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The effect of femtosecond laser treatment on the tribological properties of titanium nitride

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Abstract. The influence of femtosecond laser (fs-laser) processing on the structural-phase state of titanium nitride (TiN) coatings have been studied. It has been revealed using scanning electron microscopy and scanning probe microscopy that laser-induced periodic surface structures (LIPSS, LSFL) form on TiN coating surfaces. Depending on the laser scanning direction, two types of surface structures - parallel and cross-like ones have been obtained. Tribological tests showed that cross-like structures demonstrates better wear resistance properties in a dry friction, while parallel ones in a friction with lubricants.

1. Introduction

The development of modern technologies in the field of tribology will enable to talk about the existence of reliable and durable mechanisms that work even in fairly heavy conditions. The issue of wear resistance of materials has currently faded into the background. According to the literature, relatively high friction coefficients are the main problem of a production [1]. Analysis of the effects of friction and wear on energy consumptions, economic costs and the carbon dioxide emission levels has revealed that about 23% of the total energy consumption in the world comes from tribological contacts, and 20% of them are friction losses [2]. It was shown [3] that the effect of reducing the friction coefficient only may lead to short-term savings of up to 105,000 million Euros per year and a reduction in carbon dioxide emissions of about 200 mil tons in transport industry.

Producing laser-induced periodic surface structures (LIPSS) with multi-level roughness is prospective from the point of view of controlling the tribological properties, especially to obtain ultra-low friction coefficients [4-7]. By the example of natural phenomena, such as the gecko effect, the lotus effect and shark skin, we can talk about a successful applying a multi-level periodic relief to improve and control surface properties [8, 9]. The presence of a large number of studies on these effects, as well as the search for methods to repeat these properties and the corresponding structures on metal surfaces, only gives promises in this direction. Such studies were previously carried out on bulk materials, but with developing new commercial methods of laser exposures with ultra-short pulses, a new direction appeared - structuring coating and thin film surfaces. This allowed preserving advantages of existing coatings and additionally achieving new ones. Producing a multi-level periodic surface relief can control friction coefficients and obtain superhydrophilic or superhydrophobic surfaces. From the point of view

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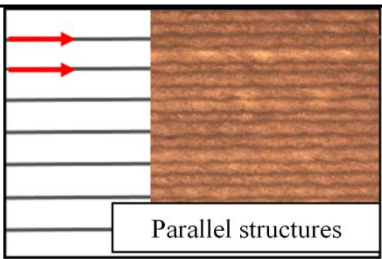
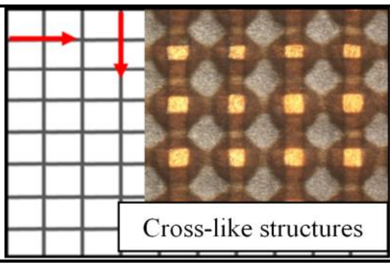
of medical use, it is possible to improve a biocompatibility of already existing materials. However, it should be noted that producing LIPSS on the coating surface is complicated by a number of additional factors comparison with bulk materials, such as coating adhesion, unrelaxed stresses at coating-substrate interfaces [5].

In this work, the surface morphology and phase composition changes in titanium nitride (TiN) coatings have been systematically studied after a fs-laser processing. The goal of present work is to investigate the LIPSS formation conditions on solid refractory TiN coating surfaces and relationships between their periodic structure parameters and friction coefficients in dry and lubricated friction conditions.

2. Experimental details

Titanium nitride coatings were deposited on a substrate of VT1-0 titanium alloy by plasma assisted vacuum-arc spraying [10]. The TiN coating surface was irradiated with an Yb-fiber laser generating ultrashort pulses with a duration of 320 fs and a wavelength of 1030 nm. The parameters of fs-laser processing are given in Table 1. These coating surfaces were ablated with three different energies: 6.2 μJ (E_{max}), 3.2 μJ (~ $E_{max}/2$) and 1.5 μJ (~ $E_{max}/4$). In this case, two types of the samples with parallel and cross-like structures have been obtained for each laser energy used.

