
**MODIFICATION OF THE SURFACE, PARTICULARLY BY BEAMS
OF CHARGED PARTICLES AND PHOTON AND PLASMA FLUXES**

**Effect of High-Power Ion Beams
on the Surface Topography and Structure of the Subsurface Layer
of Submicrocrystalline Titanium Alloys**

M. V. Zhidkov^{a, *}, A. E. Ligachev^{b, **}, Yu. R. Kolobov^{c, *},
G. V. Potemkin^{d, ****}, and G. E. Remnev^{d, *****}**

^aBelgorod State National Research University, Belgorod, 308034 Russia

^bProkhorov General Physics Institute, Russian Academy of Sciences, Moscow, 119991 Russia

^cInstitute of Problems of Chemical Physics, Russian Academy of Sciences, Chernogolovka, Moscow oblast, 142432 Russia

^dTomsk Polytechnic University, Tomsk, 634050 Russia

*e-mail: zhidkov@bsu.edu.ru

**e-mail: carbin@yandex.ru

***e-mail: kolobov@_bsu.edu.ru

****e-mail: ep.gvp@yandex.ru

*****e-mail: remnev06@mail.ru

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Abstract—The influence of high-power beams of carbon ions (the ion energy is 250 keV; the pulse duration is ~100 ns; the current density in the pulse is 150–200 A/cm²; the surface energy density of a single pulse is $j \sim 3$ J/cm² under the irradiation of the samples of the VT1-0 titanium alloy and $j \sim 1$ J/cm² for the treatment of the samples of the VT6 titanium alloy; and the number of pulses is 1, 5, 10, and 50) on the surface topography and structure-phase state of the subsurface layer of submicrocrystalline titanium alloys VT1-0 and VT6 is studied. The sample surface before irradiation is preliminarily mechanically grinded and polished. It is shown that surface defects are formed on the alloy surface after irradiation. These are craters of various shapes and geometry with diameter from fractions of micrometer to 80–100 μm. Herewith, the grain structure in the subsurface layer becomes more uniform in size and degree of grain equiaxity. A rather homogeneous structure is characteristic of the state of the VT1-0 titanium alloy; the average grain size is ~0.31 μm, while that one the VT6 alloy is ~0.9 μm. The grain growth in the transverse direction to 0.54 μm is observed after one irradiation pulse in the subsurface layer of the VT1-0 alloy (at $j \sim 3$ J/cm²), while the grain size for the VT6 alloy ($j \sim 1$ J/cm²) decreases to ~0.54 μm. The average grain size in the subsurface layer after 50 pulses reaches ~2.2 μm for the VT1-0 alloy and ~1.6 μm for the VT6 alloy. It should be noted that a rather homogeneous grain structure with equiaxial grains is formed for both alloys already after the effect of one pulse of the high-power ion beam.

Keywords: titanium alloys, high-power ion beams, craters

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INTRODUCTION

Titanium and its alloys are some of the most important construction and functional materials. The widespread use of titanium alloys in aviation, shipbuilding, chemical industry, and medicine is due to their favorable combination of high mechanical properties and small specific weight, as well as high corrosion resistance. Nevertheless, important application spheres of titanium alloys require a further increase in their operational characteristics. It is known that operational properties of wares made of metallic materials, including titanium and its alloys, are, in many aspects, determined by their surface treatment quality and the

state of subsurface layers. One promising direction in the field of development of new treatment technologies of materials is the surface modification of metals and alloys by high-power pulsed beams of accelerated ions (HPIB) [1–3].

The investigation into the interaction of high-power pulsed beams of charged particles with metallic materials has been performed for many years. The pioneering investigations into the treatment of tool alloys by a high-power pulsed beam of carbon ions (the TONUS accelerator) were performed in 1981 at the Research Institute of Nuclear Physics at the Tomsk Polytechnic Institute [1, 2]. Then, P6M9 tool steel was

selected as a target. Subsequently, experiments on the irradiation of both pure metals such as beryllium, Armco iron, electrolytic purity copper, titanium, aluminum single crystals, and steels of various brands, as well as hard alloys, were started [3–6]. The goal of these studies was to investigate the possibility of the process application of high-power pulsed electron and ion beams for varying not only the structural phase state of the surface and subsurface layer, but also physico-mechanical and physicochemical properties of wares made of metals and alloys. The experiments on studying processes associated with the structural reconstruction, physicochemical transformations, and changes in the surface topography and subsurface material layers after the HPIB effect were continued in subsequent works of both domestic and foreign scientists, and the possibility of improving the complex of operational characteristics of various metals and alloys after such surface treatment was shown [7–14]. Despite the continuing active development of this direction, many fundamental questions of the interaction of HPIB with metallic materials remain currently poorly studied. In particular, one important question is the revelation of the formation mechanism of microcraters on the surface of metallic and semiconductor materials, which is still unclear and has not been studied in detail experimentally. The peculiarities of the influence of nanosecond pulsed HPIBs on the surface and subsurface layers of submicrocrystalline and nanostructured materials and alloys, which are known to have low thermal stability of the structure and properties, are also almost unstudied.

The goal of our work was to investigate the peculiarities of changes of the surface topography and structure of subsurface layers of titanium submicrocrystalline alloys after the effect of a high-power pulsed ion beam.

