Dry Sliding Wear Mechanism of Spark Plasma Sintered Si_3N_4/SiC Composites on Steel

Zeynep Taslicukur Ozturk,
Istanbul, Turkey

Article Information

Correspondence Address
Assist. Prof. Dr. Zeynep Taslicukur Ozturk
Gedik University
Metallurgy and Materials Engineering
34876, Cumhuriyet Mah.Kartal/Istanbul, Turkey
E-mail: ztasli@gmail.com

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In the present work, Si_3N_4-based composites were characterized by a relatively low friction coefficient in unlubricated sliding against a 100Cr6 steel ball. Si_3N_4-based composites containing different amounts of SiC were produced by spark plasma sintering at 1650 °C for 5 min with a uniaxial pressure of 40 MPa in a nitrogen atmosphere. In the wear test, the load, total distance and rotating speed were selected as 60 N, 120 m and 500 rpm, respectively. The wear tracks were examined by scanning electron microscopy (SEM) to estimate the wear mechanism. The results were evaluated using the friction coefficient-distance diagram. Si_3N_4-based composites showed significant decrement in wear by improving toughness with SiC powder reinforcement.

In tribological applications, the use of engineering ceramics has been increasing. Silicon nitride ceramics are considered for use in many mechanical applications, especially for rolling contact systems (e. g. ball bearings) or for reciprocating engine parts (e. g. valves, valve seats, valve guides, and various industrial wear parts) [1-9]. In order to improve mechanical properties of monolithic Si_3N_4 ceramics, a second phase like platelets, whiskers or particles are added to the Si_3N_4 matrix [2, 10, 11]. Silicon nitride reinforced with silicon carbide is a potentially important component in a number of ceramic composites [12]. Reinforcement of ceramics by SiC is often applied to abrasive, erosive or impact wear [3]. The densification of Si_3N_4, commonly entails the use of α-Si_3N_4 powders plus rare earth oxide additives, and temperatures as high as 1700 to 1800 °C for promoting the liquid-phase sintering. Rare earth (RE) oxide sintering additives are considered as ideal additives for Si_3N_4 due to their high melting point and due to the fact that they control the $\alpha \rightarrow \beta$ phase transformation rate, the grain growth anisotropy and the aspect ratio of the β-Si_3N_4 grains [2, 9]. The SPS technique can enhance the sinterability of Si_3N_4 producing dense materials with controlled grain growth and phase transformation at reduced temperatures [2, 9, 13, 14]. Tribological materials are also required to possess higher mechanical properties like structural materials. The mechanical properties, such as strength and fracture toughness are closely related to their microstructures [4]. Tribological behavior of ceramic materials is strongly dependent on contact load, sliding speed, temperature, humidity, contact geometric configuration, lubricant, as well as microstructure of materials [2, 6, 15]. The purpose of this study is to investigate the influence of SiC particles added to Si_3N_4 matrix on the wear behaviour of Si_3N_4/SiC composite.

Experimental Procedure

α-Si_3N_4 powder (UBE Industries, SN-E10, 38 wt.-% N, > 2 wt.-% O, > 0,2 wt.-% C), SiC powder (H.C. Starck, grade UF-25, > 28,5-29,5 wt.-% C, > 2,5 wt.-% O) were used as powder precursors. High purity AlN (H.C. Starck, grade C) and Y_2O_3 (H.C. Starck, grade C) were used as sintering additives. The composition of the starting powder mixtures was given in Table 1. Powder batches were produced by wet ball milling with ethanol in a polyurethane bottle for 16 h. The slurries were dried at 100 °C in 5 h, and then sieved to a particle size < 185 µm. The powder precursors were loaded in a cylindrical graphite die with an inner diameter of 50 mm and uniaxially pressed into green compacts by using a hydraulic pressure of 40 MPa. The green compacts were sintered in DR SINTER SPS (spark plasma sintering) -7.40 MK by applying 40 MPa pressure for 5 minutes at 1650 °C in nitrogen atmosphere. The heating rate was 100 °C min^{-1}. The temperature was measured by means of an optical infrared thermometer focused on the graphite die surface.

Relative density of the samples after sintering was determined by Archimedes method. The crystal phases of the samples were investigated by X-ray diffractometry (XRD) using X'Pert Pro MRD Panalytical diffractometer. Spark plasma sintered samples were cut and polished to a 1 µm dia-

Sample	Si_3N_4	SiC	AlN	Y_2O_3
SSC10	80	10	5	5
SSC30	60	30	5	5

Table 1. Composition of starting powder mixtures [wt.-%]

mond finish. The microstructures of the samples were characterized by scanning electron microscopy (SEM) (JEOL JSM-7000 F). Hardness of the samples was tested by a micro Vicker tester (Struers Duramin A-300) with an applied load of 2 kg. The strength was measured by three-point bending. For tribological characterization, wear tests were performed under dry sliding contidition and wear characteristics were then determined based on the worn surfaces. The wear behavior of the composites was studied by unlubricated "ball-on-disc" type tribometer (Nanovea) against a polished 100Cr6 steel ball at room temperature. Related friction and wear test parameters were listed in Table 2. The wear tracks were examined by SEM to estimate the wear mechanisms.

