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Analysis of the role of the active screen in the plasma nitriding process

Thesis Booklet

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Budapest, 2019.

1. INTRODUCTION

In this thesis booklet, I review my doctoral thesis and I give a summary of the most important contents. This summary contains the outcome of the literature research, the objectives of my scientific research and also the description and evaluation of the research tasks defined by these objectives. In this theses booklet, according to the formal requirements of a doctoral thesis, I presented my scientific results in a uniform, self-explanatory system.

In this summary, I intend to introduce, explain and justify and outline, within their domain of validity, the results of my scholarly research work, which can be considered as independent initiations, formulated in thesis statements. This thesis booklet structure makes it possible to formulate the thesis statements clearly and as briefly as possible.

One of the most commonly used methods in surface treatments is the nitriding. Its aim is to create a hard, wear resistant layer on the surface of the parts. There are many variants known in the world, the oldest is the gas nitriding. As the world is constantly changing and evolving, new approaches treatments can be applied at surface. Over the years, many influencing factors force the industry and researchers to focus not only on the characteristics required of a product, but also on the economics and product quality requirements. As a result of these trends, over the decades, time has also forced the development of gas nitriding to develop further, and also new processes appeared such as salt bath nitriding and carbo-nitriding. By reducing the nitriding time, salt bath nitriding process is still popular in the industrial environment. Not only the hardness and thickness of the formed nitride layer can be taken into account, but also the post-oxidation possibilities provided by the various salt baths, which made it aesthetically important surface treatment.

However, as with all things, there are drawbacks of this method. Nowadays, it is no longer enough for a procedure to be economical, but it also has to work according to environmental requirements. This very specific requirement, namely, elimination of the cyanide content of the salt bath, encouraged the development of a plasma nitriding process.

During the literature research and under the guidance of Professor *Joseph Blucher*, I came across technical questions in the field of plasma nitriding for which only relatively imperfect results and answers can be found. Thus, in the internal system of the research topic, the specialty is that I focus on the investigation of the surface structure and the material of the active screen in active screen plasma nitriding.

To get to know the plasma nitriding process, direct current and active screen plasma nitriding was compared to explore the properties of the active screen. In the following, I will mention these shortcomings and initial results, and then I follow with the details of my research.

2. MAIN RESULTS OF LITERATURE RESEARCH

Nitriding is a thermochemical surface treatment process in which nitrogen diffuses into the surface and nitride phases can be formed with the present atoms which creates a hard, wear resistant layer [1, 2]. Nitrogen is dissolved in iron solid solutions as an interstitial alloying element and is capable of reacting and forming compounds with individual alloys and constituents of steel. The vibrational motion of the iron atoms in the steel lattice is enhanced with increasing the temperature, allowing the nitrogen atoms to diffuse into the iron lattice. After the saturation of the solid solution, the nitrogen is also able to form a compound (Fe_3N, Fe_4N) with the iron atoms, which forms a layer of higher hardness than the action of nitrogen dissolved in the solid solution on the surface of the part [3, 4]. This iron nitride formation mechanism is also characteristic of salt bath and gas nitriding.

Within the plasma nitriding process, three process variants can be distinguished: direct current plasma nitriding (DCPN), active screen plasma nitriding (ASPN), and in addition to a cathodic screen, a so-called "cathodic bias" applied separately to the workpiece, which is called active screen biased plasma nitriding (ASBPN) which name was initiated by me.

During the active screen plasma nitriding the plasma is not formed directly on the workpiece, but on a loose grid screen which is around the workpiece; which becomes an active screen by being connected to the DC circuit as a cathode, i.e. at negative potential, in the plasma nitriding process and, becomes an active maintainer of the electrical discharge generated by the electric field. As a result, the dissociated atoms from the gas molecules and the positively charged ions formed therefrom bombard the screen. The iron ions deposited therein collide with the nitrogen ions in the plasma space to form iron nitride along the surface of the active screen. There are some of these nitrides that adhere to the surface of the active screen, but there are also – and they are overwhelming majority – that fly through the loose, sloping geometry of the screen and adhere to the workpiece to be treated, which is heated by the heat radiation of the active screen and the heat of the continuous plasma flow maintained by the gas supply and the vacuum pump [5, 6].

