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The role of products of tribochemical reactions on the friction and wear properties of alumina/silicon carbide couple sliding in HFC-134a gas

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Abstract

By using a ball-on-disk type tribometer installed in a high vacuum chamber, tribological behaviour of a silicon carbide (SiC) disk sliding against an alumina (Al₂O₃) ball in CF₃CH₂F (HFC-134a) gas at pressures between 170 and 10⁵ Pa were investigated. For comparison, friction tests were also carried out in a vacuum (10^{-5} Pa) under the same experimental conditions. Surface analysis of the wear tracks on the disks was conducted using X-ray photoelectron spectroscopy (XPS) without exposure to air. It was found that there was a significant pressure influence on both the tribological properties and products of tribochemical reactions. When the pressure increased from vacuum to 10^{5} Pa, the friction and wear dramatically decreased, whereas the amount of carbon-containing compounds and fluorides on the wear track rapidly increased. The more vigorous the tribochemical reaction was, the lower were the wear rates, however, friction coefficient was dependent on the composition of the products of the tribochemical reactions. The metal fluorides, organic compounds formed were effective for reducing friction. On the contrary, the $-Si-F_x$ containing compounds formed resulted in an increase in friction. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Ceramics; Silicon carbide; Alumina; Fluorine-containing gas; Gas lubrication; Tribochemical reactions; Chemical wear

1. Introduction

Silicon carbide (SiC) is one of the most important advanced ceramics for engineering applications because of its high strength and excellent resistance to creep, and relative chemical inertness. It has been used in gas turbine blades and in turbine ceramic seals extensively [1–3]. However, experiments have showed that adhesion and friction properties of SiC depend strongly on its surface characteristics. For example, desorption of carbon contaminants from a SiC surface results in an appreciable increase in friction, and graphitisation of the surface at high temperatures associates with lower abrasion and friction [4,5].

The influence of the surrounding environments on tribological characteristics of SiC have also been investigated [6–13]. Studt [9,10] has studied the tribosystem of SiC self-mated couple lubricated with hexadecane and fatty acids (zinc dialkyl dithiophosphates with different chain length used as additives), and found that partial and complete oxidation products are formed on the SiC surface. Other researchers [11–13] have also reported that the products of tribochemical reactions of amorphous SiO_2 and $Si(OH)_4$ are produced on the SiC surface rubbing in water, and the wear rate of SiC is determined by its oxidation and hydration reaction rates. In addition, it has been found that the dependence of the friction on the adsorption or reaction layers on the SiC surfaces is more complex than that of the other ceramics, and these results are due to the different crystalline structures and atomic bonds in the ceramics.

With the development in science and technology, there is an increasing interest in use of different ceramics in a variety of applications including ball bearings and cyrogenic refrigerators, in which ceramics slide against themselves or other materials under different gaseous environments [3,14,15]. Therefore, there is an increase in the need for understanding the role of tribochemical reactions on the friction and wear properties of these friction systems. In our recent studies [16,17], we have found that fluorination reactions at the frictional interface of oxide ceramic pairs of Al₂O₃/Al₂O₃ and Al₂O₃/ZrO₂ occur when they are rubbing in CF₃CH₂F (HFC-134a) gas, and moreover, only tribochemical reactions at a moderate degree are good for obtaining both of low friction and wear. In this paper, the covalent bond type ceramic of SiC is selected as disk specimen. Friction and wear properties of a SiC disk sliding against an Al₂O₃ ball

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in HFC-134a at different pressures are investigated. X-ray photoelectron spectroscopy (XPS) is used to identify surface composition. The objectives of the present investigation are to clarify the possibility of vapour phase lubrication of Al_2O_3/SiC couple, and to understand the role of products of tribochemical reactions on tribological characteristics of covalent bond type ceramics. This knowledge will be useful for their applications in more extensive fields.

2. Experimental details

2.1. Materials and gaseous lubricant

Disk and ball specimens were SiC and Al₂O₃, respectively. The disk was made from hot pressed SiC, and its sizes were 25 mm in diameter and 7.9 mm in thickness. The ball with a diameter of 6.35 mm was a commercial product. Physical and mechanical properties of the specimens are shown in Table 1. The specimens were rinsed in acetone for 10 min, and then in hexane for 10 min by using an ultrasonic bath before friction tests. Element composition of the specimen surfaces after cleaning process is summarised in Table 2. A great amount of carbon, which can be attributed to organic contaminants, is detected on the Al₂O₃ surface even after cleaning. Moreover, a small amount of Al and N elements, and a small amount of Si and N elements are detected on the surfaces of SiC disk and Al₂O₃ ball, respectively. These results are due to Al₂O₃ and Si₃N₄, which are often added into ceramics as binders. The 1,1,1,2-tetra-fluoroethane (CF₃CH₂F, HFC-134a), which is

