The influence of laser micro- and nanostructuring on the wear resistance of Grade-2 titanium surface

IOP Publishing | Astro Ltd Laser Physics
Laser Phys. **28** (2018) 086002 (7pp) https://doi.org/10.1088/1555-6611/aac05a

The influence of laser micro- and nanostructuring on the wear resistance of Grade-2 titanium surface

V P Veiko[1], G V Odintsova[1], M Y Gazizova[2], Y Y Karlagina[1], S S Manokhin[2,3], R M Yatsuk[1], S D Vasilkov[1] and Y R Kolobov[2,3]

[1] ITMO University, Saint-Petersburg, 197101, Russia
[2] Belgorod National Research University, Belgorod, 308015, Russia
[3] Institute of Problems of Chemical Physics of RAS, Chernogolovka, 142432, Russia

E-mail: jujukarlagina@corp.ifmo.ru

Received 9 November 2017, revised 23 April 2018
Accepted for publication 23 April 2018
Published 7 June 2018

Abstract

The development of wear-resistant surfaces supports sustainable and long-term work of friction couples. In this paper, we demonstrated laser structuring as a method for increasing Grade-2 titanium surface wear resistance. Surface treatment by a nanosecond fiber laser produces nano- and microstructures simultaneously, with composition changing to combinations of Ti, O and N. Tribological tests were conducted according to certificated 'Ball-disk' method in conditions of rubbing friction and Al_2O_3-ball as a counterbody. Investigation results demonstrate an increase in wear resistance of laser-treated titanium surface by ten times. It appears both surface structure and chemical composition influence titanium durability enhancement.

Keywords: laser structuring, titanium, wear resistance, tribology, microrelief

(Some figures may appear in colour only in the online journal)

1. Introduction

Topology (surface quality) and surface composition have a substantial impact on the functional characteristics of products because the primary factor in the consistent and long-term performance of tribological pairs is the qualities of meeting surfaces. It is known that smooth surfaces allow decreasing the coefficient of friction, running-in time and wear rate [1, 2]. In the case of friction without lubrication, the probability of appearance of the adhesive mechanism of wearing increases, which has a negative effect on the efficiency of the entire tribosystem. It has been shown in various studies that the desire to create 'perfectly' smooth surfaces is not always justified and necessary, apart from being costly. In the last several years, purposeful texturing of the surface with creating multi-leveled roughness and undulation has become more popular. Formation of given texture with an exact period and level of roughness allows for a possibility of surface characteristics control, including frictional properties. In [1] it was shown that creating relief on the surface of steel using pulsed laser allows decreasing the coefficient of friction from 0.045 to 0.004 in the regime of lubricated friction. Recently similar results were presented in several other works [2–6]. During a tribological contact of such surfaces, friction appears in the conditions of hydrodynamic lubrication, and the texture of the surfaces leads to a complete division of meeting faces and an almost complete absence of wear [7].

The positive impact of surface texturing onto the tribological properties has been proven for various materials, but optimal methods of creating textures with multimodal roughness and the degree of influence of multilevel surface roughness onto the friction properties have not been determined. As a possible method for forming textures with multilevel roughness, femtosecond laser treatment is proposed, which has shown itself as an effective way of surface modification that allows creating structures with given period and level of roughness [8–11].

Commercial pure titanium Grade-2 is widely used in industry [12]. There are many papers dedicated to an improvement of tribological properties of the products made from titanium and its alloys [13–17]. That is a topical issue because titanium as a base material found applications in many different fields: from orthopedics to aerospace industry [12]. It is of great importance for titanium details and products to have excellent wear resistance and the specified value of surface roughness; for example, dental titanium implants have a substantial effective surface to prevent evulsion in use [18].

