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Some tribological properties of a carbon-derived Si_3N_4/SiC nanocomposite

M. Kašiarová[a,*], E. Rudnayová[a], J. Dusza[a], M. Hnatko[b], P. Šajgalík[b], A. Merstallinger[c], L. Kuzsella[d]

[a]Institute of Materials Research, Slovak Academy of Science, Košice, Slovakia
[b]Institute of Inorganic Chemistry, Slovak Academy of Science, Bratislava, Slovakia
[c]ARC Seibersdorf Research GmbH, Austria
[d]University of Miskolc, Material Science Institute, Hungary

Abstract

Some tribological properties of a recently developed carbon derived Si_3N_4/SiC nanocomposite have been investigated in the contribution. The material was tested in the pin-on-disc configuration, with Si_3N_4 and diamond pins, under unlubricated sliding conditions. The investigated variables were load, sliding distance, and humidity. The measured friction coefficients at 50% humidity and by using a Si_3N_4 pin were in the range of 0.6–0.7 and 0.05–0.07 when using a diamond pin. The value of this coefficient in vacuum was of about 0.6. A reduction of humidity resulted in lower friction coefficients, but the wear resistance in vacuum was significantly lower than that in air. Increasing the testing temperature (up to 500 °C) increased both, the friction and wear coefficient values. The wear patterns on the Si_3N_4 + SiC disc in the pin-on-disc test with Si_3N_4 ball revealed presence of the tribochemical reaction layers formed by SiO_2. Mechanical fracture was the wear controlling mechanism in all tested conditions.

Keywords: Friction coefficient; Nanocomposites; Si_3N_4; SiC; Wear resistance

1. Introduction

Advanced ceramic materials have been expected to be a kind of promising wear-resistant tribo-material for parts of machines and devices for high temperature applications. They have better hardness, corrosion and high temperature resistance than metallic materials. Silicon nitride is one of the most important engineering material because of its good mechanical properties, good oxidation resistance, low coefficient of thermal expansion, high hardness and good refractoriness. In order to improve mechanical properties of the monolithic Si_3N_4 ceramics, second phases like platelets, whiskers, or particles are added into the Si_3N_4 matrix. Previous studies of Si_3N_4/SiC materials revealed an improvement of their tribological (e.g., SiC platelets)[1] and high temperature properties[2,3] in comparison to the monolithic Si_3N_4. A wider application of this material is limited owing to it's high cost, therefore the recent activities are aimed to introduce new preparation methods, e.g., by substituting the expensive amorphous Si–C–N powder with cheap carbon and SiO_2. Introduction of SiC nanoparticles into silicon nitride matrix improves hardness, high-temperature strength and creep resistance. Tribological investigation of ceramics is a relatively young interdisciplinary science and there exist numerous parameters with unknown effect on the friction and wear. The major parameters given in most publications are sliding velocity, condition of the contact, load, relative humidity, material composition, surface roughness. Tribological behavior of the monolithic silicon nitride is well known for a wide range of sliding speeds (0.001–5 m/s),[4] temperatures (20–1000 °C)[5] and different environment.[6] In many works[1] the following wear behavior controlling mechanisms[4] are reported:

- mechanical or thermal induced microcracking due to fatigue assisted stresses resulting from friction at high normal loads;

* Corresponding author.
E-mail address: kasiarova@imrnov.sk (M. Kašiarová).

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- tribochemical reaction with water vapour at low temperatures and normal loads (<400 °C, <10 N); and
- tribooxidation at temperatures that depend on the composition and nature of the intergranular phases.

Hsu et al.[7] have shown that the wear rate can differ by several orders of magnitude for the same material when tested under different experimental conditions. Thus, it is very important to compare results obtained at the same experimental conditions. There is a lack of information about the effect of SiC particles on the tribological performance of Si_3N_4.

The purpose of this study is to investigate the influence of some parameters (sliding distance, load, environment, humidity and testing temperature) on the wear behaviour of Si_3N_4/SiC nanocomposite.

2. Experimental procedure

2.1. Preparation of the experimental material

The studied material was prepared at the Institute of Inorganic Chemistry of the Slovak Academy of Sciences by hot-pressing at 1750 °C for 2 h under a specific heating regime, atmosphere, and mechanical pressure in the form of discs with the diameter of 50 mm and thickness of 5 mm.[8] The used heating regime caused that the core of specimens contained unreacted carbon. It was suggested that carbon would improve friction properties of the material. So, two differently colored areas were present on the ground surfaces of the discs, the outer–lighter area and the inner–dark area.

