# THE EFFICIENCY OF ION NITRIDING OF AUSTENITIC STAINLESS STEEL 304 USING THE "ACTIVE SCREEN"

Received – Prispjelo: 2014-03-01 Accepted – Prihvaćeno: 2014-08-31 Preliminary Note – Prethodno priopćenje

The study examined layers were formed on the outer surface of austenitic stainless steel 304 under glow discharge conditions in the low-temperature and short-term ion nitriding. The outer layers analyzed in the work produced in parallel in the classical process of cathode and a novel method of "active screen", intensifying the process of nitriding.

Key words: austenitic steels, glow discharge nitriding, surface, low temperature

### INTRODUCTION

The dynamic growth of the contemporary science and technology requires the use of materials with increasingly better mechanical and service properties. Currently used metallic materials often do not fully meet the requirements associated with their purpose. This creates the need for using more expensive materials possessing these high properties in their initial state, or modifying the currently used materials. The modification of metallic materials being in use may result in changes in their mechanical and service properties either within their entire volume or only in the top layer.

Presently, the majority of studies dealing with changes in the bulk of material focus on modifying the chemical and phase composition of alloys [1, 2] or applying appropriate thermal-plastic treatment that, combined with accelerated cooling, ensures structures of the assumed mechanical properties to be obtained [3].

The properties of the top layer of metallic materials, on the other hand, are most often shaped by using various surface engineering methods, developed particularly intensively in the last three decades of the past century [4].

Owing to their good corrosion resistance, mechanical strength, heat resistance and high formability, austenitic steels have found application in many branches of industry. In the initial state, these steels exhibit, however, low hardness and abrasive wear resistance.

However, the modification of the surface of austenitic steels is, as a rule, difficult, as the dense and tight Cr2O3 oxide film formed on the steel surface prevents other elements from penetrating into the material. Nevertheless, plasma methods have been successfully developed, which allow the austenitic stainless steel surface to be modified by removing the chromium oxides from it through ion bombardment. Among them, a leading method is glow discharge nitriding [5-7]. Ion bombardment of the cathode surface makes the surface activation of steels containing considerable chromium contents occur very rapidly, whereby ion nitriding proceeds with no restrictions [7]. The simultaneous cathode sputtering accompanying the interaction of ions with the cathode, and the low nitrogen partial pressure, inhibit the growth of the nitride zone [7]. This can be counteracted by applying unconventional nitriding methods that use active screens [4].

#### MATERIAL AND TESTING METHODOLOGY

The aim of the undertaken tests was to compare the effectiveness of the active-screen nitriding process with the cathodic nitriding process. The tests were carried out on a JON-600 glow-discharge treatment stand with a cooled anode supplied by a Dora Power System MSS-10 pulse power supply.

Two variants of locating the samples in the glow discharge chamber were adopted in carrying out the nitriding process: directly on the cathode and on the cathode under the active screen (the samples were in the working chamber at the same time).

The test material was an austenitic steel from the group of nickel-chromium steels 304 acc. to standard AISI 304 and a steel from the chromium-nickel-molyb-denum steels 316L acc. to standard AISI.

The ion nitriding process was carried out following a mathematical model for experiment planning developed.

The range of the employed nitriding duration and temperature was adopted based on preliminary tests of short-duration(2 - 4 h) and low-temperature (325 - 400 °C) glow-discharge nitriding of austenitic steels [4, 8]. The most important objective determining the selection of the nitriding parameters was to achieve nitrided lay-

M. Ogórek, Z. Skuza, T. Frączek, Czestochowa University of Technology, Czestochowa, Poland

ers of satisfactory tribological properties, while retaining the good corrosion resistance of the nitrided austenitic steel (no occurring chromium nitride precipitation processes that would lead to the dechroming of the austenitic matrix).

#### **TESTING RESULTS**

During testing, the value of power supplied to the glow discharge-generating power supply was measured. On this basis, the average power used for nitriding was calculated. The power measurements were calculated from the arithmetic mean from 12 processes. For the same nitriding duration the energy expenditures were identical.

On the basis of the obtained results it was found that the cathodic nitriding of the 304 austenitic steel tested, within the adopted experiment plan, resulted in the formation of nitrided layers of a depth contained in the range from 0,3 to 2,5  $\mu$ m, while with the use of the active screen, in the range from 0,5 to 7,3  $\mu$ m. In the case of the 316L steel, similarly, the nitrided layers lay in the range from 0,56 to 2,51  $\mu$ m, and with the use of the active screen, in the range from 1,07 to 6,34  $\mu$ m.

