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Film Deposition by Duplex Treatment with Hastelloy Cage on AISI 6160 Steel

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AISI 6160 steel is used to manufacture cutting blades and springs due to its high tensile strength and good ductility. However, it has low mechanical strength and low wear resistance. In this work, a duplex treatment consisting of CCPD followed by PN was performed to improve the surface properties of the steel. In addition, XRD, optical microscopy, microhardness, and sphere-disk tribological analysis were used to verify the surface changes of the samples. The results showed a significant improvement in surface hardness and better wear resistance of AISI 6160 Steel submitted to Duplex treatment. The sample submitted to the CCPD treatment for 4 hours and, after nitriding at 500 °C for 2 hours, presented higher surface hardness and wear resistance than the sample only subjected to plasma deposition, characterizing the efficiency of the use of the duplex treatment adopted in the present work.

Keywords: *AIS I 6160, duplex treatment, hardness, wear resistance.*

1. Introduction

AISI 6160 steel is used in automotive systems, cutting blades for agricultural machinery applications[1], knives, andsprings[2] dueto its high resistance to traction and fatigue, with good ductility. However, despite having a carbon content similar to AISI 5160 steel, AISI 6160 steel contains a little more chromium and vanadium in its chemical composition. Therefore, despite the good properties of this material, it has low mechanical strengt hand wear resistance[3-5]. These disadvantages make the applications of theses teels limited in several industrial sectors.

Ionic nitriding can significantly modify the composition of the surface layers by implanting nitrogen in the structure of the metallic material[6,7]. However, plasma modification techniques have been widely used to improve the surface properties of materials and consequently increase their applicability with lower energy expenditure. Plasma nitriding (PN) produces a hardness gradient between the substrate and the coating minimizing coating/substrate pair mismatch[8-10]. The cathodic cage plasma deposition (CCPD) technique, on the other hand, is capable of producing homogeneous films on the surface of the samples through the sputtering phenomenon that occurs in the holes of the cathodic Cage[11,12]. Lately, a combination of two different treatments has been adopted to further improve the surface properties of metal parts. Duplex treatment is commonly found to be conventional nitriding (PN) followed by cathodic cage plasma nitriding (CCPN)[13], or magnetron sputtering deposition[11,14]. Araújo et al.[15], used PN post-treatment after CCPN with 5160 steel Hastelloy cage to obtain thick diffusion layers and wear-resistant chromium-nickel nitride films[15,16].

In this work, the duplex treatment composed of CCPD followed by a PN post-treatment was adopted to increase the diffusion layer and the presence of chromium nitrides and other phases from the Hastelloy steel. Surface hardening with a thick layer of nitriding and deposition are characteristics that result in increased wear resistance of the base material. Therefore, the methodology adopted in the present study aims to present a case study to promote discussion on improving the surface properties of AISI 6160 steel.

2. Materials and Methods

In this work, 4 cylindrical samples of AISI 6160 steel with elemental composition of (% by weight): 0.45 C; 0.88 Cr; 0.77 Mn; 0.005 P; 0.17 Si; 0.002 S; 0.03 Ni; 0.14 V; and iron (balance). The samples were cut 8 mm thick and 50 mm in diameter. The metallographic preparation of the samples followed the methodology adopted by Libório et al.[14].

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The deposition was performed by CCPD with a Hastelloy C-276 alloy cage with the following composition (wt.%): 47.30% Ni, 22.00% Cr, 9.00% Mo, 1.50% Co, 18.50% Fe, 0.50% Mn, 0.60% W and 0.10% C. In this process, a working pressure of 2 mbar, treatment time of 4 h, and temperature of 450 °C were used. Subsequently, two of these samples were subjected to plasma nitriding, one at 450 °C and the other at 500 °C, as shown in Table 1. A pre-sputtering step at 350 °C preceded both treatments with na atmosphere gás of 50% H_2 and 50% Argon for 30 minutes. The equipment used in this work to perform plasma deposition and nitriding is described in previous works[17-19], and the plasma processing scheme are shown in Figure 1.

