

Nitriding using cathodic cage technique of martensitic stainless steel AISI 420 with addition of CH₄

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ABSTRACT

AISI 420 martensitic stainless steel samples were nitrided by cathodic cage technique with addition of methane in the atmosphere aiming to reduce chromium nitride precipitation, to increase hardness and wear resistance without the presence of characteristic defects inherent to the ionic nitriding process. Microhardness measurements and X-ray analysis confirm the formation of a high hardness double-layer constituted by two regions: one internal region composed of carbon and another composed of nitrogen.

Keywords: plasma nitriding, carburizing, martensitic steel, hardness, cathodic cage nitriding.

1 INTRODUCTION

Stainless steel is largely used in engineering due to its high corrosion resistance, In spite of, its small wear resistance and its reduced hardness. Those two last properties are significantly improved through ionic nitriding [1, 2]. As it is known, the austenitic stainless steel nitrided at temperatures superior to 723 K produces surfaces of high wear and hardness resistance but the corrosion resistance decreases substantially, due to chromium nitride precipitation resulting in the reduction of chromium content in the stainless steel matrix. However, when it is carried out at temperatures below 723 K it produces a supersaturate solid solution of nitrogen nominated “S phase” or expanded austenite, witch confers to the surface a high hardness and wear resistance associated to an excellent corrosion resistance [3, 4].

Although there are several works published on austenitic stainless steel nitriding, the same does not happen about martensitic stainless steel nitriding [5], even if steel remains a good candidate for use in some corrosive environments applications.

In this work the objective is to demonstrate the efficiency of a new device developed in the LabPlasma-UFRN [6], in the ionic nitriding of martensitic stainless steel AISI 420, making possible to obtain a thick superficial layer with high hardness without edge effect [7, 8]. The high hardness obtained in that process is due to the appearance of a phase called expanded martensite [9-11]. This nomination is made in comparison with expanded austenite.

2 MATERIALS AND METHODS

AISI 420 martensitic stainless steel samples were machined in cylindrical form, previously annealed, grounded with 320, 400, 600 and 1200 sandpapers, meshed and smoothed over with felt disc with diamond suspensions of 1 and 0.3 μm. They were then ultrasonically cleaned in an acetone bath, before being placed into the nitriding chamber.

The conventional nitriding system consists of a power supply (maximum voltage of 1500 V, maximum electrical current of 2 A), a cylindrical stainless steel vacuum chamber (40 cm in diameter and 40 cm in height). In this work the same chamber was used with the addition of a cathodic cage, made from

stainless steel 316. Their dimensions are 0.8 mm in thickness, 112 mm in diameter, 25 mm in height and with holes of 8 mm in diameter, uniformly distributed at a distance of 9.2 mm among adjacent holes [2]. The plasma is formed on the cathodic cage that is placed on the workpiece negatively loaded, instead of the samples surface [2].

The samples were previously treated with an argon plasma during 30 minutes at a temperature of 573K. The treatment conditions were as follow: 5 hours, temperatures of 673, 723, and 773 K with nitriding mixtures of 95% N₂-5% CH₄. The flow was of 20 sccm adjusted by a controller. The treatment pressures were 250 and 500 Pa, measured by a manometer of barocel capacitance manually adjusted. After the nitriding process the samples were annealed, polished and attacked with Beraha's reagent,. Phases and composition were analyzed using X-ray diffraction (DRX). The analyses described were done using Cu K lines (wavelength: 0.154 nm), operated in 40 KV in an instrument DRX (Shimadzu, XRD-6000). Optical microscopy was used to observe morphology and thickness of nitriding layer. Finally a microhardness profile was made to confirm its thickness and evaluate its uniformity.

3 RESULTS AND DISCUSSIONS

AISI 420 Martensitic stainless steel nitriding has attracted attention due to its peculiarities and because such stainless steel is less utilized than the austenitic stainless steel. In the process of nitriding in the presence of methane, the nitrided layers thickness are inferior than that obtained by cementation, but hardness is superior to that obtained in cementation and nitriding without methane addition, as shown in the Table 1.

