

SHORT COMMUNICATION

## Effects of Temperature on Tribological Properties of $Al_2O_3$ - $TiO_2$ Coating

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### ABSTRACT

The  $Al_2O_3$ - $TiO_2$  coating for turbine blades is being used since long. During the last decades, a large number of papers have been published, but work on property assessment of  $Al_2O_3$ - $TiO_2$  coating on rotating component from tribological point of view is very less. This paper assesses the wear behaviour of  $Al_2O_3$ - $TiO_2$  coating on inconel 601. The  $Al_2O_3$ - $TiO_2$  coating on inconel 601 is deposited by plasma spray process. The SEM results show that above 300 °C, the friction coefficient decreases due to softening of coating material. The wear rate increases with increase in temperature. The coating showed brittle fracture at higher temperature. Other test results have shown the drastic changes in property due to load and temperature.

**Keywords:** Material processing, plasma spraying, microstructures

### 1. INTRODUCTION

The ceramics in coatings form for applications involving corrosion, wear, and high temperature are widely used due to their many inherent properties<sup>1-3</sup>. The plasma spraying is an established technique to produce thick ceramic coatings. Alumina-based coatings deposited by plasma spraying are widely used for a range of industrial applications due to their important mechanical properties and low production costs<sup>4-6</sup>.

The tribological behaviour of ceramics is also greatly influenced by test conditions, such as, contact load, environment, and temperature<sup>7-13</sup>. The presence of adsorbed moisture and the formation of oxide films can decrease the wear of ceramics<sup>13</sup>. The wear rate of alumina-based ceramics increases with the temperature, increasing<sup>12</sup> up to 600 °C. Tetsuya<sup>7</sup>, *et al.* found grain size also has an important effect on the wear resistance of alumina at elevated

temperatures. Therefore, an interest has been shown by the researchers in the wear of plasma sprayed ceramic coatings at elevated temperatures.

In the present study, the tribological behaviour of alumina-3 Wt per cent  $TiO_2$  coating deposited by plasma spraying was investigated by sliding against a silicon nitride ball under unlubricated conditions at temperatures ranging from room temperature to 600 °C. The wear mechanisms were discussed in terms of the observations and analyses of the wear surfaces and the debris particles.

### 2. EXPERIMENTAL

Alumina-3 Wt per cent titania coating was prepared by atmosphere plasma spraying (A-2000, Sulzer Metco AG, Switzerland). The material used was the conventional fused and crushed powder of 10-35  $\mu m$ <sup>14</sup>. The coatings were deposited onto stainless steel substrates ( $\Phi$  25 mm x 8 mm),

which were cleaned with acetone and grit blasted before plasma spraying. The coating thickness was about 480  $\mu\text{m}$ . The above experiments were described by Lin<sup>15</sup>, *et al.*

Tribological properties of the as-sprayed coatings against a 3 mm  $Si_3N_4$  ball were examined using a pin-on-disc machine. The schematic drawing of the test rig is shown in Fig. 1. On the ball bearing a normal load was fixed and the disk sample was rotated at 199 rpm (0.1 m/s). Applied load was 5 N and the sliding distance was 60 m. Wear tests were carried out in the unlubricated condition and at temperatures ranging from room temperature, i.e.,

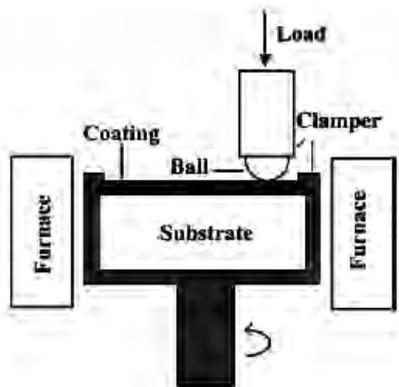


Figure 1. Experimental setup of pin-on-disc machine at IIT Roorkee.

about 27 °C to 600 °C. The disc sample was mounted on a stainless steel holder and was heated to the required temperature. The relative humidity varied from 40 per cent to 60 per cent. Prior to the test, the as-sprayed coatings were polished to a roughness of 0.08–0.15  $\mu\text{m}$ . The friction coefficient was continuously recorded during the tests. Surface profiles of the wear tracks on the disks were measured by a surface profilometer. The wear volume of the disk specimen was determined from the average cross-sectional area of the wear track obtained from three points measured perpendicular to the sliding direction. The presented friction coefficients and wear rates were the average of the three specimens. The microstructure of as-sprayed coatings was investigated by field emission scanning electron microscope (JSM-5800, JEOL, Japan). The worn surface and wear debris were examined by a SEM (EPMA-8705QH2, Shimadzu, Japan) equipped with an EDAX attachment (Oxford Instruments, Model 6841, UK).

### 3. RESULTS

#### 3.1 Friction and Wear

From Fig. 2, it can be seen that the friction coefficients of conventional  $Al_2O_3$ -3 Wt per cent  $TiO_2$  coating ranged from 0.8 to 1.0 for the whole temperature range.

