

# Friction and wear behaviour of $\text{Al}_2\text{O}_3/\text{TiB}_2/\text{SiC}_w$ ceramic composites at temperatures up to $800^\circ\text{C}$

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## Abstract

In this paper,  $\text{Al}_2\text{O}_3/\text{TiB}_2/\text{SiC}_w$  ceramic composites with different volume fraction of SiC whisker were produced, their friction and wear behaviour sliding against cemented carbide at temperatures up to  $800^\circ\text{C}$  in air and nitrogen atmospheres has been studied. The friction coefficient and wear rates were examined in relation to SiC whisker content. Results showed that the composition, temperature and atmosphere have an important effect on their friction and wear behaviours. The friction coefficient and wear rates decreased with increasing SiC whisker content both sliding in air and nitrogen atmospheres. The wear mechanism of the composites at temperature less than  $400^\circ\text{C}$  was primary abrasive wear, the higher wear resistance of composites with higher SiC whisker content under these circumstances corresponded to their higher value of hardness and fracture toughness. The mechanisms of oxidative wear dominated at  $800^\circ\text{C}$  when sliding in air atmosphere, while the wear owing to adhesion and diffusion were suggested to be the main wear mechanism when sliding in nitrogen atmosphere. © 2001 Elsevier Science Ltd and Techna S.r.l. All rights reserved.

**Keywords:** B. Composites; C. Friction; C. Wear resistance; Wear mechanism

## 1. Introduction

Ceramics have intrinsic characteristics, such as: high melting point, high hardness, good chemical inertness and high wear resistance, that make them promising candidates for high temperature structural and wear resistance components, where metallic components achieve only unsatisfactory service lives, owing to inadequate heat, corrosive or wear resistance. Components made of advanced ceramics can survive and perform well at higher operating temperature, and improve the tribological properties. Nowadays the advanced ceramics are widely used in cutting tools, drawing or extrusion, seal rings, valve seats, bearing parts, and a variety of high temperature engine parts etc. [1,2]. However, the use of single-phase ceramics, even fully densified, in wear or structural applications is limited by the variability of their mechanical strength and their poor fracture toughness. Their susceptibility to brittle fracture can lead to unexpected catastrophic failure. Improvement in mechanical properties must be achieved before

the potential of these ceramics can be fully realised. Considerable improvement in mechanical properties of the single-phase ceramic materials has been achieved by incorporating one or more other components into the base material to form ceramic-matrix composites (CMC) [3,4]. The reinforcing component is often in the form of particles or whiskers. Ceramic composites are of increasing interest with oxide matrices, particularly  $\text{Al}_2\text{O}_3$  being dominant. Some of these composites, e.g.  $\text{Al}_2\text{O}_3/\text{TiB}_2$ ,  $\text{Al}_2\text{O}_3/\text{TiC}$ ,  $\text{Al}_2\text{O}_3/\text{ZrO}_2$ ,  $\text{Al}_2\text{O}_3/\text{SiC}$ ,  $\text{Al}_2\text{O}_3/\text{SiC}_w$ ,  $\text{Al}_2\text{O}_3/\text{TiN}$ , have been used in various wear applications, tribological studies on them are also extensively carried out.

In earlier studies it has already been shown that the additions of  $\text{TiB}_2$  secondary phases to  $\text{Al}_2\text{O}_3$  matrix, in amounts higher than 20 vol.% can improve its mechanical properties, i.e. fracture toughness and flexural strength. Further improvement has also been made through the additions of SiC whisker by the authors [5]. Although many variations in composite chemistry have been formulated to improve the above-mentioned mechanical properties, few attempts have been made to improve friction and wear resistance in this manner. In this paper,  $\text{Al}_2\text{O}_3/\text{TiB}_2/\text{SiC}_w$  ceramic composites with

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different volume fraction of SiC whisker were produced, their friction and wear behaviours sliding against cemented carbide at temperatures up to 800°C in air and nitrogen atmospheres have been studied. Particular attention was paid to the effect of SiC whisker additions on the friction and wear behaviours.

