

CORROSION RESISTANCE OF NITRIDED LAYERS PRODUCED ON AISI 316L AUSTENITIC STAINLESS STEEL UNDER GLOW-DISCHARGE CONDITIONS

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Abstract: Glow discharge nitriding of AISI 316L austenitic stainless steel allows producing surface layers that improve significantly the properties of the steel, in particular its hardness and frictional wear resistance. The present studies are concentrated on the influence of the glow discharge nitriding temperature on the microstructure, phase composition and corrosion resistance of AISI 316L steel. The results indicate that the increase of the glow discharge nitriding temperature results in decreased corrosion resistance of nitrided AISI 316L austenitic steel which is due to the formation of Fe₄N and CrN nitrides in the external zone of the layer.

Keywords: austenitic stainless steel, glow-discharge nitriding, corrosion resistance.

1. Introduction

AISI 316L austenitic steel intended for medicine has a precisely specified chemical composition so as to ensure its high corrosion resistance, good biocompatibility and advantageous mechanical properties. The high corrosion resistance of this steel is associated with its ability to passivation, similar to that of chromium, which guarantees that an oxide film will form on its surface [1, 2]. Research studies conducted with the aim to improve the mechanical properties of austenitic steels have been developing along two lines. One consists of introducing nitrogen as an alloying constituent during the metallurgical process (e.g. of the AISI 304LN steel type). The other technology employs surface treatments, such as, e.g., glow discharge assisted nitriding [3-7], which increases the hardness, frictional resistance, fatigue strength and tensile strength of the steel [4, 8]. It should however be noted that when the glow discharge nitriding is conducted at a temperature above 460°C, chromium nitride precipitates at the grain boundaries, which degrades considerably the corrosion resistance of the steel [3, 8, 10-12].

2. Materials and experimental methods

Samples made of AISI 316L steel were subjected to glow discharge assisted nitriding at temperatures of 450°C and 460°C. The microstructure of the nitrided layers was examined in a Hitachi S-3500N scanning electron microscope. Metallographic cross-sections were etched in a reagent with the composition: 50 % HCl, 25 % HNO and 25 % H₂O. The microhardness (HV0.05) was measured using a Zwick Materoalprufung 3212002 microhardnessmeter. The surface roughness was examined in a Taylor Hobson Form Talysurf scanning profilometer (Series 2). The phase composition was determined with a Bruker D8 Discover X-ray diffractometer using CuK α radiation incident at various angles. The corrosion resistance was measured by the potentiodynamic method and impedance method in an aqueous 0.5 M NaCl solution using an Atlas-Solich measuring kit. The potential variation rate was 10 mV/s, except within the corrosion region where it was 5 mV/s. The potential was measured with respect to a saturated calomel electrode. In the case of the impedance method, the measurements were made at a corrosion potential with the frequency ranging from 10⁻³ Hz to 10⁵ Hz. The spectra obtained were analyzed using the 3.0 Boukamp computer program. The results are shown in the form of the Bode diagrams.

3. Results and discussion

The glow discharge assisted nitriding of AISI 316L steel yielded surface layers about 5 μ m thick. The microstructures of the layers are shown in Fig. 1. The nitrided surface layers considerably increased the microhardness of AISI 316L steel, namely from 230 HV0.05 to about 1180 HV0.05 in the layers nitrided at 450°C and to about 1200HV0.05 in those nitrided at 460°C.

Table 1 gives the roughness parameters of the AISI 316L steel surface after subjecting it to glow discharge nitriding. We can see that the slight increase of the nitriding temperature resulted in a significant increase of the surface roughness.

