Journal of Machine Engineering, Vol. 12, No. 2, 2012

laser treatment, nitriding, titanium,
wear resistance, microhardness,
phase composition

Yurij SHALAPKO[1], Natalia MASHOVETS[1]
Norbert RADEK[2], Tomasz KALACZYNSKI[3]

SURFACE MODIFICATION OF TITANIUM USING THE COMPLEX TECHNOLOGIES

In this paper the results of experimental research of microhardness, thickness, phase composition and wear resistance of pure titanium surface layer after laser treatment with different regimes and next nitriding in a glow discharge in hydrogen-free medium were presented.

1. INTRODUCTION

Titanium have unique constructional material properties – small specific gravity, low module of elasticity, high corrosive resistance and good compatibility with the the human organism [1]. At the same time, clean titanium has low hardness and small wear resistance of surface. For the increase of physical-mechanical properties the titanium alloys as alloy additions contain other elements: chrome, molybdenum, nickel, aluminium, manganese, niobium, zirconium, tin, nitrogen et al. [2],[3]. However, the metallic alloys which contain the afore-mentioned alloying elements are toxic for the human organism. In the case of application them in quality implants there is dissolution of alloying elements in a biologically active medium and their accumulation in the vitally important organs of living organism. In addition, there is damage of between crystalline border and increase wear surface [4]. It is a serious obstacle for application of such alloys in production of implants. The pure titanium have not such defects, but in the case of application in quality of implants it needs modification of surface for the increasing itd physical-mechanical and tribological properties.

Application of the low temperature nitriding in a glow discharge for strengthening of surface layer of pure titanium increase ca. 3,5 times hardness of surface layer and almost 2 orders wear resistance in an aggressive medium (Ringer's solution) at the maintenance of initial descriptions of durability[5]. New prospects for the increase of durability

[1] Khmelnitsky National University, Ukraine
[2] Kielce Technology University, Poland
[3] Faculty of Mechanical Engineering, University of Technology and Life Sciences in Bydgoszcz, Poland

of surface layer create application of complex technologies titanium with the use of laser heat treatment and next nitriding in a glow discharge.

2. METHOD OF EXPERIMENTS

Specimens, made of titanium VT1-0 (Table 1), 10mm diameter, were annealed, polished on a butt-end surface and processed on this surface the impulsive ray of laser with the diameter of bunch 0.7 mm, sometimes actions of impulse of 0.4÷0.68ms, by frequency 50 Hz, the step of plate moving with 0,4 mm, in the medium of nitrogen with an expense 25 dm^3/min, and at three different regimes:

I – without melting (power: 25W, voltage: 490V, time of impulse: 0,45ms),
II – less melting (power: 60W, voltage: 600V, time of impulse: 0,45ms),
III – more melting (power: 90W, voltage: 600V, time of impulse: 0,68ms).

Table 1. Chemical composition of VT1-0, %

Fe	C	Si	N	Ti	O	H	Impurity
0,25	0,07	max 0,1	max 0,04	99,24÷99,7	max 0,2	max 0,01	other 0,3

After laser heat treatment specimens were nitrides in a glow discharge in hydrogen-free of saturate medium on the special equipment after the regime: temperature of diffusive satiation is 670 ^{0}C, pressure in a vacuum chamber 240MPa, saturate an medium 55 at.% N2 + 45 at.% Ar, time of nitriding 6 hours. After every type of treatment, to used the methods of metallographic, the microhardness of surface, thickness of the fixed layer, distributing of microhardness on a depth and phase composition of surface, was measured with the use of device of DRON 3-M. Research of wear resistance of specimens was conducted on the machine of pin-flat friction in the medium of liquid of Ringera at pressure on-the-spot friction of 3,5MPa and sliding speed 0,057m/s. As counter-body to used the titanium VT1-0 were applied specimens with analogical treatment of surface.

Measures of tearing down was conducted using the special device through each 100m of way of friction with 0,5µm step. The microhardness of friction surface was also measured. The results of measuring were determined as a value after a 10 multiple reiteration of measuring on every specimen. Every experience on the wear of specimens after this regime of strengthening were repeated three times and as the end-point was accepted arithmetic average from three experiments.