2.2. The methods of investigation

The tests were conducted on the ground surfaces of discs on pin-on-disc tribometer (UVH) at room temperature without any lubricant. They were performed in air with a relative humidity in the range from 10 to 80%, or in the vacuum. The applied loads were 5, 10 and 15 N, sliding distances 600, 900, 1200, 1500 m and the sliding velocity was 0.1 m/s. Wear tests at higher temperatures (50, 150, 300, and 500 °C) were also made. The surface finish was characterized prior to testing by roughness R_a values ($R_a = 0.2$). The wear volume on each flat specimen was calculated from the surface profile traces (usually 4) across the track and perpendicular to the sliding direction. These data were then used to calculate the wear coefficient k, which is a dimensionless quantity defined as the wear volume W_v per sliding distance s per normal load F_N.

$$k = \frac{W_V}{sF_N}$$

The wear tracks were examined by scanning electron microscopy (SEM) to elucidate the wear process. Energy disperse spectroscopy (EDX) was used to analyze the wear track and the wear debris. The specimens were platinum coated to prevent charging during examination in SEM. A typical microstructure of the studied materials is shown in Fig. 1. Microstructure consists of hexagonal β-Si_3N_4 grains (dark), SiC nanoparticles (light, spherical), and intergranular phase (light). The nanoparticles located on the interface boundary were difficult to identify, so their volume fraction is included into the volume fraction of intergranular phases. Basic mechanical properties of the studied nanocomposite are reported in Table 1.

3. Results and their discussion

3.1. Tribological properties

Frictional behavior of the inner and outer area of specimen is shown in Figs. 2 and 3. A quick rise of the friction coefficient to the peak value, followed by a decrease to the constant value was observed in the inner area of specimen. The coefficient of friction in outer region of the sample shows a more gradual increase to a nearly constant value. We attribute the initial friction coefficient values to the contamination, which exists on the ceramic surface despite of the performed cleaning, or to the high value of surface roughness. The work of

Table 1
Mechanical properties of the nanocomposite

Mechanical properties	
Hardness	HV10 = 1706
Fracture toughness	K_{IC} (IF) = 4 MPa/m$^{1/2}$
Bending strength	σ_f = 497 MPa
Young's modulus	E = 329 GPa

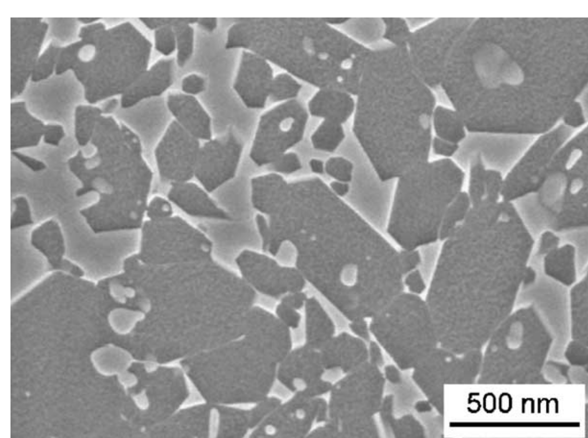

Fig. 1. Typical microstructure of Si_3N_4/SiC nanocomposite in inner region of sample.

Fisher[9] reported the dependence of the friction coefficient on surface roughness but at small sliding distances, only. Stable high values of the friction coefficient in Fisher's work were reached after sliding distances of a few meters, a distance much to short to have a practical significance. The steady state value of friction coefficient of Si_3N_4/SiC nanocomposite was observed after 150 m approximately, and this distance was decreasing with the increasing load. Therefore the chosen sliding distance between 600 and 1500 m was sufficient for estimation a real friction coefficient.

Fig. 4 reported that the course of the friction coefficient is independent on the sliding distance, while the course of wear coefficients in the inner and outer areas depends on it. The inner region of the studied material has shown that after an initial steep increasing, constant wear rate values were measured. The wear rate in the outer region decreased with the increasing of sliding distance. This can be explained by a reattachment of some of the wear debris to form the so-called "transfer film".

Influence of the applied load at a constant sliding speed does not reveal any significant change of the friction coefficient value (Fig. 5). Wear in the outer region is independent on the applied load. An increase of the wear rate with increasing of the applied load was observed in the inner region of samples. The mutual relationship between the coefficient of friction and the wear rate in vacuum, Fig. 5 has a different course. When increasing the applied load the friction coefficient and wear rate increased, too. The coefficient of wear in vacuum is one order higher than in air, the friction coefficients are lower when compared to those in air. When comparing friction coefficients of ceramics with those of metals, the ceramics could be assumed to be better than that of metals.

The influence of relative humidity on the tribological characteristics is on Fig. 6. Increasing the relative humidity caused a smooth increase of the coefficient of friction and a steep increase of the wear rate in the inner region of sample. However, the magnitude of wear rate is still smaller than the wear rate in vacuum (zero point on the x-axis). The influence of humidity (above 60%) in the outer area is enhanced in comparison with the inner area. A steep increase of the coefficient of friction and wear rate with increasing the humidity was observed. The humidity is a parameter, which has a strong influence on wear and friction. The obtained results are different than those of the most of reports, e.g. Ref. 9. It is generally assumed that water vapor interacts with the material on the wear interface and forms stable coherent interfacial films which protect the wear surfaces and reduce the wear rate. In our case, the reaction product can be easily removed from the wear interface at the applied load.

