

WEAR RESISTANCE OF POST HEAT TREATED HVOF COATINGS BASED ON CERMET - NICKEL BASED ALLOYS MIXTURE

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ABSTRACT

The aim of the present work was studied the influence of both the chemical composition and the post heat treatment on the wear resistance of composite coatings deposited by using HVOF JP-5000 gun onto AISI 1020 steel substrate. For such a purpose, prior to deposition, the NiCrBSiWFe alloy powder was mechanically mixed with different amounts of WC-12%Co powder in order to conform three coatings having the following composition: 100% NiCrBSi; 50%NiCrBSi + 50 % (WC-12%Co) and 30% NiCrBSi +70 % (WC-12%Co). An oxyacetylene flame was used to perform the post heat treatment, whose duration was of 300 seconds. The evaluation of the wear resistance of these coatings was achieved by means of abrasion wear tests according to ASTM G65-C. The hardness of the coating was determined by means of Vickers indentation using a load of 300 g. A detailed study of the wear track was carried out by Scanning Electron Microscopy (SEM) combined with Energy Dispersive X-ray microanalysis (EDX) in order to provide information of the wear mechanisms. The results have shown that the wear resistance increases with the increase of the WC-Co amount in the powder mixture. However, this increase is more significant once the post heat treatment was performed, as consequence to both the precipitation hardening, which takes place in the nickel matrix ensuring a more uniform particles distribution, and to a considerable decrease in the porosity.

Key words: WC-12% Co, cermets, HVOF, post heat treatment, wear, abrasion, sliding.

1. INTRODUCTION

Nickel-based self-fluxing alloy coatings have been used in a wide range of industrial applications on account of their combination of high wear and corrosion resistance. This kind of coatings has also become popular because of both their outstanding wear and corrosion resistance at high temperatures and their relatively low cost (Chen,2005 ; Skulev, 2005; Fernandez,2005; Gonzalez, 2007). These Nickel based alloys commonly contain chromium, tungsten, iron, boron, silicon and carbon as alloying elements. Coatings based upon self fluxing alloys generally consist of a nickel-based metallic matrix (γ phase) with several nickel, chromium, tungsten and iron-based borides and carbides. Ni-based self fluxing alloys combines a number of special properties brought by its constituents, e.g. nickel brings excellent wettability which promotes good cohesion, chromium improves the

tribomechanical properties, boron reduces the melting point, silicon increases self-fluxing properties, iron modifies the diffusion rates. Their synergetic effect thus enhances the wear resistance of the coatings (Chen, 2005; Stoica, 2005; Fernandez, 2005; Gonzalez, 2007). However, they do not always give satisfactory results for coating hardness and wear resistance. It is generally believed that W and WC additions secure good wear resistance of the coatings (Skulev, 2005). It has been demonstrated that such composite coatings produced by reinforcement of the nickel bases alloys with hard cermet phase, can offer enhanced wear resistance which is achieved by combining the relative toughness of the nickel based alloy with the hardness of the tungsten carbides. The wear behavior of such coatings depends upon the phases present and their microstructure. This coatings have been obtained using different processing as plasma, detonation gun, HVOF, however, problems associated with the partial melting of the particles causing poor bonding, both at the intersplat and coating substrate interface levels, are still encountered even with the state of the art HVOF liquid fuel system. Many efforts have been applied to improve the properties of coatings such as developing new spray methods, minimizing the size of the spray powder, parameters controlling, etc. One way of improving the coating's performance is through the use of post-deposition heat-treatment. Many post-coating treatments like flame, laser or vacuum furnace heating or infiltration with sealants have been proposed to increase the density and the lamellar cohesion of the spraying coatings. Several investigations over the last decade have established that microstructural changes associated with such heat-treatments can significantly improve the wear resistance and corrosion of thermally sprayed cermet coatings (Rodriquez, 2000; Stoica, 2005; khameneh, 2006; Rodriquez, 2001; Skulev, 2004; Shieh, 1993; Bolelli, 2007; Rodriquez,2002; Shrestha, 2005; Galvanetto,2006). This paper describes the results of research on the wear behavior of a coating of a NiCrBSi alloy mixture with differences percentages of WC-Co particles and then deposited on steel substrate by HVOH process. The wear behaviour were estimated by means of a pin-on-dick test and dry sand test. In order to compare the influence of a post heat treatment on the properties of the coatings some samples were heated using an oxyacetylene flame. The aim of the present work was studied the influence of both the chemical composition and the post heat treatment on the wear resistance of composite coatings deposited by using HVOF JP-5000 gun onto AISI 1020 steel substrate.

2. EXPERIMENTAL PROCEDURE

2.1 Coatings preparation

Two different commercially powders were used as feedstock with the characteristics given in Table 1. Both powders were mechanical blending for 5 hours in a x:y relation with different amounts of WC-12%Co powder in order to conform three coatings having the following composition: 100% NiCrBSi; 50%NiCrBSi + 50 % (WC-12%Co) and 30% NiCrBSi +70 % (WC-12%Co). Prior to the coating deposition, industrial methodology of grit blasting, cleaning, and then preheating was followed. HVOF (JP-5000 system) spraying was carried out using the parameters industrially optimized for this material in order to produce low porosity and high density. Deposited samples were post heated using an oxyacetylene flame during 300 sec, until the surface reached a glazed shine (around 1100°C), typically characteristic of the self-fluxing alloys.

