AUSTENITIC STAINLESS STEEL AISI 316 PLASMA NITRIDED BY CATHODIC CAGE TECHNIQUE

R.R.M. de Sousa[a,*]; F.O. de Araújo[b]; K.J.B. Ribeiro[c]; J.A.P. da Costa[d]; C. Alves Jr[c]

[a] Centro Federal de Educação Tecnológica do Piauí, Departamento de Mecânica, 64.000-040, Teresina, PI, Brazil
[b] Universidade Federal Rural do Semi-Árido, Depto. de Ciências Ambientais, 59625-900, Mossoró, RN, Brazil
[c] UFRN, Departamento de Física, LabPlasma, 59.072-970 Natal, RN, Brazil
[d] Universidade Federal do Ceará, Departamento de Física, Campus do Pici. 60.020-181, Fortaleza, CE, Brazil

ABSTRACT

A series of austenitic stainless steel AISI 316 cylindrical samples with different heights were simultaneously nitrided using the cathodic cage technique. In this technique the samples are placed under a floating potential inside a cage which shields the cathodic potential. A systematic study of the effect of the nitriding temperature was carried out in order to evaluate the efficiency of this technique over the uniformity of the layers formed on samples of different heights. The samples were characterized by optical microscopy, x-ray diffraction and microhardness measurements. The results were compared with those obtained from conventional ionic nitriding, and it was verified that the samples nitrided by the conventional technique were less uniform than those treated through this new technique.

1. INTRODUCTION

Conventional plasma nitriding is a well accepted industrial process because it has several advantages in relation to other nitriding techniques (using gas and salt baths) such as greater economy of gases and shorter process time, since the nitrogen diffusion speed is greater [1]. It is used for the improvement of the surface properties such as hardness, wear and corrosion resistance, increasing the useful life of the treated pieces. However there are some drawbacks as the hollow cathode effect, the border effect, non-uniform temperature and arcing, especially in the treatment of samples having a complex geometry [2-4]. Samples with different dimensions, when treated through plasma nitriding, show a thermal gradient between the treated sample surfaces and their bases [5-6]. In the case of a 10mm height sample, for instance, such a temperature difference may reach 100 ºC.

In order to correct for undesirable effects such as these, various alternatives to the nitriding process, such as pulsed plasma I3P [7,8], have been developed. In the last four years an additional technique, called active screen plasma nitriding (ASPN), in which the samples are enclosed by a screen on which a high cathodic potential is applied, has appeared. Thus the plasma acts on the screen and not on the sample surface. The components are treated under a floating potential or a low polarization voltage (e.g. 100 to 200V) through an auxiliary power supply [2].

In this work, we make use of the same principle as used in the ASPN technique, but with a modified experimental arrangement [9] shown in fig. 1 in order to enhance the sputtering and to increase the deposition rate. As the plasma is not formed directly on the sample surface but on the metallic cage the defects inherent to the conventional nitriding process are eliminated [9-12]. In addition, the temperature on the sample surface is uniform, since it is warmed up to the treatment temperature by the radiation emitted by the cathodic cage.
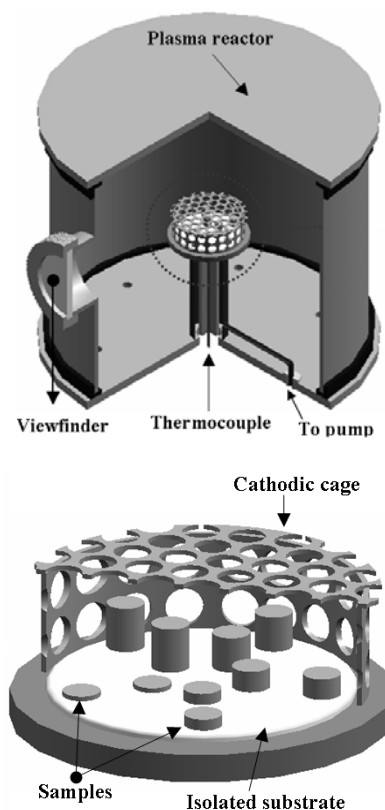

Figure 1 - The treatment reactor scheme and spatial distribution of samples used.

* romulorms@gmail.com

2. EXPERIMENTAL

The samples were machined from austenitic AISI 316 stainless steel into cylindrical shapes of height 1, 3, 5 and 10mm, previously annealed, sandpapered with 300 to 1200 grades, and polished with felt discs with 1 and 0.3μm diamante slurry, before being placed in the treatment chamber.

