Journal of the European Ceramic Society 16 (1996) 799-802
0955-2219(95)00198-0

0955-2219/96/$15.00

Effects of Silicon Carbide Nano-phase on the Wet Erosive Wear of Polycrystalline Alumina

R. W. Davidge, P. C. Twigg & F. L. Riley*

School of Materials, University of Leeds, Leeds LS2 9JT, UK

(Received 12 October 1995; revised version received 30 October 1995; accepted 30 October 1995)

Abstract

Wet erosive wear rates for alumina–silicon carbide nanocomposites have been measured and compared with those for pure polycrystalline aluminas of similar grain size. In pure materials for mean grain sizes >2 μm the dominant wear mechanism appears to be grain-boundary microfracture, leading to grain pull-out; for finer grain sizes the worn surfaces are smooth. Within both grain size ranges significant reductions in wear rate are found for the nanocomposite materials, thus extending the span of established benefits obtained through the incorporation of silicon carbide nanoparticles into ceramic materials.

Introduction

The wear behaviour of hard ceramic materials such as alumina is a subject of considerable industrial importance, even more so when 'wear' is taken to include the material removal processes of cutting and grinding. The wear of alumina is known to be strongly dependent on the microstructure, and on the mean grain size (G) in particular. For a wide range of wear and abrasion processes the wear rate decreases as G decreases.[1-10] For pure alumina with G in the range 2 to 50 μm, the predominant wear mechanism is grain-boundary fracture. For very fine-grained materials (<1 μm) plastic deformation or polishing seems to be the preferred wear mechanism and it is likely that in these cases an underlying slow tribochemical wear mechanism may become the dominant process.

A tentative model for the wear of polycrystalline ceramics of grain sizes in the range 1 to 12 μm has been proposed,[11] based on microcrack propagation. This model assumes the preferred passage of cracks along two-grain boundaries, with delays in crack propagation occurring at multiple boundary junctions for readjustment of the direction of crack propagation. This 'stop–go' crack propagation process is a product of the fluctuating stress field experienced under bombardment by multiple erodent particles.

The time (t) for the crack to progress a specified distance (d) is given by:

$$t = 2(t_f + t_j)\, d \,/\, G \qquad (1)$$

where t_f (proportional to the mean grain dimension, G) is the time to traverse a two-grain boundary face of dimension ~$G/2$ at a mean rate characteristic of the material, and t_j (a constant) is the time taken to realign at a three-grain junction. The experimentally observed wear rate (W) given by:

$$W = Ad \,/\, t \qquad (2)$$

thus becomes

$$W = AG \,/\, 2\,(t_f + t_j) \qquad (3)$$

where A is a constant related to the microstructural crack linking process and the experimental conditions of the test. t_f can be eliminated by defining a characteristic grain size (G_o) for which $t_f = t_j$. Thus:

$$t_f = (G/G_o)\, t_j \qquad (4)$$

and substitution into eqn (3) gives:

$$W = A\, G\, G_o \,/\, [2\, t_j\, (G + G_o)] \qquad (5)$$

There should therefore be a proportionality between W and $G \,/\, (G + G_o)$ with the plot passing through the origin, a prediction supported by a wide range of wear data for which the value of G_o ranges from 1 to 100 μm, depending on the nature of the process.[11] The G_o term is expected to vary with the nature of the wear process and conditions, which affect in particular the scale of the damage and the way that individual cracks propagate and link. This wear model specifically focuses on grain size effects and ignores possible contributions from other material removal processes less likely to be grain-size dependent, such as plastic-deformation-dependent and tribochemical wear. The model is therefore considered to be applicable to ceramics with grain sizes above a lower limit of ~1 μm;

---

*To whom correspondence should be addressed.

microfracture is not observed in ceramics of grain size <1 μm. Porosity has a detrimental effect on wear properties, but for levels ≤2% the effects would be expected to be small.[12]

