

## ION NITRIDING USING ACTIVE SCREEN METHOD

Received – Priljeno: 2018-12-12

Accepted – Prihvaćeno: 2019-02-25

Original Scientific Paper – Izvorni znanstveni rad

Austenitic steel grade 304 according to AISI was tested after ion nitriding in the temperature range 325 - 400 °C and time range 2 - 4 h, and after two different variants of sample arrangement in a glow discharge chamber. The first variant – the samples intended for nitriding were placed on the cathode, the second variant – the samples placed on the cathode were shielded with an active screen. In order to evaluate the effectiveness of the nitriding process variants, investigations of the diffusion depth of the obtained surface layers, surface hardness tests and tribological wear tests were carried out. It was found that using active screens increases the rate of diffusion of nitrogen into nitrided austenitic steel 304, thus increasing the thickness of the resulting surface layers.

*Key words:* austenitic steel, ion nitriding, diffusion, microhardness, abrasion, active screen method

### INTRODUCTION

Chromium-nickel austenitic steels, thanks to their good corrosion resistance, durability and easy formability, have found applications in many branches of industry. One of the many applications in which they are used is the biomedical industry [1-2].

Nitriding high-chromium steels encounters many difficulties due to the existence of an airtight film of chromium oxides on their surface, which obstructs or prevents the nitriding process. In practice, this difficulty is eliminated by preliminary surface treatment, such as etching and phosphating, or by introducing additives such as ammonium chloride or HCl into the reaction chamber or using such treatments as ionic or plasma treatments and as a result of using pre-preliminary cathodic sputtering under glow discharge conditions [3].

Ion nitriding with an active screen allows the formation of a nitrided layer of a greater thickness compared to cathodic nitriding, and with the expected functional properties. The development of this technology should move towards lowering the temperature and time of the nitriding process [4].

### MATERIAL AND RESEARCH METHODOLOGY

One of the most commonly used chromium-nickel steels, namely grade 304 steel according to AISI (X5Cr-Ni18-10 according to PN-EN 10088-1:19898) was subjected to the ion nitriding process. The chemical composition of the steel is shown in Table 1.

Table 1 **Chemical composition of 304 steel / wt. %**

Element	Min.	Max.
C	-	0,07
Cr	17,00	19,00
Ni	8,00	10,50
Mn	-	2,00
N	-	0,11
Si	-	1,00
P	-	0,045
S	0,01	0,03
Fe	rest	

The process of ion nitriding was carried out in a device for ion treatment with a cooled JON-600 anode, applying the following nitriding parameters in hydrogen-nitrogen plasma: working atmosphere pressure  $p = 150$  Pa, reactive mixture composition  $H_2$  75 % +  $N_2$  25 %, temperature range  $T = 325 - 400$  °C, time range  $t = 2 - 4$  h.

Two variants of sample arrangement were used in the glow discharge chamber of the furnace: the samples were placed directly on the cathode and the samples placed on the cathode were additionally covered with an active screen made of a perforated austenitic steel sheet.

In the first case, the surface of the samples is bombarded with ions with the energies resulting from the cathode drop value, while in the second case, strong voltage peaks appear in the area of the surface layer, which must interact with the nitrogen ions present in this area. The duration of voltage peaks favors giving ions high velocities, corresponding to the kinetic energy of several dozens or even several hundred electronvolts. These ions are driven into the material, creating in the surface layer a strong non-equilibrium zone saturated with nitrogen. This in turn favors the diffusion of nitrogen into the material. Due to the large concentration gradients, diffusion does not have to, at least at its initial stage, take place at the grain boundaries, which pro-

M. Ogórek, T. Frączek - Czestochowa University of Technology, Faculty of Materials Processing Technology and Applied Physics, Czestochowa, Poland, e-mail: ogorek.marzena@wip.pcz.pl, fraczek.tadeusz@wip.pcz.pl

motes the formation of nitrided layers with high homogeneity.

Microhardness measurements of the nitrided layers were made using the Knoop method on a Future Tech FM7 microhardness tester. The microhardness was measured using loads of 25 G, 50 G and 100 G.

The depth of nitrogen diffusion was determined on the basis of element distribution analysis on an optical emission spectrometer with a glow discharge GDS GD Profiler HR with a Grimm discharge lamp with a cathode diameter of 4 mm.

A T-05 tribological tester was used to determine the tribological properties of the produced layers. Abrasion resistance tests were performed under dry friction conditions in a roll-block arrangement.