The conventional nitriding system consists of a power supply (maximum 1500V, 2A), a vertically mounted cylindrical vacuum chamber (40cm in diameter 40cm of height) made of AISI 316 austenitic stainless steel. We made use of the same chamber with the addition of the cathodic cage made of 0,8mm thick stainless steel 316, 112mm in diameter and 25mm in height, with 8mm diameter holes spread uniformly, with 9.2mm between centers of adjacent holes [9]. A set of ten samples, two of each height, were placed on an alumina disc, as shown in fig. 1. The plasma is actually formed on the cage, which is the cathode, not directly on the sample surface.

The treatment conditions were the following: treatment time: five hours; reference temperature within the nitriding chamber: 637, 773 and 843K, as measured by a thermocouple attached to the sample holder; nitriding mixture: 80% N_2/H_2; flow rate: 20 sccm, adjusted using a mass flow controller. The treatment pressure of 250 Pa, measured by a barocel capacitance manometer, was adjusted manually.

The phase composition and texture was analyzed by a Shimadzu XRD-6000 x-ray diffractometer operated at 40 KV and using CuKα (λ = 0.154 nm). Optical microscopy was used to observe the morphology and thickness of the nitrided layers. Finally, microhardness profiling was carried out to evaluate thickness and uniformity of the layers.

3. RESULTS AND DISCUSSION

The micrographs for samples of different heights nitrided at temperatures of 673, 723 and 773 K. are presented in fig. 2, where we observe that for a fixed treatment temperature the nitrided layers thicknesses are nearly the same, while for the correspondent samples nitrided by the conventional technique, there exist a considerable variation [5, 6].

In table 01 the values of the thicknesses of layer of the treated samples are presented for the two plasma nitriding techniques. A small gradient is verified in the variation of the thickness of the layers nitrided by the new technique as a function of the height of the samples, especially for higher treatment temperatures (773 K), where the maximum variation observed in the thickness is of the order of 10%. This is unlike the thickness of the sample treated by conventional nitriding technique, where the thickness variations were of the order of 25% between two successive heights [5].

In addition, the samples treated in the cathodic cage at the same temperature did not have significant differences in the levels of hardness for different heights, as is shown in figure 3.
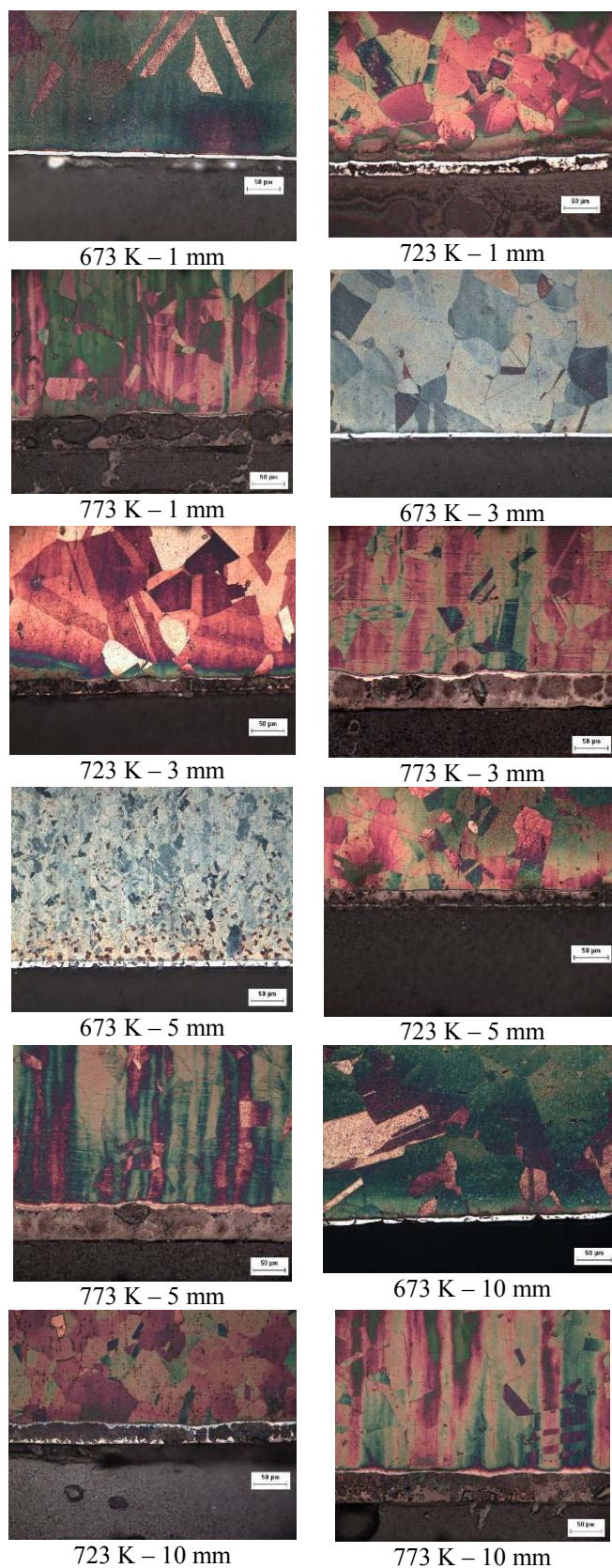

