

AFM of titanium nitride layers prepared under glow discharge conditions

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Abstract

Optimization of titanium-based materials for medical application is a complex task linking specialists in various disciplines. This is a consequence of a system of self-dependent critical conditions required for their application. It is well known that majority of processes proceeding in the human body occur at surfaces and interfaces. Hence the knowledge concerning surfaces of the applied materials is essential for understanding the occurred processes and for optimization of the applied material. In this paper the atomic force microscopy was applied for investigation of the surface topography of nitrided layers of titanium alloys in a nano and mezosopic scale. Topography of layers nitrided at temperatures: 730, 850 and 1000 °C were compared. Results indicate that the size and distribution of the grains structure formed during nitriding processes strongly depends on the preparation temperature. This observation is related with the differences in chemical composition. The most appropriate temperature from the point of view of surface homogeneity is 850 °C. This conclusion correspond with the results of biological investigations which showed that layer of titanium nitride prepared at this temperature is characterized by the maximal biocompatibility [E. Czarnowska, T. Wierzchoń, A. Maranda-Niedbała, E. Kaczmarewicz, *J. Mater. Sci.: Mater. Med.* 11 (2000) 1].

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1. Introduction

Titanium and its alloys are widely applied in various fields of human activity including medicine. This is still the most commonly used material for surgical tools and biological devices, like implants and endoprostheses [2,3]. These devices are very useful, but it should be noticed that up to now they have been far from perfect. This is a consequence of the very complex critical conditions required for their application. The main advantages of titanium-based materials in medical applications is their good biocompatibility, low density, high level of mechanical properties and corrosion resistance [4–7]. However, one of the important features limiting the application of this material in medicine is its relatively low wear resistance, leading to the release of the material elements into the surrounding cells or tissues in a biological environment [1,8–11].

Most processes proceeding in the human body occur at surfaces and interfaces of implants material. The main effective method for overcoming the above-mentioned limitation therefore concerns a surface modification, which would improve biological function. Various methods of surface engineering, like thermal spraying, PVD, ion implantation, PACVD, PIRAC, discharge nitriding are applied [9,12–23]. One of promising and widely studied method is nitriding under glow discharge. Several groups of researchers have investigated the parameters of nitrided layers produced by this method [1,14,24–30]. In most cases common investigations performed by means of electron microscopy (SEM) and X-ray spectroscopy (XPS) are applied to get information concerning layer thickness, its structure in a microscopic scale as well as its phase and chemical composition. Recently Czarnowska et al. [1] showed the change of biocompatibility of nitrided layers as a function of the nitriding process temperature. An interesting question arose as to the cause of the strong inhibitory effect of the surface layer observed for selected temperatures of preparation. This effect leads to the decrease of cell proliferation and, as authors suggested, results from the morphology of the studied layers. For this reason, in this paper

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atomic force microscopy was applied for precise investigation of surface topography of nitrided layers in a nano and mezosopic scale. We believe that such information is essential for understanding the interaction of the outer surface of these layers with a biological environment and improving the studied materials in medical applications.

2. Experimental

2.1. Specimen preparation

Plates of titanium alloy Ti–1Al–1Mn (1% Al, 0.9% Mn, 0.3% Fe, 0.3% Cr, 0.12% Si, Ti-balance) were mechanically polished and nitrided using glow discharge equipment [31]. The nitriding process was carried out at several temperatures: 730, 850 and 1000 °C. Other parameters, like time of nitriding (4 h) and nitrogen pressure (4 hPa) were kept constant during all the processes.

2.2. Study of the surface morphology

The surface of the samples before and after nitriding was examined by means of AFM (atomic force microscopy). The measurements were performed using a commercial SPM instrument, TMX 2000 Discoverer (TopoMetrix, CA). The topography of the samples was studied in contact and constant force modes. Standard Si₃N₄ tips and scanners (all from TopoMetrix) were applied. The investigations were performed under an ambient atmosphere and temperature. Several images of various areas for each sample were collected to gain better knowledge of the variations of the surface topography.

Additionally, stereometric (3D) measurements of the surface topography were performed using a Taylor Hobson scanning profilometer (Form Talysurf Series). In this method the scanning area was 100 μm × 100 μm, which is several times larger in comparison to the measurement performed by AFM.

