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The effect of roughness and pre-heating of the substrate on the morphology of aluminium coatings deposited by thermal spraying

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Abstract

Thermal spraying is a technique to deposit on previously treated surfaces metallic or non-metallic materials whose main adhesion mechanisms are mechanical and chemical–metallurgical anchorage. The preparation of the substrate comprises cleaning, development of a rough surface and sometimes preheating to guarantee mechanical anchorage at microwelding sites. To evaluate splat morphologies, test samples with aluminium coatings deposited by different thermal spray processes, namely, flame spraying, high-velocity oxy-fuel and electric arc spraying, were carried out on substrates with different roughness, with and without preheating. Coating adhesion to the substrate was also evaluated. Different splat morphologies were obtained; the results indicated that coatings on preheated substrates may have lower roughness than that recommended in the literature. Besides, although preheating was essential for the flame spray process, it may be eliminated for the electric arc and high-velocity spraying processes.

Keywords: Thermal spraying; Aluminium; Roughness; Adhesion; Substrate preparation; Pre-heating

1. Introduction

Thermal spraying consists of a group of processes used to deposit, on a previously prepared surface, layers of metallic or non-metallic materials. Fig. 1 shows the schematics of the coating layer formation following deposition by a thermal spray process [1,2].

In thermal spray processes, the deposited material is melted or heated by the combustion of gases, an electric arc or a plasma. The impact against the substrate surface flattens the particles and produces adhesion to the substrate in a direction parallel to it by interlocking of the molten or semi-molten particles with asperities of the roughened surface. Further deposition occurs onto already deposited particles, generating a layer with particular characteristics, different from any other metallurgical form [3].

While for adequate aluminium coating deposition, known technical requirements are to be met regarding substrate roughness, the effect of surface preheating is usually not considered. Thus, the aim of this work is to address the influence of surface preparation, especially the effect of roughness and pre-heating of the substrate, on the quality of aluminium coatings deposited by different thermal spray processes.

2. Theoretical background

Whenever aluminium is to be deposited as a coating, the major properties to consider are adhesion, surface preparation, porosity and oxide content, which are being discussed in details below.

The mechanical performance of a thermal spray coating depends mainly on its adhesion to the substrate and on the cohesion between the deposited particles. Analysis of coating adhesion usually considers it to be the result of a combination of three fundamental mechanisms, related to

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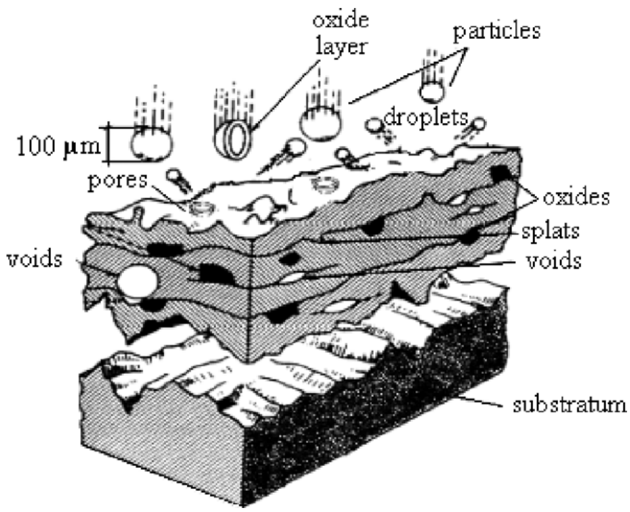

Fig. 1. Schematics of the coating layer formation following deposition by a thermal spray process (modified after Krepski [2] by Paredes [1]).

the nature of the acting forces, namely mechanical, chemical–metallurgical, and physical forces.

When the heated particles are accelerated towards the substrate, they flatten upon impact in a lenticular shape, followed by rapid cooling and mechanical anchorage to the irregularities of the surface [4]. Due to the different nature of the materials, either metallic or ceramic, the particle velocity and the heat transferred from the particle to the substrate, a different degree of micro-welding is achieved. Besides, there may occur localized melting, atomic diffusion with the formation of solid solutions or even intermetallic compounds, characterizing the mechanism of chemical–metallurgical adhesion.