EXPERIMENTAL

We selected submicrocrystalline (SMC) titanium alloys VT1-0 and VT6 fabricated by combining methods of longitudinal and transverse-screw rolling as the object of the investigation. The sample surface before irradiation was preliminarily mechanically grinded and polished to high finish using a LaboPol-5 installation (Struers, Denmark) applying abrasive paper and diamond suspensions.

Irradiation by a high-power ion beam (HPIB) consisting of carbon ions and impurity of hydrogen ions was performed using a TEMP manufacturing accelerator in the strip diode focusing geometry (Tomsk Polytechnic University) under the following modes: the ion energy was 250 keV, the pulse duration was ~100 ns at the half-height, and the pulse current density was 150–200 A/cm² (the current density in the working beam region varied considerably weakly, notably, in limits of 20%; the current density drop was more abrupt at a distance of >2 cm from the beam

center). The residual gas pressure in a chamber was in limits of $(3-4) \times 10^{-4}$ mmHg. The surface density of the energy of the single pulse was $j \sim 3$ J/cm² when irradiating the samples of the VT1-0 titanium alloy and $j \sim 1$ J/cm² when treating the samples of the VT6 titanium alloy. The number of pulses was 1, 5, 10, and 50. The pulse-to-pulse stability of irradiation modes is conditioned mainly by the spread of the accelerating voltage across the diode and, correspondingly, by the spread of the ion current density in the magnetically insulated diode. In totality, these factors and others, to a lesser extent, determine the stability of pulse-to-pulse parameters of the current density in limits of $\pm 10\%$. It should be noted that the plasma formation method applied in the diode [1] does not lead to a decrease in the current density or its spread with an increase in the number of pulses after the series of training shots.

The investigation into the topography of the surface, structure phase state, and crystallographic texture of subsurface layers of titanium alloys was performed using a Quanta 600 scanning electron microscope (FEI, Netherlands), including the use of the automatic analysis procedure of diffraction patterns of backscattered electrons (EBSD analysis).

RESULTS AND DISCUSSION

After the effect of one HPIB pulse, variously shaped and sized crater-type surface defects are formed on the titanium alloy surface (Fig. 1). For example, single one-ring (Fig. 1b) and multiple-ring (Fig. 1c) craters mainly prevail on the surface of the VT1-0 titanium alloy. They have the ring-shaped “overlap” region of the melted material in the HPIB effect zone (it is marked by a dashed line). The surface structure of such craters almost does not differ from the overlap surface structure, and both the rounded hole (no larger than 100–200 nm in depth) and bulge can form in the crater center after the HPIB effect (Figs. 1b–1d). Craters can overlap each other (Fig. 1d), and extended surface defects appear on the alloy surface under their multiple overlap (Fig. 1e). The central part of these defects contains bridges and particles of the solidified drop phase (Fig. 1f). These surface defects can be lined in the form of lines (Fig. 2a). The lines from craters usually coincide with the deformation direction during the material fabrication and grinding and polishing direction at the last stage of preparing the titanium samples before their HPIB irradiation.

The average crater size for the VT1-0 titanium alloy after the effect of one HPIB pulse is ~50 μm , while the surface density of craters reaches $\sim 2 \times 10^4$ cm⁻².

When irradiating the VT6 titanium alloy by one pulse with beam energy density $j \sim 1$ J/cm², microcraters with a substantially smaller average size (~8 μm) are formed on the sample surface (Fig. 2b); herewith, the

