be independent of the grain size, in contrast to the wear rate of alumina which increases strongly with grain size. However, the wear rate of 2 vol.% SiC nanocomposites was grain size dependent, though less so than for alumina. Ortiz Merino and Todd suggested that this was because some intergranular fracture was still observed on the worn surfaces of nanocomposites containing only 2 vol.% SiC but that the prediction might be borne out if the fracture mode could be made completely transgranular. One aim of this paper is therefore to test this prediction by investigating the grain size dependence of wear in composites containing higher volume fractions of SiC (i.e. 5 and 10 vol.%), in which fracture might be expected to be more completely transgranular.

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In addition to the geometrical mechanism in 2% SiC nanocomposites described above, Ortiz Merino and Todd⁸ also found evidence that the occurrence of surface cracking itself is suppressed when more SiC is added (10 vol.%), i.e. fewer pullouts are produced as well as each one being smaller in size. This was based on a single result, however, and the second aim of this paper is to investigate this possibility using a wider range of microstructures. The extent of crack formation has been assessed indirectly, by analysing the results of an abrasive wear test as in Ref. [8] and directly, by examining the extent of cracking beneath Vickers hardness indentations using post-indentation polishing.¹¹ The influences of SiC volume fraction, particle size and alumina grain size have been investigated.

2. Experimental procedure

2.1. Material and specimen preparation

AKP50 alumina powder (Sumitomo, Japan, 200 nm) and UF45 SiC powder (Lonza, Germany, 260 nm) were used to prepare pure alumina samples and Al₂O₃/SiC nanocomposites containing 5 and 10 vol.% SiC. The addition of 0.25 wt% MgO was used to prevent abnormal grain growth. Dispex A40 (Ciba, Bradford, UK) was used as a dispersant (2.1 wt% of the weight of the solid). Distilled water was added with the ratio of 4:1 water to powder by volume. The slurry was attrition milled (Szegvari HD-01, USA) using YSZ milling media and freeze dried (Edwards Micromodulyo, UK). The powder was then passed through a 150 μm sieve and calcined at 600 °C in air for 1 h to remove the dispersant. Dense specimens (25 mm diameter and 4 mm thickness) were fabricated by hot pressing in a graphite die for 30 min at 25 MPa in an argon atmosphere at temperatures between 1400 and 1700 °C in order to obtain a range of grain sizes.

A small grain size alumina sample ($G = 1.3 \mu\text{m}$) was also fabricated by spark plasma sintering at 1550 °C with a heating rate of 500 °C/min and no dwell time under a pressure of 40 MPa.

A 10 vol.% SiC “microcomposite” was also produced, using 3 μm α-SiC particles (Washington Mills, UK) and AES 11c alumina (Sumitomo, Japan). These were ball milled in water with Dispex A40 dispersant, spray dried (Büchi, Switzerland) and hot pressed at 1700 °C as above. The resulting grain size was 5.7 μm.

The Archimedes method was used to measure the density of the samples produced. Polished and thermally etched surfaces were coated with gold to prevent charging and SEM (JSM 840F) was used to investigate the microstructure. Grain sizes were measured by the linear intercept method with a multiplication factor of 1.56. At least 300 intercepts were counted for each measurement.

2.2. Wear test and fractographic analysis of worn surfaces

Standard samples for abrasive wear testing (17 mm × 4 mm × 4 mm) were cut from the hot pressed discs by a diamond saw and attached to resin cylinders using adhesive to enable

gripping by the testing machine. A grinding/polishing machine (MOTOPOL2000, Buehler, UK) was used to perform abrasive wear tests under standard conditions as has been described previously.⁸ Three samples of the same material were loaded in the specimen carousel. The samples were lapped on a 305 mm diameter resin-bonded alumina wheel (Kemet, UK) with an oil-based suspension of 45 μm diamond abrasive automatically sprayed every 20 s. The abrasive wear test was carried out for 4 min with a grinding wheel rotation speed of 350 rpm and a force of 22.2 N per sample. The weight before and after the test was measured and the weight loss (Δm) was calculated. The wear rate (v) was defined as:

$$v = \frac{\Delta m}{tA\rho} \quad (1)$$

where A is the contact area, t the testing time and ρ the density of the material. Thus, v is the speed with which the wearing surface is receding, quoted in $\mu\text{m s}^{-1}$. The first wear test result was discarded for bedding-in and at least three subsequent wear tests were carried out for each material.