The incorporation of nanoparticles (<1 μm) of silicon carbide (SiC) into polycrystalline alumina materials is known to confer marked improvements in their mechanical properties (for example, increased strength, toughness and creep resistance).[13–15] This paper reports the results of a preliminary investigation into whether these advantages extend to the wear properties of hot-pressed aluminas containing varying proportions of three grades of nanoparticle SiC powder. The study was based on a simple system of wet erosion, using a slurry of alumina grit in water. Direct comparisons of the wear rates for these materials with those for alumina, silicon nitride and zircon materials are possible because the experimental technique for wearing the materials was identical to that used in previous investigations.[10,16,17]

Experimental

Alumina (Sumitomo AKP53) and silicon carbide (Lonza α-SiC, UF45, UF32 and UF15 at levels of 5 and 10 wt%) powders were used. The mean particle size (d) for the alumina was 90 nm and the maximum (d_{max}) 320 nm; d for SiC ranged from 90 nm (UF45) to 110 (UF15) and d_{max} from 320 nm (UF45) to 500 nm (UF15). The powders were blended by attrition milling in water with Dispex A40 dispersant (Allied Colloids, Bradford, UK), and then freeze-dried. One sample of freeze-dried alumina without SiC additions (Al_2O_3-I) was used as a control material; a second sample of alumina was also prepared using a spray-drying alumina powder (Al_2O_3-II). Discs of material 50 mm in diameter and ~6 mm thick were hot-pressed in a graphite die at temperatures in the range 1400 to 1700°C and under pressures of 25 MPa. Selected hot-pressed discs were given subsequent annealing treatments under a non-oxidizing atmosphere at <1900°C to cause grain growth. This process was carried out under an argon gas flow (100 cm^{-3} min^{-1}) and the discs were further embedded in fine SiC powder as an oxygen getter to prevent oxidation of the nanoparticle SiC. Densities were measured by the water immersion method using BS7134. Discs of 25 mm diameter were core drilled from the larger discs for the wear rate measurements, and surfaces were ground using a steel-bonded 75 μm diamond wheel. Worn disc surfaces were gold-coated and examined by scanning electron microscopy (SEM); other surfaces were ground and polished to 1 μm diamond, embedded in SiC powder and thermally etched at 1400°C for 40 min under flowing argon, gold-coated and photographed by SEM for grain size measurements.

Wet erosion was carried out in a modified high-torque attritor mill using 0·5 to 1 mm dimension crushed fused alumina aggregate in water.[10,16] The alumina discs (25 mm diameter and ~5 mm thick) were clamped between shaped discs of hard polyurethane attached to the shaft of the mill, with ~50% of the disc exposed. The sample holder was immersed in an externally water-cooled slurry consisting of 700 g of alumina grit (of stated purity 92·2% Al_2O_3, with SiO_2 and TiO_2 as the major impurities) in 250 cm^3 of deionized water and rotated at a speed of 8 Hz with a disc track radius of 36 mm (giving a disc perimeter linear velocity of ~1.9 m s^{-1}). After wearing for 2 h (t_1) and 6 h (t_2) discs were ultrasonically cleaned in deionized water, dried for 20 min at 110°C, and weighed to ±0·1 mg (w_1 and w_2). A wear rate (R) measured in m s^{-1} was calculated on the basis of the expression:

$$R = [(w_1 - w_2) / (t_2 - t_1) / (Ap)$$

where A, arbitrarily, is the area of the exposed leading quadrant of the 6 mm thick disc rim (approximately 1/4 of the total rim area): experience showed that with this wear configuration very little wear took place on the disc faces and the face area (although much larger) was therefore ignored. This time period was chosen in order to minimize any effects of initial surface finish at short times and the significant smoothing of the grit particles at longer times. The slurry was replaced by a fresh batch after use for two tests (total time 12 h). Worn and fracture surfaces of the discs were gold-coated and examined by SEM.