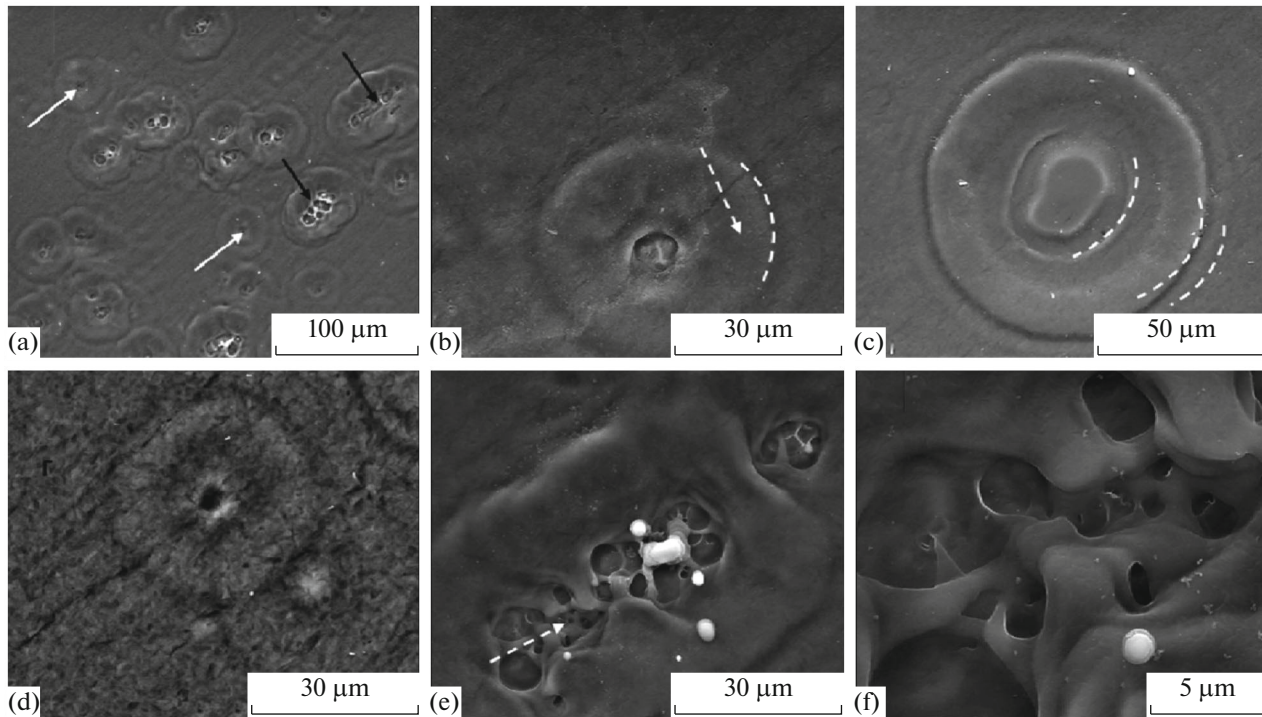


Fig. 1. Images of craters formed on the surface of the VT1-0 titanium alloy after the effect of one HPIB pulse ($j \sim 3 \text{ J/cm}^2$) formed by a scanning electron microscope (SEM). (a) White arrows show single craters, and black arrows show surface defects consisting of several craters; (b) white dashed arrows mark the “overlap” zone; (b, c) crater rings are marked by a white dashed line and (e) white dashed arrow shows the central region of the extended defects consisting of craters.

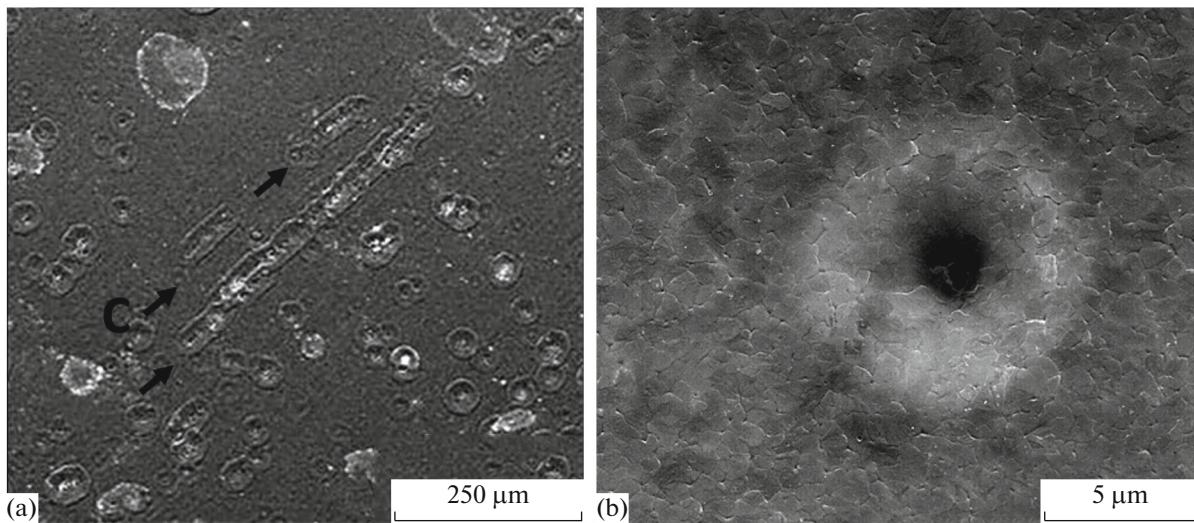


Fig. 2. (a) Surface of the VT1-0 alloy after the effect ($j \sim 3 \text{ J/cm}^2$) HPIB pulse and (b) SEM image of the characteristic microcrater formed on the surface of the VT6 titanium alloy. (a) Black arrows show banding in the arrangement of craters.

crater density on the irradiated surface is $\sim 5 \times 10^4 \text{ cm}^{-2}$. The average crater size and their surface density increase with an increase in the pulse number to 5 and 10 (Figs. 3a, 3b), but the observed crater surface density substantially decrease in the case of 50 pulses (Fig. 3c) (to $\sim 10^3 \text{ cm}^{-2}$).

The crater surface density for the VT1-0 titanium alloy, on the contrary, decreases, and pronounced craters are unobservable on the surface after the effect of 50 pulses (Figs. 3d–3f).