Wu *et al* [3] after conducting tribological studies of titanium alloys Ti64 and Ti6242 at the counterbody speed of 0.3 and 1.0 m s^{-1} note that the coefficient of friction and its fluctuation are smaller at higher counterbody speed. At high rates, the interaction area has a higher temperature, which leads to the decrease of friction forces. The sample-counterbody pair has five times higher wear factor at 0.3 m s^{-1}, and sample material has higher wear resistance (about 10^{-4} mm^3 $N^{-1}m^{-1}$). Analysis of SEM-images of counterbody trace on the sample showed that abrasive wearing prevails at 0.3 m s^{-1}. Such wear trace is broad and flat, which corresponds to higher wear factor. The wear traces at 1.0 m s^{-1} were smaller but rougher and with large pieces of counterbody material; the wear is adhesive because of a high temperature in the contact area. The results of energy dispersive x-ray spectroscopy (EDX-analysis) showed significant amounts of Ti and Al on the wear traces at given speed.

Despite various structuring methods and the variety of possible structures, there is no 'perfect' method today that would allow improving the wear resistance of construction materials surface, process complex shapes and less-accessible areas as well as leave less dirt after processing, which is essential for medical implants manufacturing.

In this article, we showed an effect of laser-induced double-level (both nano- and microscale) surface texture on tribological properties of the material under examination.

2. Methods

Unalloyed Grade-2 titanium was used as sample material. A flat surface of obtained titanium disks was polished with sandpaper based on silicon carbide until it reached roughness of $R_a = (0.75 \pm 0.02)$ μm. A picture of the sample before laser processing is shown in figure 1.

Figure 1. An image of Grade-2 titanium before laser processing.

After polishing, structuring of the obtained titanium surface was carried out with ytterbium fiber laser with $\tau = 100$ ns pulse duration and $\lambda = 1.06$ μm wavelength. The pulse repetition rate was $f = 60$ kHz. The samples surface was irradiated by subsequent scanning with a focused laser beam with a diameter of $d_0 = 50$ μm. After finishing one scanning line with a pulse repetition rate of f and scanning step M_x, the beam moved along the Y-axis to the next line with a scanning step M_y. The choice of the setup is based on the sufficient absorption of radiation at $\lambda = 1.06$ μm by metals.

Tribological tests were carried out on the automated tribometer (CSM Instruments) with a test configuration 'ball-disk' for dry friction in the air. 'Ball-disk' method is a certificated for laboratory tribological tests. As a counterbody, corundum ball with a diameter of 6 mm was used. The study of wear resistance of the samples before and after surface structural modification was carried out in two regimes, A: counterbody pressure $p = 2$ N, spin speed of counterbody $v = 10$ cm s^{-1}, slip distance $l = 360$ m, radius of counterbody rotation $r = 4.7$ mm; and B: $p = 10$ N, $v = 15$ cm s^{-1}, $l = 290$ m, $r = 3.2$ mm. The tests comply with international standards ASTM G99-959, DIN50324. In the process of testing, the coefficient of friction and friction force of the friction couples were measured. After the test, the wear of sample and counterbody was estimated using the wear factor (1):

$$W = \frac{V}{p \cdot l}, \tag{1}$$

$$V = \pi h^2 \left(r_c - \frac{h}{3}\right), \tag{2}$$

$$h = r - \sqrt{r_c^2 - \left(\frac{d_w}{2}\right)^2}. \tag{3}$$

V—the volume of removed material, mm^3, d_w—wear track diameter, mm; r_c—counterbody radius, mm;
h—height of the removed material segment, mm.

The studies of surface morphology and modified layers structure were carried out via scanning electron microscope FEI Quanta 600 with additional EDX-analysis. Compositional analysis was performed by x-ray diffraction (XRD) measurements with an x-ray diffractometer 'ARL X'TRA' employing Cu-Kα radiation at Bragg–Brentano focusing mode (lateral resolution 5 mm, information depth 2 μm).

The roughness of the surface was estimated as an arithmetical mean deviation of the assessed profile R_a, measured with profilometer Hommel Tester T8000. The roughness was calculated as an average of five measurements, the error was calculated as a standard deviation. Stated equipment was also used for measuring cross-section area of wear trace after tribological tests for calculating wear factor of the sample. The control of counterbody wear was carried out via the means of optical microscopy by measuring the diameter of the ball wear scar.

