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Residual stresses in Ti6Al4V alloy after surface texturing by femtosecond laser pulses

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Abstract. Surface topography and residual stresses in surface layers of α + β titanium alloy Ti6Al4V textured by 1030-nm, 320-fs-laser pulses were studied by scanning electron microscopy and X-ray diffraction analysis. It was found that multipulse laser processing leads to the formation of laser-induced periodic surface structures (LIPSS) on the surface of Ti6Al4V alloy. XRD studies showed that depending on the laser pulse fluence, both tensile and compressive residual stresses are formed in thin near-surface layers.

1. Introduction

Titanium alloys are broadly used as implant material in orthopaedic and dental field, employing their high corrosion resistance and good biocompatibility [1]. For further improvement of these and other characteristics surface modification is applied, like ion implantation [2], electrochemical coating [3], laser processing [1, 4, 5], etc.

Femtosecond laser processing is one of the promising methods in surface modification of biomaterials because of the unique periodic surface nano- or micro- scale topography formed after irradiation [6]. By controlling the unique periodic surface topography, surface wettability could be changed, and cell adhesion, proliferation, differentiation could be regulated [7]. It was found that such texturing surfaces can provide an improved bone-implant interface anchorage [8] for titanium or the reduction of bacterial adhesion and biofilm formation [9-11]. The main features of relief formation on titanium alloy surface were extensively studied in [1, 12, 13]; however, the residual stresses in the surface layers of such titanium alloy exposed to fs laser pulses have not been sufficiently studied [14]. The analysis of the residual stress state is of great technological importance because stresses can be beneficial or detrimental with respect to the mechanical properties of the material [15].

In this work, we study the formation of laser induces periodic surface structure (LIPSS) on Ti6Al4V titanium alloy, irradiated by 1030 nm femtosecond laser pulses, and investigate residual stresses in the near-surface layers after such laser processing.

2. Experimental details

In our experiment, α+β titanium alloy Ti6Al4V (VT6) with large-grain structure was used. This material was prepared in the form of rectangular slabs, whose surfaces were mechanically polished using a rotary machine LaboPol-5 (Struers) and then electrolytically etched (10% HF, 87% H_2O, 3% HNO_3).

Samples were arranged on a PC-controlled three-dimensional motorized translation stage and scanned in air by 1030-nm 320-fs Yb-fiber pulses with the maximal energy of 10 μJ in the TEM_{00} mode. An impulse frequency of 400 kHz and a scanning speed of 100 mm/s were used. Fs-laser radiation parameters — pulse energy E, single-pulse fluence F_0, and number of pulses N— are given in table 1.

Table 1. Irradiation modes **1–3** used in experiment.

No.	E, μJ	F_0, J/cm^2	N
1	0.4	0.08	100
2	2	0.4	100
3	6.7	1.2	100

The structure of the laser-affected surface was studied by scanning electron microscope (SEM) FEI Quanta 600, equipped by an energy dispersive spectrometer (EDX) (resolution - 0.2–0.25%). The surface topography was characterized by a Ntegra Aura atomic force microscope (AFM). X-ray diffraction (XRD) analysis was performed, using an ARL X'TRA X-ray diffractometer with CuK_α radiation. g(ψ,hkl)-method (grazing-incidence X-ray diffraction or GIXD) was applied for stress analysis in near-surface layers.

3. Results and discussion

3.1. Surface morphology

According to SEM, for mode **1** (F_0 = 0.08 J/cm^2) the laser-affected Ti6Al4V surface represents LIPSS with period of the structure is about 440±30 nm. LIPSS does not occur over the entire surface (figure 1a) because on the periphery of the laser spot the fluence is less then IR fs-laser grating formation threshold for titanium (~ 0.02 J/cm^2 [16]).

At F_0 = 0.4 J/cm^2 (mode **2**), LIPSS with period 710±40 nm form on the surface of titanium alloy (figure 1b). For the peak laser fluence F_0 = 1.2 J/cm^2 (mode **3**) exceeding many times spallative and hydro-dynamic fragmentation (phase-explosion) ablative thresholds 0.05 and 0.3 J/cm^2 [17], the resulting periodic surface structures extensively covered oxidized ablative products in the form of droplets and other debris fragments (figure 1c). The oxygen content in the surface layer is ~ 10 wt. %. The period of the structure is about 790±100 nm.