## 2. Experimental procedure

### 2.1. Materials and processing

A monolithic Al<sub>2</sub>O<sub>3</sub> (average particle size 0.5 µm) was used as the baseline material. Additions of TiB<sub>2</sub> particles (average particle size 1 µm) and SiC whiskers (diameter 1 to 3 µm, length 20–80 µm) were added to the Al<sub>2</sub>O<sub>3</sub> matrix according to the combinations listed in Table 1. The material was fabricated using colloidal and ultrasonic processing techniques. Filter pressing was used to consolidate the multi-component slurries into green bodies approximately 60 mm in diameter and 15 mm thick. Following drying, final densification of the filter cakes was accomplished by hot pressing with a pressure of 35 MPa in nitrogen atmosphere for 40–60 min to produce a ceramic disk. Details of these procedures and specific processing parameters employed are described elsewhere [5,6].

### 2.2. Material characterization

Densities of the hot-pressed disks were measured by the Archimedes method. Test pieces of 3×4×36 mm were prepared from these disks by cutting and grinding using a diamond wheel. Three-point-bending mode was

used to measure the flexural strength over a 30 mm span at a crosshead speed of 0.5 mm/min. Tests bars of dimensions 5×5×40 mm were prepared for single-edge notched beam (SENB) fracture toughness measurements. The SENB specimens were fractured in four-point with outer and inner span dimensions of 24 and 12 mm, and at a crosshead speed of 0.5 mm/min. Data for density, hardness, flexural strength, and fracture toughness were gathered on five specimens.

### 2.3. Sliding wear test

Sliding wear test apparatus used in this study was a linear reciprocating flat-on-flat high temperature tribometer designed and built by the authors [7]. The upper specimen was made of ceramic materials having a higher polished surface with a surface roughness Ra of 0.08 µm. The lower specimen was made of cemented carbide (WC/Co) with a hardness of HRA 89 and was polished to produce a final surface roughness Ra of 0.05 µm. The polished flats were rinsed with hexane, and then ultrasonically cleaned in fresh hexane, followed by ultrasonic cleaning with acetone. The ceramic specimen slide in reciprocating motion on the cemented carbide. A stroke length of 10 mm was used in all tests. Wear tests were performed with an average sliding speed of 25 mm/s and a normal load of 25 N. Each test was run over a period of 1 h. The friction coefficient was calculated by dividing the measured tangential force by the applied normal force. The wear rate is defined as the volume loss,  $V$ , divided by the applied normal load,  $P$ , times the sliding distance,  $L$ .

$$K = \frac{V}{PL} \quad (1)$$

where the  $K$  has the units of volume loss per unit force per unit distance (mm<sup>3</sup>/Nm).

The worn regions on the ceramic specimens were examined using scanning electron microscopy (HITACHI S-570). The elemental chemical composition of the worn surfaces was determined by X-ray diffraction analysis (D/max-2400). Electron microprobe analysis was used to analyze the adhesion and the element diffusion.

Table 1  
Material composition

Specimen	Composition (vol.%)		
	Al <sub>2</sub> O <sub>3</sub>	TiB <sub>2</sub>	SiC <sub>w</sub>
ABW05	76	19	5
ABW10	72	18	10
ABW20	64	16	20
ABW30	56	14	30

Table 2  
Mechanical properties of Al<sub>2</sub>O<sub>3</sub>/TiB<sub>2</sub>/SiC<sub>w</sub> ceramic composites with different volume fraction of SiC whisker

Specimen	Relative density (%)	Hardness (GPa)	Flexural strength (MPa)	Fracture toughness (MPa <sup>1/2</sup> )
ABW05	99.90	21.4	778	5.90
ABW10	99.84	21.6	750	7.60
ABW20	99.42	21.7	726	7.97
ABW30	98.93	22.0	670	8.42

### 3. Results and discussion

#### 3.1. Material properties

Results of the fracture toughness, flexural strength, hardness and relative densities of the composites with different  $\text{SiC}_w$  content are presented in Table 2. As can be seen, the fracture toughness and hardness continuously increased with increasing  $\text{SiC}_w$  content up to 30 vol.%. The relative density of the composites decreased with increasing  $\text{SiC}$  whisker content, the trend of the flexural strength being the same as that of the relative density. The decrease of flexural strength with increasing  $\text{SiC}_w$  content is likely due to the decrease in the relative density associated with  $\text{SiC}$  whisker agglomerates [5,6]. Fig. 1 shows a SEM micrograph of the fracture surface of ABW10. As can be seen, protruding whiskers and holes where whiskers were lodged prior to fracture were observed, these are evidence of whisker pullout and bridging.

