*The Korean Journal of Ceramics*, **3** [1] 47 - 51 (1997)

# Tribological Behaviour of the Si/SiC and the Si/SiC/Graphite Composites

**In-Sub Kim, Dong-Woo Shin*, You-Young So** and Byung-Ha Lee****

*National Institute of Ceramic Technology, Seoul 152-023, Korea*
**Gyeongsang National University, Department of Inorganic Materials Engineering, 900 Kajwa-Dong, Chinju Kyongnam, 660-701, Korea*
***Myung Ji University, Department of Inorganic Materials Engineering, Kyung Ki, 449-728, Korea*
(Received October 30, 1996)

The dense sintered bodies of Si/SiC composite with various Si contents could be fabricated by changing the green density in the forming process. The Si/SiC/graphite composites with various graphite contents could be also fabricated by changing a graphite content in the starting composition. Their mechanical and tribological properties were characterized and wear mechanism was also studied. The hardness and strength of the Si/SiC and the Si/SiC/graphite were decreased with increasing the contents of free Si and graphite, respectively However, the friction coefficient and specific wear rate had no specific relations to their hardness and strength. Adhesion of free Si was a main factor to determine a wear resistance of the Si/SiC composite. In the case of the Si/SiC/graphite, solid lubrication and liquid reservoir of the graphite particles played the main role of the reduction of the friction force. In the torque test to estimate the possibility of practical applications, the value of torque between the $Al_2O_3$ disk and Si/SiC/graphite disk was 1/6 lower compared with two $Al_2O_3$ disks on the basis of 100,000 cycles

***Key words :*** Si/SiC composite, Si/SiC/graphite composite, Tribological property, Friction, Wear, Lubrication

## I. Introduction

The reaction-bonded silicon carbide (RBSC) had particular advantages in view of machining and cost, since RBSC was consolidated to pore-free body with no or a little dimensional change at relatively low temperature of 1600~1800°C.[1-5] The near-net-shape fabrication of RBSC at a low temperature prompted SiC-based ceramics to the applications, such as mechanical seal ring, sleeve, sliding bearing, valve disc, and nozzle etc., despite of the sudden deterioration of mechanical properties at temperature above 1400°C due to the inevitably existing free Si (8~15 wt%).[6,7,12]

The application of structural ceramics to sliding and contacting parts that are dynamically stressed at high temperature requires to contain solid lubricant to reduce the friction coefficient and the wear resistence. The Si/SiC composite and the Si/SiC/graphite composite have been one of promising candidates for such applications. However, few have been reported on the tribological properties and behaviours of the Si/SiC composite and the Si/SiC/graphite composite by the analysis of mechanical properties and wear surfaces, whereas recent papers reported the sliding wear behaviours of Si/SiC, $Si_3N_4$, SiC, $ZrO_2$, Sialon.[8,9]

In this work we have studied the Si/SiC composite and the Si/SiC/graphite composite to determine the relation of mechanical, tribological properties and wear behaviour. Especially, the effects of the free Si and graphite content on the strength, hardness, friction coefficient, specific wear rate, and wear behaviour of the Si/SiC composites and the Si/SiC/graphite composites were analyzed

## II. Experimental Procedure

### *1. Fabrication of the Si/SiC composites and the Si/SiC/graphite composites*

α-SiC, carbon black, graphite ingot and Si metal were used as starting materials. α-SiC (80 wt%) and carbon black (20 wt%) were first dry-mixed in vibrating ball mill for 3 hrs. The graphite particles sieved with the mesh (150-425 μm) after crushing the ingot were then mixed with a mixture of α-SiC and carbon black in the impeller mixer for 15 min. Batches consisting α-SiC and carbon black were also prepared by the same procedure. The content of graphite was the vol.% of graphite particle with respect to the total volume of starting powders, i.e., the mixture of α-SiC, carbon black and graphite. The four different batches including graphite free one were pressed with 37, 74, 101, 151.5 MPa. Si infiltration was then performed at 1750°C for 10 min with heating rate of 5°C/min in vacuum atmosphere of 3 torr.