At least three micrographs of worn surfaces were taken by SEM for quantitative analysis. The area fraction of pullout caused by brittle fracture was measured by linear intercepts with lines in a grid on SEM micrographs. The pullout size was measured as the mean linear intercept multiplied by a factor of 1.27 to obtain an equivalent circular diameter.⁸ The pullout diameter was then corrected for overlap using the method developed in Ref. [8].

2.3. Indentation-polish test

Polished surfaces of alumina and nanocomposites were indented with a load of 10 kg (98 N) using a Vickers indenter. All specimens chosen for this test had an alumina grain size between 4.9 and 5.6 μm. The indented surfaces were subsequently polished using 1 μm diamond paste on a soft cloth until only the lower tip of the original indentation remained. Micrographs were taken by SEM to observe the details of surface pullout during polishing caused by prior microcracking beneath the indentations as has been described previously.¹¹

3. Results

3.1. Worn surfaces of alumina and nanocomposites

Fig. 1 shows typical worn surfaces of (a) alumina, (b) a 5 vol.% SiC nanocomposite, (c) a 10 vol.% SiC nanocomposite and (d) the 10 vol.% SiC microcomposite. The results are in accord with those reported previously.^{3,8} It can be seen that a large fraction of the worn surface of alumina has been removed by “pullout”, where a discrete piece of material is removed by brittle fracture. The nanocomposites display smoother worn surfaces with much less pullout. Intergranular fracture was responsible for pullout in alumina but pullout fracture in the nanocomposites was transgranular. The microcomposite surface is intermediate in appearance between the alumina and the nanocomposites. Most of the pullouts in this case were pro-

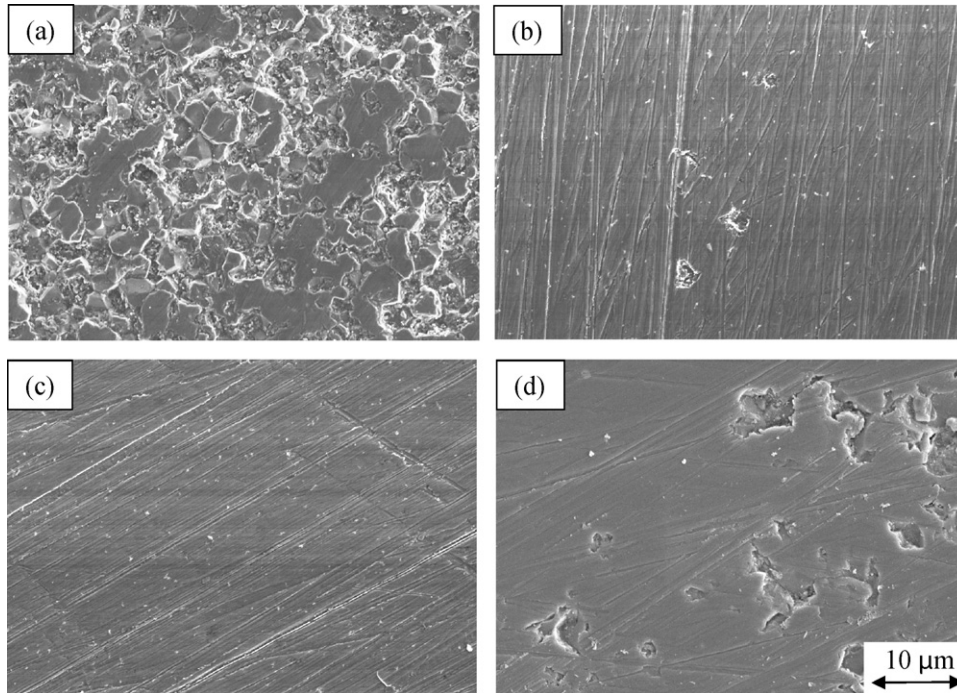


Fig. 1. Micrographs at the same magnification of worn surfaces of (a) alumina ($G = 5.6 \mu\text{m}$), (b) 5 vol.% ($G = 5.3 \mu\text{m}$), (c) 10 vol.% ($G = 4.9 \mu\text{m}$) SiC nanocomposites and (d) 10 vol.% ($G = 5.7 \mu\text{m}$) SiC microcomposite.

duced by transgranular fracture although a few larger pullouts with intergranular fracture were also found.

