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Short Communication

Some Microstructural Studies of Plasma-sprayed Stainless Steel 316L/ Al_2O_3 - TiO_2 Produced Using Two-Powder Port Configuration

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ABSTRACT

This study concerns the development of a plasma spraying technique, in particular the 2-powder port setup which allows 2 different types of feedstock to be fed separately and sprayed at the same time resulting in the fabrication of a composite coating. The materials used to produce the composite coating are stainless steel 316L and Al_2O_3 - TiO_2 . Stainless steel 316L can act as a binder and provide the coating with necessary toughness. Al_2O_3 - TiO_2 is included to provide the coating with improved hardness. The contrast in the physical and thermal properties of the two materials poses some difficulties in plasma spraying. The resulted coatings were compared with those produced from pre-mixed powders (dry milling and wet milling) with respect to their microstructures, hardness and the deposition efficiency of the process. It was found that by adjusting the powder feed rates and the positions of the powder ports, composite coatings with good integrity can be fabricated. The hardness of the coating varies with the proportion of the Al_2O_3 - TiO_2 phase, which in turn is affected by the powder feed rates. A coating containing approximately 70 vol.% Al_2O_3 - TiO_2 having low porosity and moderate amount of unmelted particles with a hardness of 259 BHN and a relatively high deposition efficiency can be produced using a 2-port setup employing feed rates of 15 and 25 g/min. for Al_2O_3 - TiO_2 and stainless steel 316L, respectively.

Keywords: plasma spraying technique, composite coating, Al_2O_3 - TiO_2 , wear resistant coating

1. INTRODUCTION

Wear damage is an underlying problem in many industries such as automotive, petrochemical, energy, textile, metal and machinery works, etc. The cost of wear is often perceived through the expense in maintenance activities. The capital drain also lies in indirect cost such as down-times of the facilities, compromised qualities of the goods

and unexpected breakdowns which may lead to breaching of customers' confidence. Although it is unlikely that wear can be eliminated from industries where operations include relative movements of solid components, it can be significantly reduced using modern surface technologies [1,2] to the point that the benefits off-set the cost of the

technologies.

Air plasma spraying (APS) is a surface coating technology that has been successfully employed to combat against wear in many applications [3-6]. The maximum temperature of a plasma jet can reach above 10,000°C [3,7]. Feedstock in the form of powder is fed into this plasma jet, where it partially melts. The pressure from the plasma jet propelled the partially molten droplets onto a substrate where the droplets deform upon impingement, thus build up a coating of a lamellar structure [8].

For a wear-resistant coating, the choice of coating materials depends largely on its intended application [9-14]. Composite materials are generally preferable due to their high hardness, tensile strength and fracture toughness [15]. In Thailand, however, such composite powders are imported as specialized wear-resistant materials at high cost. The high powder price is due partly to the processing of the composite powder deeming it unsuitable in some applications.

This study was therefore carried out with an aim to produce a hard composite coating for use in wear resistant applications. The coating was produced from 2 separate powders, namely 316L stainless steel and $\text{Al}_2\text{O}_3\text{-TiO}_2$. Microstructures and some properties of coatings produced from the two separate powders were compared with those of the coatings produced from pre-mixed powders.

2. MATERIALS AND METHODS

In this study, AISI 304 stainless steel was used as the substrate sample. The dimensions of the samples were 5.5 mm thick and 25.4 mm in diameter. The sample was grit blasted on one flat surface using 15-45 mm Al_2O_3 grit and a compressed air pressure of 7 bars. The grit blasting process is a surface preparation technique commonly used in

industries prior to the plasma spraying process in order to enhance the coating adhesion by means of contamination removal and interfacial anchorage. The surface roughness of the samples after the grit blasting process was between 5 and 7 mmRa.

