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INFLUENCE OF DIFFERENT MICROSTRUCTURAL FEATURES ON WEAR AND CORROSION RESISTANCE OF 13CR STEEL ARC SPRAYED COATING

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ABSTRACT

Wire arc spraying process is a classical technique to deposit thick coatings using metallic wires as feedstock. The process is used to protect mechanical components against wear and corrosion. 13Cr steel coatings were produced using wire arc spray with different spray distance gun-substrate. Coatings microstructure was investigated using optical microscopy and scanning electronic microscopy (SEM). Chemical composition were determined using EDS analysis. The coatings exhibit a layered microstructure consisting of 13Cr steel lamella and FeO veins between them including cracks and pores. Porosity and lamella thickness were determined by image analysis. Mechanical properties were evaluated by microhardness Vickers. The wear test was conducted by mean of ball-on-disc apparatus according to ASTM G99 standard. Corrosion resistance was analysed with electrochemical polarization test in 3% NaCl solution. The results show that corrosion resistance is strongly influenced by porosity which accelerate corrosion leading to depletion of lamellae forming the coating. However, coating post-treated with an epoxy sealing present a good corrosion resistance.

KEY WORDS

- wire arc spray,
- lamella thickness,
- porosity,
- image analysis,
- corrosion resistance,
- wear mechanism.

1. INTRODUCTION

Thermal spraying processes are surface treatment techniques using powder, wires, rods as feedstock to be melted through heat source of electrical kind as plasma and WAS or a combustion of a fuel as HVOF, D-Gun, and carrying gas to impact the melted particles of feed-stock on the surface to be treated. Each particle spreads and solidifies upon impact on the substrate forming the splat. The stacking of the successive splats on the substrate surface or on the already formed splats forms a thick coating that is beneficial for wear protection and/or repairing damaged part. The advantages of thermal spray processes being able to treat small area as well as large one of industrial parts and component with different materials making their applications growing in particular in protection against wear and corrosion in aeronautical industry, dreading machines, paper industry and offshore structures [1,2].

WAS process is usually used in industry for its flexibility, high rate of deposit, and relative cheapness method compared to the other thermal spraying processes. In this process, the wires are consumable and constitute the electrodes, by applying electric current keeping a well small distance between the tips of wire in the gun, an electric arc forms between them, resulting in a melting energy sufficient to melt the tips continuously as the wires feeding is ensured in the system [1,3].

The air jet used in WAS process plays a role of atomizing by breaking up the melted droplets and propels the particles onto the treated substrate. The atomized particles travelling from the gun to the surface impact, flatten, and finally solidify. Coating is bonded into the substrate by a pure mechanical bonding due to the low temperature of the substrate and the roughen surface. However, the lamella (splats) connected to each other by mechanical and metallurgical bonding. The lamellar microstructure of the coating makes the mechanical and thermal properties behaviour anisotropic. The properties parallel to the interface are very different corresponding to those perpendiculars to the interface [1], [4].

Thus, arc sprayed coating commonly showed lamellar microstructure with the presence of pores, oxides, interlamellar cracks, unmelted particles. It was shown that the coating quality is highly influenced by the spray parameters such as atomizing air pressure, spray distance, feeding rate of wires, current intensity and voltage. These parameters summarises the impact of the carried particle on the substrate. The velocity and the viscosity of the liquid particles (physical state) play an important role to/how build up the coating [5].

The porosity is considered as a defect in the coating, it is due to droplets flattening problem, and this latter is due to a weak impact, producing thicker splats. The impact is generally governed by the velocity and viscosity of liquid particles at the impact. It was shown that the atomizing jet pressure, and spray distance in addition to current intensity are the main affecting parameters on the quality of the coating. The structural integrity of coatings depends highly on microstructural properties of the deposition whereas an optimizing study of spray parameters is necessary to enhance coating performance [6].

Therefore, the microstructural characteristics are more critical in thermal spray coating, and require an investigation of porosity, oxidation, micro-cracks, splat thickness as the most important micro structural characteristics features of the arc sprayed coatings [7], [8].

It has been shown that the porosity is the main microstructural property which affect strongly the splat cohesion leading to a very rapid degradation of materials in wear and corrosion environment [1], [9], [10].

Fe based alloys have been used as protecting coating against wear and corrosion. Chromium in steels can enhance the oxidation resistance which makes the chromium steels as protective coatings very reliable at high temperature application such as power generation industry. Moreover, the chromium steels can be good candidates to protect materials from corrosion and wear degradation [11].

In thermally sprayed chromium steel coatings, the chromium has beneficial influence in deceasing the friction coefficient when compared to ordinary steels coatings [12].

The splat constitutes an elementary component of the thermal sprayed coating, its thickness plays an important role to determine mechanical properties, and its morphology is mainly related to the particle in-flight history before impact. This makes the behaviour of the surface coating different when it is subjected to mechanical stresses. The splat can be fractured, plastically deformed, abraded or delaminated [13]. Thus, the complex microstructure of this kind of coating affects the mechanical properties and consequently the wear resistance.

It has been found that the interlamellar oxides, create weak links between splats, which make them the preferable zones at high stresses. Consequently, delaminating of the splats is the main wear mechanism in WAS coating[12]. Regular shaped splats are the easier ones to be pulled out [14]. The fracture of splats can take place at high stresses as wear mechanism, due the severe plastic deformation at splat tips where porosity or roughness makes this mechanism worse [13].

