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Wear Behavior of a Ni/Co Bilayer Coating by Physical Vapor Deposition on AISI 1045 Steel

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ABSTRACT

Coatings by physical vapor deposition (PVD) have become highly relevant due to their wide range of applications and the rapid rate of coating formation. In this work, AISI steel 1045 was coated with two layers, Ni and Co using the PVD technique. Each coating was deposited with a thickness of 1 μm. After applying the coatings, a post-treatment was applied in an AC plasma reactor using a boron nitride blank in an Ar atmosphere at a pressure of 3 Torr, 0.3 A, and 460 V at 4, 8, and 12h. The post-treatment was characterized by optical emission spectroscopy (OES) in a range of 200-1100 nm. The main species observed by OES were Ar^+, N_2, N_2^+, and B^+. The coatings on 1045 steel and post-treatment were characterized by scanning electron microscopy (SEM) and X-ray diffraction (XRD). Also, were subjected to tribological tests to analyze wear resistance, using the Pin-on-Disk technique. The coatings on steel 1045 present remarkably better wear properties than the uncoated 1045 steel, being the sample post-treated at 4h that showed a lower wear rate.

1. Introduction

One of the steels most commonly steels used in machine components such as gears, bolts, crankshafts, etc., is AISI 1045 steel due to its good mechanical properties [1-3]. However, 1045 steel, is a medium strength steel, tends to wear and corrode easily and eventually, causes the failure of the part of steel. To try to extend the useful life of steel parts, different processes have been developed for the application of coatings and the modification of engineering materials surfaces. These processes have currently allowed the development and application of materials with quite interesting properties, due to the chemical reactions that take place on the surface, modifying the microstructure of the material and optimizing the properties of the surfaces, such as better wear resistance, corrosion, and some other properties. The coatings assisted by Plasma, have shown that they can improve the tribological and chemical properties of materials [4-8]. Being one of these processes assisted by plasma, the physical vapor deposition (PVD) by magnetron sputtering, by which multilayers of elements and/or compounds can be deposited over a matrix, increasing the wear resistance of the surface, compared against monolayers and their response [9-12].

Ni-Co alloys deposited and modified with boron (B) have great advantages, such as high hardness and wear resistance, so it is very possible to form very attractive and interesting multilayers. Now, the possible phases resulting from the integration of B, for example the interaction with Ni, is the NiB phase, is reported as useful to retain lubricants in conditions of adhesive wear, and cause a decrease in abrasive wear of the surface due to low coefficient of friction that this compound presents [13-16]. On the other hand, the compounds formed with Co, such as CoB, show a high resistance to corrosion between temperatures of 30 to 70 °C [19].

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In this work, the main purpose is to modify the Ni/Co coating deposited by PVD magnetron sputtering and later treated by Ar plasma, using a Boron Nitride (BN) as a target and carry out a detailed study of its wear behavior. The BN compound is reported to have the particularity of being structurally similar to diamond, which can be deposited over metallic layers, given the physical and chemical properties of BN, these properties can be: its high hardness, which is around the 4000 HV, low coefficients of friction, resistance to wear and corrosion. In addition, BN compounds have good resistance against oxidation and chemical stability [17, 18].