3. Results and discussion

3.1. Morphology and composition of Grade-2 titanium structured surface

Laser irradiation initiates a series of thermal and thermo-chemical processes in metal, such as oxidation, melting and

Table 1. The parameters of processing regimes.

Sample (#)	Intensity, I (10^7 W cm^{-2})	Scanning step, M_x (μm)/total number of pulses per point	Scanning step, M_y (μm)/total number of pulses per point
1	6.95	3.33/15	5.0/10
2	2.70	0.83/60	5.0/10

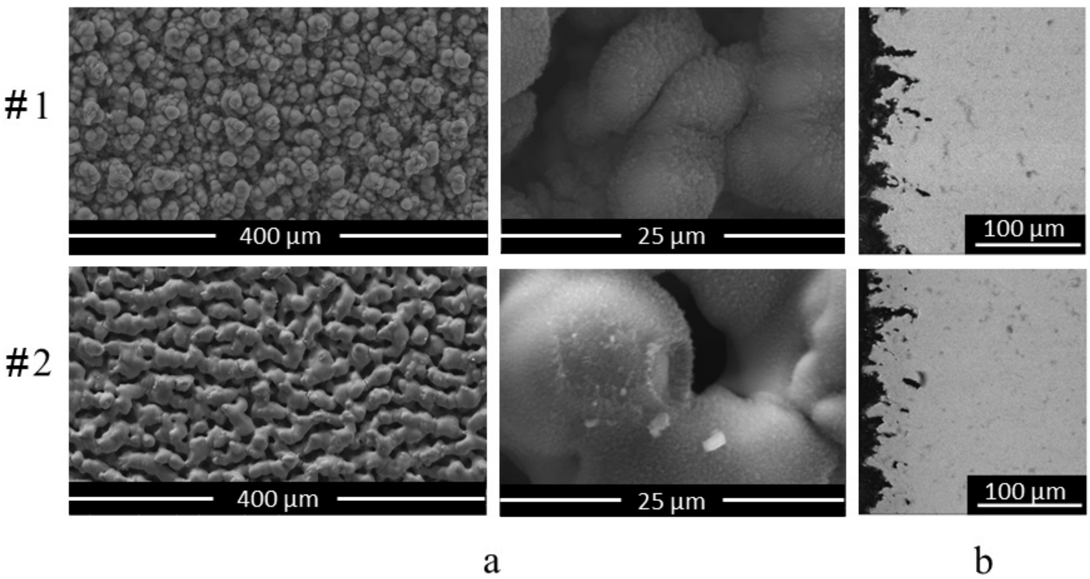

Figure 2. SEM-images (a) and structure of the modified layer cross-section (b) of the structured surface of Grade-2 titanium after laser irradiation of samples #1 and #2.

Table 2. Percentage of O and Ti on the surface of titanium before and after laser processing.

Sample	O, at.%	Ti, at.%
Pure Ti	9	91
#1	54	46
#2	53	47

ablation. These processes can be used for relief formation on the metal surface. Here, intensities sufficient for evaporating (sample #1, table 1) and melting (sample #2, table 1) of the material were used. Therefore, multi-shot irradiation led to the formation of complex relief that had both micro- and nanoroughness (figure 2(a)).

The sizes of different structural components of sample #1 were in the range of 5 to 27 μm with a $R_a = 3.40 \pm 0.02$ μm. The depth of modified layer H is 56.8 $\pm$ 2.6 μm (figure 2(b)). The microstructure of the surface of sample #2 differs in the formation of the relief consisting of extended, interpenetrating 3D microstructures with an average width of 12.4 $\pm$ 3.9 μm, $R_a = 2.91 \pm 0.15$ μm, $H = 52.9 \pm 1.9$ μm. The nanostructure of the sample surface corresponds to 255 $\pm$ 105 nm for both regimes.

The EDX-analysis of the surface after laser irradiation showed that the percentage of oxygen increased for all modification regimes (table 2).