Table 1. The irradiation parameters for TiN coatings.

Scanning direction			
Energy for Laser Processing, μJ	6,2 (E_{max})	3,2 (~$E_{max}/2$)	1,5 (~$E_{max}/4$)
Scan speed, mm/s		100	

Tribological tests were carried out in air using a Tribometer automated friction machine (CSM Instruments). Dry friction and lubricated friction regimes were applied. Engine and cutting oils were used as lubricants. The lubricants with a volume of 3 ml were added to a friction contact area before starting the tests. The test scheme is shown in figure 1.

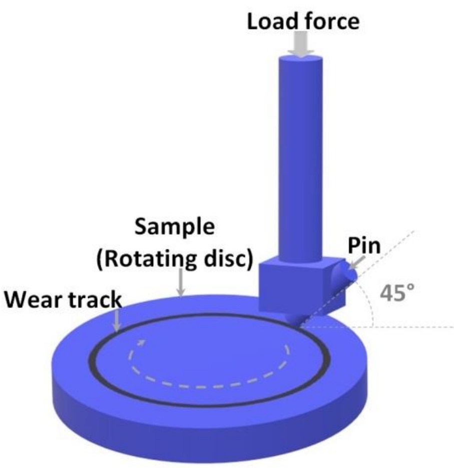


Figure 1. Scheme of tribological tests, "pin - disk".

A 6 mm pin made of steel 304 was used as a counterbody. A load was 1 N. Sample rotation speeds (v) were 5 cm/s for the friction path (l) of 450 m and 15 cm/s for l = 2000 m. Wear tests were performed in accordance with ASTM G99-959 and DIN50324 international standards. A friction track and the counterbody wear spots were analyzed after tribological tests using an Olympus GX71 optical microscope and FEI Nova NanoSEM 450 and Quanta 200 3D scanning electron microscopes (SEM). The surface structure of TiN coating was characterized before and after fs-laser processing.

X-Ray analysis was carried out in a sliding beam mode using an ARL XTRA diffractometer. The coating surface morphology and its roughness were analyzed using an NTEGRA Aura scanning probe microscope using a contact atomic force mode.

3. Results and discussion

To obtain the TiN coating surface with a parallel structure, the laser scanning steps were chosen to form a continuous structure. As a result, overlays and intersections between the laser scanning lines took place (Figure 2). It is seen in optical metallography images that structures formed along the scanning lines are heterogeneous. An increase in the laser processing energy from 1.5 μJ to 6.2 μJ, the structure becomes more uniform and well-periodic (Figure 2 a-c). So, the scanning line width, characterizing with a roughness parameter, Sm (Table 2), is 6.0 μm and 9.3 μm for 1.5 μJ and 3.2 μJ laser energies, respectively. Moreover, the statistical errors of S_m measurements were more than 60% for both processing modes. At the same time, S_m decreased up to 4.3 μm with the error less than 30% for the fs-laser processing with the energy of 6.2 μJ.

Table 2. Surface roughness parameters after fs-laser processing.

Structure	**Laser energy,** mJ	**S_a,** nm	**S_z,** nm	**S_m,** mkm
Parallel	1,5	88	468	6,0
	3,2	132	523	9,3
	6,2	61	459	4,3
Cross-like	1,5	166	417	25,0
	3,2	257	710	41,7
	6,2	321	1039	54,5

