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EFFECT OF TEMPERATURE AND SUBSTRATE ROUGHNESS ON THE ADHESION OF COATINGS OF Ni ALLOY DEPOSITED BY FLAME SPRAY

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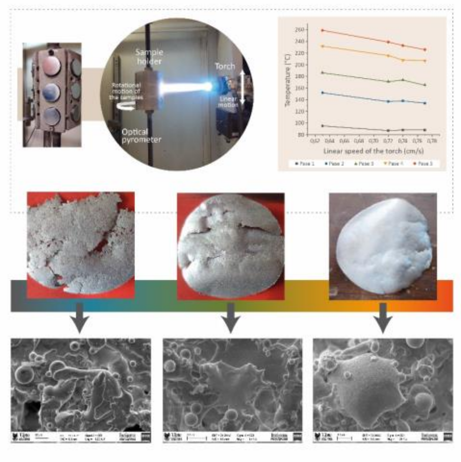

ABSTRACT

The temperature and roughness at which the adhesion occurs among an oxy-fuel thermally sprayed coating from a nickel-alloy powder and AISI 1020 carbon steel substrate is reported. The coatings were obtained using a modified Castolin Eutectic-Terodyn 2000 torch coupled to an electromechanical chamber, which allows to control the parameters of process. In order to establish the effect of the substrate roughness on the adhesion of coatings, these surfaces are prepared with SiC abrasive paper and abrasive blasting of corundum particles. The average roughness (Ra) obtained varies between 0,027±0,012 and 4,9±0,77 µm. The effect of substrate preheating temperature was evaluated for each test varying the speed of the torch and the preheating number passes, while the spraying distance and the oxidizing flame were kept constant. The spraying distance was 150 mm, while the flame used was obtained by mixing 21.8 L/min of acetylene and 59.5 L/min of oxygen. The torch speed was increased from 0.63 to 0.77 cm/s. The results indicate that the adhesion begins from an Ra value of 0,079±0,077 µm and a substrate preheating temperature of 180°C. From these values, as the roughness and temperature of the substrate increases, adhesion improves. Such studies contribute to the understanding of the mechanisms of adhesion between the coating and the substrate when flame spray is used.

Keywords: Flame spray, preheating temperature, substrate roughness, nickel alloy.

EFECTO DE LA TEMPERATURA Y RUGOSIDAD DEL SUSTRATO SOBRE LA ADHERENCIA DE RECUBRIMIENTOS DE UNA ALEACIÓN DE Ni DEPOSITADOS MEDIANTE PROYECCIÓN TÉRMICA POR LLAMA

RESUMEN

Se reporta la influencia de la temperatura de precalentamiento y rugosidad superficial del sustrato a partir de las cuales se produce la adhesión de recubrimientos de una aleación de Ni, depositados sobre acero al carbono AISI 1020 mediante proyección térmica por llama oxiacetilénica. Los recubrimientos fueron obtenidos usando una antorcha Eutectic Castolin-Terodyn 2000 modificada, acoplada a una cámara que dispone de sistemas electromecánicos a través de los cuales se controlan los parámetros del proceso. Con el fin de establecer el efecto del acabado superficial del sustrato sobre la adhesión de los recubrimientos, estos se prepararon superficialmente con papel y chorro abrasivo, el primero de SiC y el segundo usando partículas de corindón, las rugosidades medias (Ra) obtenidas variaron entre 0,027±0,012 y 4,9±0,77 µm. El efecto de la temperatura de precalentamiento del sustrato fue evaluado cambiando para cada ensayo la velocidad de la antorcha y el número de pases de precalentamiento, manteniendo constate la llama oxiacetilénica y la distancia de proyección de 150 mm. La llama utilizada se obtuvo mediante la combustión de una mezcla de 21,8 l/min de acetileno con 59,5 l/min de oxígeno, la velocidad de la antorcha fue aumentada desde 0,63 hasta 0,77 cm/s. Los resultados indican que la adhesión se empieza a dar a partir de una rugosidad media (Ra) de 0,079±0,077 µm y con una temperatura de precalentamiento del sustrato de 180°C, a partir de allí a medida que aumenta la rugosidad y la temperatura del sustrato, la adhesión es cada vez mejor. Este tipo de estudios contribuyen a la comprensión de los mecanismos de adhesión entre el recubrimiento y el sustrato, cuando se usa proyección térmica por llama.