Higher concentration of oxygen in surface and near-surface layers appears due to oxidation processes during sample irradiation, which is verified by the results of XRD (figure 3).

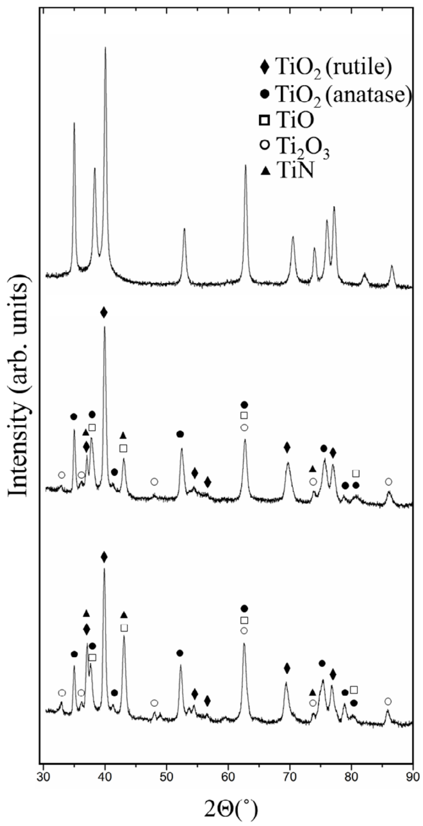

Figure 3. X-ray pattern of Grade-2 titanium surface before (a) and after laser irradiation (samples #1 (b) and #2 (c)).

Table 3. Tribological characteristics of unstructured titanium surface.

	Coefficient of friction		Wear factor (10^{-4} mm^3 N^{-1}m^{-1})	
Testing regime	Initial	Average	Counterbody	Sample
A	0.16	0.73	0.7	9.6
B	0.72	0.58	1.0	8.1

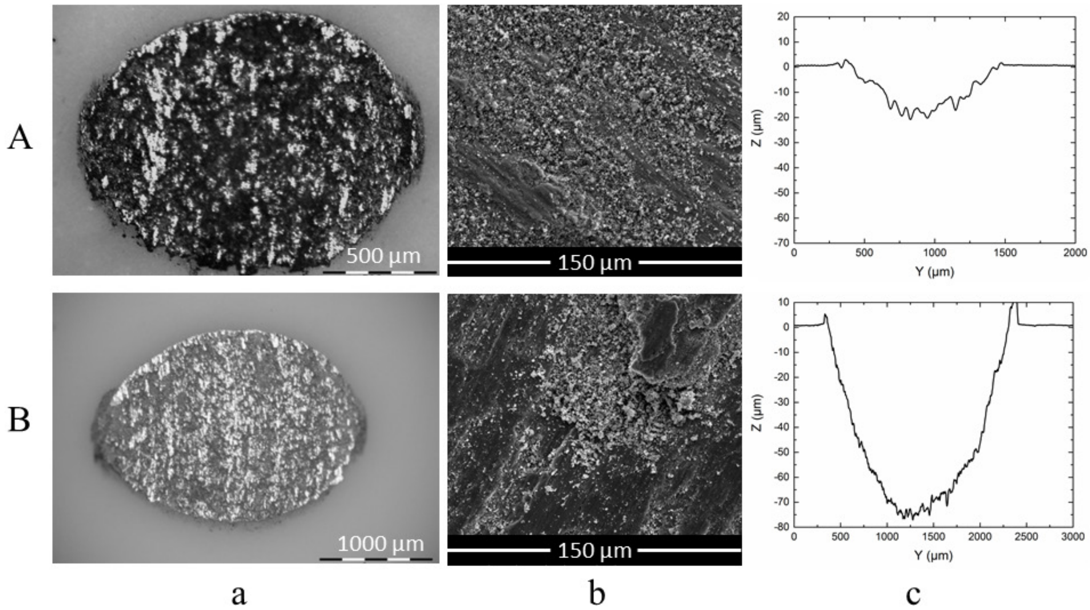

Figure 4. SEM-images of counterbody wear scar (a) and unstructured titanium surface (b) after tribological tests, cross-section profile of wear trace on the surface of Grade-2 titanium (c).