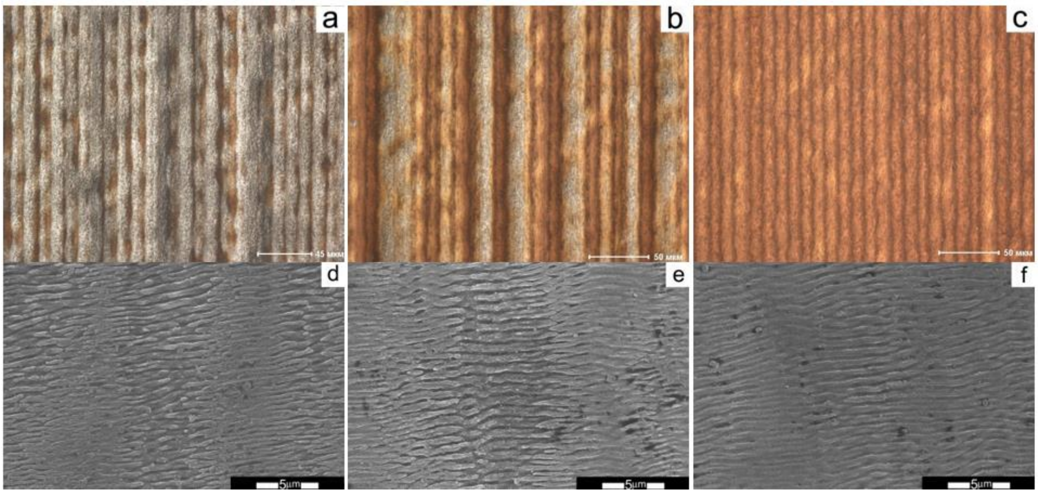


Figure 2. Microstructure of the TiN coating surfaces after fs-laser processing with different laser energies (parallel structures): a, d - 1.5 μJ; b, e - 3.2 μJ; c, f - 6.2 μJ.

SEM analysis has revealed the formation of laser-induced periodic surface structures (LIPSS) oriented perpendicular to the laser polarization direction (Figure. 2 d-f). A period of the observed structure varied in a range from 910 nm to 1050 nm, which corresponds to a wavelength of the laser applied. Structures with this periodicity are called low spatial frequency periodic structures (LSFL). A formation mechanism of them is based on the incident laser radiation interference with the surface electromagnetic waves generated by this irradiation [11].

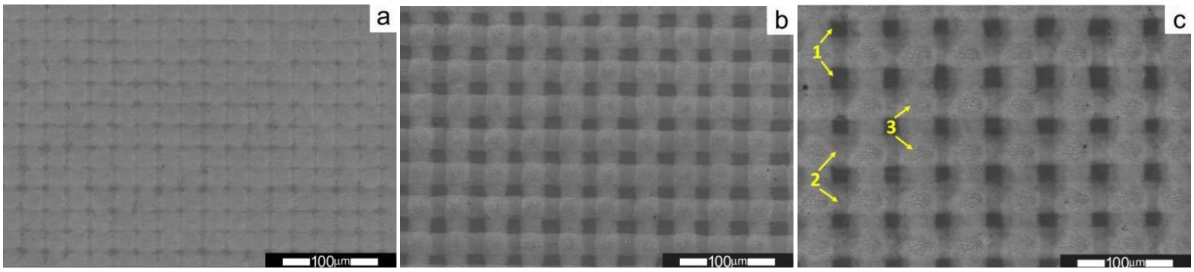


Figure 3. Microstructure of the TiN coating surfaces after fs-laser processing with different laser energies (cross-like structures): a - 1.5 μJ; b - 3.2 μJ; c - 6.2μJ.

Using SEM analysis three regions can be classified on the sample surfaces with cross-like structures: 1) dark regions of initial TiN coating surfaces without fs-laser processing; 2) regions with a single laser scanning pass; 3) regions with overlaying between laser scanning passes (Figure 3). Regions marked by 2 and 3 in Figure 3c are also characterized as periodic structures with a periodicity from 780 nm to 1060 nm.

According to X-ray analysis, the phase composition of coating was represented in the initial (unprocessed) state by two phases - α-Ti and TiN (Figure 4). For the TiN phase, X-Ray peak intensity distributions different from powder diffraction data have been found. It can indicate the presence of crystallographic texture in the TiN coating surfaces. Fs-laser processing with different laser energies led only to changes in the quantitative ratio of the phases in the coating surfaces, where there is no qualitative changes in the phase compositions. An increase in laser energy from 1.5 μJ to 6.2 μJ caused decrease in the X-Ray peaks originating from the TiN compound, which may indicate decreasing the coating thicknesses.

