WEAR RESISTANCE OF SAE 5160 STEEL DEPOSITED BY DUPLEX SIMULTANEOUS WITH HASTELLOY CATHODIC CAGE

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ABSTRACT

In this work, the cathodic cage plasma deposition (CCPD) technique was applied to SAE 5160 steel, using Hastelloy cathodic cage (nickel alloy) with the aim of increasing the surface hardness and wear resistance of this steel. The deposition treatments were carried out with the samples at cathodic potential. In this work, two different atmosphere conditions were studied (75%H_2/25%N_2 and 25%H_2/75%N_2), at a temperature of 450°C during 4 hours. The coatings formed were analyzed using X-ray diffraction, optical microscopy, Vickers microhardness and microabrasive wear tests. Both treatment conditions favored the formation of coatings with high microhardness and excellent adhesion to the substrate. The treatment carried out in the most hydrogen-rich atmosphere (75%H_2/25%N_2) showed greater layer thickness and superior wear resistance, with a reduction of around 65% in the worn volume.

KEYWORDS: *Cathodic Cage; Hastelloy; SAE 5160 Steel; Wear Resistance.*

I. INTRODUCTION

Surface treatments are widely used on carbon and low-alloy steels to increase surface hardness and wear resistance. In the literature there are several successful techniques for forming coatings with good wear resistance, such as plasma nitriding [1], [2], [3], [4], thermo-reactive deposition process (TRD) [5], [6], magnetron sputtering [7], carburizing [8].

Castillejo et. al [5] applied the TRD process to AISI D2 steel to evaluate the formation of chromium and niobium carbide coatings. The process was carried out at a temperature of 1020°C for 4 hours, varying the composition of the chemical bath by changing the amount (in weight) of ferroalloy added (S1 = 16% Fe-Nb, S2 = 30% Fe-Cr and S3 = 8% Fe-Nb + 15% Fe-Cr). All the treated samples showed a reduction in the coefficient of friction and an increase in corrosion resistance, especially sample S3, whose coating showed greater microhardness and corrosion resistance, mainly associated with the composition (NbC, Cr_7C_3 e $Cr_{23}C_6$), and the smaller crystallite size.

H.E.L. Ramos, A.R. Franco Jr., E.A. Vieira [1] studied the effect of plasma nitriding on API 5L X70 microalloyed steel under different treatment pressures (2.5, 3.9 and 4.6 Torr) at temperatures of 410°C and 440°C. The increase in treatment pressure favored the formation of the ε-$Fe_{2-3}N$ phase, which was not observed at a pressure of 2.5 Torr. It was observed that at 410°C the increase in treatment pressure led to an increase in wear resistance, while at 440°C, although the treatment at 4.6 Torr pressure showed the lowest wear rate, the behavior was not linear, with the treatment at 3.9 Torr pressure showing the worst result among the samples treated at 440°C. The worst performance of this sample is associated with the thick white layer formed. The authors observed that for greater thicknesses of nitrided layer, but with a low thickness of white layer, better wear resistance was obtained.

In this work, the use of cathodic cage plasma deposition (CCPD) was proposed as a method to increase the surface hardness and wear resistance of SAE 5160 steel. This PVD (Physical Vapor Deposition) method allows the deposition of a wide variety of films and coatings, such as transition metal oxides and nitrides [9], [10], [11], [12], [13]. The technique can be applied to a variety of substrates, but its use on steels is the most common in the literature, and papers can be found on the use of the technique on carbon steels [12], low alloy steels [14], [15], stainless steels [13], [16] and high-speed steels [17]. The treatment setup used in the work was deposition with the sample at cathodic potential, also known as duplex simultaneous [18], which enables a layer with high hardness and good wear resistance to be obtained [14], [15], However, the literature lacks studies pointing out the treatment atmosphere that optimizes the performance of the coatings for this treatment configuration.

Medeiros Filho et. al (2024) [19] studied the deposition of coatings based on Ni-Cr-Mo on SAE 6160 steel using a cathode cage made from 904L stainless steel. The coatings were deposited at 450°C for 4 hours. The authors evaluated the effect of plasma nitriding on SAE 6160 steel previously deposited using the CCPD technique. The plasma nitriding treatments were carried out at temperatures of 450°C and 500°C for 2h and 4h. The 2-hour nitriding treatments showed greater resistance to wear, these results being attributed to the large layers of compounds and diffusion zones formed in these treatments.