Table 4. Tribological characteristics of titanium after pulsed laser action.

		Coefficient of friction		Wear factor (10^{-4} mm^3 N^{-1}m^{-1})	
Processing regime	Testing regime	Initial	Average	Counterbody	Sample
#1	A	0.28	0.75	0.0019	1.6
	B	0.38	0.58	0.0018	6.3
#2	A	0.25	0.73	0.0010	0.9
	B	0.27	0.64	0.0028	7.1

It can be seen that because of laser processing, the modification of chemical composition took place: samples #1 and #2 contain Ti_2O_3, TiO, TiO_2 (in polymorphic modifications of rutile and anatase) and probably TiN. This composition is similar to the composition obtained in our previous work [19] that showed that multi-layered film forms as a result of laser irradiation, the upper layer of which consists of TiO_2 and the bottom layers consist of Ti_2O_3, TiO, and TiN.

3.2. Wear resistance of unstructured and structured titanium surface

3.2.1. Wear resistance of initial (unstructured) titanium surface. The study of tribological characteristics of unstructured titanium showed that the coefficient of friction settled after running-in has a value of 0.55–0.7 typical for this material and friction regime (table 3) [20, 21]. The increase in force resulted in the decrease of the coefficient of friction (from $\mu = 0.73$, regime A to 0.58, regime B). It is important to note that wear factor for samples decreased insignificantly with an increase of force, whereas for counterbody it increased. This seems to be connected to peculiarities of titanium behavior in case of friction, more specifically, the cold work hardening of the surface and the enrichment of surface and near-surface layers with gases from the surrounding atmosphere.

The studies of the morphology of friction trace surface, as well as wear scar of the counterbody, demonstrated typical for titanium and its alloys friction transfer of material from the sample surface to counterbody (figure 4). The presence of a significant amount of adhesive traces, deep tearing-out, and sticking indicate adhesive wear mechanism. In such wear conditions, a lot of local connections form in the contact area, the destruction of which leads to the increase of the force of friction, and, consequently, the coefficient of friction [4].

3.2.2. Wear resistance of structured titanium surface. Creating periodical multi-leveled relief on the surface of titanium did not significantly decrease the coefficient of friction for dry

