Electron Beam Nanostructurization of Titanium Alloys Surface[1]

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Abstract – Pulsed electron-beam treatment of a VT6 alloy was realized. The phase composition, the defect substructure, and the nanohardness of the Ti alloy were examined using optical microscopy, scanning and transmission electron diffraction microscopy. The regularities and mechanisms of the formation of a nanocrystalline structure responsible for the exploitation properties of the material surface layer were determined.

1. Introduction

Nanostructurization, greatly changing grain and subgrain structures, improves the exploitation properties of the majority of commercial materials [1–3]. The improvement, in this case, is attained by changing the structure and phase composition, i.e., by forming a multiphase nanostructure, rather than by alloying with expensive elements. The operating conditions of tools and device are every so often such that it is possible to improve their exploitation characteristics by nanostructuring of local material bulks (volumes of stress concentrator localization). One of the modern methods of nanostructure formation in surface layers or local material surface regions (with no considerable change in the properties of the volume as a whole) is treatment by concentrated energy flows (CEF). The CEF treatment features are superhigh (10^8–10^{10} K/s) heating rates and rather short (10^{-6}–10^{-3} s) times of high-temperature action, and this promotes its use in modification of metal materials, including Ti-based alloys, and makes the technology promising and fast-developing in modern materials science [4, 5].

The purpose of this work is to determine the regularities and mechanisms of nanostructure formation in VT6 surface layers by pulsed electron-beam treatment.

2. Experimental procedure

The material to be tested was a VT6 (Ti–6Al–4V) alloy subjected to preliminary thermomechanical treatment. The test specimens were plates of dimensions 10×10×2 mm. Low-energy (up to 20 keV) electron-beam treatment of the material was carried out on a setup "SOLO" [6] in the single pulse mode (number of pulses $N = 3$, repetition frequency of pulses $f = 0.3$ Hz) with pulse duration $\tau = 50$ μs and the energy density $E_S = 12$–30 J/cm^2. The diameter of the e-beam treatment zone is ~ 2 cm.

The structure of the irradiated surface and the cross-section of the specimens were examined by optical (OLYMPUS GX71 device with a DP70 digital camera) scanning (SEM-515 "Philips") and transmission (EM-125) electron diffraction microscopy. The mechanical characteristics of the surface layer were studied through measuring the nanohardness with a CSEM Nano Hardness Tester (the indenter load 0.5, 1.0, and 2.0 mN).

3. Results and discussion

Mechanical tests of the VT6 alloy were performed through nanoindentation before and after electron-beam treatment. The results of tests are shown in Fig. 1.

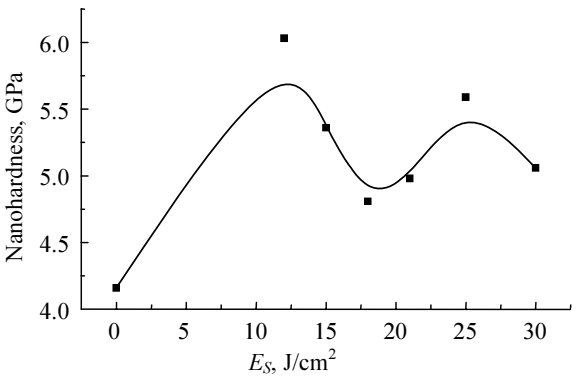

Fig. 1. Dependence of surface layer nanohardness of VT6 alloy on beam energy density

Analysis of the data presented in Fig. 1 shows that the surface nanohardness strongly depends on the mode of electron-beam treatment. The maximum nanohardness of the treated surface is reached with beam energy density $E_S = 12$ J/cm^2 and is ~ 1.5 times greater than that of the initial specimen. Electron-beam treatment with beam energy density $E_S = 25$ J/cm^2 provides somewhat lower nanohardness of the treated surface.