Table 1: Starting Powder Characteristics

Powder	Average Particle size	Particle shape	Manufacturing	Composition (wt.%)
NiCrBSiWFeC	-315mesh +15 μ m	Spherical	Atomization	15Cr, 17.3W, 3B, 4Si, 3.5 Fe,0.8C, Ni Bal.
WC-12%Co	-315mesh +15 μ m	Spherical	Agglomerate-Sintered	82.9W, 4.09 C, 11.61Co

2.2 Coating Characterization

The characterization of the coatings included cross-sectional SEM images and EDS phase analysis. A scanning electron microscope was used to examine the morphology and the microstructure of the coatings and worn surfaces by using both the secondary electrons (SE) and backscattered electrons (BSE) signals to generate images. X-ray diffractions (XRD) of the resultant coatings were undertaken on a diffractometer, using Cu-K α radiation. A 2 θ diffraction angle from 20°-100° was investigated. The microhardness of the coatings was measured using a Vickers microhardness tester with a load of 300 g applied for 15 sec. Values quoted are an average of eight indentations for each coating and they were performed on the polished cross-section of both as sprayed and post heated samples respectively.

2.3 Wear testing

The abrasive wear resistance was evaluated in accordance to the ASTM G-65. The samples of 75mm \times 25.4mm \times 6mm were coated. The coating thickness was above 600 μ m. All tests were performed with dry quartz sand 50-70 mesh (300 μ m-212 μ m) at a feeding rate of 350 g min⁻¹. The normal applied load was 130N and the wheel speed was a 200-rpm. Abrades volume loss of the coatings (>3 replicate experiments) was calculated using the weight loss measured divided by the density of the coating which were calculated by Arquimede's method for each mixture. The results are presented in table 2. Mass loss measurements during wear were determined using an analytical mass balance with a sensitivity of 0.1 mg.

Tabla 2: Coatings densities

Coating	Density gr/cm ³
As deposited 100% NiCrBSi	7.57
Post treated 100% NiCrBSi	10.51
As deposited 50% NiCrBSi 50% WC-Co	9.3
Post treated 50% NiCrBSi 50% WC-Co	9.5
As deposited 30% NiCrBSi 70% WC-Co	10.06
Post treated 30% NiCrBSi 70% WC-Co	11.70