673 K – 1 mm   723 K – 1 mm

773 K – 1 mm   673 K – 3 mm

723 K – 3 mm   773 K – 3 mm

673 K – 5 mm   723 K – 5 mm

773 K – 5 mm   673 K – 10 mm

723 K – 10 mm   773 K – 10 mm

Figure 2 - Micrographs of the samples of different heights nitrided at temperatures of 673, 723 e 773 K using the cathodic cage method.

Table 1 - Thickness of the nitrided layers for samples of height 1, 3, 5 e 10mm, at different temperatures.

Sample Height (mm)		Temperature (K)					
		673	723	773	673	723	773
		Cathodic Cage			Conventional Plasma Nitriding		
1	Thickness (μm)	7,8	22	49,1	1,3	16	24,5
3		8,5	23	50,9	2,1	17,4	28
5		10	28	52,2	3,5	23,5	36,4
10		12	32	53,4	4,4	29,5	45,8
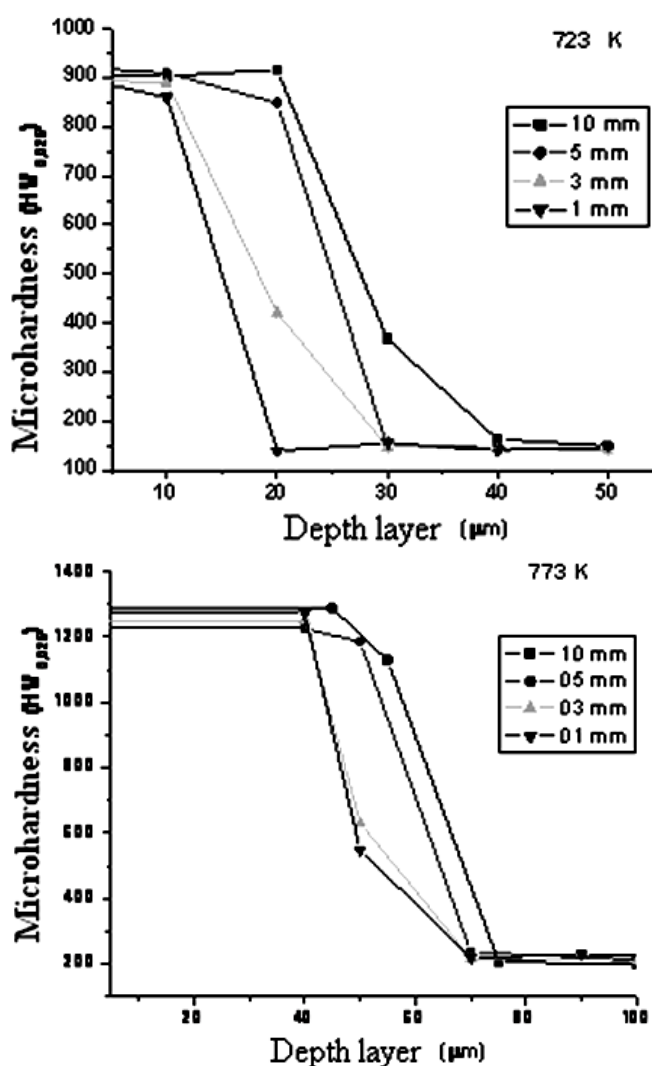
Figure 3 – Microhardnes profile as a function of the sample depth for samples treated at 723 e 773 K.

This result, allied with the uniformity in the thickness of the layer, confirms the elimination of the borders effects, a drawback observed in the conventional ionic nitriding. For the sample of height 10mm, treated at 723 K, for instance, it is observed that the microhardness begins to decrease

around a thickness of 25 μm until it reaches the value of the substrate, whereas for the sample of height 1mm such microhardness decreasing begins around 20 μm. In contrast, the samples nitrited by conventional ionic technique show a variation in the microhardness of up to 30% for samples of different heights treated at the same temperature, indicating a non uniform temperature between the surface and the base of the samples [5].

