Microstructure & Tribological Performance of Alumina-3wt% Titania Coatings Produced by APS

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Abstract:
Al2O3-3wt%TiO2 coatings were deposited by atmospheric plasma spraying (APS). The microstructure and phase composition of the coatings were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The wear and friction properties of Alumina–3wt.% titania coatings against a steel ball under dry friction conditions were examined. The microstructure result and the phase of the spray coating is analyzed and presented. In addition, wear behavior of the sprayed coating are evaluated for final coating performance. Test results showed that increasing some process parameters increased the performance of mechanical properties of coating and gave the lowest friction coefficient value of coating.

Keywords:
Plasma spray
Alumina–3wt.% Titania Coatings
Microstructure
XRD, SEM
Wear behavior.

1. Introduction

Plasma-sprayed ceramic coatings such as Al2O3– TiO2 coatings have been widely applied to structural materials and various machine parts in order to improve resistances to wear, corrosion, oxidation, erosion, and heat [1,2,3]. Plasma Thermal spraying techniques are coating processes in which melted or heated materials are sprayed onto a surface. Thermal spray coatings have a wide range of applications, for instance, by repairing machine parts damaged in service or by the production of parts with high wear resistance [4,5,6]. The Atmospheric Plasma Spraying (APS) is one of these processes. Plasma sprayed alumina–titania ceramic is one of the materials largely used in the APS process [7]. It is known for its wear, corrosion, and erosion resistance applications. In order to advance understanding the relationships between the microstructure and wear resistance of the plasma-sprayed coatings, an Al2O3-3wt% TiO2 coating was prepared by plasma spraying and its tribological behaviors against a steel ball under dry conditions was examined.

2. Experimental procedure

2.1. Materials & coating deposition

An atmospheric plasma spraying system (Sulzer-Metco 9MC equipment) was used to deposit the Al2O3-3%wt TiO2 coatings. Using two powder feeder containers, the powders were sprayed in sequence, by following (Ni-20wt%Cr)6Al powders as a bond coat and finally Al2O3-3%wtTiO2 powder with particle size of -45+15μm as a top coating. In the spraying process, three passes were sprayed for (Ni-20wt%Cr)6Al and 8 passes for Al2O3-3%wtTiO2 coating. After coating, the samples were cooled in room temperature in order to avoid internal stress occurred to the coating. Finally, the samples were collected for analysis. Plasma primary and auxiliary gases were Ar and H2: N2 was used as carrier gas. The substrates were stainless steel and prior to the plasma spraying, were degreased and grit blasted. The substrate was grit-blasted with corundum at a pressure of 3.2 bar and cleaned using ethanol in order to remove remaining dust or grease from the surface.
During the process, the material to be deposited is injected in powder form using argon as carrier gas. The main spraying parameters are listed in Table 1.

**Table 1. Plasma spraying conditions**

| APS Parameters                  | Bond Coat | Powder Coating |
|---------------------------------|-----------|----------------|
| Arc Current, A                  | 600       | 650            |
| Arc Voltage, V                  | 60        | 50             |
| Primary gas (Argon) flow rate   | 25        | 80 lpm         |
| Secondary gas (He) flow rate    | 45        | 50 lpm         |
| Powder carrier gas (Ar) flow rate| 90       | 30 lpm         |
| Powder flow rate                | 20        | 20 gpm         |
| Spray distance                  | 100       | 100 mm         |
| Passes, layer                   | 3         | 8              |
| Spray angle, °                  | 90°        | 90°            |

**Table 2. Chemical composition of AISI 304**

| Elements | % wt |
|----------|------|
| C        | 0.052|
| Si       | 0.75 |
| Mn       | 2.00 |
| P        | 0.045|
| S        | 0.030|
| Cr       | 17.5-19.5|
| Ni       | 8.0-10.5|
| Co       | 0.122|
| Cu       | 0.375|
| Fe       | Bal |