Palabras Claves: Proyección térmica por llama, temperatura de precalentamiento, rugosidad del sustrato, aleación base níquel.

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1. INTRODUCTION

The coatings of nickel and nickel alloys deposited by thermal spraying are widely used to protect metal substrates from oxidation at high temperature and abrasive wear in molds for glass manufacture, or as an interlayer or bond coat between the metal substrate and the ceramic coatings in thermal barrier for Gas-Turbine Engine Applications. Either of these cases requires that the deposited layer adhere properly to the metal substrate in order to fulfill the function for which it was developed [1-6].

Nickel-silicon alloys powders are referred as a self-fluxing materials. In thermal spraying processes from these materials, a small part of the substrate surface on which the particle is deposited reaches a molten state, or at least a diffusion of atoms between the particle and the substrate is produced. Consequently, a self-adhesive layer is obtained and its adhesion energy is greater than that reached from mechanical adhesion of coatings sprayed from other materials [3,7-9].

It is widely reported that the formation of a coating deposited by thermal spraying occurs mainly in two stages. In the first one, particles (which arrive in a liquid state and spherical shape as a product of the shear forces experienced during the passage through the gas streams in traveling from the torch to the substrate) impact with the substrate and are deformed producing a lateral flow, in ideal conditions, to give them the form of discs with a high packing factor, occurring simultaneously with their solidification. In the second stage, the layer formed by packaging of the discs has been solidified and cooled to room temperature [8-10].

During the first stage, each fully or partially melted particle experiences a primary stress or quenching stress upon impact with the substrate and solidification. The magnitude of this stress (σ_T) is proportional to the modulus of elasticity of the particle (E), its coefficient of thermal expansion (α), and the temperature gradient between the particle and the substrate (ΔT), as given by the following expression [11].

$$\sigma_T = E\alpha\Delta T \qquad (1)$$

In the second stage, a secondary or cooling stress is produced in the coating fabricate due to the difference in deformation ($\Delta\varepsilon$) occurring between the substrate and the layer, which depends on the gradient between the thermal expansion coefficients of the substrate (α_s) and coating (α_c). This is expressed by [11]:

$$\Delta\varepsilon = \int_{T_2}^{T_1} \left(\alpha_s(T) - \alpha_c(T)\right) dT \qquad (2).$$

If $\Delta\varepsilon$ is large, the stress (σ_C) produced will be sufficient to bend the coated substrate, with a radius of curvature (R), which is given by the modified Stoney equation [11,12].

$$\sigma_c = \frac{E d_s^3}{6(1-\vartheta) R d_c^2 \left(1+\frac{d_s}{d_c}\right)} \qquad (3)$$

Where, E is the Young's modulus of the substrate, d_s and d_c are the thicknesses of the substrate and coating, respectively, R is the radius of curvature of the substrate (R = 1/κ, being κ the curvature) and ϑ the Poisson's ratio of the substrate.

According to the above, the coating adhesion results from the interaction between the binding forces (by mechanical anchoring between the rough substrate surface and the deposited particles or by micro-alloys produced by the self-fluxing alloys) and thermal stresses produced during the formation of the coating, and it requires a suitable preparation of the surface as well as, the preheating of the substrate to prevent detachment of the deposited coating [13,11,14-16].

The surface substrate preparation is usually performed by abrasive blasting using particles of corundum and then when the coatings are fabricated onto thin substrates they may be deformed irreversibly. Additionally, when large pieces are covered, the amount of energy required to preheat the substrate may be excessive. Consequently, the possibility that the substrate has the roughness and the preheating temperature required to achieve good adhesion of the coating could be limited.

Therefore, the study of the preheating temperature and roughness of the substrate at which adhesion of a coating fabricated by thermal spraying is produced is of great importance because it would reduce the deterioration of coated parts and the consumption of energy used in their manufacture.