3. RESULTS OF RESEARCH

At Table 2 the results of experimental research of properties of surface layer are resulted to titanium VT1-0 after the different methods of treatment. From table 2 evidently, that the microhardness of surface layer after the different types of treatment has different, but considerably higher values as compared to the initial microhardness of 2 000MPa. In particular, the microhardness of surface after laser treatment without melting was 9 370 MPa, after laser treatment with lowl melting – 10 360MPa, after laser treatment with more melting – 11 970MPa. Application of the ionic nitriding in a glow discharge after previous laser treatment promotes the microhardness of surface at all three types of laser treatment and is, accordingly, 10 190MPa, 11 700MPa, 12 300MPa. Such takes a place a increase microhardness due to education on-the-spot nitride compounds of TiN and Ti_2N, hardness of which is considerably greater than microhardness of structure after laser treatment. The microhardness of basis is enhanceable to titanium VT1-0 after the wear of the fixed layer as compared to an initial value (2 000MPa) conditioned of hardening at a friction.

Table 2. The results of comparative tests to titanium VT1-0 on friction and wear at pressure of 3,5MPa, v=0,057m/s in the medium of Ringer's solution

№	Regime of treatment	Microhardness N_{100}		Thickness of hardened layer, h, μm	Way of friction L, m	Wear U, μm	Intensity of wear $I \cdot 10^{-8}$
		before wear	after wear				
1	Laser treatment without melting + ionic nitriding	10190	2440	170	500	228	45,6
2	Laser treatment without nitriding	9370	2390	140	100	179	179
3	Laser treatment, low melting + ionic nitriding	11700	2340	230	3200	267	8,3
4	Laser treatment with low melting	10360	2470	160	300	279	93
5	Laser treatment with more melting + ionic nitriding	12300	2340	250	5000	341	6,8
6	Laser treatment with more melting	11970	2430	180	400	378	94,5
7	Ionic nitriding: $T=667^0C$, P=240 Pa, τ=6 hour	6760	3240	250	1000	240	32,4
8	Not modified	2000	3310	-	100	400	400

In Fig. 1 microstructure of surface layer obtained after laser treatment of titanium VT1-0 after the different regimes is presented. The changes of layer structure and thickness after the different types of treatment – see Table 2, is visible. Application of nitriding in a glow discharge after laser treatment, not only increases hardness and changes phase composition of surface but also increases the thickness of the nitride layer and diminishes the gradient of hardness on a depth - Fig. 2.

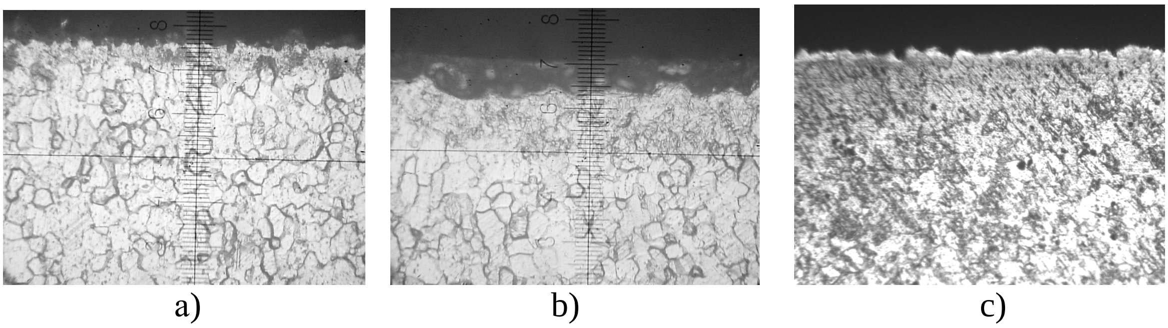

Fig. 1. The microstructure of titanium VT1-0: a) after modification of surface by laser treatment without melting, b) with melting, c) after a laser treatments without melting and with next nitriding