With the aim to elucidate the cause for this ambiguous change in the VT6 surface nanohardness, we examined the grain and intragrained structures of the material in the initial state and after electron-beam

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treatment. The grain structure evolution was studied by metallographic examination of the etched cross-section and by scanning and transmission electron diffraction microscopy. Fig. 2 shows SEM and TEM images of the grain structure of the VT6 alloy.

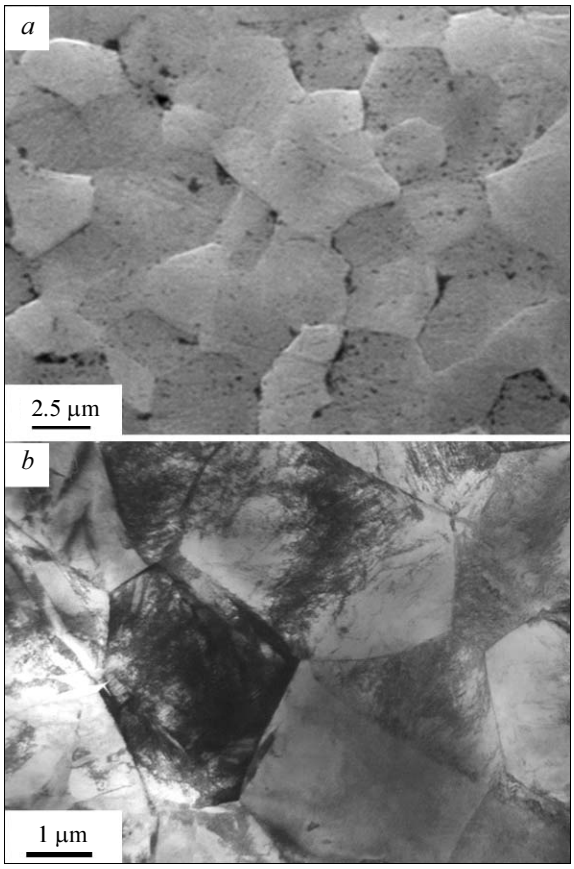

Fig. 2. Structure of VT6 alloy treated by electron beam (E_S = 12 J/cm^2): *a* – SEM image; *b* – TEM image

Statistical analysis of the grain structure shows that electron-beam treatment of the VT6 alloy at a beam energy density E_S = 12 J/cm^2 involves considerable (about double) refinement of the grain structure (Fig. 3).

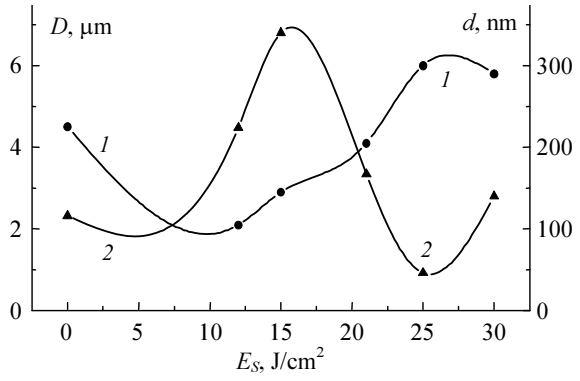

Fig. 3. Dependence of grains average size *D* (curve *1*) and average cross section size of plates of α-phase *d* (curve *2*) on beam energy density

Hence, the first peak on the dependence of the nanohardness on the beam energy density is due to the refinement of the grain structure of the surface layer, to the Hall-Petch mechanism, which takes into account hardening of a material by large-angle grain boundaries [7]. The second peak on this dependence presumably owes to the state of the intragrained structure of the surface layer.

Studies of thin foils in the initial state by transmission electron microscopy show that the test alloy is a two-phase material with grains of the α- and β-phases (Fig. 4).