2. EXPERIMENTAL PROCEDURE

The coatings were deposited using the camera Areste, developed by the GIPIMME Group at the University of Antioquia (Colombia). This system consists of a modified Eutectic Castolin-Terodyn 2000 torch, an RAYTEK infrared pyrometer, which measures the temperature of the substrate before spraying of particles, the electromechanical systems through which the linear velocity (V) of the torch and the specimen rotation is controlled, an electromagnet system to control the flow of feedstock powder, as well as the valves to control the gas flow (see Figure 1).

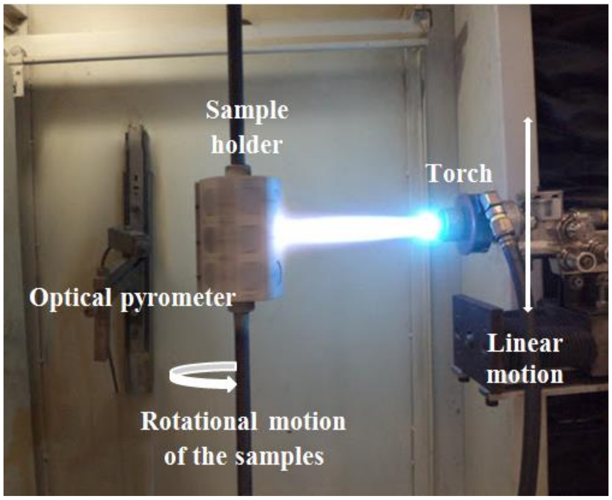

Figure 1. Experimental setup used for the spraying of the particles.

In order to spray the coatings, Eutectic-Castolin CPM 1205^{TM} powder was used; its chemical composition was analyzed by X-ray fluorescence (XRF) using a Thermo Scientific ARL OPTIM'X XRF spectrometer. Meanwhile, morphology and particle size were determined by image analysis using the Image J software, and the pictures taken by scanning electron microscopy (SEM), for which the JEOL JSM-6490LV microscope was used.

As substrates, AISI 1020 steel discs 25 mm in diameter and 6 mm thick were used. The surface was prepared with SiC abrasive paper and an abrasive blast with corundum particles, obtaining different average surface roughness. The average roughness (Ra) of the surface substrates was measured using a Mitutoyo SJ 201 profilometer. Before the spraying of coatings, the substrates were cleaned ultrasonically in an alcohol bath.

In order to determine the effect of the substrate preheating temperature on the adhesion of coatings the number of passes, and the linear velocity (V) of the torch were varied for each test, while the oxidizing flame and the spraying distance were kept constant. The flame used for this purpose was obtained from a mixture of 21.8 L/mi of acetylene and 59.5 L/min of oxygen, the spraying distance was 150 mm, while the linear speed of the torch was increased for the different tests from 0.63 to 0.74 cm/s. The rotation speed was 116 rpm for the sample holder. The powder flow rate remained constant at 48 g/min and was fed using a nitrogen flow of 17 L/min.

In Table 1, the parameters used to evaluate the influence of the roughness and the preheating temperature of the substrate, as well as the adhesion of the coatings of the nickel-base alloy are reported.

Table 1. Parameters used in the experimental stage.

Test	*Average roughness Ra[µm]*	*Speed of the torch [cm/s]*	*Preheating passes*
1	0.027±0.012	0	0
2	0.079±0.077	0	0
3		0.72	1
4		0.72	2
5		0.72	3
6		0.72	4
7		0.67	3
8		0.63	3
9	4.9±0.77	0.74	5

3. RESULTS AND DISCUSSION

The results of chemical analysis carried out to feed stock powders indicates that this material consists mainly of a nickel alloyed with silicon and iron with some impurities from aluminum and copper, as shown in Table 2.

Nickel alloys are widely used as feedstock powder to fabricate coatings and bond coats by thermal spraying process, since they protects effectively metallic substrates exposed to corrosive and high temperature environments and additionally decreases the effect of mismatch in thermal expansion coefficient between the substrate and a top layer (usually ceramic) on the adhesion. The self-fluxing effect allows the metallurgical bonding of the coating to the substrate, improving adhesion [3]. Moreover, silicon in amounts exceeding 1.5 wt %, as well as small concentrations of iron, react with oxygen to form oxides. This reduces the amount of oxygen available to react with the nickel and thus avoids oxidation and retains its ability to wet the substrate surface [3].

