Structure and Properties of AlSi/Polyester Coating

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Received 12 August 2003; accepted 15 March 2004 DOI 10.1002/app.20782 Published online in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: Powder coatings, which are formed by plasma spray technique, are being used in industrial applications. Resistance of plastics and their based composite materials to chemicals, solvents, atmospheric conditions, and high impact strength even at low service temperature increases the importance of plastic and plastic based coating applications. In this study, aluminum silicon based polyester (AlSi/polyester) coating was applied by plasma spraying technique with and without intermediate bond layer coat (NiAl). The effect of coating thickness, intermediate bond layer coat, and plasma spraying parameters on bond

strength of coating were studied experimentally. The bond strengths of the coatings were determined according to the ASTM C-633–79. Microstructures of the coating were examined by optic microscopy and scanning electron microscopy (SEM), respectively. Obtained results indicated that plasma spraying current rate, coating thickness, and spraying distance were important factors on bond strength of coating. © 2004 Wiley Periodicals, Inc. J Appl Polym Sci 93: 2437–2444, 2004

Key words: coatings; strength; structure–property relations; adhesion

INTRODUCTION

The coating of surfaces with a protective layer is extensively used to improve the surface properties of materials or to protect them for environmental factors. By this purpose, surfaces of coatings are made due to two basic principles. The first one is the coating, which is done by the diffusion bond between the coating surface and substrate. The second one is a mechanical adhesion without forming a diffusion bond between the coating surface and substrate.¹ The properties of coating surfaces, mostly the bond strengths, are directly related to the material compositions in which coatings are formed by diffusion bond. The mechanical adhesion that is not formed with diffusion bond has been changed depending on the coating spraying properties.^{1,2}

A major problem in surface design and coating performance is that many desired properties, such as adhesion between coating and substrate interface, depend strongly on chemical composition, used spraying parameter, and substrate surface.² To obtain very high bond strength, an intermediate bond layer coat must be used to eliminate excessive stress and shrinkage between substrate and layers of coating.¹ Also, the substrates must be cleaned by chemicals and sandblasting to form an anchor pattern that improves adhesion of the sprayed coating layer.² Coating provides some properties to the substrate including wear, corrosion, erosion, optical, magnetic, electric-electronic, biological, thermal attack, and tribologic properties. These properties of coatings are very important in surface engineering applications. It has also an aim of decorative attractiveness for the surfaces.³

Since plastics and their composites have resistance to corrosion, electric insulation, low density, and are durable in high impact resistance even at low service temperature, these properties have increased the importance of coatings in engineering applications. This aim different materials such as metals, ceramics, and plastic powders used as coating materials by flame and plasma spray technique. The usage of flame and plasma spraying in different industrial applications has increased the importance of these types of coatings. The plasma spraying technique has been developed to a high degree of sophistication for metallic and ceramic coating applications, but very little information has been given about plastics, their coatings, and their applications.^{4–8}

As in many spraying techniques, generally the material used during the coating process is in powder form in the plasma spraying method.^{2,3,9} The most important reason for using the plastic based powder in noncritical applications of the coating with plasma spraying technique is the low cost. And the filler and fibers in powder also increase the properties of coating without affecting the coating process.⁷

The factors that determine the coating properties are the constitution of material composition parameters

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Journal of Applied Polymer Science, Vol. 93, 2437–2444 (2004) © 2004 Wiley Periodicals, Inc.

Composition of Coating rowders		
Powder type	NiAl bonding powder	AlSi/polyester powder
Chemical composition (%) Powder dimension morphology Powder dimension ranges (μm) Density (g/cm ³)	95Ni, 5Al Spherical 44–125 3.14	48Al, 12Si, 40 polyester Complex 15–50 2.3

TABLE IComposition of Coating Powders

such as the coating process and thickness, etc. By varying these factors, the properties of resistance to corrosion, flame, bond strength, and wear strength of polymeric based powder coating can be changed and the service life can be increased. The most used plastic powder types in this method are nylon types, polyester, polypropylene (PP), polyethylene (PE), polyetra-floraethylene (PTFE), ethylene vinyl alcohol copolymer (EVA), polysulfon, polyfenilenoxide (PPO) polyester, and polyurethanes.^{1–3}

A major process advantage of this type of coating is combining particle melting, quenching, and consolidation into a single process. Also, resistance to corrosion and the bond strength of the coating are other advantages. Flexible process parameters allow using the material and components under a wide range of industrial applications.^{2,3,9}

The study reported in this article uses the plasma spraying process and explores relations between the microstructure and bond strengths of aluminum silicon polyester powder coating materials. It also investigates the structure and mechanical properties of the resulting coating.