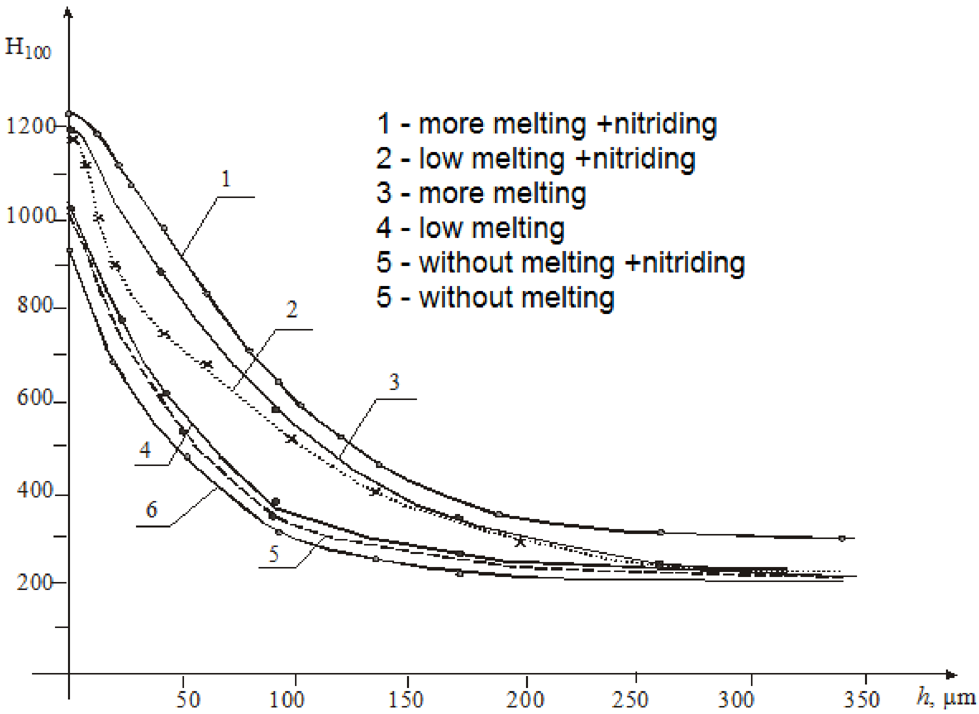

Fig. 2. Distribution of hardness on the depth of the modified layer to titanium at the different types of laser treatment and next ionic nitriding

From Table 1 it is visible that the maximal thickness of the modified layer is obtained after laser treatment with more melting and next nitriding in a glow discharge and fixed by 250µm. The thickness of the fixed modified layer at laser treatment without the following nitriding was less, and it is:

- 140µm – at laser treatment without melting,

- 160μm – at laser treatment with low melting,
- 180μm – at laser treatment with more melting.

It is discovered that the thickness of the modified layer depends on power of laser exposure and increased with its increase. Microfine structure of surface layer of titanium, appeared at laser treatment with melting is owing to increase of diffusion of nitrogen at nitriding in a glow discharge, and also is result of increase of thickness of the nitrides layer.

At Table 1 and in Fig. 3 the results of comparative tests of specimens in the medium Ringer's solution for titanium the surface which was strengthened after the different regimes are resulted. From a Table 1 it is possible to look after, that specimens which was processing of laser melting and next nitriding in a glow discharge under optimum regime had most wear resistance and longevity – a way of friction is 5 000m [5]. Intensity of wear of the fixed layer after the way of friction 5 000 m was I= $6.8\cdot10^{-8}$. At treatment of titanium after the regime by low melting and following nitriding, longevity of the layer was less than (a way of friction is 3 200m) and intensity of wear after the way of friction 3 200m was greater as I= $8.3\cdot10^{-8}$. Longevity of the layer after treatment without melting and next nitriding was considerably less – a way of friction is 500 m, and intensity of wear after 500m the way of friction I= $45.6\cdot10^{-8}$, so it is 5÷6 times more compared to the afore-mentioned regimes of treatment.

