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Investigation of the effect of nanosecond laser pulses processing on the microstructure and fatigue resistance of commercially pure titanium

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The effect has been studied of treatment with nanosecond laser pulses on the fatigue resistance of plate samples of recrystallized (grain size of the order of $2-3\,\mu$m) commercially pure titanium (grade VT1-0) under cyclic tensile loading. The results of investigations by scanning and transmission electron microscopy of the subsurface layer microstructure of the alloy under study after exposure to nanosecond laser irradiation and subsequent fatigue tests are presented.

Keywords: titanium, surface, pulsed nanosecond laser irradiation, shock−wave action, fatigue properties.

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One of the promising directions in the field of creating new technologies for processing medical−grade metal materials is surface modification using concentrated energy fluxes including laser irradiation [1–3]. The great majority of widely commercially employed techniques for material surface modification involve considerable heating of their near−surface layers or entire bulk. This may be to a high extent avoided by using the advanced method for processing metal material surfaces with short (pico—, nano—, and subnanosecond) and ultrashort (subpico— and femtosecond) laser pulses which has been intensely developed in recent years[1–3]. Processes evolving under such action, being associated with variations in the surface relief and phase−structural transformations in thin $(1-5\,\mu$m) subsurface layers, enable formation of a unique complex of physical−chemical and mechanical properties of the above mentioned regions of materials. By varying the laser irradiation parameters, it is possible to perform controllable macro— to nanoscale modification of the surface relief, including creation of multimodal roughness with which manifestation of superhydrofobic or superhydrophilic properties of the surface are associated. It is known that transition from the mode of the surface material ablation to the mode of shock−wave processing (laser peening) promotes improvement of mechanical characteristics of both the subsurface and near−surface material layers down to the depth of one micrometer to several hundreds of micrometers [1,4]. However, the question about the effect of nanosecond laser irradiation upon mechanical properties in cyclic loading of titanium alloys is so far poorly studied. This paper represents investigation of the effect of nanosecond laser irradiation on the fatigue resistance of recrystallized commercially pure titanium and also of peculiar features of variations in the recrystallized structure of the studied alloy near−surface layer during the nanosecond laser irradiation and fatigue test.

As the test material, commercially pure titanium VT1-0 was selected. Its chemical composition is given in the table.

For the fatigue test, plane samples with the double−T cross section with the base thickness of 1 mm, width of 3 mm, and length of 40 mm were used; the samples were fabricated by electrical discharge machining at setup „AQ300L“. Fatigue tests in the cyclic loading mode were performed using the equipment of the Nanostructure Material and Nanotechnologies Center of NRU „BelSU“ at the electromagnetically driven test machine Instron Electropulse E3000. As known, the loading frequency will be selected in compliance with the amplitude−frequency characteristic (AFC) that depends on the test sample rigidity. In this work, the test frequency of 15 Hz was selected from the range where there are no distortions of the AFC sinusoidal character, which are observed at room temperature at higher frequencies.

Structural investigations were carried out using transmission electron microscopes (TEM) Tecnai G2 F20 S-TWIN and FEI TecnaiOsiris at the accelerating voltage of 200 kV. Thin foils (lamellas) that are to be cut normally to the sample surface were prepared for the TEM investigation by the ion thinning technique with a focused laser beam in the column of the FEI Scios scanning electron−ion microscope.

The laser processing of the samples was performed under a layer of water about 2 mm thick using setup „Minimarker-2“ at the following irradiation parameters: wavelength $\lambda = 1064$ nm, pulse duration $\tau \sim 100$ ns, pulse

Chemical composition of the titanium–based alloy VT1-0

	Content of elements, wt.%						
	Al	Fe	Si	O	C	N	H
Required	< 0.7	< 0.25	< 0.1	< 0.2	< 0.07	< 0.04	< 0.006
Actual	< 0.01	< 0.12	< 0.01	< 0.14	< 0.01	< 0.01	< 0.01