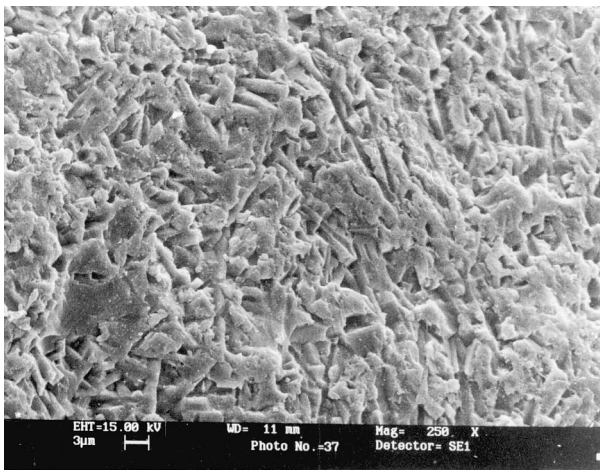


Fig. 1. SEM micrograph of the fracture surface of ABW10.

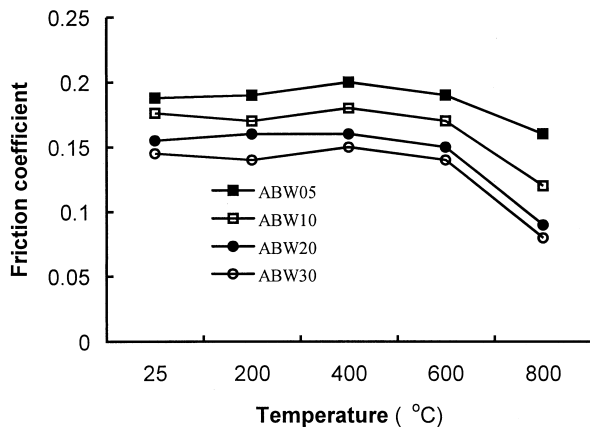


Fig. 2. Friction coefficient vs temperature for  $\text{Al}_2\text{O}_3/\text{TiB}_2/\text{SiC}_w$  composites with different  $\text{SiC}_w$  content when sliding against cemented carbide in air atmosphere.

#### 3.2. Friction coefficient and wear rates

In sliding wear tests performed in air and nitrogen atmospheres, the friction coefficients of  $\text{Al}_2\text{O}_3/\text{TiB}_2/\text{SiC}_w$  composites with different  $\text{SiC}_w$  content were a function of temperature, and are shown in Figs. 2 and 3, respectively. As can be seen, the friction coefficient was quite insensitive to the testing temperature and to the chemistry of the composites. ABW05 specimen exhibited the highest friction coefficient, and ABW30 the smallest. The friction coefficient decreased with increasing  $\text{SiC}_w$  content both sliding in air and nitrogen atmosphere, the specimens with higher  $\text{SiC}_w$  content exhibited improved friction behaviour. The friction coefficient did not vary much for all specimens at temperatures ranging from 25 to 400 °C. and decreased dramatically when sliding in air atmosphere at 800 °C. While the friction coefficient increased as the temperature increased when sliding in nitrogen atmosphere, and showed a great increase at 800 °C.

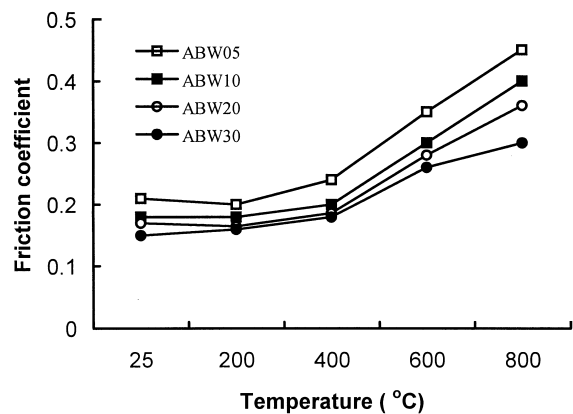


Fig. 3. Friction coefficient vs temperature for  $\text{Al}_2\text{O}_3/\text{TiB}_2/\text{SiC}_w$  composites with different  $\text{SiC}_w$  content when sliding against cemented carbide in nitrogen atmosphere.