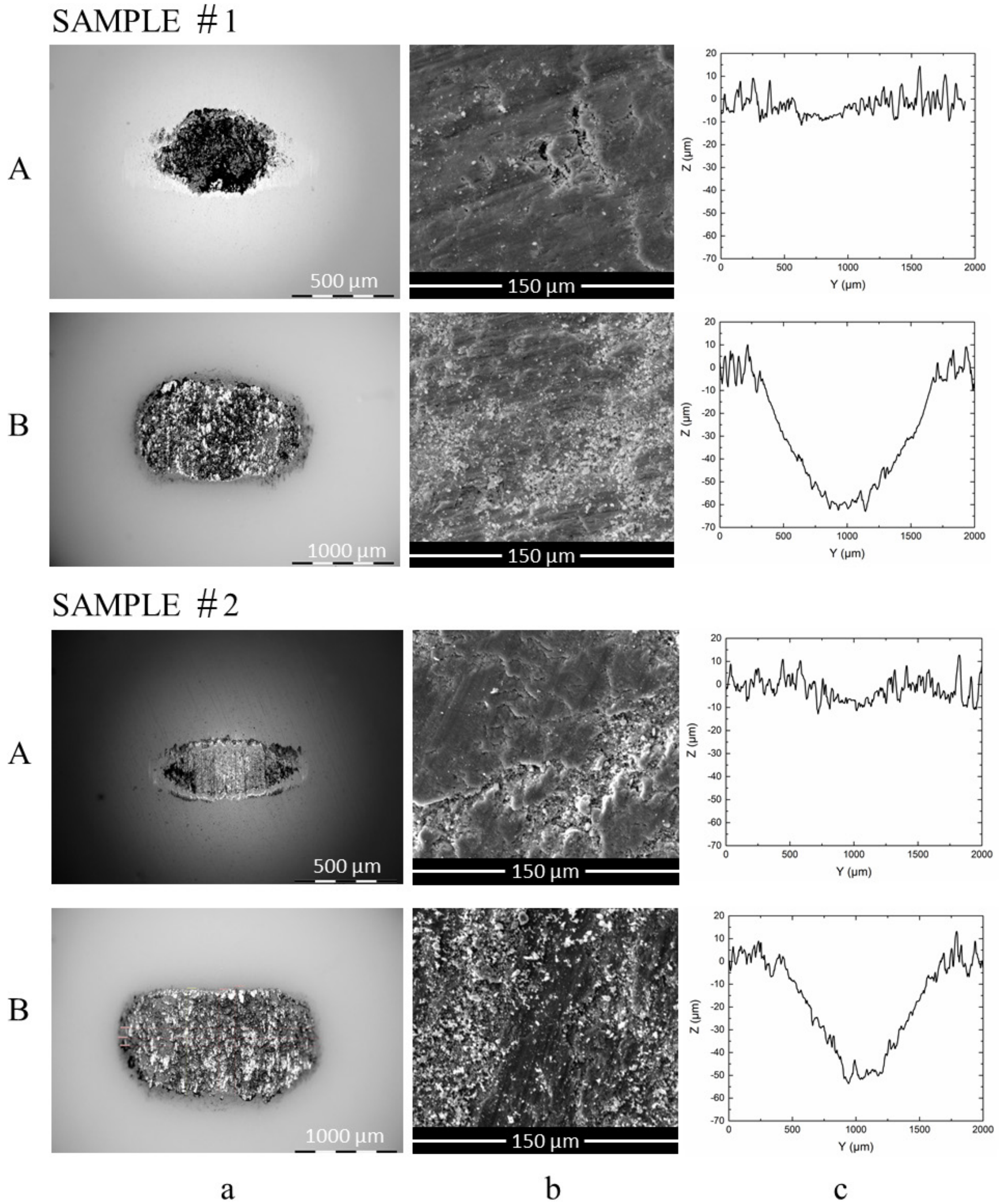

Figure 5. SEM images of counterbody wear scar (a) and samples #1 and #2 (b) after tribological tests, the cross-section profile of the wear trace on the surface of samples #1 and #2 (c).

friction over the unstructured surface (table 4). The irradiation regime did not influence the coefficient. Moreover, as for the case of unstructured titanium, the increase of force for tribological tests of samples with modified surface leads to the decrease of the coefficient of friction. For testing regime A ($p = 2$ N) μ changes in the range 0.73–0.75, for testing regime B (p = 10 N), μ changes from 0.58 to 0.64 for samples #1 and #2.

The analysis of counterbody wear factor change showed that for testing regime A counterbody wear factor decreases by about three orders of magnitude for all studied samples. However, for testing regime B, wear factor increases when compared to the correlating one for regime A, which is caused by the destruction of the modified layer when increasing the force. As a result of the damage, solid particles of titanium oxide form in the area of frictional contact, which act as abradant and increase the wear of adjacent body and the sample. On the surface of counterbody wear scar, obtained in tests for regime B, in friction couple with samples #1 and #2, there are traces typical for both abrasive (trenches, burrings, cutting traces) wear mechanism and adhesive (friction material transfer, adhesive traces, sticking) wear mechanism (figure 5(a)).

The structure of friction trace of structured samples in testing regime A indicates abrasive wear mechanism (figure 5(b)), i.e. friction occurred only on the surface of the modified layer.

The microstructure of the friction trace for testing regime B is typical for a mixed wear mechanism (adhesive and abrasive). It is important to note that abrasive wear mechanism, in this case, occurs because of titanium particles and titanium oxides.

