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ASSESSMENT OF THE EFFECTIVENESS OF 316L AUSTENITIC STEEL UNCONVENTIONAL GLOW DISCHARGE NITRIDING

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The 316L grade austenitic steel after glow discharge nitriding at a temperature of T=733 K for a duration of τ = 61,2 ks and four different variants of specimen arrangement in the glow-discharge chamber was investigated. In order to assess the effectiveness of nitriding process variants, the profile analysis examination of obtained surface layers, surface hardness tests and surface layer hardness profile examination, the analysis of surface layer structures and abrasive wear were performed. It has been found that an application of the booster screens causes depth increase of the nitrogen diffusion into the nitrided 316L austenitic steel and thus an increase of the obtained surface layers thickness.

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

Procjena učinkovitosti nekonvencionalnog nitriranja austenitnog čelika 316L. Istraživani su uzorci austenitnog čelika stupnja 316L sa 4 različita postupka u komori za nitriranje na temperaturi T=733 K u trajanju τ=61,2 ks. Za procjenu učinkovitosti varijanti nitriranja provedena je analiza profila površinskog sloja, njegove tvrdoće kao i tvrdoće profila, površinske strukture te abrazijsko trošenje. Ustanovljeni su uzroci pojačane podrške i povećanje dubine difuzije u površini austenitnog čelika 316L pri nitriranju, te povećanje debljine nitriranog površinskog sloja..

Ključne riječi: austenitni čelici, nitriranje, površinski sloj, tvrdoća

INTRODUCTION

Owing to their good corrosion resistance, mechanical strength, heat resistance and easy formability, chromium-nickel austenitic steels have found an application in the number of industry branches. One of their numerous applications is the biomedical industry. They are used for production, various types of orthopaedic and dental implants, *inter alia* and medical instrumentation [1]. Austenitic steels types 18-8 and 17-12-2L are most often used for these purposes. They have similar properties, however the 17-12-2L steel has higher resistance to pitting and crevice corrosion owing to its higher nickel content and the 2 % addition of molybdenum which, joining with chromium, stabilizes the passive oxide film by the chlorides presence [2].

In addition to good corrosion resistance, materials used for the implants require good abrasive wear resistance. Unfortunately, due to the low hardness and low tribological resistance of the austenitic stainless steels, accelerated abrasive wear of the material between the implant head and the hip joint acetabulum was observed [3,4]. Modern solutions of preventing this adverse phenomenon rely on surface engineering methods [5,6]. Commonly used methods involves nitriding [7]. However, nitriding of high-chromium steels encounters a lot of problems due to the existence of a tight oxide film on their surface, which make the nitriding process difficult or impossible. In practice, this difficulty can be overcome by solutions of surface pre-treatment, such as etching and phosphatizing, by introducing additives, such as ammonium chloride or HCl to the reaction chamber, by using various treatments, e.g. glow-discharge or plasma treatment, or finally by applying preliminary cathode sputtering under glow discharge conditions with following gas nitriding [8].

One of the nitriding methods, which eliminates the necessity of using expensive surface pre-treatment operations is glow discharge nitriding. The passive chromium oxide film cathode sputtering application during heating of the samples gives the possibility to performing austenitic steel nitriding as a single process [9].

This article describes the effect of unconventional glow discharge nitriding methods, with different variants of specimen arrangement in the discharge-glow chamber, on the properties of nitrided austenitic steel 316L surface layers.

MATERIALS AND METHODS

The glow discharge nitriding was performed using 316L austenitic steel specimens.

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The process parameters were as follows: atmosphere 75 % H_2 + 25 % N_2 vol.; atmosphere pressure p = 150 Pa; temperature T = 733 K; duration 61,2 ks.

Four variants of specimen arrangement in the glow-discharge chamber were used:

– specimens were positioned directly on the cathode,
– specimens were poisoned on a surface insulated from the anode and cathode, (plasma potential),
– specimens were positioned on the cathode and covered with a booster screen made from perforated stainless steel sheet,
– specimens were placed within the plasma potential and also covered with a booster screen.

The purpose of the booster screens application was to intensify the surface phenomena by the formation of so called hollow cathodes on their surface.

The microhardness of the nitrided layers was tested by Knopp method using Future Tech FM7 hardness tester.

X-ray phase analysis was performed on a DRON-2 X-ray diffractometer using filtered cobalt anode tube radiation.

The element distribution analysis was made on a GDS GD PROFILER HR, glow-discharge optical emission spectrometer.