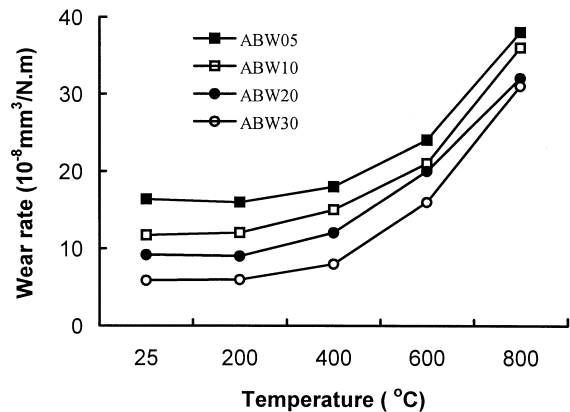


Fig. 4. Wear rates vs temperature for  $\text{Al}_2\text{O}_3/\text{TiB}_2/\text{SiC}_w$  composites with different  $\text{SiC}_w$  content when sliding against cemented carbide in air atmosphere.

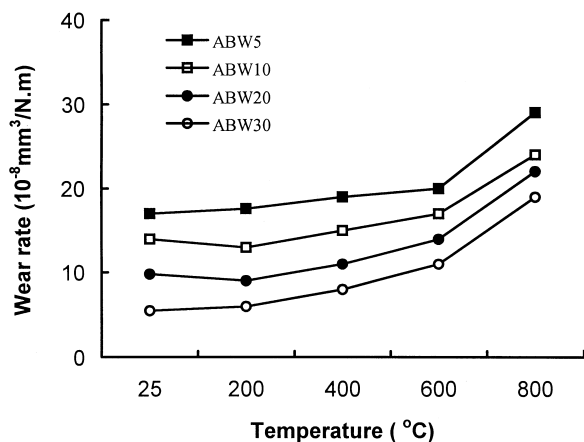
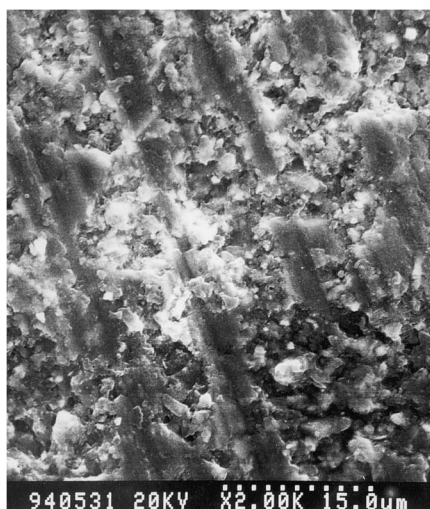


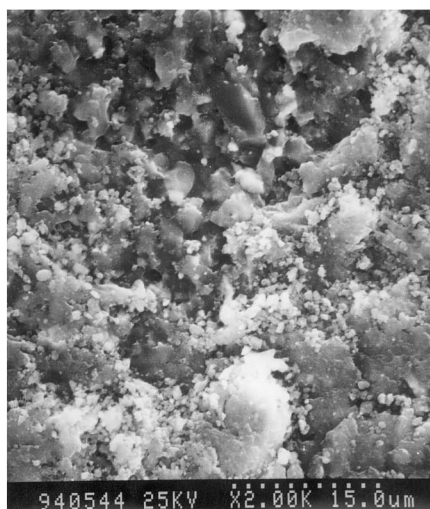
Fig. 5. Wear rates vs temperature for  $\text{Al}_2\text{O}_3/\text{TiB}_2/\text{SiC}_w$  composites with different  $\text{SiC}_w$  content when sliding against cemented carbide in nitrogen atmosphere.

