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THE EFFECTIVENESS OF ACTIVE SCREEN METHOD IN ION NITRIDING GRADE 5 ELI TITANIUM ALLOY

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A titanium alloy for biomedical applications, Grade 5 ELI, was studied, which was subjected to the ion nitriding process in the temperature range of 530 – 590 °C and during 5 – 17 h, using two variants of sample arrangement in the glow discharge chamber. The first variant – the samples intended for nitriding were placed on the cathode, the second variant – the samples placed on the cathode were shielded with an active screen. In order to assess the effectiveness of nitriding, studies were carried out on the depth of nitrogen diffusion into the substrate of the studied titanium alloy, surface hardness tests, as well as observation of the obtained microstructures and phase composition tests – in order to assess the structure of the surface layers. It was found that using active screens increases the rate of nitrogen diffusion deep into the nitrided Grade 5 ELI titanium alloy, and thus increases the thickness of the obtained nitrided layers.

Keywords: titanium alloys, biomedicine, ion nitriding, active screen method, microhardnes, microstructures

INTRODUCTION

Due to the specific physical and mechanical properties (high specific strength, high corrosion resistance, etc.) of pure titanium and titanium alloys, they are widely used in the manufacture of critical mechanical engineering parts, aerospace applications, many corrosive environments, sport equipment and medicine [1-3]. Titanium is neutral to the human body and is frequently used in the medical field to replace heart valves, joints and bones. The biocompatibility and strength of titanium make it an ideal material for dental posts and other oral prosthetics [3-5]. However, titanium alloys exhibit poor tribological properties including a high and unstable coefficient of friction and adhesive wear [6,7].

Ion nitriding titanium and its alloys effectively eliminates these shortcomings [8]. Nitriding titanium alloys can be carried out using a traditional gas method, by means of laser remelting in a nitrogen atmosphere, ion implantation or using ion techniques. Ion processes allow the constitution of surface layers with a specific structure, phase and chemical composition, which enable significant improvement in the tribological properties of titanium alloys. In this work, evaluation of the effectiveness of ion nitridingby the active screen method of Grade 5 ELI titanium alloy used in biomedicine was carried out.

MATERIAL AND RESEARCH METHODOLOGY

The biomedical titanium alloy of the so-called high purity grade Grade 5 ELI was selected for the research. This alloy is a variation of the Grade 5 alloy used since the beginning of the 1980 s, which after years turned out to be toxic to the human body due to the content of vanadium and aluminum. Compared to its predecessor (Grade 5), the Grade 5 ELI alloy has a lower content of iron, hydrogen, carbon, oxygen and nitrogen as well as higher resistance to stress corrosion, with slightly worse strength properties. The chemical composition of the examined titanium alloy (Table 1) is consistent with the certificate issued by Daido Steel Co Ltd.

Table 1 **Chemical composition of Grade 5 ELI titanium alloy / wt. %**

C	Fe	O	N	H	Al	V	Ti
0,01	0,22	0,11	0,01	0,001	6,12	4,17	rest

The ion nitriding process was carried out in a JON-600 ion treatment device with a cooled anode, using the following nitriding parameters in hydrogen-nitrogen plasma: working atmosphere pressure p = 150 Pa, the of the reactive mixture composition 75% H_2 + 25% N_2, temperature range T = 530 – 590 °C, time range t = 5 – 17 h.

Two variants of sample arrangement in the glow furnace chamber were used: the samples were placed directly on the cathode, and the samples placed on the cathode were additionally covered with an active screen made of a perforated titanium sheet (AS method).

In the first case, the surface of the samples is bombarded with ions with energies resulting from the value

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of cathodic drop, whereas in the second case, in the surface layer of the ion-nitrided surface of the sample, strong voltage peaks appear that react with the nitrogen ions found in this area. The time of occurrence of voltage peaks favors giving nitrogen ions and other active plasma components a high speed glow discharge with several times higher energy than in the case of cathodic drop. High-energy nitrogen ions are driven into the material, creating a highly non-equilibrated nitrogen-saturated zone in the surface layer of the nitrided Grade 5 ELI titanium alloy substrate. According to the Fick diffusion laws in force, this promotes the diffusion of nitrogen from the surface to the core (center) of the nitrided metallic material that is the studied titanium alloy. Due to the nitrogen concentration gradient occurring between the surface and the core (center) of the sample, diffusion does not have to take place, at least in its initial stage, at the grain boundaries, which allows the formation of nitrided layers with high homogeneity [9].

The depth of nitrogen diffusion was determined on the basis of elemental arrangement analysis on an optical emission spectrometer with glow discharge (GDS GD Profiler HR with a Grimma discharge lamp with a 4 mm cathode diameter).

Microhardness measurements of the nitrided layers were conducted using the Knoop method on a Future Tech FM7 microhardness tester. The microhardness was measured using a 490,3 mN load. Observation of the microstructures was performed with an Axiovert microscope with digital image recording.

X-ray examinations in symmetrical Bragg-Brentano geometry (X ray diffraction) were carried out using a Seifert T-T diffractometer. The research used a lamp with a copper anode that emits CuKα radiation (λ = 0,154 nm).

