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Ceramics International 40 (2014) 1165-1170

# Wear performance of SiC/G coating at elevated temperatures

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> Received 16 April 2013; received in revised form 30 June 2013; accepted 30 June 2013 Available online 5 July 2013

#### Abstract

To improve the wear performance of SiC materials, a SiC/graphite (SiC/G) coating was prepared by CVD. Wear tests were conducted at elevated temperatures, and related microstructure and mechanical characteristics were investigated by scanning electron microscopy (SEM), X-ray diffraction (XRD) and micro-hardness tester. The results show that the SiC/G coating has excellent anti-wear performance with low friction coefficient at RT owing to the lubrication of graphite. Moreover, the friction coefficient of the SiC/G coating is lower than that of the uncoated SiC samples at all tested temperatures. As the temperature rose to 250 °C, the wear mechanism of SiC/G coating was shifted to severe adhesive wear, which resulted in an increase in friction coefficient. However, fracture and oxidation dominated the friction process of SiC/G at 500 °C, which caused an increase in wear volume. In addition, the formation of silica debris was conductive to the decrease of friction coefficient. © 2013 Elsevier Ltd and Techna Group S.r.l. All rights reserved.

Keywords: D. SiC; Wear; Graphite; Coating

## 1. Introduction

SiC materials are increasingly used for wear components due to their high hardness good corrosion resistance, and excellent chemical stability [1–3]. However, the friction coefficients of SiC materials are still unacceptably high in the unlubricated conditions [4,5], which limits their application on the wear resistant materials for machine parts, such as piston, bearing, cylinder liner, etc. To improve the wear performance of SiC materials, the effects of several material modifications have been investigated. For example, it has been reported that the addition of graphite, hexagonal boron nitride and nickel to ceramic materials enhances their lubricity [6-9]. Wäsche et al. researched the tribological behavior of SiC composites containing free carbon [6]. The influence of graphite on tribological behavior neither a beneficial nor a detrimental effect has been found. It might be ascribed to uneven distribution of graphite in the microstructure. However, homogeneous SiC/graphite microstructure can be obtained by coating technology, and the coated SiC ceramic is expected to get low friction coefficient. In this way, it not only keeps the

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advantage of SiC substrate, but also improves the tribological properties of SiC effectively [10].

The chemical vapor deposition (CVD) process is widely used to deposit SiC coating [11,12]. The as-prepared SiC coating with high homogeneousness and near-stoichiometric characteristics are advantageous in coating stability [13]. Furthermore, the SiC coating prepared by CVD at a relatively lower deposition temperature (1000–1300 °C) usually consists of  $\beta$ -SiC [14–16]. The wear performance of  $\beta$ -SiC is better than that of  $\alpha$ -SiC because the cracks are more difficult to propagate in  $\beta$ -SiC phase [17,18].

In the present work, SiC/G coating was deposited on the surface of SiC substrate by CVD. Subsequently, the friction coefficients and wear volume of the SiC/G coatings and the uncoated SiC samples at elevated temperatures were measured, respectively. Moreover, the wear mechanisms of the SiC/G coating at different temperatures were also illustrated.

#### 2. Experimental details

The SiC/G coating was deposited on polycrystalline SiC samples ( $30 \times 30 \times 6 \text{ mm}^3$ , Ra of 0.05  $\mu m$ , hardness of 26 GPa)

placed inside the hot area of a CVD reactor chamber.  $H_2$ ,  $C_3H_8$ , and  $SiCl_4$  were chosen as the reactive materials. The pressure of reactor was kept at 0.1 MPa. The deposition temperature was  $1100~^{\circ}C$  and the deposition time was 3 h.

Wear experiments were carried out in a tribometer model HT-1000. Commercial SiC balls with a diameter of 5 mm, surface roughness Ra of 0.05  $\mu m$  and hardness of 26 GPa, were used as mating balls. The sliding tests were performed at room temperature (RT), 250 and 500 °C, respectively. The sliding speed was 0.2 m/s, and the applied load was 5 N. The sliding distance in each test was 1000 m, and the wear track diameter on the coating was 10 mm. Three tests were carried out for each sample, and the stationary value of friction coefficient was recorded for each test. Finally, the friction coefficients were given as the mean value of the stationary value for each sample at different temperatures. The wear volume of coatings was calculated using the following equations:

$$w = 2\pi rA \tag{1}$$

where r is the wear track radius, and A is the cross-sectional area of wear track on the coating. The A is measured by optical profiler (NANOVEA, ST400).