2. Experimental

In this work, a cold-rolled AISI 1045 steel bar with a diameter of 25.4 mm. The chemical composition of 1045 steel is shown in Table 1, which was obtained by spark spectroscopy. The steel bar was sectioned using conventional cutting processes. The dimensions of the specimens obtained were the following: diameter of 25.4 mm and 6.35 mm thickness. The specimens were subjected to a normalized heat treatment. The conditions of the heat treatment were carried out as follows: temperature of 860°C for a period of 30 minutes inside a muffle of resistors. The cooling process was carried out in air. With the heat treatment, the grain size of the AISI 1045 steel was refined and removes the residual stresses of cold rolling, which is favorable to the subsequent application of plasma treatments. Later, one of the faces was abraded with abrasive sands of silicon carbide of different grain sizes, from 120 to 600 emery paper. The face of the steel abraded was degreased with acetone and then cleaned with Nital 2 for a few seconds to remove oxides from the surface. Ni-Co coatings were applied in a magnetron sputtering chamber designed and built in in the PLASNAMAT laboratory, using Ni and Co targets. The conditions of the coatings were the following: current density of 0.3 A, pressure of 20 mTorr and gas flow of 20 sccm of Ar. The voltage applied to generate the Ni deposit was 395 V and 110 W DC, the deposit time was 50 min. For the Co coating, the voltage was 370 V and 89 W of power, applied for a time of 90 min. The Ni and Co coatings were made separately each one. In addition to the Ni-Co deposits, a plasma treatment of Ar was carried out, using a BN target, the plasma conditions were 0.3 A and 460 V at a pressure of 3 Torr, this treatment was applied on the samples, in three times, 4, 8 and 12 h. An optical fiber (solarization-resistant UV and fiber diameter of 400 μm) connected to a high-resolution spectrometer (Ocean Optics Inc., spectrometer model HR2000CG-UV-NIR) was used in the plasma monitoring process. The inlet and outlet slots were of 5 μm aperture. The process for data acquisition was performed with an integration time of 100 s. The optical fiber was placed in such a way that it could collect the light emitted by Ar plasma with BN target in a range of 200-1100 nm. The NiCoBN coatings, applied on the 1045 steel, were mechanically characterized by wear tests, carried out on a tribometer designed and built in the CICATA-IPN applying the Pin on Disc technique. The parameters used in the wear tests were the following: diameter of the steel ball was 3.17mm, load applied 10 N, distance traveled by the steel ball: 359.01 m at a speed of 100 rpm, the diameter of the track was 19.05 mm. In the wear tests, no lubricant was used, in order to accelerate the wear process on the samples. The thickness of the coatings was measured by 3D optical profiler and mechanical profiler. The coatings were also characterized by scanning electron microscopy (SEM) and X-ray diffraction (XRD), the phases of the coating were using a Bruker D2 PHASER diffractometer using Cu Kα radiation with $\lambda = 1.54184$Å. The sweep angle was between 30° and 100°.

3. Results and Discussion

3.1. Spark Spectroscopy

The chemical composition of the steel determined by spark spectroscopy, are presented in Table 1. In this table, are presented the main chemical elements in the steel alloy.

Table 1: Chemical composition (wt. %) of AISI steel 1045.

Element	Fe	C	P	S	Ni	Al	Co	Mn	Si
% present	97	0.534	0.0283	0.0079	0.0905	0.0272	0.0108	0.942	0.1770

3.2. Thickness Measurements by 3D Optical Profiler and Mechanical Profiler

The thickness of each coating was measured by the two techniques described above. The thicknesses measured by each technique did not show considerable variations. Below are the graphics of the thicknesses for each coating obtained by mechanical profiler (Figure 1). In this figure, we can observed that the profiler sweeps a length, subsequently it falls due to the difference in heights, the lower zone is the matrix and the upper one the coating, obtaining the thickness measurement of the coatings.

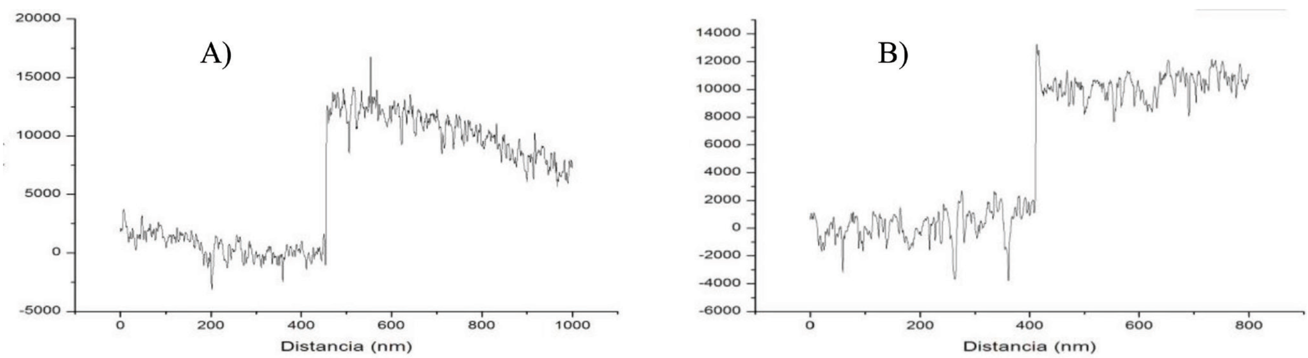

Figure 1: Graphics obtained by the mechanical profiler A) for Ni and b) for Co for the measurement of the thickness of the coatings.