Porosity and microstructural features influence the corrosion resistance and the electrochemical behaviour. P.H. Suegama and al in their study on the corrosion of thermally sprayed stainless steel on mild steel, found that corrosion is strongly influenced by porosity and oxides [9].

The objective of this work is to study the influence of the spray distance as an important parameter of electric arc spray process on microstructural features such as porosity with image analysis, and their relationship with the mechanical and electrochemical behaviours of the of 13Cr steel coatings.

2. EXPERIMENTAL DETAILS

13Cr steel wires were used as the spraying feedstock. A METALLISATION 230 type arc spraying device was applied to form thick coatings on the surface of carbon steel C35 substrates of 25mm in diameter and 8mm in thickness. The spray parameters are presented in table 1, only the spray distance parameter is considered in this study.

Substrate discs were cleaned and grit- blasting with corundum grits prior spraying, degreased using ultrasounds in acetone, then immediately sprayed due to their rapid oxidation. To ensure a good adhesion coating, a thin inner bonding coating of nickel/Aluminium alloy was deposited on the substrates.

Table 1. *parameters of arc wire spray*

Sample	E1	E2	E3
Current intensity I (A)	100	100	100
Tension V (v)	35	35	35
Spray distance (mm)	**100**	**120**	**140**
pressure (Bar)	3,5	3,5	3,5

Samples were sectioned and mounted in resin epoxy, they were then ground using silicon carbide followed by polishing with a sub-micron alumina suspension. The Examination of coating microstructure was carried out using MO and SEM on cross-sectional samples.

Porosity and lamellae thickness were evaluated using image analysis technique (Imagej software), element analysis were examined using Jeol JSM 6360LV scanning electron microscope (SEM) coupled to an energy dispersion spectroscopy (EDS).

Corrosion resistance of coated samples of different spray distances were evaluated by the mean of potentiodynamic polarization test in 3% NaCl solution.

For the friction test, only the optimized coating was tested due to the high roughness and porosity. The friction tests were conducted with a ball-on-disk test machine in accordance with the recommended procedures ASTM G99 using a different loads ranging from 2 N to 12 N. The counter faces were 6-mm diameter ball made of 100Cr6 steel. Both coated disc and counter body were cleaned in acetone and dried in air prior the wear test. The relative humidity and temperature were held constant during the test (Hr=15–20% and 20 °C, respectively). The sliding distance was 1000 m for all the tested samples. A track diameter of d=16 mm and a sliding speed of v = 0.1 m.s-1 were used. Every worn scar was characterized by SEM observation. [15]

3. RESULTS AND DISCUSSI

3.1. Microstructure

In previous work [15], it was found that XRD pattern of the present phases formed in 13Cr sprayed coating, Fe and Cr phases are the main constituent of the coating. Due to the fact that the coating was deposited on plane carbon steel substrate in air, some oxides including FeO with low intensity were determined, which can be confirmed by EDS microanalysis of cross section samples (fig. 2): it can be pointed out that only few oxide phases are formed during the interacting process of air with melted particles. The presence of chromium enhances the resistance of steels at high temperatures against oxidation [9].

Arc spray process makes the particles to crystallize when they solidify from molten state. It was being found that the average temperature obtained by Spray Watch system is varying from 1000°C to 1600°C which confirms the melting state of particles in-flight. [15]

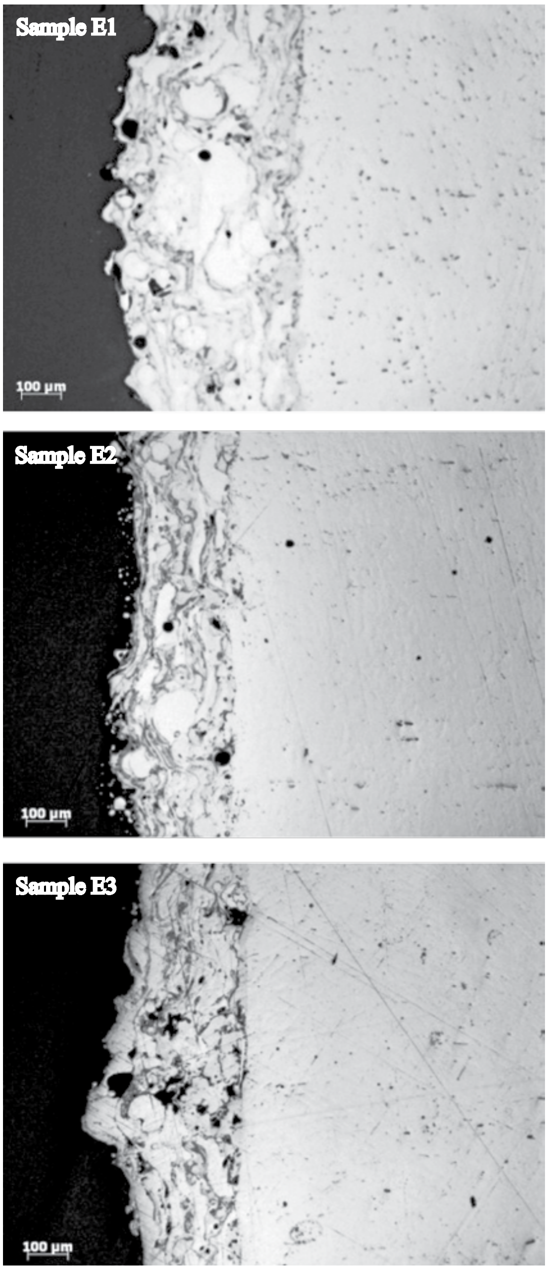

Fig. 1. optical micrographs of sprayed coatings in different spray distances

The figure 1 show the microstructure of coated samples E1, E2, and E3 corresponding to the spray distance 100 mm, 120mm, and 140 mm. All optical micrographs show lamellar microstructure with difference in thickness lamella and rate of porosity and oxides.

