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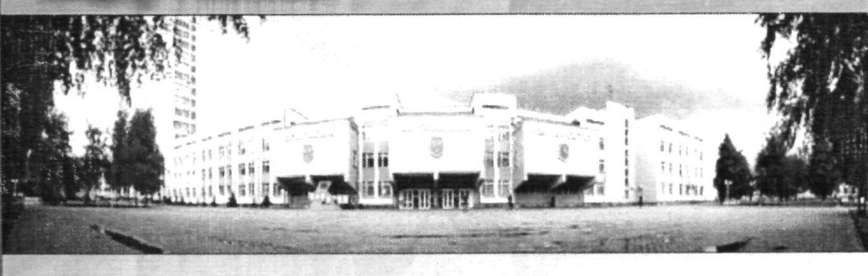

INFLUENCE OF COMPLEX TREATMENT ON THE PROPERTIES OF TITANIUM ALLOYS VT-6

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ABSTRACT

The paper presents new results on investigation of structure and physical-mechanical properties of near surface layers of titanium alloys VT-6 after (W^+, Mo^+) ion implantation and subsequent thermal annealing under 550°C during 2 hours. Using back scattering (RBS) of helium ions and protons, scanning electron microscopy (SEM) with microanalysis (EDS), (WDS), proton (ion) induced X-ray emission (PIXE), X-ray phase analysis (ERD) with a sliding beam geometry (0.5°C), Mössbauer spectroscopy (MS), measurements of nanohardness and elastic modulus, friction wear (cylinder-plate), measurements of corrosion resistance in a salt solution, we investigates VT-6 samples, and determined their fatigue resistance under cyclic loads. Two times increase of the hardness, decrease of wear and increased fatigue resistance was found, which was related to the formation of small dispersion (nanodimension) nitride, carbonitride, and intermetalloid phases.

Key words: implantation, properties, hardness

INTRODUCTION

Transition of medium energy ions through a solid is accompanied by scattering at matrix atoms and electrons, which results to deceleration and changing of ion motion direction, shifting of crystal atoms from lattice sites, accumulation of impurities in a target, sputtering of material surfaces, atomic mixing, formation of distribution profiles of implanted ions, formation of new phases. This essentially influences their mechanical and chemical properties [1 – 3]. Application of high-dose and intensive implantation results in shifting of an implanted ion concentration profile in surface vicinity, due to enhancement of a sputtering process.

In [3, 4] demonstrated that double implantation of Cu^+, No^+, Fe^+, Zr^+ into titanium alloys resulted in a change in microhardness, which was, first of all, related to surface layer hardening due to formation of martensite phases, small dispersion carbides and oxycarbides. It had already been published the works [4] in which they found almost 80% increase of the fatigue resistance in comparison with initial samples under Hf ion implantation into titanium alloys. In the middle of the 90^{-th} it was demonstrated that C, N, B implantation increased

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a cyclic life of Ti-6% Al-4% (VT-6) alloy by 4 – 5 times due to deceleration of dislocation motion and a decrease in fracture growth. Also it is known that W and mo can be applied as doping elements to increase hardness and improve servicing characteristics of construction materials [3, 4].

In this connection, investigations of high-dose and intensive W^+ and Mo^+ ion implantation (HDIII) effects on changes in physical-chemical and mechanical characteristics of titanium alloys VT-6 were of an undoubted interest.

METHODS OF SAMPLE PREPARATION AND INVESTIGATIONS

Samples of VT-6 (Ti, Al ~ 5.5 – 6.8 %, V ~ 3.5 – 4.5% of the mass content), of 15 x 15 x 2 dimensions, polished and annealed to reject residual stresses and cold working, were investigated. Metal ion implantation was performed in the vacuum-arc implantor "DIANA" under $5 \cdot 10^{17}$ cm^{-2} dose, 200 μs pulse duration, the sample surface temperature did exceed 300 °C. Ion implantation was performed in the accelerator chamber under 10^3 Pa residual vacuum. To analyze the element composition of samples we applied the RBS method for helium ions and protons of 2.035 MeV and 2.12MeV energies, respectively, and a scanning microscopy with microanalysis WDS and an ion-induced X-ray (helium) emission PIXE using the accelerator with an ion beam energy $^4He^+ \approx$ 3.1MeV (Darmstadt, Germany). The structure and surface relief analysis were performed with the help of a scanning electron microscope REMMA with a microanalyzer WDS (Selmi, Sumy) and EDS.