The thickness of the coatings were also measured by 3D optical profiler, using a Zigo profiler, model Next view, with this profiler, thickness measurements can be obtained from a 3D image based on scanning interferometry of coherence. The Figure 2 shows an image obtained from one of the coatings thicknesses, in which the red-orange area is the coating and the green-blue area is the steel matrix. The average thicknesses measured by the two techniques are: Ni 1.17 ± 0.15 µm, Co 1.28 ± 0.19 µm and NiCoBN 2.41 ± 0.21 µm.

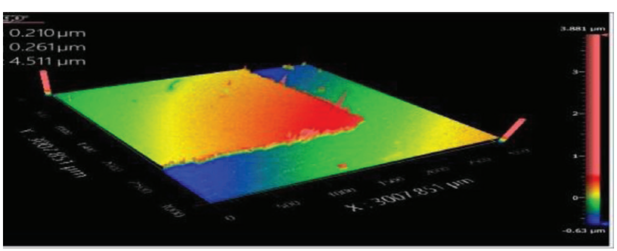

Figure 2: Measured by 3D Optical Surface Profiler of the coatings.

3.3. SEM and X-ray Diffraction

The first elemental characterization of the coatings consisted of a chemical analysis by SEM-EDX, corroborating through this technique that the deposits were applied successfully, these analyzes are presented in Figure 3 and it can observe the Ni and Co deposited on the surface of the steel.

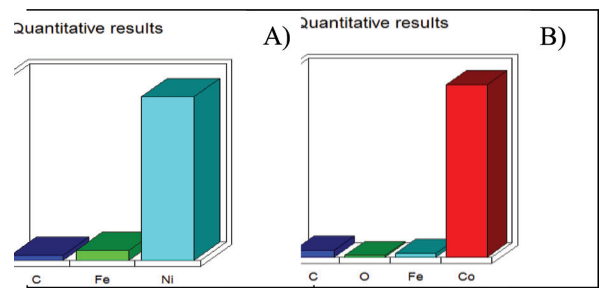

Figure 3: Chemical analysis by SEM a) For Ni coating and b) for Co coatings.

The phases identified by x-ray diffraction are shown in the diffraction pattern in Figure 4. In this pattern, BN coatings are compared at the different treatment times (4, 8 and 12 h) using BN target in the Ar plasma. The main phases found were NiCo, Co_4B, Ni_3B Co_3B and Co. We expected to form these phases which have been shown to have an influence on the behavior of the surfaces against wear. NiB phases help to reduce abrasive wear on the surface, this phase has the function to work as a kind of lubricant [13]. The CoB phases have the main function to protect the material against corrosion at medium temperatures, between 30 and 70 °C [20]. In this work, the behavior against corrosion is not analyzed. As a future work, we expected to analyze the coating in tribocorrosion tests; this is the main goal of to form the different phases of NiB and CoB and to have protection against the two mechanisms of material loss.

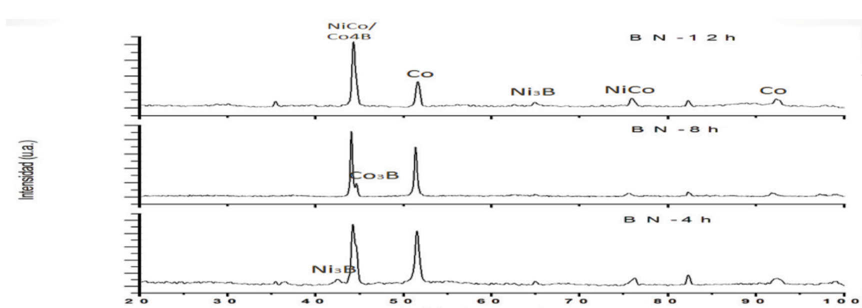

Figure 4: The main phases identified by X-ray diffraction were NiCo, Co_4B, Ni_3B Co_3B and Co.

