Influence of surface structures on torque of VT6 alloy cortical screws

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Abstract. The paper investigated the effect of the surface structure of VT6 alloy cortical screws after various modes of vacuum ion-plasma treatment on the magnitude of the torque when they are screwed and twisted into the threaded holes of titanium plates for bone osteosynthesis. It was shown that nitriding of the screw heads increases the surface hardness by 2 times, and the formation of the nitride layer - by 3 times. It was found that the maximum decrease in torque when unscrewing is observed for nitride coated screws.

1. Introduction
In modern medicine for the surgical treatment of bone fractures are widely used products for immersion osteosynthesis. The type of osteosynthesis (bone or intraosseous), as well as the shape of the product for treatment are determined depending on the nature and complexity of the fracture - these can be plates, screws, pins, nails, etc. [1-3]. The shape and structure of the bone of that section of the skeleton where it is planned to use these fixators also influence the choice of implants. In particular, devices made of titanium alloys, the designs of which include plates and fixing screws, are used for bone osteosynthesis for surgical reposition of bone fragments directly in the area of bone fracture [4]. Due to their design and biomechanical characteristics, such devices provide stable fixation of fragments in the correct position while maintaining the functional axis of the segment and perioseal blood supply, stabilization of the fracture zone to full fusion [5].

The successful use of structures made of titanium plates largely depends on the effectiveness and stability of their fixation to the bone, which in turn is determined by the material and properties of the screws. To avoid the development of contact corrosion, the same material should be used for the manufacture of screws. However, as surgical practice [3] shows, there is a need to increase the strength and wear resistance of titanium screw heads. In particular, when the screw is locked in the osteosynthesis plate, which are made of a homogeneous material with low hardness, the effect of the so-called “cold welding” occurs at the contact point of the threaded part of the screw head with the plate [4]. This, in turn, leads to complications in the removal of the structure after the termination of the fracture treatment until the thread is broken or the screw head is destroyed. Therefore, the development of a technology for modifying the surface of medical screws to give them wear-resistant properties is undoubtedly an urgent task.

Modern technological developments on the surface modification of titanium alloys in combination with the use of wear-resistant coatings open up additional opportunities for new biocompatible stabilizing structures [6–9]. Thus, vacuum ion-plasma treatment (VIPO), which contributes to the
increase of wear resistance of products, has been widely used in the manufacturing process, for example, of titanium alloy heads working in friction units paired with ultra-high molecular weight polyethylene [10-13].

Using a vacuum ion-plasma treatment, it is possible to create a modified surface layer through the diffusion mechanism of structural transformations upon saturation with nitrogen ions, the so-called nitriding; or the deposition of a nitride TiN coating carried out through an atomic-cluster mechanism [14, 15]. A combination of these two independent technological processes in a single cycle is also possible: first, nitriding, and then nitride coating. All of these processes make it possible to change the properties of both the starting material due to the formation of a modified surface layer and the created metal-modified layer-coating composite after applying TiN.

The aim of this work was to study the effect of the surface structure of the heads of cortical screws made of VT6 titanium alloy on the torque when screwing and unscrewing them into the threaded holes of titanium plates for osteosynthesis.

2. Material and methods

The objects of study were HA 4 cortical screws according to State Standard R 50582 for plates with angular stability, used in minimally invasive bone osteosynthesis for bone fractures (Figure 1). The cortical screw has a small spherical thread (∅ 4 mm) along the entire length of the body and a tapered thread on the head (∅ 6 mm) with a turnkey internal hexagon (GOST 10753). The screws were made at Federal State Unitary Enterprise “CITO” (Moscow) according to TU 9438-174-01894927-2008 from VT6 grade titanium alloy (GOST R ISO 5832-3) using standard thread cutting technology.

The surface of the screw heads was subjected to vacuum ion-plasma treatment (nitriding and coating of titanium nitride) in an upgraded Bulat-6T installation in a nitrogen-argon atmosphere for 40 min. at a temperature of 600 ° C. The screws were placed in special technological equipment so that only the screw heads were subjected to processing. After nitriding, a layer of titanium nitride TiN was applied to a part of the samples by condensation at a temperature of 600 ° C for 30 min.

To measure microhardness, roughness, and determine the depth of the nitrogen-hardened surface layer, we used “witness samples” made of VT6 alloy cut from a hot-rolled bar of ∅ 20 mm. The surface of these samples was subjected to vacuum ion-plasma treatment in the same modes as the screw heads.

The microstructure of the samples was studied on an AXIO Observer.A1m optical microscope at magnifications up to 1000 times using the NEXSYS ImageExpert Pro3 software package for processing and analysis of the obtained images. The appearance of the screw heads after the tests was investigated using a MBS-10 stereoscopic microscope with an increase of up to 100 times.

The surface roughness (Ra) was measured on a Hommel Tester T500 instrument in accordance with GOST 2789.

