Friction and Wear of Silicon Carbide at Elevated Temperatures

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炭化ケイ素の高温摩擦摩耗特性

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Friction and wear tests of sintered α -SiC were conducted from room temperature to 1200°C both in air and vacuum. The coefficient of friction was larger in air than that in vacuum but was almost independent of the temperature. The specific wear loss generally increased with an increase of temperature. However, a substantial decrease in wear loss was observed at the highest temperature for lower contact pressure tests where a thick layer of small particles was formed on the friction surface. Silicon oxide was identified in wear debris particles particularly collected from high-temperature tests. It suggests that oxidation, which is accelerated at high temperatures, generally promotes material removal, resulting in a higher wear loss. However, the wear loss could decrease when oxide-containing particles form a thick surface layer.

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1. Introduction

Silicon carbide (SiC) has high hardness and excellent chemical and mechanical properties at elevated temperatures and is therefore expected to be a candidate for tribological applications in severe and/or high-temperature environments. Potential applications include industrial nozzles, pump seals and heat engine parts.¹⁾ Further engineering applications as a corrosion or abrasion resistant material at high temperatures are also promising.²⁾

Fundamental understanding of the friction and wear mechanisms and the effect of environmental conditions such as temperature and atmosphere is essential requirement for the selection or design of a ceramic material for practical applications. Several researchers have addressed tribological characteristics of SiC/SiC pairs³⁾⁻¹⁹⁾ or SiC/other ceramics pairs.^{20),21)} Tribochemical reactions, including oxidation and decomposition of SiC, become evident at high temperatures and may have a critical effect on the tribological behaviors. It has been reported that oxidation accelerates material removal at the friction surface,^{6),8)} while it improves wear resistance for some particular cases.^{6),7)} Decomposition of SiC usually occurs in inert or vacuum environment. Graphite layer of the decomposed carbon acts as a solid self-lubricant between the contact surfaces of SiC.⁹⁾ Most of the experimental studies on the tribology of SiC were conducted at ambient or moderate temperatures. Although several studies have been reported for high temperature tribology,⁵⁾⁻¹⁰⁾ available results on the effect of temperature and atmosphere are still limited.

In the present study, a series of friction and wear tests were conducted in a plate-on-plate type test rig using a commercially available SiC ceramics. Detailed analyses were conducted for wear surfaces and debris particles. Results are compared with those in previous studies. Oxidation effects on the friction and wear mechanism are discussed mainly for two different wear modes observed at high temperatures.

2. Experimental procedures

The material used was a sintered silicon carbide (α -SiC, Nikkato, SiC-11) of 98 mass% purity with a density of 3.1

$\times 10^{-3}$ kg/m³. Wear tests of the plate-on-plate type were conducted with a dry friction test rig using a couple consisting of the same material.^{22),23)} The specimen geometry is illustrated in Fig. 1. The apparent contact area was annular-shaped with inner and outer diameters of 12 and 22 mm, respectively. The specimens were ultrasonically cleaned in acetone for 5 min before and after test. The main portion of the test rig is schematically illustrated in Fig. 2. Each specimen was mounted in a superalloy (Hastelloy X) holder. The ring specimen was rotated on the plate specimen under a constant load provided by a servo-controlled pneumatic cylinder. The specimen holders were heated by high-frequency induction coils to the required temperature. For the tests in the vacuum condition, the sealed chamber, enclosing the test section, was evacuated with a rotary vacuum pump to the residual air pressure of 7-8 Pa at room temperature or 10-12 Pa at 1000°C.

Coefficient of friction was measured at room temperature and from 200°C to 1200°C with an interval of 200°C under a constant sliding speed of 0.2 m/s. The contact pressure was provided as 0.4 and 1.2 MPa in step at each temperature. A single pair of specimens was used for all the conditions in each environment.

The wear tests were usually conducted for a sliding distance of 500 m under constant conditions of temperature,

Fig. 1. Specimen geometry: (A) plate specimen and (B) ring specimen.

Fig. 2. Schematic of the main portion of the friction and wear test rig.

contact pressure (0.2 or 0.4 MPa) and sliding speed (mainly 0.2 m/s). The wear loss was measured as a change in mass of the specimens before and after test. Wear volume was calculated as the mass loss divided by the apparent density and then specific wear loss was determined as the wear volume divided by applied load and sliding distance in a dimension of $\text{mm}^3/(\text{N} \cdot \text{m})$. It was given as an average of the values of the plate and ring specimens for each condition. The wear surfaces were observed by scanning electron microscope (SEM). Cross sectional observations by SEM were also conducted on the samples fractured perpendicular to the wear surface. Transmission electron microscopy (TEM), X-ray diffractometry (XRD) and X-ray photoelec-

tron spectroscopy (XPS) were performed on the debris particles. Cross sectional region of the vicinity of the wear surface was analyzed with an electron-probe microanalyzer (EPMA).