3.2. Relationship between microstructure and wear properties.

3.2.1. Relationship between wear rate and grain size

The wear rate of alumina depended strongly on the alumina matrix grain size (Fig. 2), increasing by more than a factor of 4 as the grain size increased from 2.6 to 5.6 μm . In the nanocomposites, the wear rate was not dependent on alumina grain size. The wear rate of all nanocomposites was

in the range between 0.25 and 0.50 $\mu\text{m s}^{-1}$. The wear rate of the microcomposite was only slightly greater than those of the nanocomposites.

3.2.2. Relationship between area fraction of pullout and grain size

The area fraction of pullout in alumina also increased strongly with grain size (Fig. 3). In contrast, the area fraction of pullout of the nanocomposites was approximately 2%, independent of grain size. This is much lower than for monolithic alumina of a similar grain size in all cases. The microcomposite result lay between those of alumina with similar grain size and the nanocomposites.

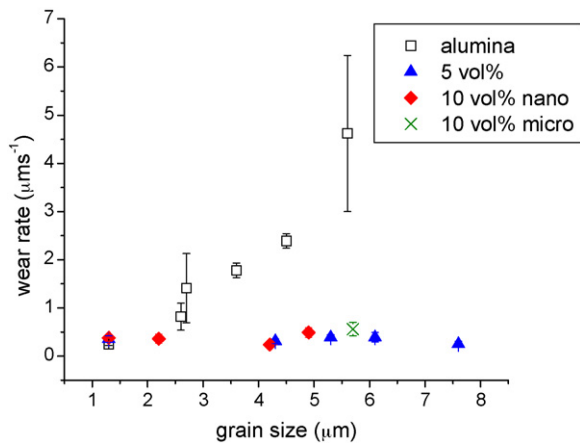


Fig. 2. Plot of wear rate against grain size for alumina and composites, showing strong grain size dependence in alumina and weak grain size dependence in the nanocomposites.

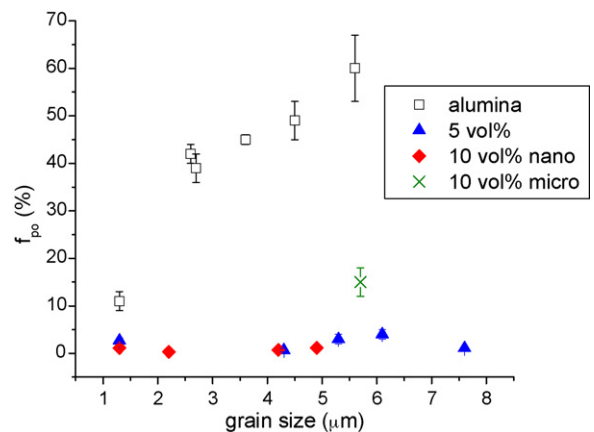


Fig. 3. Plot of area fraction of pullout on worn surfaces against grain size for alumina and composites.

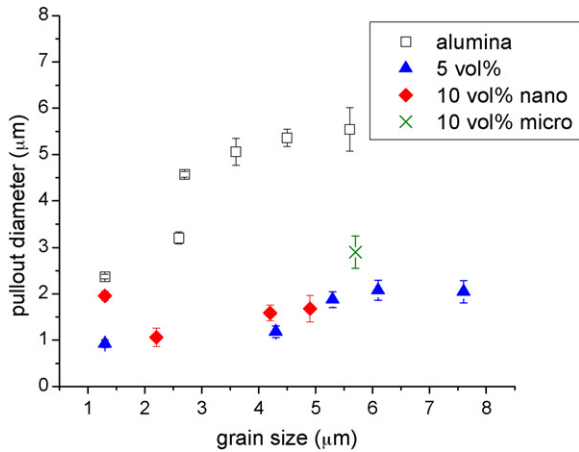


Fig. 4. Mean pullout diameter as a function of grain size for alumina and composites.