The third mechanism, considered of secondary importance, is the physical adhesion, i.e., weak chemical bonding by Van-der-Waals forces, which contributes to the inter-atomic attraction within the material.

Thus, for maximum adhesion of aluminium coatings the quality of the surface to be coated is of critical importance. In particular, the activation of the substrate must guarantee good mechanical anchorage.

Surface preparation is necessary to ensure adequate adhesion to the substrate and to allow the projected particles to be free of residual impurities. This is achieved via surface activation that comprises three stages: (a) degree of cleanliness Sa2, Sa2.5, Sa3 [5]; (b) surface roughness (obtained by abrasive or mechanical blasting); (c) substrate pre-heating.

The technology of thermal spray and, in general terms, any adhesion mechanism requires a clean substrate, free of rust, iron oxide crusts, grease, oil or moisture. The standards for cleanliness in thermal spray are met via abrasive blasting processes, where many abrasives may be used, such as steel or iron powder and aluminium oxide, although adhesion efficiency varies with the material [6,7]. Higher adhesion is achieved with abrasives, such as aluminium oxide, that promote adequate roughness with low level of anchorage on the surface.

The American Navy standard [8] recommends a roughness range of 80–100 μm for flame sprayed (FS) aluminium coatings to ensure adhesion, which is considered adequate if its mean value is greater than 13.8 MPa and if all values are above 10.3 MPa. In Brazil, the measurement of roughness is usually carried out following the ABNT P-NB-13 standard [9]. For thermally sprayed surfaces, vertical, horizontal and proportional measurements are carried out using R_y and S_m values [6,10]. Fig. 2 shows the roughness parameters of interest for the measurements.

The pre-heating of the substrate must also be part of the process of surface preparation prior to thermal spraying, being responsible for the burning and volatilization of greases, oils and moisture retained at the metal surface. Pre-heating also favors residual thermal stress reduction, which in turn favors adhesion and cohesiveness of the layer. In fact, when the particles collide with the substrate, rapid cooling occurs, followed by contraction (shrinking) of the deposited material. On the other hand, the substrate heats up upon absorption of the kinetic energy of the impact, and also of the energy transferred by the flame or the plasma. Hence, preheating may reduce or distribute these opposite tensile stresses at the substrate–coating interface.

Although technical recommendations have proven that pre-heating is valuable to guarantee adequate adhesion, it is not yet in common practice as a surface preparation technique prior to thermal spraying.

Although most authors seem to agree on the benefits of pre-heating, the temperature range recommended is still under discussion. For instance, Lyman [11] recommends 260–370 °C for flame spraying of aluminium on steel, whereas more recent publications [1,12] for the same material suggests 120 °C to increase adhesion.

In the electric arc (ASP) thermal spraying process [13–15], the protection against corrosion, specially marine environments [16], benefits from the presence of oxides (2.5 to 3.0 mass%) within the deposited layer or on its

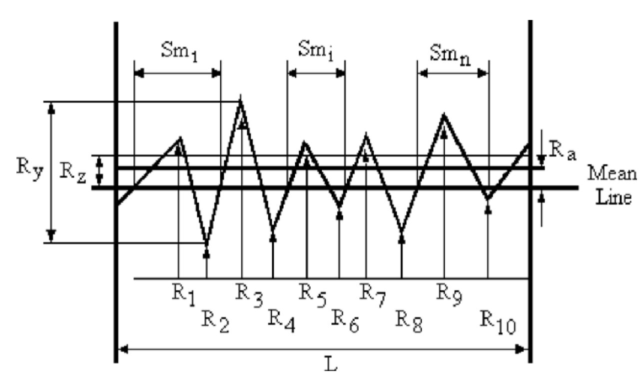

Fig. 2. Roughness parameters (as in Maranho [6]). R_a: arithmetic mean deviation; R_z: average peak-to-valley height of irregularities (10 points height); R_y: maximum peak-to-valley height of the irregularities; S_m: mean spacing of the irregularities.

surface. In addition, this level of oxide content is not detrimental to adhesion, even for a Sa2.5 surface [14,15].