To date, there are various assumptions on the nature and formation mechanisms of craters. Accord-

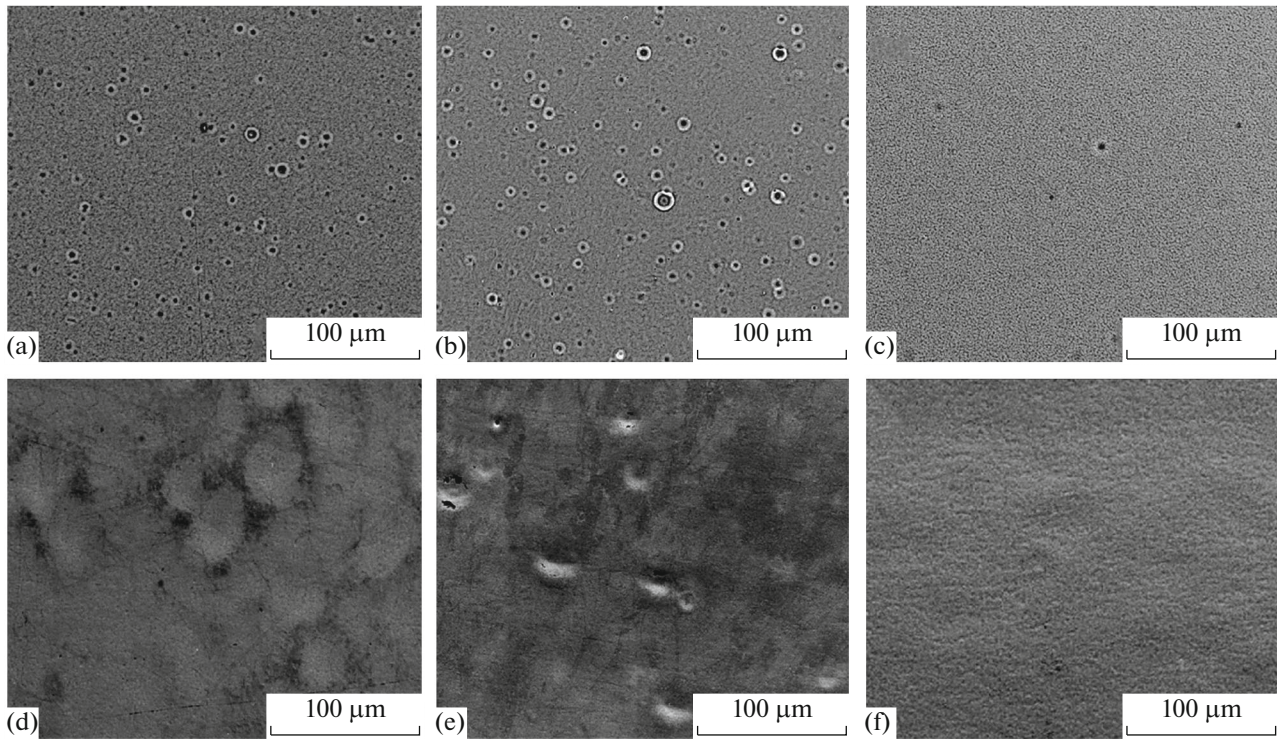


Fig. 3. SEM images of the topography of titanium alloys (a–c) VT6 and (d–f) VT1-0 after the effect of 5, 10, and 50 pulses, respectively.

ing to [15], their probable appearance mechanism is the presence of nonuniformities of the ion beam in the pulse, including those as the result of its lamination or filamentation. Due to this, local melting, boiling, and evaporation of the subsurface material layer can occur. The ion beam nonuniformity in the pulse increases with an increase in the current density, which in turn conditions the rise in crater density and sizes [16–18]. When the pulse of charged particles with a density power on the surface above 104 W/cm^2 affects the irradiated surface, melt formation is possible in the irradiation place, depending on thermal properties of the target under irradiation. The characteristic occurrence time of the melt is comparable with the irradiation pulse duration (for pulses shorter than 1 ms); i.e., it is much shorter than the crater formation time. On the contrary, when irradiation is performed by a sequential pulse series, then “washing-off” (partial or complete) of clusters appeared under the effect of the previous pulse occurs on the surface due to the effect of the subsequent pulse of generation (Figs. 3d–3f).

The crater formation can be also associated with the presence of gas impurities in the material and escape of gas bubbles on the melted surface [19, 20]. However, the presence of coarse and fine porosity filled with gases was not found in our case, and this crater-formation mechanism is low-probable but possible in principle. This mechanism cannot be

excluded, but it is still not confirmed by a rigorous experiment.

The authors of [18] proposed an HPIB model in which it was shown using theoretical calculations that craters can be formed at the power density in the pulse of $>10^{11} \text{ W/m}^2$ due to the plasma torch formation with the subsequent appearance of gravity waves and Richtmyer–Meshkov instability of the plasma–melt interface. This kind of crater-formation mechanism undoubtedly can occur.

The authors of [22] experimentally studied the internal structure of a crater on a steel surface using the preparation method of thin foils by an ion beam (the known cross-section procedure) and investigated them using transmission electron microscopy. It is shown that the subsurface crater layer is presented by columnar grains with an average size (length) of $\sim 2 \mu\text{m}$ elongated along the direction to the surface, and the grain unequianisotropy coefficient (K_{gu}) decreases with the shift from the periphery crater ring to its central part. The character of the formed structure undoubtedly evidences the occurrence of melting and ultrarapid crystallization processes in a thin surface layer of the HPIB-irradiated material during crater formation.

