The active screen influence of edge effect in plasma nitriding

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Abstract. Plasma nitriding thermochemical treatment has a beneficial effect regarding wearing resistance. One of the disturbing effects for plasma nitriding is edge effect, which modifies the properties uniformities on the edges of the treated part. Edge effect is found in the intersection area of the two surfaces adjacent negative light. The active screen has a role in modifying the plasma field for the entire part, so also in the edges areas. This overcomes and reduces the negative technological consequences of the edge effect. Different analysis for the subjected areas were made, also on the surface parts but also in depth of the parts, in it’s section.

1. Introduction

The large number of interdependent parameters, electrode voltage, pressure, temperature, gas composition and working gas dissociation degree influences the plasma nitriding process [1-4].

Due to, it presents some difficulties in controlling the negative phenomena, such as the occurrence of the double cathode effect, the effect the edge and the risk of arcing. These undesirable phenomena produce local energy concentrations and high temperature differences on the surface of the workpiece, resulting in unevenness of the diffusion layer and the white layer of chemical compounds, or in some cases even damage to the surface by local melting [5]. On the other hand, another disadvantage of plasma nitriding process is intense physical sputtering, caused by applying plasma discharge directly to the surface of the parts, which improves reactivity, but at the same time decreases surface quality by increasing the roughness and risk to surface exfoliations [6].

These disadvantages of plasma nitriding impose a number of restrictions when using this technology. These restrictions include the following: restrictions on the complexity of the parts, restrictions on the placement of different parts with different geometry on the cathode plate, temperature restrictions, electric field distribution restrictions, quality restrictions of the treated parts - chemical cleaning, lack of any peaks or adheres that can form an electric arc [7,8].

Polarized screen polishing is a technology that largely solves the difficulty of classical plasma nitration. In principle, this technology consists of changing the geometry of the electric field between the anode and the cathode [9-11]. This is accomplished by introducing a third electrode, in the form of a polarized metal screen, between the anode and the cathode. Thus, plasma discharge does not occur directly on the piece, but through the screen, thereby achieving the uniformity of the electric field on the surface of the piece. With this method, both the negative effects produced by the cathodic tension fall, occurring directly on the part and the local concentration of the discharge energy are greatly diminished [12, 13].
In the paper, the effect of the edge effect on plasma nitriding was compared for means of a polarized screen, as evidenced by the comparison of the properties of the nitrated layer on the corners of a specimen specially designed for this purpose [14].

2. Working method and materials
In order to perform the experiments the following steel type was used: 38MoCrAl09 STAS 791/88 (equivalent W1.8507, DIN 17210-69 type 34CrAlMo5). We used this steel because this is the only Romanian steel with special nitriding purpose [15-21]. The test sample was subjected to martensitic and high-tempering treatments. The chemical composition of the W1.8507 steel specimens used for the experiment was determined using the Foundry Master spectrometer and is shown in Table 1.

| Element | Fe | C  | Cr  | S  | Mo | Ni  | Al  | Cu | W  | Ti |
|---------|----|----|-----|----|----|-----|-----|----|----|----|
| Percent | balance | 0.40 | 1.37 | 0.02 | 0.15 | 0.12 | 1.07 | 0.12 | 0.06 | 0.01 |

Experiments were performed without a polarized screen (Figure 1.a) using a 50 mm diameter anode to achieve stable discharge, and polarized screen experiments (Figure 1.b), represented by a cathode grid polarized with a diameter of 65mm, and 200mm diameter anode.

The treated specimens were analyzed by SEM microscopy, chemical analysis by the EDX method, and microhardness tests on metallographic constituents.

To analyze the effect of the polarized screen on the sharp edges of the pieces, a straight triangular prism specimen was used. The base of the specimen is cylindrical in shape with a diameter of 8 mm. The upper part was machined by milling, resulting in the specimen in Figure 2.

The prismatic part is based on a rectangular triangle with the angles of 30 °, 60 ° and 90 °.

![a)](image1) ![b)](image2)

**Figure 1.** Images of the nitriding enclosure: a) Nitriding without polarized screen, detail aspects of the part after treatment; b) Polarized screen nitriding, detail aspects of the part after treatment.

The discharge parameters for the two types of treatments are shown in Table 2.

| T[°C] | P[torr] | U_k [V] | I_k [A] | I_d[A] | t[h] |
|-------|--------|---------|---------|--------|------|
| No screen | 500 | 1 | 480 | 0.1 | - | 7 |
| Screen | 500 | 2 | 470 | 0.18 | 0.04 | 7 |
Polarized screen ionic nitriding uses a 65 mm diameter cylindrical screen, stainless steel, open at both ends, upper and lower, and a cylindrical anode of 200 mm in diameter. We also used the same type of specimen as in the case of ionic nitration without a polarized screen. Dimensions are detailed in Figure 2.

