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Corrosion behavior of low energy, high temperature nitrogen ion-implanted AISI 304 stainless steel

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Abstract. This work presents the results of a low-energy nitrogen ion implantation of AISI 304 type stainless steel (SS) at a moderate temperature of about 500°C. The nitrogen ions are extracted from a Kauffman-type ion source at an energy of 30 keV, and ion current density of 100 μA cm^{-2}. Nitrogen ion concentration of 6×10^{17}, 8×10^{17} and 10^{18} ions cm^{-2}, were selected for our study. The X-ray diffraction results show the formation of CrN polycrystalline phase after nitrogen bombardment and a change of crystallinity due to the change in nitrogen ion concentration. The secondary ion mass spectrometry (SIMS) results show the formation of CrN phases too. Corrosion test has shown that corrosion resistance is enhanced by increasing nitrogen ion concentration.

Keywords. Nitrogen implantation; stainless steel; X-ray diffraction; corrosion.

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1. Introduction

Ion implantation has been used to modify the physical and chemical properties of a wide range of metals and super-alloys using different plasma techniques for ion sources and plasma surface treatment [1,2]. High-dose ion implantation is also a well-known technique to improve the mechanical properties of metals [3–6]. There have been a number of investigations about increasing microhardness, wear resistance and finally improvement of the corrosion resistance [7–9]. The AISI 304-type stainless steel (SS) which contains high chromium content is a commonly used engineering material mainly because of its corrosion resistance. High-dose ion implantation is a possible solution for producing nitride phase layers in a 304 type

Table 1. Chemical composition of AISI 304 steel (wt%) determinated by EDX analysis.

C*	Fe	Cr	Ni	Ma
0.08	71.06	20.12	7.84	0.89

Table 2. The process parameter during implantation.

Sample	1	2	3
Energy (keV)	30	30	30
Ion current (μA/cm^2)	100	100	100
Dose (ions/cm^2)	6×10^{17}	8×10^{17}	1×10^{18}
Time (s)	960	1280	1600
Temperature (°C)	500	500	500

SS. New phases or structures can be made enhancing the surface properties of the treated materials.

The microstructure and phase composition of ion-implanted surface layers are controlled by the implantation parameters (ion energy, dose rate and processing temperature), which can be maintained easily and precisely. This control was typically performed at high temperatures, high doses and different energies under high vacuum conditions.

For the treatment of stainless steel materials, it is important to improve the surface mechanical properties without degrading the corrosion resistance. Therefore, in this work, we have focused on the effects of ion implantation treatment on the corrosion resistance of the samples.

2. Materials and experimental techniques

The AISI 304 steel samples of 6 mm × 18 mm × 0.3 mm were ultrasonically cleaned using alcohol and acetone the implantation of samples was performed using the implanter of Plasma Physics Research Center of I.A. University, which could generate gas ions with maximum 30 keV and elliptic beam size 110×250 mm. Table 1 shows the chemical composition of the AISI 304 steel sample surface which is determined by using scanning electron microscopy (LEO440I) (SEM) that is equipped with energy dispersive X-ray (EDX). The given values are the average of the concentrations measured at four different points of the samples.

The implantation parameters are summarized in table 2. The background pressure of 4.8×10^{-6} Torr decreases to 1.6×10^{-5} Torr during implantation. X-ray diffraction patterns were recorded at room temperature by the X-ray (SISERTE Model 3003 PTS) diffractometer using Ni filtered CuKα1 radiation and also in the analysis the step size was 0.05°/s.

X-ray diffraction analysis was carried out to study microstructural changes and new phase formation induced by the ion implantation. The depth profiles