The observation of the obtained structures was performed on the crosscut metallographic specimens, either etched or not etched, using Carl-Zeiss Jena, Axiovert 25 metallographic microscope.

The abrasive wear resistance test under dry friction conditions was carried out on a T-05 tribological tester.

RESULTS AND DISCUSSION

The surface layers obtained after nitriding using the booster screens, on the cathode and in the so called “plasma potential”, are distinguished by a five times higher surface hardness comparing to the initial state samples. Similar hardness values were obtained in case of cathode nitriding. A lower, over triple increase in hardness compared to the initial state, was achieved in case “plasma potential” nitriding using a booster screen. The lowest surface hardness values were obtained for nitriding in the “plasma potential”. Figure 1 summarizes results of the surface hardness tests for the discharge-glow nitrided 316L austenitic steel.

The phase composition analysis shows that nitriding, on the cathode and for the variants with booster screens, causes the formation of iron nitrides and CrN, Cr_2N types chromium nitrides on the austenitic steel surface. The diffractograms obtained from the examination of specimens nitrided in the plasma potential showed saturated nitrogen solution in austenite, so called expanded austenite. Examples of diffractograms are shown in Figure 2.

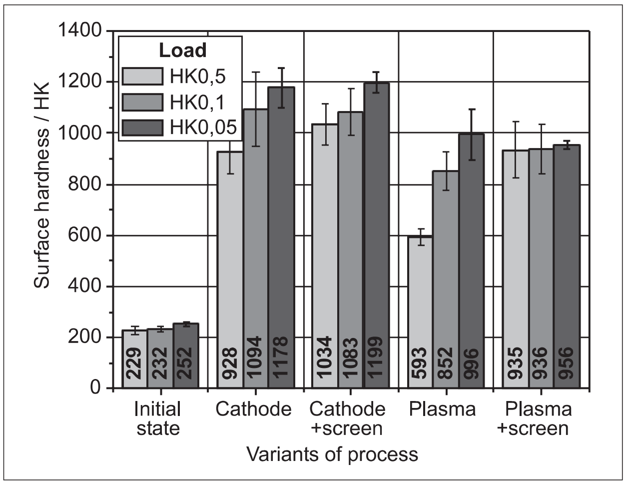

Figure 1 Surface hardness of the 316L stainless steel after glow discharge nitriding.

Table 1 **Diffusion depth and nitrogen concentration after different variants of plasma nitriding of 316L steel.**

Process variant	Nitrogen diffusion depth / μm	Max. nitrogen surface concentration / % at.	Cr:N ratio in the surface layer
Cathode	16,1	13,8	14,9 : 8,2 (1,8 : 1)
Cathode + shield	55,1	19,5	14,4 : 7,6 (1,8 : 1)
Plasma	3,0	11,5	smooth profile
Plasma + shield	41,8	10,5	14,3 : 7,7 (1,8 : 1)

Analyzing the results of 316L austenitic steel element distribution profiles in the surface layers it can be noticed that each application of the booster screens caused a greater nitrogen diffusion into the nitrided surface comparing to the respective nitriding variants without booster screens (Figure 3, Table 1). Booster screen application during nitriding specimens positioned on the cathode, increased diffusion depth into the material almost 4 times (nitrogen diffusion depth was 55,1 μm) in compare to the variant of cathode nitriding without booster screen (nitrogen diffusion depth was 16,1 μm). Application of a booster screen during plasma potential nitriding caused an approx. 14-times increase in the nitrogen diffusion depth into the material (Table 1). It should be noted, that the nitrogen diffusion layer after plasma potential nitriding shows the lowest thickness among all nitriding variants.

The highest nitrogen surface concentration after 316L austenitic steel glow discharge nitriding with different variants of specimen arrangement in the vacuum chamber was observed during cathode nitriding using a booster screen. The lowest concentration, was observed after plasma potential nitriding variants. It should be also noted that application of a booster screen during nitriding in the plasma potential practically did not change nitrogen concentration on the material surface (Table 1, Figure 3).