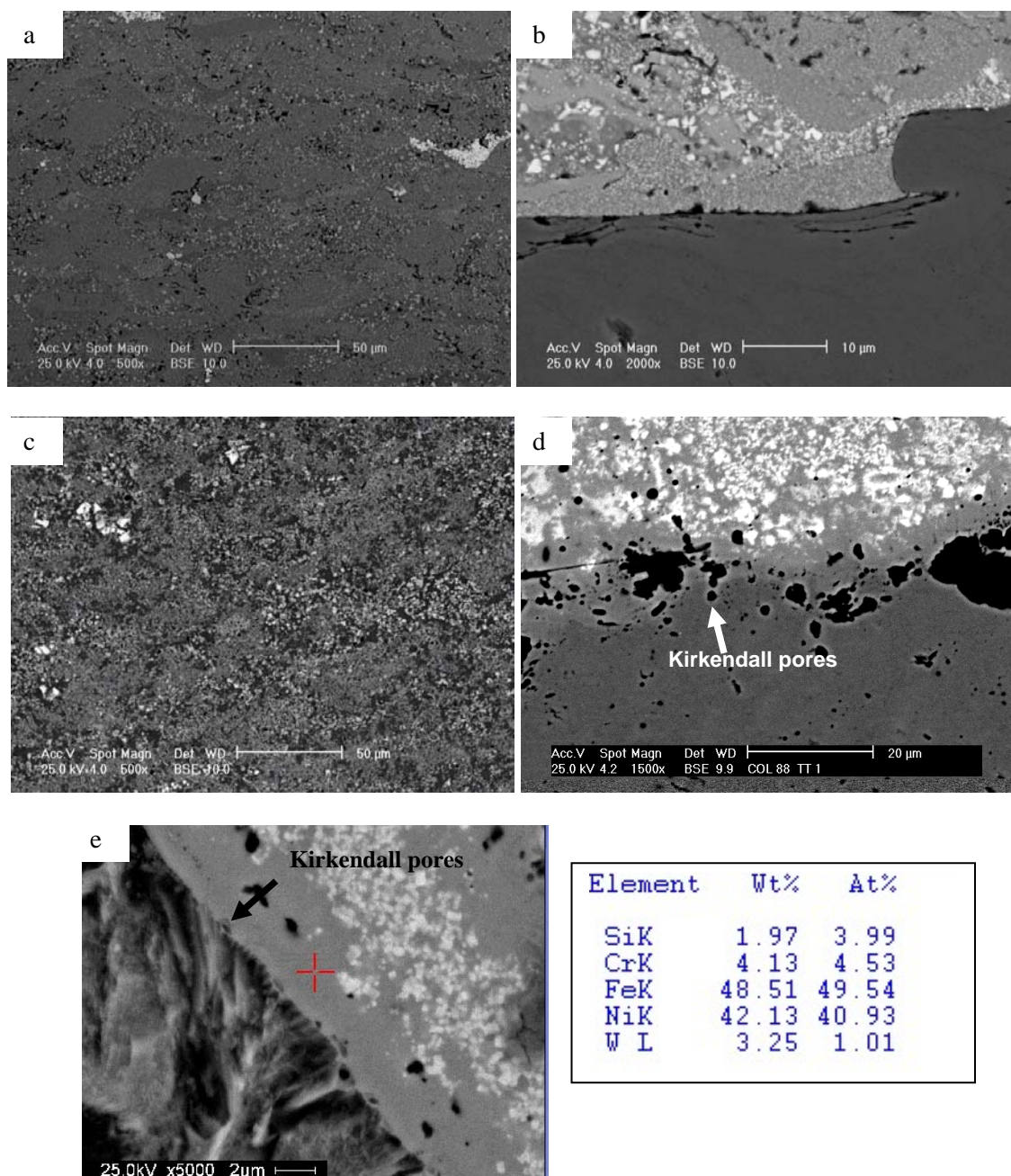
3. RESULTS AND DISCUSSION

3.1 MICROSTRUCTURAL ANALYSIS

Figure 1 a-e show backscattered SEM micrograph of the NiCr based coatings on the polished cross-section with and without post heat treatment. The coating in Fig 1 (a) shows a high density and a heterogeneous distribution of the particles into the coating. The lamellar structure, characteristic of the thermal spray coatings can be observed in this figure, nevertheless a few unmelted particles can be observed indicating that the temperature of the flame was higher enough. Figure 1 (b) shows a detail of this coating at the interface. It is more evident at this magnification, that the microstructure is formed by bright angular particles with different sizes within the dark nickel matrix. These phases have been reported earlier (Rodriquez, 2002). The lamellar structure observed in fig. 1a, has completely disappeared after post heat treatment which could indicate that an increase in cohesion resistance between the lamellas. The coating's density has increased approximately 39% after fused probably due to coalescence of porosity. As shown in figure 1(b) there is a clear interface between the substrate and the coating of the as-sprayed coating. Contrary to this, the boundary was eliminated in the heat-treated coating due to the formation of an interfacial layer of about 4 μ m thickness, this layer have been previously reported. In spite of the small size of this layer its existence was further confirmed by the elemental analysis (EDX) carried out on the substrate, neighboring the initial interface (see fig. 1e).

The EDS analysis revealed the existence of a considerable amount of Ni and Fe, suggesting that diffusion controlled processes took place, leading to a metallurgical bonding between the coating and the substrate. The

formation of a diffusion layer was the result of the Kirkendall effect and is the end result of two chemical species (Ni and Fe) diffusing in opposite directions at different rates (Kim, 2007). Nickel is the dominant species in the diffusion layer because nickel diffuses from the coating across the diffusion layer into the substrate more rapidly than iron in the opposite direction, leading to unequal fluxes of the two species from different directions (Kirkendall effect). A net diffusional mass flows from the coating to the substrate representing the diffusion layer growing by consuming the coating. As the diffusion layer moves towards the substrate, an excess of vacancies which later become pores (Kirkendall pores, see narrow in fig 1e) form in the coating neighboring the diffusion layer. As the diffusion layer moves towards the coating, the Kirkendall pores become part of the diffusion layer. Hence, it was expected that there will be improvement in the coating's adhesive strength and microhardness.



Element	Wt%	At%
SiK	1.97	3.99
CrK	4.13	4.53
FeK	48.51	49.54
NiK	42.13	40.93
W L	3.25	1.01

Fig. 1: Backscattered SEM micrograph of the NiCr based a) As deposited 500X b) Interface 2000X as deposited coating c) Post heat treated 500X d) Interface 1500X post heat treated e) Detail of the post heated coating at interface 5000X, showing EDS analysis

The microstructure showed in Fig 2 looks very different that one corresponding to the coating without mixture (See Fig 2a).