3.2.3. Relationship between mean pullout diameter and grain size

The mean pullout diameter of alumina was again strongly dependent on the grain size (Fig. 4). All alumina samples had

mean pullout diameters slightly larger than their grain sizes. The pullout diameters for the nanocomposites were much less sensitive to the alumina grain size and were smaller than the grain size in all cases except for the specimen with the smallest grain size. The pullouts in the microcomposite were only slightly larger than those in the nanocomposites with similar grain size.

3.3. Indentation-polish test

Fig. 5 shows SEM pictures of the indented surfaces following subsequent polishing with 1 μm diamond paste. In each case, the remaining part of the original indentation can be seen in the centre of the picture, and the traces of the classical radial cracks can also be seen outside the plastic zone. The dimensions of the original indentations, and therefore the hardnesses, were similar for all materials. In the case of pure alumina it is evident that whilst most of the surface has been well polished by the 1 μm diamond paste, a lot of pullout by brittle fracture has occurred in a well-defined region corresponding closely to the shape and size of the indentation before surface removal by subsequent polishing. This is assumed to be the plastic zone beneath the indentation. The 5% SiC nanocomposite shows a marked

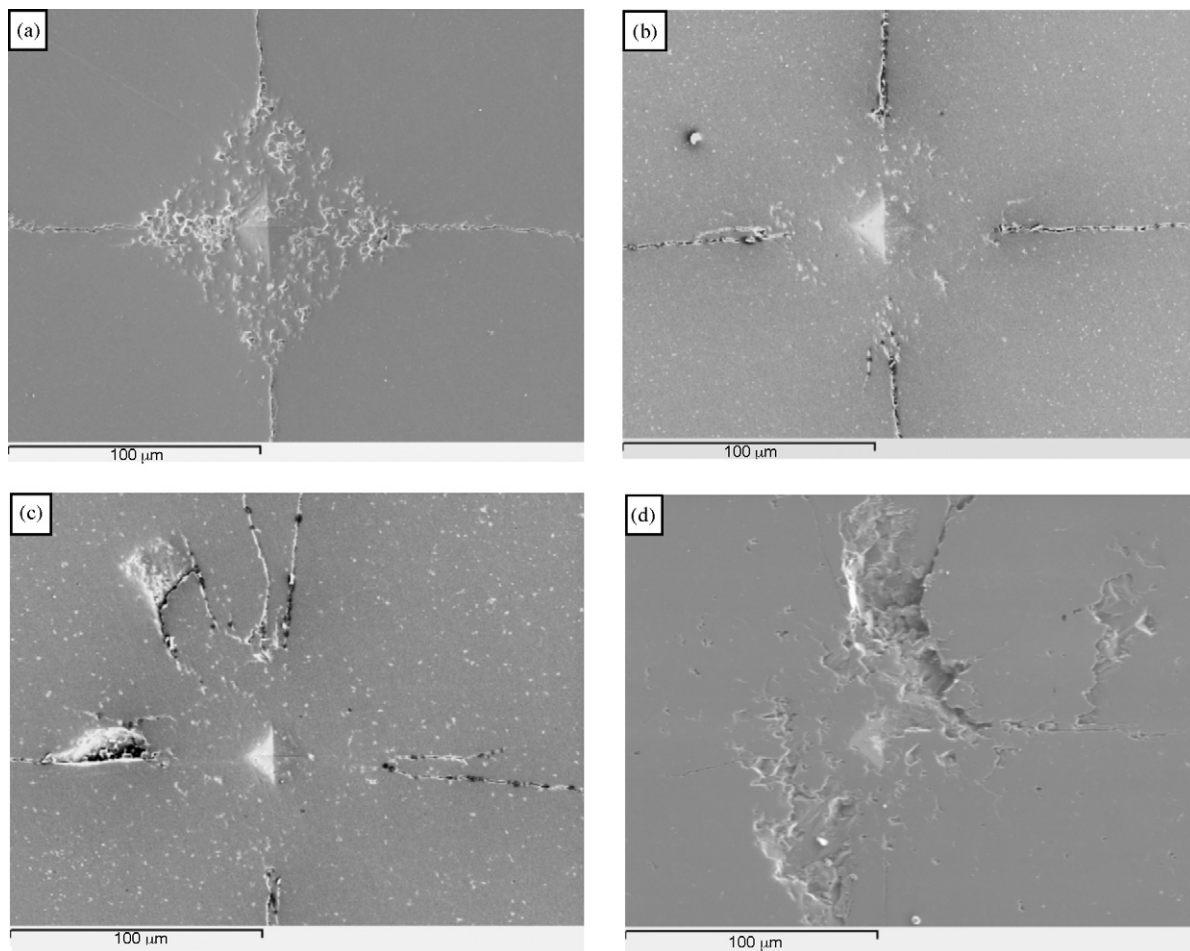


Fig. 5. Surfaces of (a) alumina ($G=5.6 \mu\text{m}$), (b) 5 vol.% SiC nanocomposite ($G=5.3 \mu\text{m}$), (c) 10 vol.% SiC nanocomposite ($G=4.9 \mu\text{m}$) and (d) 10 vol.% SiC microcomposite ($G=5.7 \mu\text{m}$) following 10 kg Vickers hardness indentation and subsequent 1 μm diamond polishing to remove most of the indentation. The figures show extensive cracking in the plastic zones of the alumina and “microcomposite” specimens, but very little plasticity induced damage in the nanocomposites. The shallow lateral cracking in the top left quadrant of the 10% SiC nanocomposite was an occasional feature observed in all materials.

reduction in pullout within this plastic zone whilst pullout is almost completely suppressed in the 10% SiC nanocomposite. The microcomposite shows extensive pullout within the plastic zone and substantial fracture in the elastically deformed area outside it.

4. Discussion

4.1. Influence of fracture mode on wear