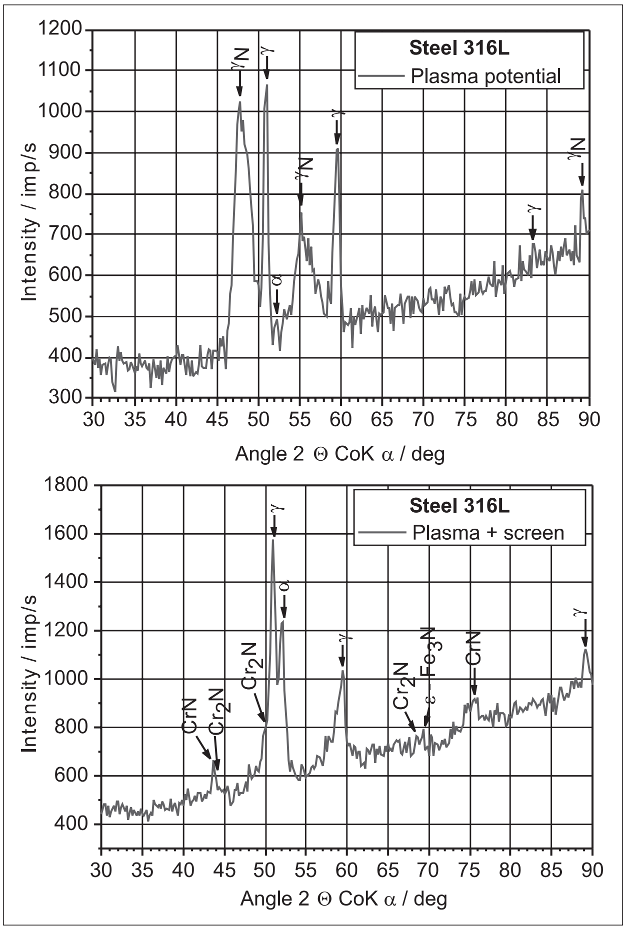

Figure 2 X-ray diffractograms of the 316L stainless steel after glow discharge.

Analyzing the nitrogen concentration with increasing distance from the nitrided surface it was observed that in the variants of cathode nitriding with and without the booster screens, nitrogen concentration in the distance of several microns from the nitrided specimen face stabilizes at a certain level. Considering the chromium to nitrogen atoms ratio it can be noticed that it amounts to approx. 2:1 (Table 1), which corresponds to A_2B compound type. This might suggest that the obtained nitrided layers are formed during precipitation of Cr_2N type chromium nitride in the austenite and nitrogen-saturated austenite matrix.

When analyzing carbon concentration as a function of the distance into the nitrided surface substrate, it was observed that carbon accumulation occurs in the nitrogen concentration drop area. This phenomenon is caused by the front of nitrogen-supersaturated austenite (*expanded austenite*) formed, displacing during nitriding, which pushes the austenite-dissolved carbon into the region of low nitrogen concentration.

Analyzing the structures of the obtained surface layers (Figure 4), a nitrided layer of variable thickness can be noticed (especially on etched microsections). Between the nitride zone and the material core, a transition zone was observed. This is probably the area, where the nitrogen concentration is not sufficient for nitrides