The plasma spraying process was then carried out using 3MBII Sulzer Metco plasma spray gun and MNC controller. Two types of powder; 316L stainless steel and $\text{Al}_2\text{O}_3\text{-TiO}_2$ were used as coating materials. The 316L stainless steel was M-683.33 powder (Fe(bal.)-16.6Cr-11.2Ni-2.3Mo wt.%) from Flame Spray Technology and was 25-53 mm in size. The $\text{AlO}_3\text{-TiO}_2$ was DURMAT 608.11 powder (Al_2O_3 (bal.)-42TiO₂ wt.%) from Durum and was 15-45 mm in size. The coating samples were classed into 7 groups according to the powder preparations. The dry milled sample was produced from a dry milled powder of 50%316L+50% Al_2O_3 (wt.) composition using a milling rotation speed of 26 rev/min. for 2 hours. The wet milled sample was produced from a wet milled powder produced using the same composition and rotational speed as the dry milled sample with PVA binder added. After the wet milling, the powder was dried at 50°C for 12 hours. Both the dry milled and the wet milled powders were sprayed with a standard single powder port using 38 g/min. feed rate and 80 psi air pressure. On the other hand, II-Port (1),(2),(3), (4), and (5) samples were sprayed using 2 port configuration without pre-mixing of the powders as shown in Figure 1. The powder feeder parameters are shown in Table 1. The plasma spraying parameters used are as follows; 3.5 inch spray distance, 70 V and 500A gun input, 50 psi of H_2 and 100 psi of Ar.

The coatings were produced to achieve a thickness of between 250 and 300 μm . The as-sprayed coatings were then cross-sectioned and polished for the microstructural study

using a scanning electron microscope (SEM). Porosities and percentages of 316L stainless steel and Al_2O_3 - TiO_2 in the coating were determined using an image analysis technique (Image Pro Plus version 5.1). Hardness values of the samples were also measured using Brinell hardness measurement on a polished

planar surface. The Brinell ball diameter was 2.5 mm and 62.5 kg load was used.

3. RESULTS AND DISCUSSION

Micrographs of the cross sections of the samples are shown in Figure 2. The coating structure consists of 3 major phases

Table 1. Powder feeder parameters.

Powder feeder parameters	316L stainless steel		Al_2O_3 - TiO_2	
	Feed rate (g/min.)	Air pressure (Psi)	Feed rate (g/min.)	Air pressure (Psi)
Dry milled	Standard single powder port using 38 g/min. feed rate and 80 Psi air pressure.			
Wet milled				
II-Port (1)	25	130	25	80
II-Port (2)	25	130	15	80
II-Port (3)	25	130	10	80
II-Port (4)	25	130	5	80
II-Port (5)	25	60	25	100

Table 2. %DE, hardness, porosity and proportion of phases of the coating samples.

Sample	%De	Brinell hardness (BHN)	%Vol		
			Porosity	SS316L	Al_2O_3 - TiO_2
Dry milled	30.7 %	233.4 ± 14.9	0.00 ± 0.00	29.7	70.3
Wet milled	23.7 %	227.5 ± 20.7	0.08 ± 0.06	30.6	69.3
II-Port (1)	-	271.5 ± 23.2	0.27 ± 0.17	12.1	87.6
II-Port (2)	52.1%	258.7 ± 32.2	0.13 ± 0.10	30.9	68.9
II-Port (3)	20.2%	274.1 ± 13.4	0.02 ± 0.00	32.2	67.8
II-Port (4)	26.6%	231.1 ± 19.5	0.07 ± 0.03	57.1	42.8
II-Port (5)	54.3%	193.6 ± 16.7	0.03 ± 0.02	79.1	20.9

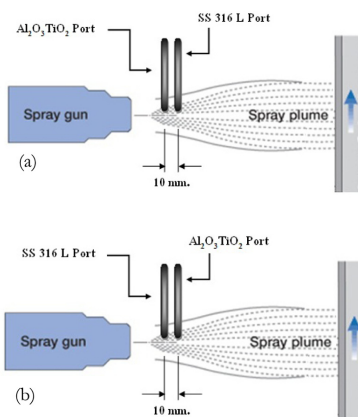


Figure 1. Powder port configuration for sample (a) II-Port (1),(2),(3) and (4), and (b) II-Port (5).

namely SS316L, Al_2O_3 and TiO_2 . The phases can be distinguished under an SEM by their deviation in the colour shade. The quantity of each phase varies according to the coating fabrication method. The 3 phases adhere well to one another; this can be assumed due to the absence of any intergranular porosity. The 3 phases pile on top of one another forming a coating with a lamellar structure.