3. Results and discussion

Figure 3 shows relationship between the coefficient of friction and temperature. The coefficient of friction varied between 0.5 and 0.8 except for a few particular cases. In general, the coefficient of friction in vacuum was smaller than that in air. Under the contact pressure of 1.2 MPa the coefficient of friction was nearly constant from room temperature to 1200°C. In air, the coefficient of friction was very low at room temperature when the applied load was 0.4 MPa. In ambient atmosphere, absorbed gas or humidity often promotes lubrication effects by some tribochemical reaction products when the applied load is not so high as severe wear taking place. Although it has been reported that graphite forms by decomposition of SiC resulting in a reduced coefficient of friction, such concentration of carbon was not detected in this study. Graphite would be more readily present on the wear surface in vacuum. However, no significant decrease in coefficient of friction has been observed in vacuum.

The specific wear loss at various temperatures is shown in Fig. 4. It is in an order of $10^{-5} \text{ mm}^3/(\text{N} \cdot \text{m})$, comparable to other ceramics including Al_2O_3 and TiB_2 .^{22),24)} For the

(A)

(B)

Fig. 3. Coefficient of friction at various temperatures: (A) under contact pressure of 0.4 MPa and (B) 1.2 MPa.

(A)

(B)

Fig. 4. Specific wear loss at various temperatures: (A) under contact pressure of 0.2 MPa and (B) 0.4 MPa.

Fig. 5. SEM micrographs of the wear surface tested in air:

(A) plan view at room temperature under 0.4 MPa, (B) its cross section, (C) plan view at 1200°C under 0.4 MPa, (D) its cross section, (E) plan view at 1200°C under 0.2 MPa and (F) its cross section.

contact pressure of 0.2 MPa specific wear loss increased significantly above 400°C but decreased at 1200°C in air. In vacuum, the specific wear loss exhibited a nearly constant value from room temperature to 800°C, and then decreased at 1000°C. When the contact pressure increased to 0.4 MPa, the wear loss continuously increased above 800°C. The general tendency is that specific wear loss increases with an increase of temperature, which is consistent with the results previously reported by Woydt and Habig,⁶ Wang et al.⁸ and Mizutani et al.⁹ However, there are two exceptions against this general tendency; namely 0.2 MPa at 1000°C in vacuum and 0.2 MPa at 1200°C in air.

Figure 5 shows micrographs of the wear surface regions in air. At room temperature, the wear surface was covered by fine particles of debris as shown in Fig. 5(A). Transgranular cracks parallel to the wear surface were often observed in cross-sectional views as shown in Fig. 5(B), suggesting that brittle microfracture is the dominant material-removal mechanism. At 1200°C, the surface morphology was dependent on the contact pressure, corresponding to the difference in the wear loss. Figures 5(C) and 5(D) reveal that numerous intergranular cracks were developed to the depth of approximately 10 μm in the surface under the contact pressure of 0.4 MPa where the wear was in the severer mode. In contrast, a thick surface layer of fine particles was observed under 0.2 MPa where the wear loss significantly decreased, as shown in Figs. 5(E) and 5(F).

In vacuum, similar difference in wear surface morphology was observed at 1000°C as shown in Fig. 6. For a contact pressure of 0.2 MPa, fine particles covered the wear surface and a very thin glassy layer without visible microcracking was formed in the cross sectional view. Although similar surface layer of fine particles was observed on the wear surface under a contact pressure of 0.4 MPa, delamination of the surface layer from the original bulk was readily observed. It should be noted that a thick layer consisting of small particles is always observed in the surface of the specimens in association with reduced wear loss at high temperatures.

XRD patterns of debris collected from the wear tests are

Fig. 6. Cross sectional view of the wear surface tested at 1000°C in vacuum: (A) under 0.2 MPa and (D) 0.4 MPa.

summarized in Fig. 7 in comparison with the original bulk material in which only α -SiC phase was identified. Diffraction patterns of wear debris exhibited a few broad peaks both in air and in vacuum. Silicon oxide, probably in amorphous state, was present when tested in air, especially at 600°C and above, while it was not clearly observed in vacuum. TEM observations of debris are shown in Fig. 8. The debris particles were in an order of 0.1 μm in size. Electron diffraction pattern of the core region of a debris particle, as shown in Fig. 8(A), was in agreement with 6H-SiC crystal

Fig. 7. XRD patterns of debris collected from the tests at various temperatures: (A) in air and (B) in vacuum.

(α -SiC). However, diffraction pattern from a surface region of the particle as shown in Fig. 8(B) exhibited amorphous features. Results of the XPS analyses are summarized in Fig. 9. Only SiC bond (100.5 eV) was observed for the surface of the bulk sample before the wear test (Fig. 9(A)). However, debris particles from the wear tests at 600°C exhibited both SiC and SiO_2 (103.5 eV) bondings (Fig. 9(B)).