Porosity within a microstructure can be easily detected by image analysis due to the high degree of contrast between the dark pores (voids) and the more highly reflective coating material.

The calculation of lamellae thickness was based on the plot of the grey scale of the segment drawn on the micrograph of lamellar coating perpendicular to the interface. The difference between the grey scale of oxide and the inner of the splat gives the splat thickness which is the distance between the oxide veins inter-splat (figure 2).

The thickness was calculated using the following equation :

$$e = \frac{\left(\sum_{i=1}^{L} (V_{i+1} - V_i) \right)}{L} \quad (1)$$

The results were : porosity : 7.4 ± 1 %, 9.2 ± %, and 11% ; lamella thickness : 5 µm, 26,15 µm, and 46,66 µm, were found correlated with respectively the spray distances gun-substrate : 100 mm, 120 mm, and 140 mm. The porosity results from stacking up the particles to form coating. It can be related to the presence of non-melted particles, or entrapped gas. However, the main factor producing pores, is the manner of impact and how to solidify on the surface to treat which are related to the temperature and velocity of the in-flight particles. The increase of spray distance in arc spray process, means more dwell time in the jet, the velocity is controlled by the pressure of carrier gas and the size of particles (corresponding to its gravity, each particles has a determined velocity) [1], [5], [7]. As the air is only used to atomise the molten particles and carry them to adhere to the surface; the particles are subjected to more cooling up by the contact air- in flight particle, thus to oxidation leading to increase their viscosity. This latter alters drastically the behaviour of particles at the impact, leading to a lower spreading degree. As it is shown in the figures 3-a, and 3-b. The porosity is resulted from the manner of flattening deduced from the splat thickness, and affected by the spray distance as an important parameter in arc spray process.

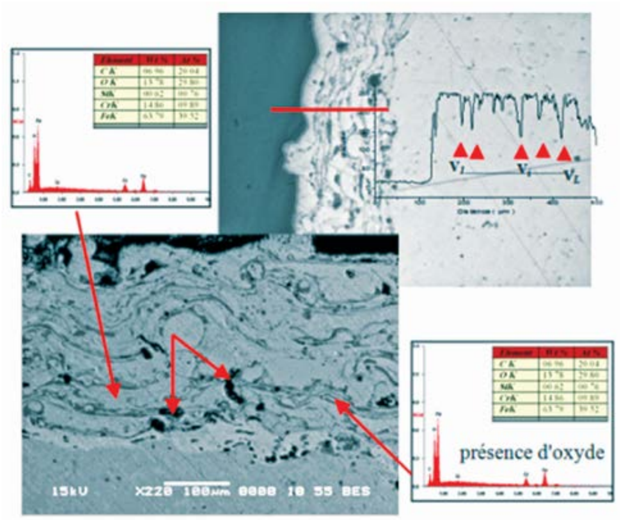

Fig. 2. back scattered SEM micrograph with microanalysis showing oxides veins b) optical micrograph used to calculate the thickness of splat with image analysis.

Coatings deposited by wire arc spray exhibit a complex microstructure, the splats are the main component, with a significant amount of pores, oxides, un-melted particles.

Figure 4-a shows fractured pulled coating and observed via SEM, most splats appear separated from their neighbours especially splats with smooth, evidence of a lack of metallurgical bonding between them. Thermal spray deposits microstructure is strongly depending on the size, chemistry, phase, and trajectory of sprayed particles in the spray jet, impacted particles could be either fully or partially molten, re-solidified, or un-melted. The coating layer contains micro-cracks (figure 4-b) witch can be attributed to thermal residual stresses. The nature of the residual stresses significantly influences various types of coating property, such as the bond strength, hardness and wear resistance.

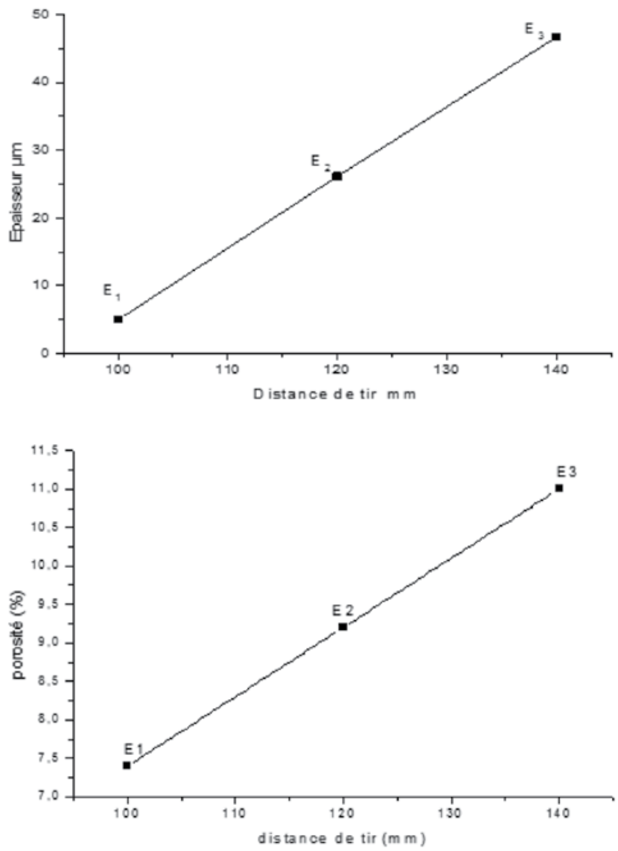

Fig. 3. influence of spray parameters on the microstructure of coating.