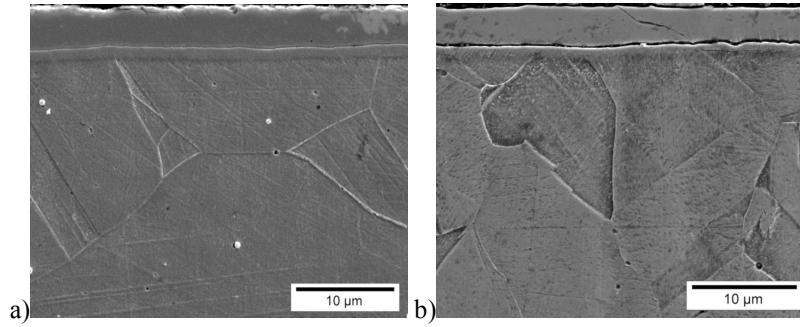


Figure 1. Microstructures of the surface layers produced on AISI 316L steel by glow discharge nitriding at a temperature of 450°C (a) and 460°C (b)

Table 1. Stereometric parameters characterizing the surface topography of AISI 316L steel surface in the starting state and after glow discharge nitriding at temperatures of 450°C and 460°C

parameter	initial state [μm]	nitriding at 450°C [μm]	nitriding at 460°C [μm]
R_a	0,0591	0,0939	0,135
R_q	0,077	0,12	0,207
R_p	0,965	0,546	5,61
R_v	0,53	0,907	1,1
R_t	1,5	1,45	6,71
R_z	1,12	1,28	4,25

R_a - mean arithmetic deviation of the roughness profile, R_q – mean square deviation of the roughness profile, R_p – maximum height of the roughness profile peak, R_v – maximum depth of the roughness profile valley, R_t – maximum height of the roughness peak ($R_t=R_p=R_v$), R_z – mean difference between the five highest peaks and the five deepest valleys of the roughness profile (acc. to ISO 4287/1-1984)

An analysis of the phase composition (Fig. 2) identified the expanded austenite, known as the S-phase, present in the nitrided layers of both types. The layer nitrided at 450°C had a surface zone of chromium nitride (CrN) that extended to a depth of 0.5 μm . The phases found in the layer nitrided at 460°C were iron nitride (Fe_4N) at a depth of 1 μm and chromium nitrides CrN and Cr_2N formed at depths down from about 1.5 μm .

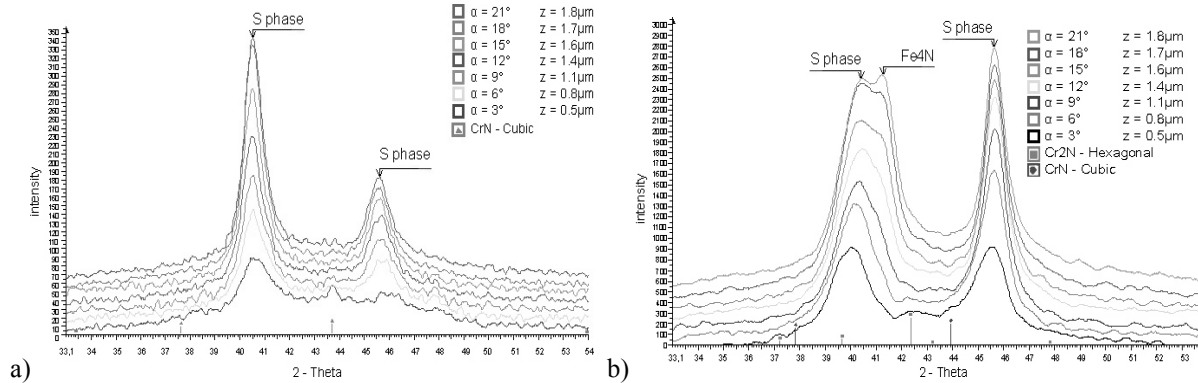


Figure 2. Diffraction patterns of the sample surfaces nitrided at 450°C (a) and 460°C (b) obtained with $\text{CuK}\alpha$ radiation striking the surface at various angles