Mean alumina grain size (G) was measured using a standard line intercept technique from SEM photographs. At least 100 grains were measured for each material, and G obtained by multiplication of the mean intercept length by the factor 1·56.[18]

Results

The alumina and alumina–silicon carbide nanocomposite materials were produced by hot-pressing and, for one composition, subsequent annealing, alumina and SiC powders using the conditions set out in Table 1, which also summarizes material characteristics and wear rates (the means of two determinations).

The microstructure of a typical nanocomposite is illustrated by Fig. 1; the uniform distribution of the nanoparticles of SiC (darker phase) at the grain boundaries and within the alumina grains is clear.

Wear rate data for the coarser-grained nanocomposite materials, compared with those for a series of pure aluminas,[10] are shown in Fig. 2. For

Table 1. Materials' fabrication conditions and properties

Material	*Hot-pressing temperature (°C)*	*Density* ($g\ cm^{-3}$)	*Density %Theoretical*	G (μm)	R ($nm\ s^{-1}$)
1 Al_2O_3-I	1450	3·95	99·3	1·3	11·8
2 Al_2O_3-II	1400	3·93	98·7	0·7	2·2
3 +5% UF45	1550	3·87	98·2	0·5	1·2
4 +5% UF45	1700	3·92	99·5	2·0	14·2
5 +5% UF45	as 3 + 1900 anneal	3·80	95·4	5·0	16·1
6 +5% UF32	1550	3·88	98·5	1·0	1·8
7 +5% UF15	1550	3·89	98·8	1·1	4·6
8 +10% UF45	1550	3·78	97·0	0·8	1·4
9 +20% UF45	1550	3·48	91·2	0·4	5·9

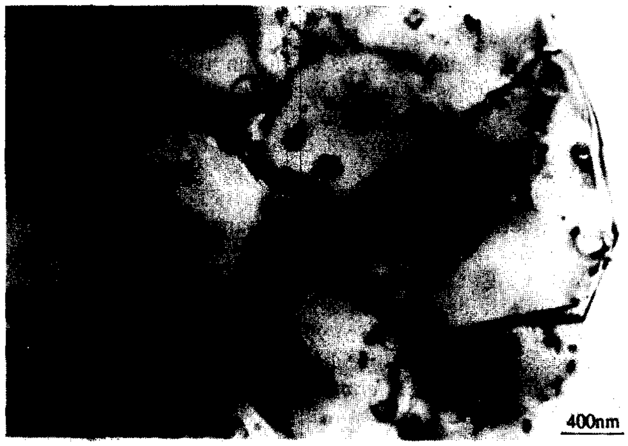

Fig. 1. Microstructure of a typical nanocomposite.

greater clarity, data for finer-grained ($G \leq 2\ \mu m$) materials, for which wear is by mechanisms other than microfracture, are shown separately in Fig. 3. Examination of the worn surfaces of the pure aluminas of grain size $\geq 2\ \mu m$ predominantly showed grain-boundary fracture; all the nanocomposites, including the 5 μm grain size material, although showing some indication of grain detachment, showed a predominantly smooth wear surface similar to that of a very fine-grained pure alumina and indicative of plastic deformation or tribochemical wear. Figure 4 is that of a worn surface of a 2·5 μm pure alumina;[10] Fig. 5 shows a typical surface of a worn 5 μm nanocomposite material.

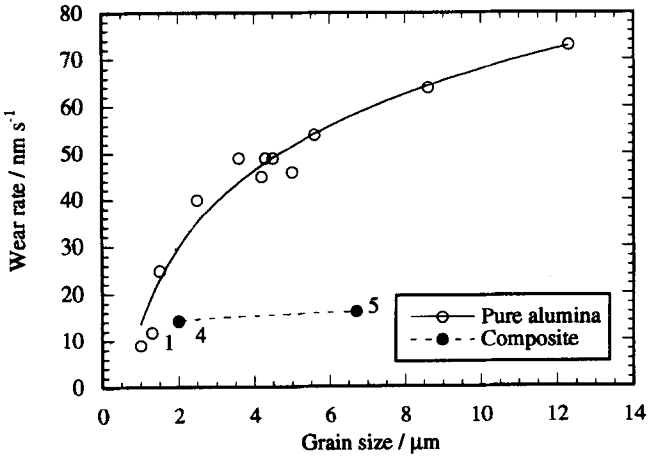

Fig. 2. Wear rate data for the coarser grain nanocomposite materials, compared with those for pure alumina.[10]