EXPERIMENTAL

Materials

AlSi/Polyester based composite powder used in this study was carried out by the plasma spraying technique. NiAl powder was also used as an intermediate bond layer. Powders used in this study were supplied by Metco (Winterthur, Switzerland). Polyester powder particles have proper morphology; aluminum and silicon particles have a complex morphology. These morphologies have an important role during the coating process to specify the homogeneity of the coating. The composition and properties of powders are listed in Table I. The surface morphologies of the coatings and NiAl bonding coat layer material were researched under light microscopy (LM) and scanning electron microscope (SEM), respectively. Some of these morphologies are shown in Figures 1a and b.

Equipment

The coatings were carried out by automatic controlled plasma spraying machine (METCO 9 MB). Plasma

spraying parameters of coatings are listed in Table II. The coated samples (substrate) were stuck with epoxy based sticker (DEVCON) and cured in the oven at 100°C. Bond strengths of coatings were determined according to the standard of ASTM C-633-79¹⁰ by using Hounsfild universal tensile test machine with a 1 mm/min test speed. The morphology and the structure of the sprayed surfaces were characterized by optical (Prior) and SEM (JEOL JSM 840 model SEM), respectively. Results of these surface evaluations for polymer based coatings with and without the bond layer coat are presented in Figure 2. Their morphologies showing the coating and interface structure are presented in Figures 3-5. Before conducting the SEM investigation, the samples were coated with gold to a thickness of 30 nm. SEM investigation operated at 20 kV, after coating the samples with gold for conductivity.

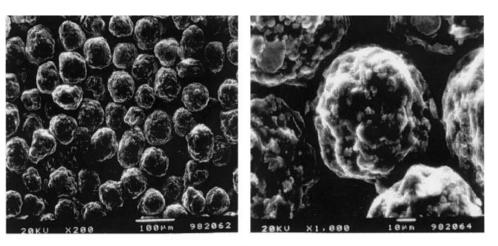
The coating of NiAl and AlSi/polyester powders

Prior to the deposition of AlSi/polyester based composite powder, substrate material was cleaned with trichloroethylene (Merck, Darmstadt, Germany) solvent. After solvent cleaning process, metal substrate was roughened using 30 μ m alumina powder to get better bond strength between substrate and AlSi/ polyester coating layer. The roughened surfaces were preheated (100–150°C). After this process, the coating operation was carried out on prepared substrates with an angle to substrate surface of approximately 60 degrees with and without NiAl bond layer coat. The substrate was controlled by air jet during the plasma spraying process bonding layer coat and top layer coatings. The NiAl bond layer process was carried out in constant parameters. Other parameters were not used (70 V, 500 A). Thickness of the bond layer coat was approximately 0.1 mm. The process parameters that were used during the AlSi/polyester powder coating are listed in Table II. AlSi/polyester coated surfaces were aircooled at indoor temperature after coating process was completed.

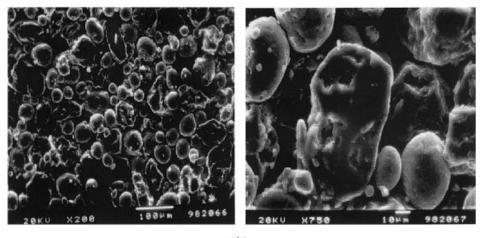
RESULTS AND DISCUSSION

The bond strength of the coatings

The strength of the adhesion between plasmasprayed surface and substrate are extremely impor-



(a)



(b)

Figure 1 Scanning electron morphology of (a) NiAl bonding powder, and (b) AlSi/polyester composite powder.

tant for coating.⁹ The bond strength values measured from the adhesion $test^{10}$ of AlSi/polyester coatings are shown in Figure 2. It is very important to control the spraying parameters, which correspond to bond strength and performance of the coating. Good adhesion plays an important role on mechanical properties that affect the life performance of the coating. According to obtained results, bond strength values measured from the adhesion test of the coating depend on the plasma spraying current and coating thickness. The plasma spraying current is an important parameter that can influence the porosity and the porosity level of the coating material.^{1,11} The ideal coating was carried out at 500 ampere of plasma current rate with NiAl bond layer coat. This may be sufficient adhesion between the substrate, NiAl bond layer, and final coating material (AlSi/polyester). It is clear that while the coatings' thickness is increasing, the bond strength values are decreasing due to excessive stress and

