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Study of the titanium VT1-0 surface degradation after cyclic loading in different structural states, including ones when coatings are formed by Micro-Arc Oxidation

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Abstract. This article considers the results of the study of the titanium VT1-0 surface degradation (Grade-4 equivalent) for recrystallized- and ultrafine-grained states after fatigue testing. Comparative analysis of peculiarities of the titanium samples degradation was performed after coating formation by Micro-Arc Oxidation. It was found, that the coating formed by Micro-Arc Oxidation shows different degradation behaviors for recrystallized and ultrafine-grained states

1. Introduction

The objective of the work was to study peculiarities of titanium degradation in different structural states, namely, for recrystallized and ultrafine-grained (UFG), both with and without coating formed by Micro-Arc Oxidation (MAO). This coating, when used for medical purposes, accelerates the integration of metallic implants with the bone tissue, reduces the period of post-surgery rehabilitation of patients as well as the amount of undesirable body reactions to a foreign implant material [1-3]. The coating is insoluble in water, alkali and weak acids; it is hygroscopic, non-inflammable and non-toxic. The coating is resistant to light and moderate temperature variations.

Such coatings have a high macro-porosity, good wear resistance and recently have been successfully used in prosthetics and stomatology. Titanium prostheses with a biocompatible coating may remain in a human body for many years. For the entire period they are under cyclic loads. Therefore, study of fatigue durability of titanium, including with coatings applied, and the degradation peculiarities analysis is now one of the objectives of interest. One should note, that the fatigue tests are widely applied to study mechanical properties of the materials, both for forecasting their durability in as similar states as real operation, and for learning the materials degradation nature [4-8].

2. Materials and methods

The studied object hereunder is titanium of technical grade purity (alloy VT1-0) in UFG and recrystallized states. UFG state was provided by mechanical processing and heat treatment with the use of longitudinal and transversal rolls enabling to produce UFG bars of titanium with the diameters 4-10 mm as per TU 1825-001-02079230-2009 [9]. During this study titanium alloy VT1-0 bars with the

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diameter of 8 mm, that were subjected to finish annealing at the temperature of 623 K for 3 hours in order to relieve internal stresses of the first kind, were used. Once processed and treated as mentioned above, the alloy features a homogeneous grain and sub-grain structure with the arithmetic mean size of structural elements of about 190 nm. A homogeneous recrystallized structure of the alloy VT1-0 (recrystallized state) was produced from UFG structure by keeping it at the temperature of 823 K during one hour. Average grain size at the recrystallized state is 2.35 μm.

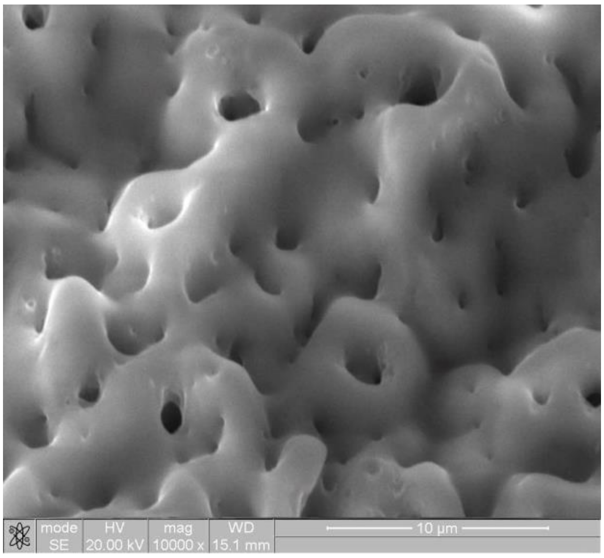

Figure 1. Image of morphology of the MAO coatings of the alloy VT1-0. Scanning-electron microscopy with x10000 magnifying at the angle of 45°.

For the fatigue properties testing, I-beam samples with the working area thickness of 1 mm and the width of 3 mm were made of bars. For half of a number of samples, both in UFG and recrystallized states, their surfaces were coated with a porous biocompatible coating with the thickness of $8 \div 10$ μm by the Micro-Arc Oxidation (MAO) [10]. The coating appearance is shown in Figure 1.

The coating composition includes silicon oxides of at least 30 mol %, calcium oxides of at least 2.5 mol %, phosphorus oxides not more than 6 mol %, sodium oxides of from 0.5 to 3.0 mol %, as well as titanium oxides of at least 20 mol %.

The coating has a porous structure resulted from electrochemical and plasma-chemical processes on the surface while local micro-arc charges are passing through it. A coating interlayer adjacent to the base is low-porous and insulates the base from the environment. Phase composition is generally represented by an amorphous glass phase with small areas of nano-crystal oxide phases.