2.2. Coating Characterization

The phase and microstructure analysis of the coating samples were measured using XRD Machine and Scanning Electron Microscopy (SEM). The wear tests were carried out on a sliding, reciprocating and vibrating test machine (SRV). The wear test mode is the reciprocal motion of a steel ball against a disc, as illustrated in Fig.1. The upper ball specimen bearing a normal load vibrates against the lower stationary disc specimen. The wear tests were performed under four applied loads (20, 40, 60 and 80 N), with a slip amplitude of 1.4 mm, a frequency of 30 Hz and a period of 20 min. For all wear experiments, the samples were unlubricated; the tests were conducted at an ambient temperature of 100°C and relative humidity of 60 ~ 70 %. The ball specimens were composed of steel with a diameter of 10 mm and the disc specimen were Al₂O₃-3wt% TiO₂ coated steel substrates with dimensions of 20x20x5 mm.

3. Results & Discussion

3.1. Morphology and structure

The samples with different setting parameters were observed for the coating morphology. Fig.2 shows an example of morphology of coated samples of coating surface. The coating features observe the molten particles condition and spread as out on top of the surface to develop coating layers. Some areas on the surface appear as semi-molten particles and they agglomerate together with molten particle to form coating layers [2,8,9]. The semi-molten particles exhibit pinholes, which are characteristic of porosity, occurred on the coated sample. The occurred porosity also may be due to the lamellae structure, which exhibit molten and semi-molten particles and will create pinholes inside the coating. The porosity may occur due to absorbed gases during spraying process. In this study, it is identified that the average porosity of the coating samples are 8.1%. Less porosity will produce better structure and bonding between the individual layers of coating. It also results in increase of density, hardness, adhesion strength and wear resistance of the coating [10,11].

![Figure 1. The schematic diagram of the SRV tester.](image1)

![Figure 2. The morphology of coating surface scanning with SEM at magnification 100µm.](image2)
When the molten particle of Al₂O₃-3wt%TiO₂ impacted the substrate, it spreads and solidifies rapidly and formed a coating. The bonding effect of particles in forming a coating is due to mechanical interlocking, chemical reaction and partial fusion of the contact surface and will lead to mechanical adherence.

As shown in Fig.4, all Al₂O₃-3wt%TiO₂ coating predominately contains γ-Al₂O₃ (Gamma alumina) coexisting with α-Al₂O₃. In view of the nucleation kinetics, under-cooling of the α-Al₂O₃ phase, resulting liquid droplets led to the nucleation of γ-Al₂O₃ nucleated rather than of α-Al₂O₃. This occurs because of lower interfacial energy between crystal and liquid [12]. Cooling rate after solidification was rapid enough to prevent subsequent transformation to α-Al₂O₃. The presence of α-Al₂O₃ in the Al₂O₃-3wt%TiO₂ coating is due to the incorporation of unmelted particles during coating process [10,11].

Fig.5 shows the steady-state friction coefficients of the nanostructured and conventional Al₂O₃-3wt% TiO₂ coatings against a steel ball under dry sliding conditions. The results showed that the friction coefficients of coatings were similar at all test loads and exhibited no great change with increasing contact load under unlubricated conditions. The friction coefficients of the Al₂O₃-3wt.%TiO₂ coatings were similar and about 0.51.

4. Conclusions

Al₂O₃-3wt.%TiO₂ coatings were deposited by atmospheric plasma spraying. Microstructure and phase properties of the as-sprayed coatings were characterized.

The difference in microstructure and properties of the coatings led to different tribological behaviours.
The Al$_2$O$_3$-3wt.% TiO$_2$ coatings contained both equiaxed α-Al$_2$O$_3$ and γ-Al$_2$O$_3$. Moreover, the coating possessed a more homogeneous microstructure and TiO$_2$ phases is rutile in this coating, which is due to the reaction between TiO$_2$ and Al$_2$O$_3$ particles during plasma spraying.

In addition, the coating possessed an improved wear resistance; it was gradually increased with increasing load. Although the friction coefficient exhibited no variation with increasing contact load.

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