DISCIPLINARUM SCIENTIA
Disciplinarum Scientia. Série: Naturais e Tecnológicas
Santa Maria, v. 26, n. 2, p. 117-123, 2025
Recebido em: 13.04.2025. Aprovado em: 27.05.2025.
ISSN 2176-462X | DOI: doi.org/10.37779/nt.v26i2.5344

CATHODIC CAGE PLASMA NITRIDING OF AISI M3:2 STEEL: EVALUATION OF THE TREATMENT ATMOSPHERE EFFECT

NITRETAÇÃO A PLASMA COM GAIOLA CATÓDICA NO AÇO AISI M3:2: AVALIAÇÃO DO EFEITO DA ATMOSFERA DE TRATAMENTO

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ABSTRACT

Samples of AISI M3:2 steel were subjected to plasma nitriding using a cathodic cage, with variations in gas flow during pre-treatment and treatment to assess its influence on the properties of the resulting coatings. After nitriding, the samples were analyzed using X-ray diffraction (XRD), Vickers microhardness testing, and optical microscopy. XRD analysis revealed the formation of iron and chromium nitrides in all treated samples. Although the layer was not particularly thick, as observed in optical microscopy measurements, there was a significant increase in microhardness, with one sample exhibiting a hardness three times greater than that of the untreated material. The results demonstrate the potential of the cathodic cage nitriding technique to improve the surface mechanical properties of AISI M3:2 steel.

Keywords: Cathodic cage; Plasma nitriding; Thermochemical treatment; Tool steel.

RESUMO

Amostras do aço AISI M3:2 foram tratadas pelo processo de nitretação por plasma com gaiola catódica, com variações do fluxo dos gases no pré-tratamento e no tratamento em si com o objetivo de mostrar a influência da composição atmosférica nas propriedades dos revestimentos formados. Para caracterização das amostras foram empregadas as técnicas de difração de Raios-X (DRX), testes de microdureza Vickers e microscopia óptica. Foi observado na análise de DRX a formação de nitretos de ferro e cromo em todas as amostras tratadas. Apesar das camadas nitretadas não apresentarem grande espessura segundo a análise metalográfica, foi verificado um aumento significativo na dureza superficial, com incrementos superiores a 200% em relação ao material não tratado. Os resultados demonstram o potencial da técnica de nitretação por gaiola catódica na melhoria das propriedades mecânicas superficiais do aço AISI M3:2.

Palavras-chave: *Aço ferramenta; Gaiola Catódica; Nitretação a plasma; Tratamento termoquímico.*

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1 INTRODUCTION

High-speed steels, with over a century since their development, continue to be widely used in cutting tools - such as bits, chisels, drills, taps, and milling cutters - due to their high hardness and wear resistance. (MESQUITA, 2016). These characteristics must be present in forming and machining tools used in the materials processing industry, as they extend tool life and contribute to cost reduction and decreased downtime due to tool replacement. (SERRA *et al.*, 2020).

Although high-speed steels have good wear resistance, this characteristic can be improved through thermochemical treatments such as plasma nitriding. Plasma nitriding is a thermochemical treatment aimed at improving the surface properties of various types of steels by forming high-hardness coatings through nitrogen diffusion and nitride precipitation. However, the plasma nitriding process presents drawbacks associated with the high cathodic potential of the sample, such as the edge effect and the occurrence of electric arc discharges on the workpieces. (ZHAO *et al.*, 2006). Oliveira (2017) in his study of plasma nitriding with an active screen on ASTM M2 steel showed the superior tribological behavior of the potential treatment, with a wear rate up to seven times lower than the conventional nitriding process.

As an innovative alternative, the Cathodic Cage Plasma Nitriding (CCPN) technique was developed, representing a technological advancement by eliminating the inherent defects of the conventional plasma nitriding process. In the CCPN process, the samples are placed on an insulating disc - typically made of alumina (Al_2O_3) - and surrounded by a cage with a cylindrical geometry (ALVES *et al.*, 2006). Since cathodic potential is applied to the cage rather than directly to the workpieces, the typical defects of conventional plasma nitriding are mitigated (BOTTONI; GRIPA; GONTIJO, 2014).