The presence of isolated or interconnected pores is another important factor to be analyzed in coatings. Thermal spray processes are known to produce coatings with a variable range of porosities, which have a direct effect on some physical properties such as thermal and electrical conductivities as well as coating cohesion and adhesion. Furthermore, continuous and interconnected porosities are undesirable in coatings designed for protection against corrosion.

All these factors are of importance when adequate coatings are to be produced by thermal spray processes and therefore their comprehension is necessary to make thermal spraying an even more widely used coating process. Literature data addressing these factors are still scarce. For instance, the influence of roughness [11, 17] was studied for the ASP and FS during the 1970s and these data are still being used, although they may be responsible for the production of higher cost coatings, which take longer to be deposited.

3. Experimental procedure

Aluminium was deposited on test specimens of different roughness ranges, with and without pre-heating (at 120 °C), via different thermal spray processes, namely, flame (FS), high-velocity oxy-fuel (HVOF) and electric arc (ASP) spraying.

Mild steel (1020) was used as the substrate and aluminium wire of 3.2 mm in diameter (Metco MAL-12, 99% purity) and aluminium powder with a granulometry of −90+45 μm (Metco 54-NS, 99% purity) were used for the coatings. The thermal spray equipment were a 12E Metco gun, a 4RP Metco and a DJ 2004, for the FS, ASP and HVOF processes, respectively. A list of the thermal spray process parameters used is shown in Table 1.

The roughness of the substrate was achieved by a 90° angle abrasive blasting with a 38A Alundun white aluminium oxide at 100, 140 or 180 mm of distance and at 100 psi of pressure for a period of 60–80 s. A Sa3 cleanliness level was reached after blasting. Five roughness measurements for each experimental condition were made by a portable rugosimeter with mechanical contact (Mitutoyo, Model Suftest 211).

The Sa3 degree of cleanliness was determined by comparison with surface quality standards published by the NACE Standard [18].

Adhesion was evaluated by tensile testing on five cylindrical specimens for each experimental condition, according to ASTM C633/79, of 360–380 μm thick coatings [19].

Evaluation of the results was carried out by an ANOVA covariance analysis following a simplified Taguchi Method [1,20].

Table 1
Control parameters of the different aluminium thermal spray processes

Sample	Control parameters of the flame spray deposition process (FS)							
	Pre-heating temperature (°C)	Distance to substrate (mm)	Material	Oxygen flow (SLPM)[a]	Acetylene flow (SLPM)[a]	Compressed air pressure		Compressed air flow (SLPM)[a]
						(psi)	(kPa)	
F1	120	300	Wire	50	45	100	689.5	55
F2	120	300	Wire	40	35	100	689.5	55
F3	120	300	Wire	50	45	80	551.6	45

Sample	Control parameters of the electric arc deposition process (ASP)							
	Pre-heating (°C)	Distance to substrate (mm)	Primary air pressure		Secondary air pressure		Voltage (V)	Current (A)	Degree of cleanliness
			(psi)	(kPa)	(psi)	(kPa)			
A1	120	300	70	482.6	50	344.7	32	160	Sa3
A2	120	300	70	482.6	60	413.7	26	160	Sa2.5
A3	120	300	80	551.6	50	344.7	32	120	Sa2.5

Sample	Control parameters of the high-velocity oxy-fuel deposition process (HVOF)								
	Pre-heating (°C)	Distance to substrate (mm)	Nitrogen flow[b]	Oxygen pressure		Oxygen flow (SLPM)[a]	Propane pressure		Propane flow (SLPM)[a]
				(psi)	(kPa)		(psi)	(kPa)	
H1	120	150	70	150	1034.2	264.6	100	689.5	68
H2	120	300	80	180	1241.1	315	100	689.5	68
H3	120	150	80	150	1034.2	264.6	100	689.5	81.6

[a] SLPM: standard liters per minute.
[b] Nitrogen flow unit as specified on the instructions manual for the DJ 2004 gun at 125 psi.