After the plasma surface treatments, the crystalline phases were identified with a Shimadzu X-ray diffractometer DRX-6000 (XRD), operating at 40 kV, and with Cu-Kα radiation (λ = 1.55418 Å). The Vickers microhardness test was performed with a Shimadzu microhardness tester model HMV 2000. A load of 25 gf was used in each test, and 4 measurements were made to obtain the mean value. In addition, a TOPCON optical microscope was also used to measure the thickness of the layers produced on the surface of samples subjected to a chemical attack with 3% Nital to produce contrast and reveal the microstructural phases of the materials. The material's wear resistance was evaluated as a function of the worn volume[20]. The parameters used to carry out the tests on the samples were 8N of load and 38.5 Hz of Ball rotation, in which caps were performed under caps at times of 2, 5, 10, 15, 20, 25, and 30 minutes. In addition, the wear volume was calculated according to the method presented in more detail yby Araújo et al.[15]. Three tests were performed on each sample to obtain the measures' average and respective deviations.

3. Results

Figure 2 shows the X-ray diffraction pattern of the samples studied. The diffractogram presents the result of the deposition of films composed of iron nitride (Fe_3N and Fe_4N), chromium nitride, and FeNi. These phases are the result of the removal of the constituent elements from the cathodic Cage and the recombination of these more abundant elements with the nitrogen present in the gaseous atmosphere. Due to the abundance of iron in the composition of the Cage and its ease of combination with nitrogen from the gaseous atmosphere, a greater formation of iron nitride phases is observed due to the high intensity of the Fe_3N peaks at positions 38,34°, 41, 07° and Fe_4N at positions 41.16°, 47.91° and 70.06°. These nitride phases increase the steel's surface hardness and reduce wear through sliding contact[15,21]. The chromium nitride and FeNi phases Consist of agglomerates that originated from the spraying process in the Hastelloy Cage during the CCPD treatment, both important for surface oxidation-reduction. According to Naeem et al.[18], post-nitriding increases the nitrogen diffusion rate in the sample, but the surface phases do not under go significant changes except for their intensities[18]. In this case, the increase in the post-nitriding temperature (from 450 to 500 °C) produces na increase in the Fe_4N phase and a reduction in the Fe_3N phase. That is characterized by the disappearance of the peak at the 38.34° position.

The optical microscopy of each treated sample is shown in Figure 3. All samples contain a layer of diffusion and deposition on the surface. It is observed that the sample only submitted to deposition by CCPD (Dep450) also presented a diffusion layer because of the nitrogen-rich atmosphere (75%) and facilitating temperature for the penetration and combination of N_2 with the iron present in the steel matrix.

Table 1. Sample nomenclature and treatment parameters CCPD and PN.

Samples	CCPD	PN			
		Pressure	Time	Gas atmosphere	Temperature
Base	2 mbar, 4 h, 75%N_2+25%H_2, 450 °C.	-	-	-	-
Dep450		-	-	-	-
DepNit450		1.8 mbar	2h	25%N_2+75%H_2	450 °C
DepNit500		1.8 mbar	2h	25%N_2+75%H_2	500 °C

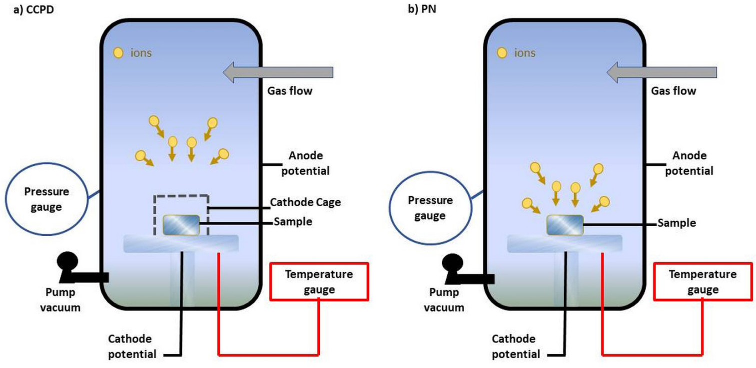

Figure 1. Scheme of plasma treatment processes: (a) CCPD, and (b) PN.