In addition to the change in surface topography, a change in the structural state of subsurface layers of titanium alloys due to the HPIB irradiation is revealed. A rather homogeneous structure with $K_{\text{gu}} \sim 2$ is char-

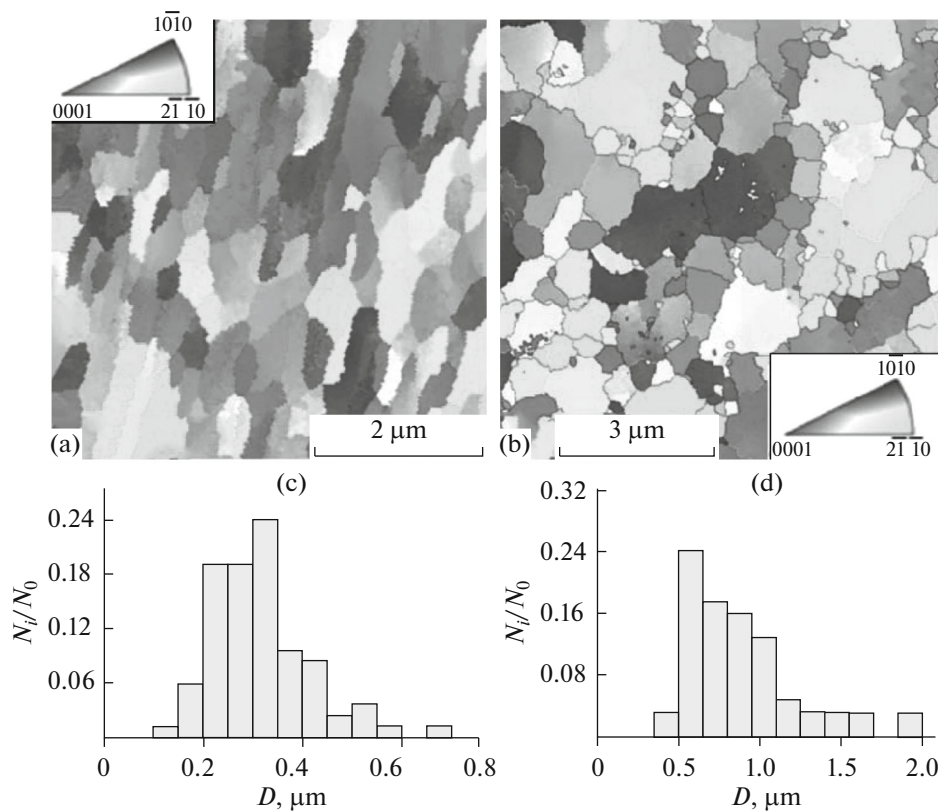


Fig. 4. Structure of subsurface layers of titanium alloys (a) VT1-0 and (b) VT6 in the initial state (the grain structure in the color gamma of the crystallographic triangle of the titanium hcp lattice according to the data of electron backscattered diffraction (EBSD analysis) and grain-size distribution histograms for alloys (c) VT1-0 and (d) VT6 in the initial state.

acteristic of the initial submicrocrystalline (SMC) state of the VT1-0 titanium alloy (Fig. 4a), and the average transverse grain size is $\sim 0.31 \mu\text{m}$ (Fig. 4c). This grain size for the initial SMC state of the VT6 alloy (Figs. 5a, 4d) is $\sim 0.9 \mu\text{m}$ at $K_{\text{gu}} \sim 1$.

The grain growth is observed in the subsurface layer of the VT1-0 alloy sample after the HPIB irradiation with an energy density of 3 J/cm^2 (Fig. 5a). Their average size in the transverse direction was $\sim 0.54 \mu\text{m}$ after one irradiation pulse (Fig. 5c). The grain size in the subsurface layer of the VT6 sample after irradiation by one HPIB pulse with $j = 1 \text{ J/cm}^2$ decreases to $\sim 0.54 \mu\text{m}$. Herewith, as is seen in SEM images recorded in the relief mode, grain boundaries and their separate grains are etched.

The average grain size in the subsurface layer after 50 pulses (Fig. 6) reaches $\sim 2.2 \mu\text{m}$ for the VT1-0 alloy and $\sim 1.6 \mu\text{m}$ for the VT6 alloy. It is noteworthy that a rather homogeneous grain structure with uniaxial grains is formed for both alloys after one HPIB pulse.

In general, our results show that thermal and shockwave processes appearing under the HPIB effect can lead to a substantial change in the structure of the surface and subsurface layers (including the materials preliminarily treated by plastic deformation) with the

formation of a microstructure more uniform in size and with a higher degree of equiaxity of grains. HPIB treatment opens up prospects for the controlled surface engineering and formation of surface ultrafine (UF), submicrocrystalline (SMC), or nanostructured (NS) states with the specified grain size and improved physicochemical properties. A change in the surface topography due to this effect, in particular, the formation of surface defects, is rather undesirable, because the craters can serve as stress concentrators on the surface or potential sources of accelerated corrosion [15].