Figure 3 shows the impedance spectra obtained for AISI 316L steel in the starting state and after glow discharge nitriding at temperatures of 450°C and 460°C. Table 2 gives the electrochemical properties of the steel in these three states. All the impedance spectra have a single time constant: a single peak occurring within the frequency range from 0.01 to 100 Hz in the untreated steel and the steel nitrided at 450°C, and within the narrower frequency range from 0.1 to 100 Hz in the steel nitrided at 460°C. This means that all the steel samples are in the passive state. The decrease of the phase angle observed in the low and high frequency regions probably indicates local corrosion. The parameter α (the quantity defining the character of the double layer: $\alpha=1$ when it has a resistive character, $\alpha=0.5$ indicates its conductive character) has a similar value in the untreated steel and in the steel nitrided at 450°C, where it is equal to 0.88 and 0.89 respectively, which is evidence that the passive layers are very stable. Similarly, the high values of the electric charge transfer resistance through the double layer

($R_t=336000 \Omega\text{cm}^2$ in the non-nitrided steel, and $R_t=380000 \Omega\text{cm}^2$ in the steel nitrided at 450°C) indicate that the corrosion resistance is high. The smaller value of the parameter $\alpha=0.81$, and the lower resistance to the electric charge transfer $R_t = 5560 \Omega\text{cm}^2$ measured in the steel nitrided at 460°C reflect its lower corrosion resistance.

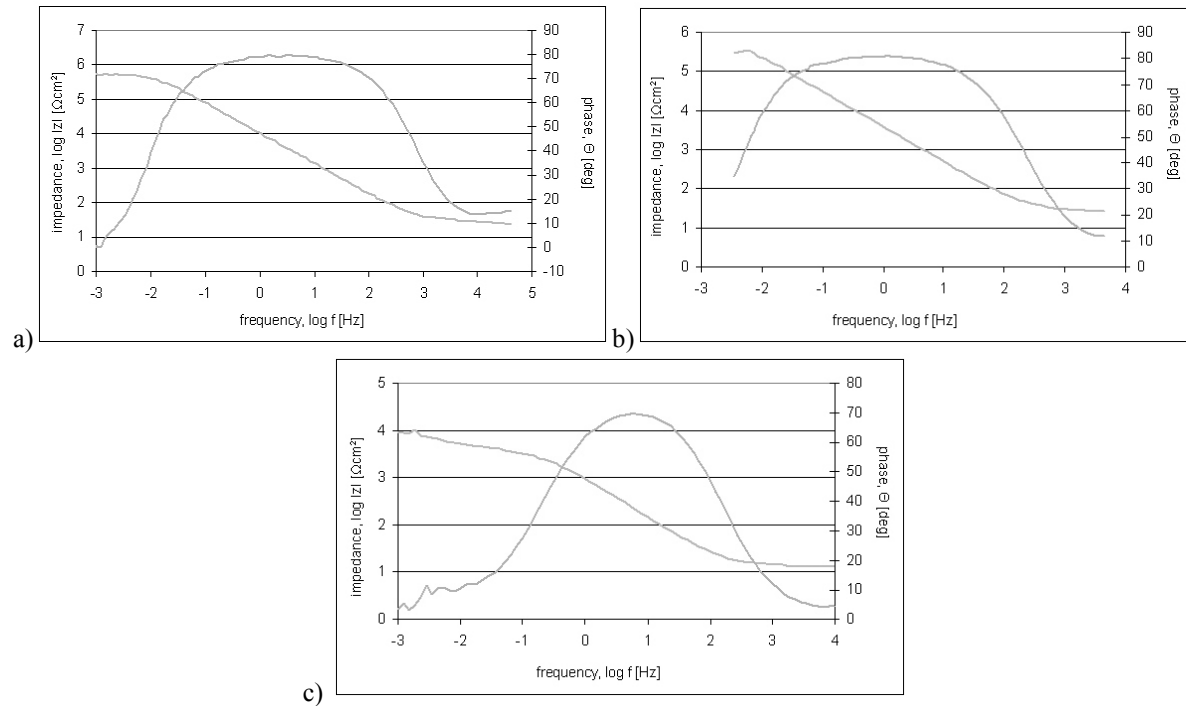


Figure 3. Impedance spectra of AISI 316L steel in the starting state (a), after glow discharge nitriding at 450°C (b) and 460°C (c)

Table 2. Characteristic electrochemical properties of AISI 316L steel in the starting state and after glow discharge assisted nitriding (impedance method)