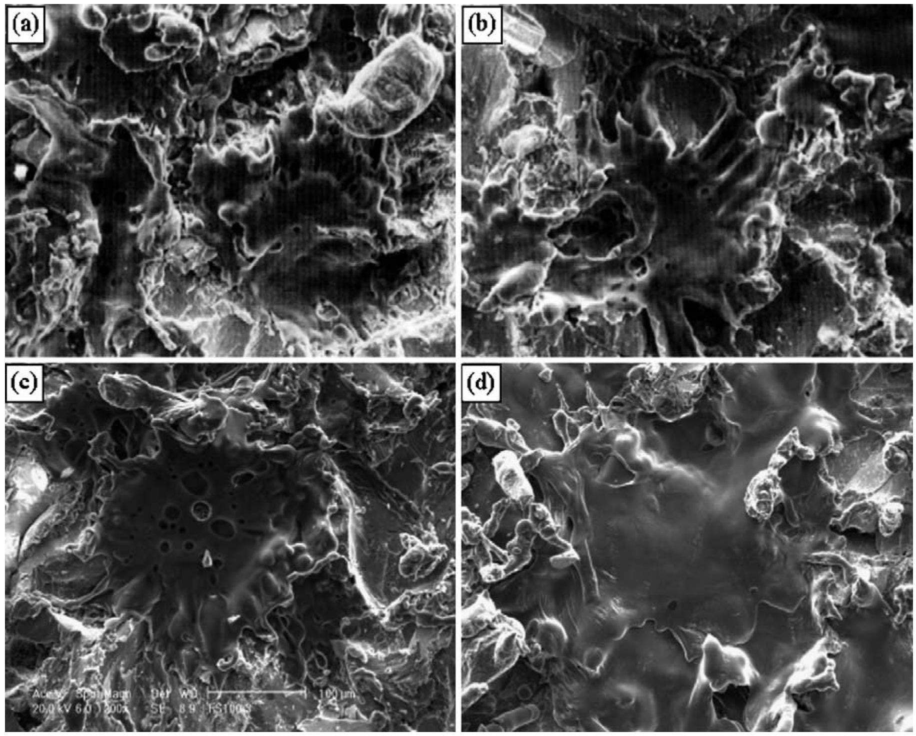

Fig. 3. Splat morphologies obtained for flame sprayed deposited coatings: (a) aluminium deposited on a R_y 70/80 μm rough substrate without pre-heating and (b) with pre-heating; (c) aluminium deposited on a R_y 50/60 μm rough substrate without pre-heating and (d) with pre-heating.

The splat formation and the morphology of the layers of aluminium coating deposited during a single and quick run of the gun over the specimen at a distance of 300 mm ("wipe test") were studied by scanning electron microscopy (SEM).

4. Results and discussion

Figs. 3–5 show splat morphologies of coatings deposited by the FS, ASP and HVOF processes, respectively. Fig. 3a, for a micrograph of a FS deposit on a substrate which had not been pre-heated, shows deposits that do not follow a typical splat morphology, and many droplets. Fig. 3b, on the other hand, shows more homogeneous splats, with pores and less droplets, being more accommodated to the substrate texture. These may indicate that for a rougher substrate without pre-heating, the particle disintegrates upon impact as the droplets are an indication of weak adhesion. Pre-heating, on the other hand, appears to favor the wettability of the substrate by the particle upon impact, enabling superior adhesion even though droplets may still occur.

