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# Cathodic cage plasma nitriding (CCPN) of austenitic stainless steel (AISI 316): Influence of the different ratios of the ($N_2/H_2$) on the nitrided layers properties

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## ABSTRACT

In this study, we assess how the ratio ($N_2/H_2$) – working atmosphere gas mixture – influences the properties of the layers produced on austenitic stainless steel surface by Cathodic Cage Plasma Nitriding (CCPN). The nitriding atmospheres contained nitrogen and hydrogen in three proportions: 20% $N_2$ + 80% $H_2$, 50% $N_2$ + 50% $H_2$, and 80% $N_2$ + 20% $H_2$ delivered at a flow rate of 20 sccm. The effect of 100% $N_2$ was also examined. The morphology, microstructure and electrochemical properties of the layer produced were studied. The samples characteristics were determined by optical microscopy, x-ray diffraction, microhardness test and electrochemical potential curves.

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## 1. Introduction

The need to increase the service life of machine components and working tools has demanded the development of various surface treatment techniques. It has further prompted a tireless quest to find an optimal combination of different processing parameters. These parameters include temperature, treatment time, pressure, pulse frequency and the combination of gases employed in nitriding processes. The ability to form isotropic layers with increased homogeneity, greater substrate adherence, higher hardness and lower porosity are necessary to achieve a leap in the quality of surface treatments techniques. Stainless steel is widely used in engineering due to its high corrosion resistance even though it has low wear resistance and low hardness. These last two properties are significantly improved by ion nitriding [1–8]. It is well-known that the nitriding of austenitic stainless steel at temperatures above 723 K produces high abrasion resistance and high hardness but it results in a decreased corrosion resistance. This is primarily due to the precipitation of chromium nitride which consequently reduces the chromium content in the matrix [1–7]. However, when nitridation is performed at lower temperatures a phase so called expanded austenite or S phase is produced. This phase has high hardness, increased wear resistance and excellent corrosion resistance [9,10].

In previous studies employing different nitriding processes the layer thickness was found to depend on elevated treatment temperature [1,8], changes in the pulse frequency [13], variation of the gas composition [5,14] and other parameters [15], and increases with working pressure [11,12] and treatment time [4]. The aim of this work was to evaluate the increase in the thickness of the nitrided layers and the resulting properties, mainly the corrosion resistance as a function of the ratio $N_2/H_2$ for a fixed working pressure.

According to some authors [16,17] the presence of hydrogen plays important role in the nitriding process mainly due to its strong deoxidation ability besides the formation of the NH molecule that favors the diffusion process of nitrogen.

Saeid Amiri Moradshahi and Masoud [18] comment that the increase of the nitrogen content in the modified layer leads to an increase in the internal tensions that can produce cracks in the nitrided layer. These cracks allow the austenite matrix to come into contact with aggressive solutions during electrochemical testing, which reduces the polarization corrosion resistance of the sample. This resistance will be further reduced if nitrides precipitate. Additionally, they state that an increase of hydrogen content in the gas mixture up to 50% vol. will lead to a lower nitrogen

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**Table 1**
Atomic chemical composition of the stainless steel AISI 316* (at. %).

| | Cr | Ni | Mo | C | Mn | Si | P | S | Al | Fe |
|---|---|---|---|---|---|---|---|---|---|---|
| % | 16–18 | 10–14 | 2–3 | 0.08 | 2.0 | 1.0 | 0.04 | 0.03 | 0.02 | Balance |

*Supplied by Villares Metals S.A.

concentration at the surface, thus causing the formation of a thicker layer free of precipitation, with low internal stresses and better corrosion resistance. They suggest that the addition of hydrogen increases the hardness and thickness of the nitrided layer by reducing the formation of oxides on the surface layer and by increasing the density of active species in the nitriding plasma. The best results were obtained with nitrogen content in the range of 15–25% vol.

S. Kumar et al. [19] carried out a series of nitriding experiments with hydrogen concentrations varying between 5 and 50% and compared the results with the treatment in a pure nitrogen atmosphere. They showed that the maximum nitrided layer thickness occurs at a concentration of approximately 15% hydrogen. They also showed that for a hydrogen concentration in the range of 5–50% there is an increase in the nitriding efficiency. They found that with the inclusion of an approximately 75% hydrogen in the nitriding gas mixture the nitrided layer thickness and microhardness values are less than for samples treated with 100% nitrogen.

In this study, we employed the cathodic cage nitriding technique which was developed in the plasma nitration (LabPlasma) UFRN [20,21].