Fatigue properties testing under tensile load for the samples of titanium alloy VT1-0 was carried out by means of the test equipment Instron Electropulse E3000 having an electromagnetic drive, in the loading mode of 50 Hz at the ambient temperature. The tests were carried out as per the tensile loading chart with a symmetric sawtooth-like cycle within the loading area of $0.9\sigma_{max} - 0.2\sigma_{max}$. The limit number of cycles for the studied samples was set to $2 \cdot 10^6$. The limit value of load at which there was no degradation after $2 \cdot 10^6$ cycles was set as a conditional limit durability of the studied material. The study of fractography of the fracture structure of broken samples was carried out by means of scanning-electron microscopes Quanta 200 3D and Nowa NanoSEM 450 in the topographic contrast mode by using secondary electrons detector.

3. Results

The samples of alloy VT1-0 were tested in recrystallized and UFG states, without coating and with coating applied by MAO, under fatigue mode. It was found, that conditional limit of the durability at $2 \cdot 10^6$ loading cycles for UFG titanium with and without coatings is 1.4 times higher versus similar recrystallized titanium. Formation of coatings by MAO, as shown herein, improves fatigue strength of the material. An increase is about $6 \div 8$ % of the original value both for recrystallized initial state and for UFG structure. After the tests having been completed the study of samples surface degradation study (at various stresses and numbers of cycles) was carried out. The stress for recrystallized state varied within the range of 475 to 490 MPa, for UFG state – from 690 to 1000 MPa. Figure 2 shows images of the degradation (fracture) surface of the samples of alloy VT1-0 in recrystallized and UFG states, including ones with coatings formed by MAO, after cyclic tensile strength tests carried out with different loads.

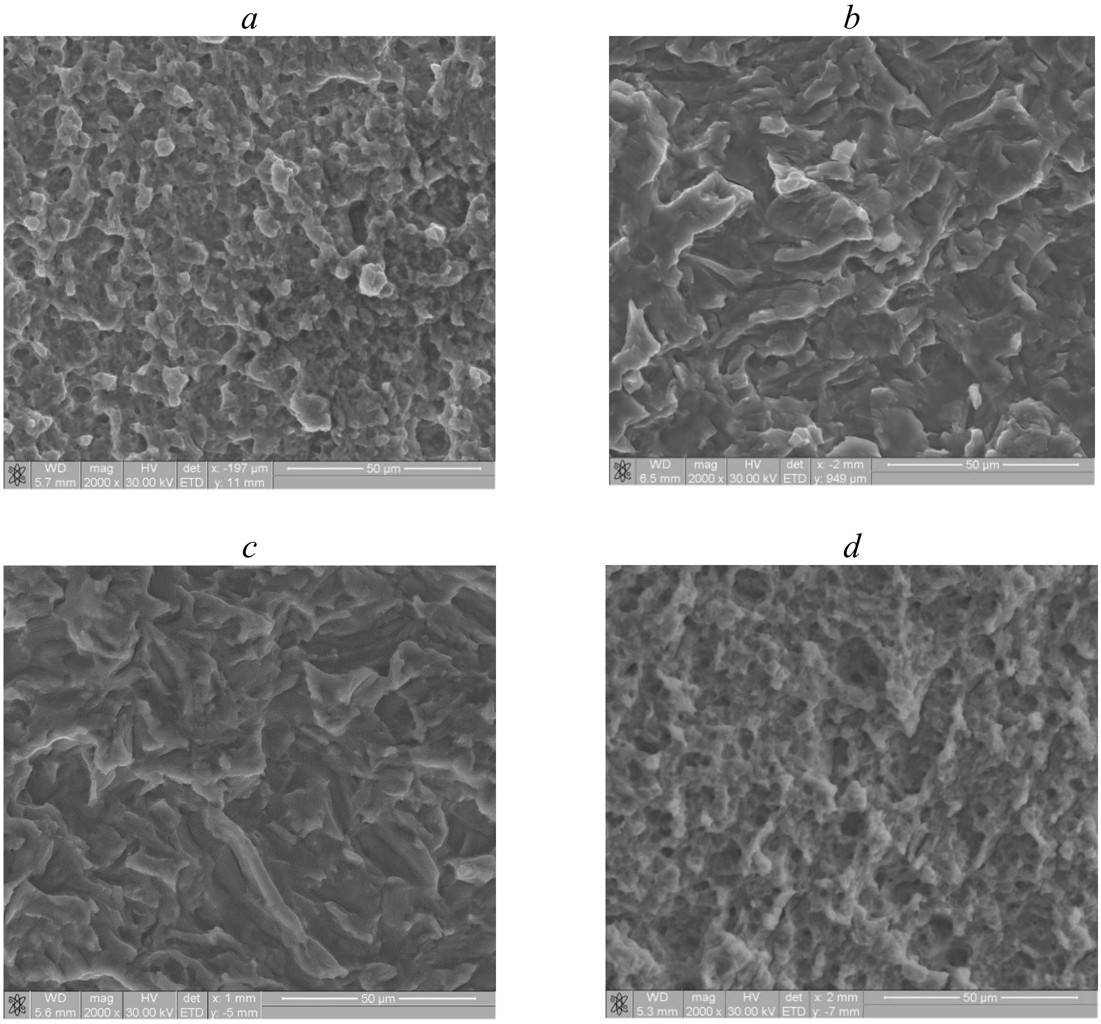

Figure 2. Electron-microscopy images of the surface degradation perpendicular to the fracture surface of the samples of titanium alloy VT1-0 after fatigue tensile tests:
a – recrystallized state, σ=515 MPa, N= 218$\cdot$10^3 cycles
b – recrystallized state + MAO coating, σ=525 MPa, N= 253$\cdot$10^3 cycles
c – UFG state, σ=650 MPa, N= 199$\cdot$10^3 cycles
d – UFG state + MAO coating, σ=800 MPa, N= 165$\cdot$10^3 cycles

