

PLASMA NITRIDING OF TITANIUM ALLOYS WITH INDIRECT PLASMATRON

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Abstract: *This article presents an experiment which consists of plasma gas nitriding of titanium alloy TI-6Al-4V. The purpose of the experiment is to improve tribological properties of titanium alloys in working conditions under sliding contact. Data obtained from the experiment is necessary for creating a technology for obtaining surface layers of a certain thickness.*

Titanium alloys are characterized by low density, high strength and corrosion resistance. This makes them an attractive material for constructing various details in shipbuilding. However, they have poor tribological properties in sliding contact, high and unstable friction coefficient, adhesion wear, and a strong tendency to be scraped when in contact with other materials. These characteristics indicate poor abrasion and adhesion resistance to wear and severely restricts their tribological use. Considerable efforts are being made to develop titanium alloy processing technologies in order to obtain a long lasting protective layer and to extend the spheres of its application. Different types of surface treatment are used to achieve the desired properties of the material. [4,6,8,10]

Plasma gas nitriding is a widely used method for increasing surface hardness and improving the tribological properties of titanium alloys. The process is characterized by diffusion of nitrogen atoms into the metal surface in the presence of a plasma medium. The nitrogen is fed into a mixture of argon and nitrogen. Nitrogen atoms (atomic mass $14.0067 \text{ g} \cdot \text{mol}^{-1}$) are much smaller than those of titanium (atomic weight $47.8670 \text{ g} \cdot \text{mol}^{-1}$), which allows them an easy penetration into the material and forming a solid solution. The concentration of nitrogen on the surface is high. Upon formation of the surface layer, the nitrogen atoms diffuse into the structure. Under certain conditions, they increase the surface hardness and greatly improve the tribological characteristics of titanium alloys for a short time. [5,7,9]

Conducting experiments and studying the structure and properties of titanium alloys after plasma gas nitriding contributes significantly to the expansion of their application.

The purpose of the experiments described in this article is to create and study a technology for obtaining a surface layer of a certain thickness.

Experiment Planning.

A certain amount of planning should be done before studying the structure and properties of titanium alloys and conducting a specific experiment. For this purpose, the researchers chose Rechtschaffner's methodology for three variables with 15 attempts. The main reason for choosing this methodology is its accessibility and simplicity. It gives the opportunity to get a real result of the experiment with a smaller number of attempts compared to the complete combination of three factors with three variable values (27). [1,2,3]

A centrally rotating composite design is used, while the output magnitude being expressed by a second order empirical polynomial:

$$(1) y = b_0 + \sum_{i=0}^k b_i X_i + \sum_{1 \leq i < j}^k b_{ij} X_i X_j + \sum_{i=1}^k b_{ii} x_i^2$$

where:

- b_0, b_i, b_{ij} and b_{ii} are regression coefficient;
- X_i and X_j are the values of the input quantities

The number of attempts in the experiment is determined by the expression:

$$(2) N = 2^k + 2k + n_0 = n_k + n_\alpha + n_0$$

Table 1 Input factors			
# of exp.	Factors		
	X1	X2	X3
1	0	0	0
2	1	1	1
3	1	0	0
4	1	-1	-1
5	-1	-1	1
6	0	0	-1
7	-1	0	0
8	0	0	1
9	1	1	-1
10	-1	1	1
11	1	-1	1
12	0	-1	0
13	0	1	0
14	-1	1	-1
15	-1	-1	-1

Where

- k is the number of parameters;
- n_0 is the number of repetitions in the middle level;
- n_α is the number of experiments in the central axes

For the specific case $k = 3$ and $n_0 = 1$.

- X1 - amperage I, A;
- X2 - amount of spent plasma forming gas N₂, l/min;
- X3 – nitriding of the sample time, t/min.

Input factor values are selected as follows:

- for X1 300, 400 and 500 A /-1,0,1/;
- for X2 2, 4 and 6 l/min /-1,0,1/;
- for X3 1, 2 and 3 min /-1,0,1/.

Table 1 shows the input factors and the number of attempts in the experiment with the specific values of the three variables for each of them.

Experiment and results



Fig. 1. Ti 64 sample, prepared for nitriding

The samples used in the present experiment are from Ti-6Al-4V alloy with dimensions 4x8x15 mm. The nitriding process is performed with an indirect plasmatron at the following input parameters:

- voltage– 60 V;
- amount of spent plasma forming gas Ar - 20 l/min;
- distance from the nozzle end to the sample – 100 mm;

According to Rechtschaffner's plan an experiment was conducted, with the input factors - current force I, A, amount of gas consumed N₂, l/min, nitriding time t,min/ - are different for each sample, according to.

Also presented are the results obtained from the experiment (table 2): phase composition, the depth of the obtained layer, the color of the sample.

