Alternative to chromium, a hard alloy powder NiCrBCSi (Fe) coatings thermally sprayed on 60CrMn4 steel. Phase and comportements

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Abstract

The purpose of this study was to investigate and to compare microstructural properties, wear resistance, and potentials of thermal spraying of NiCrBCSiFe coatings for a possible replacement of hard chromium plating in mechanical parts repair. The coatings surfaces and cross section were characterized before and after testing behavior using a scanning electron microscopy (SEM) coupled with an analyzer (EDAX). The characterization of phases was carried out by electron microprobe and X-ray diffraction. A good adherence of coating NiCrBCSi (Fe) on steel substrate is explained by formation of large diffusion zone in interface after annealing and by the nature of the structure deposit duplexes. This is explained by the split structure that we were able to characterize and which is composed of a phase lasts type boro carbide-nickel Ni$_3$(BC) and the matrix based Ni$_4$ reinforced by very fine rich Cr precipitates. These properties are enhanced by cyclic heat treatment.

Keywords: NiCrBCSi (Fe) Coatings, Hard chromium plating, Thermal spraying, HV microhardness, Wear resistance

1. Introduction

The development process wet electrolytic surface treatment is mainly based on the aqueous phase and rinsing baths. Water plays an important role in the industry of electrolytic surface treatments, and today the high cost of water and its treatment requires a reduction in consumption. During the last decade, there was a growing interest about the treatment of coatings using chromium electroplating. This is due to the toxicity of hexavalent chromium [1] to the environment, health and safety related to handling, storage and disposal of hexavalent chromium (Cr$_6^+$) [2, 3, 4, 5 and 6]. Furthermore, in terms of performance, the electrolytic bath based on ions, is currently unable to make thick deposits. The dry treatment, including treatment of steam and thermal spraying as alternatives seem very relevant and complementary to the mentioned problems [7,8,9 and 10]. Thus, alternative processes by thermal spraying have a genuine development, and are asking more and more attractive as an alternative to electroplating.

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through a wide range of materials used, good performance coatings, and the coverage massive parts used in engineering (aerospace, automotive, naval, etc...). These include thermal spraying to flame or arc (supersonic HVOF, plasma APS, etc...) where the reached temperatures are above 10000 °C and the large gas velocities are its main advantage and allow deposits to obtain denser, more adherent and more resistant to wear and corrosion. The materials are injected in the form of powder in a sting. The powder substance form of droplets is projected at high speed in the gas stream into the room where it crashed. The adhesion of the deposit is ensured by mechanical attachment on the substrate which is, in most cases, previously sandblasted. In this regard, and before renovating parts by hardfacing, which are commonly repaired by chromium electroplating, we selected three laboratory powders NiCrBCSi (Fe) base metal and an ordinary steel substrate 60CM4. The result of this study was to investigate and to compare potentials of HVOF sprayed NiCrBCSi (Fe) coating for a possible to replacement of hard chromium plating. The physico-chemical characterization, mechanical and tribological deposits made by coating and electrolytic chromium, allows revealing, that deposit hard chromium possesses a large and deep delaminating. On the other hand, deposits NiCrBCSi metal (Fe) to hard reset show better properties and performance compared to hard chromium deposits. Including the filing NiCrBCSi (Fe) type A, this may be an appropriate alternative to hard chromium and enable better protection of the environment. This is due to the existence of microstructure, composed of the Ni3B nickel boride and matrix reinforced by nano precipitates rich in chromium.

2. Experimental procedures

60CrMn4 (Euronorm) steel was used as basic substrate for coating. Three types of spraying powder of nickel based alloys A, B and C were selected having the same chromium composition Cr (11-13.5 at.%) but differ in Iron (Fe: 12-32 at.%) and Boron (B: 7-14 at.%) contents. Comparison of coating manufacturing costs for thermal spraying and for hard chromium plating is given in Refs [11, 12]. The chemical compositions of the used materials are presented in the Table 1.