In this work, the CCPD technique was applied to SAE 5160 low alloy steel to evaluate the effect of the treatment atmosphere on surface hardness and wear resistance. The treatments were performed with a cage (screen) made of Hastelloy nickel alloy, and with the samples at cathodic potential, i.e. without the use of an electrical insulator.

II. MATERIALS AND METHODS

The SAE 5160 steel samples used in this work were commercially obtained in the form of a 19.05 mm thick round bar (3/4"), with a chemical composition (%wt.) of 0.56 - 0.64% C, 1.35 - 1.65% Mn, 0.70 - 0.90% Cr, and Fe in balance. Samples with a thickness of 6 mm were extracted from the bar. The samples were cut with a water-cooled cut-off machine, sanded with SiC sandpaper to a grain size of 1200 mesh, and polished with alumina suspension (0.3 μm).

For the treatments, a cage made from the nickel alloy Hastelloy C-276 was used, with the following composition (wt.%): 47.80% Ni, 22.00% Cr, 9.00% Mo, 1.50% Co, 18.50% Fe, 0.50% Mn, 0.60% W and 0.10% C. The samples were positioned inside the cage and in direct contact with the sample holder (cathode), thus assuming its potential. The treatments carried out consisted of pre-sputtering in an inert atmosphere (50% Ar + 50% H_2 with a total flow of 90 sccm) at a temperature of 350°C for 30 minutes, the purpose of this stage was to clean and activate the surface [20]. The treatments were then conducted at a temperature of 450°C for 4 hours with a gas flow of 60 sccm, with 25%N_2 and 75%H_2 for the H25N sample and 75%N_2 and 25%H_2 for the H75N sample. The reactor used in the treatments is described in previous works [20], [21], [22].

For the characterization of the samples, Vickers microhardness tests, optical microscopy, adhesion tests, X-ray diffraction (XRD), and sliding wear test were performed. The Vickers microhardness test was carried out using an INSIZE ISH-TDV 1000 microhardness tester, with a load of 50 gf applied for 15 seconds. The average of five indentations was used to determine the surface microhardness and the microhardness profile.

Micrographs were captured using a BEL Photonics MTM-1 metallographic microscope. The cross-section of the samples was cold-embedded in acrylic resin, ground to 1200 mesh sandpaper, polished with alumina (0.3μm), and then etched with 3% nital for 10 seconds [23]. The adhesion test was conducted in accordance with the VDI 3198 standard, using an INSIZE ISH-BRV durometer to make an indentation (Rockwell-C) on the surface of each sample, followed by analysis with an optical microscope [24]. XRD analysis was performed using a SHIMADZU XRD-6000 diffractometer with Cu-Kα radiation, over a scanning range (2θ) of 20° to 100° at a speed of 1°/min, operating at 40kV and 30mA.

The fixed sphere sliding wear tests were performed using a SAE 52100 steel ball with a radius of 12.7 mm. The parameters used included a normal load of 4 N and a rotation frequency of 40 Hz for durations of 300, 600, 900, 1200, 1500 and 1800 seconds. The tests were performed without any type of abrasive liquid or lubricant. The equipment utilized for the wear test is described in the literature [22]. In this type of test, the worn surface forms a crater-shaped wear track. The crater produced in the wear test were measured using a Leica DMI8 optical microscope. The wear volume (V) was calculated according to Equation 1, where R corresponds to the radius of the ball and b is the diameter of the wear track [25].

$$V = \pi \frac{b^4}{64R^2}\left(R - \frac{b^2}{8R}\right) \approx \frac{\pi b^4}{64R} \qquad \text{for } b <<< R \qquad \text{Equation (1)}$$

III. RESULTS AND DISCUSSIONS

The diffractograms of the treated samples (H25N and H75N) are shown in Figure 1. According to the X-ray pattern of the H25N sample, the formation of three phases is observed: the nitrides CrN (ICSD 041827) [26] and Fe_4N (ICSD 060195) [27], and the intermetallic phase $Cr_{0.4}Ni_{0.6}$ (ICSD 102821) [28]. However, in the H75N sample, only the iron nitrides Fe_3N (ICSD 079981) [29] and Fe_4N (ICSD 060195) [27] were identified. Examining the composition of the coatings formed under both treatment conditions, it can be observed that using an atmosphere richer in hydrogen (75% H_2 - 25% N_2) promotes the formation of a greater number of phases containing elements from the cage (Cr and Ni), highlighting the important role of hydrogen in removing atoms from the cage (target).