In the case of a treatment temperature of 773 K, it is observed that the microhardness begins to reduce around 50μm until reaches the microhardness of the substrate, while for the samples of height 1mm, such a microhardness decrease begins around 40μm. As the samples are electrically isolated and placed on an alumina (submitted to flotation potential) disk, radiation from the cathodic cage supplies the necessary energy to heat up uniformly the sample to the desired treatment temperature. According to Arrhenius law, given by $D=D_o \exp(-Q/RT)$, where D is the diffusion coefficient, D_o is the pre-exponential factor which is temperature independent, Q is the diffusion activation energy, R the universal gas constant and T the absolute temperature, since the nitriding atmosphere is the same for the whole set of samples, the sample temperature will depend only on the diffusion coefficient. Since the conditions are the same for the all samples, the diffusion coefficient is constant for all samples during nitriding, and the surface temperature of all the samples is practically the same, independent of the sample height, with only a small thermal gradient between the bulk and the sample surface.

The analysis of the x-ray diffractograms shows a small variation in the phases presents in the case of samples of different heights nitrided at the same temperature. For a temperature of 673 K (figure 04) there are two dominant peaks characteristic of the S phase of ε – Fe_3N iron nitrite which is present in all of the samples analyzed. This contrasts with chrome nitrite, which is completely absent.
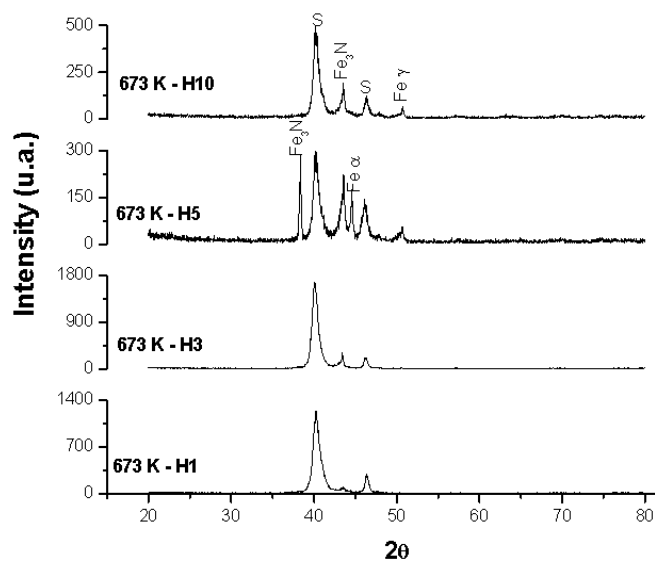

Figure 4 – X-ray spectroscopy of samples with height 1, 3, 5 and 10mm treated in 250 Pa, 80% of N2 and a temperature of 673K.

Figure 05 shows the diffractogram of the samples nitrited at 723 K. It is seen that only the sample of height 1mm presents different phases. Moreover, peaks characteristics of the expanded austenite (phase S) are once again present and γ' – Fe4N iron nitrite is beginning to form. The absence of the chrome nitrite is also observed, testifying that the precipitation of this compound occurs only at higher temperatures above 724K, leaving expanded austenite predominant. This phase has a high hardness good wear resistance and excellent corrosion resistance.
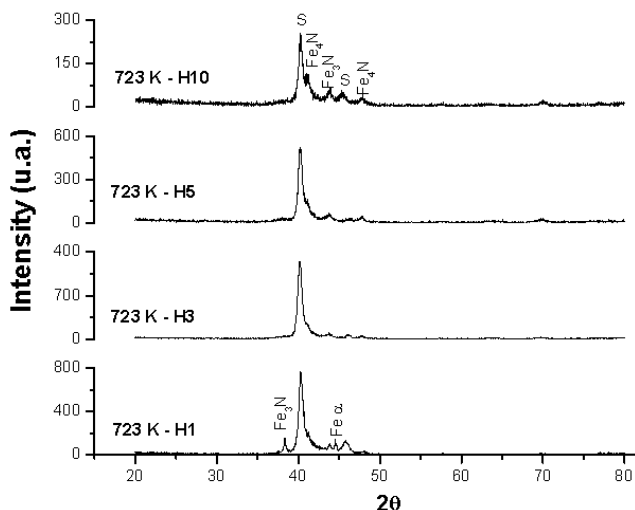

Figure 5 – X-ray spectroscopy of samples with height 1, 3, 5 and 10mm treated in 250 Pa, 80% of N2 and a temperature of 723K.