2.1. Role of the surface construction of the screen

No ideal size or geometry has been developed for the size and shape of the screen. Various studies explain how influences the distance between the workpiece and the screen, or the perforating of the screen the nitride layer formation and layer properties. There is no disclosure in the literature as to whether the active screen should cover the workpiece from all sides or whether the cover can be omitted or its design may be altered relative to the construction of its side.

2.1.1. Role of the top lid of the screen

Ahangarani et al. [7]observed that thicker layer thickness was created with 6 mm hole size screen and their top lid while the layer thickness was not changed with 8 mm hole size and both type of top lid. The thickest layer was created by nitriding with smaller hole size screen which was explained by the authors that here is the smallest chance of active particles (ions and molecules dissociated from molecules) to go through the screen holes toward the surface of workpiece.

The influences of the top lid with large hole size was investigated by *Gallo and Dong* [8].

Difference was found between the hardness of the covered and the uncovered area. The hardness of the area under the hole was third of the area covered by the mesh. In addition, a hardly appreciable glossy and matte discoloration was visible.

2.1.2. Role of the hole size of the screen

Ahangarani [9] improved his research, also worked with two different hole size he stated that the nitrided layer thickness was increased with the increasing of the temperature, moreover the hole size of the screen has no effect to the formed layer thickness. This statement is true for nitriding with high nitrogen content gas mixture, but the layer thickness was increased 2 times higher with low nitrogen content gas mixture and at lower temperature.

In some researches not the exact surface structure of the screen was determined as hole size, distance between hole sizes, but the ratio of the open area of the plate was used, which this is the ratio of the uncovered area. It called open are fraction or open are ratio [8]. This is the percentage of that area which is not filled by metal from the screen.

Nishimoto [10]used an even more special procedure for the open area ratio. Not only the hole size was changed, but also the geometry of the hole, because he did not work with circle cross-section holes, but rhombus shape was used and a big gap (or gate) was left in the cylinder screen. It can be stated that the nitrided layer thickness was decreased with the increasing of the open area ratio. The maximum hardness (780 HV) was reached with the lowest open are ratio, but the hardness of the other samples treated by the two other screens was similar, which means a significant decrease. The surface roughness had a similar tendency: the surface roughness was decreased with the increasing of the open area ratio. These results were explained by the author, the amount of the active particles can be increased because of the large open area ratio.

2.1.3. Effect of the screen material

Li [6]used copper and titanium for the material of the top lid in his research. The hardness of the sample was not changed with copper top lid, but hardness was increased with 100 HV when titanium top lid was used. Copper and titanium were found on the surface after the composition analysis.

Yazdani et al. [12]nitrided aluminium sample with steel screen. Fe_3N was formed on the surface, which came from the iron particle of the screen around the sample.

From the experimental series of *Naeem*[13]new results can be get to know about the duplex surface treatment. From the variants of the duplex surface treatments he used two different materials of screen one after another (aluminium and austenitic stainless steel). Based on the XRD-results, AlN was always created when aluminium screen was used.

It is resulted from the previous statements that the sputtered particles of the active screen can deposit on the surface of the workpiece, moreover, it can create nitride with the nitrogen.

3. REFERENCES FOR THE CHAPTER 2

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4. RESEARCH OBJECTIVES

The basics of plasma nitriding were laid more than 50 years ago. This type of surface treatment method is less widespread in Hungary, but plasma nitriding is preferred in other countries to increase the wear resistance of automotive components. Particularly intensified research has been on the topic since the spread of active screen plasma nitriding, which is obviously driven by industrial needs. Literature research has revealed that there are still influencing factors whose effects have not or not been fully investigated in the nitriding process.

Based on my experience in literature research and the suggestions of professor *Blucher* and my supervisor, I have compiled the aims and tasks of my research work according to the following structure. Thus, my research program was primarily focused on the role of the active screen. In addition, I wanted to play an important role in acquiring new knowledge on the properties of the nitride layer by applying special surface analytical methods that have so far been barely used in this field of research. The aims of my research work are briefly as follows:

- Comparison of the various plasma nitriding processes, especially for the role of the top lid of the active screen.
- Investigation of the shape and the surface structure of the screen.
- Investigation of the influence of the screen's material.
- Investigation of different type of nitrogen content measurements.