Table 2 Experiment results							
#	Factors			Power, kW	Phase composition	Layer thickness, μm	Color
	Current A	N ₂ , l/min	Time, min				
1	400	4	2	20.4	TiN	22	golden
2	500	6	3	25.5	TiO ₂ ,TiN	40	white
3	500	4	2	25.5	TiO ₂ ,TiN	36	white
4	500	2	1	25.5	TiO ₂ ,TiN	28	white
5	300	2	3	15.3	TiN	25	golden
6	400	4	1	20.4	TiN	26	golden
7	300	4	2	15.3	TiN	14	golden
8	400	4	3	20.4	TiO ₂ ,TiN	36	white
9	500	6	1	25.5	TiO ₂ ,TiN	20	white
10	300	6	3	15.3	TiN	24	golden
11	500	2	3	25.5	TiO ₂ ,TiN	60	white
12	400	2	2	20.4	TiN	32	golden
13	400	6	2	20.4	TiN	20	golden
14	300	6	1	15.3	TiN	14	golden
15	300	2	1	15.3	TiN	8	golden

It is important to note that the input factors of the nitriding mode affect the depth of the resulting nitrided layer.

After the experiment, the samples were prepared for microstructural analysis by grinding with a set of metallographic sandpaper (120, 200, 400, 600, 800, 1200, 2000) and polishing on a Metascan grinding machine.

All samples were observed with a Neophot 2 metallographic microscope and photographed with a Cannon powershot A 280 camera.

The samples have been measured for microhardness with a PMT 3 microhardness tester. The measurements were carried out in the nitrogen layer in three rows of five measurements of the depth of the surface. The mean values of the microhardness of the samples in each of the layers are shown in Table 3. Highest hardness is achieved with the nitriding modes 11, 12, 13, 14 and 15 (630 - 700 HV) and the lowest hardness - with modes 3, 4 and 5 (411 - 422 HV).

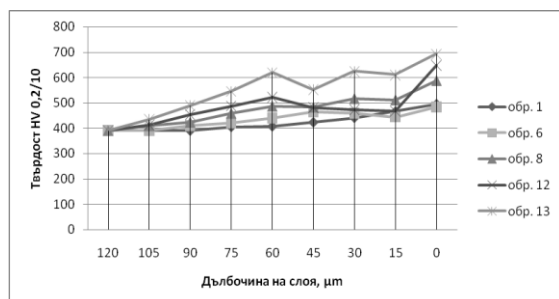


Fig. 2. Changes in microhardness in samples nitrided at plasmatron amperage 500A

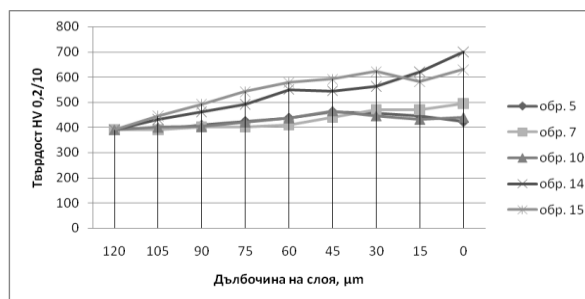


Fig. 3. Changes in microhardness in samples nitride at plasmatron amperage 400A

Microhardness measurement results of the samples are plotted in Fig. 2, 3, and 4. The criterion on which the nitrogenized samples are deposited is the amperage (A).

From the analysis of the results obtained in Fig. 2, 3 and 4 it was determined that the current variation in the 300-500A range did not significantly affect the microhardness of the nitrided layer. The other two input factors (nitriding time and nitrogen consumption) have a more significant effect.

