Damage and wear resistance of Al₄O₃–SiC microcomposites with hard and elastic properties

Hawsawi ELYAS¹, Tae Woo KIM¹, Byung-Koog JANG²,³ and Kee Sung LEE¹,††

¹School of Mechanical Engineering, Kookmin University, Seoul 02707, Korea
²National Institute of Materials Science, 1–2–1 Sengen, Tsukuba, Ibaraki 305–0047, Japan

Al₂O₃–SiC composites with different SiC contents of 0–10 wt% were fabricated and the effects on damage and wear resistance investigated. The composites were fabricated using spark plasma sintering at 1600°C in a partial vacuum (80 MPa). Hertzian indentation evaluations using a spherical indenter indicated that the hardness of the composite is improved by SiC addition. Wear resistance evaluated using the ball-on-disk method showed enhanced wear resistance for the composites, even when SiC addition was less than 2 wt%. Thus, Al₂O₃ ceramics exhibited significantly improved damage and wear resistance, even though SiC addition was as small as 2 wt%.

Key-words: Alumina, Damage, Wear, Hertzian indentation

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1. Introduction

Composites based on Al₂O₃ have been considered for applications in many fields due to their inherent properties, such as hardness, low electrical conductivity, good chemical stability, and oxidation resistance. Most research has focused on particle-dispersed Al₂O₃ composites, selecting additional material to improve their mechanical properties such as flexural strength and fracture toughness, including SiC,₁–₃ ZrO₂,₄,₅ TiN, TiC, TiO₂,₆,₇ BN,₈ and metal particles such as Cr.₉

Mullite (3Al₂O₃·2SiO₂) including Al₂O₃ systems in general have become materials of interest for structural and optoelectronic applications.¹₀,¹¹ The special features of mullite-based ceramics are related to mullite’s high refractoriness, good thermo-mechanical properties, low thermal expansion, excellent conductivity, superior oxidation resistance, and heat resistance. Since mullite has low fracture toughness and low bending strength, incorporation of nano-sized second-phase particles, such as SiC, into the mullite matrix could improve its mechanical properties. Ando et al.¹² and Takahashi et al.¹³ also showed that mullite-SiC composites exhibited self-healing characteristics.

Most literatures studied on hot-pressed Al₂O₃–SiC composites to enhance composite mechanical properties. SiC has attracted particular interest due to its structural, electronic, physical, and thermal properties, including its high thermal conductivity, electrical conductivity, and elastic modulus. Composite strength is enhanced by providing a fine-grained microstructure by adding more assisting particles. Fracture toughness can be improved through crack deflection, micro cracking, and compressive residual stress. Al₂O₃ ceramics generally have good sintering capacity, but excessive addition of carbide or nitride results in pores⁴ and cracks.

The effects of porosity, grain size, and SiC addition on the damage and wear resistance of Al₂O₃-based composites have been widely studied and analyzed,¹–³ and SiC addition in Al₂O₃ can mitigate the disadvantages of other particles, improving the damage and wear resistance of the composite.¹⁵–¹⁸ Al₂O₃–SiC composite mechanical properties are greatly influenced by the microstructure induced by SiC addition and sintering, and composites with denser and finer structures have higher flexural strength.¹¹,¹⁷ The fracture mode changes from intergranular fracture for monolithic Al₂O₃ to partial trans-granular fracture for composites containing SiC.²,³ Fracture toughness first increases with the SiC particle toughening effect, and then decreases with increases in SiC content, because the microstructure is significantly refined. Up to now, studies have focused on SiC content of about 5% or so. No research has been done on small amounts of SiC addition to Al₂O₃ ceramics. Recent literature has reported, however, that a small amount of graphene contributed to improved wear resistance and mechanical properties.¹⁹,²⁰ Ceramic nanocomposites have attracted significant attention due to their improved mechanical properties.
such as hardness, strength, wear resistance, and the possibility of super plasticity, compared with monolithic materials.\textsuperscript{18,21} These nanocomposites may be useful as structural and functional materials for a variety of applications in different fields. Retaining their superior properties at high temperatures is, however, a major technological challenge. The observed enhanced properties are strongly influenced by the dimensions of the phases present in the ceramic nanocomposite. Controlling the nanocomposites requires well-established and reliable processing methodologies. In general bulk nanocomposites are fabricated using high-cost processes.

This study fabricated Al$_2$O$_3$–SiC composites with SiC content of 0–10 wt\%, including a small amount of 2 wt\% of SiC, and investigated the effects on damage and wear resistance. The composites were fabricated by spark plasma sintering at 1600°C in a partial vacuum (80 MPa). Hardness was evaluated using the ball-on-disk method, and Hertzian indentation evaluation using a spherical indenter was used to investigate the hard and elastic behavior of the composite.

Relative hardness evaluated by analyzing the amount of permanent deformation during Hertzian indentation shows that the hardness of Al$_2$O$_3$–SiC microcomposites can be enhanced, even when the SiC addition is less than 2 wt\%. The evaluations using a spherical indenter indicate hard and elastic behavior of the composites with the addition of SiC. In the present study, it is found that the damage and wear resistance of Al$_2$O$_3$–SiC is significantly affected by a small amount of SiC content, even if it is not by nanoparticles. It is concluded that Al$_2$O$_3$–SiC microcomposites exhibit superior damage and wear resistance.

