МАТЕРИАЛОВЕДЕНИЕ В МАШИНОСТРОЕНИИ

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STRUCTURE OF COMMERCIAL TITANIUM SUBJECTED TO LOW-TEMPERATURE ION NITRIDING

The structural-phase state of commercial titanium of grades BT1-00 (VT1-00) and BT1-0 (VT1-0) has been studied in the initial state and after various types of low-temperature ion nitriding. In the initial state, the VT1-00 and VT1-0 alloys have a single-phase α-Ti structure with a hexagonal close-packed crystal lattice. The hardness of titanium is 140–150 HV 10. It has been shown that ion-beam nitriding of VT1-00 alloy at low temperatures of 350 and 450 °C leads to the formation of thin (up to 5 μm) nitrogen-hardened layers with a hardness of 160–180 HV 0.05. As a result of ion implantation at temperatures of 500 and 550 °C, a nitrogen-modified layer with a microhardness of 190–220 HV 0.05 is formed in the surface layers of the VT1-00 titanium alloy, containing a solid solution of nitrogen in the α-Ti matrix phase. Nitrogen implantation of the VT1-00 alloy at a temperature of 620 °C leads to the formation of titanium nitrides $TiN_{0.26}$, ε-Ti_2N, η-Ti_3N_{2-x} in the surface layer of titanium alloy. The microhardness of VT1-00 titanium treated with nitrogen ions at 620 °C increases to 360 HV 0.05. In the case of ion-plasma nitriding (IPN) of VT1-0 titanium at 550 °C for 5 h, a nitrogen-modified layer up to 20 μm deep is recorded in its surface layer, containing isomorphic phases: α-Ti and titanium nitride $TiN_{0.26}$. The microhardness of the VT1-0 titanium alloy subjected to IPN is 190 HV 0.01.

Keywords: *commercial titanium, ion-beam nitriding, ion-plasma nitriding, modified layer, solid solution, titanium nitrides, microhardness*

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Introduction. Titanium is one of the most common elements in nature. It combines high strength and corrosion resistance, and also has a number of design advantages over materials such as steel or aluminum. Due to this, titanium alloys are increasingly being used in various industries, namely metallurgy,

aerospace industry, electrical and radio engineering, chemical industry, shipbuilding and medicine [1]. In particular, titanium meets the requirements of biocompatibility with the tissues of the human body and is characterized by low density and low elastic modulus. The combination of these properties determined the prospects of titanium and its alloys for use as a material for medical implants [2]. At the same time, titanium, which fully meets biological requirements, is inferior to traditional materials for medical implants in strength, wear resistance and resistance to cyclic loads. In this regard, the problem of increasing the strength and tribotechnical properties of titanium while maintaining its biological inertia and corrosion resistance is very relevant. Various methods of intensive plastic deformation are often used to improve the physical and mechanical characteristics of titanium [3]. Fundamentally new opportunities in this direction are opened by the use of combined methods of high-energy exposure. In particular, the treatment of the surface layers of titanium with concentrated streams of charged particles (for example, nitrogen atoms) can significantly increase the strength and tribotechnical properties of various alloys. Currently, technologies based on the use of concentrated streams of nitrogen ions are the most promising and intensively developing methods of nitriding. The varieties of these technologies include IPN [4–7] and ion-beam nitriding [8, 9]. In particular, the authors [4–7] have shown that ion-plasma nitriding can significantly improve the durometric and tribotechnical properties of materials, including titanium alloys. Usually, ion nitriding of titanium and its alloys is carried out at high temperatures [4–6, 8]. However, high-temperature ion nitriding leads to the formation of heterophase layers modified by nitride particles, which reduces the corrosion resistance and plasticity of titanium alloys. In this regard, it was of interest to investigate the influence of various methods of low-temperature ion nitriding on the structural and phase state of commercial titanium.

Research methodology. Samples of commercial titanium grades VT1-00 and VT1-0 (GOST 19807-91 [10]) were studied. The compositions of the alloys are given in the Table. The VT1-0 alloy was studied in the as-received state after annealing to the equilibrium state, as well as after the IPN. Annealing to the equilibrium state was performed at a temperature of 550 °C for 30 min. The choice of the IPN mode was carried out on the basis of the data of the work [4], as well as taking into account the economic feasibility of ion treatment. In particular, ion-plasma nitriding of VT1-0 was carried out at the IPN installation 0.361 (Figure 1) at a temperature of 550 °C for 5 h in an argon-hydrogen medium. The temperature of the parts was recorded using a chromel-alumel thermocouple. Heating and saturation control at the exposure stage was carried out according to a given program using a specialized controller.