It should be noted that, for the controlled relief formation on the surface of materials in SMC and NS states with decreased thermal stability when it is required to conserve the unrecrystallized microstructure of subsurface layers, the laser effect by ultrashort (femtosecond and nanosecond duration) pulses is best. Despite the fact that colossal values of the peak power density can be attained under the effect of such pulses (for example, these quantities in our experiment are $\sim 10^7 \text{ W/cm}^2$ for HPIB and 10^{13} W/cm^2 for the femtosecond laser irradiation (FLI)), the depth of the thermal influence zone is extremely small under such pulse duration. No zone of the thermal influence in subsurface layers was found in our investigations per-

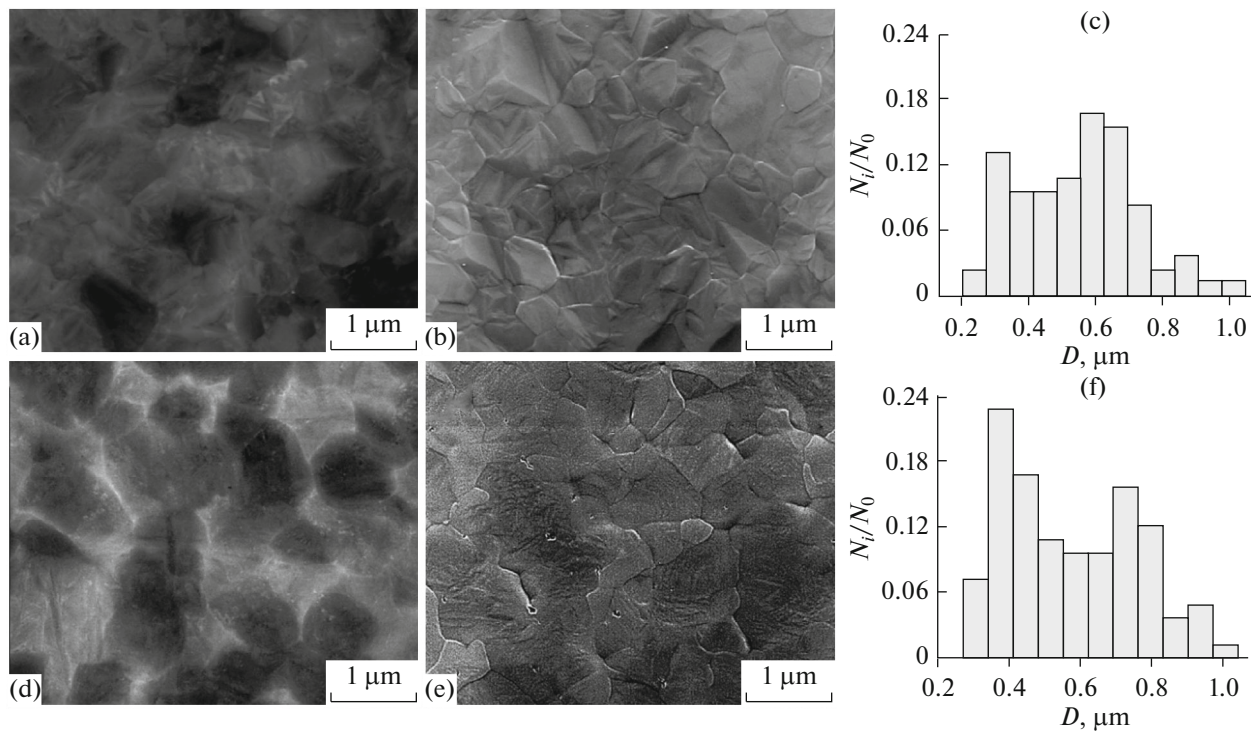


Fig. 5. Structure of subsurface regions (SEM images) and distribution histograms of the grain size for titanium alloys (a–c) VT1-0 and (d–f) VT6 after one HPIB pulse. (a, d) Detector of backscattered electrons and (b, e) detector of secondary electrons.