His fact is justified by the peak displacement of the iron phase in the diffractograms shown in Figure 2[17,22]. The displacement of the peak is evidence of the migration of nitrogen atoms into the interior of the steel structure as a solid solution, causing the emergence of uniform stresses characteristic of the $Fe_{(N)}$ phase[11,23]. This effect is barely perceptible in the diffractograms due to the low treatment temperatures adopted in this study. However, small detachments are still noticeable.

Post-nitriding at 450 °C performed on the DepNit450 sample showed na increase in the deposited layer of 32.7%, and the increase in the penetration depth of nitrogen in the inner most layer caused na increase in the diffusion layer of 14%. However, post-nitriding at 500 °C did not show a significantly higher deposition layer than that acquired at 450 °C.

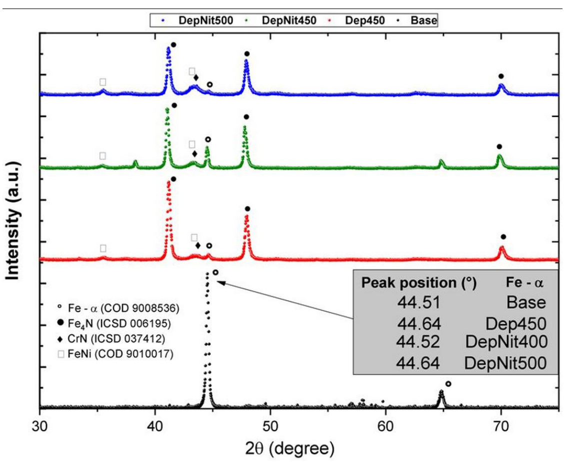

Figure 2. Diffractograms of samples submitted to duplex treatment (CCPD + post-nitriding).

On the contrary, the film of the DepNit500 sample deposited by CCPD and later nitrided showed na increase of 37.5% about the Dep450 sample. On the other hand, the DepNit500 diffusion layer obtained a depth lower than that acquired by the DepNit450 sample. This can becaused by the nitriding removal process explained by the theory of diffusion in the non-steady state (second Fick'slaw), which justifies thein efficiency of increasing the temperature to obtain thicker layers in ferrous alloys[24-26].

The Vickers microhardness tests howed increased surface mechanical strength for all plasma-treated samples. Figure 4 shows that plasma deposition with a cathodic cage produced a surface layer with a hardness of approximately 1200 Hv, representing an increase of approximately 230%. This expressive increase results from the deposition layer observed in Figure 3. In addition, the samples submitted to post-nitriding had na increase in surface hardness about the Dep450 sample. Post-nitriding at 450 °C contributed to a 42% increase in hardness compared to the Dep450 sample and 56% for the post-nitrided sample at 500 °C. These results agree with the observations made by optical microscopy, and the slight increase in hardness of the DepNit500 sample about the DepNit450 sample is justified by the reduction of the diffusion layer discussed above.

Figure 5 shows the hardness results performed in the cross-section of the samples with indentations made from the outermost part to the center of the pieces (bulk region). All samples have a hardness reduction along the depth due to phase changes caused by surface treatments. The Dep450 sample, as it has a thinner deposited film and a thin diffusion layer, showed an intense hardness reduction until it converged to the value of 380 HV, referring to the Base sample at a depth of approximately 100 µm.