The morphology of SiC/G coating and worn surface were observed and analyzed by a scanning electron microscopy (SEM, JEM-3010) equipped with energy dispersive spectroscopy (EDS). The crystalline structures of SiC coating were analyzed by an X-ray diffraction analyzer (XRD, XRD-7000). The hardness the SiC/G coating on SiC substrate was measured by a micro-hardness tester with a diamond Berkovich indenter (AHVD-1000XYZ). The adhesion strength between the coating and the substrate was studied using WDS-2005 scratch tester.

#### 3. Results and discussion

# 3.1. Characteristics of the coating

XRD pattern of the SiC/G coating obtained by CVD is shown in Fig. 1. The pattern indicates that the coating consists of  $\beta$ -SiC,  $\alpha$ -SiC and graphite. The dominant amount of  $\beta$ -SiC and a small fraction of graphite is expected, however, the formation of a small number of  $\alpha$ -SiC at a relatively lower deposition temperature (1100 °C) should be due to the deposition conditions are far from thermodynamic equilibrium [17]. Fig. 2 shows surface and cross-section SEM images of the SiC/G coating. It clearly reveals that the coating is about 30 µm in thickness, and no obvious crack can be found on surface and cross-section of the coating. The dense structure is good for the anti-wear property and the homogeneous structure guarantees the stable wear performance. Furthermore, the hardness of the SiC/G coating and the adhesion strength between the coating and the substrate are also important properties for its wear performance. The hardness measurement was performed on the SiC/G coating, and the hardness value was obtained as 21.7 GPa, which is lower than that of SiC substrate and that of CVD-SiC coating reported by

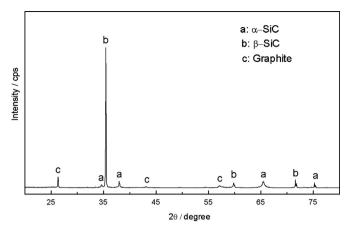
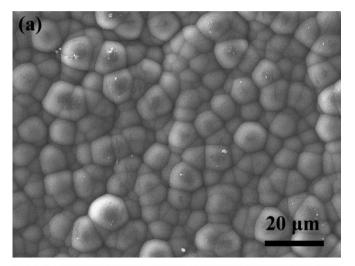


Fig. 1. XRD patterns of the SiC/G coating obtained by CVD.



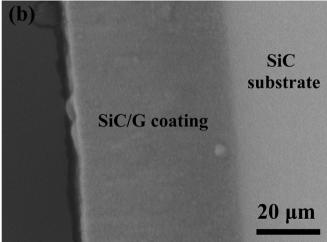


Fig. 2. SEM micrographs of the SiC/G coating: (a) surface and (b) cross-section.

literatures [17,19]. In addition, the scratch testing (Fig. 3) shows that the adhesion strength between the SiC/G coating and SiC substrate is about 32 N, which indicates that the SiC/G coating has good compatibility with the substrate.

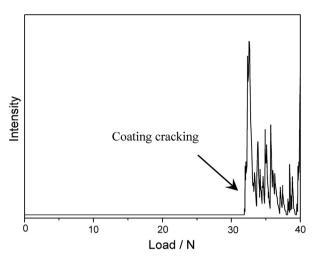


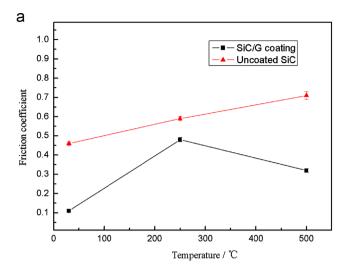
Fig. 3. The curve of acoustic emission accounts versus load for SiC/G coating obtained by scratch testing.

### 3.2. Wear performance

Fig. 4 shows the wear performance of SiC/G coating and the uncoated SiC specimen at elevated temperatures. It can be seen that the friction coefficient of the SiC/G coating against the SiC ball is about 0.11 at RT (Fig. 4a). As the temperature rises to 250 °C, it increases to the highest value (0.48). However, when the temperature is 500 °C, the friction coefficient of SiC/G coating drops to about 0.32. For the uncoated SiC sample, the friction coefficient is 0.46 at RT, which is higher than the quadruple of that of SiC/G coating. Moreover, the friction coefficient of the uncoated SiC specimen increases with increasing of the ambient temperatures. Fig. 4b demonstrates the wear volume of tested specimens at different temperatures. It obviously shows that the wear volume of both tested samples increases with the rise of ambient temperature. The wear volume of SiC/G coating is  $1.21 \times 10^{-2}$  mm<sup>3</sup> at RT, which is less than that of the uncoated SiC  $(1.76 \times 10^{-2} \text{ mm}^3)$ . As the temperature is up to 250 °C, the wear volume of the SiC/G coating and the uncoated SiC increases to  $9.69 \times 10^{-2}$ mm<sup>3</sup> and  $8.85 \times 10^{-2}$  mm<sup>3</sup>, respectively. However, when the temperature is 500 °C, the wear volume of the SiC/G coating (0.16 mm<sup>3</sup>) is lower than that of the uncoated SiC again  $(0.23 \text{ mm}^3).$ 