Table 1

Physical and mechanical properties of the specimens

Specimen	Disk	Ball
Material	SiC	Al ₂ O ₃
Density (g/cm ³)	3.21	3.80
Vickers hardness (kg/mm ²)	~ 3100	1650
Surface roughness (µm)	0.33	0.025
Vickers hardness (kg/mm ²) Surface roughness (µm)	~3100 0.33	1650 0.025

Table 2

Elemental	composition	(at.%)	of	the	specimens	surfaces	after	cleaning
process m	easured by X	PS						

Specimen	Element				
	C 1s	O 1s	Al 2p	Si 2p	N 1s
SiC disk	66.3	21.4	1.4	8.8	2.1
Al_2O_3 ball	42.3	38.4	14.6	3.4	1.3

presently used as one of working fluids in refrigerating systems, was selected as gaseous lubricant.

2.2. Friction and wear experiments

Two high vacuum systems were used in this investigation. A ball-on-disk tribometer capable of measuring load and frictional force was installed in the first vacuum chamber (see Fig. 1). After the vacuum chamber was evacuated to less than 10^{-5} Pa, gas with a fixed pressure was introduced through a variable leak valve, and then friction tests were carried out. After the friction test, the vacuum chamber was evacuated to less than 10^{-5} Pa, and the disk was directly moved into the second ultrahigh vacuum system—XPS analysis chamber (see Fig. 1) for surface analysis. When the friction test was carried out in vacuum, the vacuum chamber was evacuated to less than 10^{-5} Pa.

By using a micrometer to elevate friction arm, applied load on the ball specimen at the other end of the friction arm is easily to control accurately. Applied load and frictional force were monitored during the tests by two couples of strain-gage transducers. Rotating speed of the disk was 240 rpm (sliding velocity of about 0.30 m/s), load was 2.5 N. Each test went through 800 m sliding distance. All the friction tests were carried out at room temperature. Two to three repeated tests were conducted under the same conditions.

The surface profile of the wear track on the disks was examined by profilometry. Wear volume of the disk specimens was estimated from the diameter of the wear track and an average cross-sectional area grooved on the wear track



Fig. 1. Schematic diagram of the main component of the experimental apparatus.

obtained from four measurements perpendicular to the sliding direction. The wear volume of the ball specimens was obtained by the spherical cap loss according to an optical micrograph. Specific wear rate is defined as the total volume loss per unit load per unit sliding distance. Average value of friction coefficient and specific wear rate is reported in this paper.

2.3. Surface analysis

Element composition and chemical state analysis were performed using a micro XPS. A monochromatic Al K α (1486.6 eV) X-ray source accompanied with a neutralisation gun was used. Correction for the energy shift due to charging effects was made by assuming the C 1s line from adventitious carbon to be at the binding energy of 285.0 eV. The diameter of the analytical spot was selected smaller than the width of the wear track.

3. Results and discussion

3.1. Friction and wear behaviour

Fig. 2 shows plots of friction coefficient versus sliding distance for the Al₂O₃/SiC pair rubbing under different conditions. In vacuum (10^{-5} Pa), the initial friction coefficient is 0.68, it sharply rises to 0.96 with increasing sliding distance, and then gradually falls to about 0.60 after 800 m sliding. In HFC-134a at 170 and 10^3 Pa, the initial friction coefficients are 0.56 and 0.52, respectively. They sharply rise to 0.94 and 0.70, and then reach their steady-state values of about 0.74 and 0.68 after sliding about 100 m. In HFC-134a at the higher pressures of 10^4 and 10^5 Pa, the friction coefficients with increasing sliding distance is observed. Their steady-state values are about 0.36 and 0.24, respectively. These values

are lower than the friction coefficients in vacuum and in HFC-134a at lower pressures.

From Fig. 2, it can be seen that the initial friction coefficients under different pressures are rather low. This might be due to physisorption and/or chemisorption of molecules on the surfaces. The increase in friction coefficients versus sliding distance in vacuum and in HFC-134a gas at 170 and 10^3 Pa can be due to the removal of surface organic contaminants which have lubricating effect. Lower friction coefficients in HFC-134a at 10^4 and 10^5 Pa might be due to the products of tribochemical reactions formed at the sliding interfaces.