According to a surface profile (figure 5(c)) of the friction trace on the samples tested in regime A, the layer destruction depth (sample #1: 9 $\pm$ 0.5 μm, #2: 10 $\pm$ 0.6 μm) is less than the modified layer depth (sample #1: H = 56.8 $\pm$ 2.6 μm, #2: 52.9 $\pm$ 1.9 μm); in testing regime B, the layer destruction depth (sample #1: 60 $\pm$ 0.5 μm, #2: 50 $\pm$ 0.6 μm) becomes larger than the modified layer depth.

Thus, in regime A the destruction of the modified layer does not occur, and the wear factors of the counterbody and the sample are the lowest. Wear factors for samples #1, and #2 are $1.6 \cdot 10^{-4}$ mm^3 N^{-1}m^{-1} and $0.9 \cdot 10^{-4}$ mm^3 N^{-1}m^{-1} accordingly.

A more significant increase of the wear factor, and consequently the decrease of the wear resistance of the samples occurs when increasing the force applied to the holder of the counterbody (regime B). The wear factors for samples: $6.3 \cdot 10^{-4}$ mm^3 N^{-1}m^{-1} (#1) and $7.1 \cdot 10^{-4}$ mm^3 N^{-1}m^{-1} (#2).

4. Conclusions

Irradiation of the surface of commercially pure Grade-2 titanium with a laser of nanosecond pulse duration at intensities enough for melting and boiling of the material ([2.70–6.95] $\times$ 10^7 W cm^{-2}) with a small scanning step (0.83–3.33 μm along the X-axis, 5.0 μm along the Y-axis) leads to a formation of a two-level relief which has both micro- and nanoroughness. Apart from relief modification, the phase composition of the processed surface changes as well. According to EDX and XRD studies, TiN, Ti_2O_3, TiO and TiO_2 (in polymorphic modifications of rutile and anatase) can be found in the composition of modified layers of the surface.

The studies of tribological properties for dry friction in the air show that the formation of periodic multi-level relief on the surface of titanium does not significantly decrease the coefficient of friction. The wear resistance of modified Grade-2 titanium surfaces increases compared to initial unstructured titanium.

In the case of no breakdown of the modified layer in friction (regime A), a maximum increase of wear resistance occurs for sample surface with a typically abrasive wear mechanism; wear resistance increases ten times as compared to virgin titanium. The destruction of the modified (oxide) layer during the frictional contact has an impact on the decrease of wear resistance and the change of wear mechanism from abrasive to adhesive-abrasive, which correlates to testing regime B: the wear resistance of the modified samples is relatively similar to initial wear resistance. After the destruction of the modified layer, solid particles of titanium oxide form in the area of frictional contact and act as abradant and increase the wear of the adjacent body and the sample.

Acknowledgments

Laser structuring of the samples and XRD-analysis were financially supported by the Ministry of Education and Science of the Russian Federation in the framework of the project no. 3.3144.2017/ПЧ. Tribological tests were carried out with the financial support of Russian Foundation for Basic Research grant no. 16-32-50171/16. The study of the regimes of laser irradiation on the morphology structured surface was supported by the Russian Foundation for Basic Research grant no. 17-43-500134/17.