Conventional models for abrasive wear, such as that by Evans¹² assume that the paths of the near-surface lateral cracks responsible for pullouts in abrasive wear are determined solely by fracture in response to the stress field around the scratches made in the surface by the abrasive particles. The predicted dimensions of the pieces of ceramic removed by such cracking (“pullouts” in this work), and hence the wear rate, depend on the Young’s modulus, hardness and fracture toughness of the ceramic, but not on microstructural details such as grain size. This should be the case for ceramics with transgranular fracture such as the nanocomposites in this work, and is the basis of Ortiz Merino and Todd’s prediction⁸ that the pullout diameter, and hence wear rate and surface finish, should be independent of grain size for these materials. The results for the nanocomposites in Figs. 2–4 fully bear out this prediction and Fig. 4 shows that typical pullout diameters produced by the abrasive particles in our wear tests are in the range 1–2 μm .

Since the Young’s modulus, hardness and fracture toughness of pure alumina are similar to those of the nanocomposites,² the Evans model predicts similar wear properties. In reality, however, the intergranular fracture mode of alumina means that the paths of lateral cracks are determined by the geometry of the grain boundary network as well as by the stresses around abrasive scratches. If the alumina grains are bigger than the predicted pullout size the consequence will be significantly larger pullouts,⁸ whose dimensions will increase with the grain size, which leads in turn to increased surface pullout and wear, as observed in our results. For grain sizes smaller than the Evans model’s predicted pullout diameter, little influence of alumina grain size on wear would be expected (in the absence of other effects) and it is evident in Figs. 2–4 that the wear rate and surface finish of the alumina and nanocomposites converge at a grain size of 1–2 μm , similar to the pullout diameter in the nanocomposites, in accordance with this idea.

The 10% 3 μm SiC microcomposite showed mainly transgranular fracture within the pullouts (Fig. 5d), but with some intergranular fracture also present. The wear resistance and surface finish lay between those of the nanocomposites and alumina, as would correspondingly be expected. Conventional fracture surfaces were also predominantly transgranular for this microcomposite (Fig. 6) suggesting that mechanisms put forward for the change in fracture mode that rely on local interactions with nanoparticles are an oversimplification. Segregation of impurities to grain boundaries, as has been found to occur in several studies,^{13–15} may also play a role.