Table — Chemical compositions of the studied titanium alloys, wt.%

Alloy	Fe	Si	C	Mn	Ti
VT1-00	0.17	0.03	≤0.05	0.02	Base
VT1-0	0.19	0.10	≤0.07	0.02	Base

Figure 1 — Vacuum chamber of the ion-plasma nitriding unit 0.361

The VT1-00 alloy in the as-received state was subjected to ion-beam nitrogen treatment at the UVN-2M installation equipped with a closed electron drift source. The beam contained 70 % molecular nitrogen ions and 30 % atomic nitrogen ions [8, 9]. Implantation was carried out with an ion energy of 3 keV and an ion current density of 2 mA/cm^2. For uniform distribution of the ion beam over the treated surface, a system of mechanical scanning of the sample attachment unit was used. The alloying fluence was $\sim 3\times10^{19}$ cm^{-2}. The temperature of the samples during ion-beam treatment was 350, 450, 500, 550 and 620 °C and was controlled using a "platinum-rhodium — platinum" thermocouple. The processing time was 2 h and was selected based on the data [8, 9].

For research, samples with dimensions of 8×6×4 mm were cut out of titanium plates subjected to ion nitriding. Metallographic studies of titanium samples were carried out on an optical microscope Altami MET 1MT. An aqueous 5 % HF solution was used to identify the structure of titanium alloys. The phase composition of the samples was studied using a DRON-3.0 diffractometer in monochromatized cobalt (CoK_α) radiation at a voltage of 28 kV and an anode current of 14 mA. The transcription of radiographs was carried out using the Crystallographica Search-Match software with a PDF-2 card file. Measurements of Vickers hardness and microhardness were carried out on a Dura-Scan 20 hardness tester with a load on an indenter P equal to 10 kg, 50 and 10 g.

Research results and their discussion. Ion-beam nitriding of VT1-00 alloy. In its initial state, VT1-00 titanium has a single-phase α-Ti structure with a hexagonal close-packed (HCP) crystal lattice with parameters a = 0.2952 nm, c = 0.4688 nm, c/a = 1.5881. The average grain size is 30 μm. Undeformed titanium practically does not contain linear defects, dislocations and dislocation clusters. This is due to the relatively

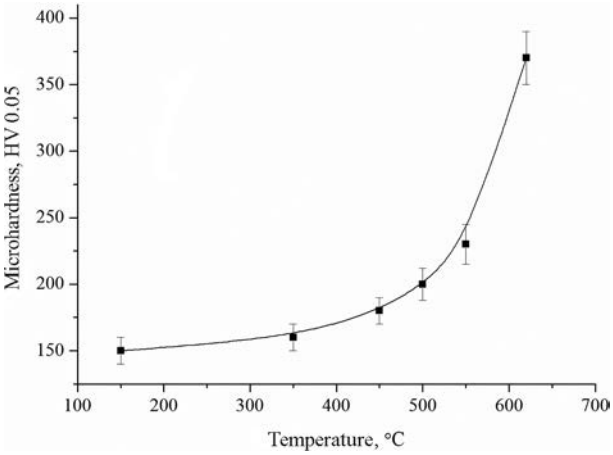


Figure 2 — Dependence of the microhardness of the nitrogen-modified surface layer of titanium VT1-00 on the ion implantation temperature

low values of the physical broadening of the diffraction lines of the matrix α-phase ($\beta_{004} \approx 1.3 \times 10^{-3}$ rad) and the alloy hardness (140–150 HV 10).

Ion implantation of VT1-00 titanium with nitrogen at temperatures of 350 and 450 °C leads to a slight increase in the microhardness of the surface layer to 160 and 180 HV 0.05, respectively (Figure 2). The phase composition of titanium treated with nitrogen ions at these temperatures practically does not change in comparison with the initial non-implanted state. The appearance of lines from new phases is not recorded on X-ray diffractograms, and therefore it can be assumed that a slight increase in the microhardness of undeformed titanium after low-temperature ion implantation is due to the formation of a solid nitrogen solution in thin surface layers in the α-Ti HCP. At the same time, due to the small thickness of the nitrogen-alloyed layer, which does not exceed 5 μm, the profile of the α-Ti diffraction lines does not undergo significant changes.