In the case of splats deposited on a smoother surface, it can be seen that there are small pores in the center of the splats and droplets around them (Fig. 3c). When this surface is pre-heated prior to deposition, a much more homogeneous splat is achieved, without pores or droplets. Therefore it is clearly shown that lower roughness and substrate pre-

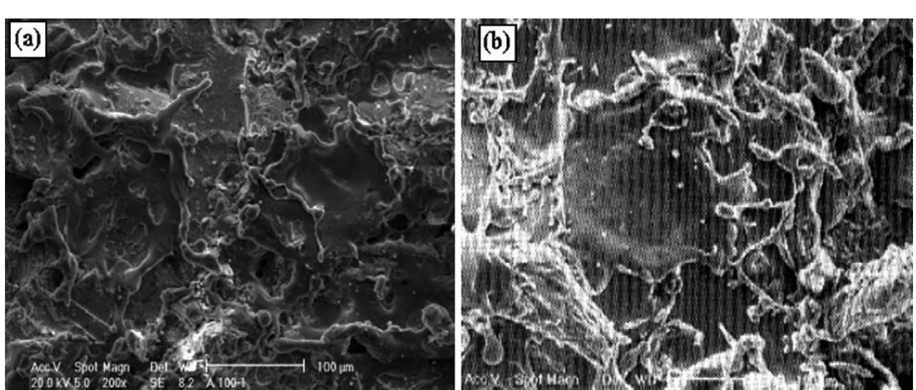

Fig. 4. Splat morphologies obtained for electric arc thermal sprayed deposited coatings: (a) Al deposited on a R_y 50/60 μm rough substrate without pre-heating and (b) with pre-heating.

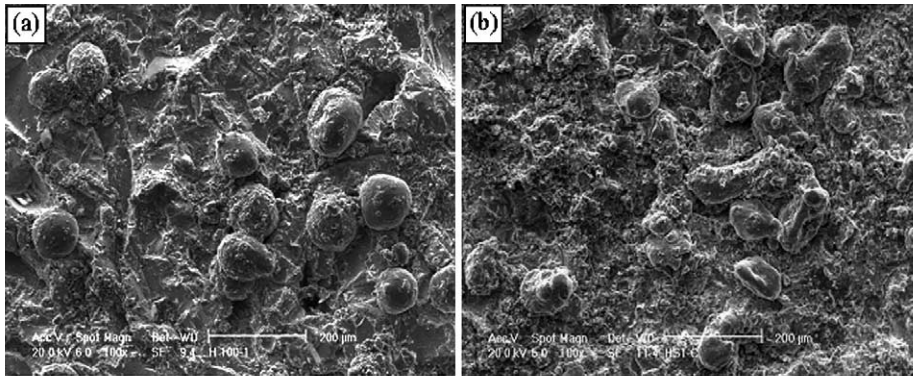

Fig. 5. Splat morphologies obtained for high-velocity oxy-fuel thermal sprayed deposited coatings: (a) Al deposited on a R_y 50/60 μm rough substrate without pre-heating and (b) with pre-heating.

heating increases wettability, allowing better mechanical anchorage of the particle to the substrate.

Fig. 4a shows particles deposited by ASP which do not have the splat appearance, with small aluminium-covered areas with many droplets. Fig. 4b shows more homogeneous splats which follow substrate texture, without pores and with less droplets. Thus it may be concluded that without substrate pre-heating, the particle arriving at high temperature does not produce a homogeneous wetting effect upon impact, since heat is quickly absorbed by the cold substrate,