Let us consider in detail the degradation peculiarities for each state.

For titanium samples in recrystallized state the surface structure formed after degradation of samples in the area of fatigue failure is specific for quasi-brittle fracture (figure 2a). The degradation occurs by the intergranular mechanism. The pore coalescence results in a brittle fracture along the boundary. At a minimum loading the degradation is more brittle and occurs at a less speed – about $10^{-5}\div10^{-4}$ mm/cycle, as evidenced by the absence of pits. The load increase up to 550 MPa (the number of cycles to degradation in this case is 50 thousand) results in a more ductile breakage, and the structure is specific for the strain rate about $5\cdot10^{-4}$ mm/cycle.

Images of the fracture surface of the samples of alloy VT1-0 in recrystallized state with the coating formed by MAO on its surface and after cyclic tensile tests are presented at figure 2 (b). The analysis of results has shown, that for all loadings (490-550 MPa) for the recrystallized samples with coating formed by MAO, there is a change in fracture pattern within the fatigue failure area. The surface structure in that area is specific for a cracked quasi-ductile fracture with typical grooves oriented perpendicular to the fatigue-crack propagation line. Such a structure is specific for more ductile materials.

Figure 2 (c) shows images of the fracture surfaces of the samples of alloy VT1-0 in UFG state after cyclic tensile strength tests. Based on the results thereof it was found that the samples with UFG structure feature considerable change of a peel neck section in the rupture area with this almost not being presented in the fatigue failure area. A strong relationship was revealed between the fatigue failure area size and the value of the applied cyclic load. Thus, in a sample subjected to medium cycle load of 615 MPa the fatigue failure area occupies 60 % of the total area of the peel neck section, while under load of 850 MPa this does not exceed 20 %. The structure of samples fracture surfaces within the area of fatigue failure is typical for quasi-ductile cracked fracture, moreover, as far as the load increases, a ductile fracture behavior are more clear, which is evidenced by a high number of sharp ridges along the rupture line.

It should be noted that formation of MAO coating on the surface of samples of alloy VT1-0 in UFG state (figure 2d) changes fundamentally the samples fracture behavior, while fatigue properties are not affected significantly though. Compared to the UFG samples, for which a quasi-ductile fracture behavior is more typical, a clearly defined quasi-brittle fracture behavior is observed for coated UFG samples. This is evidenced also by low change in the neck section area within the rupture area, which is typical for high-strength materials, as well as by the fracture surface structure. The fracture surface structure within the fatigue crack area is typical for quasi-brittle fracture of the intergranular mechanism of degradation. Pits have a facet pattern. There is a structure within the rupture area, which is similar to one of the fatigue failure area, but with rounder pits specific for more ductile degradation.

The study dealt also with adhesive properties of the MAO coatings on the surface of alloy VT1-0. As shown by the results of fatigue tests, MAO coatings perfectly withstand multi-cycle loads. Based on the analysis of images of the fracture surface of the coated sample of VT1-0, there is no coating peel-off within the fatigue failure area even in case of the sample degradation. The coating peel-off takes place only within the rupture area due to high strain the base was subjected to. Therefore, it can be concluded about high adhesive properties of the MAO coatings on the surface of alloy VT1-0.

The results also show a reinforcing effect in a near-surface area because of the coating. This is evidenced by the change of pit elongations orientation in case of fatigue failure in the near-surface layer. The pits in the near-surface layer (10-30 μm under coating) are elongated not along the direction of load, but perpendicular to the coating surface. A higher value of the fatigue strength for the coated samples is connected with reduction of surface defects, which represent the degradation development sources under cyclic loading. A critical role of the defects on surface and in thin near-surface layers in case of fatigue failure is noted in a series of studies [11-13].

4. Conclusion

1. Degradation of the samples VT1-0 in recrystallized state occurs by the intergranular mechanism and with quasi-brittle behavior. Load increase or formation of coatings by MAO contributes into a more ductile behavior of the degradation.
2. Degradation of the VT1-0 samples in UFG state occurs under combined mechanism with predominating ductile degradation of the samples. Formation of MAO coating results in change in the degradation behavior and predomination of a brittle failure by the intergranular mechanism.
3. High adhesive properties of MAO were found remaining during fatigue tests up to degradation of the samples.

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