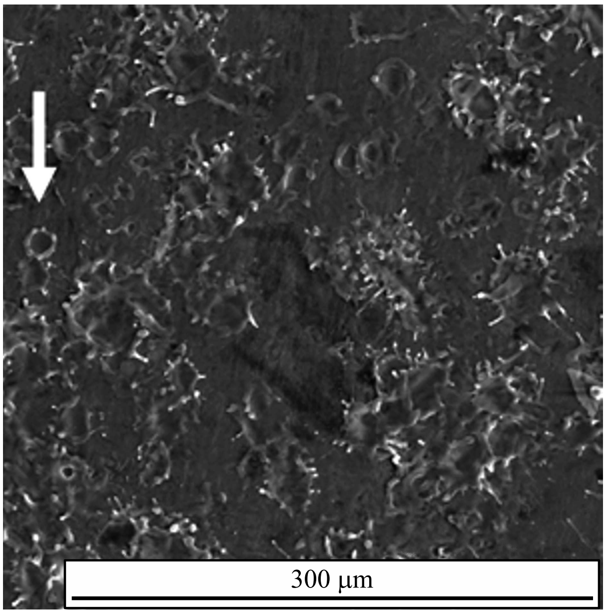

Figure 1. Surface microstructure of the alloy VT1-0 samples in the initial recrystallized state after processing with nanosecond laser pulses under a layer of water. The arrow points to an isolated crater characteristic of the single–pulse impact. The single pulse power density is $\sim 1\,\mathrm{GW/cm^2}$.

energy $E \approx 10^{-3}$ J, pulse repetition rate $\nu = 50$ kHz, laser beam scanning rate $v = 1500$ mm/s, single pulse power density $F \sim 1\,\mathrm{GW/cm^2}$. The samples were irradiated in the scanning mode with the preset by the control system maximally dense arrangement of surface craters without their superposition (only one pulse per point, free of overlapping). The spot of the laser beam focused on the surface was 35 μm in diameter.

In fabricating thin foil (lamella) from the subsurface layer of the material under study by the „cross-section" technique, scanning electron–ion microscope FEI Scios Dual Beam (Common Use Center of FRC „Crystallography and Photonics") was used. The lamellas were cut from the center of isolated craters in the form of blurred molten alloy droplets, which arose under the action of a single pulse. Along the crater periphery there is a clearly observed convex bead with elliptic edges additionally evidencing for local melting.

At the rate of laser beam scanning over the material surface (under a layer of water), which has been selected in this work, both isolated and overlapping craters may be observed by a metallographic microscope (Fig. 1). As known, emergence of the craters is connected with the fact that, when the laser pulse power exceeds the ablation threshold, a microexplosion occurs and gives rise to a crater on the sample surface and to glowing plasma jointly with solid and liquid particles flying apart. The area occupied by overlapping craters was determined by the free analytical program code „ImageJ". For this analysis, images were processed by increasing the contrast and essentially enhancing the brightness of midtones. This operation leaves visible only the craters, which enables calculation of the surface area occupied by the craters; this area appeared to be about 80%. The relief of the laser–processed region was studied at the crater cross section obtained by using a focused ion beam in the column of the scanning electron microscope (SEM). The crater depth and height of the bead surrounding it which were measured with respect to the initial surface level were 6 and 0.5 μm, respectively.

To determine the fatigue limit via the experimental data, dependences were plotted (Fig. 2) of the cycle maximal strain on the number of cycles till destruction (Woeller diagram) for the samples of recrystallized titanium VT1-0 in the initial state and after the surface preprocessing with a laser. Fig. 2 shows that in the entire range of preset strains samples withstand on the average a considerably higher strain after processing with a nanosecond laser irradiation than in the initial state prior to laser processing. Thus,