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3.4.Wear Behavior and Wear Factor

The results for the dry friction tests of the coatings and 1045 steel are presented in Figure 5. These graphics show the behavior of the friction force and the friction coefficient for the samples with NiCoBN-4h, NiCoBN-8h, NiCoBN-12h coatings, and steel 1045 normalized during the wear test. In Figure 5 A) corresponding to 1045 steel, it can be seen in the graphic that the coefficient of friction of the steel shows a continuous behavior, because the material does not have any coating and it behaved similarly during the entire wear test. The samples coated and treated by plasma show a completely different behavior than the steel 1045. In the graphics B), C) and D) observed that the friction coefficient was varying during the test. Also, the presence of ridges and valleys are also observed, which indicate that one coating was finishing and passing to another coating, the first wasted layer was the BN layer, then the Ni layer and finally the Co. The small peaks that appear are attributed to the accumulation of dust between the surface of the material and the ball of the tribometer.

The values of the friction coefficients and friction force in the three coatings and the steel 1045. As we expected, the three coatings had slightly different friction coefficients than the 1045 steel, this different friction coefficients have influence in the wear behavior of each sample. The sample coated for 8 h with BN, presented a higher coefficient of friction than all the samples, while the sample coated for 12 h presented the lowest coefficient of friction, due to the nitrogen ions improve the surface roughness of the coating and reducing de coefficient of friction, but they do not improve wear behavior, this aspect is detailed below.

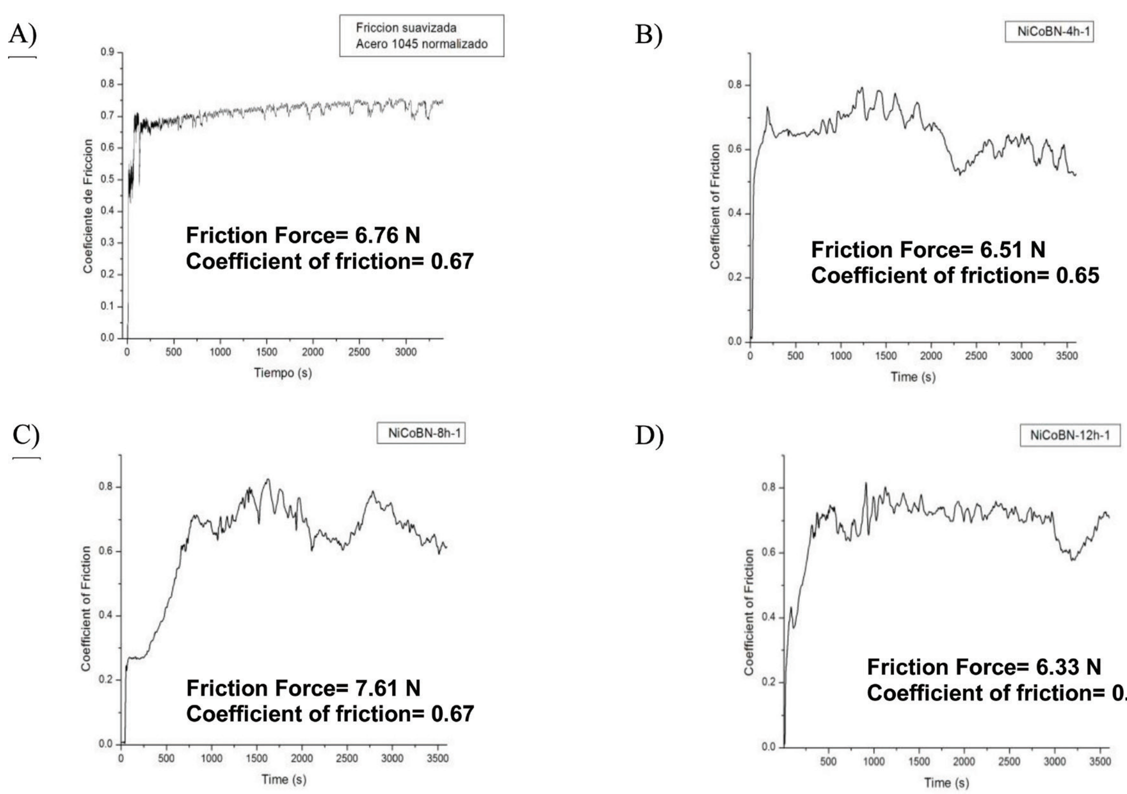

Figure 5: Friction coefficient in dry condition for A) 1045 steel normalized NiCoBN coatings B) 4h, C) 8 h and D) 12h.