An increase in the temperature of ion-beam treatment of undeformed titanium VT1-00 to 500–550 °C leads to a grow in the depth of penetration of nitrogen ions into the surface layers up to ≈10 μm (Figures 3 *b*, 4). On X-ray diffractograms near the diffraction lines of the matrix α-phase from the side of small scattering angles, the appearance of an asymmetric diffuse intensity distribution of diffracted X-rays is recorded (see Figure 3), which is caused by the formation of a solid nitrogen solution in the α-Ti HCP. The microhardness of titanium modified with nitrogen ions at 500 and 550 °C increases to 190 and 220 HV 0.05, respectively (see Figure 2). After mechanical removal of the surface layer with a thickness of ≈10 μm, the profile asymmetry disappears, which indicates the absence of nitrogen atoms in α-Ti at a depth exceeding 10 μm. Figure 4 shows the distribution of microhardness over the depth of the nitrided layer at 550 °C, obtained by the angle-lap method.

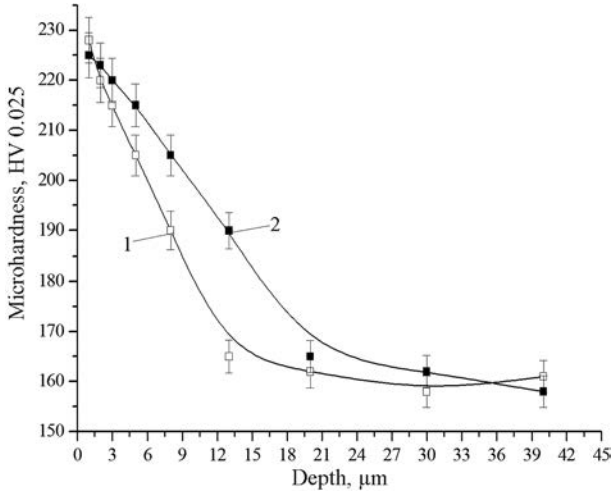
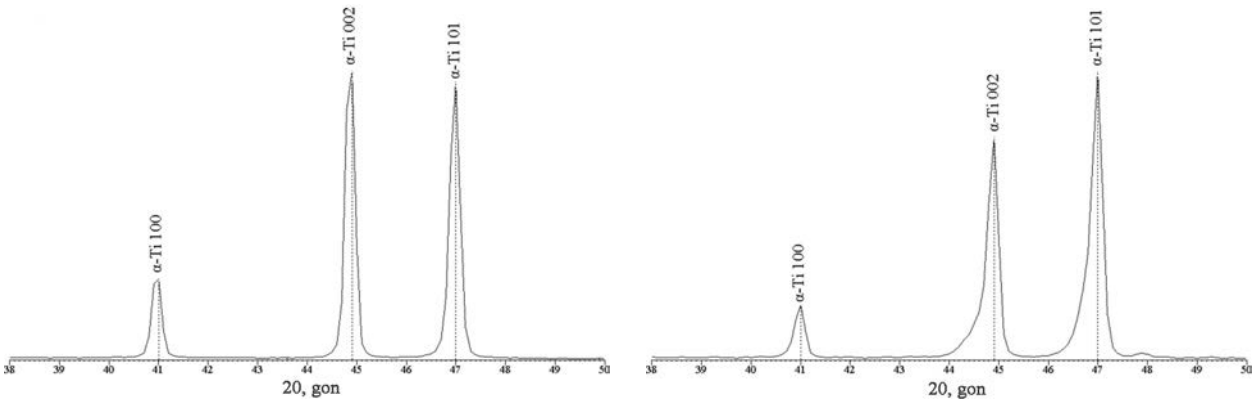


Figure 4 — Microhardness distribution by depth of nitrided layers in titanium: 1 — ion-beam nitriding at 550 °C (2 h); 2 — ion-plasma nitriding at 550 °C (5 h)

An increase in the temperature of ion implantation of titanium VT1-00 to 620 °C leads to a significant change in the phase composition of the nitrogen-modified layer. In particular, the X-ray diffractogram of the titanium VT1-00 implanted with nitrogen at 620 °C (Figure 5) shows the diffraction lines of α-Ti titanium nitrides $TiN_{0.26}$ (HCP crystal lattice; spatial group $P6_3/mmc$; $a = 0.2956$ nm; $c = 0.4765$ nm), ε-Ti_2N (tetragonal crystal lattice; spatial group $I4_1/amd$; $a = 0.4140$ nm, $c = 0.8805$ nm), η-Ti_3N_{2-x} (spatial group $R\bar{3}m$; $a = 0.2980$ nm; $c = 2.1660$ nm), as well as weak lines of titanium oxynitride $TiO_{0.34}N_{0.74}$ (spatial group A; $a = 0.9961$ nm; $b = 0.3796$ nm; $c = 0.9802$ nm). The for-



a *b*

Figure 3 — Fragments of X-ray diffractograms (CoK_α) of titanium VT1-00 in the initial state (*a*) and after ion-beam nitriding at 550 °C (*b*)