Table 1. Chemical composition of various spraying powders (at%).

| Element (at%) | C  | B  | Si  | Cr  | Fe  | Al  | Ni  |
|--------------|----|----|-----|-----|-----|-----|-----|
| Coating A    | 2.8| 14.3| 7.1 | 13.6| 12  | -   | Base|
| Coating B    | 5.2| 7.4 | 4.1 | 13.2| 22.4| -   | Base|
| Coating C    | 6.5| 6.8 | 4.8 | 10.9| 32.4| -   | Base|
| Bond coat    | -  | -  | -   | -   | -   | 25  | Base|
| Hard chromium plating | -  | -  | -   | Base| -   | -   | -   |

Preliminary SEM investigations of these powders have revealed the presence of several phases for various powders. In the NiCrBCSi(Fe) top coatings we have revealed the presence/pure nickel with dispersives Ni3B of boride. The typical morphology of type-A spraying powder is given in micrography, illustrated in fig.1.a. This micrograpy obtained by SEM shows crosses sections of NiCrBCSi (Fe) powders particles. At high magnification, observation of cross sections powder particle (fig.1.b) shows that it is completely dendritic structure. This last, is due to the rough state preparation of the powder NiCrSiCB (Fe) by the atomization process, this him gives a aspect property refractory that allows it to withstand relatively high temperatures.
The new composition of Al–Ni bond-coating appears like two types of aggregates Fig. 2.a. The first are rich in Aluminium (90 at% Al) (Fig. 2.b) and the second are rich in Nickel (97.5 at% Ni) (Fig. 2.c), with difference in particle percentage.

Before starting any experiment, the samples were cleaned, degreased and grit blasted according to usual procedures. For all the specimens, the roughness $Ra$ was about 0.46 μm, a suitable value for better adhesion properties.

Both Al-Ni bond-coating and NiCrBCSi (Fe) top coating were deposited with the Sulzer Metco 5P1I Gun thermal spray at high velocity. Table 2 compares some characteristic parameters of the used spraying technique for each coating.
Table 2. Deposition conditions for spraying powders.

| Characteristics parameters of Sulzer Metco 5PII Gun thermal spray. |
|---------------------------------------------------------------|
| Heating of substrate (°C)                                      | 200          |
| Spraying powders rate (l b/h)                                 | 15           |
| Spraying distance (mm)                                        | 200          |
| Gas flow rate $\text{C}_2\text{H}_2$ (l mn$^{-1}$)            | 34           |
| Gas flow rate $\text{O}_2$ (1 mn$^{-1}$)                      | 32           |
| Aires pressure (1 b/in)                                       | 20           |

Samples of conventional hard chromium (CHC) coated steel (350 μm thickness) were provided by an electroplating Spanish industry, using conventional rules (50–60 A dm$^{-2}$, 60 °C, 350 g L$^{-1}$ chromic acid). The thicknesses coat's for deposits (bond-coating and top coating) obtained by thermal spray is respectively 100 μm and 350 μm deposited on a cylindrical substrate steel with, 10 mm of diameter[13].

Microstructure examinations of all used powders and coatings were performed using standard LEICA SEM coupled with an X-ray analyzer (EDX). Cross sections of powder particles were mounted in conducting resin and polished up to 1 μm finish etched reagent consisting of (25 ml FeCl$_3$, 25 ml HCl and 100 ml H$_2$O). The observations were performed using respectively secondary electrons (SE) and backscattered electrons (BSE) signals. Crystallographic structures of powder particles and the resulting phases of the coatings were obtained using X-ray diffraction. Chemical composition profiles were systematically determined through the interfaces between deposited layers and steel substrate using “CAMECA-SX50” microprobe. TiB$_2$ compound and Cr$_7$C$_3$ carbide were used for calibrating boron and carbon while standards pure elements were used for the others. Atomic number, absorption as well as fluorescence effects were corrected using software derived from POPE analysis [14]. Vickers hardness values were determined on coating polished cross-sections under a load of 0.3 kg (i.e., HV$_{0.3\,\text{kgf}}$). Six measurements were performed on three different samples of each nature and the results were also averaged. Finally, wear and friction properties were determined using a simple block-on ring tribometer “developed in LSIMP, INP Grenoble laboratory” which has an aided computer for data acquisition and treatment. Curves representing friction coefficient evolution vs cycle’s number were simultaneously plotted during tests. The sample block was a cylinder of 10 mm diameter whereas the coating represents 5 mm in thickness. The ring of 178 mm diameter was hard steel (100Cr6) with a nominal hardness of about 63 HRC. The tests were carried out under dry friction conditions in ambient air using a line speed of 71 m.mn$^{-1}$ and with load increments of 20 N every 2 min up to 160 N.