Table 2. Chemical composition of the starting powder.

Chemical composition (wt %)				
Ni	*Si*	*Fe*	*Al*	*Cu*
97.62 ± 0.08	1.79 ± 0.07	0.34 ± 0.017	0.18 ± 0.023	0.07 ± 0.013

The particle size analysis carried out from images of powder particles by SEM (see Figure 2) indicates that the size distribution is d_{10}=20 mμ and d_{90}=70 μm, d_{50}=31μm. Additionally, the particles show a rounded morphology, which facilitates its flowability during the thermal spray process, allowing coatings with a homogeneous structure and low porosity, reducing the accumulation of stresses during the cooling of the coating [3].

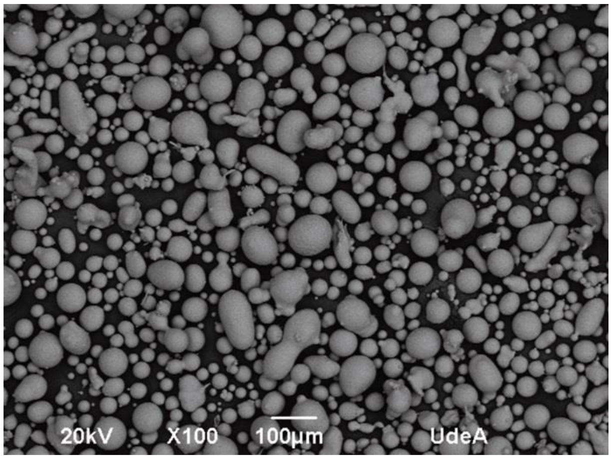

Figure 2. Micrography of the powder CPM 1205TM.

In Figure 3, the preheating temperature of the substrate is presented in terms of the linear velocity of the torch (*V*) and number of passes. This illustrates that the preheating temperature increases significantly with the number of passes and decreases slightly with increasing linear velocity of the torch. An analysis of the results obtained in the various tests described in Table 1 is presented below.

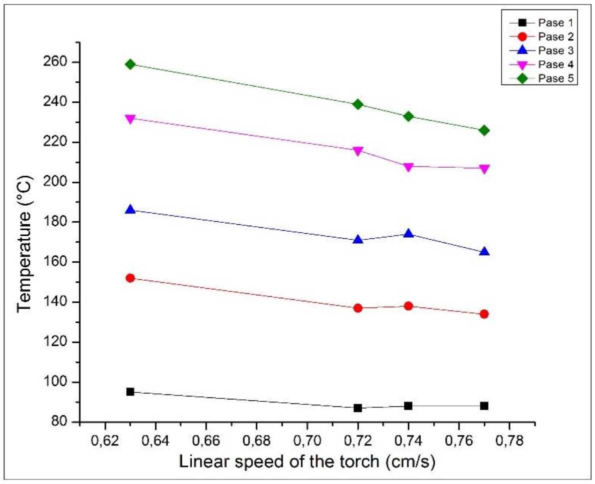

Figure 3. Preheating temperature of substrate depending on the linear velocity of the torch and the number of passes.

In test No. 1, conducted on a substrate having an average roughness (Ra) of 0.027 ± 0.012 μμ and without preheating (at room temperature), the particles of the nickel alloy deposited formed a discontinuous layer with multiple cracks and craters which did not adhere to the substrate surface, as shown in Figure 4 (a) and (b). Analysis at high magnification of the surface of the layer showed that the particles are flower-shaped, as illustrated in Figure 4 (c) and (d). This indicates that during the impact with the surface substrate the particles were in a liquid state.

It is important to clarify that the splashes shown in Figure 4 (c) correspond to the face of the layer that was in direct contact with the substrate, while those shown in Figure 4 (d) belong to those particles deposited on the opposite face of the coating. The splashes obtained from particles sprayed directly onto the substrate without preheating were most irregular than those sprayed onto layers previously deposited, which had a cumulative heat due to the thermal spraying process.