To determine the tightening torque for screwing in and stragging when the screws are unscrewed was determined according to the procedure based on GOST R 50581 (RF) [16] and ASTM F543-13 (USA) for medical bone screws [17] The screws were inserted through the holes of a limited compression contact plate from VT6 titanium alloy, and into a wooden block clamped in a vice, into which holes were pre-drilled, then with the help of a hexagonal screwdriver, the screws were screwed completely into the plate (Figure 1).
Linden was chosen as the material of wooden bars serving as an imitation of the bone structure of the cortical bone, since it has the most similar mechanical characteristics - $\sigma_0 \sim 110-140$ MPa, $E \sim 12-16$ GPa [18]. To apply torque, a torque wrench with digital indication KD10-6.3 was used. The measurement range is 2-10 N ∙ m, the resolution is 0.01 N ∙ m, the relative error in measuring the torque of the force is not more than ± 2%. During torsion, the axis of the screw and the device for applying torque coincided.

Torque was applied so that the screw head rotated with a uniform angular velocity in the range of 1-5 rad / min with a gradual increase in axial load during the first four turns of the screw. In this case, the axial load did not exceed 11 N. The test screws were tightened with a moment of about 6 N ∙ m ($M_1$). When the screw was unscrewed, the torque was fixed when it was strained ($M_2$), then the relative change in the torques was determined. Part of the bars with plates and screws was immersed in a 0.9% aqueous solution of NaCl simulating the human body under natural aeration at room temperature for 30 days. After holding in the solution, the torque was also measured when the screws were unscrewed.

3. Results and discussion

At the first stage of the work, the effect of vacuum ion-plasma treatment on the structure and surface properties of VT6 alloy products was studied.

Initially, the structure of cortical screws made of VT6 alloy was studied, in the initial untreated state and after vacuum ion-plasma treatment (Figure 2). For this, the screws were cut along the axis.

The structure of the screws in the initial state is represented by $\alpha$ and $\beta$ phases with a predominant fraction of the $\alpha$-phase of a polyhedral shape (Figure 2 a).
Since in the process of nitriding, the products are heated to 600 °C, which corresponds to incomplete annealing temperatures for \((\alpha + \beta)\) -titanium alloys [19], polygonization processes take place in the particles of the primary \(\alpha\)-phase and become more globular (Figure 2 b). The particle size is \(5 \div 7\) microns. The resulting structure complies with the requirements of GOST R ISO 5832-3 for the wrought alloy Ti-6Al-4V used in medicine.

At the next stage of operation, cortical screws were subjected to vacuum ion-plasma treatment, the modes of which were selected on the basis of previous studies. Together with the screws, the test pieces were also pre-polished to a roughness value \(Ra = 0.06\ \mu m\). The polished state makes it possible to more accurately determine the change in its roughness during processing, as well as evaluate the structural changes in the surface [15].

The studies showed that the microhardness of the VT6 alloy sample in the initial polished state is 3600 MPa (Figure 3 a). After vacuum ion-plasma nitriding, the microhardness on the surface increases approximately 2 times and amounts to 6300 MPa. The increase in surface hardness is due to the formation of a solid solution of the introduction of nitrogen into \(\alpha\) titanium [11, 14]. An analysis of microstructures shows that in vacuum ion-plasma nitriding without nitride deposition, the most dense and “clean” surface is achieved (Figure 4 b).

![Figure 3](image1.png)

**Figure 3.** Microhardness (a) and roughness (b) of the surface of witness samples of VT6 alloy depending on the regimes of vacuum ion-plasma treatment.

![Figure 4](image2.png)

**Figure 4.** The microstructure of the surface of the samples of a bar of VT6 alloy after various modes of vacuum ion-plasma treatment: a) the initial polished state; b) after nitriding; c) after TiN coating.
The deposition of titanium nitride on the surface of a pre-nitrided sample and immediately after polishing leads to an increase in the surface microhardness by a factor of 3 compared with the initial state, to 11600 MPa (Figure 3 a). The condensation deposition of titanium nitride (TiN) after nitriding leads to the formation of defects on the surface in the form of craters and a droplet phase (Figure 4 c), which is formed in the flow of titanium-nitrogen plasma [11].

Depending on the mode of vacuum ion-plasma treatment, the surface microgeometry changes (Figure 3 b). If nitriding practically does not affect the surface roughness (Ra = 0.07 μm) compared with the initial state (Ra = 0.06 μm), then after applying titanium nitride, the roughness index increases by more than 3 times to 0.20 μm (Figure 3 b) due to the presence of the droplet phase (see Figure 4).

To assess the depth of the nitrided layer, the method of measuring microhardness on oblique thin sections was used. The measurements were carried out on samples subjected to nitriding and complex vacuum ion-plasma treatment. Based on the measurement results, we plotted the distribution of the microhardness of the samples along the depth of the modified layer (Figure 5).

The studies showed that after nitriding, the depth of the modified layer is 17 μm (Figure 5), and for the sample on the surface of which nitride was deposited, the presence of a nitride zone with a depth of about 3 μm is typical.