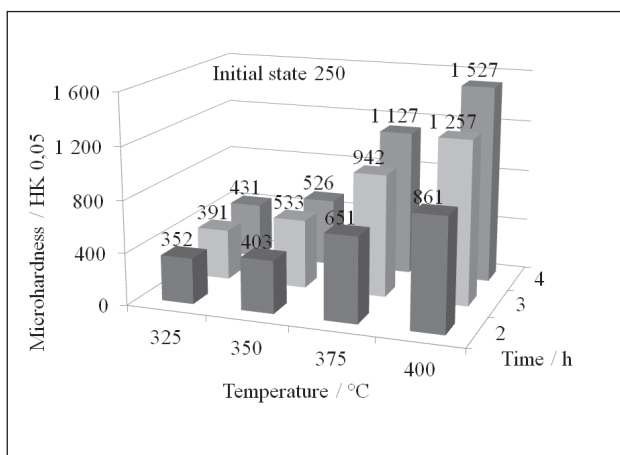
## TEST RESULTS

The surface microhardness tests of the nitrided layers showed an increase in the hardness of the tested austenitic steel after the conducted cathodic ion nitriding process, with reference to the hardness of this steel in the initial state. The use of the active screen method in the ion nitriding process resulted in a further increase in

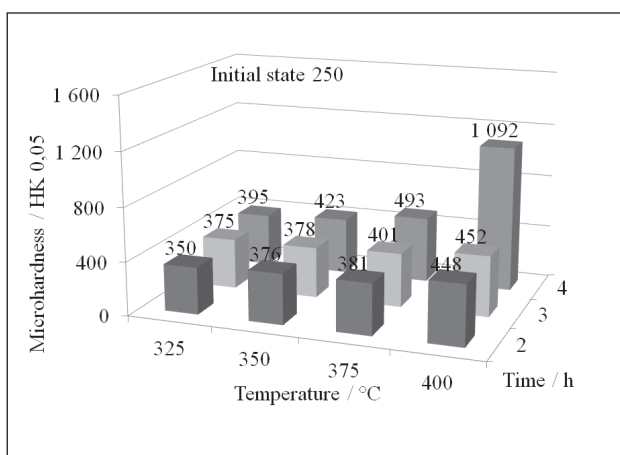
hardness. The surface layers produced in the ion nitriding process have a hardness of about three to five times higher than the initial state. Using the active screen supporting the process of short-term and low-temperature ion nitriding caused a further increase in hardness, which increased six-fold in relation to the initial state. The results of hardness measurements after ion nitriding of steel 304 are shown in Figures 1a and 1b.

Based on analysis of the chemical composition profile, the nitrogen diffusion depth was determined, depending on the scheme adopted in the experimental design parameters. Analysis of the obtained test results showed that during cathodic nitriding of austenitic steel 304, nitrided layers with a depth in the range from 0,3 to 2,5  $\mu\text{m}$  were produced, while the active screen method resulted in the formation of layers with a depth in the range of 0,5 – 7,3  $\mu\text{m}$  (Figures 2a and b). The obtained test results allow one to conclude that using the active screen method results in a nearly two- to six-fold increase in the diffusion depth of the obtained nitrided layers in relation to the cathodic process carried out under the same conditions.

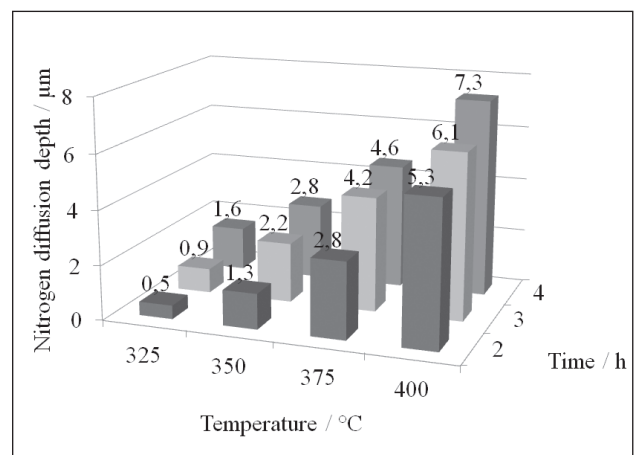
Examination of the weight loss of the samples after the tribological test showed an increase in abrasion resistance of the material after nitriding as compared to the initial state - each of the conducted nitriding processes improves the abrasion resistance. Using the ac-