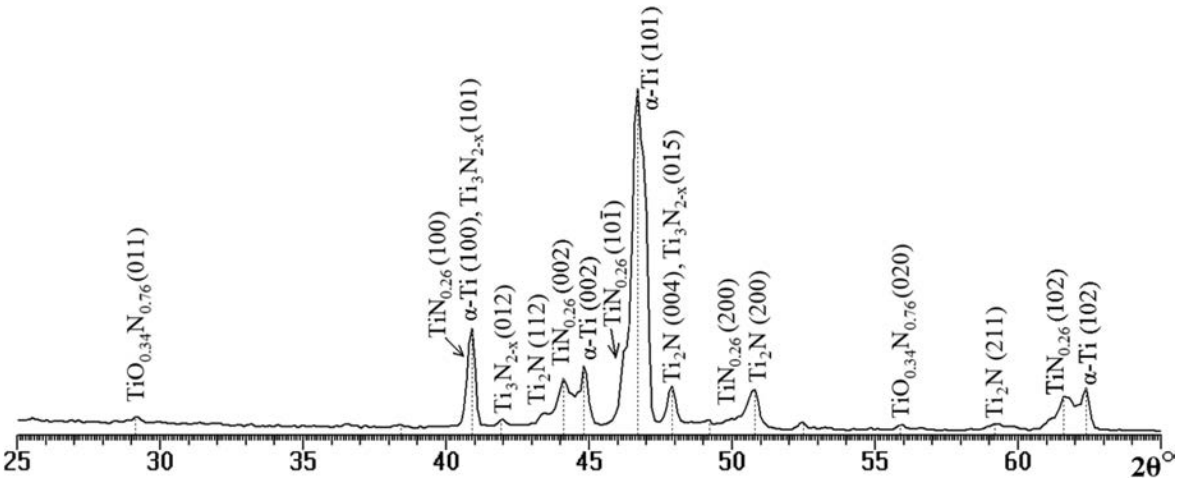


Figure 5 — Fragment of an X-ray diffractogram (CoK_α) of titanium VT1-00 subjected to ion-beam nitriding at 620 °C

mation of ε- and η-nitrides is consistent with the data obtained by other authors during high-temperature ion treatment of titanium [11–13], and the formation of a small amount of oxynitride is apparently caused by radiation-stimulated contamination of the surface layer of the titanium VT1-00 by residual gases in the chamber of the ion implantation unit. The microhardness of the surface layer of the titanium VT1-00 after high-temperature ion-beam nitriding increases to 360 HV 0.05.