3.5. Wear Factor

The volume loss was calculated measuring the area removed in the track left by the steel ball on the surface sample with a roughness tester (Mitutoyo, model SJ 410), these measurements were made in different areas of the surface on track and the average of the removed area from the coating by the steel ball was obtained during the wear test. The volume was calculated by applying equation 1.

$$V = A \times \pi \times D \tag{1}$$

Where:
V = volume removed.
A = area removed (measured with a roughness tester).
D = diameter of the track left by the steel ball on the surface sample.
The wear factor was calculated to analyze the behavior of each samples coated with NiCoBN using equation 2.

$$K = \frac{W}{FVT} \quad (2)$$

Where:

K = Wear factor.

W = Wear measured in volume removed.

F = Load applied.

V = Linear velocity between the sliding parts (steel ball and surface).

T = Operation time.

The calculated values of the wear factors are shown in Figure 6. In this graphic we can observed that the 1045 steel, as we expected, presented the highest wear factor, it means, the steel 1045 wears out more compared against steel coated with NiCoBN at different times. Steel with BN-4h coating exhibited the lowest wear rate compared to the other two coatings treated with BN target. Originally, it was expected that the coating treated with BN-12 h, would be the one with the least wear behavior, but the calculations of the wear factor, shown the opposite. The low resistance against wear is attributable to the high content of N in the coating, when nitrogen increases in the chemical composition of the steel, N favors the formation of the white layer, this layer is characterized for its high hardness, but also this layer is very easily detached from the material, causing that the dust released during the wear test to induce the abrasive wear mechanism by the particles with high hardness, producing the high wear rate in this specimen with the coating applied for a longer time [20] .

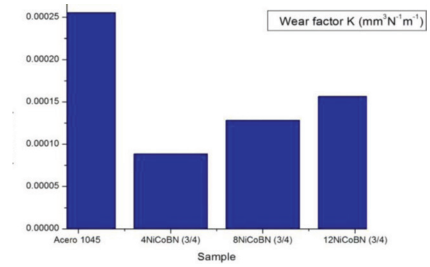

Figure 6: Wear factor of the steel 1045 and the coatings NiCo treated with BN at different times.

The following micrographs (Figure 7) present the track of wear left by the steel ball during the Pin on Disc test on the coated samples, analyzing this images, we can deduced more about the wear mechanism of each coating, highlighting that the mechanisms that operate, are mainly abrasive and adhesive wear on the surface. In micrograph B) it is observed dust residues which function as abrasive particles on the coated steel. These particles, according to the SEM analysis, are mainly iron oxide. In micrographs E) and F) of the BN-12h coating, it is observed that large portions of material were detached, which is representative of fatigue wear, in which it is observed that, during repeated sliding and trapping, it will eventually produce cracks and detachment of material, as we can observe in the micrographs, causing that the volume removed to be greater than in other coatings. In the 8 and 12 h coatings, according to the calculated wear factor, the wear was greater than in the 4 h coating, this behavior can be explained by the low adherence of the BN layer and the white layer to the NiCo coatings, this can be observed in the micrographs C), D), E) and F), the material removed in powder adhered to the steel ball, leaving traces on the steel.

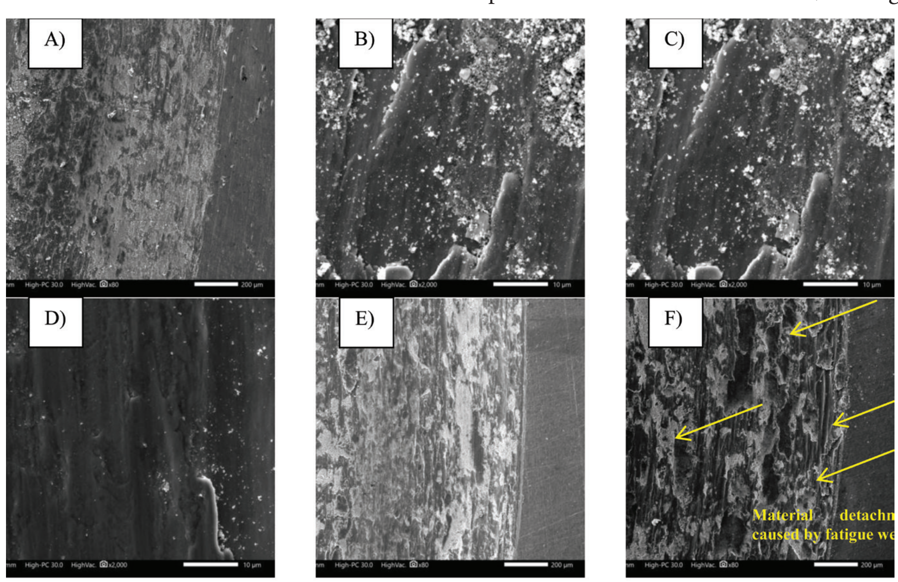

Figure 7: SEM micrographs of the wear track on the coatings A y B) NiCo BN-4h, C y D) NiCoBN-8h y E y F) NiCoBN-12h.