### *2. Measurement of mechanical properties*

The bulk density was measured by the Archemedes method, after removing the excess silicon remained on the surface of composites. The values obtained from four specimens in each batch were averaged. The reaction-bonded specimens were cut into bars (3 mm×3 mm×45 mm) prior to test of 3-point flexural strength with a 25 mm span and crosshead speed of 0.5 mm/min. Hardness was determined by Rockwell indentation technique. Each polished surface was indented 10 times using Rockwell indentor with a major load of 15 N. The reason of choosing the cone-shaped Rockwell indentor with angle of 120° instead of Vickers indentor in the tetrahedral shape is attributed to relatively larger indentation mark. The small Vickers indentation of ~50 μm diagonal under 15 N could represent only the hardness of a specific constituent of the composite, since some grains of SiC and graphite are much larger than the indentation mark of Vickers indentor[10]. However, the diameter of Rockwell indentation is about 400 μm with the load of 15 N. Thus, Rockwell technique can provide more reliable results with narrower deviation compared with those obtained

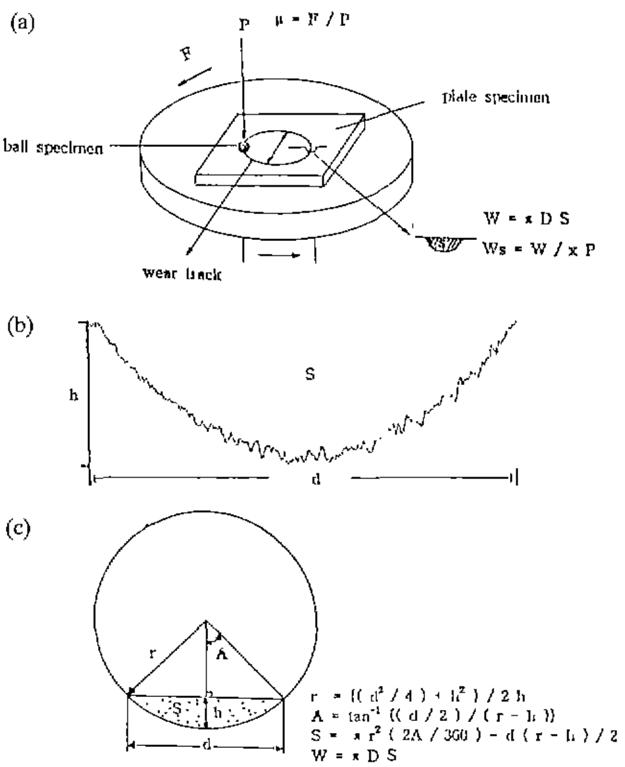


**Fig. 1.** Schematics of (a) the ball-on-plate type wear test, (b) the form of wear track obtained from the surface roughness analyzer, and (c) the cross section of wear track that is estimated by assuming as a part of the cross section of circle.

from Vickers indentation.

### 3. Measurement of the friction coefficient and specific wear rate

The sliding friction and wear tests were performed on a ball-on-plate type tribosystem in a wet condition, as schematically shown in Fig. 1(a). The sintered SiC balls with diameter of 10 mm were used as a counter-sliding material. Vickers hardness and grain size of the ball were 24.5 GPa and 3~4 μm, repectively. SEM micrographs of the fracture and chemically etched surfaces are shown in Fig. 2. The contacting surface of the Si/SiC/graphite composite plate with 35 mm×45 mm×6 mm was lapped with diamond paste of 0.01 to 0.08 μm. The 'wet' wear test was then carried out in water bath at the room temperature, with an applied load of 5 N and sliding velocity of 500 mm/sec. Sliding distance was limited to 2.1 km. The coefficient of friction was calculated from the frictional force. The depth and width of four different wear tracks marked on the surface of plate were measured using the surface form analyzer.[11]

The specific wear rate was then calculated from the wear volume predicted by the assumption shown in Fig. 1(c). The surface roughness analyzer can define the form of wear track in terms of depth (h) and width (d), as demonstrated in Fig. 1(b). The cross section of wear track (S) can be roughly estimated by assuming as a part of circle in Fig. 1(c). From the simple geometric relation, the radius of circle $(r)=\{(d^2/4)+h^2\}/2h$, angle $(A)=\tan^{-1}\{(d/2)/(r-h)\}$, and then $S=\pi r^2(2A/360)-d(r-h)/2$. The total wear volume (W) can be calculated from the average radius of wear track (D), i.e., $W=\pi DS$. The specific wear rate (Ws) of the composite can be finally estimated from the sliding distance (X) and applied load (P), i.e., $Ws=W/(PX)$, where X is obtained from the angular velocity and sliding distance.