Friction coefficient and specific wear rate of the ball and disk specimens in HFC-134a gas with increasing pressure for 800 m sliding are shown in Fig. 3. It can be seen that the friction coefficient slightly increases, and then sharply reduces when pressure exceeds 10^3 Pa. With increasing pressure, the specific wear rates of the ball and disk tend to reduce. In vacuum, the specific wear rate of Al₂O₃ ball is higher than that of SiC disk, it becomes lower than that of SiC disk when rubs under HFC-134a environment. Compared to the wear in vacuum (10^{-5} Pa), when they are sliding in HFC-134a gas at 10^5 Pa, the specific wear rates of the ball and disk reduce by factor of 1000 and 10, respectively.

We also note that the feature of the wear tracks formed on the SiC disks in vacuum and in HFC-134a is different. A coarse worn surface (see Fig. 4a) with some wear debris was observed while the couple sliding in vacuum. In contrast, relatively smooth surfaces (see Fig. 4b) with very little debris were observed in HFC-134a at 10^4 and 10^5 Pa.

3.2. Results of surface analysis

Table 3 gives the atomic concentration of the elements inside the wear tracks on SiC disks. For comparison, the atomic concentration of outside the wear track under HFC-134a gas at 10^5 Pa is also shown. No F element is



Fig. 2. Friction coefficient of Al_2O_3/SiC couple in vacuum and in HFC-134a gas at different pressures plotted as a function of sliding distance (load: 2.5 N, sliding velocity: 0.30 m/s).



Fig. 3. Friction coefficient and specific wear rate of Al₂O₃/SiC couple sliding in HFC-134a as a function of gas pressure (load: 2.5 N, sliding velocity: 0.30 m/s, sliding distance: 800 m).

detected on the wear track in vacuum, and only few F element exists on the outside wear track even the disk exposure to the gas at the highest pressure of 10^5 Pa. Correspondingly, a great amount of F element is detected on all the wear tracks formed under HFC-134a gas. With an increase in pressure, the atomic concentration of C 1s increases. These results can be due to tribochemical reaction between Al_2O_3/SiC ceramics and HFC-134a molecules at the frictional interface.

Fig. 5 presents XPS spectra of C 1s obtained from narrow scans inside the wear tracks in vacuum and in HFC-134a gas at different pressures after 800 m sliding. The XPS C 1s spectrum of the outside wear track under HFC-134a gas



Fig. 4. Typical surface profile of the wear tracks on SiC disks after sliding in (a) vacuum and (b) HFC-134a gas (load: 2.5 N, sliding velocity: 0.30 m/s, sliding distance: 800 m).

Table 3 Main elemental composition (at.%) of the wear tracks on SiC disks measured by XPS

Pressure of HFC-134a gas (Pa	C 1s	O 1s	Al 2p	Si 2p	N 1s	F 1s
Vacuum	<u> </u>					
10^{-5}	27.2	40.0	20.8	11.4	0.6	_
170	33.8	28.6	3.2	23.1	0.8	10.5
10 ³	45.8	17.1	0.9	28.4	0.6	7.2
10^{4}	55.8	17.4	2.0	14.8	1.3	8.7
10 ⁵	65.1	11.0	1.8	13.2	0.5	8.4
Outside the wear	track					
10 ⁵	66.7	21.3	1.0	9.0	1.7	0.3

at 10^5 Pa is also given. Changes of C 1s spectral features depend on the gas pressure are observed. All the C 1s spectra are doublet main peaks, which can be attributed to carbide (~282.2 eV) and adventitious carbon (~285.0 eV) [18]. Compare the C 1s spectrum in vacuum (10^{-5} Pa) to those in HFC-134a gas, the intensity at binding energy 285.0 eV at lower pressures of 170 and 10^3 Pa are smaller, but they are higher at higher pressures of 10^4 and 10^5 Pa. Moreover, at 10^5 Pa, an obvious new peak at binding energy of about 287.1 eV, which can be attributed to alcohols/ethers or C-CF_x [18,19], appears, indicating that organic compounds are formed on the wear track. Unfortunately, the concentration of the organic matters formed is difficult to determine



Fig. 5. C 1s spectra of the wear tracks on SiC disks *after* sliding in vacuum $(10^{-5} Pa)$ and in HFC-134a gas at different pressures (load: 2.5 N, sliding velocity: 0.30 m/s, sliding distance: 800 m).



Fig. 6. Si 2p spectra of the wear tracks on SiC disks *after* sliding in vacuum $(10^{-5} Pa)$ and in HFC-134a gas at different pressures (load: 2.5 N, sliding velocity: 0.30 m/s, sliding distance: 800 m).

precisely by XPS because of the existence of organic contaminants on the worn surfaces.