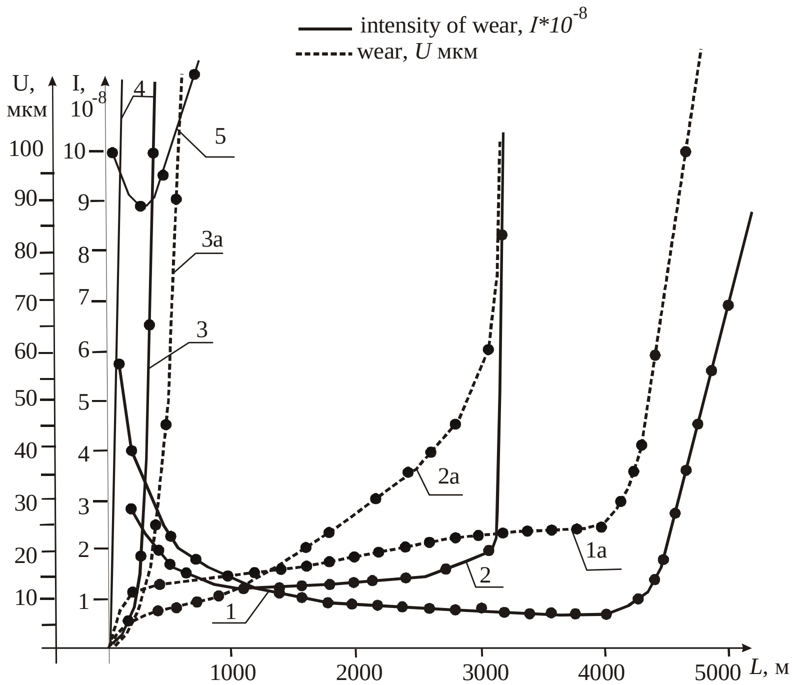

Fig. 3. Wear U and intensity of wear of $I\cdot10^{-8}$ depending on the way of friction at the different types of laser treatment and nitriding: 1 and 1a is more melting + nitriding; 2 and 2a is low melting + nitriding; 3 and 3a – without melting + nitriding; 4 - without melting; 5 is more melting.

Results of presented research are shown that intensity of wear of specimens which was strengthened laser treatment without the next nitriding, on an order higher from compared to the specimens with nitriding after laser treatment. It is explained a presence on-the-spot of friction of nitrogen and nitride compounds which arise up after nitriding, them by anti-friction properties and higher hardness and corrosive resistance of the modified layers.

In Fig. 3 it is shown the kinetics of wear of specimens is resulted to titanium VT1-0, that strengthened after the different regimes, depending on the way of friction. The specimens which was strengthened on complex technology (curves 1 and 2) at the beginning of tests had a period of bedding on a way by 500 m and subsequent period of normal wear with permanent intensity on a way to by 3 000 m for specimens were processed with low melting and 4 000m for specimens which was processed with more melting. In future intensity of wear was sharply increased in connection from elimination of the modified layer.

A specimen which was not nitriding after laser treatment, on the way of friction 200 ÷300m appears full wear, a layer is modified from subsequent catastrophic wear.

4. CONCLUSIONS

Presented in this paper research was shown that application of complex technology of strengthening titanium VT1-0 with the use of laser treatment and next nitriding in a glow discharge allowed considerably to promote (ca. 2 orders) wear resistance of surface layer in the aggressive medium compared to the not modified titanium.

REFERENCES

[1] ZAGORODNII N.V., 2000, *Titanium alloys in total hip arthroplasty*, Journal of Traumatology and Orthopedics, 2/73-75.

[2] SOLONINA O.P. GLAZUNOV S.G., 1976, *Tanium alloys*, Metallurgiya, Moscow, 448.

[3] ZWIKKER W., 1979, *Titanium and its alloys*, Metalurgiya, Moskwa, (trans. edited. O. E.Eutina and S.G. Glazunova), 512.

[4] HENCH L.L., 1998, *Bioceramics*, J. Am. Ceram. Soc., 81, No 7, 1705-1727.

[5] KAPLUN V.G., MASHOVETS N., ROSENBERG A.A, SHEYKIN S., 2011, *Influence of low temperature nitriding in glow discharge to wear-resistence a pair of "titanium-titanium,* Bulletin of the engine, 1, 67-73.