## III. Results and Discussion

### 1. Mechanical and tribological properties

The mechanical and tribological properties of the Si/SiC composite and Si/SiC/graphite composite are given in Table 1. The volume fraction of free silicon ($V_{si}$) existing in the Si/SiC composites can be estimated by the simple equation using the theoretical densities of SiC

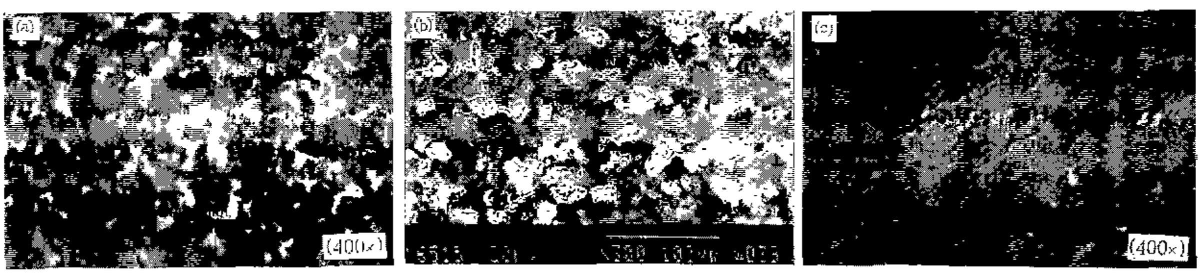


**Fig. 2.** Micrographs of wear surface in wear test of SiC ball on the Si/SiC composite plate.

**Table 1.** Mechanical and Tribological Properties of the Si/SiC Composite and the Si/SiC/graphite Composites

| Composites \ Properties | | Rockwell Hardness 15 N | Flexural Strength MPa | Friction Coefficient μ | Specific Wear Rate $mm^2kgf^{-1}$ |
|---|---|---|---|---|---|
| Si/SiC | 15.82 vol% Si | 97.3 | 371 | 0.08 | $4.0\times10^{-8}$ |
| | 16.62 vol% Si | 97.1 | 363 | 0.08 | $2.4\times10^{-8}$ |
| | 17.64 vol% Si | 96.8 | 351 | 0.09 | $4.3\times10^{-8}$ |
| | 19.11 vol% Si | 96.1 | 278 | 0.12 | $7.6\times10^{-8}$ |
| Si/SiC/graphite | 20 vol% graphite | 94.6 | 192 | 0.05 | $1.5\times10^{-8}$ |
| | 25 vol% graphite | 92.3 | 184 | 0.04 | $2.0\times10^{-8}$ |
| | 30 vol% graphite | 92.0 | 175 | 0.03 | $2.4\times10^{-8}$ |

(3.21 g/cm$^3$) and Si (2.33 g/cm$^3$),[8)] i.e., $V_{si}$=3.648-1.136D, where D is the bulk density. Those of the four different Si/SiC composite specimens were 15.82, 16.62, 17.64, 19.11 vol.%.

The 3-point flexural strengths and Rockwell hardnesses of the Si/SiC composites and the Si/SiC/graphite composites were decreased with increasing the Si content, as we expected from low bending compressive strengths of free Si and graphite. Flexural strength of the Si/SiC/25 vol.% graphite was 185 MPa, that is much higher than the values reported (120~130 MPa) for the commercial products. The reason for the superior flexural strength is because of the more uniform microstructure. It seemed to be originated from the finer SiC particles (2.5~13 μm) used as a starting material compared with those of 30 to 60 μm used by others. The friction coefficient of the Si/SiC composites increased with decreasing hardness and flexural strength, whereas that of the Si/SiC/graphite composites decreased with decreasing hardness and flexural strength. Also the specific wear rate of the Si/SiC composites and the Si/SiC/graphite composites didn't have a specific relation with their hardness and flexural strength.