5. OVERVIEW OF THE RESEARCH

My research programme was divided for three main topics in accordance with the objectives as follows:

1) comparison of the various plasma nitriding processes
2) influences of the hole size and the open area ratio of the active screen
3) role of the screen's material.

In the following, the experimental details and the measurements method of my research were summarized together with the analysis and evaluation of results. These results provide a sufficient base and justification for the thesis statements.

5.1. Comparison of the various plasma nitriding processes

The material used in this study was $\varnothing 20 \times 6$ mm, grade C45E unalloyed steel, the average hardness of the base material was measured as 250 ± 22 HV0.05. The samples were plasma nitrided in 490 °C, for 4 hours on 2,8 torr pressure with two different gas mixtures, one is a low nitrogen content (25 % N_2 + 75 % H_2), the other one is a high nitrogen content (95 % N_2 + 5 % H_2) gas mixture. Various arrangements of the plasma nitriding equipment are shown in Fig. 1. The active screen plasma nitriding experiments were made with and without the top lid of the screen.
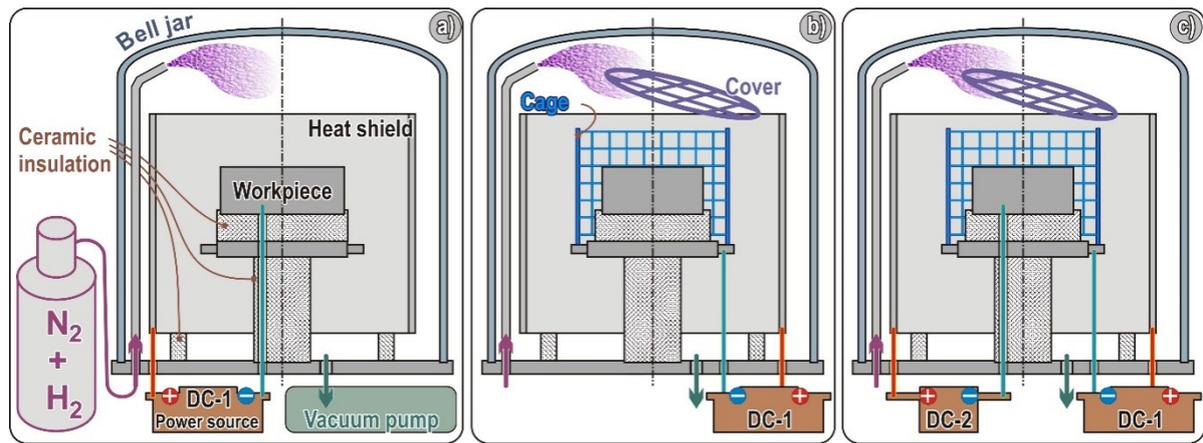
Figure 1.   Plasma nitriding arrangements (a) DCPN, (b) ASPN, (c) ASBPN

It is necessary to introduce an effective compound layer thickness for the evaluation of the metallographic layer thickness which is explained in Fig. 2. Its justification is given by the fact that in the cross-sectional images the boundary of the compound layer is uneven. The entire area of the compound layer was taken into consideration from the metallographic image for the evaluation. In the following it is called for compound layer thickness based on this method, 3 different metallographic images were used for determine the average effective compound layer thickness.
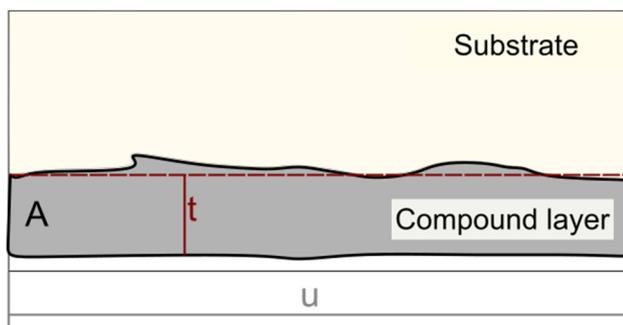
1. step: capturing the contour of the compound layer
2. step: calculating the scale-limited area A in an image analysis program
3. step: calculation of the t effective compound layer thickness from the ratio of the A area and the u width

Figure 2.   Definition of the effective compound layer thickness, t

The thinnest layer thickness, measured by scanning electron microscope (SEM), was formed independently from the composition of the gas mixture with ASPN arrangement. The layer thicknesses were almost the same with DCPN and ASBPN arrangements, but the thickest layer was formed with the high nitrogen content gas mixture in ASBPN arrangement. The top lid did not influence the thickness.