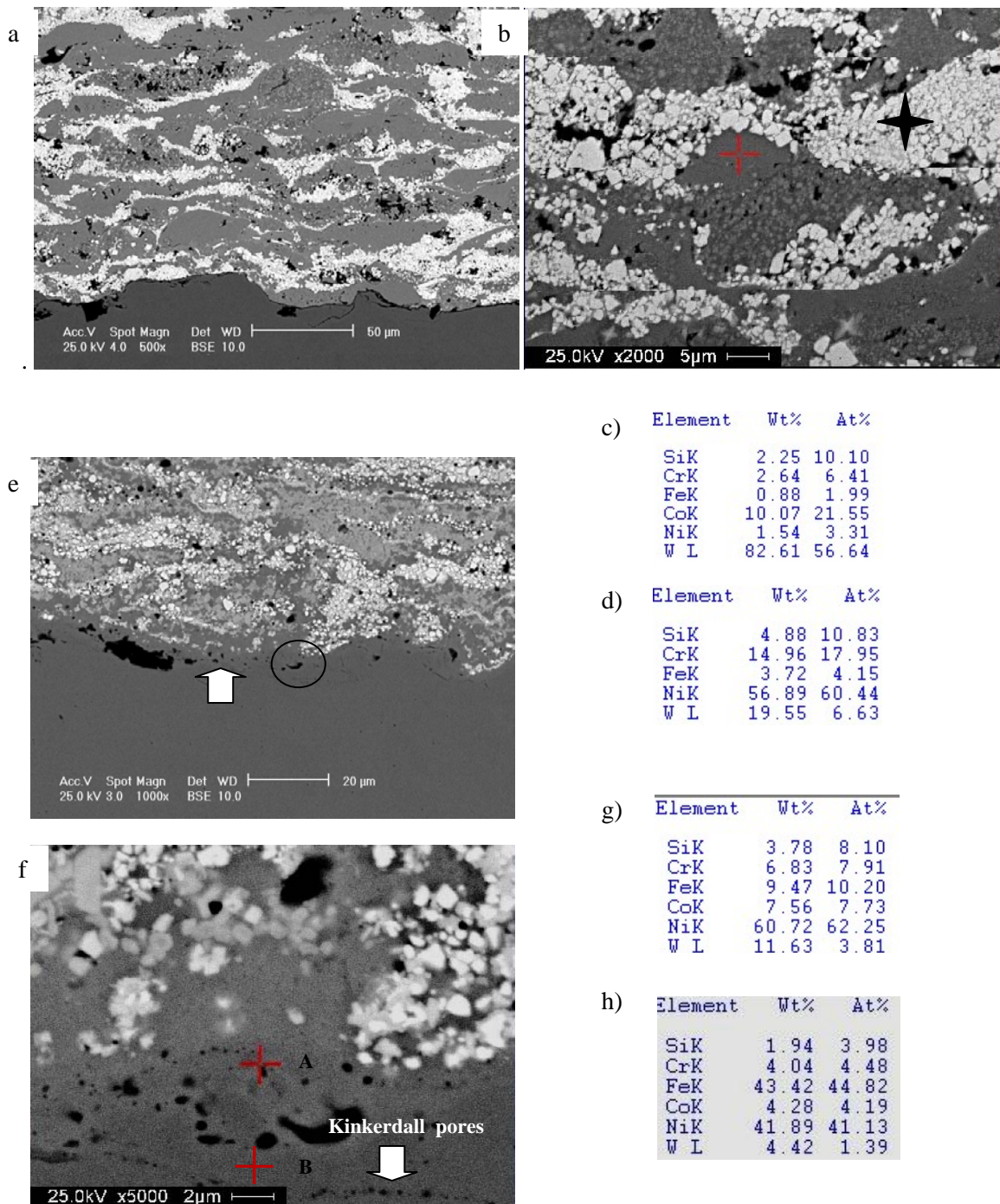


Fig. 2: Backscattered SEM micrograph of the 50% NiCrBSi 50%WC-Co coating a) As deposited 500X b) 2000x c) EDS analysis in white phase d) EDS analysis in black phase e)Interface 1000X in post treated sample f) Diffusion zone at 500X g)EDS taken in red mark h)EDS taken in white mark

The lamellar structure is more evident due to the colors contrast of the phases. There is some intersplat avoids as a consequence of two kinds of powders mechanically mixed and probably inadequate spray parameters that have been used. Nevertheless there are few unmelted drops, indicating that the particle's temperature was higher enough. A definite interfacial line is observed between the substrate and coating. Fig 2b corresponding to a magnification of Fig 2a (see black circle) and EDS analysis (fig. 2c) done in the white phase (indicated in this figure with black star) revealed the presence of W and Co corresponding to WC-Co alloy. Different sizes and morphology of carbides can be observed. Fig. 2d shows an EDS analysis taken in dark phase in the same Fig 2b (see red pointer), which reveals the components of the Ni based alloy. Upon microstructural examinations, the splat boundary layer was experienced some changes as consequence of post heat treatment, as shown in Fig 2e. The splat boundary layer became less define as well as the interface coating-substrate and no interlamellar voids were observed. The coating obtained after fused treatment was dense and less porous than the sprayed coating due to coalescence of individual splats. An increase in hardness to about 17% was measured on the 50-50 fused coating, which was attributed to both the densification of the coating and the formation of Ni-rich hardphase particles. This was confirmed by XRD, which differentiated from the sprayed coating and with the appearance of $\text{Cr}_2\text{Ni}_3\text{B}_6$, $\text{Fe}_3\text{Ni}_3\text{B}$ and $\text{Ni}_{2.9}\text{Cr}_{0.7}\text{Fe}_{0.36}$, which were not present in the sprayed coatings. The spectra of the heat-treated coating show a number of complex tungsten carbide phases. These phases ($\text{Ni}_2\text{W}_4\text{C}$, FeW_3C , and $\text{Fe}_6\text{W}_6\text{C}$) have been produced on the expense of primary tungsten carbide (WC), secondary tungsten carbide (W_2C), and metallic tungsten (W). In addition to these phases, the spectra reveals reactions within the matrix, which resulted in new phases such as nickel boride (Ni_4B_3), chromium borides (CrB_6 , Cr_2B_3), and chromium carbides (Cr_3C_2 , Cr_7C_3). A diffusion layer of approximately 6- μm at the coating/substrate interface is observed in Fig. 3e, which is in good agreement with the result reported in the literature for WC-Co and γ Ni-B-Si composite coatings post heat treated. Kirkendall pores also can be observed in this sample. It appeared that Ni and Cr diffused into the substrate and Fe dissolved into liquid NiCrBSi(Co) alloy. The iron diffusion from substrate toward coating is confirmed by semi quantitative EDS analysis (see fig. 2g and point A in fig. 2f). The iron percentage present at the beginning of the diffusion zone is about 9.5 %w meanwhile nickel is about 60%w. The semi quantitative analysis EDS has indicated that at the bottom of the diffusion layer, nickel has almost 42%w and iron has 43%w (sees fig. 3h and point B in fig. 3f). This high nickel's value near to the substrate indicates an effective diffusion of this element has occurred. The densification of this coating after post heat treated is negligible. The principal mechanism of coating's densification after post heat treatment is the coalescence of individual splats and this is probably less effective due to the differences in the chemical composition of the phases.