### *2. Wear mechanism of the Si/SiC composites*

The wear surfaces of the Si/SiC composites are given in Fig. 2, where the worn regions with a fine-polished surface (a), the pitted channels around SiC grains (b), and the regions with free Si adhesion (c) are shown. In the initial wear stage, the worn surface was finely polished. In the intermediate wear stage (b) the pitted channels around SiC grains began to appear, while free Si regions disappeared. And increments of the friction coefficient were caused by adhesion of free Si between the SiC grains (c). From these results and the dependence of free Si on the friction coefficient and the specific wear rate, we can propose the wear mechanism of the Si/SiC composites, as suggested schematically in Fig. 3.

### *3. Wear mechanism of the Si/SiC/graphite composites*

The wear surfaces of the Si/SiC/graphite composite are shown in Fig. 4, where the wear track in contrast to the

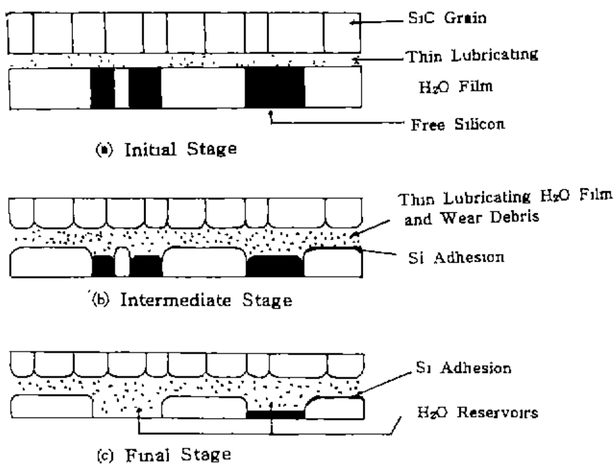


**Fig. 3.** Representation of possible sliding mechanism in wear of SiC ball on the Si/SiC composite plate.

unworn regions (a), and the morphology around the graphite grains existing within the wear track (b and c) are shown. The contrast of darkness between the worn and the unworn surfaces (a) was caused by the sticking of graphite debris to the surrounding SiC grains and Si regions (b). Some areas of the Si/SiC matrix were coated severely with the wear debris of graphite during wear test, as shown in Fig. 4(c). From these results and the dependence of friction coefficient and the specific wear rate on the graphite content, we can propose the wear mechanism of the Si/SiC/graphite composite, as suggested schematically in Fig. 5.

The soft constituents of composite, i.e., free Si and graphite, were worn first in progressing the wet wear test (b), and the surface of SiC grains nearby large graphite particle was coated with the wear debris, which consist mainly of graphite (c). The pitted graphite grains, which were surrounded with SiC grains, could contain the water. Therefore, the higher graphite content in the Si/SiC/graphite composite disk made the enlargement of lubricating graphite area in the sliding surface, the increase of the amount of graphite debris and the water content in the pitted regions resulted in the lower coef-