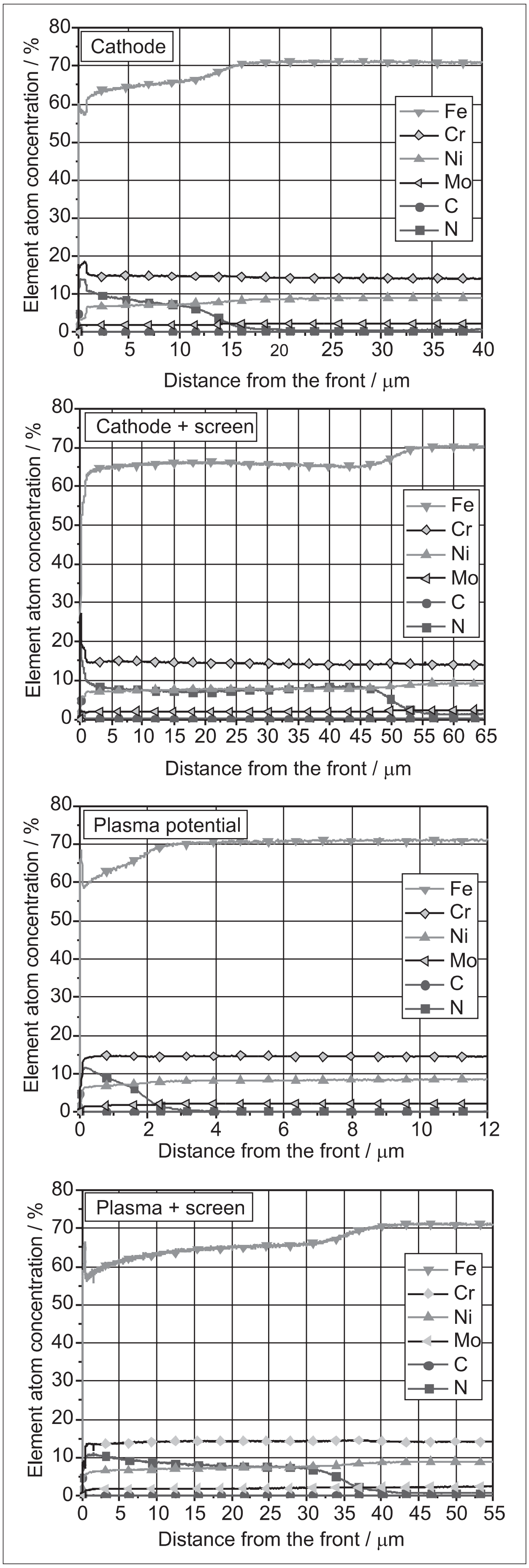

Figure 3 Elements depth profile in the surface layer after different variants of plasma nitriding.

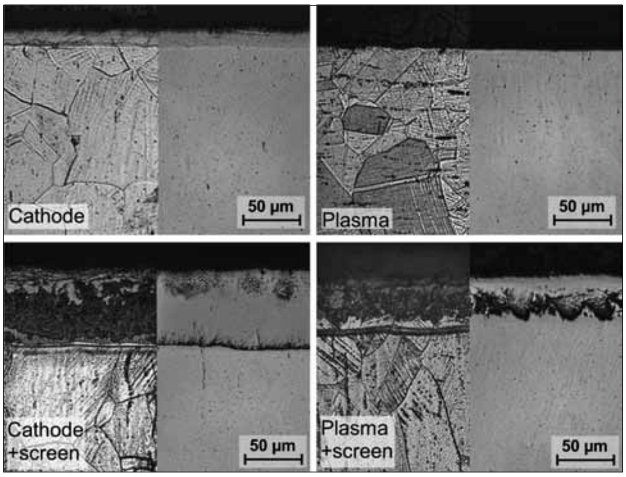

Figure 4 Microstructures of the surface layer after 316L steel plasma nitriding.

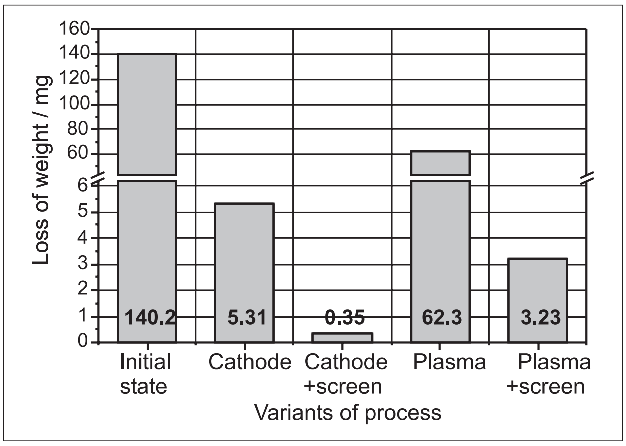

Figure 5 Wear test results of the 316 steel surface layer after glow discharge nitriding.

forming, therefore nitrogen-supersaturated austenite (*expanded austenite*) occurs there.

Analyzing the measurement results of specimen mass loss after the abrasion test we can be noticed that each nitriding variants significantly improves the abrasive wear resistance (Figure 5). The greatest increase in abrasive wear resistance was observed for the variants with booster screens application.

CONCLUSIONS

The 316L austenitic steel surface layers after different variants of glow discharge nitriding can be obtained in a single process. The booster screens applied for the surface phenomena intensifying, as the results of the formation of hollow cathodes on their surface, which cause primarily the increase of the nitrided specimens placed under the booster screens temperature. Application of the booster screens in the nitriding process causes an increase in the nitrided layers thickness, an increase in surface hardness and an enhancement of abrasive wear resistance. The effectiveness of the steel nitriding process described in this article can be understood as the result of the undertaken researches, expressed by the relation of the effects achieved versus the incurred outlays.

An increase in effectiveness may occur, when the identical effects have been achieved at lower outlays or unchanged outlays have produced better effects. The application of the booster screen in the glow discharge nitriding process did not change the basic parameters preset (such as: steel grade, atmosphere, pressure, time, temperature), but it did contribute to a clear (sometimes several times) increase of such thermochemical treatment indicator , that is the nitrided layer thickness, surface hardness, or abrasive wear resistance. Therefore, it might be possible that further studies also for other steel grades and process conditions, will allow, owing to the booster screens application, a new unconventional method to be developed, which would be characterized by higher effectiveness comparing to the current nitriding methods.

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