Results and Discussion

XRD patterns of 1650 °C under 40 MPa pressure for 5 min spark plasma sintered samples SSC10 and SSC30 were shown in Figure 1. The main phases were α-Si_3N_4, β-Si_3N_4, SiC and SiAlON in both composite materials. Also, Y_2O_3 could be detected in SSC10 composite.

Mechanical properties of SSC10 and SSC30 composites sintered at 1650 °C under 40 MPa for 5 minutes were given in Table 3. Fracture toughness value of SSC30 was 6.0 MPa × $m^{1/2}$, while the fracture toughness value of SSC10 was 5.8 MPa × $m^{1/2}$. Higher toughness exhibited better wear resistance. The specimen was more resistant to wear when it had a high bending strength and hardness. The fracture toughness of Si_3N_4-based composites played an important role for wear behavior [15, 16]. The fracture toughness of silicon nitride was influenced by the microstructure characteristics (grain diameter, aspect ratio of grains, amount of elongated grains). The composite showed decrement in wear by improving toughness with SiC powder reinforcement. Tatarko et al. reported that the wear rate of Si_3N_4 ceramic composite decreased with the increasing fracture toughness. The hardness values of SSC10 and SSC30 were 21.4 GPa and 22.5 GPa, respectively. This can be explained by the presence of harder SiC particles in the Si_3N_4 matrix [2].

Because of the high density of the samples, the pores were not observed from the micrographs given in Figure 2. SiC particles in the powder composite hindered the α-β-Si_3N_4 phase transformation and the growth of β-Si_3N_4 grains into the Si_3N_4 matrix.

Counterpart material	100Cr6
Counterpart hardness	65 HRC
Friction velocity	0.1 m × s^{-1}
Normal load	60 N
Total sliding distance	120 m
Surrounding atmosphere	ambient air
Lubrication method	none

Table 2. Parameters used for the wear tests

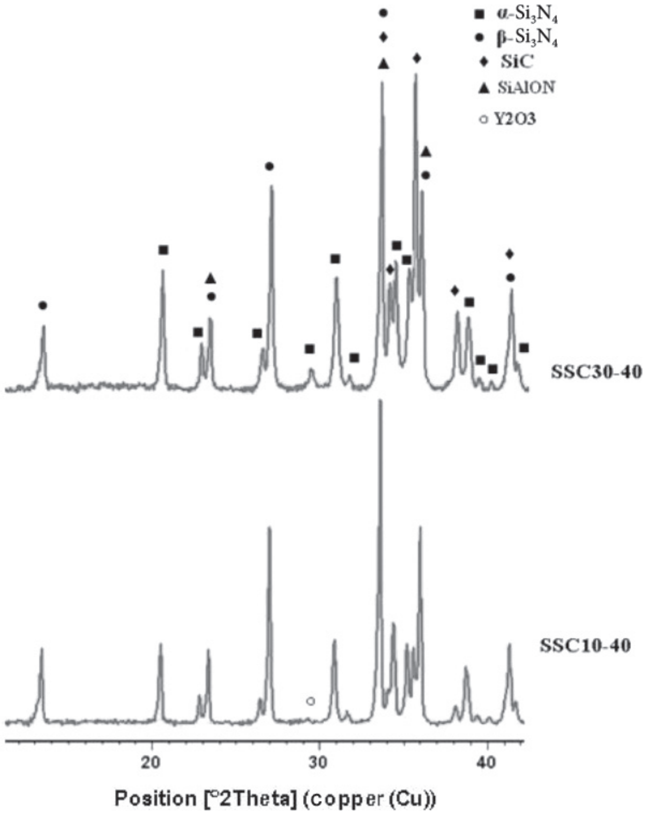

Figure 1. XRD patterns of SSC10 and SSC30 samples obtained by reactive spark plasma sintering at 1650 °C under 40 MPa pressure for 5 minutes in nitrogen atmosphere