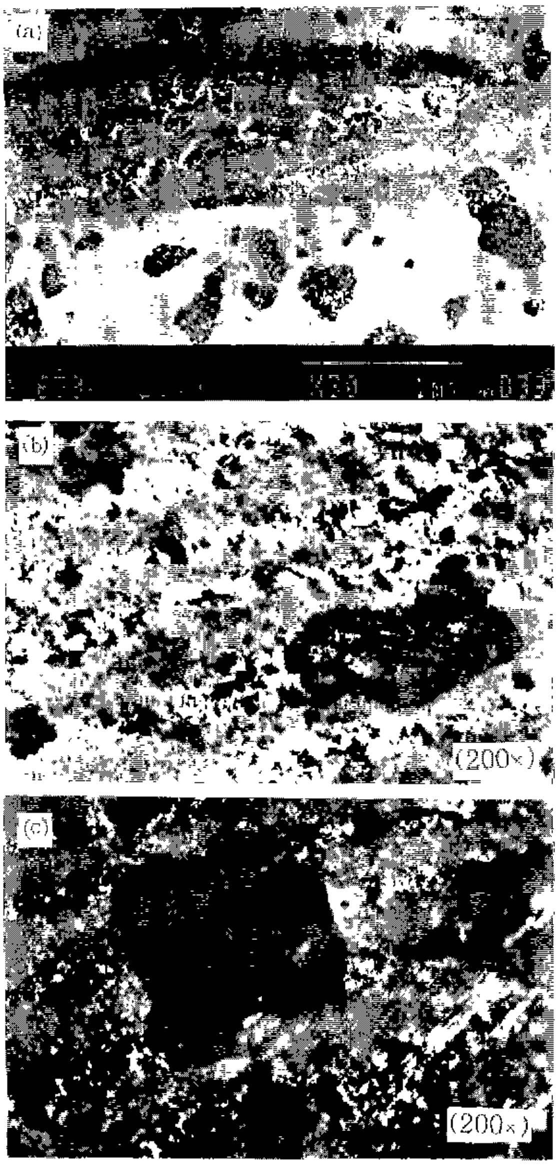


Fig. 4. Micrographs of wear surface in wear test of SiC ball on the Si/SiC/graphite composite plate

ficient of friction, when they were used in the moisture environment.

### *4. Torque tests for applications*

The torque tests were carried out to estimate the possibility of practical application of Si/SiC/25 vol% graphite composite which had good tribological properties. This is one of wear test methods to measure the friction force between two disks. Tap seal valves can be one of the candidates in practical applications of the Si/SiC/graphite composites. Ceramic disks, such as $Al_2O_3$-$Al_2O_3$, $Al_2O_3$-SiC and SiC-SiC have been commercialized for a tap seal valve. One of some problems of the $Al_2O_3$-$Al_2O_3$ disks in tap seal is a rapid increasing rate of friction force in 50,000 to 100,000 cycles. SiC ceramics with good tribological properties were substituted for $Al_2O_3$ ceramics to solve this problem. So, torque tests of $Al_2O_3$-$Al_2O_3$, $Al_2O_3$-SiC and $Al_2O_3$-Si/SiC/graphite systems were carried out by disk-on-disk type wear tester. Their results are shown in Fig. 6. Torques of $Al_2O_3$-$Al_2O_3$ system were rapidly increased with increasing the number of cycles. In this case, there was 60% torque increment in 200,000 cycles on the basis of initial stage. Torque of $Al_2O_3$-SiC are slowly increased with increasing the number of cycles. The torque was just a half compared with $Al_2O_3$-$Al_2O_3$ on the basis of 100,000 cycles. Torque between $Al_2O_3$ disk and Si/SiC/graphite disk was not changed from the initial stage to the final stage. The torque was 1/6 compared with $Al_2O_3$-$Al_2O_3$ on the basis of 100,000 cycles. Therefore, the Si/SiC/graphtie composite can be a very promising sliding material when low torque is especially required

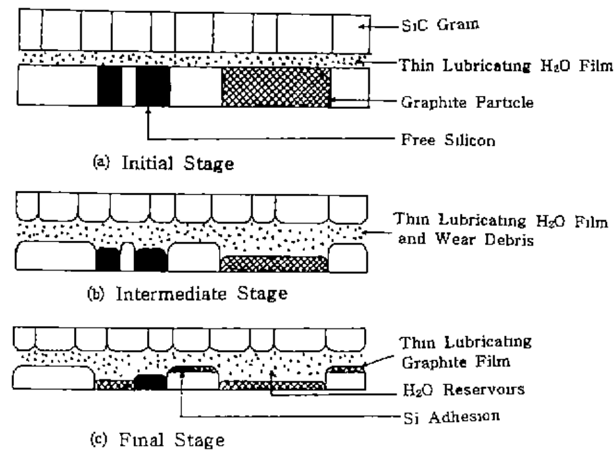


Fig. 5. Representation of possible sliding mechanism in wear of SiC ball on the Si/SiC/graphite composite plate.

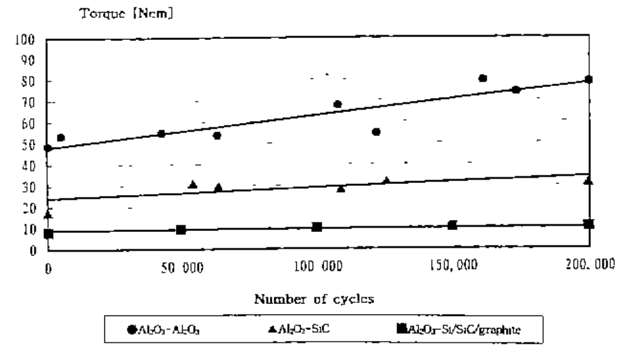


Fig. 6. Torque tests of $Al_2O_3$-$Al_2O_3$, $Al_2O_3$-SiC and $Al_2O_3$-Si/SiC/graphite.