Increasing the percentage of CW-Co in the mixture the coating looks as showed in fig. 3a. The lamellar structure is evident and there is a majority presence of the white phases due its composition is 70% wt. Some porosity and interlamellar avoids can be observed. After fused, the gray phase has almost disappeared as well as the interlamellar avoids, less porosity and lower size of the pores can be observed. Fig. 4c corresponds to the interdiffusion layer. Three EDS analysis have been taken in the points identified as A, B and C in this figure. As shown the results of this analysis the iron has a high level in the zone A, very close to the coating, meanwhile the nickel is present in the bottom of the diffusion layer, close to the substrate.

3.1 MICROHARDNESS

The hardness of the coatings under study, with and without post heat treatment obtained through Vickers test is shown in Fig. 4. We can observe that the post heat treatment improved the microhardness of all coatings. The best improvement was for the 100%NiCr coating which reach an increase of almost 42% after post heat treatment. This behavior was previously explained in an earlier work (Rodriguez, 2002). The hardness' increase of the others two coatings were of approximately 17%. This improvement of hardness after post heat treatment can be explained by the formation of hard phases and a decrease of tensile residual stress in coating layers formed during thermal spray. It is noticeable that the post heat treatment of 100%NiCr coating gives a similar value of microhardness compared to 50-50% coating post treated.

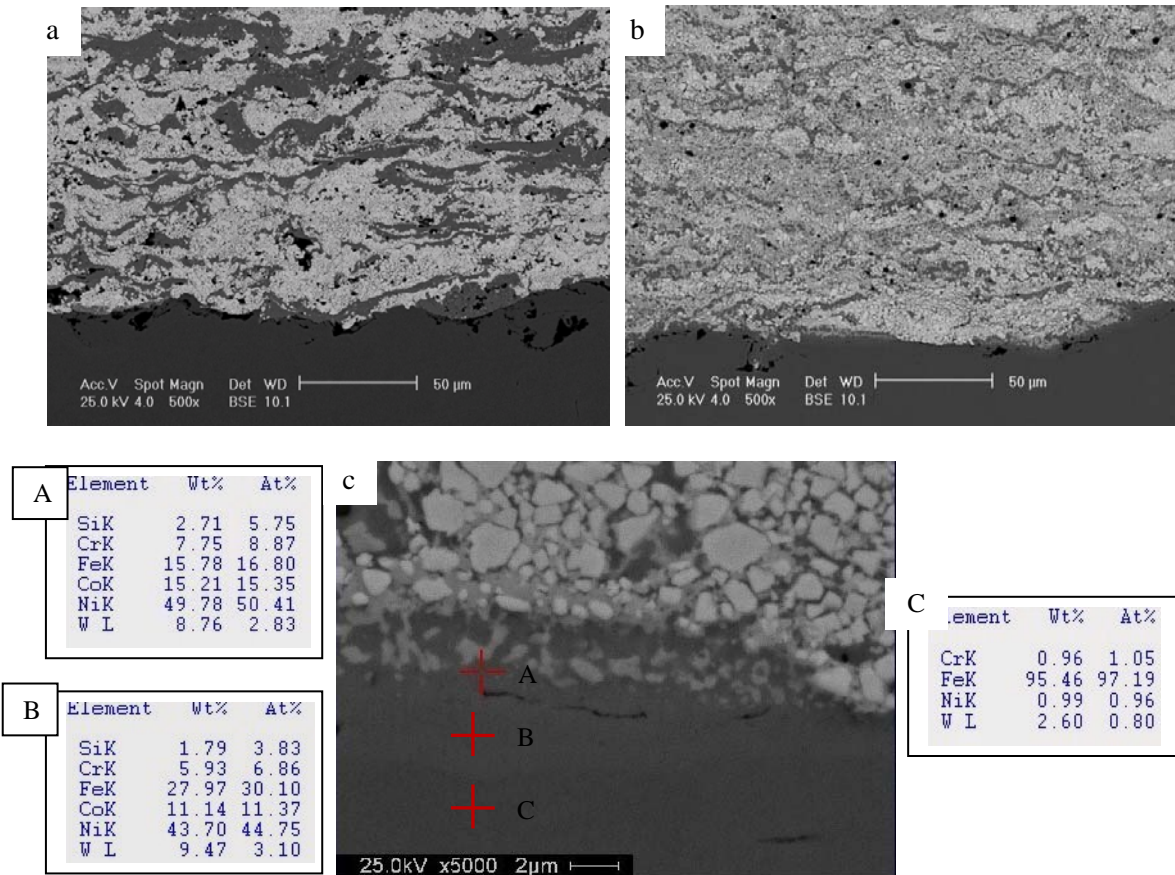


Fig. 3: Backscattered SEM micrograph of the 30% NiCrBSi -70%WC-Co coating a) As deposited 500X b) After post heat treatment 500X c)Diffusion zone at 5000X