The analysis of the presented data indicates that, in the identical conditions, the use of the active screen for the 304 steel and the adopted testing range results in a 2 - 6 time increase in the depth of obtained nitrided layers compared to the cathodic process.

The surface microhardness of the obtained nitrided layers was measured by the Knoop method with asemiautomatic hardness tester using loads of 245,2 mN (25 G) and 490,3 mN (50 G), respectively. The layers produced in the cathode nitriding process for both 304 steel and 316L steel are characterized by surface hardness four to five times higher compared to the initial state. The use of active screens aiding the short-term ion nitriding process resulted in an sixfold increase in hardness relative to the initial state (steels 304 and 316L, Figure 1).

An abrasive wear resistance test under dry friction conditions in the roller-block arrangement was carried out on a T-05 tester. The abrasive wear resistance test was performed under a load of 13,73 N and for a duration of 2 hours (Four 30 minutes' cycles). A measure of abrasive wear resistance was the loss of specimen mass after each of the tribological test cycles. The analysis of the specimen mass loss measurement results after the abrasive wear resistance test showed that both the cathode ion nitriding process and the process using the active screen improved the abrasive wear resistance relative to that of the material in the initial state. The use of the active screen increases the abrasive wear resistance of the material compared to classic cathode ion nitriding (Figure 2).

In the presented photographs of the obtained microstructures (Figure 3) we can notice that each of the applied variants of short-term glow discharge nitriding of steels 304 and 316L has caused the formation of a tight,

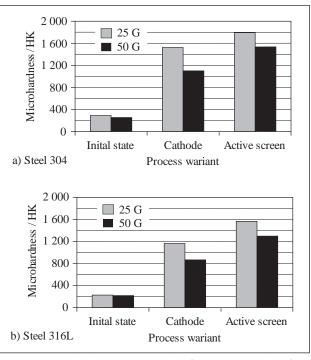


Figure 1 Microhardness tests results of austenitic steel surface after glow discharge nitriding; a) 304, b) 316L

uniform nitrided layer on the material surface, which has a characteristic zonal structure formed as a result of the concentration decreasing into the nitrided surface. By comparing the obtained structures we find that the use of the aiding screens has resulted in the formation of a nitrided layer of a considerably greater thickness, caused by the greater depth of nitrogen diffusion into the nitrided substrate.

## ECONOMIC ASPECTS OF ION NITRIDING BY THE "SCTIVE SCREEN" METHOD

Electric energy costs are one of the components determining the overall costs of ion nitriding. The costs involved in the heating process and the specimen exposure period are the same for the both variants of the nitriding process. During cathode nitriding, the cost of electric energy needed for producing a 2,5 µm-deep diffusion layer amounts to 4,3 EUR (400 °C, 4 hrs). At the same time, for ion nitriding using the «active screen» method, the cost of producing a 2,8 µm-deep diffusion layer is, respectively, 3,3 EUR (350 °C, 4 hrs) and 2,6 EUR (375 °C and 2 hrs). The juxtaposition of these costs in the case at hand shows clearly that the optimal variant is the ion nitriding process conducted in the shortest possible duration (in the range of conditions defined by the parameters of the carried out). Increasing the diffusion layer thickness should therefore involve first the increase of the nitriding temperature and only then the increase of the specimen exposure duration.

The empirical relationships provided in study [4] allow also one to found that reducing the specimen exposure duration (from 4 to 2 hrs), while simultaneously increasing the nitriding temperature (by 25 °C) does not significantly affect the tribological properties.

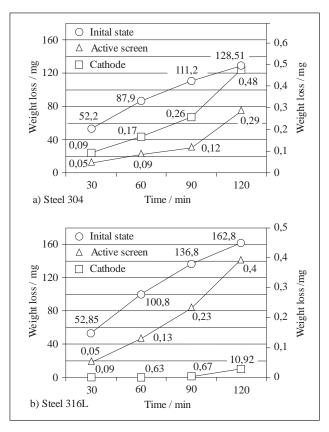


Figure 2 Wear resistance after short-term and lowtemperature nitriding: a) 304 steel, b) 316L steel

The reduction of the specimen exposure duration will not only contribute to the reduction of electric energy consumption but also result in a decrease in labour costs. Thus, considering the reduction of electric energy consumption and the shortening of the overall nitriding process duration (the cost of a man-hour – the heating, nitriding and cooling period) it can be concluded that only these two cost types alone demonstrate that ion nitriding using the «active screen» method is cheaper by 31 % compared to classic cathode nitriding.

#### SUMMARY

At this stage of investigation it was only possible to calculate the partial effectiveness. The presented data shows that the process of short-term and low-temperature ion nitriding using the aiding screen is more effective that the classic cathode process.