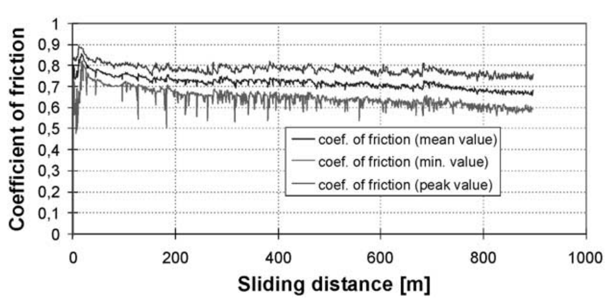

Fig. 2. Change of the friction coefficient with sliding distance in the inner region.

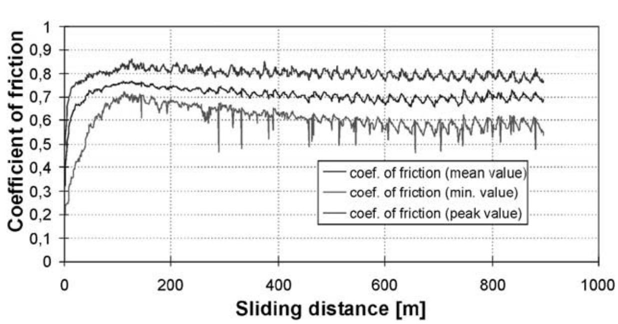

Fig. 3. Change of the friction coefficient with sliding distance in the outer region.

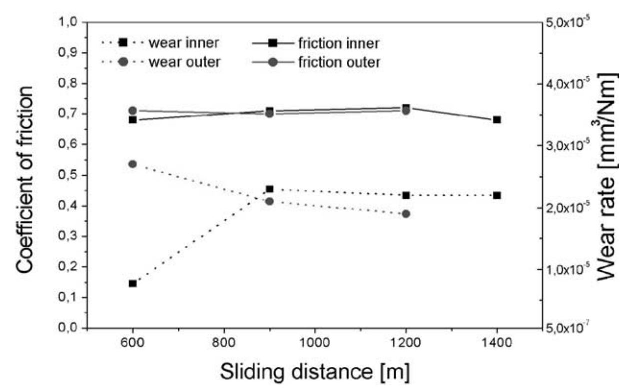

Fig. 4. Dependence of the friction coefficient and wear rate on sliding distances in the inner and outer area of the specimen.

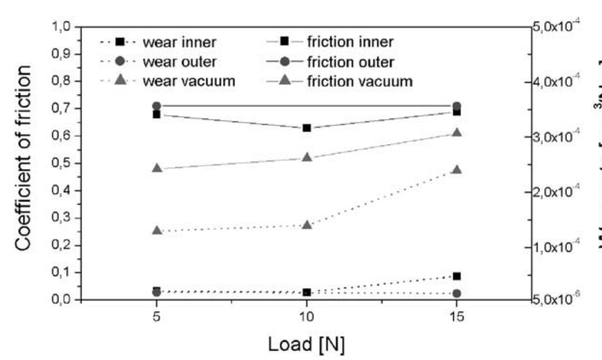

Fig. 5. Tribological characteristics in the inner and outer region at 50% humidity, and in the outer region in vacuum.

Microscopic examination of the friction track revealed the presence of debris within the wear track. Two types of debris could be identified: free individual rounded particles and more or less dense agglomerates forming flakes or platelets. Figs. 7 and 8 compare the friction tracks in the inner region of specimens loaded with 15 N, sliding distance of 900 m and humidity 55% (Fig. 7) and 81% (Fig. 8).

The EDX analysis revealed that debris are predominantly formed by SiO_2. However, the formation of SiO_2 debris was observed also in vacuum, what indicates that the product of wear is formed from the intergranular Si_2N_2O phases, predominantly. Thus, the wear in our case is predominantly mechanical damage of the worn surface and to a lesser extent caused by tribochemical reactions.

3.2. Tribological characteristics at higher temperatures

Tribological tests made at higher temperatures revealed (Fig. 9) that up to 150 °C the friction coefficient had increasing values, at higher temperatures (up to 500 °C) has an almost constant value. The wear rate increased with the testing temperature quite monotonously. The fact that the friction coefficient grows already at temperatures much lower than the temperature of glassy phase softening can be explained by the fact that due to friction the surface temperature in the contact area of the ball is significantly higher than that in the bulk of material.