A very small amount of (Fe,Cr) oxide formed from SS316 during plasma spraying can also be observed in some regions in all samples. The amount of this oxide is minimal due to the short time the powder particle spent

at high temperature and therefore cannot be analyzed accurately using a quantitative technique such as Image analysis. For the purpose of this paper, it can be correctly stated that the percentage of (Fe,Cr) oxide is lower than 0.1% by volume in all samples.

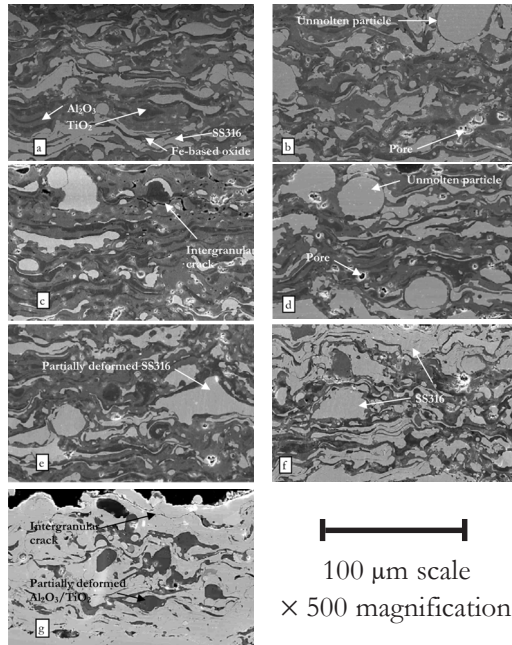


Figure 2. SEM micrographs of the coating samples; a) dry milled, b) wet milled, c) II-Port (1), d) II-Port (2), e) II-Port (3), f) II-Port (4) and g) II-Port (5).

A small amount of porosity, typical of the plasma spraying process, is also evident in all samples. The volume percentage of the porosity measured using an image analysis technique is shown in Table 2. Due to the uneven distribution of the porosity, the values are presented with large deviations.

Unmelted particles are also present. Unmelted particles are generally undesirable and can occur if a powder particle underwent a low degree of melting during plasma spraying, thus could not significantly deformed when it impinged upon the substrate surface but nevertheless remained within the coating as a non-flattened particle.

Observations on individual samples

show that the dry milled sample possesses a structure with evenly distributed phases, a minute amount of porosity and a small amount of unmelted particles, see Figure 2(a) and Table 2. The coating splats reveal a high degree of deformation essential for good bonding. There are no cracks observed in the coating and at the substrate interface. The volume percentage of the hard $\text{Al}_2\text{O}_3\text{-TiO}_2$ phase measured using an image analysis technique is approximately 50.

The wet milled sample generally exhibits well distributed phases. There are, however, a higher proportion of the unmelted particles compared to the previous sample; see Figure 2(b). There are also some large pores a few microns in diameter. The presence of the unmelted particles and the pores is due to the coarseness of the spraying powder as a result of the wet milling process. During plasma spraying, the powder absorbs a large amount of heat from the plasma jet and transforms to a partially molten state. In this case, the larger powder limits the heat that can be absorbed per unit volume of powder. The powder transformed to a partially molten state on the outside but the inside of the grain remained solid. Upon impingement, the solid core became the unmelted particle. If the spraying powder contained some pores, this too may remain in the coating.