Figs. 4 and 5 show the wear rates of all specimens sliding against cemented carbide as a function of temperature in air and nitrogen atmospheres respectively. As can be seen, the wear rates were approximately constant for all specimens both sliding in air and nitrogen atmospheres at temperatures ranging from 25 to 400°C, and showed a gradual increase when the temperature was greater than 600°C. The specimens with higher  $\text{SiC}_w$  content resulted in low wear rates both sliding in air and nitrogen atmosphere.

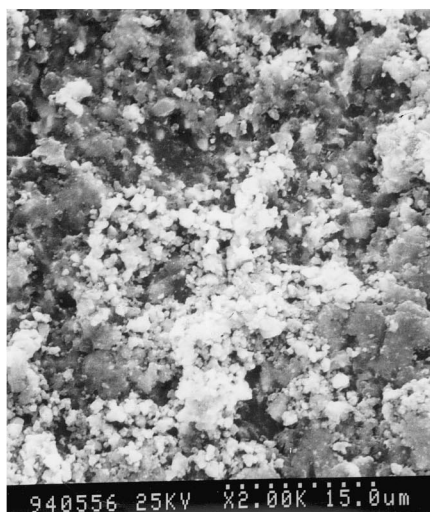
The results confirm that the tribological behavior and wear resistance of  $\text{Al}_2\text{O}_3/\text{TiB}_2/\text{SiC}_w$  composites tested depends on the composition, temperature and atmosphere. Both the friction coefficient and wear rates are approximately constant for all specimens at the temperature less than 400°C. The specimens with higher  $\text{SiC}_w$  content exhibit improved friction behaviour and wear resistance at temperatures up to 800°C.



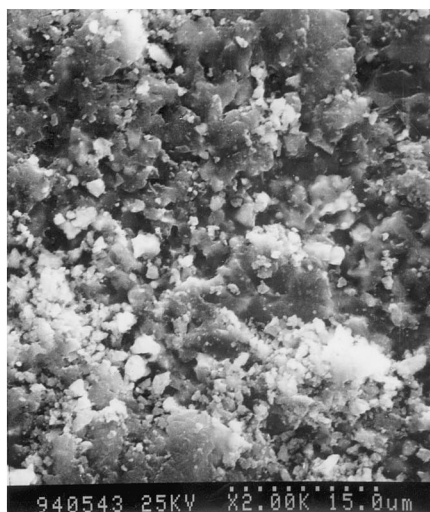
(a)



(b)



(c)



(d)

Fig. 6. SEM micrographs of the worn surface of ABW10 specimen sliding in air and in nitrogen atmosphere at 200 and 400°C (a) 200°C in air; (b) 400°C in air; (c) 200°C in nitrogen; (d) 400°C in nitrogen.

### 3.3. Wear surface studies

The SEM micrographs of the worn surface of ABW10 specimen sliding in air and in nitrogen at 200 and 400°C are shown in Fig. 6, respectively. These micrographs are indicative of abrasive wear. It can be seen that the wear track was covered with compacted wear debris, clearing of the specimens in an ultrasonic bath with hexane can remove some of the loose debris. Also there were numerous scratches on the wear surface. This suggests that the primary wear mechanisms under these conditions are abrasive wear. The wear resistance was found to improve with increasing  $\text{SiC}_w$  content. The fact can be understood according to Evans's equation [8]:  $K \propto H^{-1/2} K_{IC}^{-3/4}$ , where  $K$  means the wear rates,  $H$  means the hardness,  $K_{IC}$  means the fracture toughness. Therefore, composites with greater  $\text{SiC}_w$  content exhibited higher hardness and fracture resistance, leading to the least wear scratches and the best wear resistance. The higher wear resistance of specimen ABW30 corresponded to its highest values of hardness and fracture toughness.

The SEM micrograph of the worn surface of ABW05 specimen sliding in air at 800°C is shown in Fig. 7. The wear track shows clearly a thin oxide film. When compared to Fig. 6, the presence of the oxide film becomes obvious. X-ray diffraction analysis of the worn surface showed the presence of  $\text{TiO}_2$  and  $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$  on the wear track, as shown in Fig. 8, no  $\text{B}_2\text{O}_3$  phase can be detected owing to its low melting point (577°C). It is obvious that  $\text{TiO}_2$  and  $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$  are newly formed phases, and are resulted from the oxidation of  $\text{TiB}_2$  and  $\text{SiC}_w$ . The oxidation formulas are as follows [9]:

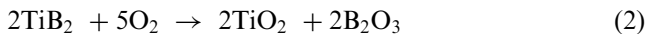
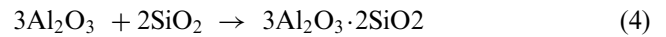
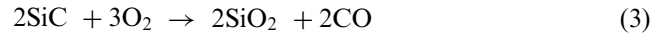


Fig. 7. SEM micrograph of the worn surface of ABW05 specimen sliding in air at 800°C.