The use of the active screen in the nitriding of steel 304 increased the depth of obtained nitrided layers by 2 - 6 times relative to classing nitriding, depending on the process parameters. The adopted parameters of the active screen ion nitriding process caused the formation of nitrided layers on the 316L steel surface, which were 2 - 3 deeper compared to the cathode process.

The adopted parameters of nitriding steels 304 and 316L caused an increase in the surface hardness of the nitrided steels. When nitriding specimens located directly on the cathode, the hardness increased by 4 - 5 times relative to the initial material. Using the active

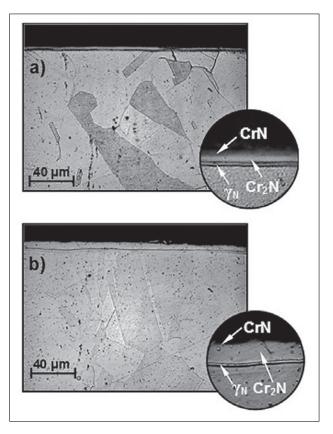


Figure 3 Microstructures of the 304 steel surface layer after short-term glow discharge nitriding. Sample position: a) cathode, b) cathode + screen

screen resulted in a sixfold increase in the hardness of the nitrided surfaces compared to the initial state.

The assumed nitriding process parameters enhance the abrasive wear resistance. For the steel grades examined, either of the processes (cathode, cathode + screen) improves the abrasive wear resistance as compared to the material in the initial state. In addition, an improvement in abrasive wear resistance is visible for the active-screen nitrided specimens over the cathode nitrided specimens.

The produced surface layers have a zonal structure and are composed of the following zones, in succession: a CrN nitride zone, a Cr2N nitride precipitation zone, and a transition zone built of the phase of  $\gamma$ N, or nitrogen-supersaturated austenite.

The preliminary analysis of the costs of the nitriding process with the "active screen" method shows that this method, as compared to classic ion nitriding, enables nitrided layers of comparable magnitudes (or even greater) to be obtained at costs lower by 31 %. In the economic assessment of the both processes, only electric energy costs and the costs of operation of the specific equipment by a technical worker were taken into account. It can be expected, therefore, that considering all costs will result in a further difference in costs between the compared nitriding processes.

## REFERENCES

 Kawałek A., Rapalska-Nowakowska J., Dyja H., Koczurkiewicz B.: Physical and numerical modelling of heat treatment the precipitation-hardening complex-phase steel (CP), Metalurgija, 52 (2013) 1, 19-22.

- [2] Kaputkina L. M., Prokoszkina W. G., Siwka J., Svjazin A. G., Koczurkiewicz B., Knapinski M.: The individual and cumulative effect of C and N on the hardening and structure of high-nitrogen hot plastic worked steels, Materials Science Forum, 539-543 (2007) 1-5, 4968-4974.
- [3] Dyja H., Knapiński M., Kwapisz M., Snopek J.: Physical Simulation of Contrelled Rolling and Accelerated Cooling for Ultrafine Grained Steel Plates, Archives of Metallurgy and Materials, 56 (2011) 2, 447-454.
- [4] Frączek T: Niekonwencjonalne niskotemperaturowe azotowanie jarzeniowe materiałów metalicznych, Wyd. WIPMiFS, Częstochowa 2011.
- [5] Borgioli F., Galvanetto E., Bacci T., Pradelli G.: Influence of the treatment atmosphere on the characteristics of glowdischarge treated sintered stainless steels, Surface and Coatings Technology 149 (2002), 192–197.

- [6] Gui-jiang Li, Qian Peng, Cong Li, Ying Wang, Jian Gao, Shu-yuan Chen, Jun Wang, Bao-luo Shen: Effect of DC plasma nitriding temperature on microstructure and drysliding wear properties of 316L stainless steel, Surface & Coatings Technology 202 (2008), 2749–2754.
- [7] Roliński E., Sharp G.: Wybrane zastosowania azotowania i azotonawęglania jonowego w Stanach Zjednoczonych, Inżynieria Powierzchni, (2007) 3, 3-8.
- [8] Ogórek M., Frączek T., Skuza Z.: Effectiveness of the ion nitriding of steels X5CrNi18-10, X2CrNiMo17-12-2. Innovations in management and production engineering, Editor: Ryszard Knosala, Oficyna Wydawnicza Polskiego Towarszystwa Zarządzania Produkcją, Opole 2013, 128-138.
- **Note:** The professional translator for English language is Czesław Grochowina, Studio Tekst, Poland