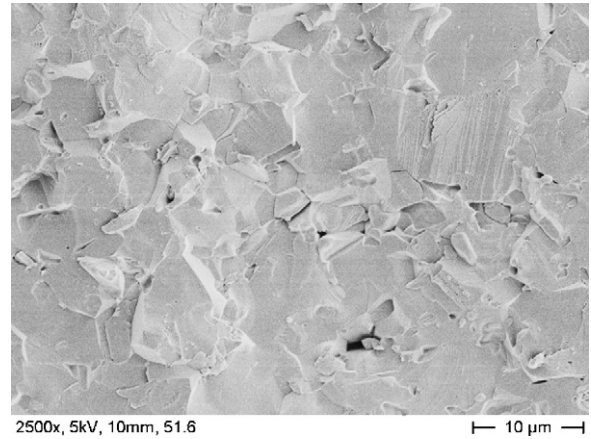


Fig. 6. Conventional fracture surface of the 10 vol.% SiC microcomposite showing predominantly transgranular fracture mode.

We conclude that the present results fully support the suggestion⁸ that the change in fracture mode on adding SiC to alumina makes a major contribution to the resulting improvement in wear resistance because of the simple geometrical consequence that each surface pullout through brittle fracture will then be smaller in size.

4.2. Primary suppression of subsurface cracking by SiC nanoparticles

Although the pullout dimensions are important in controlling the wear properties of ceramics, other factors are also influential, most obviously the rate at which such pullouts are produced. For the conditions in our wear test, in which the material removal rate is such that steady state abrasive wear is quickly established and no significant tribolayer accumulates, it can be shown on a phenomenological basis that⁸:

$$\frac{f_{po}}{(1 - f_{po})(1 - z/2)} = \frac{nAd_0}{v} \quad (2)$$

where f_{po} is the area fraction of pullout at steady state, v is the wear rate, A is the mean area of the individual pullouts (assumed prismatic in shape for simplicity), d_0 is the mean depth of the pullouts when created (note that, once formed, a pullout becomes shallower as the sample surface around it wears away), n is the rate at which pullouts are produced by brittle fracture per unit load bearing area per unit time, and z is a function of f_{po} only, given in Ref. [8]. For small amounts of pullout, Eq. (2) reduces to:

$$f_{po} \approx \frac{nAd_0}{v} \quad (3)$$

Experimentally, d_0 is proportional to the pullout diameter,⁸ and since all other quantities apart from n have been measured in our experiments, n can be deduced from Eq. (2) for each material. The results are shown as a log–log plot of n against $1/(1 - f_{po})$ in Fig. 7, the significance of $1/(1 - f_{po})$ being that it is proportional to the mean stress applied to the load bearing surface between the pullouts. In alumina, the pullout rate increases steadily

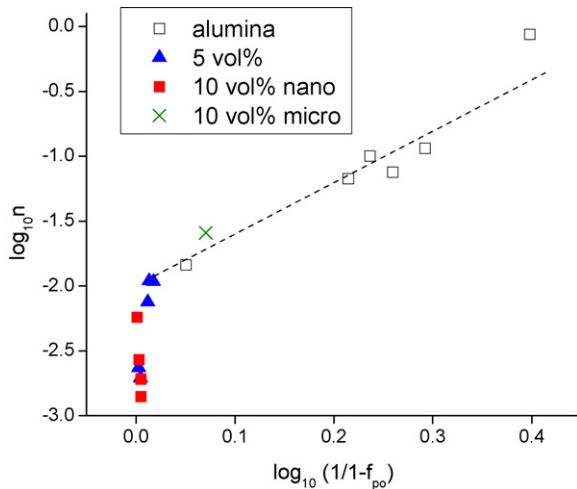


Fig. 7. Logarithmic plot of pullout formation rate, n ($\mu\text{m}^{-2}\text{s}^{-1}$) against $1/(1 - f_{po})$ for alumina and composites. The dotted line is the best fit straight line to the alumina points. The nanocomposite points tend to lie below this line, indicating the suppression of the cracking responsible for pullout.

with f_{po} , presumably because of the accompanying increase in pressure on the load bearing area and because the specimens with more pullout correspond to those with bigger grain sizes. The addition of nanosized SiC particles, and especially with the higher content of 10 vol.%, suppresses pullout formation severely, by a factor of between 2 and 10 compared with the finest grained alumina used here. The effect of the nano-SiC cannot be attributed to differences in pressure on the load bearing area or grain size. The 3 μm SiC microcomposite did not exhibit the same effect; its behaviour was similar to that of alumina in this respect.