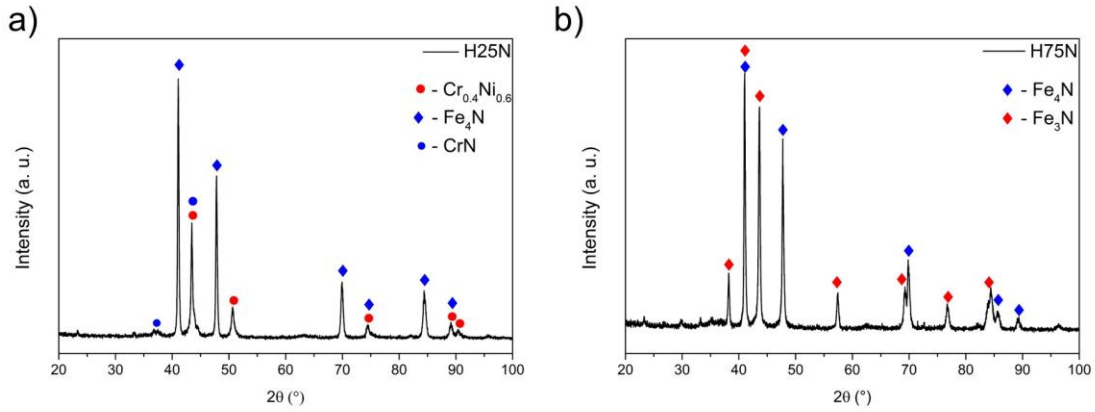

Figure 1 - XRD Patterns of the samples (a) H25N and (b) H75N.

Figure 2 shows the surface microhardness of the untreated and treated samples. The untreated SAE 5160 steel exhibits a surface hardness of 402 ± 13 $HV_{0.050}$, while the H25N and H75N treated samples demonstrate an increase in hardness of 138% and 173%, respectively. This increase in the microhardness of the treated samples is attributed to the formation of hard phases such as Fe_3N, Fe_4N and CrN [2], [30]. The microhardness values obtained align with similar works in the literature [15], [31]. Medeiros Filho et al. (2023) [15] reported a surface hardness of approximately 1200 $HV_{0.025}$ by applying simultaneous duplex treatment to AISI 6160 steel using a Hastelloy cage.

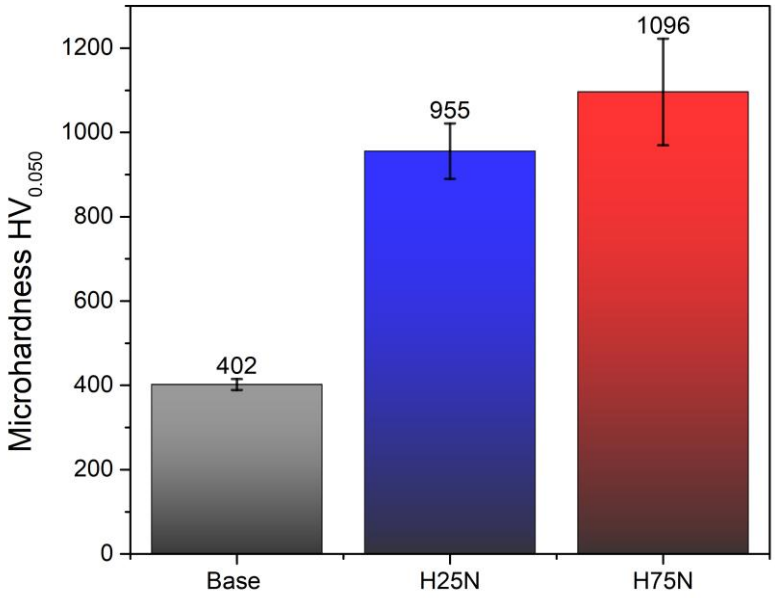

Figure 2 - Microhardness of the Base, H25N and H75N samples.