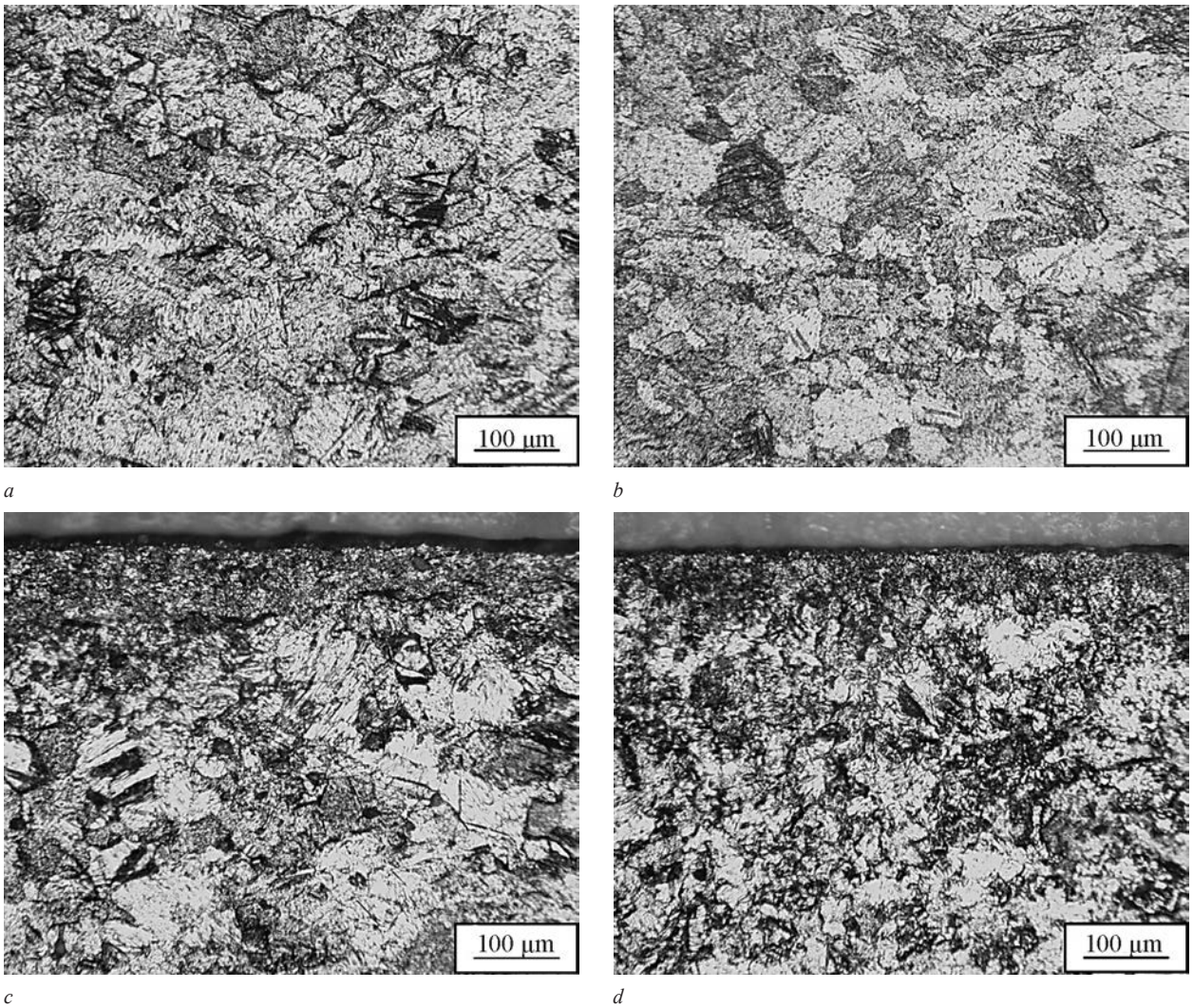
Thus, ion treatment of titanium VT1-00 at 350–550 °C leads to the formation of a solid nitrogen solution in the matrix phase α-Ti in a thin surface layer, which is accompanied by a grow in the surface microhardness from 160 to 220 HV 0.05. An increase in the temperature of ion-beam treatment with nitrogen to 620 °C leads to the formation of a modified layer containing various nitride phases and having increased microhardness.

IPN of VT1-0. Microstructures of commercial titanium VT1-0 samples in the initial state (as-received state) and after various types of processing are shown in Figure 6.

In the initial state, titanium VT1-0 has a structure with an average grain size of 25–30 μm (see Figure 6 *a*). The titanium hardness is 150 HV 10. The alloy contains α-Ti with a HCP crystal lattice with parameters a = 0.2952 nm, c = 0.4688 nm, c/a = 1.5881 nm (Figure 7). In addition, an insignificant amount of Ti_3O_5 titanium oxide is registered in the alloy (see Figure 7 *a*, *b*). Preliminary annealing of the VT1-0 alloy at 550 °C (0.5 h) leads to the some expected enlargement of the grain structure compared to the initial state (see Figure 6 *b*). The average grain size of annealed titanium is 40–50 μm (see Figure 6 *b*). The hardness of titanium samples after annealing is 130–140 HV 10.