3.1 Microhardness

Vickers microhardness values of 13Cr coatings of different spray distance are shown in (figure 5). The values are the average of ten measurements taken along the polished cross section of the sample. It is well noted that the value was not uniform due to the inhomogeneity of the coating microstructure. The molten particles leave the gun with velocity of the order of 50 m.s^{-1} to cross the spray distance of 100 mm [15]. Very short dwell time of in-flight particles : solidification takes place since the particle have impacted on the surface of substrate or the flattened particles with rapid cooling leading to ultrafine grain size (about 1 µm of size) [1]. Inclusion of re-solidified particles in the coating are observed, they exhibit a high microhardness.

In the wire arc spraying process, the wires are melted into particles by an electric arc formed between them. There are no hot gas jets associated with wire arc spray. Unlike other thermal spray processes, droplet/particle temperatures begin to decrease immediately after the particles are formed or atomized in the arc zone, leaving the wire tips as they melt. The dwell time that produces increased heating in other processes only serves to cool particles in wire arc spray, because the atomizing jets are only used to accelerate the particles toward the surface to be coated. However, since the particles are still hot from the melting process, interactions between air and the hot particle surfaces do still occur. Thus, the hardness of the coating depends on the spray distance since the increasing of dwell time (spray distance), makes the droplets solidifying and increasing in their viscosity and thus, altering the flattening of particles during impact, producing thicker splats with more pores and less cohesion.

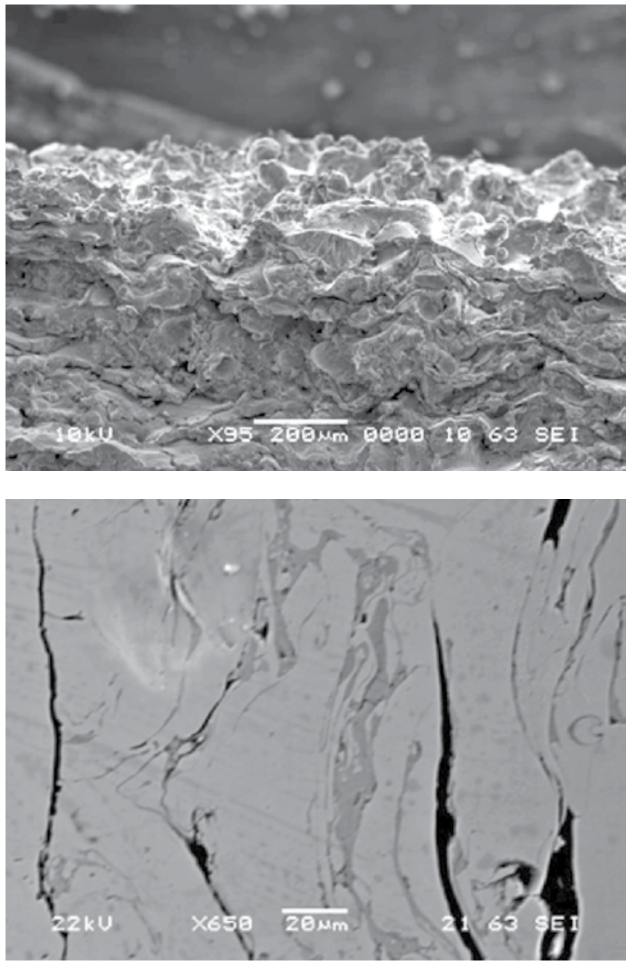

Fig. 4. a) SEM observation of a pulled coating fracture
b) SEM observation of microcrack existing in the interface. [15]

The oxides disperse upon impact and become included in the deposit, as the case in the other processes [1]. It is noted that the interlamellar areas exhibit microhardness relatively higher than the inner of splat.

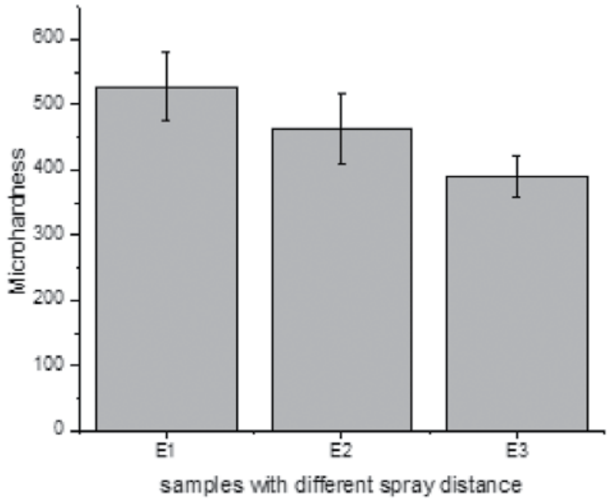

Fig. 5. microhardness of different coatings according to their spray distance.