TEST RESULTS

The study of the depth of nitrogen diffusion deep into the nitrided substrate of the Grade 5 ELI titanium alloy, as the basic feature determining nitriding effectiveness, was used to determine the effectiveness of the ion nitriding process using the active screen method. It was found that increasing the temperature, as well as extending the duration of the nitriding process, results in an increase in the depth of nitrogen diffusion in the Grade 5 ELI titanium alloy selected for biomedical research (Table 2). Applying the active screen method increases the intensity of the nitriding process because depending on the parameters of the nitriding process using the active screen, a 2 - 4,1 - fold increase in the thickness of the nitrided layers produced with respect to cathodic nitriding was found.

Table 2 **Thicknesses of nitrided layers obtained on Grade 5 ELI titanium alloy**

Cathode

Surface layer depth / μm 530		Temperature / °C		
		560	590	
Time / h	5	0,18	0,27	0,69
	8	0,33	-	0,91
	11	0,4	0,84	1,1
	14	0,53	-	1,88
	17	0,96	1,18	2,06

Cathode+AS

Surface layer depth / μm 530		Temperature / °C		
		560	590	
Time / h	5	0,54	1,11	2,09
	8	0,82	-	2,73
	11	1,36	1,66	3,2
	14	1,77	-	3,21
	17	2,09	2,32	5,45

The microhardness tests of the nitrided surface layers showed an increase in the hardness of the studied titanium alloy after the cathodic ion nitriding process, in relation to the hardness of this alloy in its initial state. The use of the active screen method resulted in a further increase in hardness. The surface layers produced in the cathodic ion nitriding process are characterized by a hardness 1,2 times higher than the initial state. Using an active screen supporting the ion nitriding process resulted in a further two-fold increase in hardness compared to the initial state.

The microstructure studies allow the authors to state that introducing the active screen results in the formation of a compact titanium nitride zone on the tested titanium alloy (Figure 1a), similar to the ion nitriding process on the cathode (Figure 1b).

In addition, introducing the active screen enables the production of a nitride layer also on the surface of the

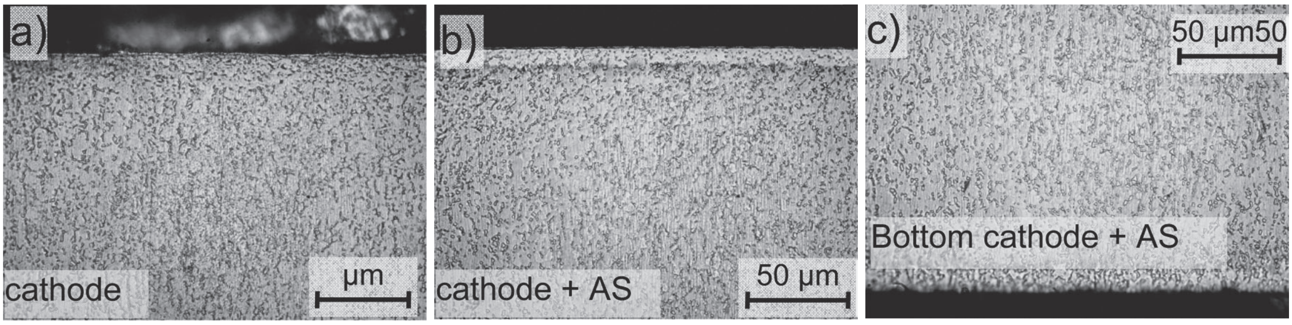


Figure 1 Microstructure of nitrided layer on Grade 5 ELI titanium alloy substrate for different variants of sample location in glow discharge chamber: a) cathode, b) cathode + screen, c) cathode + screen - bottom of sample. Process temperature T = 530 °C, time t = 17 h