My 1ˢ thesis statement was defined on the base of those summarized conditions of the measurements and experimental details of my research, which are seen in the chapter 5.1.

With each gas mixture, the ASPN samples had the lowest hardness values, which were between 510 and 560 HV0.05. For DCPN and ASBPN, the hardness increased more than twice, but with the use of the active screen, it has been just slightly doubled.

To comparing the formed phases after the nitriding with both gas mixture, Fe_3N was created on all of the sample with 95 % N_2 + 5 % H_2 gas mixture. In the highest amount was formed on the compound layer of the ASBPN sample, while with low nitrogen content gas mixture only Fe_4N was created. It is observed in the measurement results, the phases are the same with or without the top lid of the active screen.

My 2ⁿᵈ thesis statement was defined on the base of those summarized conditions of the measurements and experimental details of my research, which are seen in the chapter 5.1.

The nitrogen depth profile (from the surface to the substrate) was measured with glow discharge optical emission spectroscopy (GDOES). I defined an evaluation method for the depth profiles to determine the compound layer thickness, which initial and end point are demonstrated in the Fig. 3.

My 3ʳᵈ thesis statement was defined based on the summarized condition of the measurements and experimental details of the research, which are seen in the chapter 5.1.
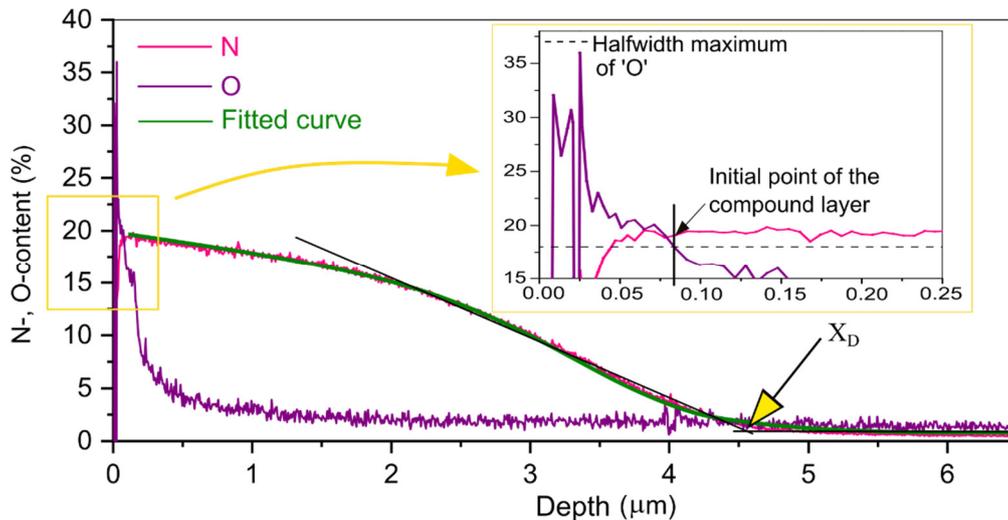

Figure 3.   Determination of the initial and end point of the thickness of the compound layer on the GDOES depth profile of nitrogen and oxygen

Equation (1) can be properly fitted on the nitrogen depth profile, and the end point calculated according to Eq. 2.

$$N(x) = a\mathrm{e}^{-bx} + \frac{c}{2}\left(1 + \tanh\left(-\frac{x+d}{\varepsilon}\right)\right) + g \qquad (1)$$

$$x_{\mathrm{D}} = d + \varepsilon - 2\,a\varepsilon\frac{b\varepsilon - 1}{2\,ab\varepsilon + a\,\mathrm{e}^{bd}} \qquad (2)$$

where,

$N(x)$ – nitrogen content
x – location coordinate (in diagram: depth from the surface)
a – abscissa of the initial point of the exponential curve
b – initial decay in $x = 0$ point
c – abscissa of the initial point of the sigmoid curve
d – inflexion point of the sigmoid curve
ε – smoothness parameter of the sigmoid curve which determine the tangential of the inflexion point
g – correction factor
x_D – end point of the compound layer

5.2. Effect of the surface construction of the screen to the formation of the nitrided layer

I defined an accurate characterization for the surface construction of the screen, in which the open area ratio is one of the main concepts. The *open area ratio* is defined as a missing or removed part of the metal plate calculated in ratio. The open area fraction can be between 0 and 100%. The *perforating* is the missing part of the plate.