Si 2p, F 1s and Al 2p spectra of the wear tracks on SiC disks under different gas pressures are shown in Figs. 6-8, respectively. For comparison, the XPS Si 2p and F 1s spectra of the outside wear track under HFC-134a gas at 10⁵ Pa are also presented. The Si 2p main peak (Fig. 6) of the outside and the wear track in vacuum is at binding energy of 99.8 eV, which corresponds to Si peak in carbides [18]. A small peak at binding energy of 102.2 eV corresponding to Si in Si₃N₄ [18] (added as binders) seems to exist. Si 2p spectra of the wear tracks in HFC-134a at 170-10⁴ Pa are very similar in shape, a peak at binding energy 104.8 eV, which can be attributed to $Si-F_r$ bonding [18] appears, its intensity is the highest at 10^3 Pa. However, at 10^5 Pa, the peak of Si-F_x bonding disappears, and a peak at binding energy 100.4 eV, which may be attributed to organic silicides [18] is detected. Conclusively, $-SiF_x$ containing compounds are produced on the worn surfaces when the friction couples rub in HFC-134a at 170–10⁴ Pa.

From Fig. 7, it can be seen that the F 1s peak on the outside wear track is at about 686.0 eV, which can only be attributed to $Si-F_x$ bonding [18]. This result proves that the chemisorption of HFC-134a molecules occur on the SiC disk. However, the amount of the chemisorption



Fig. 7. F 1s spectra of the wear tracks on SiC disks *after* sliding in HFC-134a gas at different pressures (load: 2.5 N, sliding velocity: 0.30 m/s, sliding distance: 800 m).

molecules is quite little (Table 3). All the main peaks of F 1s on the worn surfaces are at the binding energy of about 687.3 eV, which are higher than F 1s peaks in Si–F_x bonding (686.0–686.6 eV) and AlF₃ (686.3 eV) [18]. These F 1s peaks can be attributed to the overlap of organic fluorine-containing compounds (688.4–689.0 eV) and Si–F_x/metal fluorides (686.0–686.6 eV) [18]. In addition, F 1s peak at binding energy of about 689.0 eV, which is due to –CCF₃ [18] is observed on the SiC worn surface at 10⁵ Pa.

From Fig. 8, the formations of partially fluorinated products of $AIO_x F_y$ (74.3–76.3 eV) at 170 and 10³ Pa, and completely fluorinated product of AIF_3 (76.5 eV) [18] at 10⁴ and 10⁵ Pa are verified. From the atomic concentration shown in Table 3, it can be seen that only a small amount (<4%) of Al 2p exists on the worn surfaces in HFC-134a gas, this existence of Al element is due to the wear of the Al₂O₃ ball transferring onto the SiC worn surfaces.

According to data given in Table 3 and the XPS spectra shown in Figs. 6 and 7, the following data processing is carried out. Except for the carbon coming from carbide, the atomic concentration of carbon from contaminants and formed organic matters is labelled as C'. We use the atomic concentration of Si as a basic element to standardise the



Fig. 8. Al 2p spectra of the wear tracks on SiC disks *after* sliding in vacuum $(10^{-5} Pa)$ and in HFC-134a gas at different pressures (load: 2.5 N, sliding velocity: 0.30 m/s, sliding distance: 800 m).

concentrations of C', F and $-SiF_x$ bonding (simplified as C'/Si, F/Si and $-SiF_x/Si$). In this paper, C'/Si and F/Si values are used as parameters to quantitatively characterise the reaction degree between SiC and HFC-134a molecules.

In Fig. 9, C'/Si, F/Si and $-SiF_x/Si$ values are shown as a function of gas pressure. With an increase in pressure, C'/Si



Fig. 9. Variation in the relative concentration of C'/Si, F/Si and $-SiF_x/Si$ of the wear track on SiC disks with increasing pressure of HFC-134a gas (load: 2.5 N, sliding velocity: 0.30 m/s, sliding distance: 800 m).

Table 4 Products of tribochemical reactions for a SiC disk sliding against an Al₂O₃ ball in HFC-134a gas at different pressures^a

	0 1
Pressure of HFC-134a gas (Pa)	Products of tribochemical reactions
170	$-SiF_x$ containing compounds (main)
10 ³	$-CF_x$ containing organic compounds (little) AlO _x F _y (little)
10 ⁴	-SiF _x containing compounds -CF _x containing organic compounds AlF ₃ (little)
10 ⁵	$-CF_x$ containing organic compounds AIF ₃ (little)

^a Load: 2.5 N, sliding velocity: 0.30 m/s, sliding distance: 800 m.

and F/Si values show increasing trend, and these trends become more obvious when the pressure exceeds 10^3 Pa, indicating that tribochemical reactions are more vigorous, and more carbon/organic compounds and fluorides are formed at higher pressures. It is interested to note that $-\text{SiF}_x/\text{Si}$ value rises with increasing pressure, but it falls sharply when the pressure exceeds 10^3 Pa, showing that the products of tribochemical reaction are dependent on the gas pressure.