TABLE II		
Parameters of Plasma Spraying Process		
of AlSi/Polyester Coating		

Coating material	Aluminum silicon based polyester powder
Argon flow rate (l/min)	80
Hydrogen flow rate (l/min)	8
Carrier gas flow ratio (argon, 1/min)	4.5
Nozzle diameter (mm)	6
Injector angle	60°
Plasma gun type	METCO 9 MB
Nozzle and electrode	W cathode, Cu anode
Plasma type	$Ar + H_2$
Substrate	SAE 1035 steel
Plasma current (I, ampere)	450, 500, 550
Arc voltage (V)	60, 70
Plasma gun type	$Ar + H_2$
Nozzle distance (mm)	50-90
Powder feed rate (g/min)	30
Coating thickness (mm)	0.5, 1, 1.5, 2

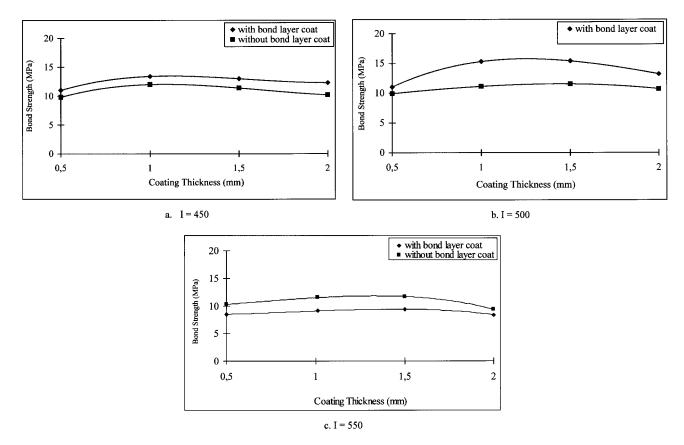


Figure 2 The bonding strengths of plasma sprayed AlSi/polyester coatings: (a) I = 450, (b) I = 500, and (c) I = 550.

shrinkage between substrate and layers of coating. This may be due to slow cooling and exothermic reaction between bond layer coat and top layer coat. NiAl also can reduce cracks in the coating micro structure due to slow cooling^{12,13} The change of bond strength of AlSi/polyester coatings depending on the coating thickness under different plasma current rates with and without bond layer coat are shown in Figures 2a, b, and c.

The bond strengths of the coatings without NiAl bond layer coat are weak due to inefficient adhesion between the substrate and top coating material. For strong adhesion, an intermediate bond layer coat must be used in this type of application. Bond layer coat not only decreases stress but also prevents micro-cracks and provides a better adhesion between the layers of coating, including the substrate. The micro-cracks that occur during fast cooling decrease the bond strength and service performance. Especially when the coating thickness is over 1.5 mm, bond tensile strengths are decreased. The increasing of thickness decreases the bond strength due to stress that is applied to the coating. Thus, stress from the surface to coating inside move with a larger area and the break out with less force. Better results were obtained when plasma current was 500 A. As is shown in Figures 2a, b, and c, the bond strengths of the coatings

strongly depend on thickness and spraying current. All coatings showed high results when thicknesses were 1–1.5 mm. In 450 and 550 plasma spraying coatings, the bond strengths are not strongly dependent on NiAl bond layer coat and indicate the importance of thickness of the coatings. This may be due to very few or an absence of microstructural cracks and gaps depending on the thickness. This is verified by the morphology of surfaces that were taken by SEM (Fig. 5a).

