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The structure of the surface layer in titanium VT1-0 after high-cycle fatigue tests

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Abstract. Using the methods of optical and scanning electron microscopy, transformations that occur in the structure of commercially pure titanium VT1-0 subjected to electric-current pulse treatment and high-cycle fatigue testing to failure, were analyzed. The paper reports that as a result of the energy impact on the samples, its fatigue life increases by a factor of 1.3. It is revealed that the improvement of the fatigue life is possible due to the change in the size of crystallites in α-titanium, as well as to the decreased number of internal stress field concentrators in the surface layer.

1. Introduction

The fracture of metallic materials in conditions of any stresses is caused by the processes of plastic deformation, the formation and propagation of cracks. During fatigue testing it is associated with a sharply increasing temperature of a sample, leading probably, as a consequence, to processes of dynamic ageing, polygonization and recrystallization. The extent, to which these processes can be developed, significantly depends on both the nature of the external impact and the initial structural state of the material [1-7].

An efficient procedure to modify the structure and phase composition in a material is its electric-current pulse treatment [8-10]. A number of studies carried out on diverse structural classes of steel emphasizes that the impact of an electric-current on a material at a certain stage of fatigue stress improves the efficiency of samples by a factor of 1.5-2 [11-14].

Titanium alloys are widely applied industrial materials, since the unique combination of physical and mechanical characteristics such as high specific strength, sufficient technological plasticity and high corrosion resistance makes them rather competitive constructional materials. However, their use is restricted by low resistance to abrasive and erosive wear, low hardness, liability to seizure if applied in friction areas, and insufficient wear resistance.

This study is aimed at analyzing changes in the defect substructure, registered in samples of titanium alloy VT1-0 under regular stressing and after electric-current pulse treatment, as well as identifying the mechanisms relevant for improvement of fatigue life [15-17].
2. Test materials, equipment and research methods

Samples of titanium alloy VT1-0 (foreign analogue – alloy Grade 2) with chemical composition (up to 0.18 Fe; to 0.07 C; to 0.04 N; to 0.12 Si; to 0.004 H; 0.3 % - other impurities; balanced by Ti, mass %) was used as the material for research. Fatigue tests were carried out using a special device according to the scheme of asymmetric cantilever bending. Samples with a thickness of 4 mm and a width of 12 mm were made according to the Russian Standard (GOST 25.502-79) and had a symmetric stress concentrator with a minimal narrow spot in the center (5 mm). The temperature of tests was 300 К, the frequency of the bending stress of the sample was 10 Hz.

When fatigue testing, ultrasound monitoring was carried out using a universal unit to control the speed of ultrasound (Institute of Strength Physics and Materials Science, SB RAS), which was measured on a distressed sample. Electric-current treatment was carried out after a sharp change in the speed of ultrasound (180,000 cycles). The parameters of the electric-current treatment were as follows: the amplitude of the current pulses was 2 kА, the maximum current density in the zone of the stress concentrator was 100 А/mm$^2$, the frequency of the current pulses was 70 Hz, and the time of treatment was 2 minutes. After completion of the electric-current treatment a high-cycle fatigue testing to failure of the samples was carried out.

Using SEM methods (Tesla BS-301), the fractured surface was analyzed. The phase composition and state of the defect substructure were studied using transmission electron diffraction microscopy methods (JEM-2100). Foils for the purpose of research were prepared by ion thinning of plates cut of a solid sample, in parallel and as close as possible to the fractured surface using electric-spark method.

3. Results and Discussion

It is determined that the electric-current treatment causes an increase in cycles to failure from 281,333 to 359,532. This corresponds to an increase in fatigue life by a factor of 1.3.

The structure of the surface layer in as-delivered titanium fractured in the process of fatigue testing (281,333 cycles) has a relatively thin (maximum 4.5 µm) surface layer with a nano-crystalline grain and a sub-grain structure. Grains in this layer are on average $58.6 \pm 21.5$ nm. Grains form layers that are disoriented relative to each other and parallel to the surface of the sample. A ring structure is observed in the electron-diffraction micro-pattern, presented in figure 1 a. This fact demonstrates, firstly, the small size of crystallites, forming the electron-diffraction micro-pattern, secondly, large-angle disorientation of these crystallites, i.e. the formation of a grain and sub-grain structure in the surface layer of commercially pure titanium as a result of fatigue testing.