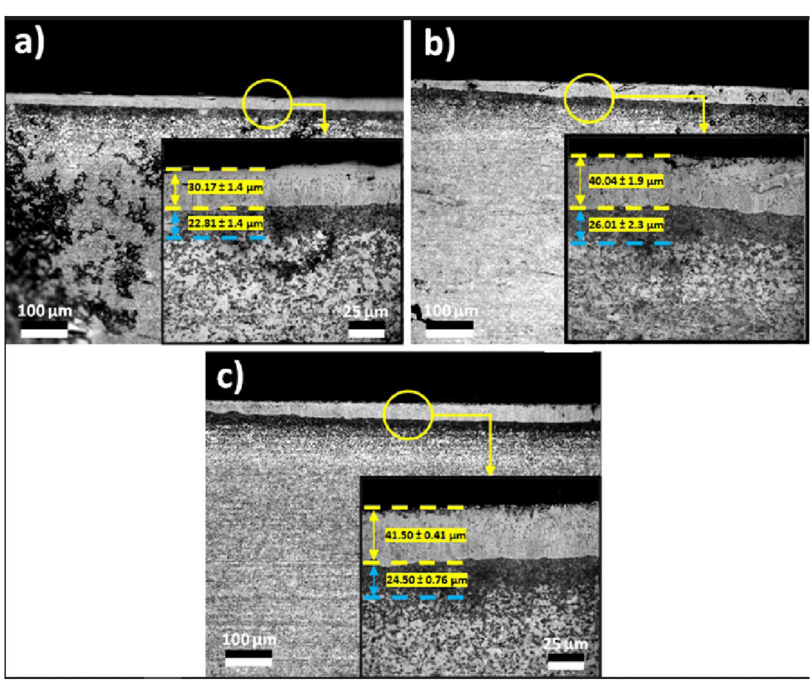

Figure 3. Optical microscopy of plasma-treated samples: (a) Dep450, (b) DepNit450, and (c) DepNit500.

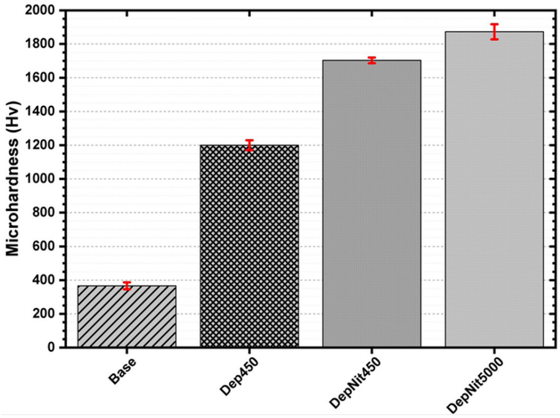

Figure 4. Surface hardness of Base samples and samples subjected to plasma treatment (CCPD and duplex treatment).

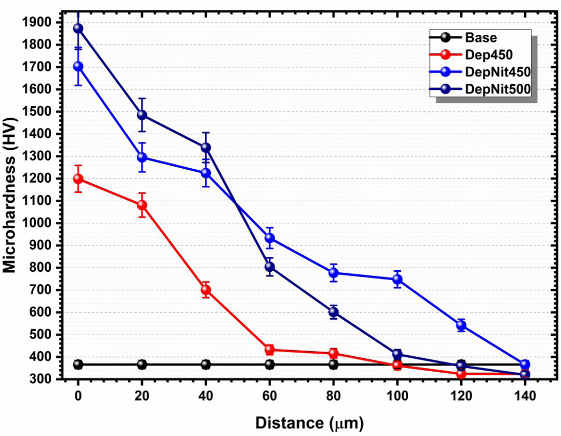

Figure 5. Profile hardness with measurements taken from the surface towards the center of the sample.

The DepNit500 sample showed superior hardness in theinner regions and a smoother decay than Dep450, which resulted in hardness convergence to 380 HV at 120 µm. On the other hand, the DepNit450 sample, with greater thickness of the deposited and diffusion layer, showed less loss of mechanical strength as a function of the transverse depth of indentation, as shown in Figure 5.

Figure 6 shows the wear volume results obtained for the base material and material streated with Hastelloy deposition at 450 °C at 4 hours and Hastelloy deposition at 450 °C at 4 hours plus conventional nitriding at 450 °C and Hastelloy plus Nitriding conventional at 500 °C (both nitriding at 2 hours of treatment). With this, it is possible to verify that, in comparison with the Base sample, all the treatments performed contributed significantly to the increase in the material's wear resistance. The base material obtained a wear volume value of 4.3 x 10^{-1} mm^3 in 30 minutes of testing.

The DepNit500 sample obtained a lower wear volume in the sphere-disk test, that is, a higher wear resistance. The values presented in Figure 6b show little variation over time, resulting in a value of 3.64 x 10^{-4} mm^3 in the final 30-minute period.