The SEM micrograph of the worn surface of ABW30 specimen sliding in air at 800°C is shown in Fig. 9. As can be seen that ABW30 specimen displayed a similar lubrication process at 800°C, the oxide growth in the wear track appears to be present to a much higher degree compared to ABW05 owing to its higher volume content of  $\text{SiC}$  whiskers, and there is a lot of small cracks on the oxide film. X-ray diffraction analysis of the worn surface of ABW30 tested in air at 800°C also showed the presence of  $\text{TiO}_2$  and  $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ .

The friction coefficient between two smooth bodies sliding under elasticity-loaded conditions in an elliptical contact can be decreased as [10,11]:

$$\mu = A \frac{\tau}{P^{1/3}} \left( \frac{3}{4E'} \right)^{2/3} \quad (5)$$

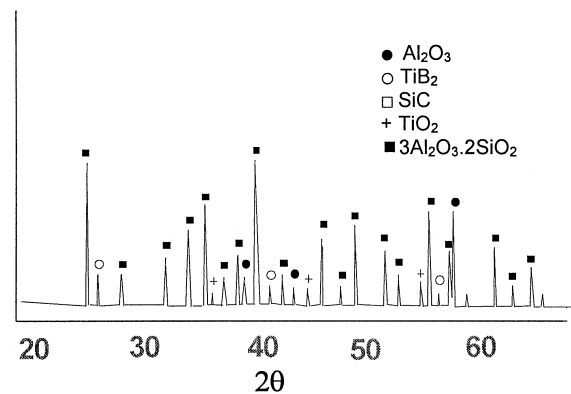


Fig. 8. X-ray diffraction analysis of the worn surface of ABW05 tested in air at 800°C.

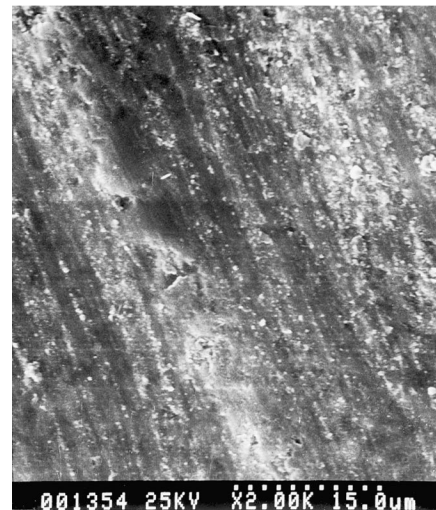


Fig. 9. SEM micrograph of the worn surface of ABW30 specimen sliding in air at 800°C.