The tests were performed using a three-face Berkovich indentation with a nanohardness measuring device Nano Indenter-II (MTS Systems Corp., Oak Ridge, TN, USA). In the process of testing with a high accuracy a dependence of the indenter top displacement on a load was registered. An accuracy of the print depth measurement was equal to ± 0.04 nm, that of the indenter load - ± 75 nH. The device is able to perform about 3 load and displacement measurements per minute. To decrease vibrations the device was positioned at a vibration isolating table. In every test the indenter was loaded/unloaded three times, each time reaching higher load, which did not exceed 5 mH (≈ 0.5 Hz) with 150 mH depth.

RESULTS AND DISCUSSION

The results of phase analysis performed for VT-6 samples before and after W and Mo ion implantation demonstrated that the surface layer of VT-6 alloy composed: α-Ti, β-Ti, Al_3Ti. After irradiation ions W and Mo the intensity of diffraction lines was redistributed. One should note that basic intensities changes occurred with phases $Al_{0.67}Cr_{0.08}Cr_{0.08}$ and Al_3Ti. One can see that after implantation a wide set of elements: C, O, Al, Ti, V, Fe, Mo, W was found in this sample. Treatment of these spectra according to a standard program allowed us to obtain an element concentration over the surface layers.

After annealing of the implanted samples basic changes were related to Al_3Ti, in particular, a separated peak (111) Al_3Ti appeared in the diffraction pattern. Results taken in a small angle geometry (0.5° angle) demonstrates that in the region between (001) and (100) α-Ti the emission intensity increased, which seems to be conditioned by additional line (111) Al_3Ti (*Fig.1b*, *Table 1*). *Table 1* presents the results of PCA spectra treatment and phase analysis for VT-6 samples.

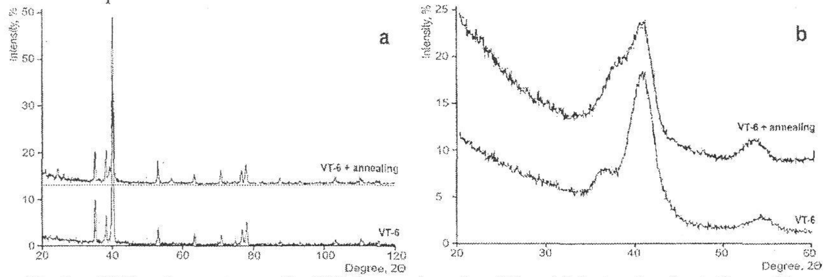

Fig.1 – Diffraction patterns for VT-6 samples after W and Mo ion implantation with $5\cdot10^{17}$ cm^{-2} dose, 60 keV, after annealing at 550 °C during 2 hours (*a*) and taken in a small angle geometry at 0.5° slope angle in the regions (100) and (101) α-Ti, and additional lines (111) Al_3Ti (*b*)

Table 1 – Results XRD-analysis for samples of VT-6 after implantation of W and Mo with energy 60 kV dose of $5x10^{17}$ cm^{-2}

№	Degree	Cleavage spacing.	Rel. intensity.	Phase	HKL	Ref. degree	Intensity
1	20,76	4,2785	12,24	Al_3Ti $Al_3Ti_{0.8}V_{0.2}$	002 002	20,705 20,747	15 6
2	35,260	2,5453	29,41	α-Ti	100	35,123	25
3	38,460	2,3406	100,00	β-Ti	110	38,514	100
4	39,300	2,2924	63,73	Al_3Ti $Al_3Ti_{0.8}V_{0.2}$ $Al_{0.67}Cr_{0.08}Ti_{0.25}$	112 112 111	39,150 39,345 39,395	100 100 100
5	40,280	2,2389	78,43	α-Ti	101	40,205	100
6	53,200	1,7217	27,45	α-Ti	102	53,051	13
7	56,940	1,6171	83,33	Al_3Ti $Al_{0.67}Cr_{0.08}Ti_{0.25}$	1019 112	56,910 56,936	12 5
8	63,440	1,4662	60,78	α-Ti	110	63,007	11
9	71,000	1,3275	76,47	β-Ti	211	70,728	17
10	76,740	1,2419	58,82	α-Ti	112	76,293	9
11	82,620	1,1678	23,53	β-Ti	220	82,528	4
12	93,080	1,0620	23,53	α-Ti	104	92,829	1
13	110,900	0,9360	23,53	α-Ti	211	109,17	4
14	115,120	0,9550	0,9134	α-Ti	114	114,42	3