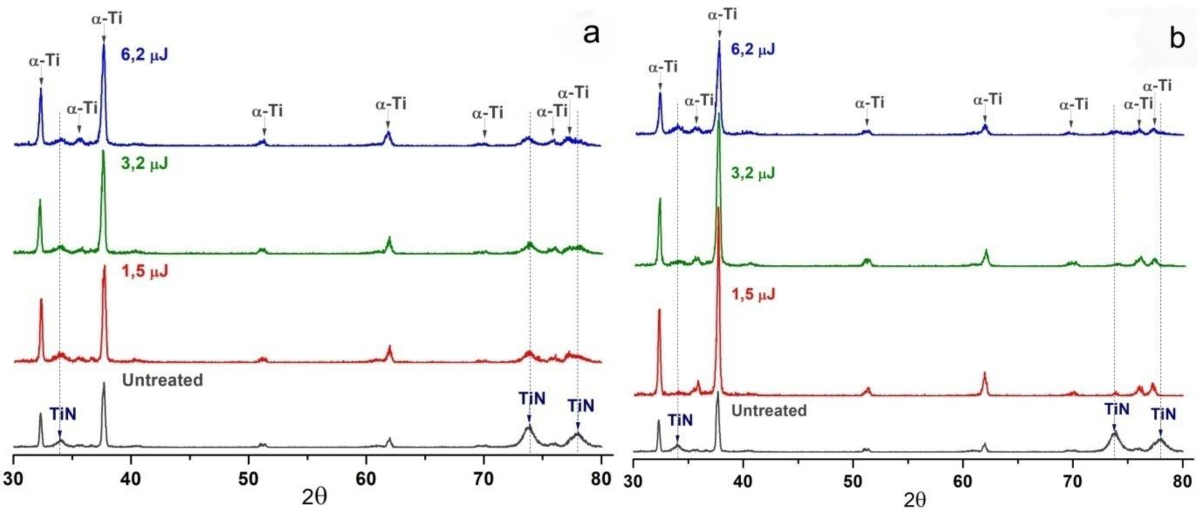


Figure 4. X-ray analysis of the TiN coatings before and after fs-laser processing with different energies: a - cross-like structures; b - parallel structures.

The surface roughness was estimated using two parameters - an arithmetic mean deviation, Sa, of the profile and a maximum height of the profile roughness, Sz (by 10 points). At the lowest laser energy (1.5 μJ), the minimum Sa values were 88 nm and 166 nm for the parallel and cross-like structures, respectively (Table 2). An increase in the laser energy by a factor of approximately 2 led to increasing in Sa values by 44% and 55% for the parallel and cross-like structures, respectively. The following increase in the laser energy from 1.5 to 6.2 μJ also contributed to an increase in the Sa parameter by 93% for cross-like structures, whereas Sa decreased by 31% for parallel structures. It should be noted that cross-like structures demonstrated a more developed surface relief in comparison with the parallel ones. Thus, the change in roughness parameters for parallel structures lied in the Sa range from 61 nm to 132 nm, and Sz range from 459 nm to 523 nm, and for the cross-like structures Sa and Sz varied in ranges from 166 nm to 321 nm and from 417 nm to 1039 nm, respectively.

The tribological properties of structured TiN coating surfaces have been studied in regimes of dry friction and friction with lubricants using engine and cutting oils. All the tribological test results are presented in Table 3. Dry friction test demonstrated relatively high friction coefficients for all the TiN coatings after fs-laser processing, and even for the unprocessed TiN coating surface. The friction path in this regime was ~310 m, but some tests had to be stopped earlier due to a coating destruction leading to a catastrophic wear. The coating destruction led to the formation of TiN solid particles acting as abrasive agents. It caused a drastic increase in a wear rate for the coating itself. The coating destruction have been found in the samples in initial state, the samples processed by the laser energy of 6.2 μJ, and the samples with parallel structures processed with the laser energy of 3.2 μJ. Cross-like structures demonstrated a well-developed relief: protrusions, grooves, holes, which can serve as "storage" places for the wear products forming in frictional contact areas, and thereby increase wear resistance. This can also be confirmed by the fact that, at the laser energy of 3.2 μJ, parallel structures collapsed at l = 35 m, whereas the cross-like structures was not even possible to test. In this case, only local filling of the grooves with the counterbody wear products has been found on the friction track surface. The friction paths to fracture for the samples after fs-laser processing with the laser energy of 6.2 μJ were 260 and 150 m for the cross-like and parallel structures, respectively.