Only the optimised coatings of spray distance E1 were considered in this part of study. As sprayed samples have rough surfaces as a characteristic property of wire arc spray coating even for the optimised one, as shown in fig. 6. The SEM observation of the samples under investigation shows a rough surface due to coating forming by stacking of flattened particles, showing a good melting state at the impact, with inclusion of unmelted particles, this latter can contribute to produce higher porosity and roughen surface, that's why samples must be polished to eliminate the effect of roughness on wear test; it was reduced from 20 µm to 5 µm.

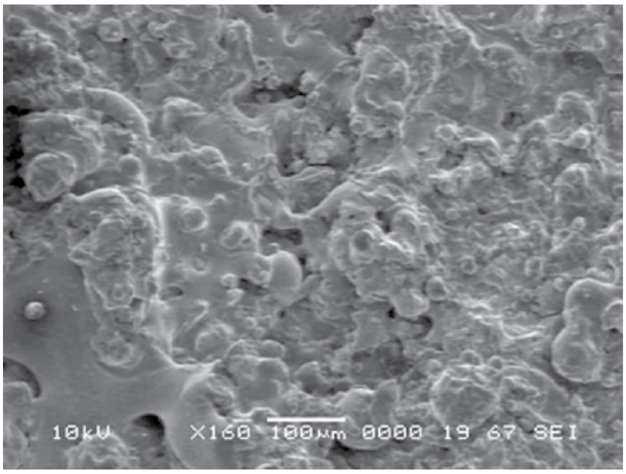

Fig. 6 top surface of 13Cr coating as-sprayed observed by SEM for 100 mm of spray distance (E1). [15]

The most important governing parameters for sliding wear are load and sliding velocity. The influence of load on coefficient of friction of 13Cr coating is presented in Fig 8.

It is observed that coefficient of friction increases by increasing applied load. According to Greenwood and Williamson [11], the real contact area is proportional to the normal load. Based on the classical theory of adhesion, the frictional force is defined as [16] :

$$F = \tau_a A_r \text{ and } \frac{F}{(L)} = \frac{\tau_a A_r}{(L)} = \mu_a \quad (2)$$

where τa is the average shear strength during sliding and (L) is the applied load. µa is adhesion induced friction coefficient.

For elastic contact of a spherical indenter and a homogenous half-space, the contact area Ar can be estimated as :

$$A_r = \pi\left(\frac{3(L)R}{4E}\right)^{2/3} \quad (3)$$

Where E is the effective elastic modulus and R is the effective radius of curvature.

Combining equations. (2) and (3), the friction coefficient corresponding to the pure elastic adhesion is given by :

$$\mu_a = \pi\tau\left(\frac{3R}{4E}\right)^{2/3} L^{-1/3} \quad (4)$$

Where μ_a is the coefficient of friction, τ is the average shear strength during sliding and L is the applied load. E is the effective elastic modulus and R is the effective radius of curvature. Thus the coefficient of friction is inversely related to the cube root of the applied load and the observation of Fig. 8 well conforms to this relation. The adhesion is the dominant friction mechanism [17]. where the coefficient of friction decreases with the applied load.

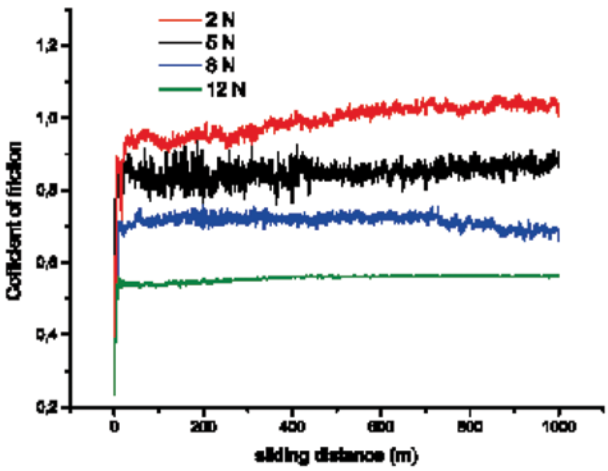

Fig. 7. Evolution of the friction coefficient along the sliding distance for the loads tested. [15]

In the previous work, it was shown that wear is increased by the increase of loads; this can be attributed to the increase of normal load and shear stresses, causing plastic deformation and material displacement leading to material removal at high loads. [15]

It is generally difficult to clearly establish the dominant sliding wear mechanism; given that this wear is known to occur mainly by adhesion [18], [19], delamination [20] and/or fretting [21] ; in the present case, to identify wear mechanism, worn surfaces were observed by SEM.

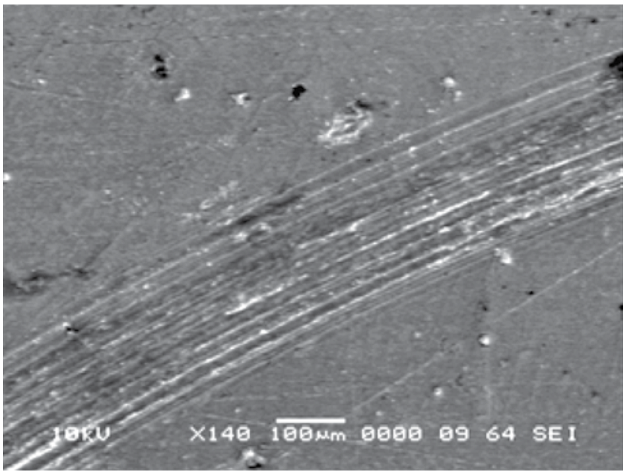

***Fig. 8** abrasive wear of C35 steel substrate 2N 40 mm/s [15]*

The wear of wrought mild steel (uncoated sample) was observed in fig.8. It can be seen the grooves made by the abrasion of hard ball under load of 2N due to the big difference of microhardness of two bodies. However, for coatings with higher hardness and their complex microstructure will certainly behave differently.