## 2. Materials and methods

Austenitic stainless steel AISI 316 in the annealed state (microhardness 240 HV) was used as the material in this study. Its elemental composition is given in Table 1. The samples were plasma nitrided via the cathodic cage technique at 723 K for 5 h. The nitriding atmosphere was a mixture of nitrogen and hydrogen. We used the same equipment employed in conventional nitriding with the addition of a cathodic cage. A schematic diagram of the experimental arrangement is shown in Fig. 1. A direct current voltage source with a maximum voltage of 1.5 × $10^3$ V and a maximum current 2.0 A was employed.

A cathodic cage (detailed in Fig. 1) made of 0.80 mm thick austenitic stainless steel (AISI 316) was placed inside the stainless steel cylindrical vacuum chamber. The vacuum chamber has a diameter of 30 cm and a height of 40 cm. The cathodic cage is 112 mm in diameter, 25 mm in height and has 8-mm diameter holes. The holes are uniformly distributed at 9.2 mm on center. In this configuration, the cage wall is the cathode and the chamber wall is the anode, the sample is placed on an insulating alumina disc at floating potential, so the plasma is consequently formed on the cathodic cage wall and not directly on the sample surface. To remove the passive layer of oxide and/or grease residues on the sample surface all samples were pretreated in 200 Pa hydrogen atmosphere at 573 K for 20 min. Furthermore, the samples were treated for 5 h at a temperature of 723 K and pressure of 250 Pa at a total flow rate of 20 sccm in one of the following atmospheres: 20% $N_2$ + 80% $H_2$, 50% $N_2$ + 50% $H_2$, 80% $N_2$ + 20% $H_2$, and 100% $N_2$. To reduce the risk of oxidation post-treatment the samples were cooled to 353 K inside the chamber under nitrogen flow.

The samples were then cut, embedded, polished, and etched with aqua regia and Beraha II. X-ray diffraction analysis was performed on a Shimadzu XRD-6000, using molybdenum radiation (Mo-$K_\alpha$, $\lambda = 0.07094$ nm) at 40 kV and 30 mA. Optical microscope (Olympus BX60M) was used to record the resultant morphology and thickness of the nitrided layers. Microhardness tests were performed using a Pantec 1000 with a 100 g weight. Potentiodynamic anodic polarization curves and pitting potentials were obtained at 298 K, in a 0.1 M $H_2SO_4$ and a 3.5% NaCl solutions at a scan rate of $1.0 \times 10^{-3}$ V/s. A saturated calomel electrode was used as reference electrode to which all potentials are referred. Electrochemical impedance spectroscopy (EIS) was conducted at corrosion potential of the system by varying the ac frequency from 100 MHz to 10 mHz with the perturbation within 5 mV and 15 points per decade. The tests were performed at the temperature of 298 K and for the curve fitting data analysis was used the Z-view and Ivium softwares.

## 3. Results and discussion

### 3.1. Characterization of plasma nitrided layers

The Fig. 2 shows cross-sectional micrographs of stainless steel AISI 316 samples nitrided in different atmospheres. The nitrided layer thickness and microhardness values are presented in Table 2. The greatest layer thicknesses were found for samples treated with 80% $N_2$ and 100% $N_2$. This differs from samples treated in plasmas with less nitrogen content (20% $N_2$ and 50% $N_2$). The thickness of the nitrided layer increased with increasing nitrogen content up to 80% but decreased when a pure nitrogen atmosphere was used. Additionally, in our studies the microhardness was also higher for

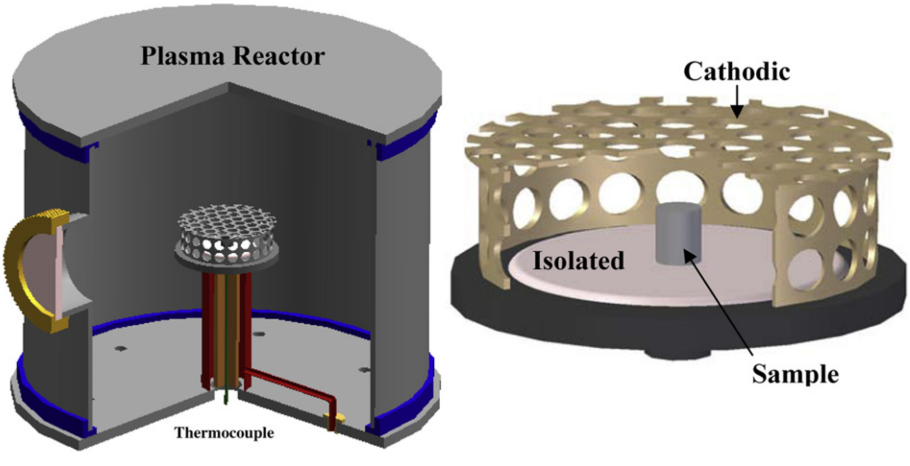

**Fig. 1.** Schematic view of the reactor ion nitriding, showing the sample inside the cathode cage.

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