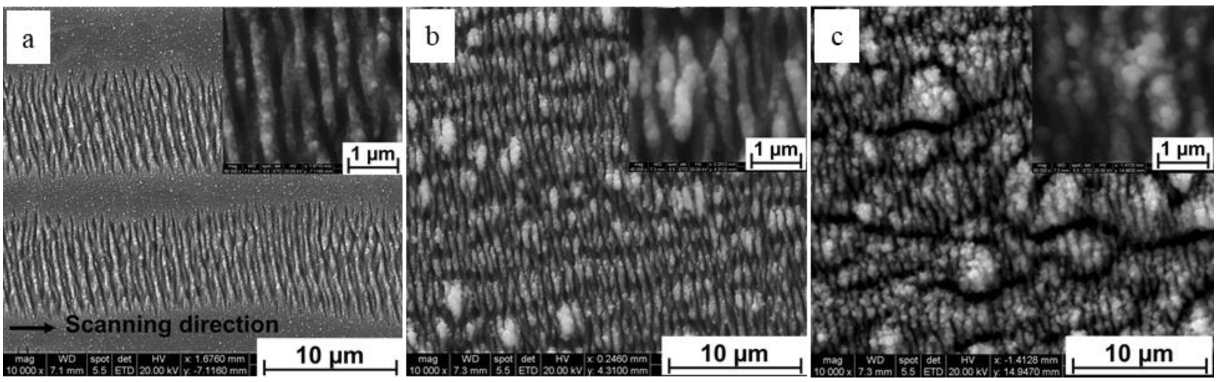

Figure 1. SEM images of samples of Ti6Al4V alloy affected by 100 fs laser pulses: (a) F_0 = 0.08 J/cm^2; (b) F_0 = 0.4 J/cm^2 and (c) F_0 = 1.2 J/cm^2.

Figure 2 shows the surface profilograms for all the sampled measured using the AFM. As can be seen from figure 2 and table 2, the surface roughness increases with increasing laser pulse fluence.

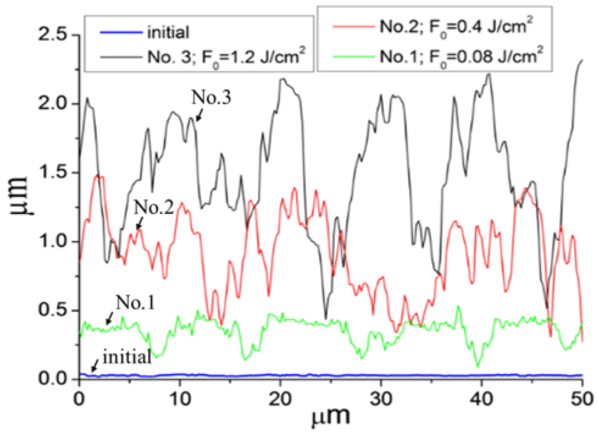

Figure 2. Profilograms of examined specimen surfaces.

Table 2. Surface roughness parameters.

	Sa, nm	Sz, nm
initial	3	63
No.1	73	320
No.2	300	1217
No.3	429	1761

S_a - average roughness.
S_z - ten-point height.

3.2. Residual stresses

At first, the residual stresses in the near-surface layers (depth ~ 14 μm) were estimated by using the conventional $\sin^2\psi$ method. In our work diffraction line (213) of α-Ti was analyzed. The main stress components $\sigma_\varphi = \sigma_1 + \sigma_2$ were estimated using equation (1):

$$\sigma_\varphi = E \cdot m / (1+\vartheta) \tag{1}$$

, where E – Young modulus, υ - Poisson coefficient, m is the slope of the plot $\varepsilon(\varphi,\ \psi)$ *versus* $\sin^2\psi$.

It was found that all laser treated samples exhibit near-zero residual stresses in the subsurface layers with a thickness of 14 μm. Measured stress value σ_φ for the initial state is ~ 7 MPa. σ_φ for samples after laser processing in modes **1–3** it is about -4 MPa, 2 MPa, and 17 MPa, respectively. Relatively low values of the measured stresses do not exceed the value of the instrumental error of ± 30 MPa.