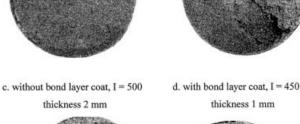
Microstructure of the coatings

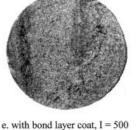
The typical surface morphologies microstructure of coatings are presented in Figures 3b, c, d, e, f, 4a, b, c, d, and 5a, b, c, respectively. The microstructures of the coatings were examined by optical and SEM. Fracture surfaces show adhesive and cohesive fracture types in Figure 3. The evaluation was done according to the fracture surface types. Coatings that were carried out without NiAl bond layer coat are given in Figures 3a, b, and c. These types of coatings had adhesive fracture type tends to cohesive type. In general, the basis of bond strength mechanisms is mechanical adhering in this type of coating.



a. without bond layer coat, I= 450 thickness 1.5 mm







thickness 1.5 mm

f. with bond layer coat, I = 550 thickness 2 mm

thickness 1 mm

b. without bond layer coat, I = 500

thickness 1.5 mm

Figure 3 Light microscopy fracture surface morphologies of AlSi/polyester coatings with and without NiAl bonding layer coat: (a) without bond layer coat, I = 450, thickness 1.5 mm; (b) without bond layer coat, I = 500, thickness 1.5 mm; (c) without bond layer coat, I = 500, thickness 2 mm; (d) with bond layer coat, I = 450, thickness 1 mm; (e) with bond layer coat, I = 500, thickness 1.5 mm; and (f) with bond layer coat, I = 550, thickness 2 mm.

Three of these coatings (in Figs. 3d, e, and f) were performed by using NiAl bond layer coat. However, due to the increase of coating thickness, the tendency can be towards cohesive rather than being adhesive. This is due to increasing of plastic deformation with the thickness increase. Thus, cohesive fracture can occur. The increase of the coating thickness causes inefficient adhesion between bond layer coat and substrate due to increasing large volume toward inside of the coating. If the plasma spraying current rate occurs at 500 ampere and the coating thickness at 1.5 mm, the fracture will occur towards all long surfaces. Figures 3d, e, and f show the fracture surface of coatings that have NiAl bond layer coat. Although it can also be seen from this figure that bond layer coat is used,

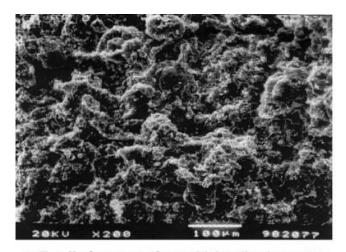
there can still be cohesive fracture occurring locally due to plasma spraying current rate at 450 and 550 ampere.

Without using the NiAl bond coat layer, the cooling of the coating shows an effect of decreasing the mechanical properties. Especially during the coating of material, the fast cooling makes a brittle structure that breaks off faster and makes cracks, gaps, and local plastic deformation during the test examinations. So the microstructure of the coatings was found to be important.

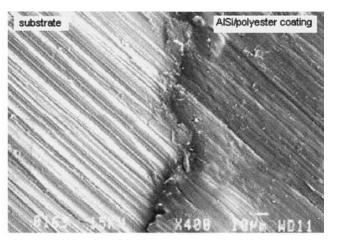
Fracture surfaces of AlSi/polyester coating taken by SEM are given in Figures 4a, b, c, and d, respectively. Figure 4a shows the fracture surface by SEM with NiAl bond layer coat. As it is shown, the surface has ductile structure. With different expansion between the composite coating material phases, the elasticity of the coating structure decreases due to the excessive stress and shrinkage inside of the coating. NiAl bond layer coat provides thermal expansion equilibrium and slow cooling.^{2,11,12} So bond strength of coating is quite obvious. Also, plasma heat is very important on mechanical properties. This is due to efficient wettability and makes the coating homogenous.

The main factor that affects bond strength is micro-cracks. Stress and pores structure can cause nonhomogenous structure in polyester based composites. The coating that has high bond strength indicates the importance of the intermediate bond layer. Because it provides equilibrium to the thermal expansion, it eliminates excessive stress and shrinkage.^{2,12,14} Without NiAl bond layer, coat failure occurred at the coating; substrate interface and surface can be brittle (see Figs. 3a, b, and c). Also, coating indicates cohesive strength by using NiAl bond layer coat, and fracture surface can be ductile (Figs. 3d, e, and f).