The II-Port (1) sample shows a structure largely consisting of $\text{Al}_2\text{O}_3\text{-TiO}_2$, approximately 87.6% by volume, see Figure 2(c) and Table 2. This is because in this plasma spraying process, the powder ports were setup such that the $\text{Al}_2\text{O}_3\text{-TiO}_2$ port at the feed rate of 25 g/min. was optimized for the spray plume. When SS316L was fed through the second port, the heat had already been mostly consumed in melting the $\text{Al}_2\text{O}_3\text{-TiO}_2$ powder thus most of the SS316L powder could not melt and therefore could not readily bond to the underlying coating

upon impingement resulting in the coating containing a small amount of SS316L phase.

For II-Port (2), II-Port (3) and II-Port (4) samples, the feed rates of the Al_2O_3 - TiO_2 powder were reduced to 15, 10 and 5 g/min., respectively. This reduction results in samples containing more SS316L phase at 30.9, 32.2 and 57.1 vol.%, respectively. The unmelted particles are still present in large numbers due to the non-optimized powder port position, i.e. the SS316L powder has less time in the plasma plume than if it were fed through the first port, see Figure 1(a).

For II-Port (5), the powder ports were swapped so SS316L was fed through the first port. The air pressures in both powder feeders were adjusted in order to maintain the powder stream within the plasma plume. In this case, SS316L entered the plasma at an optimum position resulting in a well deformed structure with a low number of the unmelted particle. The Al_2O_3 - TiO_2 on the other hand did not melt well and remained largely as unmelted particles. Most of the Al_2O_3 - TiO_2 powder was also lost during spraying resulting in a low volume percentage of Al_2O_3 - TiO_2 at 20.9%.

When one considers the physical properties of the coatings, it can be seen that the dry milled sample possesses a good uniform structure. This coating, however, requires a pre-mixing process which adds to the cost of the feedstock. II-Port (2), (3) and (4) samples also show coating structures with good integrity, meaning there are no cracks and large amounts of porosity present. The unmelted particles can possibly be reduced by reducing the SS316L powder size. The absence of the powder processing step provides a good advantage with respect to the cost.

The Brinell hardness number is employed as a loose indication of the coating's

resistance to wear. Although the relationship between the surface hardness and its wear properties is not straightforward, it will serve as a guideline for this study. The hardness of the coating is generally proportional to the volume percentage of Al_2O_3 - TiO_2 . This finding is in accordance to previous work by Jiansirisomboon et al. (2006) [16]. Percentage porosity and pore size also affects the hardness although, in this study, not to as great an extent as the Al_2O_3 - TiO_2 . The Brinell hardness number is presented in Table 2. Samples containing a low amount of Al_2O_3 - TiO_2 , such as the II-Port (5) sample, are not suitable as a wear resistant coating.

Deposition efficiency (%DE) is a number expressing the powder usage efficiency. %DE varies with coating parameters including the type of powder, the powder feed rate and the processing technique and its parameters. It is also strongly dependent on the geometry of the substrate. In this study, the size of the substrate sample is much smaller than a typical industrial component affecting the calculated %DE. Hence, the values reported in this paper can serve only as a comparison between the samples and should not be taken as definite data. %DE in Table 2 is calculated as weight of the coating/weight of the powder used $\times 100\%$. When %DE is taken into account, II-Port (2) sample stands out as a spraying condition suitable for producing a coating with good hardness and powder economy. Further work is required to improve the coating structure, particularly to reduce the amount of the unmelted particles, and also to study wear behaviour of this coating in detail.

5. CONCLUSIONS

In this study it was found that a plasma-sprayed stainless steel 316L/ Al_2O_3 - TiO_2

coating can be produced from 2 separate powders of stainless steel 316L and Al_2O_3 - TiO_2 as well as from pre-mixed dry milled and wet milled powders. When the 2-port setup is used to produce the coating from 2 separate powders, the positions of the ports and the powder feed rates affect the proportion of stainless steel 316L and Al_2O_3 - TiO_2 in the coating. A coating containing ~70 vol.% Al_2O_3 - TiO_2 , a hardness of 259 BHN and a relatively high deposition efficiency can be produced using a 2-port setup employing feed rates of 15 and 25 g/min. for Al_2O_3 - TiO_2 and stainless steel 316L, respectively.

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