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SHORT-TERM LOW-TEMPERATURE GLOW DISCHARGE NITRIDING OF 316L AUSTENITIC STEEL

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The AISI 316L austenitic steel after glow discharge nitriding at temperature of $T = 673$ K and duration of $\tau = 14,4$ ks, for two different variants of specimen arrangement in the glow-discharge chamber was investigated. In order to assess the effectiveness of nitriding process, the surface layers profile analysis examination, surface hardness and hardness profile examination, the analysis of surface layer structures and corrosion resistance tests were performed. It has been found that application of a booster screen effects in a nitrogen diffusion depth increment into the 316L austenitic steel surface, what results in the surface layer thickness escalation.

Key words: austenitic stainless steel, glow discharge nitriding, surface layer, hardness

Kratkotrajno niskotemperaturno nitiranje austenitnog čelika 316L. Ispitani su uzorci od AISI 316L austenitni čelik tretirani na dva različita načina u komori za nitiranje nakon nitiranja na temperaturi $T=673$ K u trajanju $\tau=14,4$ s. Kako bi procijenili efikasnost procesa nitiranja provedena je analiza profila površinskog sloja, ispitana je površinska tvrdoća kao i tvrdoća profila, te analiza strukture površinskog sloja i ispitivanje korozijske otpornosti. Pronađena je primjena efekta pojačane podrške u povećanju dubine difuzije kod nitiranja u površinu austenitnog čelika 316L, što rezultira povećanje debljine površinskog sloja.

Ključne riječi: austenitni čelici, nitiranje, površinski sloj, tvrdoće

INTRODUCTION

Owing to good corrosion resistance, mechanical strength, heat resistance and high formability, chromium-nickel austenitic steels have found application in a number of the industry branches. One of their numerous applications is biomedical industry. Austenitic steels are used for inter alia, medical instruments and various types of orthopaedic and dental implants fabrication [1,2]. For these purposes austenitic steels type 18-8 and 17-12-2L are oftentimes used. They have similar mechanical properties, however 17-12-2L steel has higher pitting and crevice corrosion resistance owing to its higher nickel content and 2 % molybdenum addition, which in combination with chromium, stabilizes the passive oxides film in the presence of chlorides [3]. Besides to good corrosion resistance, materials used for implants require high abrasive wear resistance. Unfortunately, due to austenitic stainless steels low hardness and low tribological resistance, accelerated abrasive wear of the materials between the head and the acetabulum in a hip joint implant was observed [4,5]. Modern methods for preventing this adverse phenomenon are based on the surface engineering field [6,7]. Method commonly used for material surface modification is nitriding [8]. However, nitriding of high-chromium steels encounters lot of problems due to oxides film existence on the steel surface, which brings the nitriding

process about difficult or almost impossible. In practice, this problem is solved by surface pre-treatment, e.g. etching and phosphatizing, introducing additives such as ammonium chloride or hydrochloric acid to the reaction chamber or by using various treatments e.g. plasma treatment and finally by applying preliminary cathode sputtering under glow discharge conditions with gas nitriding afterwards [9]. Nitriding method, which eliminates the necessity of an expensive surface pre-treatment operations is glow discharge nitriding. The cathode sputtering of a passive chromium oxide film during material heating gives the possibility to realize the austenitic steel nitriding as a single process [10]. This investigation describes the effect of glow discharge nitriding, on the 316L austenitic steel surface layer properties.

MATERIALS AND METHODS

Glow discharge nitriding of chromium-nickel-molybdenum austenitic steel AISI 316L was performed in a JON-600 glow discharge treatment device with cooled anode, according to the following parameters: atmosphere composition 75 % H_2 + 25 % N_2 vol.; pressure $p = 150$ Pa; temperature $T = 673$ K; duration $\tau = 14,4$ ks.