The analysis has revealed a lamellar structure of the material close to the nanostructured surface layer and a lot of flexural extinction contours in it. This is a good illustration of a large number of sources (concentrators) of internal stress fields in the surface layer, which cause bending and torsion in the crystal lattice of the material.

Indexing of the electron-diffraction micro-pattern shown in figure 1 b, demonstrates a multi-phase state of the surface layer in the material. A principal phase is the α-modification of titanium, the reflections of which form a ring structure. Therefore, nanocrystalline grains are formed by α-modification of titanium. An analysis of the dark-field of the surface layer in the reflections of the crystal lattice of α-titanium revealed a speckled contrast throughout crystallites (figure 1 c). This fact may be evidence of a defect substructure in nano-crystallites of α-titanium. Besides reflections of α-titanium, there are reflections of the titanium oxide phase detected in the electron-diffraction micro-pattern. Titanium oxides are located mainly along the edges of α-titanium crystallites; the particles are rounded with sizes varying in the range of 10 nm (figure 1 d). It should be noted that the particles of the oxide phase are found only in the nanostructured surface layer of the sample. Oxide particles were not detected throughout the sample. It can be assumed that particles of the oxide phase are formed, because atoms of oxygen penetrate along the small and large-angle edges of the crystallites during fatigue testing of the material.

It was found that after fatigue testing of samples subjected first to electric-current pulse treatment (359,532 cycles), the submicrocrystalline structure (average size of crystallites is 422.7 ± 400 nm) is
formed in a relatively thin (maximum 4 µm) surface layer of commercially pure titanium VT1-0 (figure 2). Therefore, electric-current pulse treatment of commercially pure titanium VT1-0 during fatigue testing results in a multiple increase in dimensions of crystallites in the surface layer. The other special feature of the grain structure in the surface layer is the comparatively high dispersion of grain sizes. It is assumed that the reason for this is the recrystallization processes of the material that occur during electric-current pulse treatment.

Figure 1. The cross section structure in a sample of commercially pure titanium VT1-0 fractured in the process of fatigue testing (281 333 cycles); а – bright-field image of the surface layer; b – electron-diffraction micro-pattern, taken on the zone in the oval (а); c – dark-field image of zone (а), taken in [002]α-Ti (reflection 1, shown by the arrow in b); d – dark-field image of zone (а), taken in [020]Ti₃O₅ (reflection 2, shown by the arrow in b).

Another feature of the samples of commercially pure titanium VT1-0, fractured during fatigue testing in conditions of electric-current treatment, is a rather low level of bending torsion in the crystal lattice of the material. Thus, this treatment of commercially pure titanium during fatigue testing decreases the number of sources (concentrators) of the internal stress fields in the surface layer of the material.

The principal phase in the layer of titanium fractured during fatigue testing with electric-current treatment is α-titanium, the second phases is the particles of titanium oxides located along the edges of α-titanium grains and subgrains. The oxide phase particles vary from 10 to 35 nm, significantly
exceeding the size of the oxide phase particles formed in the surface layer of titanium subjected to fatigue testing without energy impact.

**Figure 2.** The cross section structure in a sample of commercially pure titanium VT1-0 fractured in the process of fatigue testing after electric-current treatment (359 532 cycles); a – bright-field image of the surface layer; b – electron diffraction micro-pattern of the foil section (a); c – dark-field image of the foil section (a), obtained in [011]α-Ti (reflex 1 indicated by the arrow in b); d – dark-field image of the foil section (a), obtained in [200]Ti$_2$O (reflex 2 indicated by the arrow in b).

4. **Conclusions**
To sum up, electric-current pulse treatment of commercially pure titanium VT1-0 results, firstly, in a multiple increase in sizes of α-titanium crystallites in the surface layer of the material due to recrystallization processes, secondly, reduces the number of sources (concentrators) of internal stress fields in the surface layer of the material, thus reducing the number of potentially dangerous spots of crack formation; thirdly, it causes a significant increase in size of the oxide phase particles formed in the surface layer of commercially pure titanium during fatigue testing.

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