References

[1] Segu D Z, Choi S G, Choi J H and Kim S S 2013 The effect of multi-scale laser textured surface on lubrication regime *Appl. Surf. Sci.* **270** 58–63
[2] Myshkin N K and Petrocovets M I 2007 Friction, lubrication, wear. Physical basics and technical applications of tribology *Phys. Mat. Lit.* (in Russian)
[3] Wu Z, Deng J, Zhang H, Lian Y and Zhao J 2012 Tribological behavior of textured cemented carbide filled with solid lubricants in dry sliding with titanium alloys *Wear* **292–3** 135–43
[4] Qin L, Lin P, Zhang Y and Zeng G D Q 2013 Influence of surface wettability on the tribological properties of laser textured Co–Cr–Mo alloy in aqueous bovine serum albumin solution *Appl. Surf. Sci.* **268** 79–86
[5] Xing Y, Deng J, Zhang K, Zhang G and Gao H 2014 Effect of femtosecond laser pretreatment on wear resistance of Al_2O_3/TiC ceramic tools in dry cutting *Int. J. Ref. Met. Hard Mater.* **43** 291–301
[6] Kovalchenko A, Ajayi O, Erdemir A and Fenske G 2011 Friction and wear behavior of laser textured surface under lubricated initial point contact *Wear* **271** 1719–25
[7] Ding Q, Wang L and Hu L 2015 Tribology optimization by laser surface texturing: from bulk materials to surface coatings *Laser Surf. Eng.* **16** 405–22
[8] Vorobyev A Y and Guo C 2007 Femtosecond laser structuring of titanium implants *Appl. Surf. Sci.* **253** 7272–80
[9] Romera G R B E, Huis in't Veld A J, Meijer J and Groenendijk M N W 2009 On the formation of laser induced self-organizing nanostructures *CIRP Ann.* **58** 201–4
[10] Kolobov Y R, Zhidkov M V, Golosov E V, Vershinina T N, Kudryashov S I, Makarov S V, Ionin A A and Ligachev A E 2016 Phase composition and structure of femtosecond laser-produced oxide layer on VT6 alloy surface *Laser. Phys. Lett.* **13** 076103
[11] Kolobov Y 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 2015 Structural transformation and residual stresses in surface layers of $\alpha + \beta$ titanium alloys nanotextured by femtosecond laser pulses *Appl. Phys.* A **119** 241–7
[12] Leyens C and Peters M 2003 Structure and properties of titanium and titanium alloys *Titanium and Titanium Alloys* ed C Leyens and M Peters (New York: Wiley) pp 2–3
[13] Zhecheva A, Sha W, Malinov S and Long A 2005 Enhancing the microstructure and properties of titanium alloys through nitriding and other surface engineering methods *Surf. Coat. Technol.* **200** 2192–207

[14] Ettaqi S, Hays V, Hantzpergue J J, Saindrenan G and Remya J C 1998 Mechanical, structural and tribological properties of titanium nitrided by a pulsed laser *Surf. Coat. Technol.* **100** 428–32

[15] He D, Zheng S, Pu J, Zhang G and Hu L 2015 Improving tribological properties of titanium alloys by combining laser surface texturing and diamond-like carbon film *Tribol. Int.* **82** 20–7

[16] Guo B, Zhou J, Zhang S, Zhou H, Pu Y and Chen J 2008 Tribological properties of titanium aluminides coatings produced on pure Ti by laser surface alloying *Surf. Coat. Technol.* **202** 4121–9

[17] Sant C, Daia M B, Aubert P, Labdi S and Houdy P 2000 Interface effect on tribological properties of titanium–titanium nitride nanolaminated structures *Surf. Coat. Technol.* **2** 167–73

[18] Guéhennec L L, Soueidan A, Layrolle P and Amouriq Y 2007 Surface treatments of titanium dental implants for rapid osseointegration *Dent. Mater.* **23** 844–54

[19] Ageev E I, Andreeva Y M, Karlagina Y Y, Kolobov Y R, Manokhin S S, Odintsova G V, Slobodov A A and Veiko V P 2017 Composition analysis of oxide films formed on titanium surface under pulsed laser action by method of chemical thermodynamics *Laser Phys.* **27** 046001

[20] Kovaleva M G, Kolpakov A Y, Poplavskii A I, Goncharov I Y, Prozorova M S, Arseenko M Y and Smolyakova M Y 2012 Tribological characteristics of nanosized carbon coatings obtained by the pulsed vacuum-arc method on the modified tini surface *J. Frict. Wear* **33** 260–5

[21] Beresnev V M, Grudnitskii V V, Nemchenko U S, Kaverin M V, Makhmud A M, Kirik G V, Smolyakova M Y, Kolesnikov D A and Komarov F F 2012 Triboengineering properties of nanocomposite coatings Ti–Zr–Si–n deposited by ion plasma method *J. Frict. Wear* **33** 167–73