Table 3. Tribological test parameters for the TiN coating after fs-laser processing with different laser energies. Respective friction paths are marked.

Structure	Laser energy used, mJ	Dry friction	Engine oil, l=450 m	Cutting oil, l=450 m	Cutting oil, l=2000 m
Parallel	1.5	0.46	0.55	0.09	0.04
	3.2	1.07	0.11	0.09	0.07
	6.2	0.90	0.12	0.06	0.05
Cross-like	1.5	0.85	0.09	0.13	0.06
	3.2	0.97	0.13	0.12	0.11
	6.2	0.98	0.13	0.13	0.05
Ti+TiN	-	0.86	0.14	0.14	0.14

Tribological tests using lubricants have shown that the friction coefficient for the initial state of the TiN coating remained stable during the test and was 0.14, regardless of the lubricants used and friction paths (table 3). This friction coefficient behavior allowed talking about positive effect of the structure itself, but not only about the lubricant properties and their additives. Worthy to note analyzing the tribological results using lubricants that there is no coating destruction in the samples for all the fs-laser processing regimes used as well as lubricant types. Tests of cross-like structures in engine and cutting oils at l = 450 m have not revealed any significant decrease in friction coefficients in comparison with the TiN coatings without fs-laser processing. The similar behavior was observed for the parallel structures during friction in the engine oil. Moreover, the structure after fs-laser processing with the energy of 1.5 μJ led to an increase in the friction coefficient up to 0.55. An analysis of the results obtained on the TiN coatings with the parallel structures and in the cutting oil showed reduction in the friction coefficient. The lowest friction coefficient was 0.06 after fs-laser processing with the energy of 6.2 μJ. It is also interesting to note that an increase in the friction path from 450 m to 2000 m and an increase in the rotation speed during friction in the cutting oil enabled to reduce the friction coefficient. Its values varied in a range from 0.04 to 0.07 depending on the laser energy. Thus, the lowest friction coefficient of 0.04 was detected for the parallel structure after fs-laser processing with the energy of 1.5 μJ. The friction coefficient tended to continuously decrease with increase in the friction path that suggests that a running-in stage may not have completed yet (Figure 5). Also, low values of the friction coefficients were observed for TiN coatings with the parallel structure after fs-laser processing with the energy of 6.2 μJ, and for TiN coatings with the cross-like structure after fs-laser processing with the energies of 1.5 μJ and 6.2 μJ (Table 3). This friction coefficient behavior is probably due to structural features related with the lubricant retention in the friction contact area at elevated speeds as well as the formation of a thin layer of the lubricant distancing the friction surfaces.

4. Conclusion

The fs-laser processing of the TiN coating surfaces enabled to form laser-induced periodic surface structures with a period close to a laser wavelength. Cross-like structure demonstarted a more developed relief in comparison with the parallel structures. It has been shown that there is no change in the phase composition of the coating surfaces after fs-laser processings, being represented with the α-Ti and TiN phases. The influence of periodic surface structures on tribological properties has been studied. Cross-like structures have been found to be better in dry friction, but parallel structures showed the lower friction coefficients in tests using cutting oil as a lubricant. Moreover, an increase in friction paths and speeds yields friction coefficients in a range from 0.04 to 0.07 for parallel structures. The lowest friction coefficient was measured for the TiN coating surface with parallel structures after fs-laser processing with the energy of 1.5 μJ.

Acknowledgments

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