## IV. Conclusions

The dense Si/SiC and Si/SiC/graphite composites were fabricated by the reaction bonding process with infiltrated Si metal. The wear properties of the composites were characterized by the 'wet' wear test using SiC ball. Adhesion of free Si was a main factor of friction and specific wear rate in the wear test of the Si/SiC composite. Also solid graphite lubrication and liquid reservoir in pitted graphite grains played a main role of the reduction of friction force in the Si/SiC/graphite composite, i.e , a combined solid lubricating effect of graphite as a solid lubricant and a water reservoir of pitted graphite grains caused the reduction of friction coefficient with increasing the graphite content. The friction coefficient was linearly changed from 0.12 of the Si/SiC composite to 0.03 of Si/SiC/30 vol% graphite composite. The specific wear rate exhibited the lowest value of $1.5\times10^{-8}$ $mm^2/kg_f$ at 25 vol% of graphite. From the results, the optimum content of graphite in the Si/SiC/graphite composites can be concluded to be ~25 vol% when they were used as mechanical and wear resistant parts. Torque between the $Al_2O_3$ and Si/SiC/graphite disks was 1/6 that of the $Al_2O_3$-$Al_2O_3$ disks on the basis of 100,000 cycles.

### Acknowledgements

This study is supported by Korean Ministry of Education through 1995 Research Fund.

## Reference

1. G. R. Sawyer and T. F. Page, "Microstucture Characterization of "REFEL" (Reaction-Bonded) Silicon Carbides," *J. Mat. Sci.*, **13**, pp. 885-904 (1978).
2. P. A. Willerment, R. A. Pettand and J. J. Whalen, "Development and Processing of Injection-Moldable Reaction-Sintered SiC Compositions," *Am. Ceram. Soc. Bull.*, **57**[8], 744-747 (1978).
3. B. John and J Wachtman, "Structural Ceramics," Academic Press INC., Vol. **29**, pp. 91-163 (1989).
4. P. Popper, "The Preparation of Dense Self-Bonded Silicon Carbide," Special Ceramics, Heywood, 209-219 (1960).
5. W. B. Hilling, R. L. Mehan, C. R. Morelock and V. J. Decarlo, W. Laskow, "Silicon/Silicon Silicon Carbide Composites," *Ceram. Bull.*, **54**[12], 1054-56 (1975).
6. C. W. Forrest, P. Kennedy and J. V. Shenann, "The Fabrication and Properties of Self-Bonded Silicon Carbide Bodies," *Special Ceramics* Vol. **5**, pp. 99-123 (1972).
7. C. B. Lim and T. Iseki, "Transport of Fine-Grained β-SiC in SiC/Liquid Si System," *Adv. Ceram. Mater.*, **3**, 291-293 (1988).
8. M. IwaSa and S. Kakiuchi, "Mechanical and Tribological Properties of $Si_3N_4$-BN Composite Ceramics," *Yogyo-Kyokai-Shi*, **93**[10], 97-101 (1985).
9. D. C. Evans. "Self-lubricating Bearings." Industrial Lubrication and Tribology, **33**[4], 132-138 (1981).
10. I. S. Han, J. H. Yang and D. S. Suhr, "Properties of Reaction-Bonded Silicon Carbide Depending on Graphite Additions," *J. Kor. Ceram. Soc.*, **30**[1], 69-77 (1993).
11. M. Iwasa and M. Kinoshita, "Wear Behaviour of Silicon Nitride Ball,' *J. Ceram. Soc. Japn*, **97**[2], 195-199 (1989).
12. M Shinivasan and M Kasprzyk, "The Effect of Microstructure on the mechanical Properties of Reaction-Sintered Silicon Carbide," Presented at Fall Meeting of Basic Science Division of American Ceramic Society, New Orieans, November (1979).
13. K. Miyoshi and D. H. Buckley, "Friction Deformation and Fracture of Single Crystal Silicon Carbide," *Powder Metall.*, **8**, 113-27 (1961).
14. K. Miyoshi and D. H. Buckley. "The Generation and Morphology of Single Crystal Silicon Carbide Wear Particles under Adhesive Conditions," *Wear*, **67**[3], 303-19 (1981).