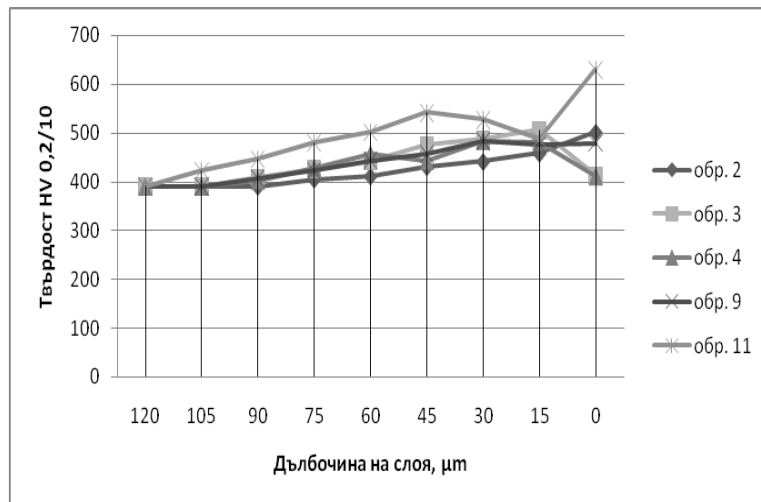


Fig. 4. Changes in microhardness in samples nitrided at plasmatron amperage 300A

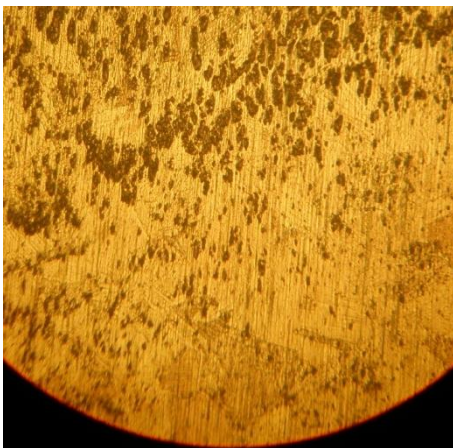


Fig. 5. Sample 2 nitrided layer /TiO₂,TiN/, magnification 100

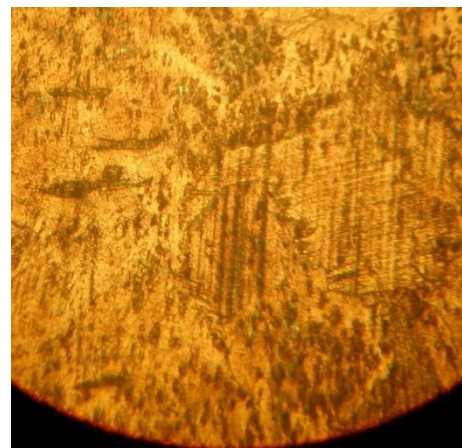


Fig. 6. Sample 7 nitrided layer /TiN/, magnification 100

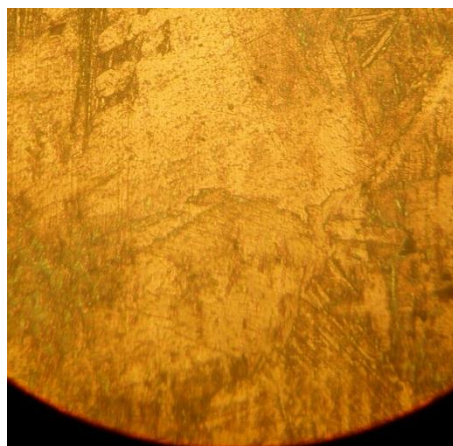


Fig. 7. Sample 8 nitrided layer /TiO₂,TiN/, magnification 200

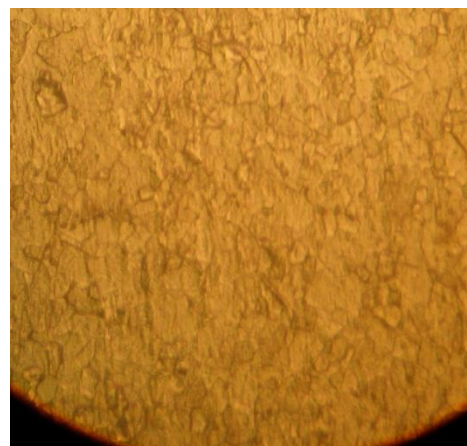


Fig. 8. Sample 14 nitrided layer /TiN/, magnification 200

Fig. 5, 7 and Fig. 6, 8 show a nitrided layer of TiO₂, TiN and TiN at a magnification of 100 and 200 times. It appears that in different nitriding modes the depth of the nitrided layer changes. Main factor is the amount of nitrogen and the time of the nitriding process. The amount of consumed gas N₂ also has an effect upon the plasmatron and the microhardness of the nitrided layer.

Plasma gas nitriding with indirect plasmatron has a number of advantages as a technique for changing the properties of the surface layer and improving the tribological characteristics of titanium alloys.

The experiments demonstrated that layer of TiO₂ or TiN can be identified, which determines the color of the surface layer of the nitrided samples. The depth of the resulting layer depends mainly on the time of nitriding and the consumption of plasma-forming gas N₂, and to a lesser extent on the change in the current's power within the range of 300-500A.

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ПЛАЗМЕНО АЗОТИРАНЕ НА ТИТАНОВИ СПЛАВИ С ИНДИРЕКТЕН ПЛАЗМОТРОН

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Ключови думи: *Титанови сплави, Плазмено газово азотиране, Повърхностно уякчаване, Titanium alloys, Plasma gas nitriding, Surface hardening*

Резюме: *Настоящата статия представя експеримент, състоящ се в плазмено газово азотиране на титанова сплав Ti-6Al-4V. Експериментът има за цел подобряване трибологичните свойства на титановите сплави в условията на работа при плъзгащ контакт. Данните, получени от експеримента ще послужат при създаване на технология за получаване на повърхностен слой с определена дебелина.*