Table 1: Microhardness and thickness of composite layer of nitriding samples under different conditions.

Pressure (Pa)	Temperature (K)	Microhardness (HV)	Thickness (µm)
250	673	1000	-
250	723	1400	23,7
250	773	440	32,8
500	673	1006	-
500	723	1600	6,6
500	773	1310	20,6

Figure 1 presents optical micrographs (1a and 1b) and SEM pictures (1c and 1d) of the nitrided samples.

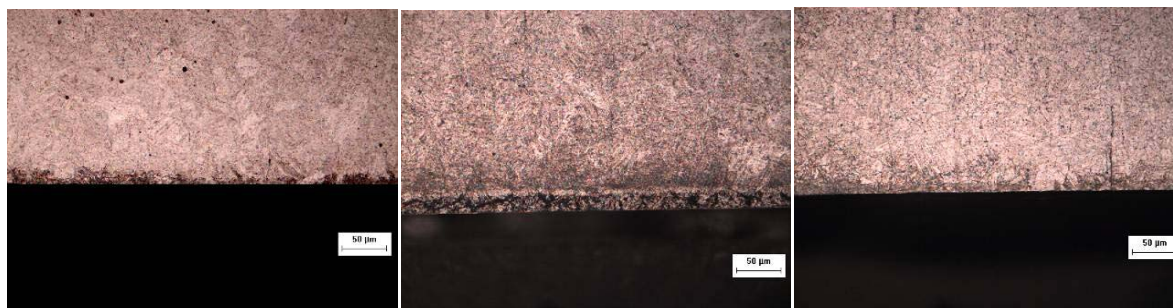


Figure 1(a): Nitrided samples under a pressure of 250 Pa, at temperatures of 673, 723, 773 K respectively.

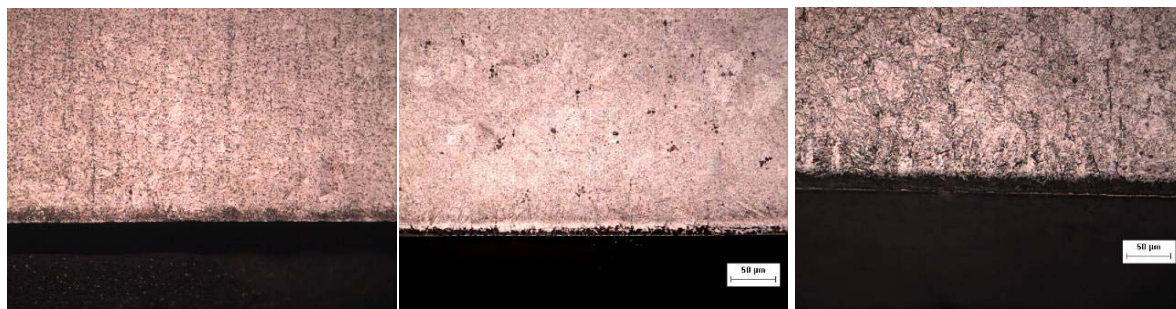


Figure 1(b): Nitrided samples under a pressure of 500 Pa, at temperatures of 673, 723, 773 K respectively.

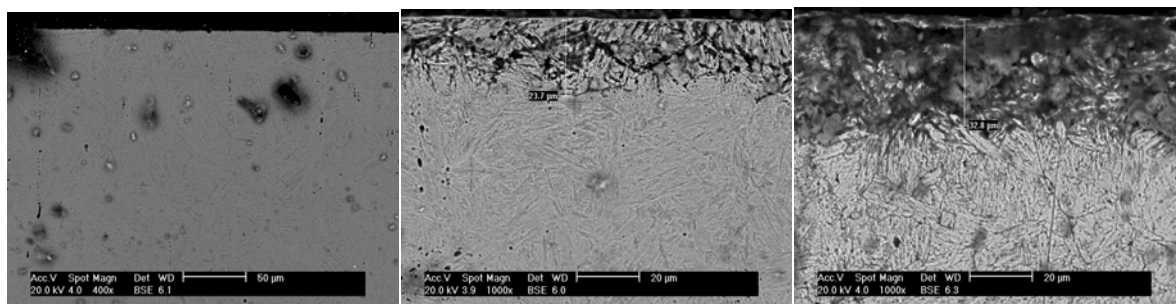


Figure 1(c): Nitrided samples under a pressure of 250 Pa, at temperatures of 673, 723, 773 K respectively.