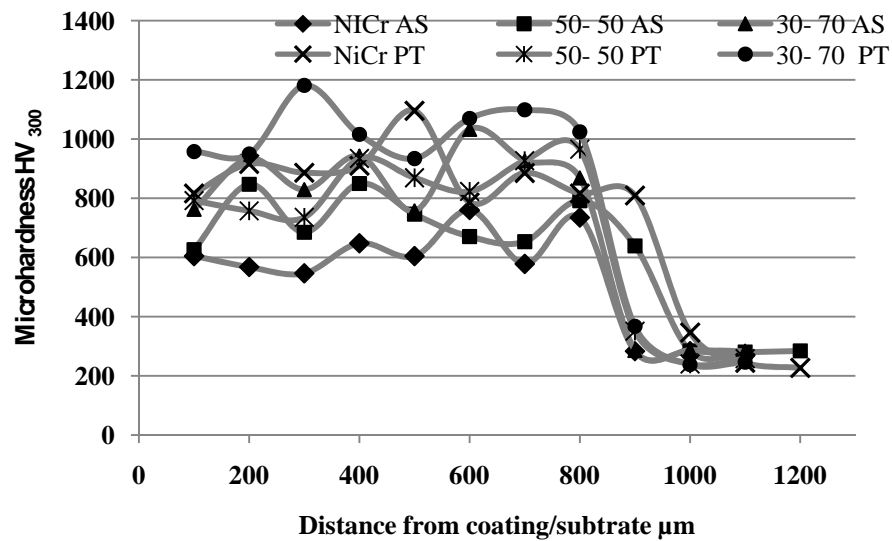


Fig. 4: Variation of coating microhardness with the distance from the coating/substrate

Tabla 4 : Porosity

Coating	Porosity	% Variation
NiCr AS	0.9205	
NiCr PT	0.2165	71.91
50-50 AS	1.2068	
50-50 PT	0.4043	59.17
30- 70 AS	1.5375	
30- 70 PT	0.6205	67.30

3.2. ABRASION WEAR BEHAVIOR

Abrasive wear resistance of the WC–Co–NiCrBSi composite coatings was evaluated by a dry sand rubber wheel technique according to ASTM G-65 standard. The wear rates of the coatings are shown in Fig 5 as a function of heat treatment and test time. In all cases the volume loss is proportional to the time test. The 100% NiCr coating presented the worst behavior against abrasive wear for all times studied. The wear resistance improved as the WC-Co particles were increased in the mixture. The best behavior against abrasive wear was reached by 30%NiCrBSi-70% WC-Co (30-70) composite coating, with and without post heat treatment, for all times studied. The results shown in Fig. 6 clearly demonstrated that heat treatment led to improvements in abrasive wear resistance for all coatings under study. the 100%NiCrBSi post treated coating, which reduced its wear rate by more than 3 times, 4 times and 5 times for test times at 5, 10 and 30 minutes respectively, in comparison with that of as-sprayed coating. The wear rate of 30-70 samples was reduced by 2.9, 5.44 and 3.20 times in the same times conditions. In the same way the 50-50 sample after flame post heat treatment diminished its abrasion wear in 3.5, 4.1 and 5.36 times for the test carry out at 5, 10 and 30 min respectively, compared with as spray coatings. After 30 minutes the abrasion test, wear traces were observed at a magnification of 500X using SEM in secondary electrons mode as can be seen in Figure 6a-f. Fig. 7a corresponding to the 100% NiCrBSi coating as sprayed. Grooving is clearly present in the matrix. It can be observed that the soft Ni matrix was easily worn and suffered preferential binder removal. Traces of the original grooves produced by original machining are not visible instead of deep grooves produced by the quartz sand are present in this worn surface. Micro cutting and some brittle particles are still visible on the worn surface.