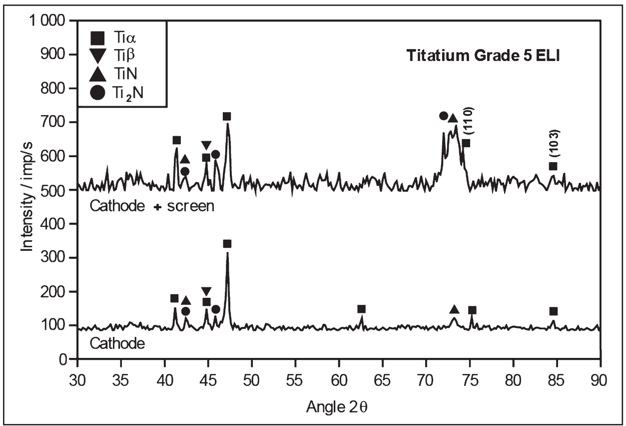
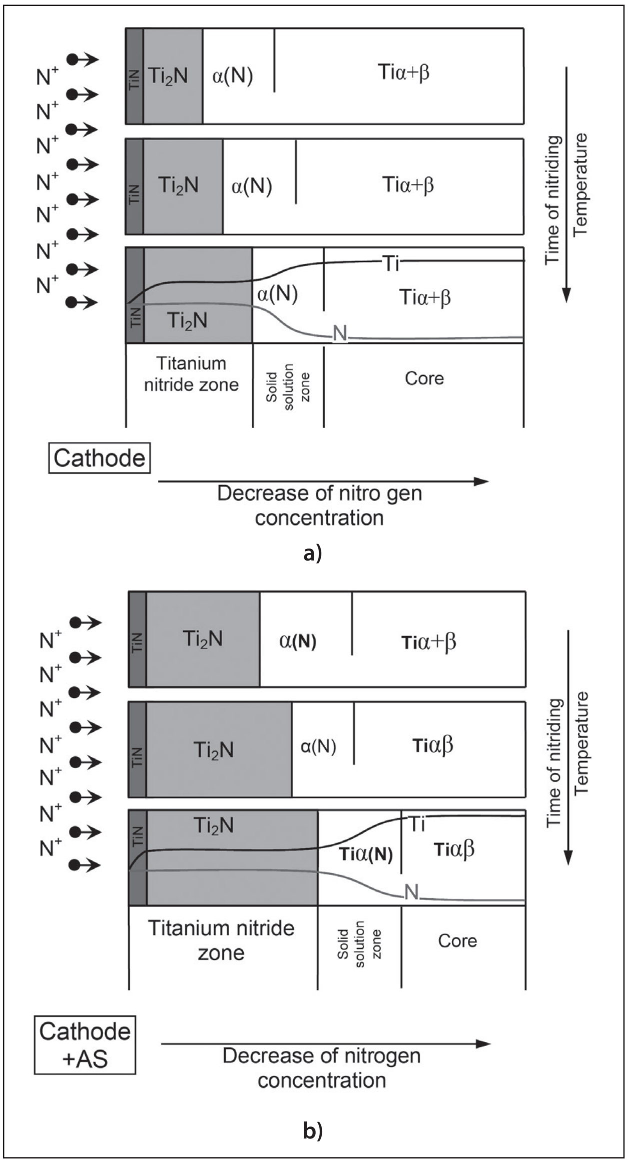


Figure 2 Diffractograms of nitrided layer on Grade 5 ELI titanium alloy substrate after various ion nitriding variants. Process temperature T = 560 °C, time t = 11 h



a)

b)

Figure 3 Model of surface layer construction after ion nitriding of Grade 5 ELI titanium alloy: a) cathodic nitriding, b) nitriding using active screen - AS method.

element in direct contact with the cathode - on the bottom of the sample (Figure 1c). Until now, the nitride layer was formed there only during nitriding at a much higher temperature over 700 °C [10, 11].

Analysis of the results of the phase composition tests of the layers produced on the Grade 5 ELI titanium alloy substrate allows the authors to state that in the ion nitriding process, a TiN and Ti_2N nitride zone is formed on the surface of this alloy. Introducing an active screen during nitridingon the cathode increases the growth rate of the nitride layer as evidenced by the large number of intense reflections coming from the TiN and Ti_2N phases (Figure 2). The use of an active screen produced a small amount of TiN nitrides. This is due to the intensification of surface processes under the active screen.

Analysis of the results of the microstructure and phase composition studies of the nitrided layers on the Grade 5 ELI titanium alloy was the basis for developing models of their construction (Figure 3 a and b). The obtained surface layers have a zonal structure and consist successively of aTiN nitride zone, Ti_2N nitride separation zone as well as αN phase. It should be noted that a clear increase in the depth of the nitrided layers is obtained by using an active screen.

Introducing an active screen during nitridingon the cathode increases the growth rate of the nitride layer. Using an active screen produced a small amount of TiN nitrides. This is due to the intensification of surface processes under the active screen.

SUMMARY

The adopted parameters of the ion nitriding process resulted in the formation of dense nitrided layers on the surface of the Grade 5 ELI titanium alloy, while the use of an active screen caused a two to four-fold increase in the thickness of the obtained nitrided layers compared to nitriding on the cathode.

Each of the adopted nitriding parameters increased the hardness of the nitrided titanium alloy. During the nitriding of samples located directly on the cathode, there was a single increase in hardness compared to the material in the initial state. The use of an active screen resulted in a further more than double increase in the hardness of the nitrided surfaces compared to the initial state.

The obtained surface layers have a zonal structure and consist successively of a TiN nitride zone, Ti_2N nitride separation zone and transition zone composed of the αN phase. In the summary of the analysis of the test results, it can be stated that the use of an active screen ensures a clear increase in the depth of nitrided layers on the Grade 5 ELI titanium alloy substrate. The improvement in the intensity of surface processes is not only caused by an increase in temperature. It was found that the temperature of the substrate under the active screen in the nitriding process on the cathode was higher by approx. 120 °C. An increase in temperature does not significantly increase the depth of the nitrided layer in the cathodic nitriding process.

It can be assumed that the increase in the depth of the nitrided layer, due to the use of an active screen, is influenced by factors other than temperature such as an increase in the concentration of ions and other plasma components actively participating in the nitriding process, as well as the increase in the kinetic energy of nitrogen ions that act on the surface of the nitrided substrate generate more defects accelerating the diffusion of chemisorbed nitrogen ions.

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