3. Results and discussions

3.1. Section SEM analysis
In order to analyze the effect of the polarized screen on the sharp edges of the parts and the structural changes in the outer thin layers, we performed sectional analysis on characteristic areas for the three angles of 30˚, 60˚ and 90˚ as well as the sides.

![Figure 2](image)

**Figure 2.** Sequence of specimens preparation for metallographic analyzes: a) Plasma nitrided specimen; b) sectioned specimen perpendicular to the edges of interest; c) metallographic blueprint with the positioning of the analyzed areas.

In order to perform the microstructural analysis of the outer layer of the specimen we performed the sectioning of the specimen perpendicular to the vertical edges of the right triangular prism (Figure 2.b). Morphological analyzes were performed using the microscope in the areas noted in Figure 2.c. S.E.M. images of the studied areas on the sharp angled sample section, nitrated without polarized screen, from areas A-C, according to the scheme in Figure 2.c., are shown in Figures 3, 5, 7.

![Figure 3](image)

**Figure 3.** SEM micrographs of classical nitride test specimen in zone A at magnifications: a) 1000x; b) 2500x; c) 5000x; d) the mass concentration of the elements per section.

| El. | [%] |
|-----|-----|
| Fe  | Bal.|
| N   | 7.98|
| C   | 2.19|
| Al  | 1.86|
| Cr  | 2.22|
| Mo  | 0.77|
Figures 4, 6, and 8 presents S.E.M. images for the polarized screen nitrided sectioned sample, images taken in the same area of Figure 2.c.

**Figure 4.** SEM micrographs of polarized screen nitrided specimen in zone A at magnifications: a) 1000x; b) 2500x; c) 5000x; d) the mass concentration of the elements per section.

**Figure 5.** SEM micrographs of the classical nitrided test specimen in zone B at magnifications: a) 1000x; b) 2500x; c) 5000x; d) the mass concentration of the elements per section.

**Figure 6.** SEM micrographs of polarized screen polarized nitrided specimen in zone B at magnifications: a) 1000x; b) 2500x; c) 5000x; d) the mass concentration of the elements per section.
Figure 7. SEM micrographs of the classical nitrided test specimen in zone C at magnifications: a) 1000x; b) 2500x; c) 5000x; d) the mass concentration of the elements per section.

![SEM micrographs of classical nitrided specimen](image1)

| El. [%]              |
|----------------------|
| Fe       | Bal.  |
| N        | 22.38 |
| C        | 2.89  |
| Al       | 1.27  |
| Cr       | 1.36  |
| Mo       | 0.46  |

Figure 8. SEM micrographs of polarized screened nitrided specimen in zone C at magnifications: a) 1000x; b) 2500x; c) 5000x; d) the mass concentration of the elements per section.

![SEM micrographs of polarized nitrided specimen](image2)

3.2. Section EDX analysis

Analyzes for the chemical composition of the surface obtained by sectioning were performed in the same areas as the SEM micrographs.

![EDX analysis](image3)

Figure 9. Analysis of the distribution of N, Al, Cr and Mo elements along a line in the 90-degree angle zone after the nitriding treatment: a) without polarized screen; b) polarized screen.

4. Results interpretation

4.1. Nitrided specimen without grid

Analysis of SEM images of plasma nitrided samples without polarized plasma shows the presence of severe irregularities (adhesions, rust, microcracks and microbumps). This is caused by the
phenomenon of cathodic spraying which, due to its high intensity, creates rough surfaces. The most visible irregularities occur in the corner and edge areas.

Edge effect occurs due to the intersection of negative lights and their supply energies on corner and edge areas. This effect increases the temperature at the corners, which influences the mass transfer, creating uneven diffusion areas and too much decarburation. The unevenness of the diffusion layer obtained in the corner areas can be seen in Figures 3, 5, 7.

Superficial decarburization favorable to nitration is due to the chemical spraying effect around the temperature of 600 °C, meaning moderate, with the formation of chemical compounds of the type CnHm in the plasma volume. They create vacancies in the crystalline network to facilitate the penetration of nitrogen atoms. Instead, in classical nitration, in the edge areas the physical spraying process is intensified and the chemical spraying decreases. Thus, superficial decarburization greatly increases, and there is a risk of thin and fragile nitride forming, which reduces the mechanical properties of the layer obtained.