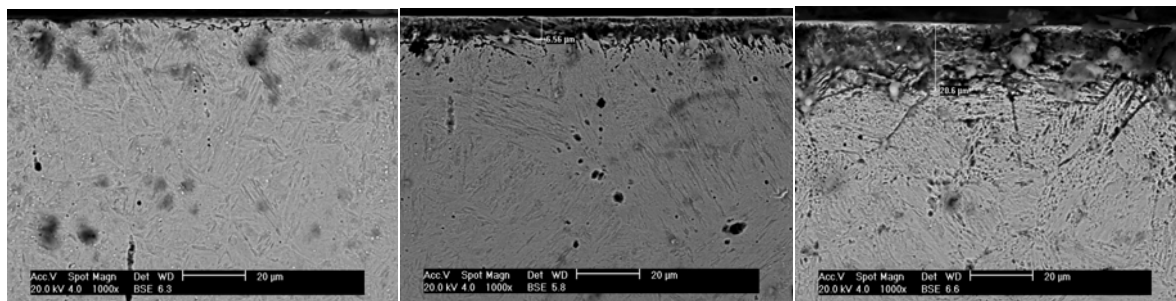


Figure 1(d): Nitrided samples under a pressure of 500 Pa, at temperatures of 673, 723, 773 K respectively.

The images done by SEM (1c and 1d) confirm the thickness of the composite layer for the nitrided samples at temperatures 723 and 773 K. For the sample nitrided at temperature of 673 K the composite layer do not appear.

The microhardness profiles presented in figures 2 and 3 show a microhardness increase and gradual variation for samples nitrided at temperature of 723K (250 Pa) and for temperatures of 723 and 773K (500 Pa). The sample treated at a 773 K and in 250 Pa does not present superficial hardening due to its fragility induced by tempering temperature. In contrast, the sample treated at 773 K and under the pressure of 500 Pa shows high microhardness produced by the increase of carbon introduced in the sample due to the higher pressure, increased the minimum temperature for the occurrence of the tempering fragility. It is worth to mention that the microhardness value for the sample nitrided at temperature of 723K (250 Pa) decreases gradually up to a depth of 60 µm, where it reaches that of the substrate. Whereas for the samples nitrided at temperature of 723 and 773K (500 Pa), such decrease is faster, reaching the value of the substrate around 30 µm.