Figure 3 shows the microhardness profile of the treated samples. It can be observed that H25N sample, treated with a lower percentage of nitrogen, exhibits a smoother hardness gradient [15], while sample H75N shows an abrupt hardness gradient [31].

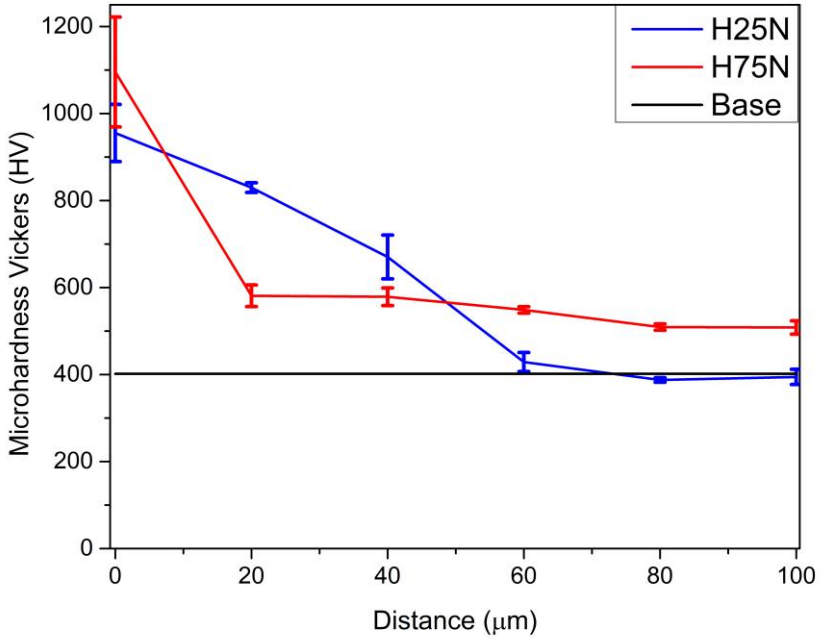

Figure 3 - Microhardness profile of the treated samples.

In the micrographs of the cross-section of the treated samples (Figure 4), it can be observed that the sample with the lower proportion of nitrogen (H25N) exhibited a greater layer thickness of 47.66 ± 2.48 μm, consisting of two distinct regions: a compound layer with a thickness of 24.81 ± 0.75 μm, followed by a diffusion zone of 21.83 ± 2.47 μm. In contrast the sample H75N presented only the compound layer with a thickness of 12.99 ± 1.34 μm, without the presence of a diffusion zone. The microhardness profile (Figure 3) confirms the layer thicknesses observed in the micrographs and reinforces the absence of a diffusion zone in the H75N sample. The presence of this intermediate layer (diffusion zone) acts as a transition from the hard coating to the soft substrate, contributing to the layer's adhesion and wear resistance [32], [33].

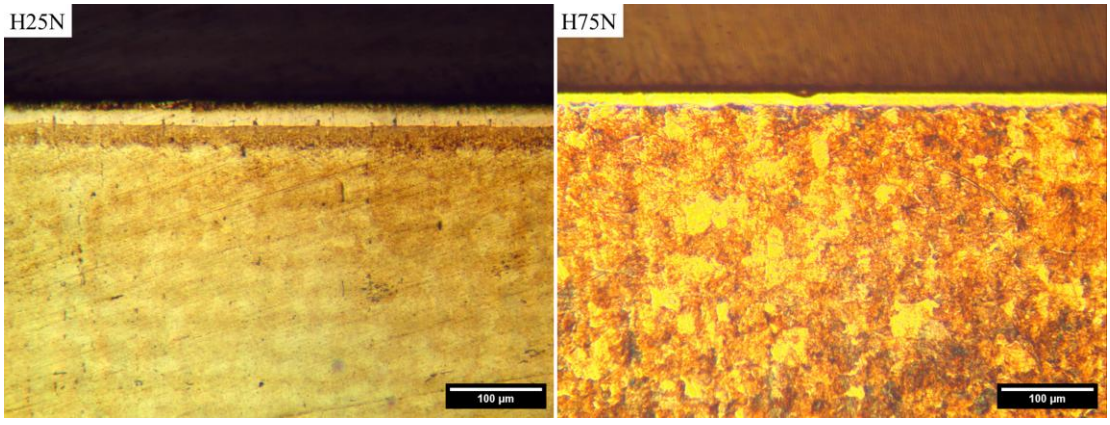

Figure 4 - Micrographs of the cross section of the treated samples.