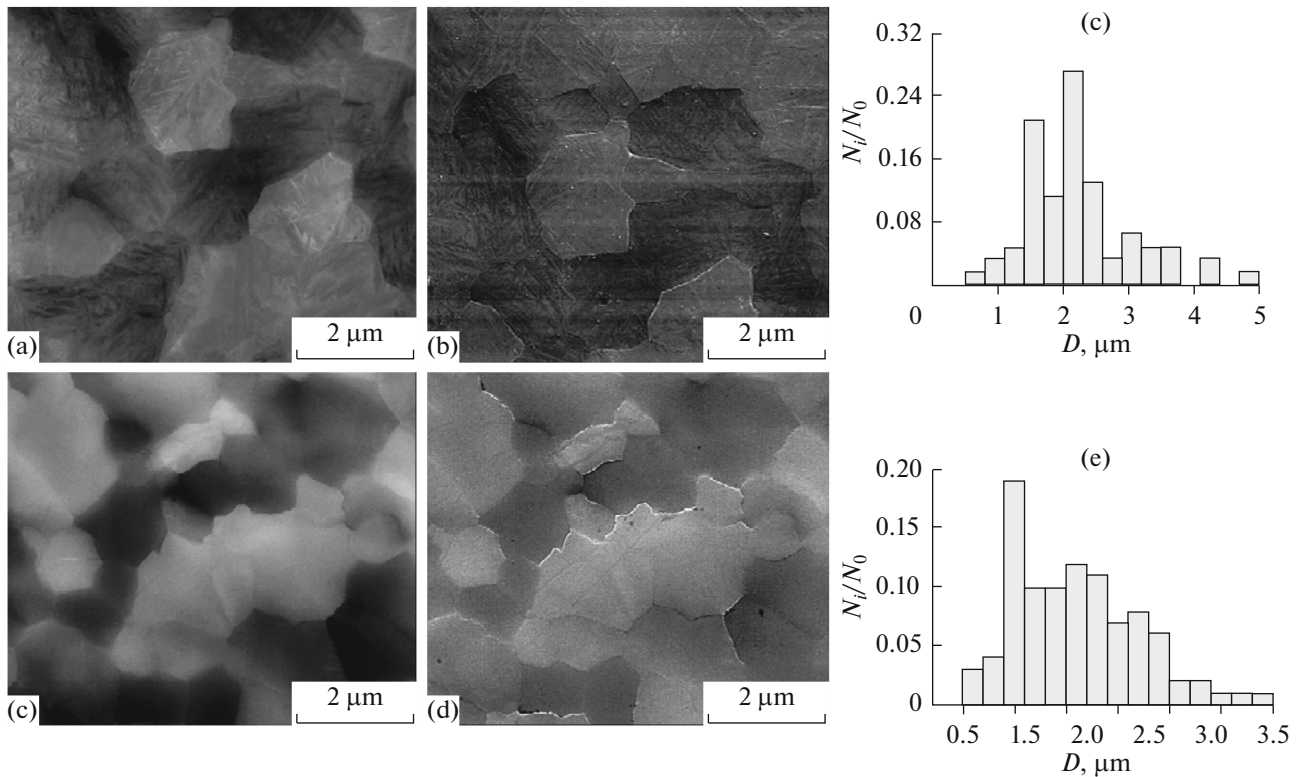


Fig. 6. Structure of subsurface regions (SEM images) and distribution histograms of the grain size for titanium alloys (a–c) VT1-0 and (d–f) VT6 after 50 HPIB pulse. (a, d) Detector of backscattered electrons and (b, e) detector of secondary electrons.

formed previously for SMC VT6 and VT1-0 alloys at close energy densities (~ 1 and 3 J/cm^2) [23, 24].

CONCLUSIONS

(i) The surface irradiation of VT1-0 and VT6 titanium alloys by the high-power pulsed beam of carbon ions leads to the formation of surface defects in the form of craters of various shapes and geometries with diameters from hundredths of a micrometer to $80\text{--}100 \mu\text{m}$.

(ii) HPIB treatment of submicrocrystalline titanium alloys VT1-0 and VT6 causes a substantial variation in the structure of the surface and subsurface layers with the formation of microstructure grains more uniform in size and degree of uniaxiality.

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CONFLICT OF INTEREST

The authors claim that they have no conflict of interest.