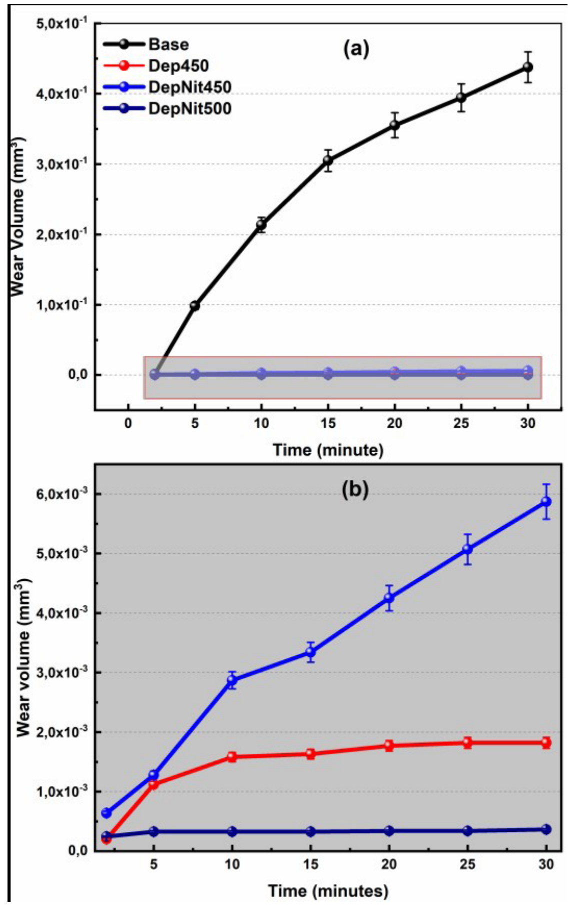

Figure 6. Wear of the samples submitted to the sphere-disk test: (a) Comparison between the Base sample and the treated samples; (b) enlarged result in the gray region.

On the other hand, the highest wear volume among the treated samples was observed in DepNit450 with a value equal to 5.8 x 10^{-3} mm^3. The deposition treatment with Hastelloy at 450 °C (4hrs) resulted in intermediate values about the other surface treatments. After 10 minutes of testing, a wear volume is observed with Constant values for the Dep450 sample, reaching a value equal to 1.8 x 10^{-3} mm^3. The uniformity and hardness of the composite layer formed in the plasma treatment contribute to the increased wear resistance of the part. However, due to the fragility of the layer, its peeling can occur and, consequently, the formation of abrasive particles at the contact interface of the tribological couple, causing na increase in the volume worn as observed in the DepNit450 sample.

4. Conclusion

The duplex treatment adopted in this study with plasma deposition with a Hastelloy cathodic Cage and post-treatment of conventional plasma nitriding on AISI 6160 steel presented surface modifications capable of improving the hardness and making the studied steel applicable in situations that require wear resistance. Among the results obtained in this work, it can be concluded that:

- The methodology adopted for the production of surface modification by duplex treatment (CCPD+PN) proved viable for forming iron and chromium nitrides and increasing the diffusion layer due to the greater insertion of nitrogen in the internal regions of the material.
- Optical microscopy results showed that PN post-treatmentat 450 °C increases the diffusion and deposition layer. However, at 500 °C, the diffusion layer is reduced, characterized by the depth of the nitrogen reached inside the sample.
- The Vickers microhardness tests howed a significant increase in surface hardness in samples submitted to duplex treatment. The hardness profile of the DepNit450 sample showed convergence to the base material with greater depth. That represents a surface hardening gradient favorable to the durability of parts treated in this treatment condition.
- The result of the sphere-disk test showed that all samples submitted to plasma treatment showed a reduction in surface wear. The DepNit500 sample showed higher wear resistance due to the high surface hardness combined with the higher layer thickness produced by the duplex treatment.

Therefore, the method applied in this study with PN post-treatment showed favorable surface hardness results for more severe AISI 6160 steel applications. In addition, the DepNit450 sample, despite having a slightly lower surface hardness than DepNit500, showed a smoother hardness decay which may result in greater wear resistance for longer tribological tests. However, the results showed that the sample submitted to the Duplex treatment composed of CCPD with Hastelloy at 450 °C (4h) followed by PN at 500 °C (2h) is efficient for significantly increasing hardness and reducing surface wear of AISI 6160 steel.

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