# 3.3. Worn surface analysis

Fig. 5a shows the worn surface of the uncoated SiC sample at RT. It obviously indicates that the worn surface is smooth, only some slender grooves can be observed on the surface. It implies that mild abrasive wear is main wear mechanism in the friction process. When the temperature rises to 250 °C, a large amount of sheet debris can be observed on the worn surface of the uncoated SiC sample (Fig. 5b), which should be attributed to the adhesion wear. However, the feature of grains pullout presents on the worn surface as the temperature is up to 500 °C (Fig. 5c). It reveals that fracture and serious abrasive wear dominate the friction process.



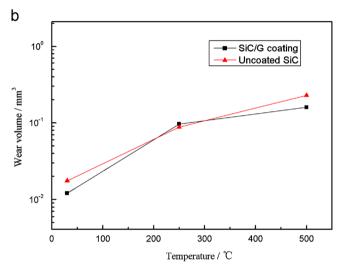


Fig. 4. (a) Friction coefficient of the SiC/G coating and the uncoated SiC sample at different temperatures; and (b) wear volume of the SiC/G coating and the uncoated SiC sample at different temperatures.

For the SiC/G coating, as shown in Fig. 6a, a narrow and shallow wear track generated on the SiC/G coating, which means only a mild wear occurred on the test coating at RT. The higher magnification image (Fig. 6b) indicates that the worn surface presents the characteristics of smooth and clean, which implies that slight adhesive wear was predominately wear mechanism at RT. Furthermore, lamellar structure can be observed in some zones. According to EDS analysis, the lamellar structure is confirmed to be graphite. As is well-known, graphite could act as a lubricant. It should make contribution to the decrease of the friction coefficient and wear volume of tested coating.

As the temperature rises to  $250\,^{\circ}$ C, the wear track is significantly increased in width and fracture appears on the edge of wear track (Fig. 7a), which implies the rising temperature exacerbated the wear of SiC/G coating. Fig. 7b is the higher magnification image of worn surface of the SiC/G coating at  $250\,^{\circ}$ C. It shows a surface on which there are relatively large and thin flakes appeared to be only weakly attached. In addition, some cracks also can be observed clearly on the surface. The initiation

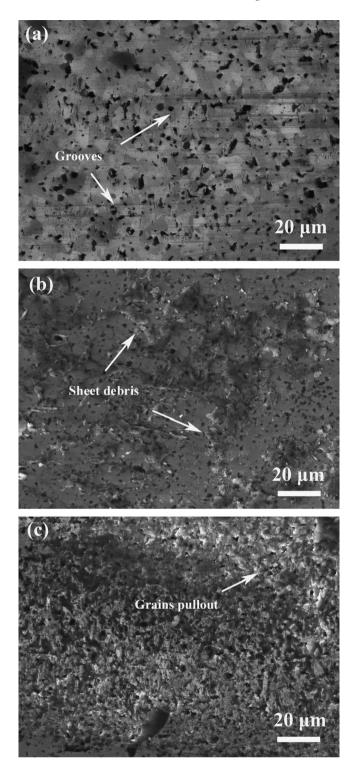
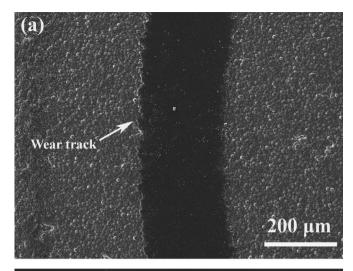


Fig. 5. SEM micrographs of worn surface on the uncoated SiC samples at: (a) RT; (b) 250 °C; and (c) 500 °C.

and propagation of cracks should be attributed to the increased friction coefficient which resulted in relatively high shearing stress to the tested coating. However, "dusting wear" of graphitic materials, which is widely believed to be caused by the interaction between the unsaturated covalent bonds of carbon atoms created by wear, should be the main reason for the high friction coefficient of the SiC/G coating at 250 °C [20]. In the temperature ranging



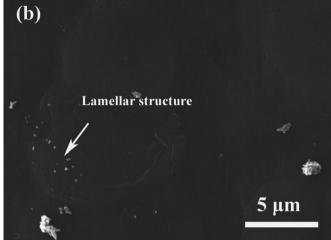
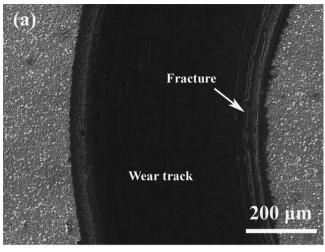