Eq. (3) shows that the area fraction of pullout at the surface, f_{po} , is approximately proportional to n , and, via A and d_0 , to the cube of the pullout diameter. Detailed comparison of the pullout diameters for the nanocomposites and aluminas in Fig. 4 shows that despite the effectiveness of the SiC additions in suppressing crack initiation, the simultaneous reduction in pullout diameter remains the dominant contribution to the improved wear properties for most grain sizes.

The above evidence of the ability of the SiC nanoparticles to suppress brittle fracture is indirect but the more direct results of the indentation-polish test confirm it. The confinement of the pullouts produced in alumina during 1 μm diamond polishing to the plastic zone of the indentation provides clear evidence that plastic deformation is responsible for the initiation of the cracks that lead to pullout. Its virtual absence in the indentation plastic zones of the nanocomposites confirms the powerful ability of the nanosized SiC additions to suppress crack initiation.

It is interesting that the investigation of damage beneath single scratches of Wu et al.¹⁶ by sectioning using focused ion beams (FIB) did not reveal any cracking within the plastic zone in alumina. This indicates that the incipient cracks resulting from plastic deformation were too small to be detected at the magnifications used or were obscured by redeposited material during milling. In the current work, mechanical polishing was used to section the materials instead of FIB. Though sufficiently gentle

to avoid crack nucleation in undamaged areas, this technique was evidently sufficiently aggressive to propagate the incipient cracks in the plastic zone thus forming pullouts that were more easy to detect in the SEM.

As with the reduction in n , Fig. 5d shows that the addition of larger, 3 μm SiC particles does not have the same effect. Indeed, the extensive cracking in the elastic region around the indentation shows that the larger SiC particles can actually aid cracking, probably because of the increased effect on fracture of the considerable thermal microstresses present when the particle size is larger.¹⁷

The results are consistent with the suggestion of Ortiz Merino and Todd⁸ that cracking is suppressed because intragranular SiC nanoparticles block the formation of long twins and/or dislocation pileups during plastic deformation (Fig. 8)