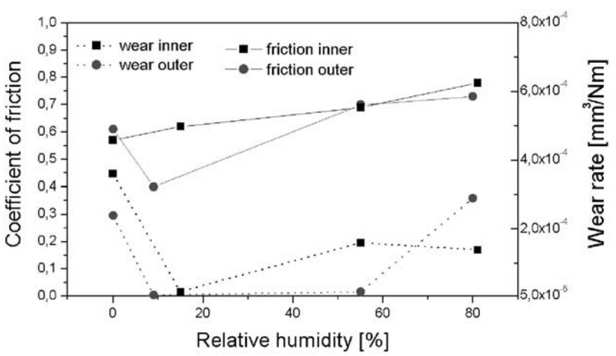

Fig. 6. The influence of relative humidity (zero point on the *X*-axis) on the coefficient of friction and wear rate.

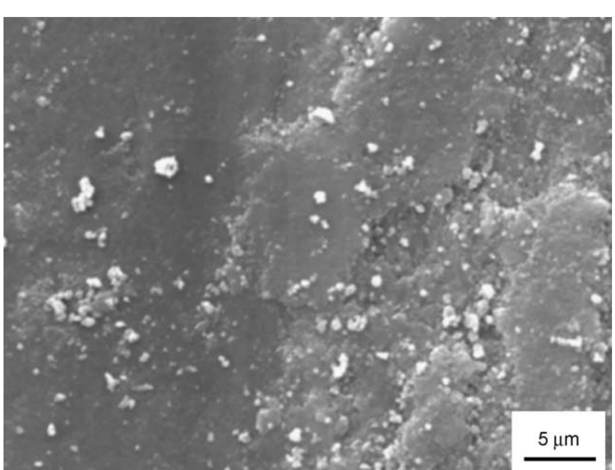

Fig. 7. Wear trace in the inner region of specimen at 0.1 m/s, 15 N, 900 m and relative humidity 51%.

4. Conclusions

Influence of the tribological parameters: sliding distance, load, humidity, and testing temperature on the resulting tribological parameters of the studied nanocomposite was determined.

From the achieved results it follows that:

- an influence of the friction coefficient on the sliding distance was not revealed and that its

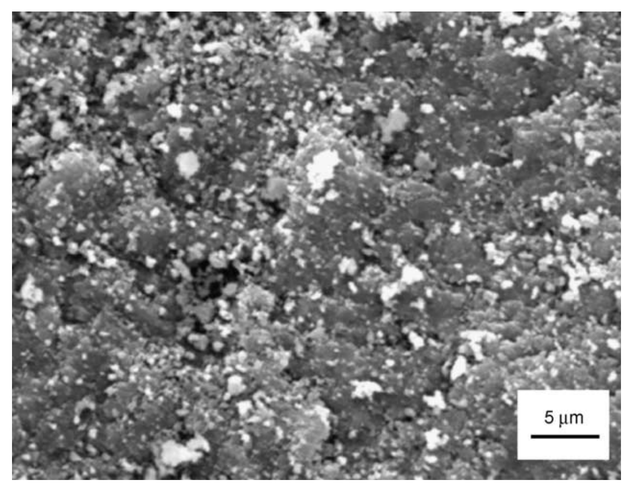

Fig. 8. Wear trace in the inner region of specimen at 0.1 m/s, 15 N, 900 m and relative humidity 81%.

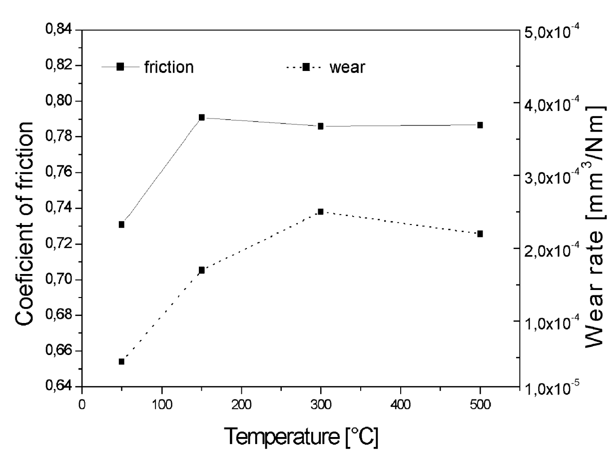

Fig. 9. The dependence of the friction coefficient and the wear rate on testing temperature.

influence on the wear rate in the outer and inner part of the specimen were different;

- it was found that the applied load did not influence the friction coefficient values, but it influenced the formation of debris and thus the wear rate;
- the relative humidity is a parameter which influences the friction coefficient as well as the wear rate very significantly;
- the prevailing wear mechanism was the mechanical fracture on the material surface; and
- increasing the testing temperature slightly increases both, the friction coefficient and the wear rate.

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