In thermal spray processes, splats with a flower shape (which is undesirable due to the difficulty of achieving a a good packaging between them and therefore it is difficult to achieve coatings with compact structure) has two possible causes: i) the impact of particles on the substrate at very high speed and ii) the impact with a substrate at a temperature below the critical temperature, Tc, or the transition temperature, Tt [4,17,18]. Because the process used to deposit oxyacetylene flame particles is low speed (<40 m/s) [9,19], in this case, the cause of the cracking and craters is not the high kinetic energy of particles upon the impact with the substrate, but rather the impact of the molten particles on the substrate (without preheating) at a temperature below Tc or Tt.

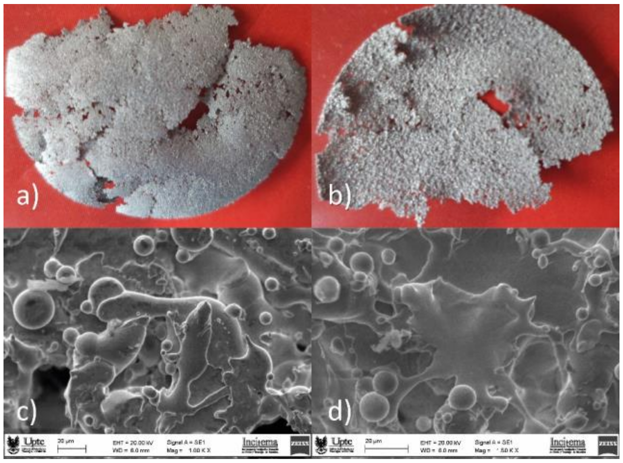

Figure 4. Images of the layer obtained with the parameters referred to as test No. 1. a) The surface was in direct contact with the substrate. b) The upper surface of the layer. c) A micrograph of the splash layer in direct contact with the substrate. d) A micrograph of the splash on the upper surface of the layer.

The influence of the preheating temperature of the substrate on the morphology of splash and the thermal stresses produced during spraying of this coatings has been widely studied [4,20,21]. It has been reported that when heating the carbon steel substrate at a temperature of 200°C, the particles of molybdenum (Mo) deposited change shape from flowers into discs [20,21]. However, it has not been possible to determine values of the transition temperature (Tt) for the materials used in this study; steel substrates on which nickel alloy had been deposited.

The primary stress or quenching stress (σ_T) experienced for each particle fused upon impact with the substrate was determined using expression (1), for which the values of E=210 GPa and α=13x10^{-6} K^{-1} reported in the literature were used [11], obtaining a thermal stress (σ_T) value of 3.9 GPa. Importantly, the temperature gradient experienced by the particle is determined from the melting temperature of the nickel (since, as shown in Figure 4 (c), the particles arrive as a liquid upon impact with the substrate) and the ambient temperature of the steel substrate. The thermal stress of 3.9 GPa produced by the quenching of each particle would be sufficient for shrinkage, making stacking difficult and consequently favoring the formation of cracks and craters because of separation between particles, as evidenced in Figure 4 (a) and (b).

Other researchers have reported similar values for the maximum thermal stress (σ_T= 3.6 GPa) in nickel alloy particles deposited on substrates without preheating and experimentally measured the total stress that causes this thermal shock in the coating, finding values of 55 MPa. The difference between the values of thermal stress calculated and experimentally measured mainly arises because the calculated value corresponds to the quenching stress produced in each particle, while the experimentally measured value was formed on the layer by the accumulation of particles. In the second case, cracks and craters between particles contribute to the release of much of the thermal energy to which each particle was subjected [22].

With regards to the effect of the substrate roughness on the adhesion of the sprayed coatings, some authors have agreed that the layer shrinks during cooling, producing a secondary cooling stress which is parallel to the substrate surface and can be minimized by roughness in the form of grooves [3,23].

According to the above results and the fact that the roughness of the substrate is very low (an Ra value of 0.027 ± 0.012 µm), the deposited layer did not adhere. Thermal stresses (σ_T) occurring during the process exceeded the anchoring between the particles and the substrate surface. The small difference in thermal expansion coefficients between the substrate (α_S=1.17x10^{-5} C^{-1}) and the coating (α_c=1.3x10^{-5} C^{-1}) [24-26] makes cooling stress (σ_c) small and therefore does not contribute significantly to the release of the deposited layer.