3. Results and discussions

3.1. Coatings structural characterization

3.1.1 Nature of deposits

The cross-sectional images of deposits obtained by coating and electroplating, are observed in Fig. 3. The thickness and hardness of deposits obtained by coating and electrolytic are respectively 350 μm; 1090 HV$_{0.3\,\text{kgf}}$ and 350 μm, 793 HV$_{0.3\,\text{kgf}}$. SEM cross section micrographs of type A deposit are illustrated in Fig. 4a. This figure reveals after spraying, the typical morphology of deposits with three layer zones: the NiCrBSiC(Fe) top coating (I), the Al-Ni bond coating (II), the steel substrate (III) and an intermediate layers at the interfaces (I/II) and (II/III). The NiCrBSiC(Fe) coating (Fig. 4b) have a duplex structure, with large balls incorporated in a second phase presenting a darker electronic contrast. The morphology of bond coat (II) is more compact and more homogeneous. It clearly has a fine grain structure at compounds of small balls of electronic contrast. This layer is less porous with a very small size of about 20 μm compared to top coating (I).
The hard coating is in general, intended to function under cyclic working conditions (heating and cooling). For this reason we applied a post-annealing by simulating to the work temperature of certain machine parts [15]. It makes us possible to follow the evolution of phases in bond coating and on the interfaces according to the thermal cycles. After application of several cyclic heat treatments, these layers tend to be homogeneous and wide on the interfaces level, particularly in the interface of bond coating/top coating (II-III) noticed after four thermal cycles. In addition, the bond coating becomes finer and the layer coating records a reduction in the rate and the face of pores. This can be explained by existence of a rich Aluminium phases in the coating.

The atoms of last diffuse bond coating towards the most porous zones of the top coating by effect mass transfer after their softening during post annealing. This established by fact of the melting point of the rich aluminium phases which is close to the temperature of post–annealing at 650° C.

3.1.2 Analyzed phases formed in deposits alternative

The results of quantitative electron probe microanalysis of phases determined in top coating show the existence of two majority phases, the first which is clear containing a constant content of B and Si and can be considered as a carbo-boride type Ni3(BC) which is derived from Ni3B boride. The second phase has a dark grey contrast; observed with high magnification (fig 5.a) shows fine dark precipitates embedded with Ni matrix bases. Their precise nature could not be given because they are very fine for the quantitative electron probe microanalysis, but the qualitative
analysis on quantity shows that they are precipitates which are rich in Cr (fig.5.b). This makes it possible to bring them closer with Cr(BC) boro-carbides.