For the samples treated 773 K (figure 6) we found a reduction in the phase S with consequent appearance of CrN and γ'–Fe4N, what confirm that for temperatures higher than 723 K the phase S (expanded austenite) is decomposed having as products CrN and γ' – Fe4N.
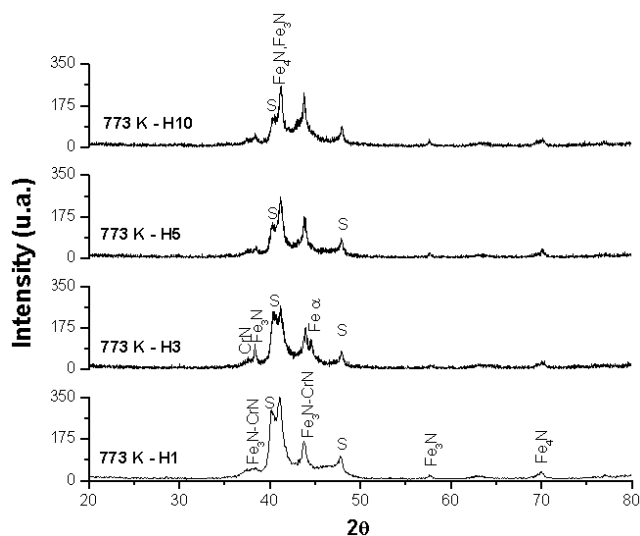

Figure 6 – X-ray spectroscopy of samples with height 1, 3, 5 and 10mm treated in 250 Pa, 80% of N2 and temperature of 773K.

4. CONCLUSIONS

In the analysis of the x-ray difractograms of austenite stainless steel AISI 316 samples, nitrided through the cathodic cage technique the phases present are those usually present when treated by conventional plasma nitriding [13], detaching the phase S (expanded austenite), which shows a small variation in relation to the height of the samples. However, there is not a great temperature variation inside of the cathodic cage, as seen from the small variation in the thicknesses of the layers for samples of different heights nitrided at the same temperature, as is confirmed through the micrography and also a small microhardness variation. The cathodic cage nitriding technique produces layers of microhardness compatible with those produced by conventional plasma techniques, but with superior and more uniform thicknesses, and without the problems associated with the conventional nittriding, such as the well known border effect [14].

REFERENCES

1. ALVES JR., C. *Nitretação a Plasma: Fundamentos e Aplicações.* Editora da UFRN, Natal, 2001.
2. LI, C.X.; GEORGES, J.; LI, X.Y., *Surface Engineer*ing 18 (2002) 453.
3. LI, C.X.; BELL, T., *Wear* 256 (2004) 1144.
4. LI, C.X.; BELL, T., *Corrosion Science* 46 (2004) 1527.
5. ALVES JR., C.; SILVA, E.F.; MARTINELLI, A.E., *Surface and coating Technology* 139 (2001) 1.
6. SOUSA, R.S., *Influência da Geometria das Peças e Parâmetros do Processo sobre as Características da Camada Nitretada por Plasma.* Tese de Doutorado (2005). Universidade Federal do Rio Grande do Norte, Natal (RN).
7. THORWART, G.; MANDL, S.; RAMCHEMBACK, B., *Surface and Coating Technology* 128 (2000) 116.
8. ALVES JR., C.; RODRIGUES, J.A.; MARTINELLI, A.E., *Surface Technology* 122 (1999) 112.
9. ALVES Jr., C.; ARAÚJO, F.O.; RIBEIRO, K.J.B.; COSTA, J.A.P.; SOUSA, R. R. M.; SOUSA, R.S., *Surface & Coatings Technology* 201 (2006) 2450.
10. LI, C.X.; BELL, T.; DONG, H., *Surface Engineering* 18 (2002) 174.
11. GEORGE, J., *Heat Treatment of Metals* 28 (2001) 33.
12. LI, C.X.; BELL, *Heat Treatment of Metals* 1 (2003) 1.
13. SUN, Y.; LI, C.X.; BELL, T., *Journal of Materials Science* 34 (1999) 4793.
14. SOUSA, R.R.M.; ARAÚJO, F.O.; RIBEIRO, K.J.B.; MENDES, M.W.D.; COSTA, J.A.P.; ALVES JR., C., *Materials Science and Engineering A* 465 (2007) 223.