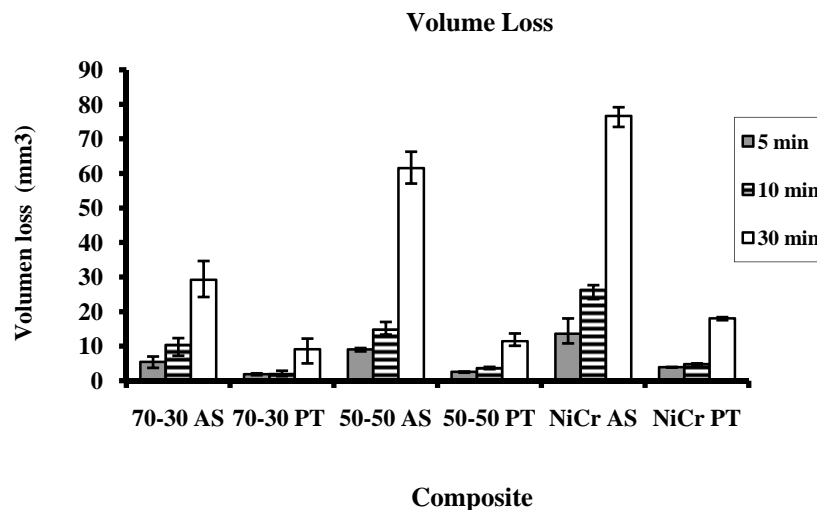


Figure 5. Wear resistance of the composite coating

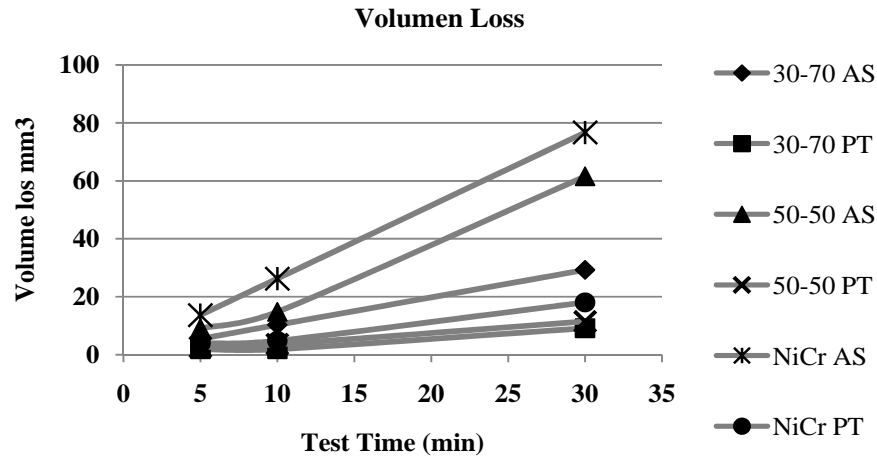
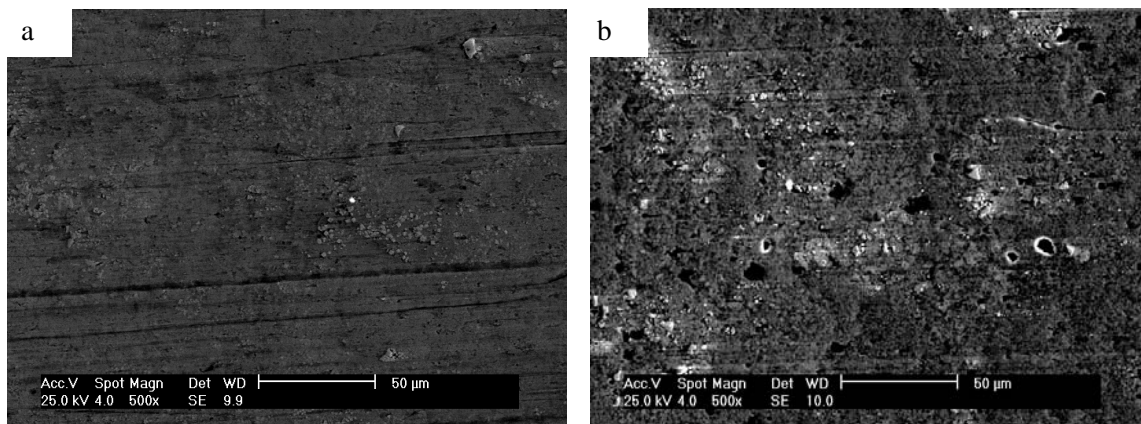


Fig 6: Volumen Loss

It seems to be that during the wear process, the quartz abrasive scraped out the relatively soft Ni matrix between hard phases particles leading these hard particles exposed and as the test continue they are eventually breaking-out by the quartz abrasives. The toughness of the matrix avoid micro-cutting. Meanwhile, another important element for the abrasion resistance of cermets is the bond strength between the hard particles and the matrix (Liao, 2000). This implies that the low bonding strength between hard phases and the Ni matrix and the relative low toughness of the matrix should be the responsible by the lower wear resistance of this coating compared to the others studied. After post heat treatment the worn surface looks very different (See Fig. 7b). The grooves produced by machining can be easily observed indicating that unnoticeable wear process has occurred. In some areas of the track material has been removed as metallic pieces, leaving behind round holes. The effect of this flame post heat treatment on the wear resistance is probably due to a better cohesion between lamellas and hard phases, less porosity, additional carbide phases in the splat boundary layer and an increased of the hardening of Ni matrix as a consequence of the rapid cooling. In fact, the hardness of the as sprayed coating increased 42% after post heat treatment.