As a result, a thin layer with a crushed grain structure up to 40–50 μm deep is recorded on the surface of the sample at 550 °C for 5 h in the VT1-0 alloy (see Figure 6 *c*). This layer is formed due to the recrystallization processes in the thin surface layer, as well as due to the decoration of grain and subgrain boundaries with nitrogen atoms during the IPN process. A change in the ratio of the intensities of the diffraction lines 002 and 101 of the matrix titanium phase also testifies in favor of the recrystallization processes during ion-plasma processing (see Figure 7 *a*, *c*). The processes of recrystallization of the thin surface layer in the alloy during IPN can occur due to deformation (strain hardening) of the thin surface layer during pre-machining (grinding, etc.) of titanium, also as a result of subsequent heating of the surface during ion-plasma treatment. In the recrystallized layer after ion-plasma treatment, α-Ti and titanium nitride with a low nitrogen content $TiN_{0.26}$ are recorded (see Figure 7 *c*). In fact, the $TiN_{0.26}$ nitride phase (like the $TiN_{0.3}$ phase) has a crystal lattice isomorphic to α-titanium HCP crystal lattice (space group $P6_3/mmc$) and, as a result, the $TiN_{0.26}$ nitride can be considered as a saturated solid solution of nitrogen in α-Ti. At the same time, the parameter **a** of the crystal lattice of the $TiN_{0.26}$ phase practically coincides with the value of the parameter **a** for the α-Ti lattice, and the parameter **c** of the $TiN_{0.26}$ phase significantly exceeds the value of the parameter **c** for titanium. This fact indicates the predominant arrangement of nitrogen atoms in the octahedral pores of the HCP lattice of the α-phase. The depth of the nitrogen-alloyed layer is 12–16 μm (see Figure 4). The microhardness of the sample after the IPN is 195–200 HV 0.01. Figure 8 shows an X-ray diffractogram from the surface layer of titanium VT1-0, subjected to IPN and mechanical grinding to a depth of 12 μm. It can be seen that the diffraction lines from α-Ti have a characteristic asymmetric shape, which is due to the presence of nitrogen dissolved in the matrix phase in the surface layer. After grinding of the titanium VT1-0 to a depth of 20–25 μm, the profile asymmetry disappears, which indicates the absence of nitrogen atoms in the α-Ti. As a result of the IPN of the annealed VT1-0 alloy, as in the case of an unburned alloy, a recrystallized layer is also recorded on its surface, the depth of which is ≈40–50 μm (see Figure 6 *d*).

The phase composition of the modified layer includes the α-Ti matrix phase and low-nitrogen nitride $TiN_{0.26}$ (see Figure 6 *d*). The microhardness of the nitrided layer is 190–195 HV 0.01.



a *b* *c* *d*

Figure 6 — Characteristic microstructures of titanium VT1-0 after various types of processing: *a* — as-received state; *b* — after annealing at 550 °C; *c* — alloy in as-received state + IPN (550 °C, 5 h); *d* — alloy after annealing + IPN (550 °C, 5 h)

Comparing the results of phase analysis of commercial titanium subjected to low-temperature ion nitriding by various methods, it can be noted that as a result of ion-beam nitriding at temperatures of 350–550 °C, thin modified layers containing a solid solution of nitrogen in α-Ti are formed in the thin surface layers. As a result of prolonged ion-plasma treatment for 5 h at 550 °C, a solid nitrogen solution in α-Ti is transformed into a low-nitrogen phase $TiN_{0.26}$ with the HCP lattice to the isomorphic lattice of α-Ti. When the ion nitriding temperature rises to 620 °C, the formation of ε-Ti_2N and η-Ti_3N_{2-x} nitrides is recorded in the nitrogen-modified layer, which is accompanied by a significant increase in the surface microhardness to 360 HV 0.05.

Thus, ion-beam and ion-plasma nitriding of commercial titanium VT1-0 and VT1-00 at a processing temperature of 550 °C (for 2–5 h) leads to the formation of thin nitrogen-modified layers, mainly containing the isomorphic solid nitrogen solution in α-titanium and a low-nitrogen nitride phase with the HCP lattice. The microhardness of the nitrided layer is ≈200 HV 0.05. It should be noted that the formation of the solid nitrogen solution in the matrix phase is accompanied by the appearance of powerful compressive stresses in the surface layer, which leads to an increase in the cyclic durability of materials [8, 14]. In this regard, low-temperature nitriding of titanium can be a promising way to increase its structural reliability, which is very important for critical items of the aerospace industry and medicine. An increase in the temperature of ion nitriding of titanium to 620 °C is accompanied by the formation of a strong heterogeneous structure in its surface layer containing highly nitride phases. The specified structural-phase state may be of interest for the formation of wear-resistant layers.

Conclusion. The structural-phase state of commercial titanium VT1-00 and VT1-0 in the initial state and subjected to different types of ion nitriding at different temperatures has been studied. It is shown that low-temperature ion-beam treatment of titanium at 350–550 °C for 2 h leads to the formation of a nitrogen-modified surface layer up to 10 μm thick containing a solid nitrogen solution in the HCP matrix α-phase lattice. The microhardness of the nitrogen-modified surface layer of the alloy increases to 200–220 HV 0.05 compared to the initial