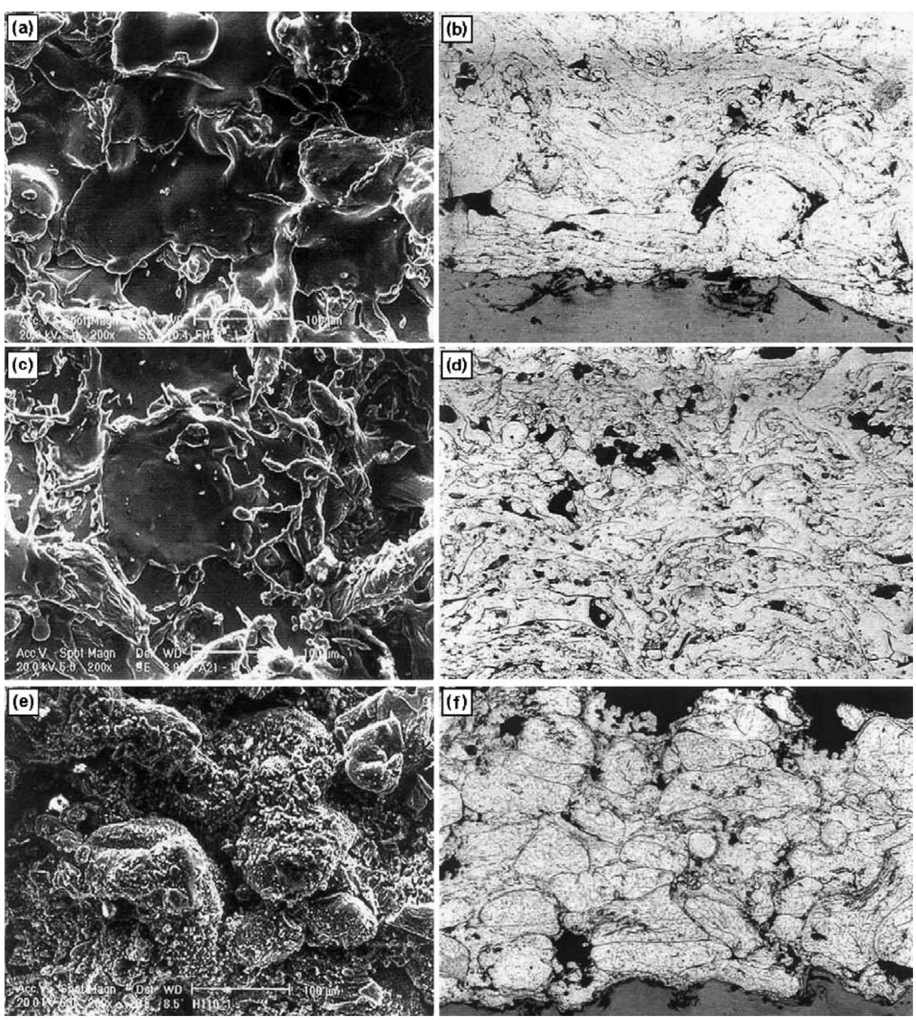

Fig. 6. Micrographs (200×) of typical aluminium coatings deposited on pre-heated substrates by: (a) FS in top view and (b) in cross-sectional view (average thickness of 386 μm); (c) ASP in top view and (d) in cross-sectional view (average thickness of 390 μm); and (e) HVOF in top view and (f) in cross-sectional view (average thickness of 396 μm).

favoring particle disintegration and the formation of droplets, and consequently a lower adhesion. Preheating, on the other hand, reduces thermal gradient favoring wetting upon impact due to more homogeneous splat formation and droplets that do not cause significant harm to adhesion.

Fig. 5a shows particles deposited via the HVOF process on a substrate without pre-heating and with less roughness. In their path from the powder deposit towards the gun, the flame and the substrate, the particles did not melt (no splat formation). This happens because the powder is fed to this gun by the nitrogen gas, and the particles achieve even higher velocities when traveling within the venturi inside the gun. When the particles pass through the flame (oxygen and propane), the heat supplied is not enough to provoke melting of the aluminium oxide film (melting point of 2200 °C). In fact, the particles are kept at the substrate surface only due to their deformation upon impact, with a typical shape distinct from that of a splat, and some particles disintegrate upon impact on the substrate or other particles already anchored. Since the particles do not melt, roughness is mainly dependent on the granulometry of the original powder and on the morphology of the substrate surface, and thus the lower the roughness of the substrate, the lower the roughness of the coating surface. Thus, the roughness of the surface reproduces the profile of the powder and of the fragments, as it will be seen in Fig. 6e.

In Fig. 5b, which also does not show the presence of splats, there is evidence that the larger particles keep their texture whilst the smaller ones fragment upon impact into micro-particles. Hence, it may be concluded that the HVOF process does not produce wettability, only mechanical anchorage, which is favored by lower porosity situations, and that pre-heating only causes less fragmentation of particles and higher adhesion to substrate.