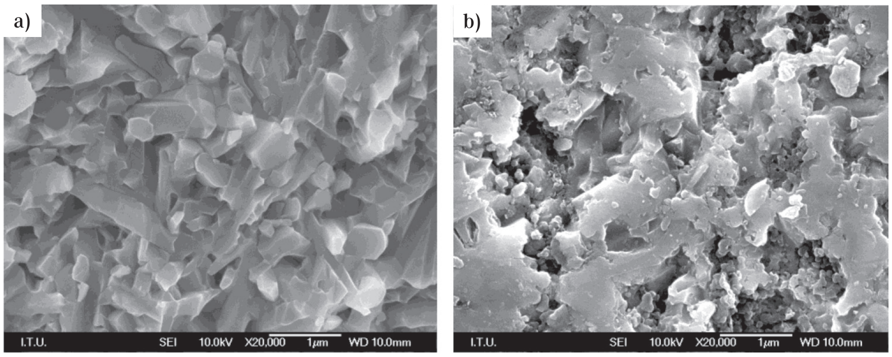

Figure 2. Microstructures of the composites spark plasma sintered at 1650 °C under 40 MPa for 5 minutes, a) SSC10, b) SSC30

Sample	Hardness	Elastic modul, E [GPa]	Theoretical density [%]	Density [g × cm^{-3}]	Bending strength [MPa]	Fracture toughness [MPa × $m^{1/2}$]
SSC10	21.4	335	99.6	3.28	317	5.8
SSC30	22.5	379	99.3	3.27	367	6.0

Table 3. Mechanical properties of SSC10 and SSC30 composites sintered at 1650 °C under 40 MPa for 5 minutes

The friction coefficients of Si_3N_4/SiC composites against 100Cr6 steel ball as a function of distance were shown in Figure 3. The SSC30 ceramic composite showed a lower friction coefficient than SSC10 ceramic composite. The friction coefficient values of SSC10 were nearly the twice of the friction coefficient values of SSC30. The addition of SiC particles to Si_3N_4-based composites showed a lower friction coefficient during sliding.

The wear occurs by microfracture under dry conditions. The tribochemical wear mechanism was often accompanied by material transfer and adhesion, mostly from softer steel to the ceramic specimen. The presence of tribochemical films on ceramic and metals under sliding or fretting conditions were reported many times [3, 4, 6, 8, 15]. Although their strong dependence on environmental and operating conditions was confirmed, details of the mechanisms and chemical reactions are still not understood. The situation is complicated by the many influential parameters and variations in test conditions [17]. The wear of Si_3N_4 as a result of the adhesion with the steel ball followed by the pull-out of the Si_3N_4 grains. The microstructure of silicon nitride ceramics has a significant effect on their mechanical and tribological properties. The grain morphology, in particular, is a significant factor for controlling wear resistance [5]. Under dry frictional conditions, the wear of Si_3N_4 ceramic in the ceramic steel sliding contacts was thought to be mainly caused by the adhesion between the rubbing surfaces and the microfracture of the ceramic [18]. The wear microstructures of the samples SSC10 and SSC30 were shown in Figure 4. The wear tracks in SSC10 specimen were deep, intense and wider compared to SSC30 specimen. As the SiC powder content increased to 30 wt.-%, material removal via delamination was seen to be less.

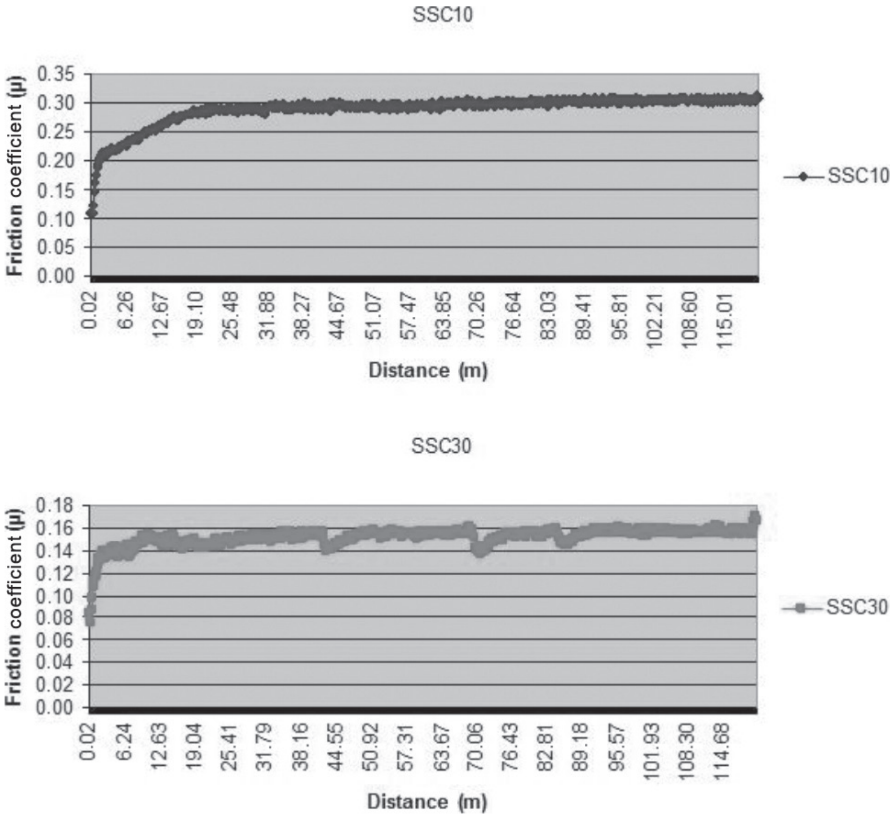

Figure 3. Friction coefficients of Si_3N_4/SiC composites against 100Cr6 steel ball as a function of distance