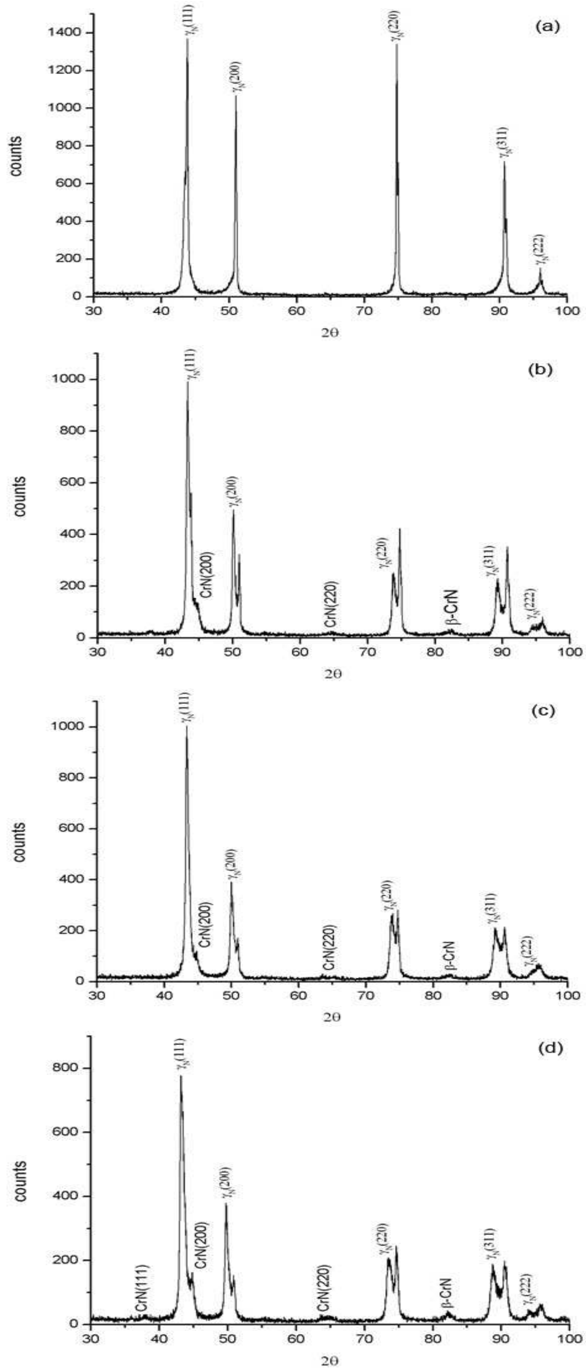

Figure 1. XRD patterns for (**a**) untreated sample and different doses of (**b**) 6×10^{17} ion/cm^2, (**c**) 8×10^{17} ion/cm^2 and (**d**) 1×10^{18} ion/cm^2.

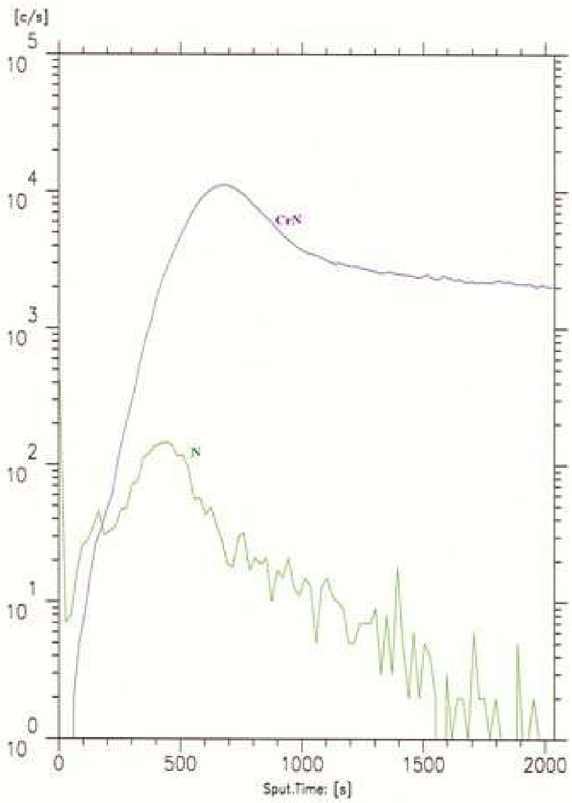

Figure 2. SIMS depth profile of nitrogen and CrN corresponding to the dose of 8×10^{17} ion/cm^2.

of different samples were performed by secondary ion mass spectrometry (SIMS) technique by means of Cameca IMS 6F instrument. The Cs^+ ions are used as primary ions and negative secondary nitrogen ions have been detected. In SIMS analysis the primary ions at 15 keV energy and 2 nA ion current were scanned on 150 $\times$150 μm^2 sample surface.

The potentiodynamic polarization test was performed in a 3 wt% NaCl solution made of analytical grade reagents and distilled water. The corrosion current was calculated using softcorr III code. The scanning rate was 0.5 mV min^{-1}.