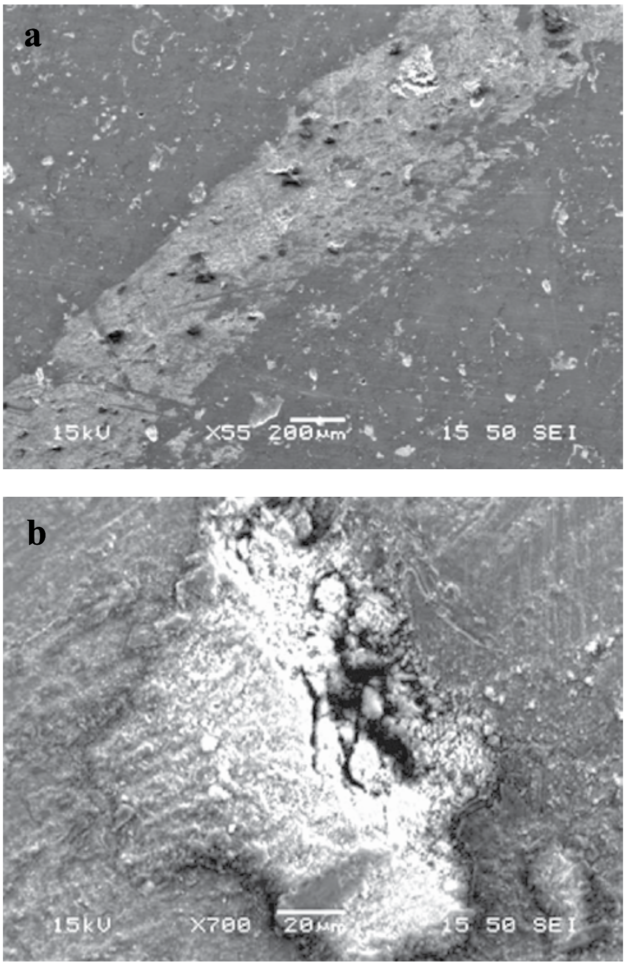

***Fig. 9 a)** worn track on coating under 2N.*
***b)** fracture of splats near the macropore. [15]*

At lower load 2N, there is no full contact between the pin and coated disc. Because of this, near the worn region as seen in Fig9-a, it is possible to observe unworn depth region under the surface. however, fracture of splats can take place near the macro-pore as it is observed in Fig9- b

At 5N of load, the surface worn presented in the figure 10-a show the debris wear produced during friction. The dominant mechanism in this wear process is the delamination of splats which form the coating. Debris appear like-flake produced by this mechanism has high dimensions because it comes from the detachment of single splats, though in other cases it could be generated by the elimination of bits of coating containing more than one splat. [22]

Debris are namely the fraction of delaminated splats of 13Cr coating into ultrafine particles due to their different primary grain size. This may lower the friction coefficient and decrease the wear rate.

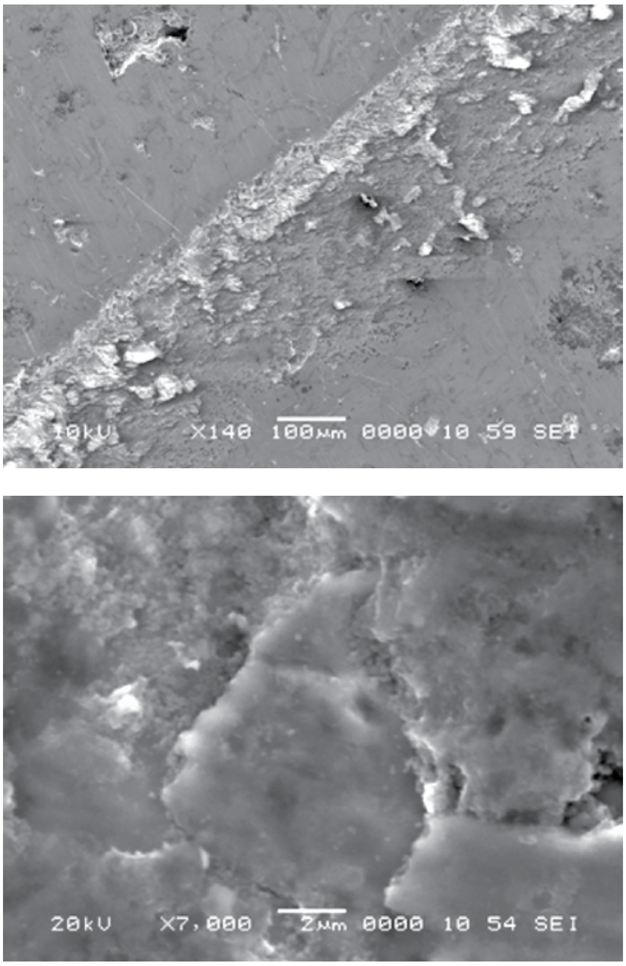

***Fig. 10 a)** worn track of coating under 5N.*
***b)** worn track showing the scale-like under 8N. [15]*

It was reported that the sliding wear of thermal spray coatings could be attributed to splat delamination [14] due to the weak links caused by the oxides veins[23], as it is described in microstructure section and confirmed by EDS microanalysis, and RDX [15].

