EFFECT OF PARTICLE SIZE AND BONDING LAYER ON PLASMA SPRAYED Al$_2$O$_3$-13\%TiO$_2$ COATINGS

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ABSTRACT
To date, plasma sprayed alumina titania have been widely used as wear resistance coatings in textile, machinery and printing industries. Previous studies showed that the coating microstructures and properties were strongly depended on various parameters such as ceramic composition, grain size powders and spray parameters, thus, affecting the melting degree of the alumina titania during the deposition process. This paper discusses the effect of Al$_2$O$_3$-13\% TiO$_2$ particle size powders and the presence of Ni-Al as a bonding layer on commercial marine grade mild steels using a plasma spray technique. The optimum plasma spray parameters were identified for both nanoparticle and microparticle powders in order the get the best coating properties in term of microhardness, surface roughness, coating porosity and specific wear rate. It was found that microparticle powders exhibited better surface roughness than the nanoparticle powders. On the other hand, the nanoparticle powder coating gave an excellent microhardness, with less coating porosity and better wears resistance than those of microparticle powders, which was likely due to the microstructure that consisted of bi-modal structures, resulting greater strengthening effect on both fully and partially melted regions. By sacrificing the surface smoothness, the presence of Ni-Al bonding layer on the other hand, was able to generate a good quality coating with less porosity percentage.

Keywords: Al$_2$O$_3$-13\%TiO$_2$, bonding layer, plasma spray.

1. INTRODUCTION
Atmospheric plasma spraying is an important industrial technique for depositing protective coatings to improve component performance in wear, corrosion, thermal barrier and electrical insulation. Plasma sprayed alumina titania has been widely used as wear resistance coatings in textile, machinery and printing industries (Wang et al. 2007). Attractive properties associated with ceramic coating have been documented for bulk materials. It is also anticipated that if properly deposited, ceramic coating could also provide improved properties for various applications. Early researches on these kind of coatings were only limited to the microstructure and phase transformation studies. The focus was more on the mechanical properties and their relationship with the microstructures (Tian et al., 2009). Previous studies have shown that the coating microstructure and properties were strongly depended on various parameters such as ceramic composition, grain size powder, spray parameters and powder manufacturing method of the ceramic (Habib et al., 2006; Jordan et al., 2001; Zhang et al., 2007) thus, giving great influence on its melting degree.

In recent years, nanoparticle powders were used in plasma spray. These powders could form coatings with both partially and fully melted regions. This bi-modal structure could provide better mechanical properties particularly in wear and crack growth resistance, as well as increasing the adhesion strength between substrates and coating (Wang et al., 2009a; Zhang et al., 2008). However the process must be carefully controlled in order to retain the nanostructure in the final coating.

The aim of this study focuses on the particle size of alumina-13\%wt titania and the presence of a bonding layer on the mechanical properties. The effect of the microstructures on the wear resistance, the microhardness and the average surface roughness were also discussed.

2. EXPERIMENTAL PROCEDURE
Two types of Al$_2$O$_3$-13\%TiO$_2$ powders were used in this study. They were Al$_2$O$_3$-13\%TiO$_2$ microparticles with grain size of 10 to 25 \textmu m from HAI Advanced Material and nanoparticles powder with the agglomerated size of 20 to 60 \textmu m obtained from Inframat Advanced Materials. The steel substrates with a dimension of 50 mm x 30 mm x 4 mm were sectioned in accordance to ASTM A36 type steel. The surface of the substrate was sandblasted prior to coating. The Al$_2$O$_3$-13\%TiO$_2$ powders in this study were deposited on the substrates by using a SG 100 Plasma Gun from Praxair spray systems that was mounted on a programmable robot. The spray parameters for the coating process were shown in Table 1.