![Figure 5. Change in the microhardness of samples of VT6 alloy after vacuum ion-plasma surface treatment.](image)

At the next stage, tests were performed on HA 4 cortical screws made of VT6 titanium alloy, processed in various modes, for torque when screwing and unscrewing them into the holes of the plates and into a wooden block imitating the cortical structure of the bone. The appearance of the screws is shown in Figure 6.

![Figure 6. Cortical screws from VT6 alloy for bone osteosynthesis after vacuum ion-plasma treatment of the surface of the heads: a) nitriding; b) TiN coating.](image)
Since Russia does not have any regulatory documentation for this kind of testing, the Russian standard GOST R 50581 [16] and the standard of the American international organization for testing and materials ASTM F543-13 [17], which establish torsion test methods, were taken as the basis to determine the tearing torque and angle of rotation until the destruction of the metal bone screw. However, these regulatory documents do not provide any regulatory indicators for torques when the screw contacts the osteosynthesis plate. According to GOST R 50581, the minimum breaking torque of cortical and spongy screws varies from 0.2 to 6.2 N ∙ m depending on the type of screw [16]. Based on these values, a torque of about 6 N ∙ m was applied to tighten the test screws in the plate openings.

Initially, we studied the change in torque when screwing and unscrewing the screw body into a wooden block without using a plate. Since the screw is not self-tapping, holes for the diameter of the thread of the screws were previously made in the bar. When testing used screws with the original state of the surface.

The test results showed that the torque when tightening and stragging the screw when screwing it in and out directly into a wooden block is 0.09 N ∙ m, which is an order of magnitude lower than the minimum breaking torque values specified in the standards for cortical screws [16, 17]. Therefore, when testing with a plate, the contribution to the torque of screwing and unscrewing the screw body into a wooden block was not taken into account.

The tests were carried out on three screws, subjected to each type of processing, as well as in the original untreated condition. One part of the screws was tested under normal conditions (in air), when the screws were removed immediately after screwing into the plate and bar, the other part of the screws after screwing was immersed in a 0.9% aqueous NaCl solution for 30 days. Based on the obtained measurement results, the relative change in torque during unscrewing was determined, and the obtained values were averaged (Figure 7).

The results of tests conducted in air showed that the screws with the untreated surface of the heads were strained when unscrewing from the plate with greater force than when tightened - the torque increased on average by ~ 4% (Figure 7).

![Figure 7. Torque values when screwing in (■) and unscrewing (○) screws in the holes of the plate and wooden block depending on the surface treatment of the heads after testing in air (a) and after holding for 30 days in 0.9% aqueous solution of NaCl (b), as well as the relative difference in torques.](image-url)
Surface treatment of the heads leads to a decrease in torque when unscrewing by 12% and 8% for nitrided screws and with nitride coating, respectively (Figure 7 a). The difference between the obtained values is most likely associated with an increase in the roughness of the nitrided surface due to the formation of a droplet phase (Figure 4 c).

After keeping in a 0.9% aqueous NaCl solution for 30 days, the greatest difference in the torques during screwing in and out was shown by the screws, on the heads of which titanium nitride was applied - 27% and 26% for TiN-coated screws and pre-nitrided ones, respectively (Figure 7 b). Vacuum ion-plasma nitriding of screw heads also leads to a decrease in torque by 18.5%. When testing screws with an untreated surface of the heads, the change in torque was the smallest - 10.2% (Figure 7 b).

Inspection of the screws after all tests showed signs of wear between the threads (Figure 8). Also, the threads on the screw heads underwent a partial deformation, with more deformed turns observed in untreated screws (Figure 8 b).

**Figure 8.** Appearance of screw heads with various surface conditions, tested for screwing and twisting into a titanium plate after holding for 30 days in a 0.9% aqueous NaCl solution (x8):
in the initial state before tests (a) and after (b);
with TiN coating before tests (c) and after (d);
after nitriding before tests (e) and after (f)
The greater change in the torques after holding in solution compared with the test under ordinary conditions is apparently due to the difference in the friction forces in air and liquid environments. Probably, the solution acted as a lubricating fluid when it entered the microscopic slots during deformation of the screw and plate threads.

The results indicate a positive effect of surface treatment of screw heads when they are unscrewed from the osteosynthesis plate - the torque values (and therefore the applied force) decrease, and the screws with titanium nitride coating show the most noticeable decrease (~ 30%). This result, apparently, is explained by an increase in the surface hardness during the deposition of nitride, which contributes to an increase in the resistance of the surface of the screw heads to plastic deformation when they are screwed into the threaded hole of the plate.

4. Conclusion

Studies have shown that vacuum ion-plasma treatment does not lead to significant changes in the internal structure of medical screws made of VT6 alloy. In this case, after nitriding, the hardness of the surface of the screws increases by 2 times compared with the initial state (from 3600 MPa to 6900 MPa) due to the formation of a solid solution of nitrogen incorporation into the titanium crystal lattice in the surface layer, and the presence of titanium nitride TiN - by 3 times to 11,600 MPa. This leads to a decrease in torque when moving the screw both in air and after maintaining the structure in a 0.9% NaCl aqueous solution. The most noticeable reduction in torque is shown by titanium nitride coated screws.

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