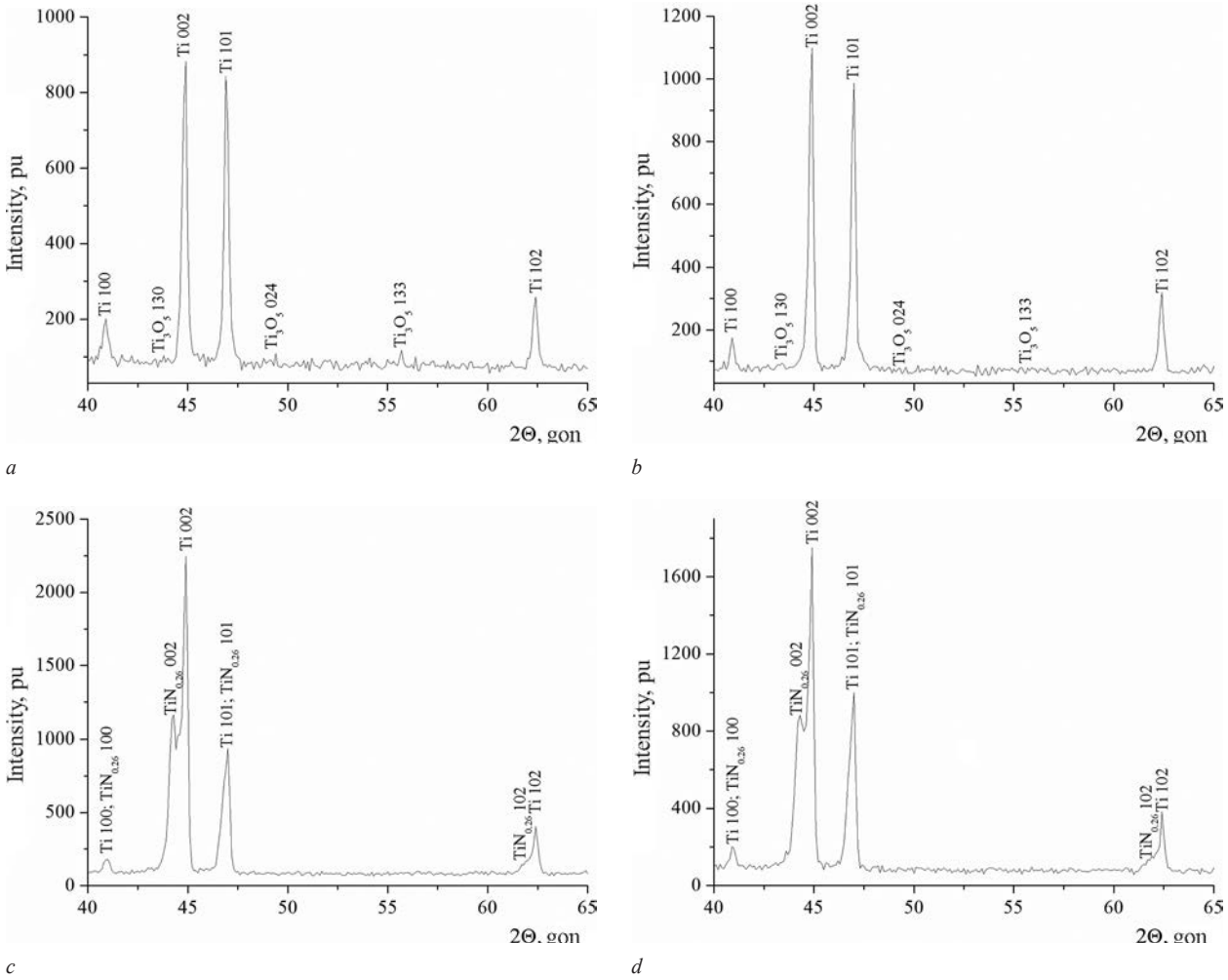


Figure 7 — Fragments of X-ray diffractograms (CoK_α) from the surface layers of titanium alloy VT1-0 samples after various types of processing: *a* — as-received state; *b* — after annealing to the equilibrium state; *c* — alloy in as-received state + IPN (550 °C for 5 h); *d* — alloy after annealing to the equilibrium state + IPN (550 °C for 5 h)