The hardness of the coatings was measured using a HMV-2T Vickers microhardness tester by applying a
300 g load on the cross section of samples for 15 s. The readings were taken at 15 different locations for an average value. Abrasive wear test was conducted by using a pin on disc tester model TR-20LE from Ducom. The samples also known as pins with 10.0 mm in diameter, slide on 120 grit SiC sand paper under 5 N load in a dry condition. The test was conducted for 20 m sliding distance. After the test, samples were ultrasonically cleaned with ethanol, dried and were then measured for weight loss. The surface roughness was measured by a SV-C3100 (Formtracer) from Mitutoyo with the length of 15mm with a pitch of 0.001 mm and a speed of 2.0 mm/sec.

3. RESULTS AND DISCUSSION
3.1 Coating Microstructure and Surface Morphology

Figure 1 shows the backscattered SEM image of Al$_2$O$_3$-13%TiO$_2$ micro and nanoparticles. The agglomerated nanoparticles were spherical in shape with lengths from 20 to 60 µm while the microparticles were irregular with length from 10 to 25 µm. The difference in the powder morphology is due to the different powder manufacturing processes (Wang et al., 2007). Even though they have different morphologies alumina and titania were seen evenly distributed as shown in Figure 1.

The SEM micrographs of coating surfaces were shown in Figure 2. It was proven that the Al$_2$O$_3$-13%TiO$_2$ coatings exhibited splat shapes. The structure was formed during the plasma spray, whereby the molten droplets hit the substrate at a high velocity resulting splat morphology (Singh et al., 2011). The splat size of the microparticle was found slightly smaller than the nanoparticles due to density difference. By having small droplet splats, microparticles showed an increase in voids and pores due to the splat boundaries which appeared dark areas as shown in Figure 2 (a). This finding was in a good agreement with Dejang et al. (2010) and Wang et al. (2009b). Figure 3(a) and (b) show the cross sections of the of micro and nanoparticle coatings. The microparticles exhibited greater randomly distributed pores within the partially melted region. This was most likely due to an imperfect contact within partially melted ceramic particles or gas entrapment formation (Du et al., 2005). In contrast, the nanoparticle powders possessed coatings with little pores in which the partially melted and fully melted regions were alternately piled-up. In some areas the two regions were mixed together without distinct boundaries as shown in high magnification SEM images in Figure 3(c). According to previous studies, the bi-modal microstructure was formed due to selective melting of TiO$_2$ nanoparticles during plasma spray (Hassanuddin et al., 2011). With low conductivity and high porosity of agglomerated nanoparticle powders, the heat surface during plasma spray was difficult to transfer into the powder’s interior (Wang et al., 2009a) making the nanoparticle remained inside the coating structure. This microstructure gave a better coating performance and was consistent with several established studies on nanostructures coatings produced by the nanoparticle powders (Gell et al., 2001; Hassanuddin et al., 2011; Jordan et al., 2005; Zhang et al., 2008).

<table>
<thead>
<tr>
<th>Spray parameters</th>
<th>Unit</th>
<th>Bonding layer</th>
<th>Al$_2$O$_3$-13%TiO$_2$ layer</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>M1</td>
<td>M2</td>
</tr>
<tr>
<td>Plasma power</td>
<td>kW</td>
<td>30</td>
<td>35</td>
</tr>
<tr>
<td>Primary gas pressure</td>
<td>Psi</td>
<td>50</td>
<td>100</td>
</tr>
<tr>
<td>Secondary gas pressure</td>
<td>Psi</td>
<td>60</td>
<td>60</td>
</tr>
<tr>
<td>Carrier gas pressure</td>
<td>Psi</td>
<td>30</td>
<td>40</td>
</tr>
<tr>
<td>Powder feed rate</td>
<td>Rpm</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Coating speed</td>
<td>mm/s</td>
<td>250</td>
<td>250</td>
</tr>
<tr>
<td>Layer</td>
<td>Cycle</td>
<td>2</td>
<td>10</td>
</tr>
<tr>
<td>Pre-heat</td>
<td>Cycle</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Spray distance</td>
<td>mm</td>
<td>100</td>
<td>100</td>
</tr>
</tbody>
</table>