The material used in this study was ⌀20×6 mm, 42CrMo4tempered steel, the average hardness of the base material was measured as 420±45 HV0.01. The constant temperature of the samples was regulated by the applied voltage, which was proportional to the open area ratio (the percentage of open holes in the active screen), as can be seen in Table 1.

Table 1. The applied plasma nitriding conditions and the surface structure of the screen

Sample no.	Hole size (mm)	Open are ratio (%)	T (°C)	t (hour)	p (torr)	Gas mixture (%)
d4.5	4.5	50				
d8	8	61				
d12	12	50	490	4	2.8	75 % N_2 + 25 % H_2
d18	18	74				
d25	25	61				
d45	45	87				

The layer thickness does not show any significant difference for the perforating, but it has a connection with the open are ratio. In contrast, the perforating and the open area ratio did not influence neither the maximum hardness, nor the hardness and the thickness of the diffusion zone.

Because of the sameness of the compound layer thickness the iron phase under the nitrided layer was in the same amount in all of the samples. It did not influence the determination of the iron-nitride phases. While the hole size and the open area ratio did not influence the layer thickness, until then it has an effect for the relative amount of the iron-nitride phases. The γ'-phase (Fe_4N) is decreasing, the ε-phase (Fe_3N) is increasing with the decrease of the open area ratio

Atomic force microscope was used for measurements of the surface roughness. The peaks and dales, which is a typical form of the surface texture, were in different amount and dispersion. Comparing the skewness (S_{sk}) and the open area ratio, a close correlation is shown.

The 4th, 5th and 6th thesis statements were defined on the base of those summarized conditions of the measurements and experimental details of my research, which are seen in the chapter 5.2.

5.3. The effect of the screen material to the composition of the nitrided layer

The material used in this study was ⌀20×6 mm, 42CrMo4 tempered steel, the average hardness of the base material was measured as 420±45 HV0.01.The active screen used for the plasma nitriding was made from a 1.0330 type unalloyed steel, hole size was 5 mm, the distance between the holes was 8 mm, the dimensions of the screen were ⌀140×85 mm. Two electroplated coating materials were used for the screen: nickel and

chromium. The samples were nitrided for 5 hours at 510 °C using 75 % N_2 + 25 % H_2 gas mixture with 2.8 torr gas pressure. The screens were used twice in a row.

After nitriding, the sample surface appearance remained polished as it was at the end of the preparation. Compound layer was not formed on any surfaces, the hardness was the same with the original.

The so-called maximum height of the scale-limited surface is within 70 μm. It had to be considered during the evaluation of the depth profile of the nitrided layer.

Secondary neutral mass spectrometry (SNMS) depth profiling was used to analyse the Fe, Cr, Ni, C and O concentration changes from the sample surface, the nitrogen content was measured with X-ray photoelectron spectroscopy (XPS). The depth of the deposited Ni was ~290 nm after the first, and ~420 nm was after the second nitriding cycle with the same Ni-coated screen. The bonding of the N was investigated in the intersection point of the Ni and Fe depth profiles. The N did not bond with the Fe, so iron-nitride did not form. The most of the detected nitrogen (84 %) was molecular (N_2) on the sputtered layer. In this depth N-C were presented in few amount (16 % and 23 %), but it has not a hardening effect to the surface.

The SNMS profile of the Cr and O after the ASPN process with Cr screen was same, moreover the amount of the O was higher. It means, the Cr created a chromium-oxide layer on the surface. The thickness of the chromium-oxide layer was ~168 nm after the first, ~198 nm was after the second nitriding cycle with the Cr screen. XPS-analysis shows the N create a bond with the Fe, but this amount is not enough to form an Fe_3N or Fe_4N phase.

It can be stated from the comparing these three types of measurement method, that the particle from the screen's surface can be deposited in the surface of the sample to create different surface texture, but did not react with the N and it block the nitriding of the steel.