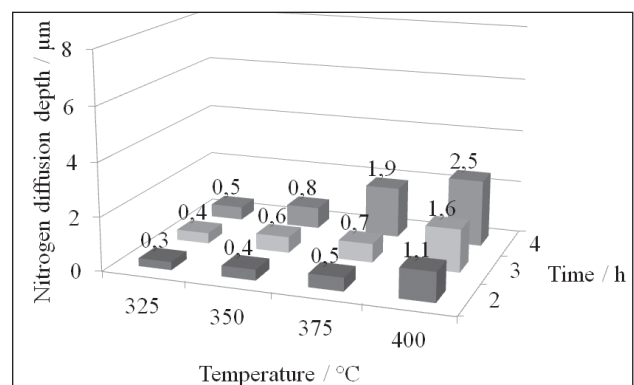
**Figure 1a** Microhardness of obtained nitrided layers using 50 G load for nitrided samples using active screen method



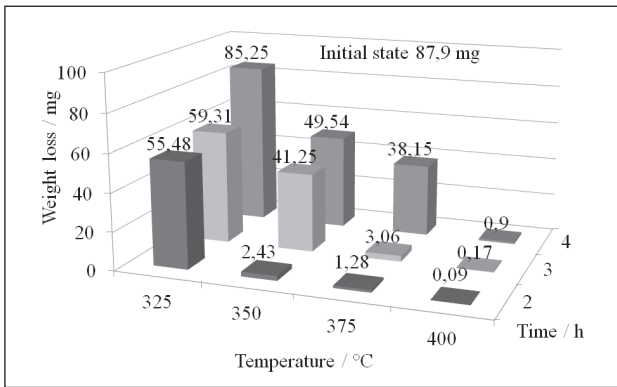
**Figure 1b** Microhardness of obtained nitrided layers using 50 G load for cathodic nitrided samples



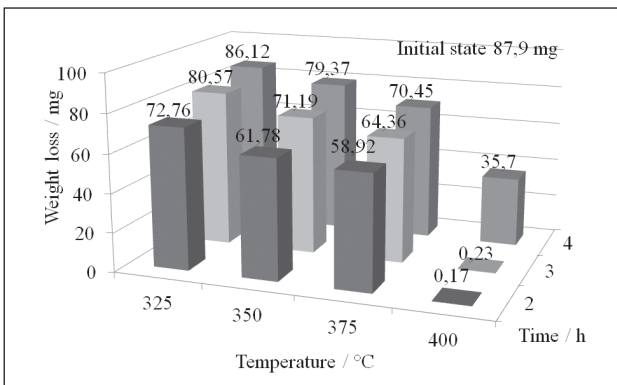
**Figure 2a** Nitrogen diffusion depth for nitrided samples using active screen method



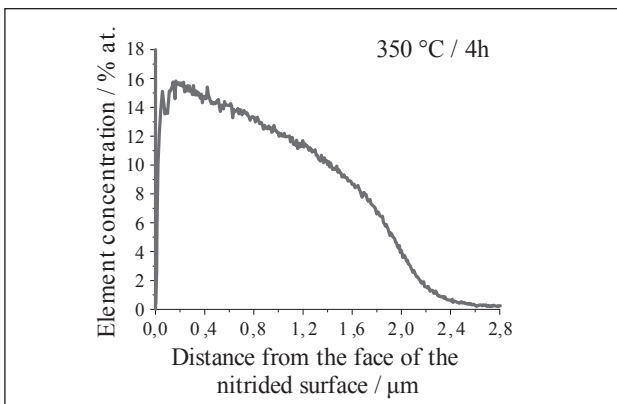
**Figure 2b** Depth of nitrogen diffusion for cathodic nitrided samples



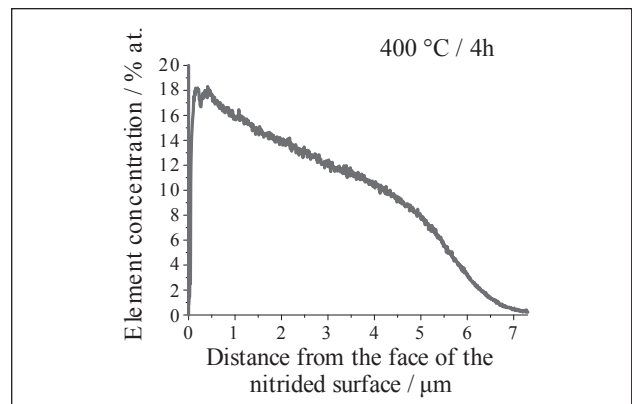
**Figure 3a** Abrasive wear after 60 min of tribological testing for samples nitrided using active screen method