*M1 - Microparticle powder without bonding layer.
M2 - Microparticle powder with bonding layer.
N1 - Nanoparticle without bonding layer.
N2 - Nanoparticle with bonding layer.
The decreasing percentage of pores contributed by both micro and nanoparticle powders were confirmed based on image analysis measurement as shown in Table 2. Coatings from microparticle powders exhibited 8.6% of porosity, while nanoparticle coatings exhibited 5.0% of porosity. The presence of the bonding layer decreased the coating porosity as shown in sample M2 and N2 (Table 2) as a result of better thermal expansion between the bonding layer and Al$_2$O$_3$-13%TiO$_2$ coating. These pores and micro cracks were formed due to the tensile residual stress (quenching stress) that generated during a rapid cooling process of the droplets when spreading out over the substrate (Wang et al., 2009b). The presence of the bonding layer increased the surface roughness of the coating either by using microparticle or nanoparticle powders. This was due to the particle size of the bonding layer that relatively large and produced coarser surface (Yilmaz et al., 2009). Fig. 5 shows the coefficient of friction ($\mu$) evolution as function of sliding distance. The plot can distinguish two regions called running-in and stabilization regions which related to different wear mechanisms. As the roughness of the materials is reduced in the running-in period, the friction coefficient increases due to increase of the contact surface. Then, the value of the coefficient of friction stabilizes representing the wear behavior of the considered material couple (Guessasma et al. 2006). The running and stabilization regions were similar when varying each of the samples which demonstrate that friction mechanisms were the same for all the samples.

Table 2: Percentage of pores, surface roughness and microhardness of the coating prepared with and without Ni-Al bonding layer.

<table>
<thead>
<tr>
<th>Sample</th>
<th>% Pores</th>
<th>$R_a$ (µm)</th>
<th>Microhardness (HV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>M1</td>
<td>8.6</td>
<td>3.70</td>
<td>1013.2</td>
</tr>
<tr>
<td>M2</td>
<td>6.3</td>
<td>4.30</td>
<td>1015.9</td>
</tr>
<tr>
<td>N1</td>
<td>5.0</td>
<td>6.83</td>
<td>1054.7</td>
</tr>
<tr>
<td>N2</td>
<td>3.9</td>
<td>7.70</td>
<td>1082.5</td>
</tr>
</tbody>
</table>
Figure 3 SEM images of coatings by using (a) microparticle powders (b) nanoparticle powder sand (c) high magnification of nanoparticle coating.

Figure 4 Specific wear rates ($K'$) of optimised plasma sprayed coatings.

However, in the beginning of the test, the coefficient of friction of the microparticle coating was about 0.8, whereas the coefficient of friction of nanoparticle powders was about 0.7. The value was then stabilized as the sliding distance more than 0.5 m for nanoparticle coating. While microparticles coating was then stabilized after sliding distance 2.5 m. These results were probably due to coating microstructure and porosity level as describe by Bounazef et al. (2004).

Figure 5 The coefficient of friction of the coating prepared by using the optimum parameters of plasma spray.

4. CONCLUSION

Different particle size of microparticle and agglomerated nanoparticle Al$_2$O$_3$-13% wt TiO$_2$ powder were successfully deposited by using plasma spray method. The behaviors of these coatings were investigated and the following conclusions are made.

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The microparticle powder was found to produce coatings with small droplet forming splotches compared to the nanoparticles, thus increased the voids or pores of the splot boundaries. The cross sections of the coatings showed that the microparticles produced higher pores with 8.6% compared to the nanoparticle coating which only generated 5% pores. The presence of bonding layer had reduced the pores and voids of both microparticle and nanoparticle coatings.

Microparticle coatings also gave better surface roughness than the nanoparticle coatings. In contrast, the presence of the bonding layer exhibited rougher surfaces.

Using the best plasma spray parameters on both types of powder coatings showed that the agglomerated nanoparticle powders (N1) coatings possessed higher microhardness than the microparticle powder (M1) coatings.

However, the wear rate of the coatings did not show any significant difference on all types of coatings. The coefficient of friction also suggested that friction mechanisms were the same for both nanoparticle and microparticle coatings.

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