The 7th and the 8th thesis statements were defined on the base of those summarized conditions of the measurements and experimental details of my research, which are seen in the chapter 5.3.

6. RESULTS OF THE RESEARCH IN THESES POINTS

In Chapter 1-5. I summarized my research results in a unified, self-explanatory system, which I achieved in the course of my research work, and described in detail in my doctoral dissertation. This summary is also intended to introduce, explain, and justify the thesis-like presentation of my independent scientific work, that is, to formulate it in itemized, thesis statements, by assigning my source publications. This thesis booklet structure makes it possible to formulate my thesis statements in a genre-specific (i.e. clear and as short as possible) manner.

Thesis 1: [S2] [S10]

I elaborated an evaluation method for the measuring of the compound layer thickness from metallographic examination images. The essence of this new method is to define the effective compound layer thickness.

Thesis 2: [S1] [S7] [S8]

The use or disregard of the screen cover results the same kind nitrided layer, that is It does not influence on the layer thickness, hardness and the composition of the iron-nitride phases of the compound layer.

Thesis 3: [S2] [S8]

I elaborated an evaluation method for the determination of the compound layer depth profile measured by GDOES-analysis of the unalloyed and low alloyed steel. The thickness of the compound layer can be determined within 0,2. The initial point of the compound layer is at the half-width maximum of the oxygen depth profile, and the end point is at the intersection of the tangent of the nitrogen profile's inflexion point and the baseline approaching to zero.

Thesis 4: [S3] [S4]

The hardness of the nitrided layer remains with both of the changing of the perforating and the open area ratio of the screen, namely these two surface construction features do not influence the hardness of the nitrided layer.

Thesis 5: [S4] [S5]

The open area ratio of the screen influences the formed phases in the nitrided layer. The γ'-phase (Fe_4N) is decreasing, the ε-phase (Fe_3N) is increasing with the decrease of the open area ratio.

Thesis 6: [S3] [S4]

The skewness (S_{sk}) is increasing with the increase of the screen open area ratio.

Thesis 7: [S6] [S7] [S9] [S10]

In the case of active screen plasma nitriding with a nickel-coated screen, only nickel from the surface of the screen is adhered to the workpiece surface at hundreds of nm thickness and iron nitride phases are not formed.

Thesis 8: [S6] [S9] [S10]

During anew use of a nickel-coated screen for active screen plasma nitriding, the nickel will be deposited in a thicker layer on the workpiece surface than when it was first used. This phenomenon is a consequence of the porosity formed during the first application of the nickel plating.

7. OWN PUBLICATIONS FOR THE THESIS STATEMENTS

[S1] Dobránszky János, **Kovács Dorina**: Szemlézés a rozsdamentes acélok gyártásának euró-pai kutatásaiból. Bányászati és Kohászati Lapok – Kohászat, 149,1, 6-10 (2016)

[S2] **Kovács D**., Kemény A., Dobránszky J., Quintana I.: Effects of plasma nitriding on tem-pered steel. IOP Conference Series: Materials Science and Engineering, 426, 012027 (2018)

[S3] **Kovács D**., Kemény A., Bonyár A., Dobránszky J.: The effects of screen sizes on the sur-face properties of tempered steel treated by active screen plasma nitriding. IOP Conference Series: Materials Science and Engineering, 416, 012040 (2018)

[S4] **Kovács D**., Dobránszky J., Bonyár A.: Effect of different active screen hole sizes on the surface characteristic of plama nitrided steel. Results in Physics, 12 (2019) 1311–1318.
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[S8] **Kovács D**., Quintana I., Dobránszky J.: Effects of different variants of plasma nitriding on the properites of the nitrided layer. Journal of Materials Engineering and Performance, 28 (2019:9) 5485–5493.

[S9] **Kovács D**., Bonyár A., Dobránszky J., Fodor T., Takáts V.: Influence of the material of ac-tive screen on the surface properties of plasma nitrided low alloy steel. Surface and Coat-ings Technology. – beadva

[S10] **Kovács D**., Szabó A., Kemény A.: Az aktív ernyő anyagának szerepe a plazmanitridálás során. Acta Materialia Transylvanica. – bírálat alatt