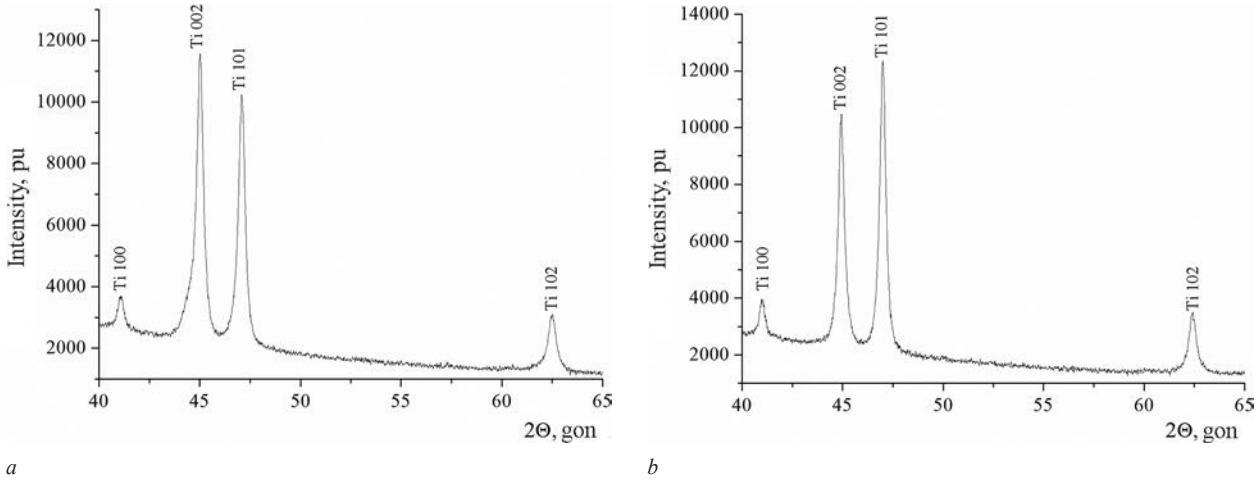


Figure 8 — Fragments of X-ray diffractograms (CoK_α) from the surface layers of titanium alloy VT1-0 samples in as-received state after IPN (550 °C for 5 h) and subjected to mechanical grinding to a depth of 10–12 μm (*a*) and 20–25 μm (*b*)

state. High-temperature ion implantation of titanium with nitrogen at 620 °C leads to the formation of nitride phases in the modified layer and an increase in the titanium microhardness by 2.5 times compared to the initial state.

The IPN of titanium VT1-0 at 550 °C for 5 h is accompanied by the formation of modified layers up to 20 μm deep containing the low-nitrogen phase $TiN_{0.26}$ isomorphic to α-titanium. The layer microhardness is ≈200 HV 0.05.

It is concluded that low-temperature ion nitriding can be used as a method to increase the structural reliability of commercial titanium, which is widely used in the aerospace and chemical industries, as well as in medical applications.

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СТРУКТУРА ТЕХНИЧЕСКОГО ТИТАНА, ПОДВЕРГНУТОГО НИЗКОТЕМПЕРАТУРНОМУ ИОННОМУ АЗОТИРОВАНИЮ

Исследовано структурно-фазовое состояние технического титана марок ВТ1-00 и ВТ1-0 в исходном состоянии и после различных видов низкотемпературного ионного азотирования. В исходном

состоянии сплавы ВТ1-00 и ВТ1-0 имеют однофазную структуру α-Ti с гексагональной плотноупакованной кристаллической решеткой. Твердость титана составляет 140–150 HV 10. Показано, что ионно-лучевое азотирование сплава ВТ1-00 при пониженных температурах 350 и 450 °С приводит к формированию тонких (до 5 мкм) упрочненных азотом слоев, имеющих твердость 160–180 HV 0,05. В результате ионной имплантации при температурах 500 и 550 °С в поверхностных слоях титанового сплава ВТ1-00 формируется модифицированный азотом слой с микротвердостью 190–220 HV 0,05, содержащий твердый раствор азота в матричной фазе α-Ti. Имплантация азотом сплава ВТ1-00 при температуре 620 °С приводит к образованию в поверхностном слое нитридов титана $TiN_{0,26}$, ε-Ti_2N, η-Ti_3N_{2-x}. Микротвердость обработанного ионами азота при 620 °С титана ВТ1-00 возрастает до 360 HV 0,05. В случает ионно-плазменного азотирования (ИПА) титана ВТ1-0 при 550 °С в течение 5 ч в его поверхностном слое регистрируется модифицированный азотом слой глубиной до 20 мкм, содержащий изоморфные фазы: α-Ti и нитрид титана $TiN_{0,26}$. Микротвердость подвергнутого ИПА титанового сплава ВТ1-0 составляет 190 HV 0,01.

Ключевые слова: *технический титан, ионно-лучевое азотирование, ионно-плазменное азотирование, модифицированный слой, твердый раствор, нитриды титана, микротвердость*

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