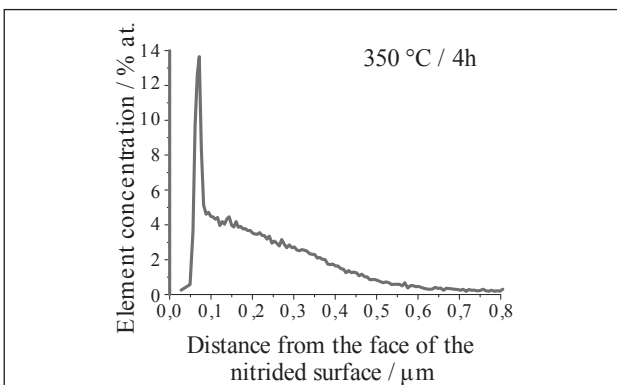
**Figure 3b** Abrasive wear after 60 min of tribological testing for cathodic nitrated samples



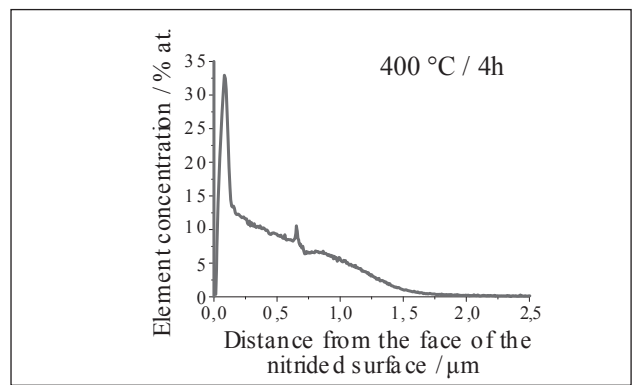
**Figure 4a** Nitrogen concentration in surface layer during ion nitriding using active screen method - temperature 350 °C, time 4 h



**Figure 5a** Nitrogen concentration in surface layer during ion nitriding using active screen method - temperature 400 °C, time 4 h



**Figure 4b** Nitrogen concentration in surface layer during cathodic ion nitriding - temperature 350 °C, time 4 h



**Figure 5b** Nitrogen concentration in surface layer during cathodic ion nitriding - temperature 400 °C, time 4 h

tive screen method in the ion nitriding process increases the material resistance to tribological wear, also in comparison to classical cathodic nitriding. During all the cathodic nitriding processes at 325 °C, a slight increase in abrasion resistance was found, due to the formation of very thin surface layers at this temperature, which are quickly rubbed off by the load used for the test (13,73 N). The test results for abrasive wear after 60 minutes of tribological testing are shown in Figures 3a and b.

By analyzing the results of the distribution of elements (Figures 4 a and b, Figures 5 a and b), it can be noticed that short-term nitriding on the cathode caused diffusion of nitrogen into the surface of the material to a depth of 0,8µm for the temperature of 350 °C and 2,5µm for the temperature of 400 °C. Use of the active screen increased the nitrogen diffusion depth almost three-fold (for the temperature of 350 °C - 2,8 µm and for the temperature of 400 °C 7,3µm).

Considering the nitrogen concentration along with the distance from the face of the nitrided surface, it can be noticed that the nitrogen concentration at a certain short distance from the face of the nitrided sample stabilizes at a certain level, which is particularly evident during short-term nitriding with the active screen. In turn, taking into account the ratio of the concentration of chromium atoms to nitrogen atoms (Table 2), we can see that it is 2:1, which corresponds to the A<sub>2</sub>B type compound. This may indicate that the obtained nitrided lay-

ers in this region are composed of  $\text{Cr}_2\text{N}$  chromium nitride precipitates in the austenite matrix and austenite saturated with nitrogen. However, the chromium concentration decreases at the surface of the nitrided material itself. The ratio of chromium to nitrogen in this area is approximately 1:1, which may indicate that  $\text{CrN}$  nitrides occur on the surface of the nitrided material itself. In the transition area between the nitrided layer and the substrate material, the nitrogen concentration falls below the limit value of formation of nitride precipitates in the matrix, which results in the presence an area of austenite saturated with nitrogen  $\gamma\text{N}$  (expanded austenite).