Two variants of specimen arrangement inside the glow-discharge chamber were investigated:

- variant 1: specimens were positioned directly on the cathode,

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- variant 2: specimens were placed on the cathode and covered with a booster screen (perforated stainless steel sheet)

Considering first variant, a specimens surfaces were ion bombarded with energies resulting directly from the cathode potential drop (several hundreds volts), whereas in the second variant, near the surface layer area, high charged potential peaks, which interact with the nitrogen atoms were observed. High charged potential peaks existence lead to ions acceleration which corresponds to energy of several hundreds electron volts. Ions were implanted into the material surface layer generating highly nitrogen saturated non-equilibrium zone, which affects nitrogen diffusion into the material increment. Considering high concentration gradients, it was observed that diffusion at the first stage proceeds not through the grain boundaries, what effects in more homogeneous nitrogen layers forming. Example of the surface layer potential osciloscopic analysis of samples placed on the cathode and covered with a booster screen is shown in the Figure 1.

Nitrided layers microhardness and microhardness profiles were measured by Knopp method on a Future Tech FM7 hardness tester. Surface microhardness measurements were performed for different loads: 25 G, 50 G and 100 G, while surface layer microhardness profiles for 10 G load.

X-ray phase analysis was made on a DRON-2 type X-ray diffractometer using filtered cobalt anode tube radiation of average wavelength $\lambda_{CoK_{\alpha av.}} = 0,17902$ nm. Element distribution analysis was performed on a GDS GD PROFILER HR glow-discharge optical emission spectrometer with a Grimm discharge tube with $\varnothing 4$ mm cathode. Corrosion resistance tests were performed using AMEL 7050 potentiostat and an electrochemical test chamber. A platinum wire mesh was used as the counter electrode, while saturated calomel electrode (SCE) as a reference electrode. The corrosion medium was $0,5 \text{ mol/dm}^3$ NaCl water solution.

RESULTS AND DISCUSSION

Microhardness obtained from cross sections of cathode nitrided samples was three to four times higher com-

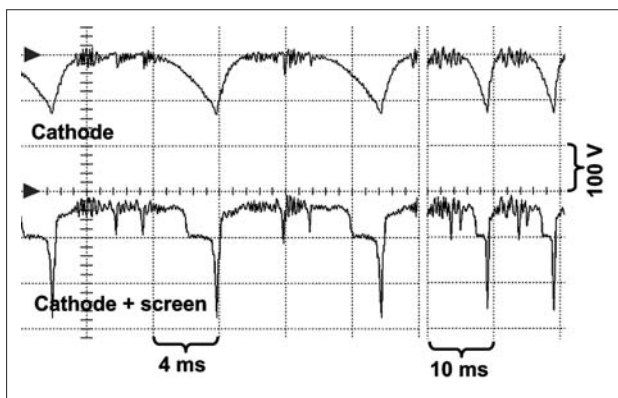


Figure 1 Potential osciloscopic analysis performed for sample cathode area.

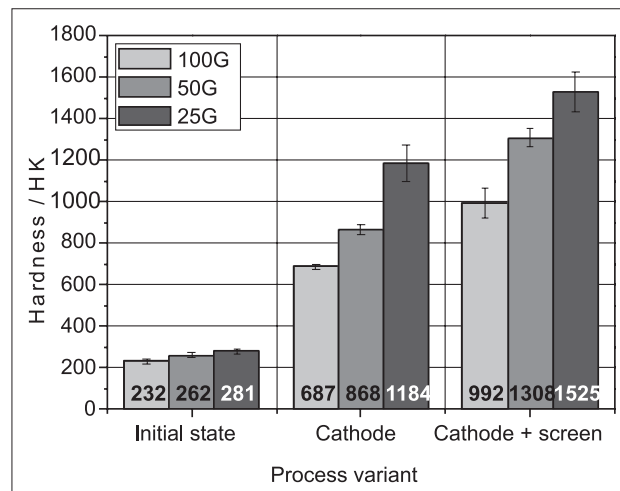


Figure 2 Microhardness tests results of 316L austenitic steel surface after glow discharge nitriding.

paring to the material initial state. Application of the booster screen caused surface layer increment even four to five times. Surface layer hardness drops present when hardness test load increase can be explained by penetration distance increment so obtained results are nitrogen lower concentration zones resultant hardness. Microhardness test results of 316L steel after glow discharge nitriding are shown in the Figure 2.