		Resistance of the environment	Resistance of the layer
AISI 316L initial state	$R_t [\Omega\text{cm}^2]$	16,55	$3,36\text{E}+05$
	$C_{CPE} [\text{F}/\text{cm}^2]$		$8,21\text{E}-06$
	α_{CPE}		0,8827
AISI 316L nitriding at 450°C	$R_t [\Omega\text{cm}^2]$	16,04	$3,80\text{E}+05$
	$C_{CPE} [\text{F}/\text{cm}^2]$		$2,60\text{E}-05$
	α_{CPE}		0,8916
AISI 316L nitriding at 460°C	$R_t [\Omega\text{cm}^2]$	12,97	$5,56\text{E}+03$
	$C_{CPE} [\text{F}/\text{cm}^2]$		$3,548\text{E}-05$
	α_{CPE}		0,8141

The results obtained by the potentiodynamic method agree with the impedance results (Fig. 4). The nitriding at a temperature of 450°C increases the corrosion resistance of the AISI 316L steel samples: the current density decreases, whereas the corrosion potential increases. In the layer produced at 460°C , the corrosion potential is shifted towards negative values, with the current density values remaining unchanged. Hence, an increase of the nitriding temperature to 460°C slightly decreases the corrosion resistance of the steel.

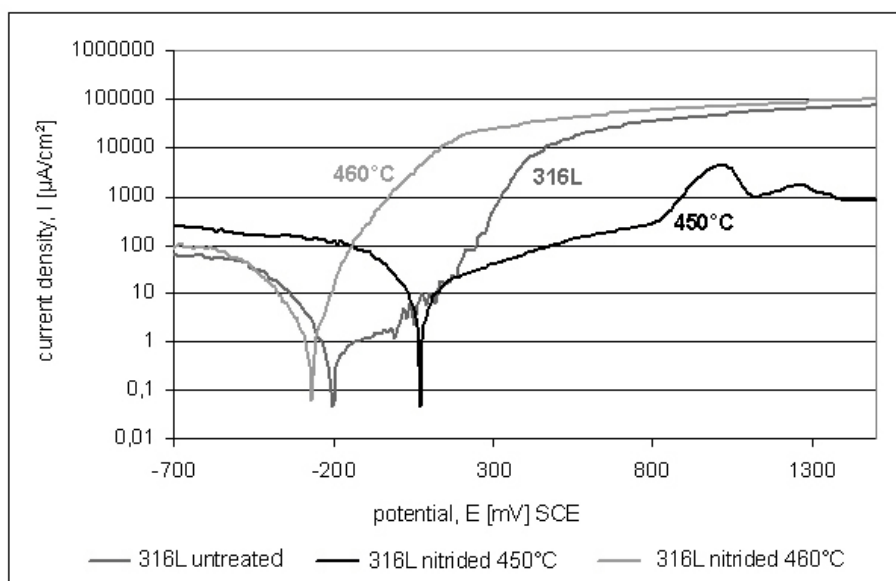


Figure 4. Polarization curves measured for AISI 16L steel in the starting state and after glow discharge nitriding at 450°C and 460°C

Table 3. Characteristic electrochemical properties of AISI 316L steel in the starting state and after glow discharge assisted nitriding (potentiodynamic method)

	current density i [$\mu\text{A}/\text{cm}^2$]	corrosion potential E [mV]
AISI 316L initial state	13,5	-204
AISI 316L nitriding at 450°C	10	73,1
AISI 316L nitriding at 460°C	10,4	-276

4. Conclusions

Glow discharge assisted nitriding applied to AISI 316L steel yields diffusion surface layers of high surface hardness with the possibility of modifying their thickness, phase composition and surface roughness depending on the nitriding process temperature. The best corrosion resistance was achieved with the nitrided layers forms during glow discharge nitriding at a temperature of 450°C, which is composed of expanded austenite, known as the S-phase, and a thin surface zone of chromium nitride (CrN). The increase of the nitriding temperature to 460°C results in chromium nitride and iron nitride being formed leading to a decrease of the corrosion resistance.

5. References

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