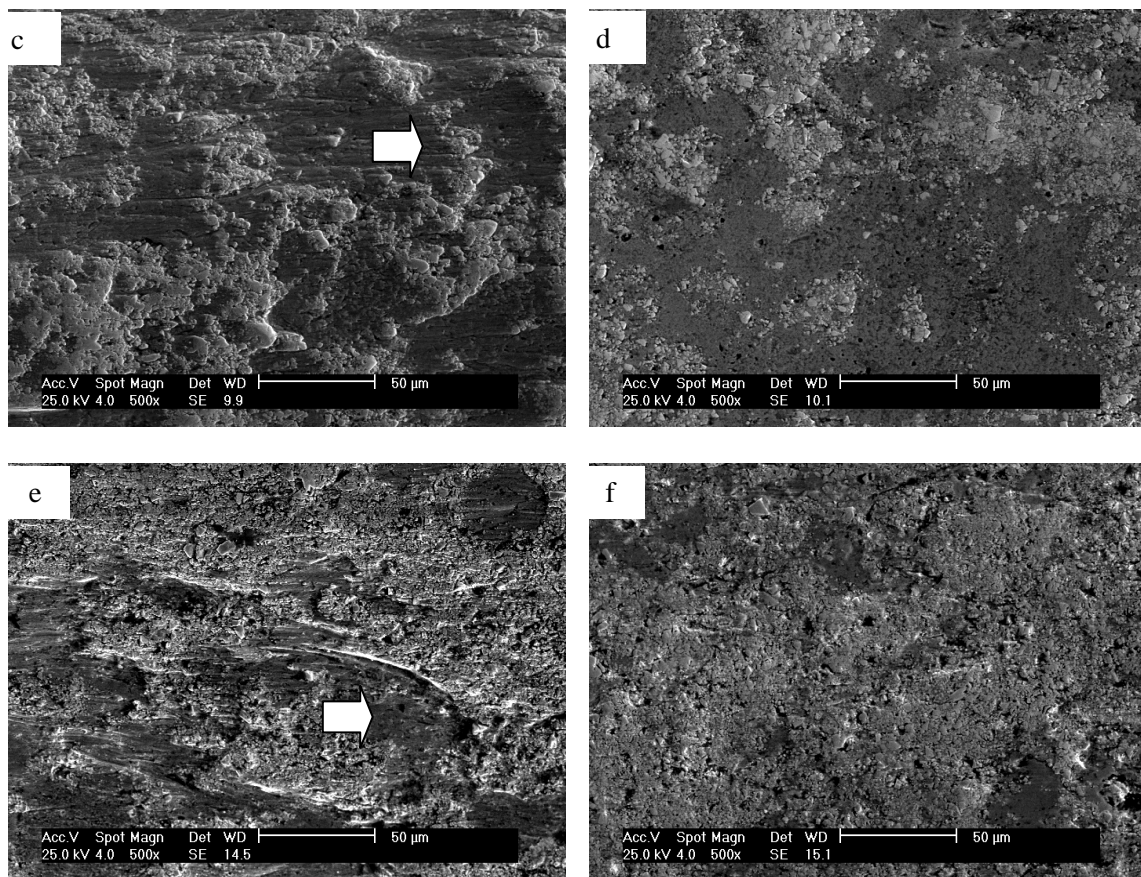


Fig. 7 :SEM micrographs at 500X, showing the characteristic morphology of wear scars after 30 minutes test a)100%NiCrBSi as sprayed b) NiCrBSi after post treated c) 50-50 as sprayed d) 50-50 post treated e) as sprayed f) 30-70 post treated

All these factors improved the coating performance. The probably wear mechanisms in this sample were abrasion of the Ni matrix and pull out of the hard phases. The mixed 50-50 improved the wear resistance of the coating in 19% compared with the 100% NiCrBSi coating. The worn surface is presented in Fig. 7c where probably the wear mechanism would be similar to the coating without mixing but in this case the mayor fraction volumetric of carbides and bigger size of these carbides prevents the matrix removal and posterior pull out of carbides. This increased carbide volume fraction made a major contribution to the improved coating performance [13]. Nevertheless, the sizes of the abrasive grains are larger than the hard particles and so the abrasive particles cut or plough the coating, breaking hard particles or pulling them out. In the wear process the quartz abrasive scraped out the relatively soft NiCrBSi(Co) matrix between hard phases first as the scratches interrupted by WC particles are shown in matrix (see arrow in Fig 7c). In summary, the wear loss of this coating results from three distinct mechanisms: the wear of the matrix, the wear of the large phases, either by micro-cutting or micro-cracking, and the extraction of the small phases from the matrix (normally by the particle/matrix interface). The post heat treatment also improved the wear resistance of the 50-50 coating in 5.36 times and the microhardness in a 16%, compared to the same coating without treatment. But the wear behavior of this coating is only 0.63 times better than de 100%NiCr coating post treated. It indicates that in spite of the higher fraction volume of carbides present in the 50-50 sample, the flame treatment is more effective in the sample without mixed. It could be due to the flame post heat treatment promoted the formation of additional hard phases (demonstrated by XDR analysis) and the better cohesion between splats boundary. The higher the bonding strength, the better the wear resistance will be [17,20]. In this case the matrix's microstructure (see Fig. 7d) looks with a better cohesion between hard phases and its removal because of the effect of the quartz abrasive it is not as easy as occurred in the coating without

treatment. The main mechanism of wear is by removal of the binder phase by the abrasive particles, followed by undermining of the carbide particles leading to their pullout.

As the binder matrix diminishes in the 30-70 coating as deposited, the volume fraction of the hard phases is higher and this implies a better abrasive wear resistance. Some researcher's [19] suggest that wear resistance is increased in the same proportion as the volume of the hard phases. A large carbide size means that the binder phase must be removed to a greater depth before the particle itself can be removed and, therefore, larger particles are more resistant to pullout. This coating behavior was 2.6 times better than the coating without mixture but the 100%NiCrBSi coating post heat treated was 1.62 times better than this 30-70 as sprayed coating. This is very important when costs are involved.

Nevertheless, the abrasion wear best performance was reached by the 30-70 post treated which behavior was 3.2 times better than the same coating without treatment and 1.2 times better than the 50-50 flame treated coating and 1.97 times better than the 100%NiCrBSi flame treated coating. It is noticeable that the microhardness improvement as a consequence of flame post treatment was just 2.1% indicating that the hardness necessarily is not directly related with wear resistance. Thus, there is certainly a role of hardness; harder is better, but it is not a direct proportionality (Budinski, 2004). The performance of coatings was highly dependent on the changes within the coating microstructure. In this case the wear mechanism would be similar to the coating without post treatment but a less rate because the matrix is hardened due to the rapid cooling during the post heat treatment which avoids its wear and then the easy removal of hard phases.

4. CONCLUSIONS

The results have shown that the wear resistance increases with the increase of the WC-Co amount in the powder mixture. However, this increase is more significant once the post heat treatment was performed, as consequence to both the precipitation hardening, which takes place in the nickel matrix ensuring a more uniform particles distribution, and to a considerable decrease in the porosity. The abrasion wear best performance was reached by the 30-70 post treated which behavior was 3.2 times better than the same coating without treatment and 1.2 times better than the 50-50 flame treated coating and 1.97 times better than the 100%NiCrBSi flame treated coating.

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