REFERENCES

- Logachev, E.I., Remnev, G.E., and Usov, Yu.P., The acceleration of ions from an explosive-emission-plasma, *Pis'ma Zh. Tekh. Fiz.*, 1980, vol. 22, no. 6, pp. 1404–1406.
- Didenko, A.N., Kuznetsov, B.I., and Remnev, G.E., Study of the influence of high-current electron and ion beams irradiation on the surface properties of tool steels, in *Tezisy dokladov VI Vsesoyuznoi konferentsii po primeneniyu elektronno-ionnoi tekhnologii v narodnom khozyaistve* (Abstracts of the All-Union Conf. on the Application of the Electron–Ion Technology in the National Economy), Tbilisi: NIET, 1981, pp. 110–111.
- Didenko, A.N., Remnev, G.E., and Ligachev, A.E., The processes of strengthening and improving the performance of alloys irradiated by high-power ion beams, in *Tezisy dokladov VI Vsesoyuznogo simpoziuma po sil'notochnoi impul'snoi elektronike* (Abstracts of the VI All-Union Conf. on High-Current Electronics), Tomsk, Inst. Sil'notochn. Elektron. Sib. Otd. Akad. Nauk SSSR, 1986, pp. 163–166.
- Didenko, A.N., Remnev, G.E., Chistjakov, S.A., and Ligachev, A.E., Definition of recoil pulse and removal of target mass under effect of high power beams (HPIB), in *Proc. 6th Int. Conf. of High-Power Particle Beams*, Kobe, Japan, 1986, pp. 77–81.
- Pogrebnyak, A.D., Remnev, G.E., Chistyakov, S.A., and Ligachev, A.E., Modification of metal properties under the effect of high-power ion beams, *Izv. Vyssh. Uchebn. Zaved. Fiz.*, 1987, vol. 30, no. 1, pp. 52–65.
- Pogrebnyak, A.D., Remnev, G.E., Kurakin, I.B., and Ligachev, A.E., Structural, physical and chemical changes induced in metals and alloys exposed to high power ion beams, *Nucl. Instrum. Meth. Phys. Res. Sect. B*, 1989, vol. 36, no. 3, pp. 286–305.
- Didenko, A.N., Ligachev, A.E., and Kurakin, I.B., *Vozdeistvie puchkov zaryazhennykh chastits na poverkhnost' metallov i splavov* (Effects of Charged Particle Beams on the Surface of Metals and Alloys), Moscow: Ergoatomizdat, 1987.
- Valeev, A.N., Pogrebnyak, A.D., and Plotnikov, S.V., Radiatsionno-mekhanicheskie efekty v tverdykh telakh pri obluchenii vysokointensivnymi impul'snymi elektronnymi i ionnymi puchkami (Radiation-and-Mechanical Effects in Solids under High-Intensity Pulsed Electron and Ion Beams), Ust'-Kamenogorsk: VKTU, 1998.
- Shulov, V.A., Remnev, G.E., and Kashcheev, V.N., Effect of ion-beam treatment by high-power pulsed beams on the physical and chemical state of the surface layers and fatigue strength of the EP7181 alloy, *Fiz. Khim. Obrab. Mater.*, 1992, no. 6, pp. 28–35.
- Shulov, V.A., Nochovnaya, N.A., Remnev, G.E., Pellerin, F., and Monge-Cadet, P., High-power ion beam treatment application for properties modification of refractory alloys, *Surf. Coat. Technol.*, 1998, vol. 99, pp. 74–81.
- Pogrebnyak, A.D., Ivanov, Yu.F., Lebed', A.G., Valyaev, A.N., Renk, T., Tompson, M.O., and Zhao, V., The result of intense pulsed ion-beam action on the properties of carbon and stainless steel, *Metallofiz. Noveish. Tekhnol.*, 2000, vol. 22, no. 10, pp. 18–24.
- Xian-xiu, M., Sheng-zhi, H., Teng-cai, M., Ying-min, W., and Zhen-min, L., Microstructure and wear resistance of high-speed steel treated with intense pulsed ion beam, *Nucl. Instrum. Meth. Phys. Res. Sect. B*, 2005, vol. 239, pp. 152–158.
- Mei, X.X., Sun, W.F., Hao, S.Z., Ma, T.C., and Dong, C., Surface modification of high-speed steel by intense pulsed ion beam irradiation, *Surf. Coat. Technol.*, 2007, vol. 201, pp. 5072–5076.
- Wang, X., Lei, M.K., and Zhang, J.S., Surface modification of 316L stainless steel with high-intensity pulsed ion beams, *Surf. Coat. Technol.*, 2007, vol. 201, pp. 5884–5890.
- Pogrebnyak, A.D. and Kul'ment'eva, O.P., Structure-phase transformations in near-surface layers and properties of metal materials after the pulse effect of particle beams, *Fiz. Inzh. Poverkhn.*, 2003, vol. 1, no. 2, pp. 110–136.
- Anishchik, V.M. and Uglov, V.V., *Modifikatsiya instrumental'nykh materialov ionnymi i plazmennymi puchkami* (Modification of Instrumental Materials by Ion and Plasma Beams), Minsk: BGU, 2003.
- Korotaev, A.D., Tyumentsev, A.N., Pochivalov, Yu.I., Ovchinnikov, S.V., Remnev, G.E., and Isakov, I.F., Structure-phase state of the surface layer of metal targets under the effect of high-power ion beams, *Fiz. Met. Metalloved.*, 1996, vol. 81, no. 5, pp. 118–127.

18. Volkov, N.B., Maier, A.E., and Yalovets, A.P., On the mechanism of cratering on solid surfaces exposed to an intense charged particle beam, *Tech. Phys. Russ. J. Appl. Phys.*, 2002, vol. 47, no. 8, pp. 968–977.
19. Brekhovskikh, V.F., Rykalin, N.N., and Uglov, A.A., Revisiting the possible influence of gas content in metals on the laser beam area, *Dokl. Akad. Nauk SSSR*, 1970, vol. 190, no. 5, pp. 1059–1062.
20. Demidov, B.A., Efremov, V.P., Ivkin, M.V., Ivonin, I.A., Petrov, V.A., and Fortov, V.E., Determination of the dynamic characteristics of aerogels in the energy-release zone of a high-power electron beam, *Tech. Phys. Russ. J. Appl. Phys.*, 1998, vol. 43, no. 10, pp. 1239–1246.
21. Korotaev, A.D., Tyumentsev, A.N., Pinzhin, Yu.P., and Remnev, G.E., Features of the morphology, defect substructure, and phase composition of metal and alloy surfaces upon high-power ion beam irradiation, *Surf. Coat. Technol.*, 2004, vol. 185, pp. 38–49.
22. Zhidkov, M., Ligachev, A.E., Potemkin, G.V., Manokhin, S.S., Remnev, G.E., and Kolobov, Yu.R., Study of the structure of crater at the surface of 12Cr18Ni10Ti steel irradiated by high-power pulsed ion beam, *Inorg. Mater. Appl. Res.*, 2018, vol. 9, no. 3, pp. 376–378.
23. Kolobov, Yu.R., Golosov, E.V., Vershinina, T.N., Zhidkov, M.V., Ionin, A.A., Kudryashov, S.I., Makarov, S.V., Seleznev, L.V., Sinitsyn, D.V., and Ligachev, A.E., Structural transformation and residual stresses in surface layers of alpha plus beta titanium alloys nanotextured by femtosecond laser pulses, *Appl. Phys. A*, 2015, vol. 119, no. 1, pp. 241–247.
24. Kolobov, Yu.R., Zhidkov, M.V., Golosov, E.V., Vershinina, T.N., Kudryashov, S.I., Makarov, S.V., Ionin, A.A., and Ligachev, A.E., Phase composition and structure of femtosecond laser produced oxide layer on VT6 alloy surface, *Laser Phys. Lett.*, 2016, vol. 1, no. 7, paper 076103.

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