The obtained test results confirm the zone structure of the surface layers obtained on austenitic steels, described in the literature data [4], both as a result of nitriding on the cathode and using the active screen method (Figures 6). In the construction of the obtained layers, we can specify two zones: the subsurface zone of nitride precipitates in the metallic matrix and the austenite zone saturated with nitrogen.

Table 2 **Depth of nitrogen diffusion and Cr:N concentration ratio after two variants of ion nitriding**

Process	Process variant	Depth of nitrogen diffusion, $\mu\text{m}$	Proportion of Cr:N concentration on surface layer	Proportion of Cr:N concentration in surface layer
350 °C / 4 h	Cathode	0,8	3,5:3,6 (1:1) CrN	9,6:4,7 (2:1) $\text{Cr}_2\text{N}$
	Active screen	2,8	13,66:13,69 (1:1) CrN	14,64:7,32 (2:1) $\text{Cr}_2\text{N}$
400 °C / 4 h	Katoda	2,5	13,9:12,02 (1,1:1) CrN	16,23:8,03 (2:1) $\text{Cr}_2\text{N}$
	Active screen	7,3	10,3:10,7 (1:1) CrN	14,6:7,7 (2:1) $\text{Cr}_2\text{N}$

## SUMMARY

The adopted parameters of the short-term and low-temperature ion nitriding process resulted in the formation of compact nitrided layers on the surface of the 304 steel, wherein the use of an active screen caused a two to six-fold increase in the thickness of the obtained nitrided layer compared to nitriding on the cathode.

Each of the adopted nitriding parameters increased the hardness of the nitrided steel. During the nitriding of samples located directly on the cathode, the increase in

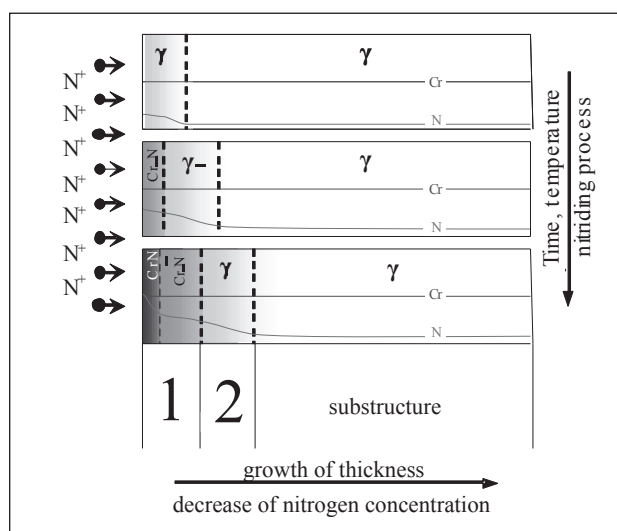


Figure 6 Schematic diagram of the structure of nitrided layers obtained on austenitic steel substrate 304 in short-term and low-temperature processes; where: 1 – nitride emission zone, 2 – nitrogen supersaturated austenite zone [4]

hardness was almost double in relation to the material in the initial state. Using the active screen resulted in a further, even six-fold increase in hardness of the nitrided surfaces in relation to the initial state.

The obtained surface layers have a zone structure and are composed successively of a zone of  $\text{CrN}$  nitrides, a zone of  $\text{Cr}_2\text{N}$  nitride precipitates and a transition zone built from the  $\gamma\text{N}$  phase, i.e. austenite supersaturated with nitrogen.

## REFERENCES

- [1] M. Jurczyk, J. Jakubowicz, *Bionanomaterials*. Poznan University of Technology Publishing House, Poznan 2007, pp. 22-30.
- [2] S. Kannan, A. Balamurugan, S. Rajeswari, *Electrochemical characterization of hydroxyapatite coatings on  $\text{HNO}_3$  passivated 316L SS for implant applications*, *Electrochimica Acta* 50 (2005), 2065-2072.
- [3] J. Baranowska, W. Serwiński, A. Zleliński, *Surface treatment of austenitic stainless steel*, *Materials Engineering* 5 (1999), 279-281.
- [4] T. Frączek, *Unconventional low-temperature glow discharge nitriding of metallic materials*, *Czestochowa University of Technology, Faculty of Production Engineering and Materials Technology Publishing House, Czestochowa* 2011.

Note: The professional translator for English language is Christine Frank-Szarecka, Poland