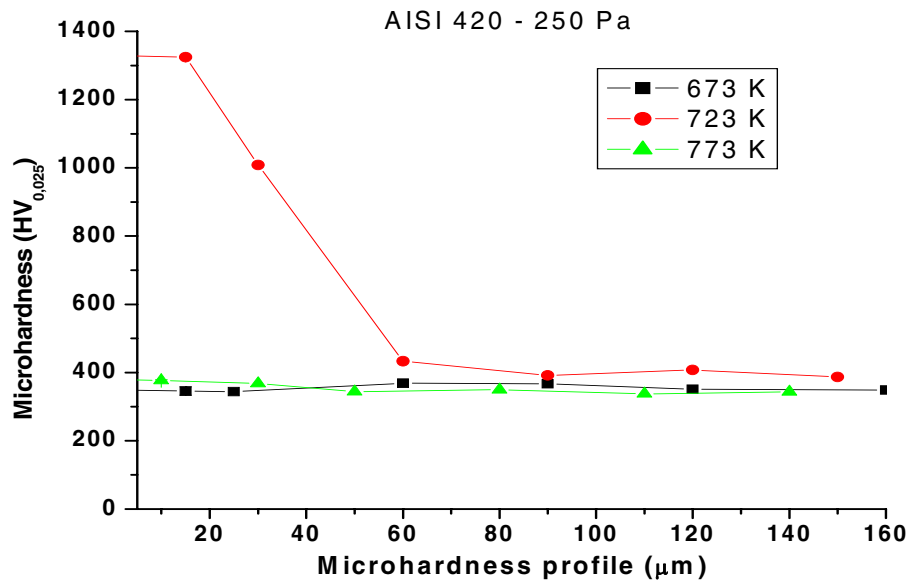


Figure 2: Microhardness depth profile of nitrided samples under a pressure of 250 Pa.

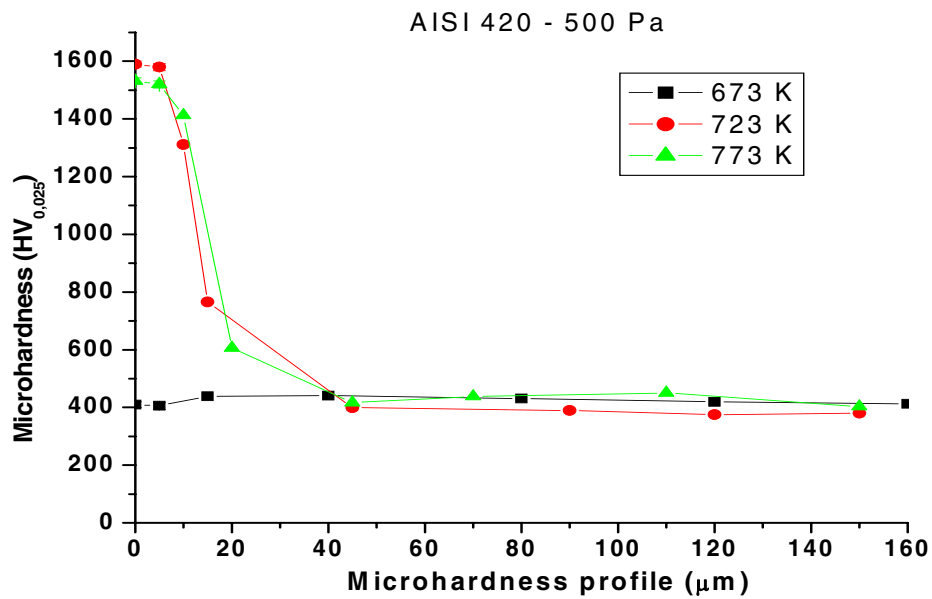


Figure 3: Microhardness depth profile of nitrided samples under the pressure of 500 Pa.

