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## APPLICATION OF ACTIVE SCREEN METHOD FOR ION NITRIDING EFFICIENCY IMPROVEMENT

### ZASTOSOWANIE INNOWACYJNEJ METODY „ACTIVE SCREEN” DLA POPRAWY WYDAJNOŚCI PROCESU AZOTOWANIA JONOWEGO

Paper presents the research of austenitic steel AISI 304 after ion nitriding at 400°C and at  $t = 4$  h, for the two different variants of samples distribution in the working plasma reactive chamber tube. In order to assess the effectiveness of ion nitriding variants emission spectroscopy – GDOES, surface hardness tests, microstructure research (LM) of nitrated layers were made. It has been found that the use of active screens increases the surface layer thickness and depth of nitrogen diffusion into austenitic steel 304.

*Keywords:* glow discharge nitriding, active screens, austenitic steels, nitrogen diffusion depth

Badaniom poddano stal austenityczną gatunku 304 wg AISI po azotowaniu jarzeniowym w temperaturze 400°C i czasie  $t = 4$  h, dla dwóch różnych wariantach rozmieszczenia próbek w komorze jarzeniowej. W celu oceny efektywności wariantów procesu azotowania przeprowadzono badania głębokości dyfuzji metodą GDOES, badania twardości powierzchniowej, badania odporności na zużycie ściernie oraz analizę mikrostruktur otrzymanych warstw wierzchnich. Stwierdzono, że zastosowanie ekranów wspomagających powoduje wzrost głębokości dyfuzji azotu w głąb azotowanej stali austenitycznej 304, a tym samym zwiększenie grubości otrzymanych warstw wierzchnich.

#### 1. Introduction

The world's production of crude steel in 2014 amounted to 1662 million Mg, having increased since the preceding year by 1.2% (in Poland, 8.8 million Mg with an increase by 9.8% since 2010) [1]. Regardless of the progress in science and technology, steel has remained one of the key constructional and engineering materials.

New constructional solutions that require improved mechanical and service properties of materials to reduce energy consumption and operational costs, while complying with the environmental proportion rules, has driven an intensive development of e.g., surface engineering methods in recent decades [2, 3]. The practical application of new, innovative solutions is conditioned, however, by the capability to achieve the desired material properties at costs lower than in the currently functioning industrial methods. One of the measurements of the economic aspect of a technological process is the effectiveness, which is defined as the ratio of the obtained effect to the incurred outlay [4].

Nitriding is one of the thermochemical treatment types which belongs to the group of the fastest developing surface engineering methods. This process enhances the service properties of machines and parts, especially their tribological properties and corrosion resistance. Such a potential for designing

the surface layer characteristics seems to be particularly attractive for austenitic steels, among the others whose low hardness and abrasion resistance significantly limit their application [5].

The process of high-chromium steels nitriding is, however, difficult to carry out due to the occurrence of a thin film of chromium oxides on the steel surface, which prevents nitrogen penetration into the material. A method that enables the austenitic steel nitriding process to be effected is ion nitriding. It allows the film of oxides to be removed by cathode sputtering [6]. The conditions of glow-discharge nitriding are defined by the parameters, such as: time, temperature, pressure voltage and current intensity, and chemical composition of the atmosphere [7].

The development of glow-discharge heating techniques and associated glow discharge- radiator and resistance-glow discharge heating, make it possible to shorten the heating duration. A reduction in energy intensity might be provided by developing technologies realized at low temperature and with a short process duration, as well as energy efficient vacuum technologies, and the modification of the CVD and PVD methods [8].

Active-screen ion nitriding enables formation of nitrated layers with greater depth and expected service properties. The development of this technology should proceed by reducing the temperature and duration of the nitriding processes [8].

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## 2. Materials and methods

The aim of the tests is to compare the effectiveness of the active-screen ion nitriding process with the cathodic ion nitriding process. The tests were realized in a JON-600 glow-discharge treatment stand with a cooled anode supplied with a Dora Power System MSS-10 pulse power supply (Fig. 1).