Fig. 6 compares micrographs of typical aluminium coatings deposited on pre-heated substrates by the various processes studied. In Fig. 6a and b, for the FS process, the surface morphology shows homogeneous splats and few droplets, and the cross-section shows small-sized pores and a few coarser pores. Fig. 6c and d, for the ASP process, show very low porosity, many droplets and nests of pores in the cross-section. Besides, more refined splats are obtained, compared to the FS process. Fig. 6e and f, for the HVOF process, display voids and larger and fragmented particles that fulfill the gaps between deformed particles. The cross-section shows aluminium grains that have not melted and the chemical attack reveals the internal substructure of the grains, with more attacked grain boundaries, related to their thin oxide layer. It has also been noticed that this layer is discontinuous and that the high adhesion of the layer may be a consequence of the melting of the smaller particles between larger particles, acting as micro-welding sites and helping the anchorage of the larger particles. Furthermore, the porosity is finely and homogeneously divided, although large voids between large, not-melted aluminium particles may be seen.

Table 3
Roughness values for the substrate and the aluminium surface layer

Roughness—R_y (μm)	Roughness—R_y at the aluminium surface layer (μm)		
	FS	ASP	HVOF
70/80	105.4	120.8	187.6
50/60	82.2	92.2	120.2

Table 2 shows substrate roughness and deposited layer adhesion values along with the statistical analysis carried out, where the F parameter is an indication of the relative importance of the various experimental conditions (different processes, with or without substrate pre-heating). From these results, it can be inferred for non-pre-heated samples that as substrate roughness decreases, adhesion increases slightly in the layers deposited by FS and ASP (high F values) but remains constant for HVOF deposited layers (low F values). For pre-heated substrates, the results show significant adhesion increase for all processes. Furthermore, only the ASP and HVOF processes achieve higher values than those required by the standard.

Adhesion decreases when substrate roughness increases for the FS and ASP processes mainly due to two factors: (i) the high velocity of the particles, which allows an increase in the anchorage of the particles on the substrate, and (ii) the lower effective area of particle transference (smaller attack radius), which significantly decreases the droplets respon-

Table 2
Roughness and adhesion values

Process and sample identification		Layer thickness (μm)	Roughness R_y (μm)	Adhesion (MPa)			
				Without pre-heating	Statistical analysis[a]	With pre-heating	Statistical analysis[a]
FS	F1	382	70–80	9.2±1.2	(F=10–27%)	17.2±0.8	(F=7–37%)
	F2	386	60–70	10.2±1.7		18.5±0.7	
	F3	386	50–60	11.7±1.3		23.6±0.5	
ASP	A1	397	70–80	13.7±1.1	(F=15–30%)	18.5±1.5	(F=10–38%)
	A2	396	60–70	15.8±0.8		20.4±2.0	
	A3	390	50–60	17.9±2.0		25.7±1.8	
HVOF	H1	386	70–80	27.7±0.5	(F=1–2%)	38.3±0.7	(F=4–18%)
	H2	390	60–70	28.3±1.4		39.8±2.4	
	H3	396	50–60	28.1±0.8		45.2±2.2	

[a] Parameter F of the statistical analysis [20].

sible for the porosity and, therefore, contributes to increase adhesion.

Regarding the roughness of the aluminium coatings, Table 3 shows that with reduction of substrate roughness, the roughness of the deposited layer is reduced by 22%, 24% and 36% for the FS, ASP and HVOF processes, respectively.

5. Conclusions

Only coatings deposited by ASP and HVOF on surfaces of higher roughness and without pre-heating comply with the adhesion requirements set by the standard. For the aluminium coating deposited by FS, standard values can be met only if the substrate is pre-heated.

Reduction of roughness from 70/80 to 50/60 μm does not yield significant adhesion loss in any of the studied thermal spray processes.

For the ASP and HVOF processes, the reduction of roughness causes an increase of adhesion, even for non-pre-heated substrates.

In all, reduction of substrate roughness reduces coating roughness, which is an important benefit when machining is going to be carried out later on this layer, for instance, in the assembling of components with strict size requirements.

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