In test No. 2, as in the above, particles projected onto the substrate without preheating experienced an excessive primary or quenching stress, which produced flower-shaped splashes and strong cracking in the deposited layer, as shown in Figure 5 (a) and (b). However, although the splashes continue to exhibit a flower shape, as shown in Figure 5 (c) and (d), they are less irregular than evidenced in the test sample No. 1. Likewise, it is observed that the deposited layer fractured much less than the previous sample, which can be explained by the approximately three-fold increase in roughness, favoring the wettability of the deposited particles [17]. Thus, the splashes are less irregular than those formed in test No. 1 (see Figures 4 (c), 4 (d), 5 (c) and 5 (d)). Overall, it was established that, in this case, the primary stress or quenching stress produced in the particles remain primarily responsible for the failure of layer adhesion.

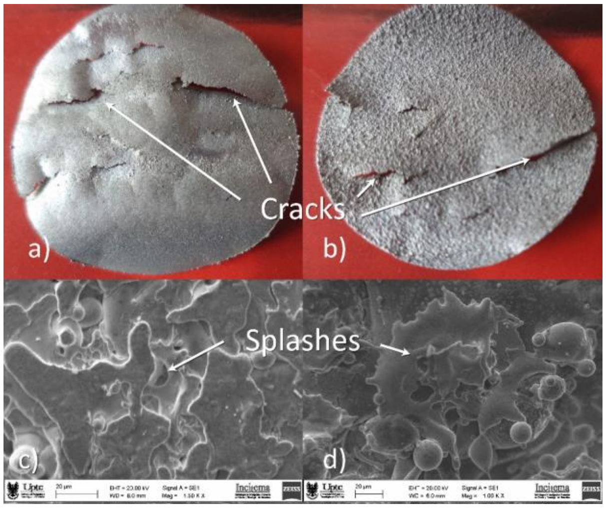

Figure 5. Images of the layer obtained with the parameters referred to as test No. 2. a) The surface was in direct contact with the substrate. b) The upper surface of the layer. c) A micrograph of the splash layer in direct contact with the substrate. (d) A micrograph of splash on the upper surface of the layer.

Compared to the two earlier tests, the application of a preheating pass for test No. 3 increases the substrate temperature to approximately 87 ºC, which reduces the quenching stress produced by thermal shock in each particle at the time of impact with the substrate to 3.7 GPa. This thermal shock reduction would be sufficient to inhibit cracking of the layer, which cannot completely separate the particles and takes the form of striations in the coating face that was in contact with the substrate. Additionally, the curvature of the deposited layer was evident in the peripheral view of the sample (see Figure 6).

The curvature of the layer shown in Figure 6 is caused by the difference in the expansion experienced by the layer and the substrate cooling. This indicates that as the cracking of the layer is reduced, cooling stress becomes important for the integrity and adhesion of the coating because the energy associated with such stresses is no longer easily dissipated through the cracks.

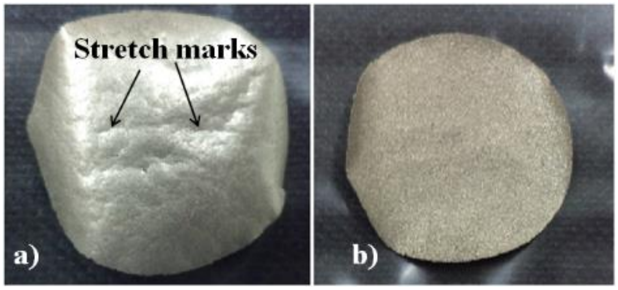

Figure 6. Images of the layer obtained with the parameters referred to as test No. 3. a) The layer was in direct contact with the substrate. b) The top surface layer.

In tests No. 4, 5 and 6, the number of passes for preheating the substrate was increased to 2, 3 and 4, thereby increasing its temperature to 137, 171 and 216 °C, respectively, while maintaining the same surface roughness (Ra = 0.079± 0.077 μm) used to deposit the layers in tests 2 and 3. The results obtained establish that the layers deposited on substrates with two or three passes of preheating continue without adhering, while the coating with four preheating passes adhered to the substrate, as shown in Figure 7.