Fig. 6. SEM micrographs of worn surface on the SiC/G coating at RT, (a) low magnified; and (b) high magnified.

between 150–200 °C, researchers have observed a normal-to-dusting wear transition in ambient air for graphite and have attributed it to desorption of water vapor from graphite surface [21]. Similarly, The SiC/G coating in the present work exhibits low friction coefficient and excellent anti-wear performance at RT because of the self-lubricating effect of graphite. Nevertheless, the moisture and other hydrated contaminations which absorbed on the SiC/G coating surfaces are eliminated as the ambient temperature rises to 250 °C. It means that the graphite in the tested coating suffered the transition of normal-to-dusting wear and cannot act as lubricant to improve the wear performance any more. Therefore, the worn surface of SiC/G coating is characterized by typical adhesive wear.

At 500 °C, the worn surface of the SiC/G coating is not flat any more, and lots of wear debris attached on the surface can be observed (Fig. 8a and b). According to EDS analysis (Fig. 8d), this debris, ranging in size from hundreds of nanometers to several micrometers, is composed of silica. The formation of silica debris should because small SiC particles broke away from the coating to form debris which was oxidized subsequently under the combined effect of friction



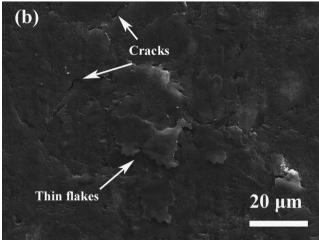


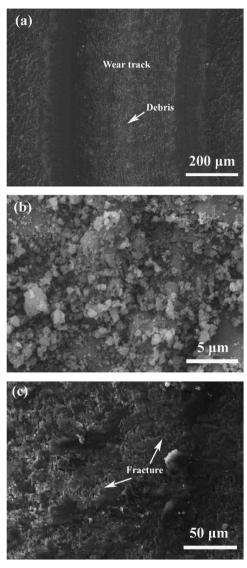
Fig. 7. SEM micrographs of worn surface on the SiC/G coating at 250  $^{\circ}$ C, (a) low magnified; and (b) high magnified.

heat and high ambient temperature. Furthermore, after the debris was removed, a more detailed view of the worn surface (Fig. 8c) shows features of fracture. It implies that the wear of tested coating becomes more serious under those test conditions, which can be fundamentally attributed to the following reasons. (i) The elevated temperature aggravated the oxidation of SiC/G coating and probably resulted in the decrease of mechanical strength. The fatigue cracks were easy to initiate and propagate which resulted in the SiC grains broke and separated from the coating to form debris. (ii) At 500 °C, the graphite in the SiC/G coating could be oxidized and the loss of graphite means the absence of self-lubricating. It is interesting to note, the elevated temperature aggravated the wear of the SiC/G coating, but the friction coefficient at 500 °C was lower than that at 250 °C. It should because the fine silica debris spread on the wear track, which being soft in nature reduced the friction coefficient [22,23].

### 4. Conclusions

The SiC/G coating was deposited on SiC substrate by CVD, and the wear performance of SiC/G coating sliding against SiC

was investigated at elevated temperatures. The results show that the SiC/G coating has excellent anti-wear performance with low friction coefficient at RT. Moreover, the friction coefficient of the SiC/G coating was lower than that of the



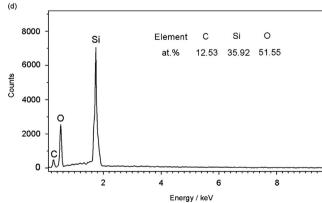


Fig. 8. (a) SEM micrograph of worn surface on the SiC/G coating at  $500\,^{\circ}$ C; (b) SEM micrograph of debris on the SiC/G coating; (c) a detailed view of the worn surface on the SiC/G coating at  $500\,^{\circ}$ C after the debris was removed; and (d) EDS analysis of the debris.

uncoated SiC samples at all tested temperatures. As the temperature rose to 250  $^{\circ}$ C, the wear mechanism of SiC/G coating was shifted to severe adhesive wear, which resulted in an increase in friction coefficient. However, fracture and oxidation dominated the friction process of SiC/G at 500  $^{\circ}$ C. The formation of silica debris was conductive to the decrease of friction coefficient at 500  $^{\circ}$ C.

# Acknowledgments

This work has been supported by the Natural Science Foundation of Shanxi Province (Grant no. 2012JQ6020), the Doctor Foundation of Xian University of Science & Technology of China (2011QDJ017) and the National Natural Science Foundation of China (Grant no. 51201131).

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