2.3. Chemical composition

The chemical composition of the nitrided layers was determined using a Cameca Su-30 type X-ray microanalyser and by means of X-ray photoelectron spectroscopy (XPS) Escalab-210 (VG Scientific).

2.4. Corrosion resistance

The corrosion resistance test was performed using the potentiodynamic method (Atlas-Sollich, Atlas'98 type potentiostat). Prior to measurements the samples were kept in a test solution for 24 h in order to stabilize the corrosion potential. The polarization curves were determined at a temperature of 37 °C in Hank's solution (Table 1). The potential of the surface varied from –1000 mV towards anodic potentials up to 5000 mV, with rate 50 mV/min. A saturated calomel electrode (SCE) was used as the reference electrode.

Corrosion resistance was defined on the basis of corrosion current density, corrosion potential and polarization resistance. Lower corrosion current density and higher corrosion potential and polarization resistance indicate better corrosion resistance of the samples.

Table 1

Chemical constitution of the Hank's solution [36]

Ingredients	Concentration (g/l)
NaCl	8
CaCl ₂	0.14
KCl	0.4
NaHCO ₃	0.35
Glucose	1
MgCl ₂ ·6H ₂ O	0.1
Na ₂ HPO ₄ ·2H ₂ O	0.06
KH ₂ PO ₄	0.06
MgSO ₄ ·7H ₂ O	0.06

3. Results and discussion

3.1. Surface topography of nitride layers

The surface topography of the Ti–1Al–1Mn titanium alloy and its nitrided layers obtained during nitriding at temperatures: 730, 850 and 1000 °C, were studied by means of two methods: scanning profilometer and AFM. The results enable us to analyze the influence of the nitriding temperature on the change of surface topography. The divergences between the results obtained by means of these two techniques are discussed below.

The values of typical stereometric parameters determined from the measurements by scanning profilometer for titanium alloys before and after nitriding are presented in Table 2. The scale of changes of all presented parameters indicates a strong influence of the nitriding process on the formation of the surface topography. Evidently the nitriding process causes development of the real surface area. This effect increases with the temperature of nitriding. The highest value of real surface area is therefore observed for the sample after glow discharge assisted nitriding at 1000 °C. The surface corrugation is characterized by the square mean deviation (S_q) equals to 0.489 μm, and the maximum height of roughness (S_t) equals to 3.23 μm. These values are two times higher than these calculated for the nitrided layer prepared at 730 °C, and three times higher than the appropriate values obtained for titanium alloy without nitriding. It is also interesting to compare these data with the simultaneously performed measurements of the change of peak density on the surface. This parameter exhibits the opposite behavior, i.e. strongly decreases with the temperature of the nitriding process. The maximal value (106 535 mm^{–2}) is noted for titanium alloy without nitriding, while the peak density significantly decreases

Table 2

Stereometric parameters characterizing the topography of the surface of the Ti–1Al–1Mn titanium alloy and nitrided layers

Parameters	Ti–1Al–1Mn	730 °C	850 °C	1000 °C
S_a (μm)	0.081 ± 0.003	0.175 ± 0.011	0.253 ± 0.001	0.395 ± 0.028
S_q (μm)	0.113 ± 0.006	0.229 ± 0.016	0.322 ± 0.008	0.489 ± 0.029
S_p (μm)	0.559 ± 0.043	0.88 ± 0.326	1.24 ± 0.177	1.92 ± 0.04
S_v (μm)	0.555 ± 0.107	0.87 ± 0.172	1.12 ± 0.054	1.31 ± 0.063
S_t (μm)	1.11 ± 0.057	1.75 ± 0.325	2.36 ± 0.173	3.23 ± 0.087
S_{ds} (peaks/mm ²)	106535 ± 4261.3	51251 ± 3412.6	45688 ± 3287.7	39632 ± 3786.7

S_a : arithmetic mean deviation of the roughness profile; S_q : square mean deviation of the roughness profile; S_p : maximum height of the peak of the roughness profile; S_v : maximum depth of the valley of the roughness profile; S_t : maximum height of the roughness ($S_t = S_p + S_v$); S_{ds} : peaks density.

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