EDX analysis on the polarized screened prism specimen shows a fluctuating N content on the surface ranging between 3.47% and 8.09%, due to the edge effect, which leads to a low absorption of N on the surface. In the section, the EDX analysis shows an N content between 9.43% and 6.11%.

4.2. Polarized Screen nitrided sample
In the case of prismatic polarized screen plasma nitrided samples, the surface SEM photographs, presents edges and corners areas with a coral deposition, uniformly and relatively compact on the surface.

The quality of the deposition is due to a much less intense and evenly distributed physical spray uniformly spread across the surface. This characteristic of physical spraying is due to a uniform distribution of the electric field around the workpiece by the presence of the polarized screen. It flattens the electric field on the surface of the workpiece by distributing the same amount of energy over a larger surface without concentrating it on sharp edges or corners. Due to the decrease in physical spraying and the increase in chemical spraying nitrogen diffuses on a greater depth inside the workpiece. Thus, the edge effect is diminished and a uniform diffusion layer with a higher thickness is obtained.

The use of the polarized screen also has a beneficial effect on the roughness and the resulting surface has a relatively small number of apparent defects of the following types: adhesions, roughness, exfoliations and unevenness of the formed layer, as seen in Figures 4, 6, 8.

From the SEM photographs of the microstructure obtained in section, it is observed that the thickness of the diffusion layer is relatively uniform, as opposed to classical nitriding treatment, when the angular areas exhibit a thickening of the diffusion layer and the adjacent areas a thinning thereof. This is also due to the protection provided by the polarized screen at the electric arc, which leads to pinching of the parts and local overheating, and can change the diffusion parameters.

5. Conclusions
The comparative analysis of the results obtained with ionic nitration with the two techniques (classic and polarized screen) for the prismatic pieces, with angles of 30˚, 60˚ and 90˚, led to the following observations, which are presented below:

Analysis of SEM images of plasma nitrided samples without polarized screen shows the presence of severe irregularities (adhesions, rust, microcracks and microbumps). This is due to the phenomenon of cathodic spraying which, due to its high intensity, creates rough surfaces. The most visible irregularities occur in the corner and edge areas.

In the case of prismatic ionic nitride samples with the help of the polarized screen, SEM photographs of surfaces, edges and corners are observed to form a uniformly and relatively compact layer of coral surface. Using the polarized screen also has a beneficial effect on the roughness, the resulting surface has a relatively small number of apparent defects of the following types: adhesion, roughness, exfoliation and unevenness of the deposition.
Edge effect occurs due to intersection of negative lights and their supply energies on corner and edge areas. This effect increases the temperature at the corners, which influences mass transfer, creating uneven diffusion areas and far too intense decarburization.

Superficial decarburization, favorable to nitration is due to the chemical spraying effect around the temperature of 600 °C, so moderate, with the formation of chemical compounds of the type CnHm in the plasma volume. They create vacancies in the crystalline network to facilitate the penetration of nitrogen atoms. Instead, classical nitriding, in the edge areas, intensifies the physical spraying process and lowers the chemical spraying. Thus, superficial decarburization is greatly increased and there is a risk of formation of thin, fragile nitrides, which reduce the mechanical properties of the layer obtained.

EDX analysis on the polarized screened prism specimen shows fluctuating N content on the surface, ranging from 3.47% to 8.09%, due to the edge effect, which leads to a low absorption of N on the surface. In the section, the EDX analysis shows an N content between 9.43% and 6.11%.

In the case of polarized screening nitriding, the layer quality is due to a much less intense physical spraying and evenly spread across the surface. This characteristic of physical spraying is due to a uniform distribution of the electric field around the workpiece by the presence of the polarized screen. It flattens the electric field on the surface of the workpiece by distributing the same amount of energy over a larger surface without concentrating it on sharp edges or corners. Due to the decrease in physical spraying and increased chemical spraying, nitrogen diffuses to a greater depth inside the workpiece. Thus, the edge effect is diminished and a uniform diffusion layer with a higher thickness is obtained.

From the SEM photographs of the microstructure obtained in the section it is observed that the thickness of the diffusion layer is relatively uniform, unlike in the case of classical nitriding treatment, when the angular zones exhibit a thickening of the diffusion layer and the adjacent areas a thinning thereof. This is also due to the protection provided by the polarized screen at the electric arc, which leads to pinching of the parts and local overheating, and can change the diffusion parameters.

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