Element distribution profiles of 316L steel surface layer (Figures 3) show that short-term glow discharge nitriding causes a $2 \mu\text{m}$ distance nitrogen diffusion. Booster screen application increases nitrogen diffusion into the surface three times ($6 \mu\text{m}$), comparing to the respective nitriding variants without booster screens. Nitrogen concentration in the surface layer for both nitriding variants of specimen arrangement in the chamber was nearly the same. When analyzing the concentration of nitrogen to the distance from the nitrided surface face, in the variants of the cathode nitriding both with and without booster screen, nitrogen concentration at the distance of several micrometers from the nitrided specimen face stabilizes at a certain level. Considering the chromium to nitrogen atoms concentration ratio, it amounts approx. 2:1 (Table 1), which corresponds to A_2B compound type. That might suggest the obtained nitrided layers are built from precipitates of Cr_2N . However chromium to nitrogen atoms ratio at the material face was approx 1:1, what might suggest of CrN presence. In transient zone of nitrided layer and substrate, nitrogen concentration decrease below nitrides forming boundary, what effects in nitrogen-saturated austenite (*expanded austenite*) presence [11].

X-ray diffraction analysis (fig. 4) of 316L steel surface layer after glow discharge nitriding considerably confirms the elements profiles analysis. Analyzing the cathode nitrided samples surface diffractograms, CrN and Cr_2N phases peaks were noticed. Extra peaks in the transient zone (nitrogen-supersaturated austenite \tilde{a}_N) show that thickness of nitrides formation zone decrease.

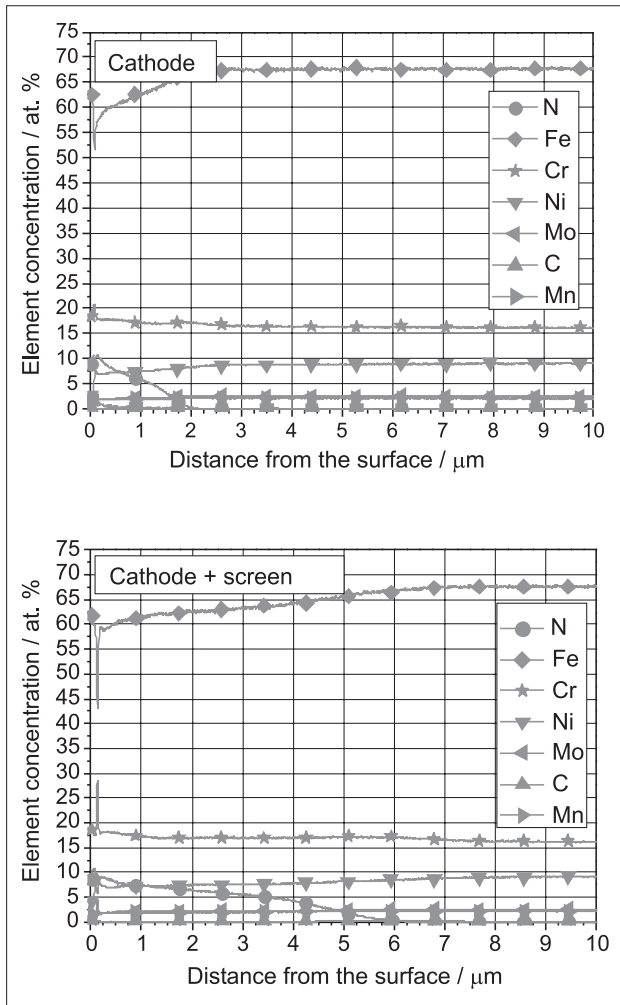


Figure 3 The depth profile of elements in the surface layer after glow discharge nitriding of the 316L.

Table 1 Nitrogen concentration and diffusion depth of 316L steel after short term glow discharge nitriding

Process variant	Nitrogen diffusion depth / μm	Cr : N ratio on the surface	Cr : N ratio on the surface layer
Cathode	2,1	10,1 : 9,2 (1,1 : 1)	17,5 : 8,1 (2,1 : 1)
Cathode + screen	6,1	12,5 : 10,5 (1,2 : 1)	17,8 : 8,5 (2,1 : 1)

Glow discharge nitriding using a booster result in nitrides zones increment, therefore nitrides phases peaks are generally noticed in the surface layer diffractograms.