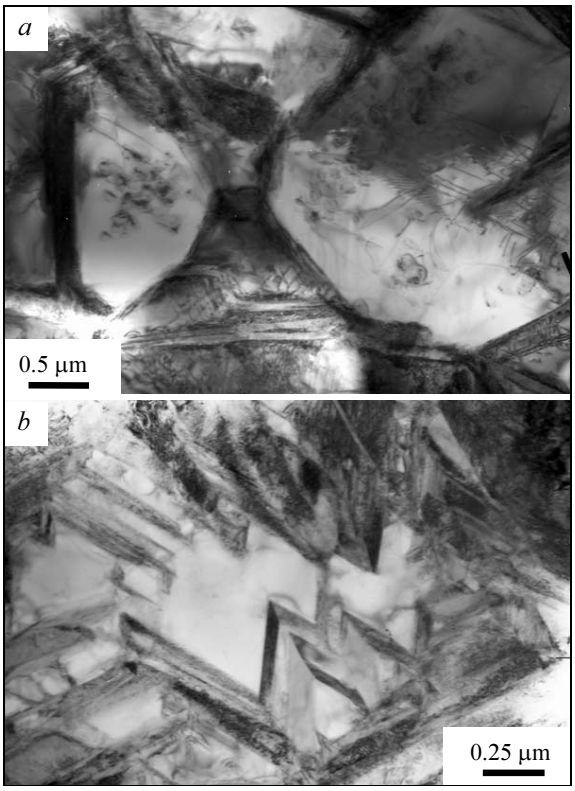

Fig. 4. TEM images of VT6 alloy structure before electron-beam treatment

After electron-beam treatment with E_S = 12 J/cm^2, the initial stage of α-titanium recrystallization is fixed. This process involves the formation of grains with size of 2–5 μm (Fig. 3). In individual fine grains, a lamellar structure with crystals of cross sectional dimension 150–300 nm is found. The structure results apparently from the $\alpha \Rightarrow \beta \Rightarrow \alpha$ transformation.

With E_S = 15…18 J/cm^2, a two-phase ($\alpha + \beta$) polycrystalline structure is fixed. The partial $\alpha \Rightarrow \beta \Rightarrow \alpha$ martensite transformation is accompanied by the formation of packet and lamellar martensite crystals (Fig. 5).

With E_S = 25 J/cm^2 (the second peak on the dependence of the nanohardness on the beam energy density), the superhigh heating and cooling rates of the surface layer causes a polymorphous $\alpha \Rightarrow \beta \Rightarrow \alpha$

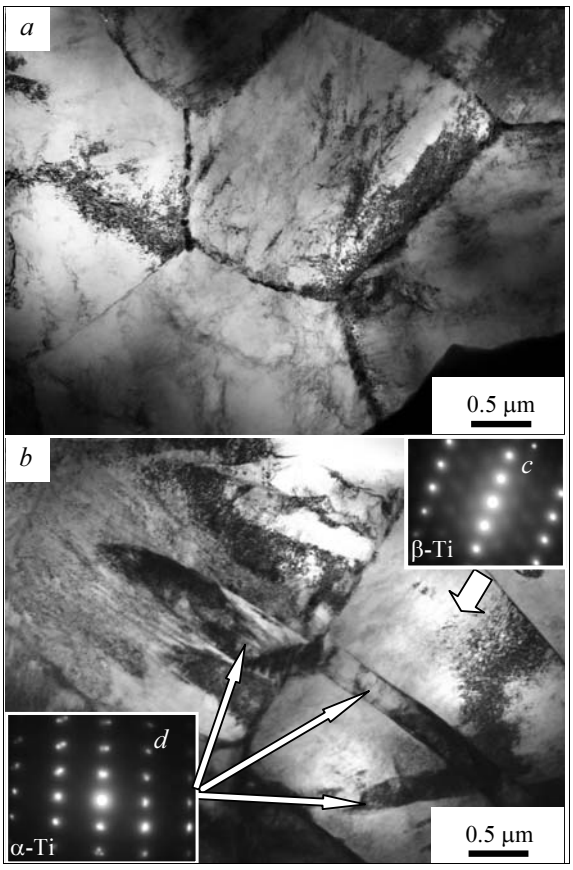

Fig. 5. TEM image of VT6 alloy structure treated by electron beam (E_S = 15 J/cm^2); *a*, *b* – TEM bright field image; *c*, *d* – diffraction pattern. Arrows show crystals of α-Ti and grains of β-Ti (*b*)