2. Experimental procedures

2.1 Fabrication of Al$_2$O$_3$–SiC microcomposites

Commercial starting powders of Al$_2$O$_3$ (Taimei Co, TM-DAR) and fine SiC (Ibiden Co.) were selected for composite preparation, with Y$_2$O$_3$ (Rare Metallic Co. 99.9% purity) powder added to improve sintering. Table 1 shows the initial material proportions for the Al$_2$O$_3$–SiC composites. Each composition shown was mixed as a slurry with isopropyl alcohol for 24 h using a ball mill with 200 mesh screen. The powders were put into graphite molds with 30 mm diameters, and spark plasma sintering was performed (Syntex Inc., Japan) at 1600°C for 20 min in a partial vacuum (80 MPa). The sintered body was fractured and the fracture site observed by Field Emission Scanning Electron Microscope (FE-SEM).

2.2 Damage and wear resistance

Figure 1 shows the Hertzian indentation and ball-on-disk wear test methods used in this study. The same tungsten carbide (WC) spherical indenter was used for both tests. 25.4 mm diameter samples of the tested composite material were prepared, and the surfaces were polished to a 1 μm finish using diamond suspension.

The Hertzian indentation test was carried out to induce contact damage on the polished Al$_2$O$_3$–SiC composites using a universal testing machine (Instron 5567, UK). The top surfaces of each sample were contacted with a WC spherical indenter, radius $r = 3.18$ mm, and the load was increased to $P = 2000$ N, and then reduced to $P = 0$ N. Displacement data was collected through an amplifier, analog-digital converters, and a digital signal processor after measuring by extensometer. The constant load was driven at a crosshead speed 0.2 mm/min during indentation testing and unloading. All tests were conducted in air. The relative hardness and elastic modulus were evaluated by analyzing the permanent deformation and tangential slope of the unloading curve, respectively, during Hertzian indentation.

Indentation damage was observed by optical microscope. The local plastic deformed regions were observed using Nomarski illumination,\textsuperscript{22,23} but ring cracks occurring on the surfaces were observed without Nomarski illumination. The surfaces were gold coated to assist visualizing the developed damages precisely.

Wear resistance was evaluated using the ball-on-disk method. Wear tests were performed with a rotating ball and stationary disk sample fixed on a stainless jig. The rotating circle was 20 mm diameter, and the rotation speed was maintained at a constant 62.8 cm/sec for 1800 s under a fixed load. Samples were contacted with a WC sphere, $r = 1.98$ or 3.18 mm at load $P = 58.8$ N, and the friction coefficient was measured by friction load sensor. The weight of each sample was measured before and after the wear test, and the wear loss was calculated.

| Al$_2$O$_3$–SiC wt% | Al$_2$O$_3$ (%) | SiC (%) | Y$_2$O$_3$ (%) | Theoretical density (g/cm$^3$) | Sintered density (g/cm$^3$) |
|---------------------|--------------|--------|--------------|-----------------------------|-----------------------------|
| 0 wt%               | 100          | 0      | 0            | 3.98                        | 3.98                        |
| 2 wt%               | 96.8         | 2      | 1.2          | 3.97                        | 3.97                        |
| 5 wt%               | 92           | 5      | 3            | 3.95                        | 3.95                        |
| 10 wt%              | 84           | 10     | 6            | 3.93                        | 3.93                        |

Fig. 1. Schematic diagrams of (a) Hertzian indentation and (b) a wear test by the ball-on-disk method.
3. Results and discussion

Table 1 shows the theoretical and sintered density of Al₂O₃ and Al₂O₃–SiC composites. The theoretical density was calculated using the lever rule for the Al₂O₃–SiC mixture with Al₂O₃ and SiC density = 3.98 and 3.21 g/cm³, respectively. As the proportion of SiC increases, composite density decreases because of the lower theoretical density of SiC. The mixtures were all sintered at 1600°C under a partial vacuum (80 MPa), and fully densified samples were obtained, i.e., no pores were observed any composition.

Figure 2 shows typical microstructures of sintered Al₂O₃–SiC composites as a function of the SiC amount. All samples consisted of equiaxed grains of Al₂O₃ and finely dispersed SiC in an Al₂O₃ matrix. The sintered Al₂O₃–SiC composites show full densification. SiC surfaces are easily oxidized and a thin layer of silica rests on the SiC surface. A small number of amorphous silicate-type phases were also formed due to a reaction between the silica on the SiC surface and oxide impurities (e.g. Al, Fe, Ca) present in the initial powder. Amorphous silicate phases, as well as Y₂O₃, act as sintering aids, accelerating composite sintering through a mechanism similar to liquid-phase sintering. The sample grain size decreased quickly with increasing SiC content, since the SiC particles allow pinning, which suppressed alumina grain growth.