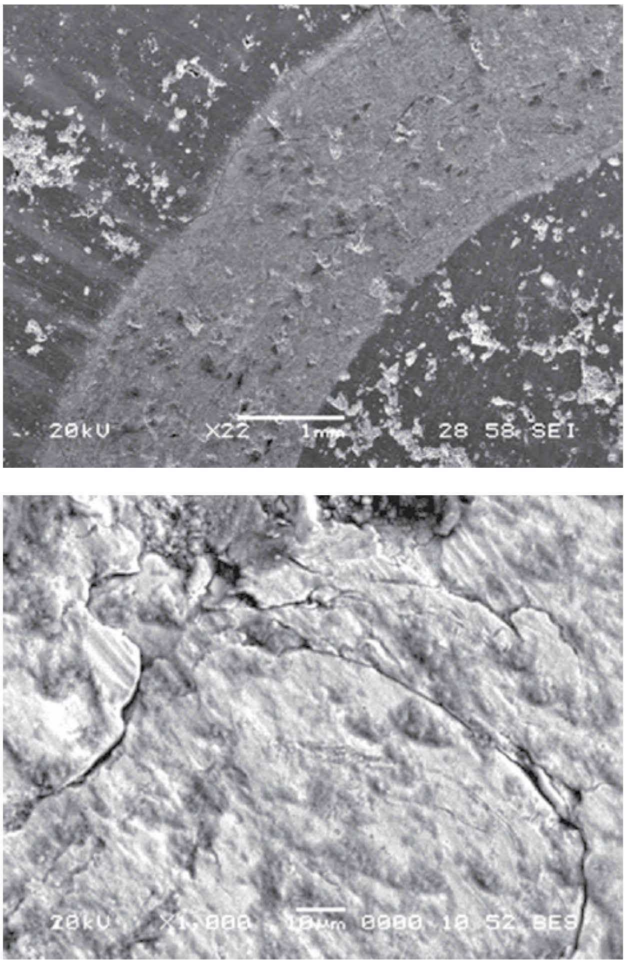

Fig. 11 a) worn track under 12 N of load
b) worn track showing disturbing crack around splat. [15]

At load of 8N, it can be seen that worn track has a scale-like appearance (fig10-b): splat in contact with the counter ball will be subjected to shear stress, leading to delamination within oxide veins. Fig.11-b shows that some cracks distribute around the scaling pits, and a few slight ploughs exist on the coating surface.

As shown in Figure 11-a, the wear rate is the highest for the 12N of load. The wear process of 13Cr coating mainly involves the brittle splat delamination.

It is well known that the wear rate is tribo-system property, this is for what, worn surface of ball was investigated. Figure 12-a shows worn surface of the counter ball at 5N of load with the presence of grooves that can be explained by the complexity of coating microstructure, especially inhomogeneity of microhardness in the coating constituents including hard oxides and resolidified particles with a relatively rough surface. The latter can be understood from the oscillation of COF evolution curves at loads ranging from 2N to 8N.

However, the sphere of counter body 106Cr at 12N was subjected to an adhesive wear, Figure 12-b shows particles adhered into surface detached from coating, due to cold welding between the two surfaces in contact, and in another place a removal area from the surface of ball may due to adhesion with the coating.

3.4. Corrosion test:

The ptentiodynamic polarization curves are represented in figure 13-a, the curves exhibit similar polarization behaviour. It can be seen that the corrosion potential Ecorr is shifted to positive value for the optimized coating corresponding to the distance of 100 mm of spray distance. While the corrosion current density Icorr decreases with the same criterion: spray distance. This parameter is very important for the formation of sprayed coating.

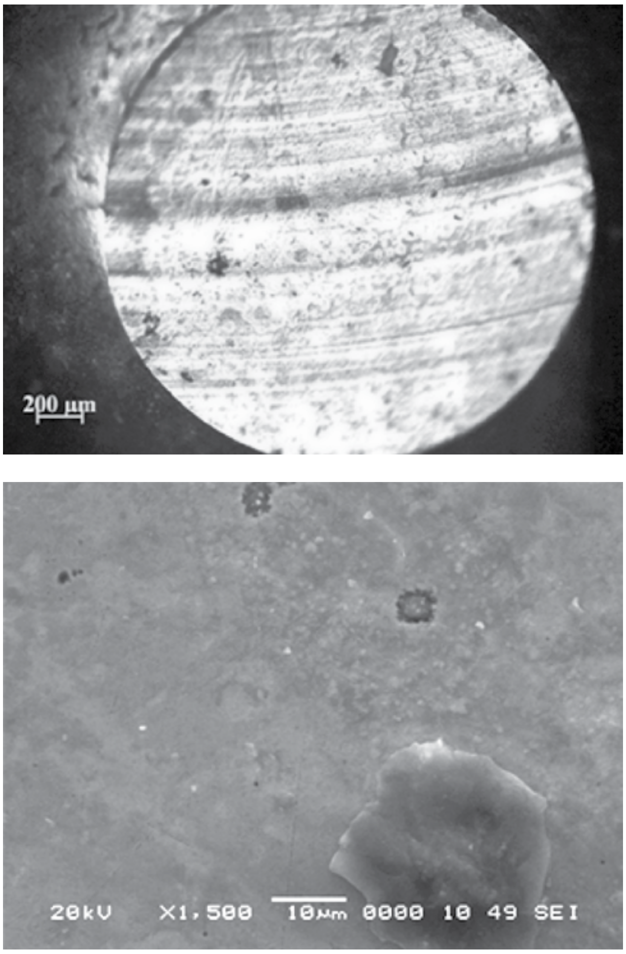

Fig. 12 observation of worn ball indicating transferred material from the coating , and removal of material from the ball by the adhesive wear.