The present work aims to study the effects of cathodic cage plasma nitriding on AISI M3:2 high-speed steel, a material that has been scarcely explored in the literature. Considering its potential for the manufacture of tools used in the mechanical manufacturing industry, the influence of the treatment atmosphere on the composition and hardness of the formed layers was analyzed, seeking to understand the material's behavior under different nitriding conditions.

2 METHODOLOGY

The material used in this study was AISI M3:2 high-speed steel, obtained in the form of a cylindrical rod, from which the samples were extracted. Sample preparation involved grinding with sandpaper of grain sizes ranging from 180 to 1200 mesh, followed by polishing with 3μm diamond paste on a felt polishing cloth. Before each treatment, the samples were cleaned in an ultrasonic bath with an acetone solution for 10 minutes and then dried with a hot air jet.

For the nitriding treatments, a cathodic cage made of AISI 316 stainless steel was used, with dimensions of 80 mm in diameter, 50 mm in height, and 5 mm in thickness. The cage featured holes with a diameter of 8 mm and a center-to-center distance of 9.2 mm between adjacent holes. In cathodic cage nitriding, the samples are placed on an alumina disk to maintain a floating potential. An alumina disk with a diameter of 30 mm and a thickness of 3 mm was used in the treatments.

Table 1 - Parameters used during pre-sputtering.

Sample	Gas flow			Time (h)	Temperature (°C)	Pressure (mbar)
	Ar	H_2	N_2			
CCPN_1	100	0	0	1	350	1.5
CCPN_2	50	50				2.5
CCPN_3	100	0				1.5
CCPN_4	100	0				1.5

Source: Authors (2025).

The treatment conditions used in this study are shown in Tables 1 and 2, where the composition of the treatment atmosphere varied across the four conditions analyzed. Conditions 1 and 2 (CCPN_1 and CCPN_2) share the same treatment parameters (sputtering), but pre-sputtering was performed with different atmospheres (Table 1). Samples 1, 3, and 4 underwent the same pre-sputtering process, but the treatment atmosphere consisted of a mixture of argon, hydrogen, and nitrogen in different proportions (Table 2).

Table 2 - Parameters used in cathodic cage plasma nitriding (sputtering) treatments.

Sample	Gas flow			Time (h)	Temperature (°C)	Pressure (mbar)
	Ar	H_2	N_2			
CCPN_1	10	10	40	4	450	3
CCPN_2	10	10	40			3
CCPN_3	12	18	30			3.5
CCPN_4	18	12	30			3

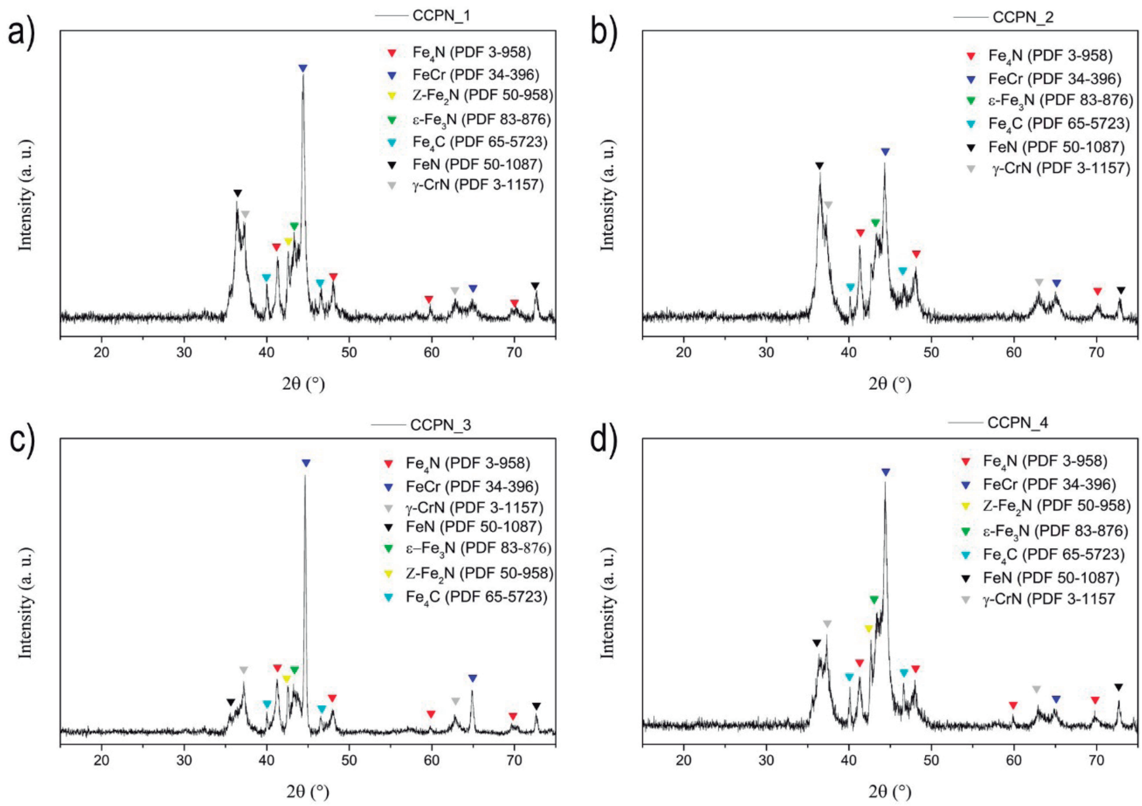
Source: Authors (2025).