It is clear from Fig. 2 that the friction coefficient decreases as the temperature increases. It is found

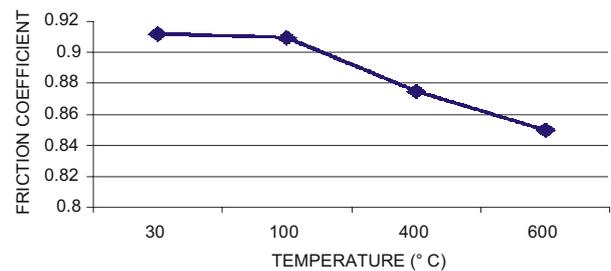


Figure 2. Effect of temperature on friction coefficient of the coating.

that from room temperature to 100 °C, friction coefficient was more or less constant but after that, graph showed a sliding trend. Figure 3 shows the effect of temperature on the wear rates of the as-sprayed coatings.

At room temperature, the wear rate of coatings was too small to be measured accurately, and gradually it increased with increase in temperature.

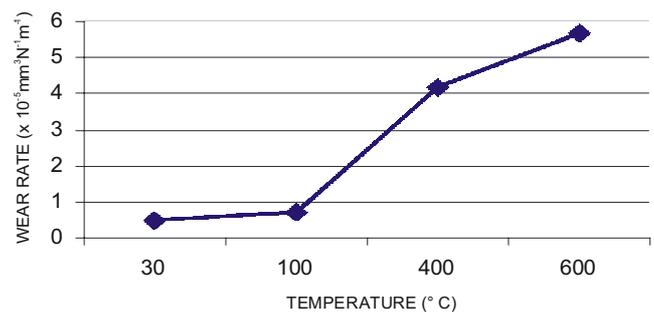


Figure 3. Effect of temperature on wear rate of the coating material ( $\times 10^{-5} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$ ).

#### 3.2 SEM of Worn Surface and Wear Debris

Figure 4 shows the micrographs of the worn surfaces of a conventional  $Al_2O_3$ -3 Wt per cent  $TiO_2$  coatings sliding against a silicon nitride ball at room temperature. The worn surfaces of coatings

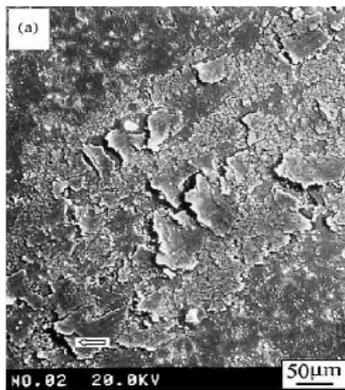


Figure 4. SEM micrographs of worn surface of  $Al_2O_3$ -3 Wt per cent  $TiO_2$  coating surface at room temperature.

were covered with a discontinuous compact layer. The EDS analysis revealed that the compact layer contained large amounts of silicon but no nitrogen, suggesting that silicon nitride was oxidised and transferred to the worn surface of the coatings<sup>7,18</sup>. There were many cracks existing on the worn surface of the coatings and the compact layer was in the process of delamination as flake-like debris, due to the effect of the cyclic stress. In some places, the delamination occurred in coatings, and large craters were found. It was thought that the cracks would propagate along areas of weakness in the underlying material under cyclic stress due to the strong adhesion between the transferred surface layer and the underlying material, i.e., a fatigue process was involved<sup>19</sup>. Since the inter-splat bonding is weak in the plasma-sprayed ceramic coating<sup>20,21</sup>, the delamination was along the splat boundary, and accordingly, a large crater was produced. It was inferred that the wear rate of the coating would increase if the sliding distance was increased.

It can be seen that the morphology of the worn surface of the coating is strongly dependent on the testing temperature. From Fig. 5, it can be seen that at 100 °C and 400 °C, smooth areas were absent in the coating and the worn surface was covered with a layer of fine wear debris particles [Figs 5(a) and 5(b)]. At 100 °C, most of the wear debris particles were < 1 μm and were globular, whilst their size was up to several μm at 400 °C and their morphology was angular. It was found that the composition of the wear debris at 100 °C was similar to that at 400 °C and it was mainly alumina.

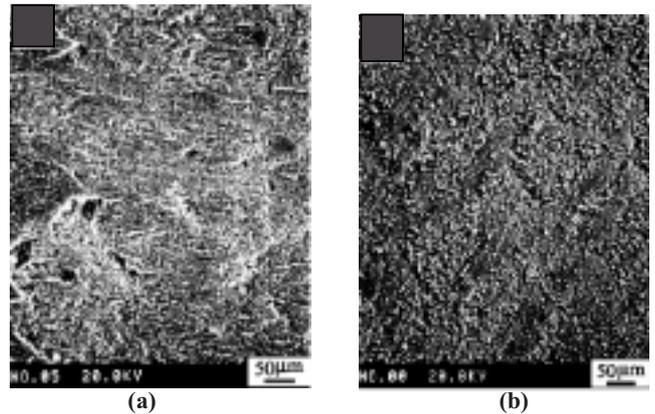


Figure 5. SEM micrographs of worn surface of  $Al_2O_3$ -3 Wt per cent  $TiO_2$  coating surface at: (a) 100 °C, and (b) 400 °C.