As the temperature increases with each preheating pass, the primary quenching stresses experienced by each of the particles decreases to 3.6 to 3.5 and 3.4 GPa, respectively. With this effect, the striations evident in the surface layer that was in direct contact with the substrate are less severe, as illustrated by Figure 7 (a) and (b). This indicates that the splashes, which occur in substrates without preheating and give rise to cracks, become less severe with increasing substrate preheating temperature. With a substrate preheated to 216 ºC, the stress produced by such splashes does not exceed the anchoring forces of the coating to the substrate.

Additionally, it is evident from the layers deposited in tests No. 4 and 5, shown in Figure 7 (a) and (b) that curvature increases with the preheating temperature of the substrate. This occurs because when the striations produced by the quenching stress are less severe, the coating does not have large energy dissipation. Thus, the cooling stress that generates the differences in deformations undergone by the substrate and the layer becomes more critical to coating integrity.

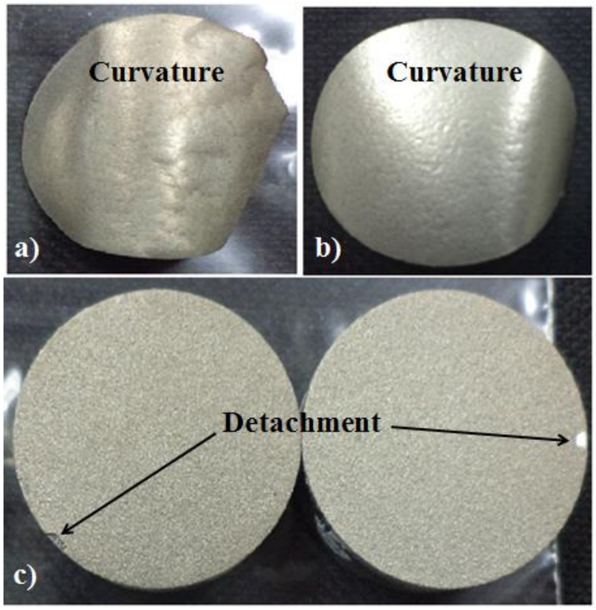

Figure 7. Images of the samples corresponding to tests No. 4, 5 and 6 (a) and (b) the surface layers which were prepared in tests No. 4 and 5, respectively, were in direct contact with the substrate. c) The coating deposited according to test No.6.

In the sample prepared in test No. 6, cooling stresses generated small detachments in the periphery of the coating, but not enough to generate curvature of the layer or total detachment from it, as shown in Figure 7 (c).

The above results indicate that the preheating of the carbon steel substrate at a temperature close to 216 ºC promotes the adhesion of the layer, even with low average roughness (Ra = 0.079± 0.077 μm).

To identify the preheating temperature of the substrate from which the thermal shock experienced by each of the sprayed particles is sufficiently reduced for the coating to adhere, two tests (No. 7 and 8) on substrates with the same roughness (Ra=0.079 ± 0.077 μm) were performed. This was carried out as in the previous experiments but with preheating temperatures between 171 ºC (test No. 5) and 216 ºC (test No. 6). In tests No.7 and No. 8, all three passes for preheating were remained, but the torch speeds slightly decreased (from 0.72 to 0.67 and 0.63 cm/s respectively), resulting in preheating temperatures of 180 and 186 ºC.