The reactor used in the treatments consists in a vacuum chamber connected to a voltage source, a vacuum pump, and a set of instruments for monitoring and controlling the treatment. For the characterization of the samples, Vickers microhardness, optical microscopy and X-ray diffraction (XRD) tests were performed. For the microhardness tests, an INSIZE model ISH-TDV 1000 microdurometer was used, with a load of 50 gf. The mean and standard deviation of 20 indentations made on each sample were used as the results. To measure the layer thickness, micrographs were taken of the cross-section of the samples using a BEL PHOTONICS model MTM-1A metallographic microscope. XRD analysis was carried out using a SHIMADZU XRD-6000 diffractometer operating at 40 kV and 30 mA, using Cu-Kα radiation, the 2θ scan range measured was 15° to 75°, with a speed of 2°/min.

3 RESULTS AND DISCUSSION

Figure 1 presents the X-ray diffraction patterns of the samples treated by cathodic cage plasma nitriding, where the formation of iron nitrides (PDF 50-1087, PDF 50-958, PDF 83-876, and PDF 3-958), chromium nitride (PDF 3-1157), as well as the FeCr (PDF 34-396) and Fe_4C (PDF 65-5723) phases can be observed in all treated samples.

Figure 1 - Diffractograms of treated samples.



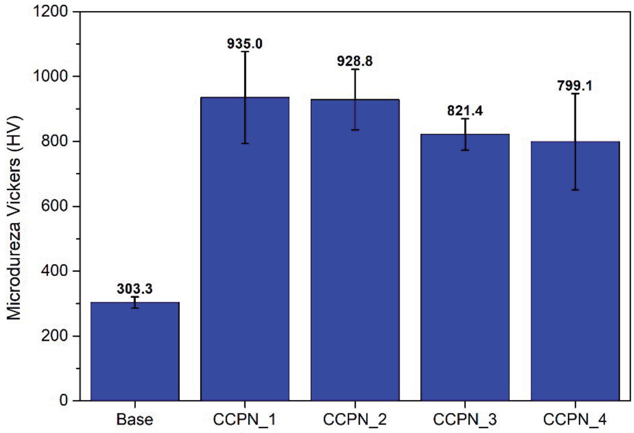
Source: Authors (2025)

A comparison of the diffractograms of the CCPN_1 and CCPN_2 samples reveals a decrease in the intensity of the FeCr peak at 2θ = 44.3°, as this phase does not contain nitrogen in its composition. This change in peak intensity can be attributed to the reduction of argon in the pre-sputtering process, since lowering the partial pressure of argon in the atmosphere decreases the sputtering rate (SABURI *et al.*, 2003; SAIKIA *et al.*, 2016). In the samples with the same pre-sputtering, we can see that the CCPN_3 sample shows less intensity in the peaks of the nitride phases (FeN, Fe_2N, Fe_3N, Fe_4N and CrN), which may be related to the increase in hydrogen in the atmosphere.