Microstructures of 316L steel after glow discharge nitriding (Figure 5) show, that both variants of short-term glow discharge nitriding result in formation of tight, uniform layer with a characteristic zonal structure which forms as an effect of nitrogen concentration decrement within the surface layer. Structures comparison gives conclusions that the application of a booster screen lead to surface layer thickness increment and influence on layer properties improvement.

Surface layer hardness profiles (Figure 6) correlate with the profile analysis of the nitrogen diffusion depth

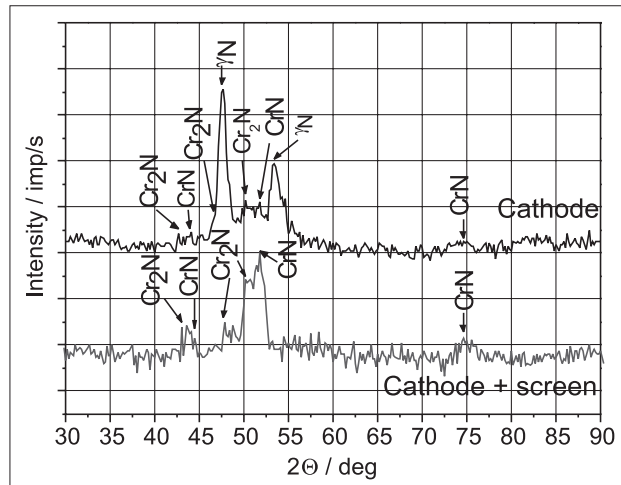


Figure 4 X-ray diffractograms of the 316L stainless steel after glow discharge.

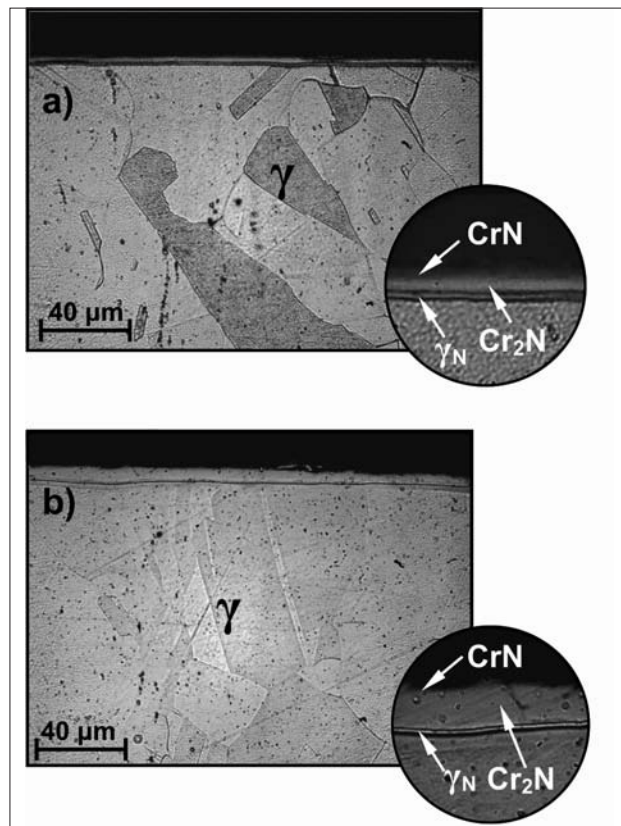


Figure 5 Microstructures of 316L steel surface layer after short-term glow discharge nitriding: a) cathode, b) cathode + screen.

into layer. Material hardness decrease with distance from the surface, what causes nitrogen concentration decrease in the surface layer and zonal structure of nitrided layers. Additionally, hardness profiles confirm advantageous effect of booster screen application on the surface layer properties.