3. Results and discussion

XRD patterns of the ion-implanted and untreated samples are shown in figure 1. From figure 1a, it is clear that the untreated sample presents a single phase γ-Fe with sharp peaks which are typical of annealed materials. From figures 1b, c and d, it can be seen that for the ion-implanted samples, all peaks were shifted to lower angles and were broadened considerably. The shift and broadening of the peaks are

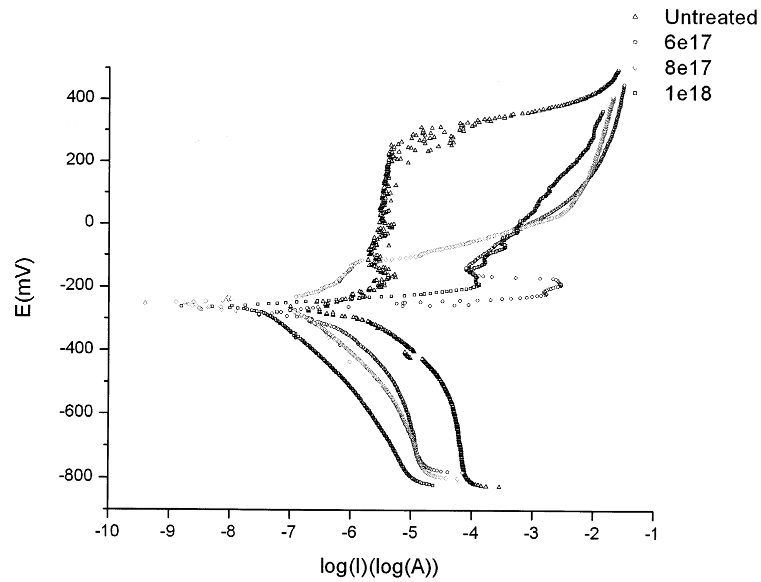

Figure 3. Potentiodynamic polarization curves acquired from the AISI 304 in 3 wt% NaCl solution for different doses.

associated with the 'expanded austenite' (γ_N) phase [10,11] produced by nitrogen supersaturation and associated stress caused by the nitrogen remaining in solid solution in the fcc lattice. Also it can be concluded that the ion-implanted CrN samples produced at 500°C is composed mainly of the CrN phases and the degree of crystallinity change depended on the N doses. It is suggested that CrN would be precipitated at a temperature of 500°C [12]. It should be noted that although the X-ray penetration depth is estimated to be approximately 2.5 μm under our diffraction condition, XRD results can be representative of the whole layers due to the homogeneity of γ phase's constituents of the layers seen from figure 1.

The SIMS results in figure 2 show the depth profile of nitrogen and CrN of 8×10^{17} ions/cm^2 implanted sample. The CrN depth profile presents nearly a Gaussian distribution throughout the bulk, according to the nitrogen profile. It can also be observed from figure 2 that the maximum nitrogen penetration is lower than that of CrN. We suppose that the nitrogen diffusion to the bulk was disturbed by CrN phase formation which acts as a diffusion barrier.

The results of the electrochemical corrosion measurement for four samples are plotted in figure 3. All the treated samples show improved corrosion resistance, a low corrosion current at the equilibrium potential, as well as a very low corrosion current. For example, at -260 mV, the corrosion current is 2.436 μA for the untreated sample, but this current diminishes to about 885.1, 43.75, 31.8 nA for implanted samples at 6×10^{17}, 8×10^{17}, 1×10^{18} nitrogen ions cm^{-2} respectively. Corrosion tests were performed at 0.5 cm^{-2} areas.

4. Conclusions

It is well-known that the corrosion introduced by nitrogen depends on the steel composition and especially on Cr contents has a suitable amount of Cr on its surface to enhance the corrosion resistance of AISI 304. As is well-known, anticorrosive effect can be obtained when Cr is in excess of 20% in an iron matrix.

Electrochemical tests showed that the ion implantation prompted the formation of a stable passive layer probably due to the presence of CrN and $\gamma_{\rm N}$ phases. There was no difference in chemical composition of the AISI 304 after and before implantation.

There were beneficial effects by the treatment of corrosion resistance. The ion implantation had no negative effect on the corrosion resistance of the AISI 304.

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