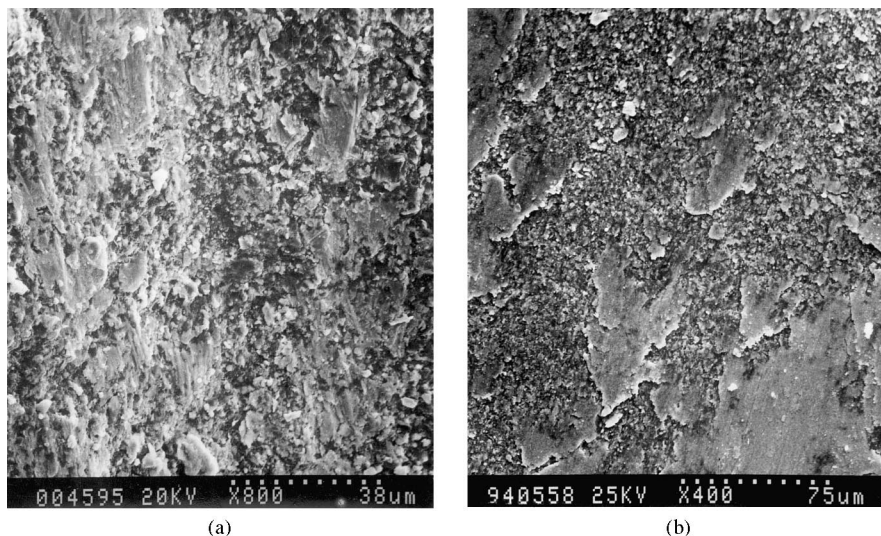


Fig. 10. SEM micrographs of the worn surface of ABW10 and ABW30 composite sliding in nitrogen at 800°C: (a) ABW10; (b) ABW30.

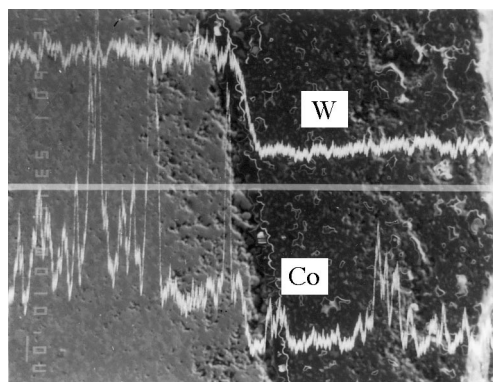


Fig. 11. Cross-section of ABW10 specimen after sliding in nitrogen atmosphere at 800°C, the dashed line represented the EDX line of scanning analysis results of the Co element.

where  $A$  is a constant determined by contact geometry,  $\tau$  is critical shear stress at the interface, which may be an oxide film,  $P$  is the normal load, and  $E'$  the effective elastic modulus of the contact materials. Thus, for a given contact geometry Eq. (5) shows that the friction coefficient varies linearly with critical shear stress. The oxide film on the wear surface had a much smaller critical shear stress than the substrate and thus resulted in a reduced friction coefficient according to Eq. (5). So the formation of this lubricious oxide film on the wear surface may result in a decrease in friction coefficient when sliding in air at 800°C.

The SEM micrograph of the worn surface of ABW10 and ABW30 composite sliding in nitrogen atmosphere at 800°C are shown in Fig. 10. An adhesive and deformed layer can be seen, the wear track shows clearly some adhering materials. Thus the possibility of adhesion and diffusion wear should be considered the main reason for high wear rates at these conditions. There was experimental evidence of adhesion and diffusion of the

Co element of cemented carbide to the specimen. EDX analysis of the cross-section of ABW10 after sliding in nitrogen atmosphere at 800°C was shown in Fig. 11, the dashed line represented EDX line of the scanning analysis results of the Co element. It can be seen that the Co of cemented carbide diffused rather a long way into the ceramic composite. The Co had a low melting point and hardness, so the adhesion and diffusion of Co to the ceramic specimen may accelerate the wear rates. This may explain the higher wear rates when sliding in nitrogen atmosphere at 800°C.

#### 4. Conclusions

$\text{Al}_2\text{O}_3/\text{TiB}_2/\text{SiC}_w$  ceramic composites with a different volume fraction of SiC whisker were produced to further optimise its wear resistance. Particular attention was paid to the effect of SiC whisker additions on the friction and wear behaviours. Results showed that:

1. The fracture toughness and hardness continuously increased with increasing  $\text{SiC}_w$  content up to 30 vol.%, while the relative density and flexural strength decreased with increasing SiC whisker content.
2. The friction coefficient and wear rates decreased with increasing  $\text{SiC}_w$  content both sliding in air and nitrogen atmospheres at temperatures up to 800°C.
3. The wear mechanism of  $\text{Al}_2\text{O}_3/\text{TiB}_2/\text{SiC}_w$  composites at temperatures less than 400°C is primary abrasive wear, the greater wear resistance of composites with higher  $\text{SiC}_w$  content under these circumstances corresponds to their higher value of hardness and fracture toughness.

4. The mechanism of oxidative wear dominates at 800°C when sliding in air atmosphere. While the wear owing to adhesion and diffusion was suggested to be the main wear mechanism when sliding in nitrogen atmosphere at 800°C.

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