Certain authors analyzed these phases in interface and in layer coating, obtained under different operating conditions. Among others, the work of sampath and al. [15] noticed the formation of fine precipitates which are rich in Cr embedded with a matrix of nickel bases (Ni-Cr with 25 at% Cr) obtained after a fast cooling by using a plasma gun VPS (vacuum plasma spraying). These precipitates were analyzed by technique EXAFS (Extented X-ray absorption fine structure), noticed their great aptitude for adhesion and their good wear resistance to corrosion under aggressive conditions. Certain authors [16] noticed the formation of borides Ni2B, Cr2B and carbides Cr7C3 in (NiCrBSi) coating performed by plasma arc. Certain authors [17,18] noted the formation of (Ni Cr Fe) with matrix phase of nickel bases and any secondary phases type: CrB2 Fe2B and M7 (BC)3. In the coating with nickel base similar to the our coating [19] carried out by using spraying coating with presence of boride bond coat. They obtained by thermal boridation process, with noted the formation of various borides standard (FeX Cry Ni (l-x-y)z B with z = 2,3 or 4 without specifying the values of x and y. The quantitative electron probe microanalysis of phases obtained in the bond coating (II) is given in table 2. It shows the existence of three differently contrasted clear majority phases and a phase of dark electronic contrast in small quantity. The first one has a matrix content rich in Ni and their content of composition different of Al 5 at % and 10 at% respectively. This difference in chemical composition is due to the chemical heterogeneity of coating obtained at raw state spraying. The third phase formed is rich in Al and contains significant quantities of Si and C (7at% Si and 5 at% C) with no Ni content. In the level of interface zone bond coating / top coating (II-I), the specific chemical analysis made it possible to show the evolution of the phases formed profile while going from the layer of coating towards the bond coat. Thus the phases formed in this interface are made up of solid solution < Niγ>, one of them rich in Ni and the other with the following elements: Si Cr Fe. After annealing at 650 °C we noted a change in the deposit and in level of interfaces after a fourth thermal cycle. The results of quantitative EDX analysis of phases in interfaces zones (I/II and II/III) given in Fig. 6 and Fig. 7 after a post annealing, show the formation of new phases of the interfaces to each zone. In interface I/II (see figure 6) analyzed the Al3Ni, Ni-AlNi and Al13Ni2 phases (Fig. 6.b). These phases are mentioned in the binary diagram Al-Ni. Certain authors noted in similar deposit (Ni Cr B Si and Ni Cr B Si Fe) elaborated under other operating conditions, the formation of these latter phases formed between the various interfaces substrate, bond coat and layer coating. That was highlighted well by work [16] on NiCrBSi powders deposited on an aluminium alloy substrate (from 5 to 10 at% Si) by the laser plasma process.
In the interface of substrate / bond coating (III-II) found an aluminum intermetallic phase (A1_3Ni_2) with dark contrast, identified in retrodiffused electrons (Fig.7). This phase is well characterized in binary alloyings Al-Ni. In addition in these zones, we noticed the formation of new secondary phases nickel bases that rich in Al (20 at%) and the phases of the NiAl type. On the other hand, on the level of bond coating, the matrix becomes homogeneous and has a higher Al concentration than that formed after spraying. It passes from 10 at% to 20 at%, that confirms the widening of interfaces zones in consequently of the elements diffusion on both sides of the interface. Also, we could observe a profile of iron diffusion towards nickel and nickel towards iron. This zone of interdiffusion is spread out over a distance from 40 μm. In the homogeneous zone, approximately 5 μm thickness with dimensions of the substrate, the contents of alloy elements are close to the nominal composition of alloy. After this zone and opposite in a raw state of the deposit, iron extends at a very broad distance from approximately 25 μm inside the deposit, thus showing a significant diffusion of this element after the four thermal cycles. The other elements such as silicon, boron and chromium diffuse more towards the periphery of the deposit.

3.2 Mechanical characterization

3.2.1 Aptitude for indentation
Fig. 8.a shows the profiles of hardness depth for all non annealed samples. The hardness of the substrate was ~ 240 HV$_{0.3}$ and that various for different sprayed coating (B, C and A) is has 620 HV, 750 HV and 1090 HV$_{0.3}$ respectively.

![Graph showing hardness profiles](image)

(a) coatings not annealed

(b) after annealing for coating A

Fig. 8. Micro hardness profiles Hv of the coatings.

After annealed at 650 °C (fig.10.b), the best result obtained for typeA deposit, it has been shown that hardnes for the bond coat decreased from ~ 95 HV$_{0.3}$ to ~ 75HV$_{0.3}$ proportionally with the number of thermal cycles, in particular in interface (II/III) starting from the fourth thermal cycle. Also, it was showing, with according to the number of thermal cycles, an important a improvement of hardness in typeA top coating interface (I/II) and it was changing from about 1090 HV$_{0.3}$ to about 1160 HV$_{0.3}$. This is, due to a more homogeneous of microstructure and distribution of pores, oxides and inclusions. Trivalent chromium process permits to obtain deposits of hardness, equivalent to those resulting from hexavalent solutions, but the behavior in regards with corrosion remains very poor [20]. Coatings manufactured implementing physical vapor deposition process exhibit very high hardness (i.e., 2000–3000 HV) [21] However, the performance of these coatings remains in some cases limited because of their low thickness (1–10 μm), their moderate load capacity and their abrupt interface [22].