3.6. Optical Characterization by Optical Emission Spectroscopy (OES)

OES measurements for Ar plasma at a pressure of 3 Torr and an electrode gap of 6.35 mm were performed at a current of 0.3 A and 460 V. The emission spectra obtained under these conditions are presented in Figure 8. In these emission spectra, the main species identified were Ar^+, N_2, N_2^+, C^+ and B^+. Other species identified were Co_2 at 210 and 360 nm at the start of the discharge. For the plasma spectrum at 12 h, it is observed that the intensity of the peaks between 600 and 1000 nm decreases as the time of application of the coating increases from 0 h to 12 h. Doing a relationship between the tribological behavior and the plasma at 12 h, we can explain that quantity of N present in the plasma at 12 h, nitride the surface of the steel and the coating, which influenced the wear of the sample was higher than the other samples coated.

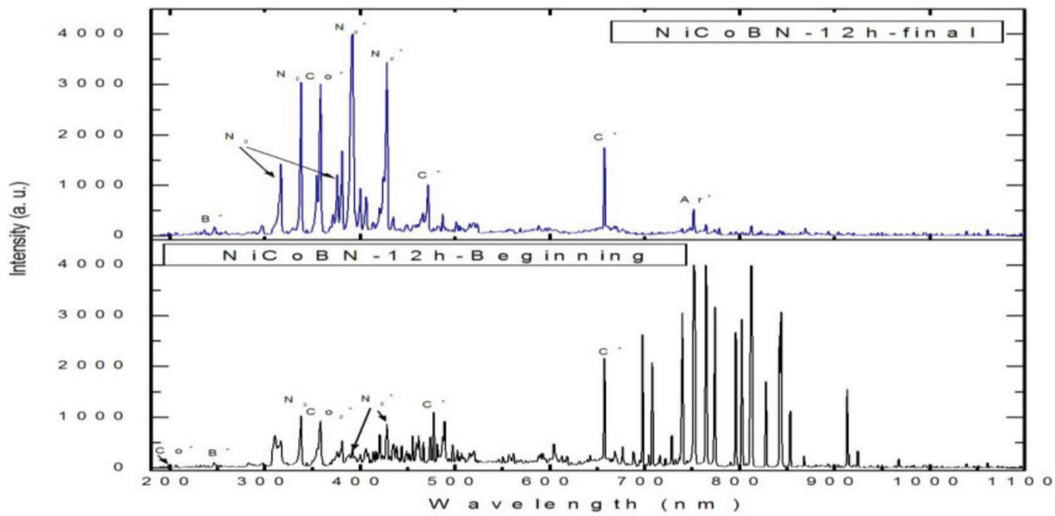

Figure 8: The OES of Ar-BN plasma at 3 Torr at the beginning and at the end of the treatment.

Conclusions

In this work, different treatment times with a BN target were applied to AISI steel 1045 coated with NiCo-coated by magnetron sputtering, in order to evaluate the tribological behavior using the Pin on Disc technique under dry conditions. The plasma treatment to modify the NiCo coatings was carried out with Ar plasma with a BN target. The results obtained are summarized below:

The thickness of the NiCo coatings was 2.41 μm +0.21 μm obtained by PVD magnetron sputtering assisted by Ar plasma with DC. Subsequently, treatments with Ar plasma with AC and a BN target were applied at three times, 4, 8 and 12h.

The surface characterization by X-ray diffraction, the following compounds were identified: NiCo, Co_4B, Ni_3B Co_3B and Co.

According to the results of tribological tests, NiCoBN coatings treated with Ar-BN plasma were more effective against wear than steel 1045 normalized.