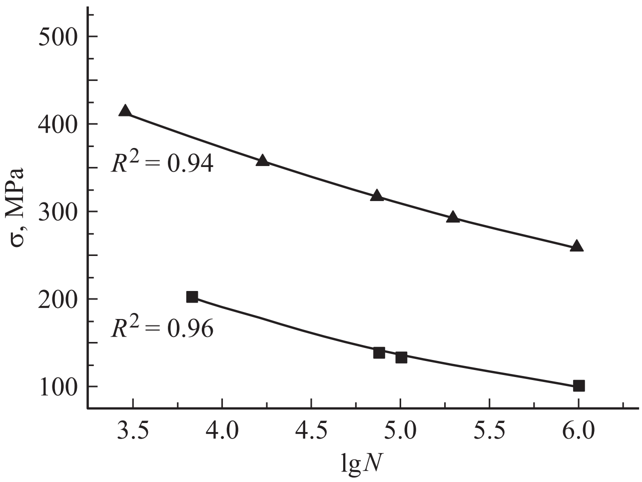

Figure 2. Fatigue curves for the alloy VT1-0 samples in the initial recrystallized state (squares) and after processing with nanosecond laser pulses (triangles) in water (at the single pulse power density of $\sim 1\,\mathrm{GW/cm^2}$).