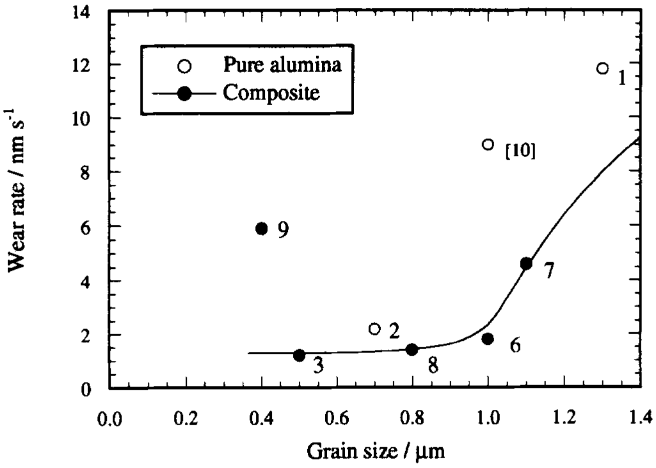

Fig. 3. Wear rate data for the finer grain nanocomposite materials, and fine grain size pure alumina.

Fig. 4. Typical surface of a worn 2·5 μm grain size pure alumina.

Fig. 5. Typical surface of a worn 5 μm grain size nanocomposite material.

Discussion

There is a clear improvement in the wear behaviour of the nanocomposites in comparison with that of the pure aluminas of equivalent grain size. For nanocomposites of $G \geq 2\ \mu m$ the wear rates were reduced by a factor of 2 to 3. The data for the finer-grained materials are less conclusive, but the wear rate appears to be reduced by a factor of 1·5 to 2. The deleterious effect of a high porosity level (9%) is indicated by the poor wear resistance of sample 9 prepared with the addition of 20% of SiC powder, and therefore less easy to densify fully.

One of the best established effects concerning Al_2O_3/SiC composite materials is the 'strengthening' of the alumina grain boundaries by the nanophase. This is manifested, for example, by relatively smooth transgranular fracture paths in the nanocomposites, compared with those of pure alumina, and the smoother surfaces during polishing (indicative of a reduced degree of grain pull-out).[15] These effects of the SiC nanoparticles on wear rate are also associated with a change in the nature of the wear process, from that typical of a large grain material (where there is relatively easy material removal by grain-boundary fracture) to that typical of a smaller-scale material removal process with mechanisms involving much slower localized plastic deformation or atomic removal of material. The nanocomposites thus have much better wear resistance than that predicted by the microfracture model, because of inhibition of the grain-boundary fracture process by the SiC particles. However, even when the worn surfaces of the small-grained pure alumina and the nanocomposites are similar, the wear rate of the nanocomposite is much slower. It is known that dislocation substructures form in nanocomposites[13] and these may reduce the efficiency of processes involving plastic deformation.

Conclusions

The incorporation of nanoparticle SiC into dense polycrystalline alumina leads to a significant reduction in wear rate and parallels the action of this type of SiC in improving other mechanical properties of ceramic materials. Although grain size is the single most important parameter in determining the wear rate of pure polycrystalline aluminas, the presence of nano-phase SiC has (at equivalent grain sizes) a significant additional beneficial effect. The presence of the fine SiC particles in the sintering alumina powder also inhibits grain growth so that, for given densification conditions, the nanocomposites tend to possess desirable fine grain sizes.

Acknowledgements

The support of the EPSRC through research grant GR/J86513 is acknowledged. The nanocomposites were prepared as part of BRITE-EURAM Project BRE CT920358, and the Partners are thanked for provision of materials.