Figure 5 shows the micrographs of the indentation carried out on the treated samples. The qualitative evaluation of the coating adhesion was conducted in accordance with the VDI 3198 standard [24]. Observing the area surrounding the indentation, radial cracks can be seen in the H25N sample, while the H75N sample exhibits no significant crack formation or delamination. Both coatings can be classified as HF1, indicating acceptable fracture behavior. The literature reports studies where

coatings deposited using the cathodic cage deposition technique, even when samples are at cathodic potential, demonstrate excellent adhesion to the substrate [22], [34].

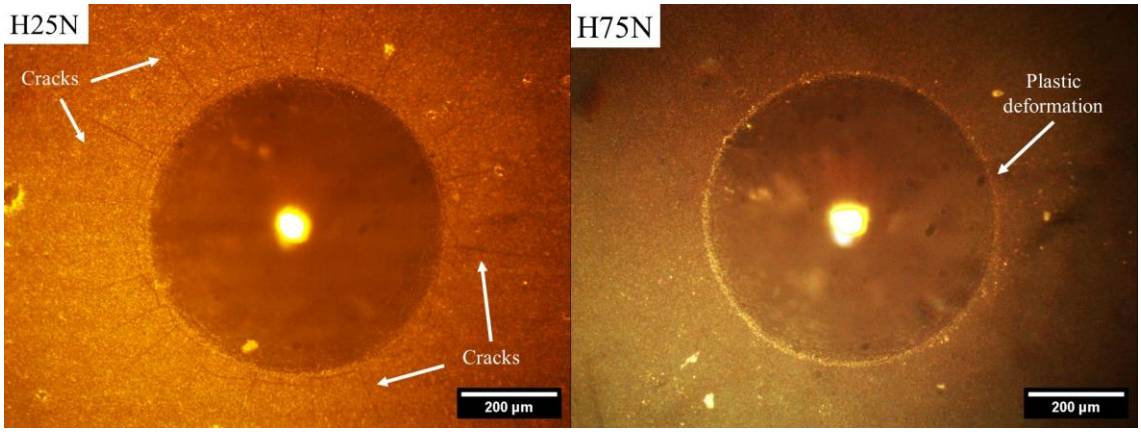

Figure 5 - Optical microscopy taken after the Rockwell hardness test.

Figure 6 shows the wear volumes of the treated samples. The deposition treatment with 75% nitrogen in its atmosphere (H75N sample) resulted in a higher wear volume than the base material, with a worn volume of $3.54x10^{-2}$ mm^3 compared to $3.41x10^{-2}$ mm^3 for the base material. The higher wear volume may be associated with the abrupt hardness gradient presented by the H75N sample. It can be stated that the best treatment parameter was H25N, which had a wear volume of $1.20x10^{-2}$ mm^3, representing a reduction of 64.8% compared to the base material. The better behavior of the H25N sample is explained by the presence of an intermediate layer and its consequent smooth hardness gradient. The hardness gradient influences the adhesion of the coating to the substrate when subjected to dynamic stresses, such as in the wear test. Fracture of the coating quickly exposes the substrate to contact and adds hard particles to the tribological pair [35].

During the wear tests, the coefficient of friction was monitored, as shown in Figure 7. Despite the lower wear volume shown by the H25N sample, it exhibited a higher friction coefficient than the base material during the initial phase of the test (the first 1000 seconds). Both the Base and H25N samples showed an average friction coefficient of 0.55 ± 0.03. The H75N treatment also starts the treatment with a higher coefficient of friction than the base material, but there is a reduction after the first 700 seconds of the test. The H75N sample presented the lowest average coefficient of friction with a value of 0.50 ± 0.05. Although it didn't have the lowest coefficient of friction, sample H25N proved to have the best performance of all the samples due to its smaller wear volume.