On the basis of Figs. 5–9, it can be concluded that tribochemical reactions occur when the Al₂O₃/SiC pairs slide in the presence of HFC-134a gas. Different tribochemical products are formed depending on the gas pressure, which are summarised in Table 4. When the pressure ranges from 170 to 10^3 Pa, $-SiF_x$ containing compounds are the main products of tribochemical reactions. In contrast, when the pressure increases to 10^5 Pa, more alcohols/ethers and fluorine-containing organic compounds are formed at the wear interface.

3.3. Discussion

It is well known that tribochemical reactions are promoted by high contact pressure and temperature generated at the friction interface [20–22]. Nascent surfaces are also playing an important role on tribochemical reactions [23–25]. It is believed that a monolayer of adsorbed gas is enough for tribochemical reactions, and the nascent surfaces of ceramics are covered with molecules of a monolayer in few seconds when exposed to gases at 10^{-4} Pa.

In this case, it is interesting to note that there is a significant environmental pressure influence on the products of tribochemical reactions, even when the pressure far exceeds 10^{-4} Pa. When the Al₂O₃/SiC pair slides at relatively low pressures of $170-10^4$ Pa, $-\text{SiF}_x$ containing compounds are the main tribochemical products. When the pair slides at higher pressure of 10^5 Pa, a great amount of organic matters are formed at the wear interface.

Fig. 10 shows the variation in friction coefficient (Fig. 10a) and specific wear rates (Fig. 10b) with increasing F/Si value of the wear track on the SiC disk. The variation



Fig. 10. Variation of friction coefficient and specific wear rate with increasing F/Si value of the worn surface on SiC disks (a) friction coefficient; (b) specific wear rate (load: 2.5 N, sliding velocity: 0.30 m/s, sliding distance: 800 m).

of $-SiF_x/Si$ value with increasing F/Si value is also shown in Fig. 10a. The friction coefficient slightly increases, and then sharply reduces when the F/Si value exceeds 0.46, and this is the same as the trend of $-SiF_x/Si$. That is to say, an increasing content of $-SiF_x$ containing compounds correlates a higher friction coefficient. Connecting Fig. 10a with Fig. 9, it can be concluded that the organic matters formed are effective for reducing friction. On the contrary, the $-SiF_x$ containing compounds formed cause higher friction.

Variation trends of the specific wear rates of both the disk and ball specimens are the same; they reduce with an increase in F/Si value. Clearly, these tribochemical reactions effectively reduce the wear of ceramics, and more vigorous reactions are beneficial for obtaining a lower wear rate.

When ionic bond type ceramics Al_2O_3 and ZrO_2 disks rub against an Al_2O_3 ball in HFC-134a gas, it has also been found that fluorination reactions of ceramics occur at the wear interface, and only moderate degree tribochemical reactions are good for obtaining both low friction and wear [16,17]. SiC is a covalent bond type ceramic. Its tribochemical reactions in HFC-134a are more complicated than those of oxide ceramics, the composition of the products depends more on gas pressure. Although more vigorous tribochemical reactions lead to a lower wear, the $-SiF_x$ containing compounds formed cause higher friction.

4. Conclusions

- There is a significant gas pressure influence on both the tribological properties and products of tribochemical reactions of the Al₂O₃/HFC-134a/SiC tribosystem.
- 2. When gas pressure increases from vacuum 10^{-5} to 10^{5} Pa, friction coefficient of Al₂O₃/SiC pair decreases from 0.66 to 0.22, wear rates of SiC disk and Al₂O₃ ball are reduced by about factors of 10 and 1000, respectively.
- 3. More fluorides and carbon-containing matters are formed on the worn surfaces when the gas pressure exceeds 10^3 Pa. When the pressure ranges from 170 to 10^3 Pa, $-SiF_x$ containing compounds are the main tribochemical products. When the pressure increases to 10^5 Pa, alcohols/ethers and organic fluorine-containing compounds are mainly formed on the worn surface.
- 4. Organic compounds are effective for reducing friction, whereas the formed $-SiF_x$ containing compounds lead to a slightly increase in friction. All the products of tribochemical reaction are effectively for reducing wear.

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