References

1. Evans, A. G. & Wilshire, T. R., Quasi static solid particle damage in brittle solids—I: Observations, analysis and implications. *Acta. Metall.*, **24** (1976) 939–56.
2. Cho, S. J., Hockley, B. J., Lawn, B. R. & Bennison, J., Grain-size and R-curve effects in the abrasive wear of alumina. *J. Am. Ceram. Soc.*, **72**(7) (1989) 1249–52.
3. Rice, R. W. & Speronello, B. K., Effect of microstructure on rate of machining of ceramics. *J. Am. Ceram. Soc.*, **59**(7–8) (1976) 330–3.
4. Wiederhorn, S. M. & Hockley, B. J., Effect of material parameters on the erosion resistance of brittle materials. *J. Mater. Sci.*, **18** (1983) 766–89.
5. Wu, C. Cm., Rice, R. W., Johnson, D. & Platt, B. A., Grain size dependence of wear in ceramics. *Ceram. Eng. Sci. Proc.*, **6**(7–8) (1985) 995–1011.
6. Rice, R. W., Micromechanics of microstructural aspects of ceramic wear. *Ceram. Eng. Sci. Proc.*, **6**(7–8) (1985) 940–58.
7. Marshall, D. B., Lawn, B. R. & Cook, R. F., Microstructural effects on grinding of alumina and glass ceramics. *J. Am. Ceram. Soc.*, **70**(6) (1987) C139–40.
8. Gee, M. G. & Almond, E. A., The effects of surface finish on the sliding wear of alumina. *J. Mater. Sci.*, **25**(1A) (1990) 296–310.
9. Cho, S. J., Moon, H., Hockley, B. J. & Hsu, S. M., Wear transformation phenomenon in alumina during sliding. In *C-MRS Int. Symp. Meeting 1990*, Vol. 5, ed. B. Wu. North-Holland, Amsterdam, 1991, pp. 387–9.
10. Miranda-Martinez, M., Davidge, R. W. & Riley, F. L., Grain size effects on the wet erosive wear of high purity polycrystalline alumina. *Wear*, **172** (1994) 41–8.
11. Davidge, R. W. & Riley, F. L., Grain size dependence of the wear of alumina. *Wear*, **186–187** (1995) 45–9.
12. Miranda-Martinez, M., Davidge, R. W. & Riley, F. L., The reduction of erosive wear rates of advanced technical ceramics. In *Ceramics in Energy Applications*, Proc. Institute of Energy's 2nd Int. Conf., London, 20–21 April 1994. The Institute of Energy, London, 1994, pp. 239–52.
13. Niihara, K., New design concept of structural ceramics—ceramic nanocomposites. *Ceram. Soc. Jap.*, **99**(10) (1991) 974–82 (Centennial Memorial Issue).
14. Zhao, J., Stearns, L. C., Harmer, M. P., Chan, H. M., Muller, G.A. & Cook, R. E., Mechanical behaviour of alumina–silicon carbide 'nanocomposites'. *J. Am. Ceram. Soc.*, **76** (1993) 503–10.
15. Walker, C. N., Borsa, C. E., Todd, R. I., Davidge, R. W. & Brook, R. J., Fabrication, characterization and properties of alumina matrix nanocomposites. *Br. Ceram. Proc.*, **53** (1995) 249–64.
16. Miranda-Martinez, M. & Riley, F. L., Wet abrasive wear of technical ceramics. *Br. Ceram. Trans. J.*, **90** (1991) 118–21.
17. Wootton, A., Miranda-Martinez, M., Davidge, R. W. & Riley, F. L., The wet erosive wear behaviour of fine-grain zircon ceramic. *J. Eur. Ceram. Soc.*, in press.
18. Mendelson, M. I., Average grain size in polycrystalline ceramics. *J. Am. Ceram. Soc.*, **52**(8) (1969) 332–46.