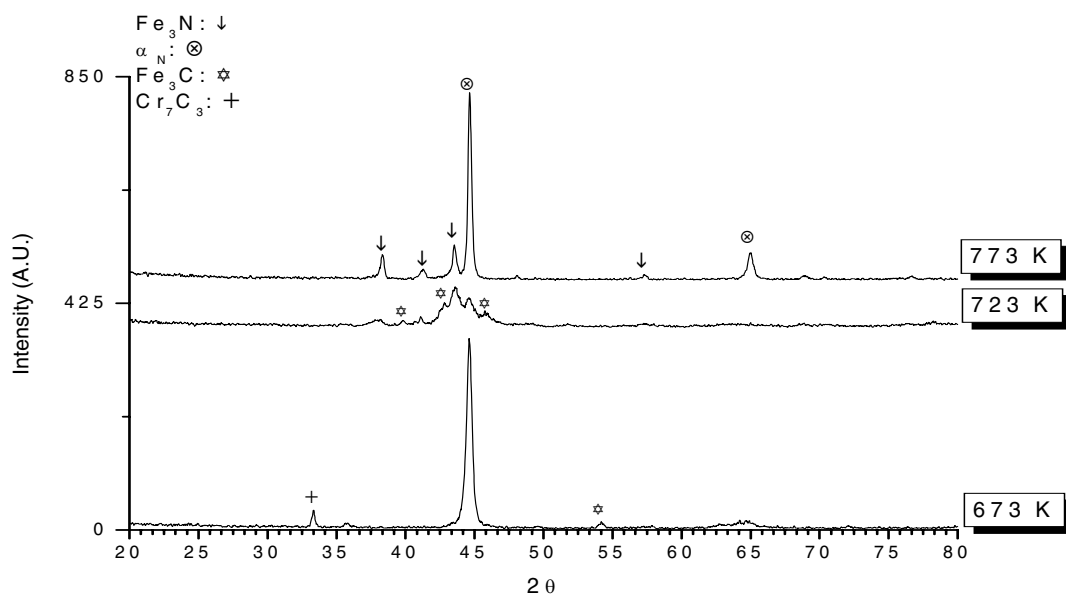


Figure 4: X-ray diffractograms of samples nitrided under 250 Pa at temperatures of 673, 723, 773 K.

X-ray diffraction patterns practically confirm the total elimination of chromium nitride. The carbides presence is due to the higher percentage of carbon on AISI 420 martensitic stainless steel. Also it was observed the presence of α_N phase, nominated expanded martensite, which is responsible for the high microhardness.

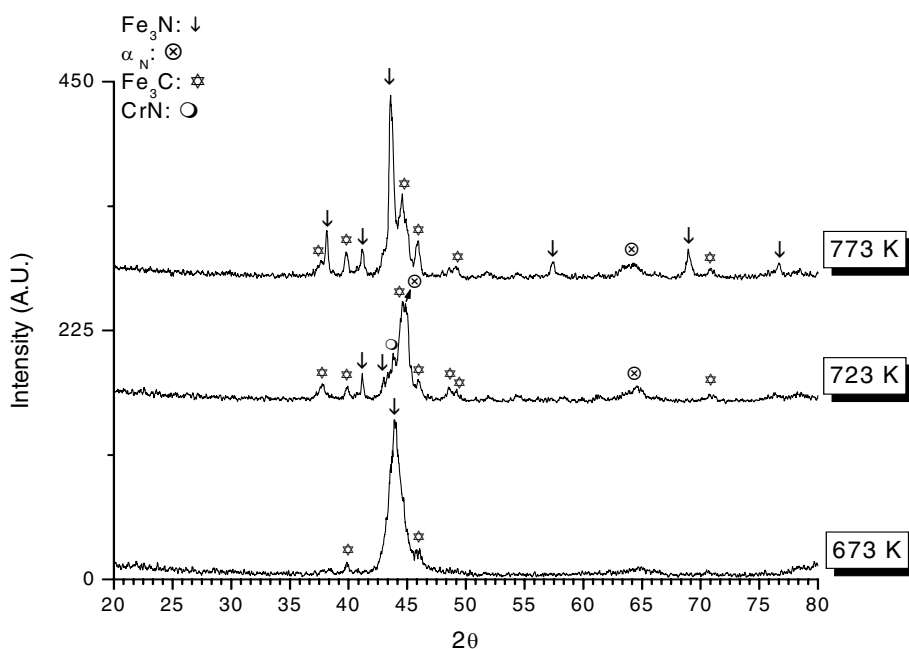


Figure 5: X-ray diffraction patterns of nitrided samples under 500 Pa at temperatures of 673, 723, 773 K.

4 CONCLUSIONS

Addition of low amounts of methane in the nitriding atmosphere during the cathodic cage plasma nitriding produces considerable changes in the properties of martensitic stainless steel. It produces a dual compound layer, the inner layer being saturated by carbon and the outer one being saturated by nitrogen with greater thickness and hardness than that obtained by conventional nitriding process or cementation. The process also eliminates inconveniences associated to DC conventional nitriding as, for instance, the edge effect. The increase in hardness is possible due to the elimination of chromium nitride precipitation and the consequent increase of expanded martensite, without reducing the corrosion resistance.

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