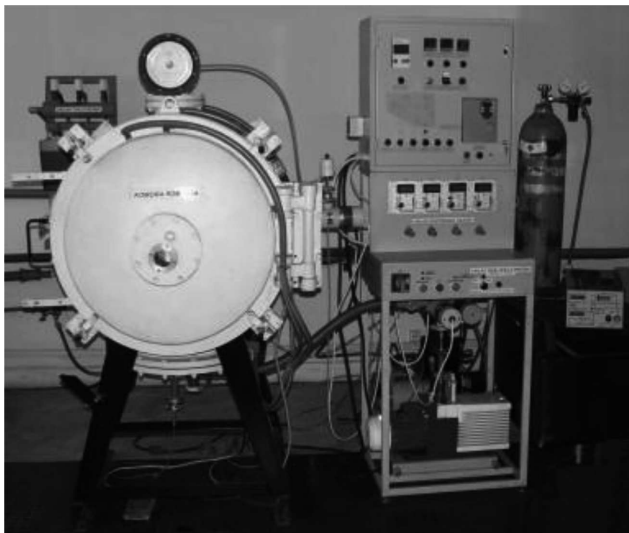


Fig. 1. Active screen ion nitriding equipment – JON-600

Two variants of samples placement in the glow discharge chamber were realized directly in the cathode and in the cathode covered with active screen. Samples were nitrided at the same time in the reactive chamber.

The test material used for the research was an austenitic steel acc. to standard PN-EN 10088-1:1998 (AISI 304).

The ion nitriding process was realized according with experiment planning mathematical model (Fig. 2).

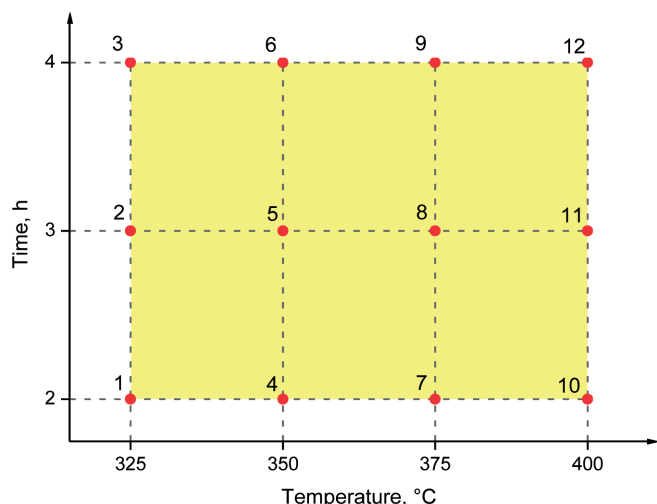


Fig. 2. Experiment mathematic planning diagram

The range of the employed nitriding duration and temperature was adopted based on preliminary short term tests (2÷4 h) and low-temperature (325÷400°C) [4, 10]. The most important objective determining the selection of the nitriding parameters was to achieve nitrided layers of satisfactory tribological properties, while retaining the good corrosion resistance

of the nitrided austenitic steel (no occurrence of chromium nitride precipitations that would lead to the de-chromization of the austenitic matrix).

Distribution profile studies of elements in the surface layer was realized the optical emission spectrometer from HORIBA Jobin glow discharge Yvon GD-Profilier HR (*GDOES RF Glow Discharge Optical Emission Spectroscopy*). Grimm's discharge lamp with a diameter of 4 mm cathode.

The surface microhardness of the obtained nitrided layers was measured by the Knoop method with asemi-automatic hardness tester under loads of 245.2 mN (25 G) and 490.3 mN (50 G) respectively.

An tribological wear resistance test under dry friction conditions in the roller-block system was performed on a T-05 tester. The 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.

## 3. Results and discussion

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 similar.

On the basis of the obtained results it was found that the cathodic nitriding of the 304 austenitic steel tested, within applied experiment plan, resulted in the formation of nitrided layers of a depth in the range from 0.3 to 2.5  $\mu\text{m}$ , while with the use of the active screen, in the range from 0.5 to 7.3  $\mu\text{m}$  (Fig. 3).

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.

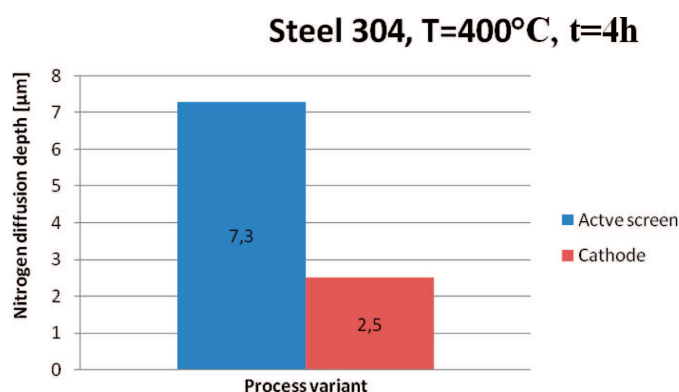


Fig. 3. The depth of diffusion of nitrogen

The layers produced by with the cathode nitriding process for both 304 steel are characterized by surface hardness four to five times higher compared to the initial state. The use of active screens for the short-term ion nitriding process resulted in an sixfold increase in hardness relative to the initial state (Fig. 4).