Figure 2 presents the values of microhardness obtained for each treatment condition. The reference sample (Base sample) exhibited a microhardness of 303.3 ± 17.3 HV. All treated samples showed an increase in surface hardness of more than 2.5 times compared to the untreated sample, consistent with findings reported in the literature (SOUSA *et al.*, 2008; ABREU *et al.*, 2020).

The surface hardening of the treated samples is associated with the formation of high-hardness nitrides, as observed in the diffractograms in Figure 1.

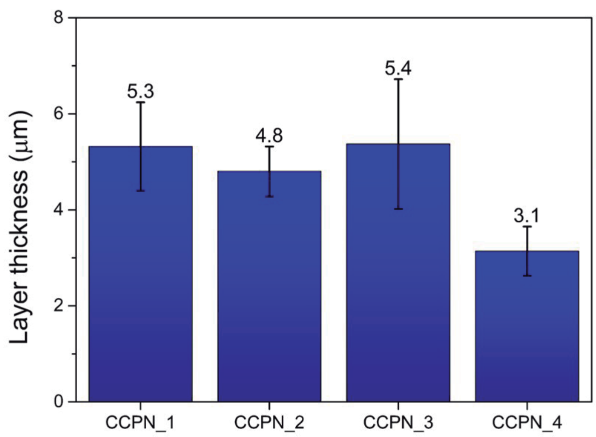
Figure 2 - The microhardness Vickers of the untreated sample of AISI M3:2 steel (Base) and the samples treated by cathodic cage plasma nitriding



Source: Authors (2025)

Figure 3 presents the layer thickness values obtained in the treatments, it can be observed that the CCPN_4 samples have a notably lower layer thickness, indicating that the high argon content in the treatment may be detrimental to layer growth. In addition, it can be seen that the coatings formed had uniform layers (Figure 4), which can be attributed to the floating potential of the sample, preventing the edge effect (SOUSA *et al.*, 2009). Furthermore, according to Raza *et al.* (2018) and Liu *et al.* (2023), pre-sputtering does not influence the layer thickness, acting only to activate and clean the substrate surface. Additionally, a higher gas pressure proportion tended to promote the growth of the nitride layer, which is associated with an increased sputtering rate due to a higher probability of particle collisions (BOIDIN *et al.*, 2016).

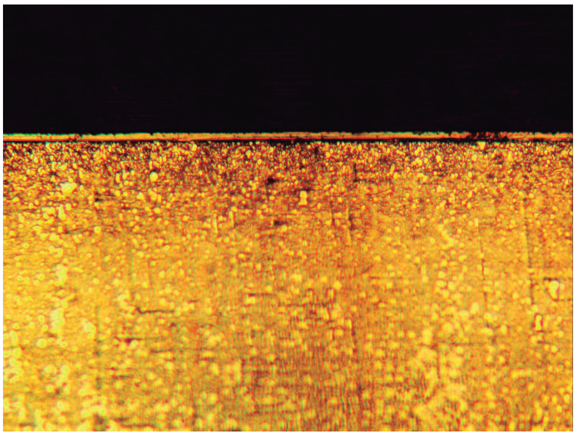
Figure 3 - Thickness of the nitrided layer obtained in plasma nitriding treatments with cathodic cage.



Samples	Thickness layer (µm)
CCPN_1	5,32 ± 0,92
CCPN_2	4,80 ± 0,52
CCPN_3	5,37 ± 1,35
CCPN_4	3,14 ± 0,51

Source: Authors (2025).

Figure 4 - Micrograph of the cross section of sample CCPN_3, which obtained the greatest layer thickness.



Source: Authors (2025).

4 CONCLUSIONS

In this work, the effect of the atmospheric composition during pre-sputtering and sputtering on the properties of the formed layers was studied. Based on the results obtained, it can be concluded that the nitriding process on M3:2 steel significantly increased the surface microhardness of the samples due to the formation of iron nitrides (Fe_4N, ε-Fe_3N, ζ-Fe_2N, and FeN) and chromium nitride (γ-CrN).

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