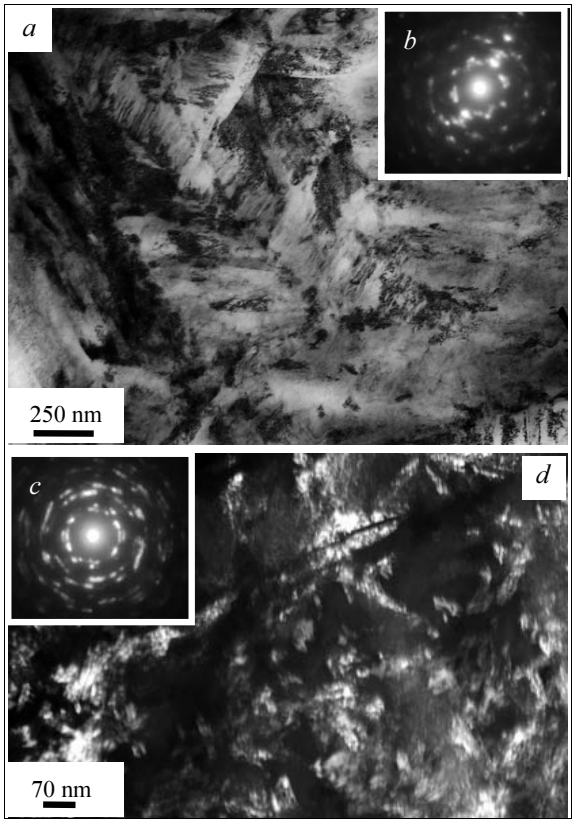

Fig. 6. TEM image of VT6 alloy structure treated by electron beam (E_S = 25 J/cm^2); *a* – TEM bright field image; *b*, *c* – diffraction pattern to (*a*) and (*d*) correspondingly; *d* – TEM dark field image obtained in [101] α-Ti reflection

martensite transformation in the grain volume with the attendant formation of crystallites of cross section dimension 10–20 nm (Fig. 6).

Analysis of the foregoing examination of the VT6 alloy treated by the pulse electron beam shows that in a certain range of the e-beam parameters, the high-rate heating, melting, crystallization, and quenching initiated by the treatment make it possible to form a nanocrystalline structure with α-phase crystallites of cross-sectional dimension 10–20 nm in the surface layer.

Judging from the morphological character, this structure is formed by the martensite $\beta \Rightarrow \alpha$ transformation.

The nonmonotonic change in the surface nanohardness of the material treated with different electron beam energy densities is governed by a complex response of the VT6 structure to the pulsed action. With a relatively low beam energy density (the first peak on the dependence of the nanohardness on the electron beam density, Fig. 1), the decisive factor responsible for the nanohardness is considerable refinement of the material grain structure (the so-called Hall-Petch effect). The second peak of this dependence is due to the formation of nanocrystalline structure in the surface layer by the polymorphous $\beta \Rightarrow \alpha$ transformation.

4. Conclusions

Pulsed electron-beam treatment of the VT6 titanium alloy was realized. The phase composition, the defect substructure and the hardness of the treated specimen surface were examined. The nonmonotonic character of the changes in the strength properties of the VT6 alloy treated by the electron beam was revealed. The mechanisms responsible for this effect were elucidated. The mode of electron-beam treatment that ensures a nanocrystalline structure formation in the VT6 surface layer was determined. It is shown that the formation of the nanocrystalline structure results from the polymorphous $\beta \Rightarrow \alpha$ transformation by the martensite mechanism.

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