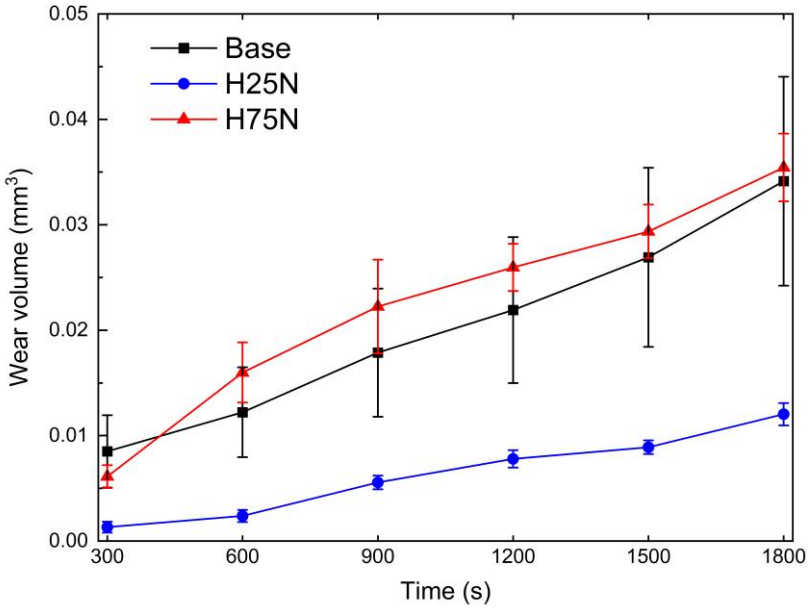

Figure 6 - Wear volume of samples Base, H25N and H75N.

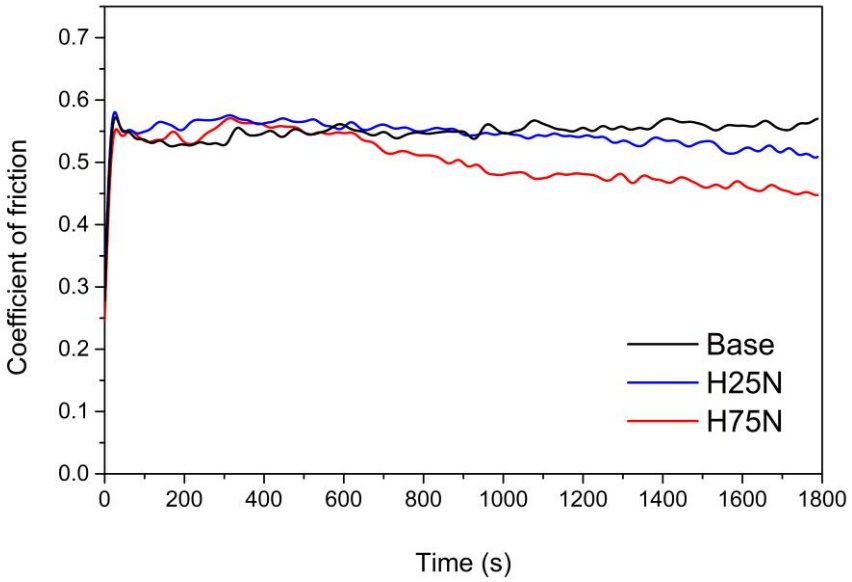

Figure 7 - Monitoring the coefficient of friction of the Base, H25N and H75N samples.

IV. CONCLUSIONS

This study evaluated how the composition of the atmosphere of treatment influenced the wear resistance and surface hardness of SAE 5160 steel treated using the CCPD technique with a Hastelloy cage. Based on the results obtained, it can be concluded that:

1. Both treatment conditions produce coatings with high hardness and good adhesion to the substrate (HF1), however the H25N sample showed a smoother hardness gradient, in contrast to the H75N sample.

2. The H25N treatment condition showed the greatest layer thickness (47.7 μm), as well as the formation of $Cr_{0.4}Ni_{0.6}$ and CrN phases (in addition to Fe_4N) which are associated with the elements originating from the cage.

3. The H25N sample exhibited better wear resistance with a 65% reduction in worn volume compared to the untreated sample, this result is associated with the high hardness and layer thickness of the coating combined with the smooth hardness gradient.

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