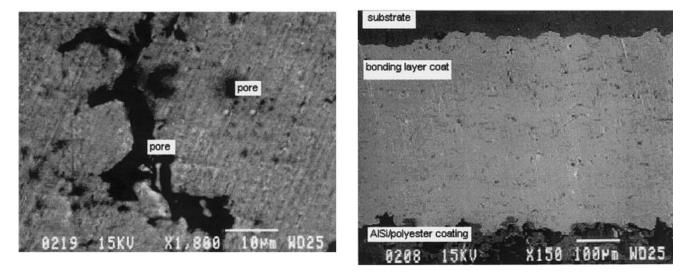
The properties of the coatings are also influenced by spraying distances, plasma current, and thickness.¹³ It is seen that if the thickness of coating gets thicker than 1.5 mm, the bond strength decreases. This can be explained with increasing of the microcracks and excessive stress between coating layers. However, both coatings carried out with the NiAl bond coat layer show micro-cracks in coatings. It is believed that coating performed using bond layer coat has fewer cracks than coating performed without bond layer coat. Increasing of coating thickness increases the porosity in the structure that indicates mechanically weak bonding between substrate, bond layer coat, and top coat (Figs. 4b and c). The bond strengths of the coatings with NiAl bond increases strong mechanical adhesion between the substrate and top coating materials (Fig. 4d). For high bond strengths, an intermediate bond layer must be used in this type of applications. Layers like NiAl not only decrease stress but also prevent mi-



a. Tensile fracture surface, with bonding layer coat



b. without bonding layer coat (cross section)



c. without NiAl bond layer coat, (cross section)

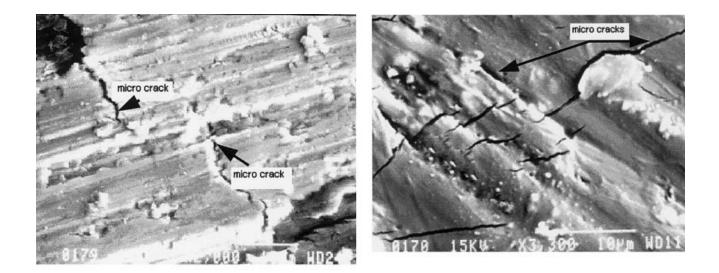
d. with NiAl bond layer coat (cross section)

Figure 4 Scanning electron morphologies and cross section of AlSi/polyester based coating: (a) Tensile fracture surface, with bonding layer coat; (b) Without bonding layer coat (cross section); (c) Without NiAl bond layer coat (cross section); and (d) With NiAl bond layer coat (cross section).

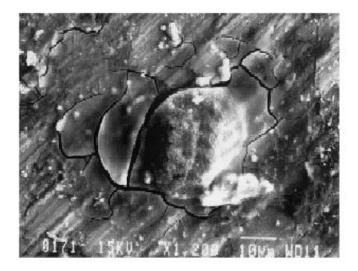
cro-cracks and provide strong mechanical adhering between the surfaces. SEM morphology also showed micro-cracks (Figs. 5a, b, and c). As it is shown in this figure, the adhesion of coating is not well without NiAl bond layer coat. With NiAl bond layer coat, strong adhesion can be determined (Fig. 2b).

When the thickness increases, then the gaps also increase. The distance between the nozzle and substrate material also affects the coating bond strength. It is believed that when the spraying distance increases between nozzle and substrate during the powder particle transfer, the melting of the powder occurs homoge-

neously.¹⁴ The presence of gaps of the coating that were carried out without using bond layer coat are much less than the coatings that were carried out using bond layer coat. It is thought that this is due to not many cracks. From the studies with SEM, the cracks ratio could not be determined properly. Nevertheless, when NiAl bond layer coat is used, the presence of micro-cracks observation gets low (Fig. 5a). The cracks were monitored much more in the AlSi/polyester coating without using NiAl bond layer coat (Figs. 5b and c). So the bond layer coat is a very important factor that determines the properties of coatings.



a. I=500, with NiAl bonding layer coat



c. I=500, without NiAl bonding layer coat

Figure 5 Scanning electron morphologies and micro-cracks of AlSi/polyester coating without NiAl: (a) I=500, with NiAl bonding layer coat; (b) I=500, without NiAl bonding layer coat; and (c) I=500, without NiAl bonding layer coat.

CONCLUSION

The effect of plasma spraying parameters, coating thickness on the bond strength, and morphologies of AlSi/polyester coatings were investigated. The following results were obtained.

AlSi/Polyester powder can be carried out onto metallic surfaces by using plasma spraying technique. The plasma spraying current and thickness of the coating play an important role on bond strength. The presence of NiAl bond layer coat increases the bond strength of coating and decreases micro-cracks and gaps. Bond layer coat affects cohesive fracture. Also, when NiAl is used, ductile fracture occurs. Without NiAl bond layer coat, brittle fracture obtained.

b. I=500, without NiAl bonding layer coat

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