A reasonable interpretation of these analyses is as follows: Debris particles are partially oxidized and consist of very fine SiC crystalline in the core region and SiO_2 amorphous coatings in the surface. The SiC crystallite size is very small and therefore XRD peaks are very broad. In vacuum, oxide formation was observed by XPS (Fig. 9(C)),

Fig. 8. Transmission electron microscope photographs of the wear debris particles collected from the test at 600°C in vacuum.

but not by XRD (Fig. 7). This difference suggests that the oxidation slightly occurs even in vacuum because of the rather low vacuum of only rotary pump level. It is also substantiated by the observations of amorphous surface region by TEM.

SiC is normally very brittle and plastic deformation of SiC is restricted. Microstructural features also suggest that microfracture is the most probable mechanism for material removal even at high temperatures. It is reasonable to consider that oxidation promotes material removal because the wear loss in air was generally higher than that in vacuum as shown in Fig. 4. Since oxidation becomes more profound at high temperatures, the increase in the wear loss at high temperatures is also attributed to oxidation effects. Oxidation of SiC ceramics is usually evident at temperatures above 900°C in air, forming a dense oxide product which protects SiC from further oxidation. Since frictional contact provides additional heat, oxidation could be observed even in the wear surfaces tested at room temperature.²⁴⁾ Furthermore, repetitive contact would readily remove the protective oxide film which usually forms on the SiC bulk surface in case of static oxidation.

Woydt and Habig,⁶⁾ and Wang et al.⁸⁾ consistently suggested that oxidation could promote material removal because oxidation product, that is SiO_2 , would be much softer than the original SiC material. However, detailed analyses in this study reveal that debris particles consist of SiC core and SiO_2 coatings. This implies that the oxidation occurred after debris generation rather than that the material removal took place following to the initial oxidation of the bulk surface. On the other hand, it has been reported that the presence of oxygen could cause grain boundary oxidation.²⁵⁾ If so, then it may accelerate the intergranular crack growth of SiC at high temperatures, resulting in an in-

Fig. 9. X-ray photoelectron spectroscopy of SiC: (A) bulk surface before the wear test, (B) debris particles at 600°C in air, and (C) debris particles at 600°C in vacuum.

crease in the wear loss by intergranular microfracture. This effect seems more critical to the increase in the wear loss at elevated temperatures.

In contrast, under a contact pressure of 0.2 MPa, a significant decrease in wear loss was observed at the highest temperature, against the general tendency that the wear loss increases with an increase of temperature. SEM observations revealed that a thick layer containing very fine particles consistently formed on the wear surfaces. Figure 10 shows elemental concentration maps by EPMA of the cross section of the wear surface under 0.2 MPa at 1000°C in vacuum. Oxygen was substantially concentrated in the surface layer.

Similar wear loss decrease associated with a surface layer formation has been reported for alumina and mullite ceramics.^{23),26)–28)} It has been concluded that plastic deformation accompanied by dynamic recrystallization during the sliding wear forms the fine-grain-size surface layer and that it acts as solid lubrication layer due to its low deformation resistance.^{23),29)} The surface layer of SiC however contains substantial amount of oxygen suggesting that the layer consists of partially oxidized wear debris instead of recrystallized fine grains. Regardless of the formation mechanism, a layer of fine-grain-size particles could have low deformation resistance by grain boundary slide or rotation mechanisms. Soft SiO₂ coating of the particle may en-

Fig. 10. Electron-probe microanalysis of the cross section of the wear surface region at 1000°C in vacuum: (A) silicon and (B) oxygen. In the top layer, O is richer but Si is less rich.

Fig. 11. Effect of sliding speed on specific wear loss of SiC at 1000°C in air under a contact pressure of 0.4 MPa.

hance the deformability. If a soft layer stays on the hard substrate, wear can be reduced by a solid lubrication effect of the layer. For higher contact pressure case, this type of layer could not be formed stably because such layer was easily

removed away from the wear surface resulting in a higher wear loss.

Figure 11 depicts the effect of sliding speed on the wear loss of SiC at 1000°C in air. The specific wear loss simply decreased with an increase of sliding speed. At higher sliding speed tests, a thick layer of fine-grain-size particles were observed at the wear surface. It is not clear whether this was directly related to the effect of sliding speed itself or that of temperature because local surface temperature also depends on the sliding speed. Although the sliding conditions required for this low wear mode are not well defined, lower contact pressure (or applied load) and higher sliding speed are the preferable conditions for contact with low wear loss at high temperatures.

4. Conclusions

Friction and wear tests of a sintered SiC ceramic were conducted from room temperature to 1200°C. Effects of temperature, atmosphere and test parameters on the tribological characteristics of SiC were investigated. The coefficient of friction was not much dependent on temperature, especially under a higher contact pressure. The coefficient of friction in air was larger than that in vacuum. Specific wear loss generally increased with an increase of temperature, and was higher in air than in vacuum. Partial oxidation was evident at 600°C and above from the detailed analyses of wear debris. These results suggest that oxidation generally accelerates intergranular microcracking and increases the wear loss. However, there could be a particular case that the wear loss significantly decreases when the contact pressure is low and the sliding speed is high, where thick surface layer of oxide-containing particles is formed.

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