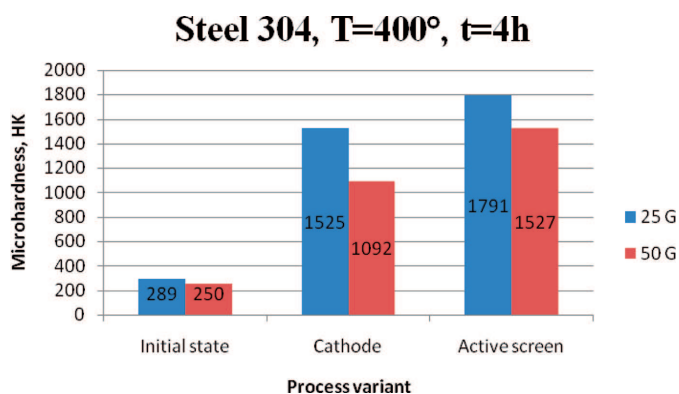


Fig. 4. Microhardness tests results of austenitic steel surface after glow discharge nitriding

The analysis of the specimen mass loss 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 in relation to that of the material in the initial state. The application of the active screen increases the abrasive wear resistance of the material compared to conventional cathode ion nitriding (Fig. 5).

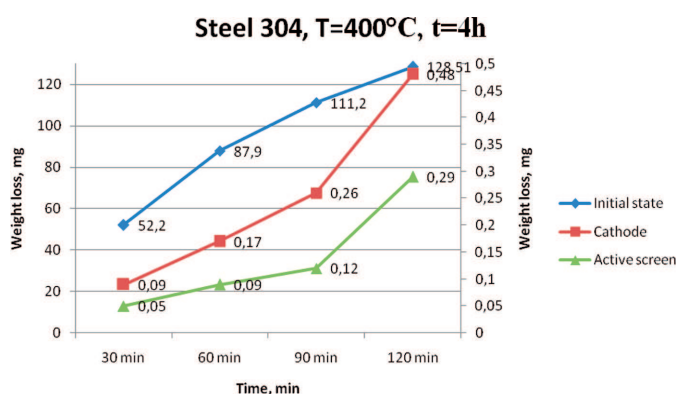


Fig. 5. Wear resistance of ASIS 304 steel after the short-term and low-temperature ion nitriding

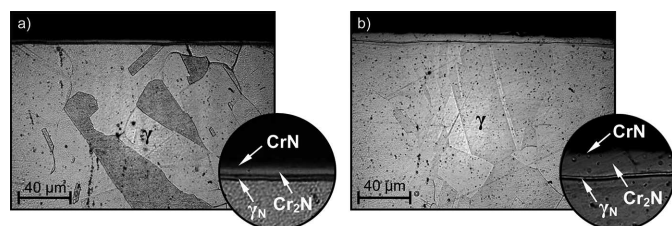


Fig. 6. Microstructures of the 304 steel surface layer after short-term glow discharge nitriding. Process variant: a) cathode, b) cathode + active screen

In the presented figures of microstructures (Fig. 6) it can be noticed that each of the applied variants of short-term glow discharge nitriding of steels 304 caused the formation of a tight, uniform nitrided layer on the material surface, which has a characteristic zonal structure formed in a result of the

concentration decreasing into the nitrided surface. While comparing the obtained structures it can be found that the use of the active screens has resulted in the formation of a nitrided layer of a considerably greater thickness, caused by the nitrogen diffusion depth into the nitrided substrate increment.

#### 4. Summary

Application of active screen method for ion nitriding of the X5CrNi18-10 steel according to selected parameters results in a 2÷6 time increment in the depth of nitride layers compared to the cathodic process.

Parameters of nitriding steels 304 and 316L caused an increase in the surface hardness of the nitrided steels. When nitriding specimens were located directly on the cathode, the hardness increased by 4-5 times relative to the initial material. Application of the active screen resulted in a significant (six-fold) hardness increment of the nitrided surfaces compared to the initial state.

The selected nitriding parameters improve the abrasive wear resistance. For the steel grades examined, either of the processes (cathode, cathode + active screen) improves the abrasive wear resistance in compare to the initial state material. 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 composed with following zones, CrN nitrides zone, a Cr<sub>2</sub>N nitrides precipitation zone and a transition zone built of the  $\gamma_N$  phase or nitrogen-supersaturated austenite.

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