#### 4. DISCUSSIONS

Tribochemical reaction of  $Al_2O_3$ -3 wt per cent  $TiO_2$  coating is considered to be an important factor that influences the wear of ceramic materials<sup>13,18</sup>. At room temperature, the worn surfaces of the conventional  $Al_2O_3$ -3 Wt per cent  $TiO_2$  coating were covered with a compact layer consisting of silicon oxide for the tests performed in an atmosphere with a humidity level of 40-50 per cent. Compared with alumina, silicon oxide is softer<sup>17</sup>. This softer layer was effective in distributing contact stress, and the shear and slipping in the soft layer could act to accommodate the velocity mismatch between the two wear surfaces. Thus, the presence of the softer layer on the worn surface prevented the coating from undergoing brittle fracture.

Consequently, the coating wear at room temperature was very less but, fatigue delamination occurred along the splat boundaries in coatings under the cyclic stress due to the strong adhesion between the transferred layer and the coating. As for silicon nitride, the humidity in the atmosphere affects the formation of oxides. The activation energies of reactivity of silicon nitride with wet oxygen and dry oxygen are  $488 \pm 30 \text{ kJ}\cdot\text{mol}^{-1}$  and  $375 \pm 25 \text{ kJ}\cdot\text{mol}^{-1}$ , respectively, and the oxidation rate of  $Si_3N_4$  is much more accelerated in the presence of water vapours<sup>22</sup>.

Another effect of humidity is to increase the adhesion between the wear debris particles and the worn surface/wear debris particles<sup>23-25</sup>, which is beneficial to compact the particles to form a

dense layer. The adsorption tendency of water is reduced as the temperature is increased. As a result, the worn surfaces of conventional  $Al_2O_3$ -3 Wt per cent  $TiO_2$  coating were not covered with the compact layer at 100 °C and the friction stress could not be distributed. Brittle fracture occurred on the worn surface of the coating, and alumina wear debris particles were produced. However, adsorbed water on the surface cannot be completely removed<sup>26</sup> 100 °C. Therefore, the wear debris still remained a part of its adhesive capability and was trapped on the wear track.

During the subsequent sliding, the wear debris that remained on the wear track was subjected to continuous fracture and was composed of fine particles. For temperatures up to 400 °C, the adhesion capability of the wear debris further decreased. Under the effect of centrifugal force, the wear debris particles could be swept away from the contact zone after these were produced, and further fracture of the wear debris could not happen. Thus, wear debris consisted of coarse particles at 400 °C.

It is generally assumed that the presence of wear debris in the contact zone increases wear. But, this is far from truth in dry friction, especially for ceramic materials<sup>27,28</sup>. Fretting experiments have clearly shown that wear debris decreases the wear of materials<sup>27</sup>. Compared with ceramic materials, wear debris is looser and can also respond kinematically to the velocity mismatch, and accordingly, causes less damage than that occasioned by locked asperities of the mated material with identical hardness<sup>28</sup>. In this study, the hardness of the  $Si_3N_4$  ball was about 1600 HV<sub>0.2</sub>, which is much higher than that of the coating (830 HV<sub>0.2</sub>). Thus, the wear of the coatings from the particle abrasion was less than that from the direct contact between the sliding surfaces, though loose wear debris particles could not protect the coating surface at 100 °C as a compact layer did at the room temperature. As expected, the brittle fracture enhanced with an increase in the temperature, and accordingly, the wear rates of the conventional  $Al_2O_3$ -3 Wt per cent  $TiO_2$  coating also increased with temperature due to the desorption of moisture.

Wear resistance of the materials is closely related to their microhardness and microstructure,

such as porosity and grain size. A high hardness, low porosity, and small grain size are desirable to improve the wear resistance of polycrystalline ceramics<sup>5,29,30</sup>. On the worn surfaces of the coating, the existence of pits indicate that brittle fracture mostly occurs along the grain boundary, which is directly related to the strength of grain boundaries<sup>8</sup>. The strength of grain boundaries is weakened by the residual stresses from the thermal expansion/elastic anisotropy. According to the Hall-Petch relationship, the stresses increase with the grain size, resulting in higher stress in the grain boundaries of coarser grain size materials and an increasing tendency for intercrystalline fracture<sup>29</sup>. Below 700 °C, the increase in the wear rate of alumina with grain size was interpreted in term of decrease in strength of grain boundaries with grain size<sup>8</sup>.

## 5. CONCLUSIONS

In this study, wear tests at elevated temperatures have been conducted for both conventional  $Al_2O_3$ -3 Wt per cent  $TiO_2$  coatings. The wear rates of coating sliding against a silicon nitride ball increased with an increase of temperature in the range room temperature to 600 °C. At room temperature, the wear of coating was too low to be measure of using the test condition because a layer consisting of oxidation products of silicon nitride appeared on the worn surface of both the coatings. When the temperature was increased up to 100 °C, brittle fracture occurred on the worn surface of the coating, which was enhanced with the increase in temperature due to the desorption of the moisture on the worn surface.

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