3.3 Tribological Performance of the coatings.

3.3.1. Friction behaviour

The friction results of coating, substrate, and hard chromium plating is given in figure 9.a. It shows average value of the friction coefficient for all deposits and substrate at high load and constant speed (P=60N and v=1.3 m/s). The evolution of the friction coefficient records a fluctuation (signals) at least similar for all deposits during all friction tests. We notice a fast increase which corresponds to a friction between asperity of the antagonists, followed by a short stage and the coefficient seems more stable with the formation of the third detached body of antagonists. Then a significant fluctuation, whose frequency depends on the nature of coating and experimental conditions. On figure 8, we notice type-A coating with a low average value on friction coefficient (0.41) compared to all deposit reference hard chromium plating and of the substrate. The coating which recorded a coefficient of larger friction under our experimental conditions is the type-C coating (0.55). Value of friction coefficient of hard chromium plating is about 0.31, which is close for the value of the deposit type A. This coating is with a value slightly lower than the steel substrate (0.67). This explained by the cellular structure of the type-A coating and by the faster elimination of particles than the other coating. That is, we can say that the friction coefficient is better for the coatings than the substrate. Indeed, the latter allow the creation of the particles between the antagonistic parts which form a protective shield against wear [23].
3.3.2. Wear behavior

The wear rate of the type-A coating annealed and not annealed, is presented as a function of cycles in Fig. 9b. The wear rate of all coating given for speed $v = 71 \text{ m/mn}$ according to the load after annealing, shows that:

The best wear dry resistance of type-A coating, compared to the type-C, type-B, hard chromium plating and the reference coating “satellites 6”. Indeed, this reduction of the wear rate after the post annealing is due to the duplexe structure and chromium borides precipitate $\text{Cr(BC)}$ in which the maximum rate in the matrix $\text{Ni(\gamma)}$ s reached after four cycle annealings at 650°C, this reinforces the hardening of the matrix [24, 25].

The good friction coefficient which decrease for the low loads up to a value close to 0.27, which is stabilized from the fourth thermal cycle and the the formation of new phases to the interface reinforcing the adhesion of coating. Therefore the low roughness of surface ($R_s < 7 \mu m$) noted after post-annealing, is due to the significant reduction of the rate porosity in the coating.

![Friction coefficient of deposits and Wear rate of type A coating, according to the loads.](image)

(A) Friction coefficient of deposits (B) Wear rate of type A coating, according to the loads.

Fig. 9. Tribological proprieties of coatings.

Abrasion resistance and friction coefficient, permit the improvement of the wear and friction properties [26] of coatings deposited by thermal spraying appear also as alternatives to hard chromium plating [27] in spite of the realization of the parts in a unit way, of the limitations in minimum dimensions of the parts to be coated, and of the limitations in the minimum internal diameter of the cylinder that can be coated (i.e., $\Phi_{\min}=40–80 \text{ mm}$) [28]. Faced with this combination of difficulties and limits found in these processes, both environment-friendly and more flexible coatings implementing thermal spraying process have been used for a few years, providing a better productability. It should be noted that the sales turnovers relating to thermal spraying process know a remarkable increase, [29 and 30]. The emergence and the development of the high-velocity oxy-fuel (HVOF) thermal spraying process over the last decade has enabled the production of very dense metallic and cermets coatings, offering an effective substitute for hard chromium plating for certain jet engine components, among other components. [31, 32, 33, 34 and 35]. With HVOF systems, the feedstock material is heated to near or above its melting point by a high-velocity combustion gas stream (resulting from continuous combustion of fuels including propane, propylene, methane or hydrogen) and deposited [34, 35].

4. Conclusion

The first results obtained with deposits NiCrBSiC (Fe) define a set of properties that make it a potential alternative to hard chromium with an interesting range of properties:

- A high hardness of the deposit after cyclic heat treatment
- An excellent resistance to wear and friction
- A good corrosion resistance
- An excellent adhesion to steel

However, even if some chemical results are very promising, it remains necessary to validate other couples materials/processes in different conditions in the areas of corrosion. Moreover, it remains necessary to conduct
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