It is known that the classical $\sin^2\psi$ method cannot be used to study thin surface-adjacent layers and layers with a high stress gradient [15]. For very thin surface-adjacent layers, the X-ray diffraction measurement of residual stress is possible by using grazing-incidence X-ray diffraction (GIXD). In our work, the g-$\sin^2$ geometry (g(ψ,hkl) method) was applied for the measurement of the interplanar spacings.

It is known, that for a biaxial stress state, the general strain-stress relation can be written in the form [15]:

$$\frac{\varepsilon_\psi^{hkl}}{S_1^{hkl}} = g(\psi, hkl) \cdot \sigma_{11} + (\sigma_{11} + \sigma_{22}) \tag{2}$$

with

$$g(\psi,hkl)=\frac{1/2S_2^{hkl}}{2S_1^{hkl}}\cdot sin^2\psi$$

Where $2S_1^{hkl}$ and $1/2S_2^{hkl}$ are diffraction elastic constants (DECs) for different diffraction peaks *hkl*.

In our work, five diffraction lines, 100, 101, 102, 110 and 103, were analyzed. DECs were calculated in ISODEC [18]. Using equation (2) and plots $\varepsilon_\psi^{hkl}/S_1^{hkl}$ *versus* $g(\psi,hkl)$ (figure 3a), macro-stresses component σ_{11} was determined.

Figure 3b shows the depth profile of residual stresses for the samples after femtosecond laser processing in mode **1-3**. Processing with the energy density of 0.08 J/cm^2 (mode **1**) and 0.4 J/cm^2 (mode **2**) leads to the formation of compressive residual stresses in surface layers with a depth of 2.3 µm. The maximum value of compressive stresses at a depth of ~ 2.3 µm for mode 2 is 340 MPa. Treatment with an energy density of 1.2 J/cm^2 (mode **3**) leads to the formation of tensile residual stresses (figure 3b).

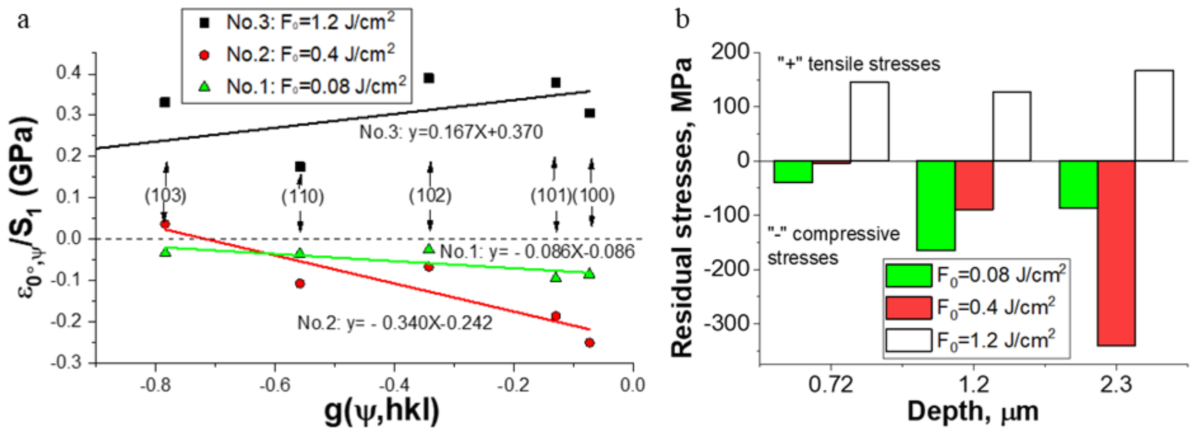

Figure 3. Stress analysis employing the *g(ψ,hkl)* method (a) and residual stresses distribution in near-surface layers of samples of Ti6Al4V alloy after femtosecond laser processing (b).

4. Conclusion

Multipulse femtosecond laser processing of Ti6Al4V titanium alloy with a single-pulse fluence in the range from 0.08 to 1.2 J/cm^2 leads to the formation of periodic surface structure. The period of the structures increases with increase of fluence. It is shown that laser processing with 0.08 and 0.4 J/cm^2 leads to the formation of compressive residual stresses in the surface layers (up to 2.3 µm). After processing 1.2 J/cm^2, tensile residual stresses are formed in the surface layers of the Ti6Al4V titanium alloy.

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