The calculations of the wear factor for the BN coatings, showed values lower than that of the steel 1045. Steel 1045, showed a wear factor of $2.55\times10^{-13}\,mm^3N^{-1}m^{-1}$, while NiCoBN treated during 4h, its wear factor was 9.14 $\times10^{-14}\,mm^3N^{-1}m^{-1}$, these data indicate that the coatings modified with BN, reduce the wear process by an order of magnitude, compared with steel 1045.

As future work, the wear of the coatings will be evaluated with lubricant, we expected to significantly improve the performance of the surfaces against wear. Tribocorrosion tests are also expected, NiB and CoB compounds have been applied mainly to protect the substrate against corrosion.

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References

[1] R. Charadia, M. Regis, E.L. Dalibon and S.P. Brühl, CONAMET/SAM (2015). http://ria.utn.edu.ar/bitstream/handle/123456789/889/GIS63.pdf?sequence=1&isAllowed=y

[2] R. Günzel, M. Betzl, I. Alphonsa, B. Ganguly, P.I. John and S. Mukherjee, Surface and Coatings Technology **112**, 307 (1999). https://doi.org/10.1016/S0257-8972(98)00776-2

[3] T. Hirsch, T.G.R. Clarke and A. da S. Rocha, Surface and Coatings Technology **201**, 6380 (2007). https://doi.org/10.1016/j.surfcoat.2006.12.005

[4] F. Mahboubi & M. Fattah, Vacuum **79**, 1 (2005). https://doi.org/10.1016/j.vacuum.2005.01.002

[5] F. Hakami, M.H. Sohi and J.R. Ghani, Thin Solid Films **519**, 6792 (2011). https://doi.org/10.1016/j.tsf.2011.04.054

[6] S. Kikuchi, T. Fukuoka, T. Sasaki, J. Komotori, K. Fukazawa, Y. Misaka and K. Kawasaki, Materials Transactions **54**, 344 (2013). https://doi.org/10.2320/matertrans.MBW201205

[7] I. Rosales, H. Martinez and R. Guardian Applied Surface Science **371**, 576 (2016). http://dx.doi.org/10.1016/j.apsusc.2016.03.048

[8] García Lorente J.A. and Rodríguez Trias R. Recubrimientos tribológicos por PVD. Ingeniería Química, ISSN 0210-2064, N° 418, p. 107-110 (2004).

[9] K. Chu and Y.G. Shen, Surf Eng **24**, 402 (2008) http://dx.doi.org/10.1179/174329408X282541

[10] E. Martínez, J. Romero, A. Lousa and J. Esteve, Surface and Coatings Technology **163–164**, 571 (2003). https://doi.org/10.1016/S0257-8972(02)00664-3

[11] M. Berger, U. Wiklund, M. Eriksson, H. Engqvist and S. Jacobson, Surface and Coatings Technology **116–119**, 1138 (1999). https://doi.org/10.1016/S0257-8972(99)00151-6

[12] F. Ge, X. Zhou, F. Meng, Q. Xue and F. Huang, Tribology International **99**, 140 (2016). https://doi.org/10.1016/j.triboint.2015.10.019

[13] K. Krishnaveni, T.S.N. Sankara Narayanana, S.K. Seshadri, Surface and Coatings Technology **190**, 115 (2005). https://doi.org/10.1016/j.triboint.2015.10.019

[14] G.G. Gawrilov, *Chemical (Electroless) Nickel Plating*, Portcullis Press Ltd., Surrey (1979).

[15] G.O. Mallory, J.B. Hadju (Eds.), *Electroless Plating: Fundamentals and Applications*, AESF, Orlando (1991).

[16] W. Riedel, *Electroless Plating*. ASM International, Ohio (1991).

[17] M. Keddam, M. Kulka, N. Makuch, A. Pertek and L. Małdzinski, Applied Surface Science **298**, 155 (2014). https://doi.org/10.1016/j.apsusc.2014.01.151

[18] U. Fantz, Plasma Sources Science and Technololgy **15**, S137 (2006). http://dx.doi.org/10.1088/0963-0252/15/4/S01

[19] I. Hacisalihoglua, F. Yildiz and A. Alsaran, Wear **384-385**, 159 (2017). https://doi.org/10.1016/j.wear.2017.01.117

[20] Z. Sukackienė, L. Tamašauskaitė-Tamašiūnaitė, V. Jasulaitienė, A. Balčiūnaitė, A. Naujokaitis and E. Norkus, Thin Solid Films **636**, 425 (2017). https://doi.org/10.1016/j.tsf.2017.06.034

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