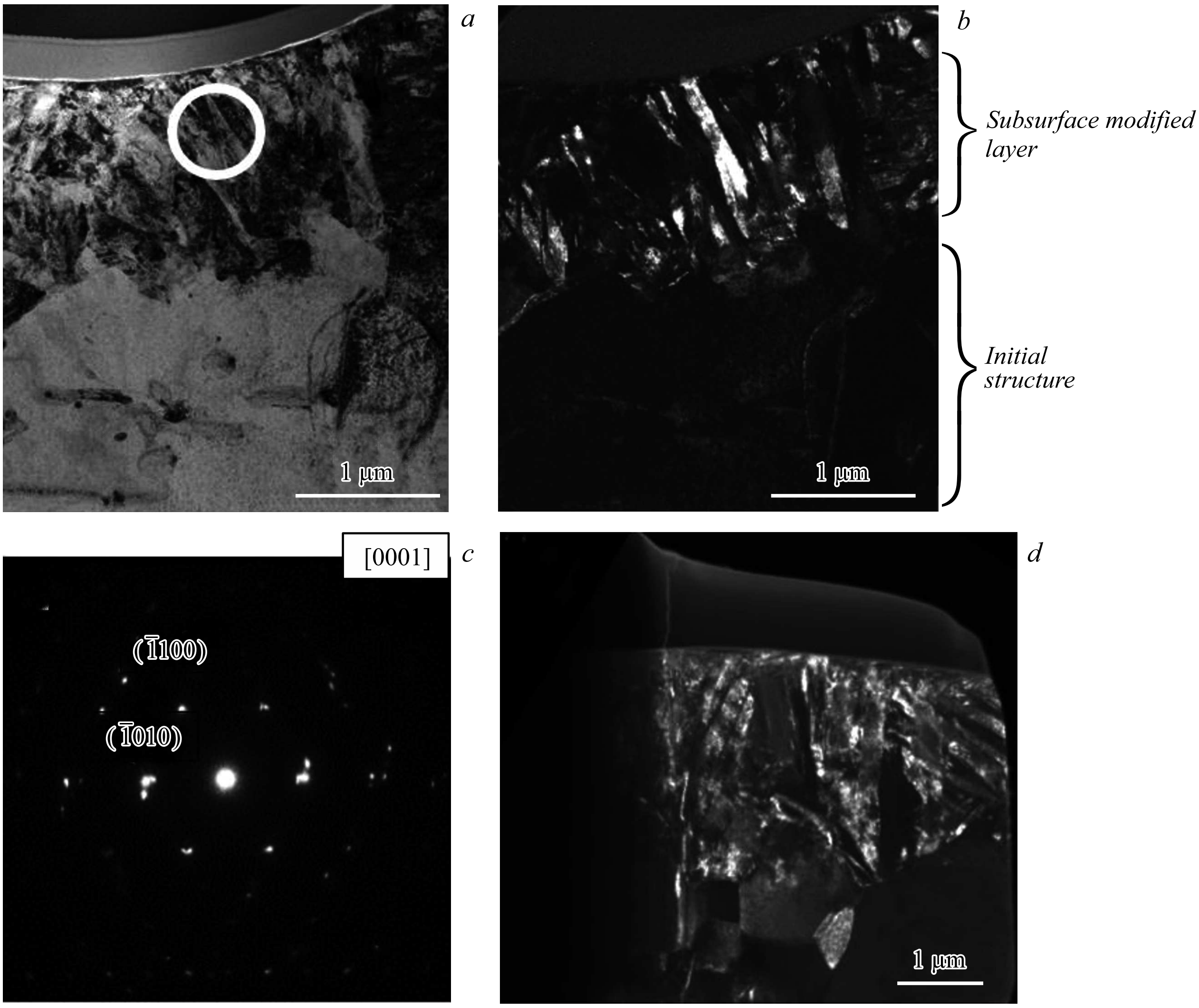

Figure 3. Microstructure of the commercially pure titanium (VT1-0) sample in the initial recrystallized state after pretreatment with a nanosecond laser beam under water (at the single pulse power density of $\sim 1\,\mathrm{GW/cm^2}$) and fatigue test ($1 \cdot 10^6$ cycles, the cycle strain of 100 MPa). *a* — bright–field TEM image; *b* — dark–field TEM image ; *c* — microdiffraction (the region it has been obtained in is marked in part *a*); *d* — a dark–field image of the initial recrystallized sample (prior to irradiation) given for comparison (transmission scanning electron microscopy).

modification of subsurface layers of the studied samples with the nanosecond laser irradiation results in an essential increase in the resistance to fatigue destruction.

TEM images obtained after the nanosecond laser irradiation and fatigue test exhibit in the near–surface layer a modified layer about $1-2\,\mu$m deep (Fig. 3, *a*, *b*). This layer was formed as a result of processing by nanosecond laser pulses under a layer of water since no differences were detected in its microstructure as compared to that after laser irradiation and prior to the fatigue test (Fig. 3, *d*). The studied alloy microstructure consists in crystallites of the elongated plate shape about 300 nm wide and $2-3\,\mu$m long. The crystallites are elongated mainly in the direction perpendicular to the sample surface just as it should be in crystallization with heat removal in the given direction. Based on the microdiffraction pattern (Fig. 3, *c*) it is possible to conclude that the crystallites misalignment is of the small–angle character (the misalignment angle below 15°). The arrangement of reflexes does not fit to the twin misalignment since in the case of twinning in the considered crystallite orientation [000$\bar{1}$] (Fig. 3, *c*) there should be observed twin reflexes located symmetrically with respect to the (1010) plane [5]. Thus, closely located reflexes from planes of the ($\bar{1}$010), ($\bar{1}$100), (0$\bar{1}$10) and other types evidence for the presence of small–angle boundaries between neighboring crystallites. We may assume that such a misalignment of the crystallites is connected with their nucleation in the molten alloy on a polycrystalline substrate with a well pronounced crystallographic texture. Thus, the crystalline substrate on which the elongated monocrystalline structural elements (crystallites) are crystallized and grow, prescribes the crystalline–structure matching between the substrate and observed blocks with small−angle mutual misalignment. This is typically observed during the growth of blocked monocrystals from liquid phase on a solid−phase crystalline substrate. Probably, it is possible to obtain various crystalline structures with a preset crystallographic orientation depending on the rolling texture that mainly prescribes the type of the crystallographic plane (on which the crystallization nucleus will be formed).

It has been revealed that processing of experimental samples of recrystallized commercially pure plate−shaped titanium VT1-0 by nanosecond laser pulses results in essential elevation of the conditional fatigue limit under a cyclic tensile load at the cycle number of up to 10^6. We have established that this effect is connected with formation in the thin (about $2\,\mu$m) subsurface layer under the impact of nanosecond laser pulses of a plate−type microstructure with elongated (mainly normally to the sample surface) microstructure elements about 300 nm in transverse size and $2-3\,\mu$m in length. Formation of the above−characterized submicrocrystalline structure confirms the earlier observed manifestation of crystallization of a thin subsurface layer melted with a laser beam in the direction perpendicular to the sample surface (in the heat removal direction).

Structural grinding of metals and alloys to the nanoscale level is known to prevent in at least one direction nucleation and evolution of cracks that occur in thin near−surface layers in the case of fatigue in the studied range of loading cycles.

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Conflict of interests

The authors declare that they have no conflict of interests.

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