Porosity is the main defect affecting the corrosion behaviour of thermal spray coatings. Especially for the interconnected pores [9], [24] , the electrolyte can reach the substrate and accelerate the corrosion of this latter as the coating is considered cathodic one . The figure 13-b shows the potential curves of optimized coated sample without and with epoxy sealing. The sealed sample show high resistance corrosion with a shift of Ecorr to higher value, with decrease of Icorr to low current density value. [25]

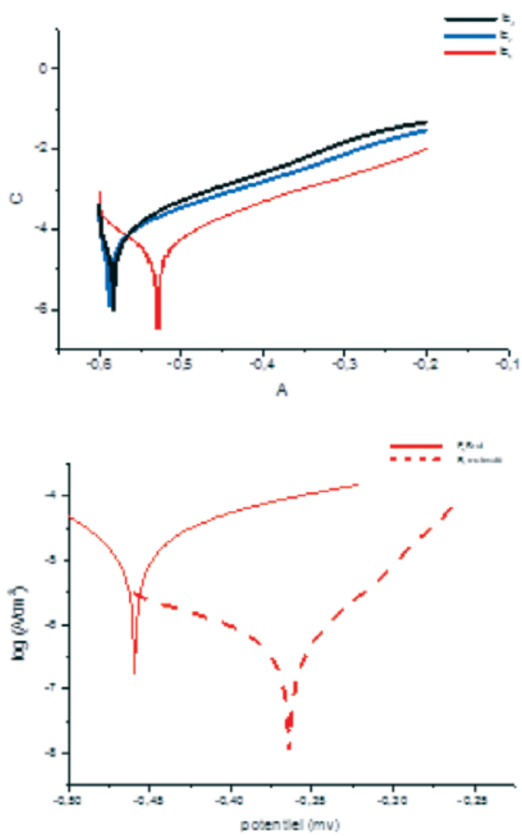

Fig. 13. potentiodynamic curves of a) coating with different spray distance b) optimized coating with sealing epoxy b) without sealing epoxy

The immersion of the coated sample in 3% NaCl solution for a week, permitted to reveal the mechanism of the corrosion. The observation of the coated sample after the test by optical microscopy, showed that the attack of corrosion was in the substrate by the removal of material from the substrate, by the forming of galvanic corrosion between the coating and substrate, created by the contact between the electrolyte and the substrate through the interconnected pores. Furthermore, the attack was along the interlamellar zones, the oxide veins between the lamellae.

It was found [26], that corrosion of thermally sprayed stainless steel coating , occurred selectively at the boundaries of the splats, at the pores and the oxide veins as poor zones in chromium where the corrosion attack the coating itself, leading to deplete the single or blocky splats following the pores as showed in the figure 14

Fig. 14. optical micrograph showing the degradation of coating by corrosion atack in both coaitng and substrate

4. CONCLUSIONS

13Cr steel wire was sprayed on mild steel substrates using wire arc spraying process under different spray distances. The produced coatings exhibit a lamellar microstructure, with different inclusions: splats, oxides, pores.

The microstructural features of deposited coating are different dependently with the spray distance parameter.

The main features are the porosity and splat thicknesses were determined by image analysis software. It was found that porosity is correlated with the splat thickness, this latter reveals the manner of flattening at the impact; witch is related to the spray parameters governing the in-flight particles.

The microhardness in each coating is not uniform, however the mean value is dependent with spray distance, this can be related to the influence on the interlamellar cohesion and the manner how the particles are solidified at the impact.

Corrosion resistance of the coatings is strongly influenced by porosity and the oxides rate. It was found that the corrosion attacks the coating through the oxides being in inter-splats resulting in dissolution of oxides, and attack the substrate through the interconnected pores forming a galvanic corrosion. The degradation of substrate in the interface may lead to coating depletion. However, sealing treatment of surface coating by epoxy can enhance considerably the corrosion resistance.

The interlamellar oxides and porosity in the coating are not undesired features only in the corrosion protection application, but also in wear protection: the oxides decrease the cohesive strength, the zones where the microcracks take place and propagate inducing lamellae delamination. Porosity may affect the wear to become worse not to only increase cracking propagation but cause splat fracture especially with the presence of macro-pores.

It is very common that in a real contact more than one wear mechanism is acting at the same time. Abrasion mechanism can act as wear mechanism especially at loads ranging from 2N to 8N. At high load 12N, a transfer of small particles appears between the two bodies in friction leading to an adhesive mechanism of wear accompanied by delamination. The wear and corrosion behaviours of 13Cr arc sprayed coatings are very sensible to microstructural features such as porosity and splat thickness, resulting from the spray condition. That›s why, a deposit by thermal spray processes need an optimization to obtain the best quality of coatings as desired in application.

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