Potentiodynamic experiments (Figure 7) results show, that nitriding process parameter set affects on potentiodynamic curves shift to the minus potential comparing to the initial state. Corrosive current after nitriding on the cathode was nearly the same to material

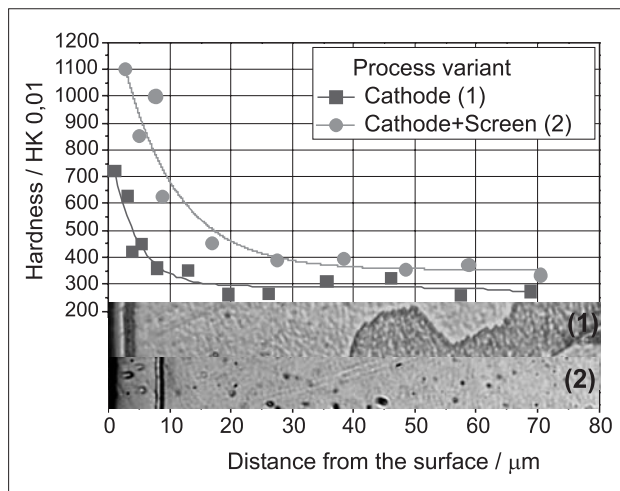


Figure 6 Microhardness profiles of 316L steel surface layer after different variants of glow discharge nitriding.

initial state, whilst after booster screen nitriding corrosive current increased. Potentiodynamic curves were in the passive range, which proofs material from the local pitting corrosion. When analyzing macrostructure after corrosion tests (Figure 8), deep great pinholes were observed on the initial material surface. After cathode nitriding amount of pinholes increased, however pins repassivation resulted in their depth and size decrement what was observed on the potentiodynamic curves as a passive range peak. Local pitting corrosion was not observed on the material surface after booster screen nitriding, whereas change of corrosion characteristic (pitting corrosion to uniform corrosion) was observed.

SUMMARY

1. Proper short-term glow discharge nitriding conditions preset (atmosphere composition, pressure, time, temperature) results in formation of the tight, uniform nitrided layers.
2. Application of a booster screen effects in three-fold surface layer increment.
3. Each nitriding parameter causes increment of nitrided steel surface layers. Surface layer after cathode nitriding was $3 \div 4$ times higher comparing to material initial state.
4. Surface layers formed after glow discharge nitriding exhibit zonal structure of CrN , Cr_2N and expanded austenite γ_{N} transient zone.
5. Short-term glow discharge nitriding according to set conditions proofs nitrided material for a local pitting corrosion.

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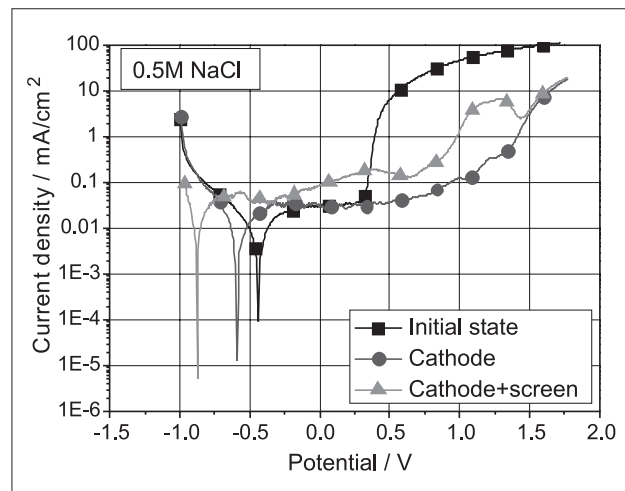


Figure 7 Potentiodynamic curves of nitrided 316L stainless steel

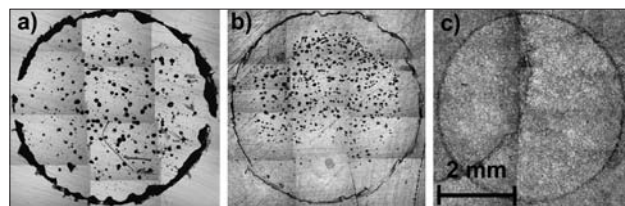


Figure 8 Macrostructure of glow discharge nitrided 316L steel surface after corrosion test: a) initial state, b) cathode, c) cathode + screen

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