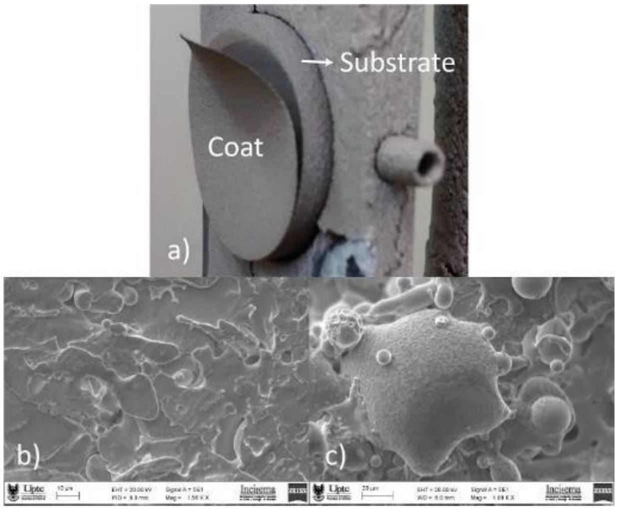

Figure 8. (a) Coating deposited according to the parameters of test No. 7. (b) The deposited splashes for test No. 8, which were in direct contact with the substrate (c) Splashes on the upper surface layer deposited according to the parameters for test No. 8.

The results indicate that the layer deposited onto the substrate preheated to 180 °C has already partially adhered to the substrate, as shown in Figure 8 (a). The splashes of the layer in direct contact with the substrate preheated to 186 ºC exhibited a less severe shape than those of the cooler substrates, as shown in Figure 8 (b). Even those splashes corresponding to the upper face of the coating are beginning to be disc-shaped, as reported elsewhere for particles deposited on substrates preheated to Tc or Tt [20,21].

According to the above results, it can be established that the Tt value for the nickel alloy particles deposited on a substrate of carbon steel is between 186 and 216 ºC.

Finally, the coating deposited on a substrate preheated at 233 ºC (slightly above the value established as the Tt) and with an average roughness of Ra of 4.9 ± 0.77 μm, according to the parameters for test No. 9, presented good adhesion to the substrate, as shown Figure 9 (a). The structure of particles that are in direct contact with the substrate surface exhibit a preferentially lamellar shape, illustrated in Figure 9 (b). Under these conditions, particles were introduced even in the more irregular valleys thanks to good wettability of the splashes that experienced a low degree of spatter [17].

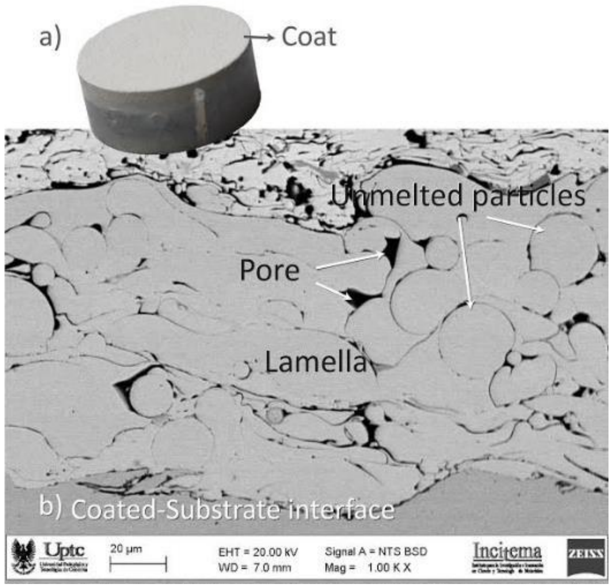

Figure 1. The coating obtained from the parameters corresponding to test No. 9. (a) The overview, and (b) cross-section of coating.

4. CONCLUSIONS

Particles of a nickel alloy Eutectic- Castolin CPM 1205TM were deposited by flame spray on AISI 1020 steel substrates varying the average surface roughness (Ra) from 0.027 ± 0.012μm to 4.9 ± 0.77μm and with preheating temperatures from 25 to 233 ºC in order to establish the conditions at which the particles begin to adhere to the substrate.

The results obtained establish that, in carbon steel substrates with roughness from 0.079 ± 0.077μm, Tc is in a range between 180 and 216 ºC. Under these conditions, the splashes left by the particles upon impact with the substrate are not as irregular as those obtained with lower roughness values and lower temperatures, favoring wettability of particles on the substrate and thus their adherence to it.

Thus, it can be concluded that the Tc for substrates of carbon steel with low surface roughness (Ra 0.079 ± 0.077μm) on which particles of nickel alloys are deposited is approximately 200 ºC and that as the preheating temperatures and roughness values of the substrate slightly increases, the coating adhesion will improve.

5. ACKNOWLEDGEMENT

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