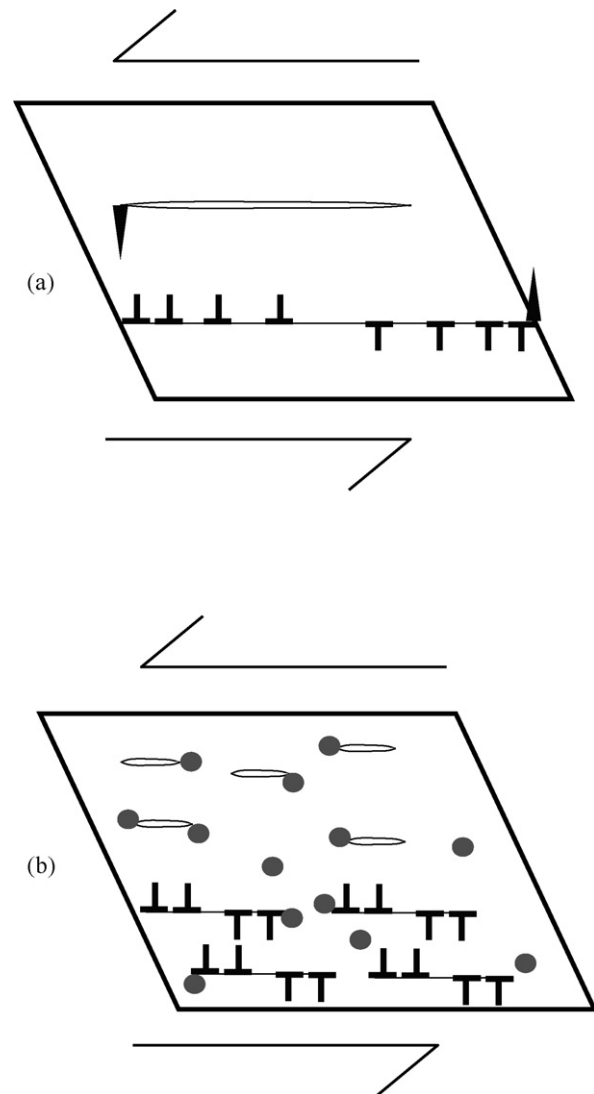


Fig. 8. Schematic illustration of suppression of crack initiation by intragranular SiC particles. (a) In alumina, stress concentrations at the heads of long twins or dislocation pileups cause crack initiation. (b) In the nanocomposite, the SiC particles block the development of twins and pileups, reducing their length and therefore the stress concentration so that cracking is suppressed.

in a mechanism similar to that of slip homogenisation in metals. The stress concentrations at the heads of twins and pileups have been shown to be capable of initiating cracks in alumina,^{18,19} and increase with the length of the twin or pileup. The introduction of obstacles such as SiC particles on the slip or twinning plane limits the length of such stress concentrators and, although more of them are then required to achieve a given plastic strain, the stress concentration associated with each one is reduced. If enough nanosized SiC is added (>5 vol.% for our 260 nm SiC powder), the twin or pileup length becomes sufficiently small to suppress crack nucleation. This suggestion derives directly from the TEM examination of deformation structures beneath scratches and indentations by Wu et al.^{16,20} which demonstrates convincingly that long basal twins observed in alumina are absent or reduced in penetration in the nanocomposites.

The planar spacing, s , of equiaxed particles of diameter d is given by:

$$s \approx d \sqrt{\frac{1}{f}} \quad (4)$$

Our results show that with $d \approx 260$ nm microcracking begins to be suppressed for volume fractions of approximately 0.05 and above, giving $s = 1.2 \mu\text{m}$, i.e. at the point where the particle spacing starts to become significantly smaller than the grain sizes of the specimens used here. Larger SiC particles, such as those in our $3 \mu\text{m}$ SiC microcomposite are widely spaced, with $s = 9.5 \mu\text{m}$ for the 10 vol.% microcomposite used here, and are mostly on the alumina grain boundaries and therefore are not expected to have this effect, in agreement with the experimental results. Conversely, there is evidence that smaller SiC particles than those used here can suppress cracking with lower volume fractions,²¹ presumably because of their smaller separation according to Eq. (4).

Although the present results focus specifically on damage during severe abrasive wear, it can be recognised that the general conclusions relating to the suppression of surface crack initiation are in accord with other properties of alumina/SiC nanocomposites reported in the literature, such as a lower density of surface flaws in polished surfaces²² and the consistently improved strength relative to alumina.

5. Summary

1. The grain size dependence of the wear rate and surface finish in abrasive wear has been investigated in alumina and alumina/SiC nanocomposites containing 5 and 10 vol.% SiC. The wear properties of the nanocomposites were almost completely independent of the alumina grain size, whereas the wear properties of the alumina deteriorated rapidly with increasing grain size. The properties appeared to converge at an alumina grain size approximately equal to the mean diameter of surface pullouts by brittle fracture occurring during the wear test. The results confirm that much of the improvement in wear properties of the nanocomposites comes from a reduction in the mean size of the individual pullouts as a geo-

metrical consequence of the change in fracture mode found on adding SiC to alumina.

2. Quantitative analysis of the wear test results showed that the addition of high volume fractions of SiC nanoparticles to alumina also reduces wear by directly preventing the brittle fracture responsible for surface pullout. Examination of damage beneath Vickers hardness indentations confirmed this and showed that cracking and pullout in alumina is nucleated by plastic deformation. The results support the idea that SiC nanoparticles within the alumina grains block the development of twins and dislocation pileups in the alumina and thus limit their length and associated stress concentration to below those required for crack nucleation.

Acknowledgement

The authors are grateful to Dr. Jinyong Zhang and Professor Zheng Yi Fu of Wuhan University of Technology for providing the fine grained alumina specimen made by SPS.

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