Figure 3 shows the load-displacement curves for Al₂O₃–SiC composites with different SiC contents under Hertzian indentation using a WC ball r = 3.18 mm at maximum load P = 2000 N. Indentation loading caused fractures only in local areas and provided the predicted macroscopic mechanical properties. The load-displacement curves were continuous during loading and unloading. The permanent deformation after unloading became smaller as the SiC content increased, and the tangential curve slope is almost the same irrespective of SiC content. The residual displacement due to permanent deformation after complete unloading is related to hardness, and the tangential curve slope is related to the elastic modulus. The composite became harder with increasing SiC addition, therefore, without affecting the high elastic modulus.

Figure 4 shows optical micrographs of contact damage on composite surfaces using WC ball r = 3.18 mm at load P = 2000 N. Classic ring cracks were observed in all the composites, irrespective of SiC contents. The cracks are propagated as full circles, but their depth appears to decreases slightly with increases in SiC content as the density of the ring-cracks increase. These results imply that crack propagation is hindered by SiC addition. Local quasi-plasticity was not evident, which indicates that the composite had hard and elastic properties. Figures 3 and 4
show hard and elastic properties with the addition of SiC to the composite.

**Figure 5** shows the relative hardness ratio and Young’s modulus as a function of SiC content in Al$_2$O$_3$–SiC composites. The relative data are obtained from residual displacement after complete unloading for hardness and the tangential slope on the unloading curve for the elastic modulus in each graph of Fig. 3. Young’s modulus shows almost the same data up to 10 wt% of SiC, as seen in Fig. 5(b). The addition of SiC to the Al$_2$O$_3$ exhibits 1.018–1.053 times the initial Young’s modulus. The values are thus similar to each other, because the elastic modulus of Al$_2$O$_3$ is 380 GPa and that of SiC is 410 GPa.

On the other hand, SiC addition increases the relative hardness continuously, almost doubling it over the addition range investigated, as shown in Fig. 5(a). This is consistent with the increased SiC hardness compared to Al$_2$O$_3$.

**Figure 6** shows the representative indentation stress–strain curves of the fabricated composites. Indentation strain increases with increases in indentation stress. Since the increase is nonlinear, however, local yielding must occur during spherical indentation. Indentation stress and strain at 0.05–0.06 a/r (contact area/ball radius) shows that SiC addition reduces strain, indicating higher damage resistance.

**Figure 7** shows friction coefficient changes of the fabricated composites as a function of sliding distance for the wear test using WC ball $r = 1.98$ mm with $P = 58.8$ N and rotation 60 rpm. Initial friction coefficients for 2 wt% SiC composites are lower than those for 0, 5, and 10 wt% SiC. Thus, the sliding contact shows better initial wear resistance for 2 wt% added SiC composites than for the other additive amounts, while the friction coefficient increases continuously for the Al$_2$O$_3$ without SiC addition with increasing wear. Al$_2$O$_3$ with 5 and 10 wt% SiC composites shows a constant friction coefficient during wear testing. This implies that Al$_2$O$_3$–SiC composite wear resistance is successfully controlled by a small addition of SiC.

**Figure 8** shows the mass changes for the wear test on fabricated composites as a function of SiC content using WC ball $r = 1.98$ mm with $P = 58.8$ N and a rotation of 60 rpm. Wear loss of Al$_2$O$_3$ after 1800 s testing was 0.04 g. Figure 8 shows that wear resistance was significantly improved even for small additional SiC, with minimum mass change occurring for 2 wt% SiC content composites. These results are consistent with Figs. 3 and 5. Since composite hardness is closely related to wear resistance, mass loss is reduced by SiC addition due to improved hardness.

**Figure 9** shows the width of the wear scars on composite surfaces using a video microscope for different SiC addition. Figures 9(a)–9(d) show wear test results for 0, 2, 5, and 10 wt% added SiC, respectively. Wear scar has minimum width for 2 wt% SiC, a result consistent with Fig. 8.

**Figure 10** summarizes the wear width after testing from Fig. 9. Wear width is significantly reduced with SiC addition, indicating high wear resistance of the Al$_2$O$_3$–SiC (2 wt%) composite.
4. Conclusions

This study investigated the effects of SiC addition to Al₂O₃ ceramics on indentation and wear resistance using tungsten carbide balls to induce load on composite surfaces. Composites included 0, 2, 5, and 10 wt% of SiC.

Damage and wear resistance were evaluated by Hertzian indentation and wear test using the ball-on-disk method.

1. Increased hardness and elasticity maintenance were observed with SiC addition.
2. Damage resistance was most significantly improved for 2 wt% SiC addition, whereas relative hardness doubled with 10 wt% addition compared to unmodified Al₂O₃ ceramic.

3. Friction coefficient, mass loss, and wear width change results for the wear test indicate that Al₂O₃ wear resistance can be significantly enhanced by small SiC addition, i.e., 2 wt%. The friction coefficient was significantly reduced and wear resistance significantly improved with a small amount of SiC addition.

Thus, Al₂O₃–SiC composites exhibit superior damage and wear resistance, and enhanced wear resistance can be achieved with a relatively small SiC addition of 2 wt%.

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