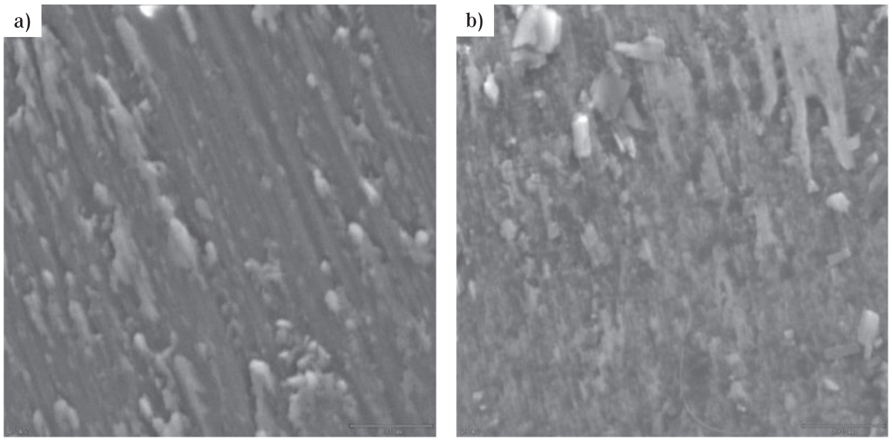

Figure 4. Wear microstructures of the spark plasma sintered samples at 1650 °C under 40 MPa for 5 minutes, a) SSC10, b) SSC30

Conclusions

The following conclusions could be listed from the results obtained in this study:

- Because of the reinforcement effects of SiC, the obtained composites showed increased mechanical properties such as high hardness and fracture toughness. The samples had relative densities higher than 98 %. α-Si_3N_4, β-Si_3N_4, SiC and SiAlON were the main phases. SiC particles in the powder composite hindered the α-β-Si_3N_4 phase transformation and the growth of β-Si_3N_4 grains into the Si_3N_4 matrix.
- Si_3N_4-based composites were characterized by a relatively low friction coefficient in unlubricated sliding against 100Cr6 steel ball.
- The adding of SiC particles increased the fracture toughness of Si_3N_4-based composites. The fracture toughness of Si_3N_4 played an important role in their wear behavior. The addition of SiC particles to Si_3N_4-based composite increased its wear resistance.
- The wear tracks in SSC10 specimen were deep, intense and wider compared to SSC30 specimen. As the SiC powder content increased to 30 wt.-%, material removal via delamination was seen to be less.

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Abstract

Trockengleit-Verschleißmechanismus von funkenplasmagesinterten Si_3N_4-SiC-Kompositen gegenüber Stahl. In den diesem Beitrag zugrundeliegenden Forschungsarbeiten wurden Si_3N_4-basierte Kompositwerkstoffe bei einem relativ niedrigem Reibungskoeffizient unter ungeschmiertem Gleiten gegen einen Ball aus 100Cr6 Stahl charakterisiert. Die Si_3N_4-basierten Komposite mit verschiedenen Mengen von SiC wurden durch Plasmafunkensintern bei einer Temperatur von 1650 °C für 5 min bei einem einachsigen Druck von 40 MPa in einer Stickstoffatmosphäre hergestellt. In dem Verschleißtest wurden die Last, der Gesamtabstand und die Rotationsgeschwindigkeit mit 60 N, 120 m und 500 U × min^{-1} gewählt. Die Verschleißspuren wurden mittels Elektronenmikroskopie untersucht, um den Verschleißmechanismus abzuschätzen. Die Ergebnisse wurden mittels eines Reibungskoeffizient-Entferungs-Diagramms ausgewertet. Die Si_3N_4-basierten Komposite zeigten eine signifikante Abnahme des Verschleißes mit verbesserter Zähigkeit bei einer Verstärkung mit SiC-Pulver.

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The Author of This Contribution

Dr. Zeynep Taslicukur Ozturk, born in 1981, received her PhD in 2010 from Yildiz Technical University, Istanbul, Turkey. Presently, she works as Assistant Professor in the Department of Metallurgical and Materials Engineering, Gedik University, Istanbul, Turkey. Her areas of interest focus on the synthesis of high-tech ceramics.