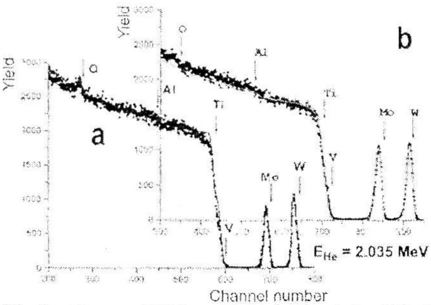

Fig.2 – Energy RBS spectra obtained for VT-6 samples after double W and Mo ion implantation (*a*) and after the subsequent annealing at 550°C (*b*)

Figure 2a, b shows the energy spectra RBS taken for VT-6 samples after double implantation of Mo and W ions for two various doses. In these spectra one can find Al, Ti, V, O, C elements, as well as implanted Mo and W ions. *Table 2* presents the results of element analysis for VT-6 irradiated with $2\cdot10^{17}$ cm^{-2} dose over the sample depth, which were taken using a standard program. As it is seen from the Table, a maximum W concentration reached about 4.44 at.%, its maximum occurring at 8.5nm depth (for $2\cdot10^{17}cm^{-2}$ dose). Mo concentration amounted about 11.65 at.% with the maximum at 15.5 nm depth. Also V (~2.91 at.%), Ti (37–87.57 at.%), Al (7.15–9.52 at.%) were found. An oxygen peak (16 at.%) occurred at about 23.5 nm, carbon (42.53 at.%) – at 7 nm. When the dose increased to $5\cdot10^{17}$ cm^{-2}, maximum W concentration reached 11 at.%, Mo concentration increased to 38 at.% (see *Table 3*).

Table 2 - Element concentration over VT-6 sample depth with implantation dose $2\cdot10^{17}$ cm^{-2}.

Depth, A	Elemental composition (at%)					
	W	Mo	V	Ti	Al	O
408.4	0.00	0.00	2.17	26.44	9.70	61.69
958.4	4.44	11.11	2.19	43.53	9.57	29.16
2524.1	0.00	0.00	2.17	88.14	9.69	0.00
4089.8	0.00	0.00	2.17	88.14	9.69	0.00
160658.9	0.00	0.00	2.17	88.14	9.69	0.00

Table 3 - Element concentration over VT-6 sample depth with implantation dose $5\cdot10^{17}$ cm^{-2}

Depth, A	Elemental composition (at%)							
	W	Mo	Fe	V	Ti	Al	O	C
85.2	11.06	,00	0,95	3,54	36,02	6,96	0,00	41,47
224.3	7.08	38,44	0,95	3,62	42,67	7,24	0,00	0,00
364.6	0.80	12,10	1,03	3,98	59,59	8,67	13,83	0,00
740.1	0.21	1,28	1,01	4,08	76,11	8,92	8,39	0,00
1483.9	0,10	1,05	1,00	4,03	79,90	8,88	5,04	0,00
156303.3	0,11	1,04	1,02	4,12	84,52	9,19	0,00	0,00

Figure 3a, b shows W and mo ion profiles obtained from the RBS energy spectra after implantation with $2\cdot10^{17}$ cm^{-2} dose and subsequent thermal annealing at 550 °C during 2 hours. The thermal annealing resulted on smearing of the profile, decreasing of the peak W and Mo concentration.

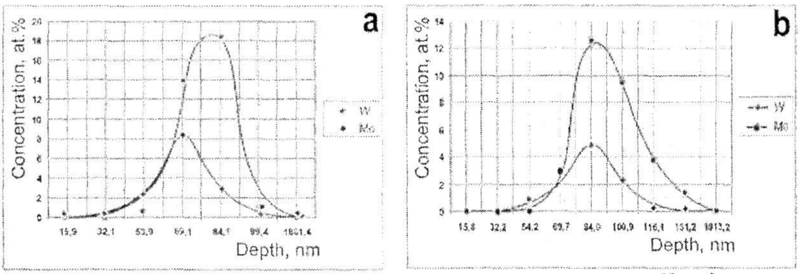

Fig.3 – Profiles of W and Mo ions obtained after implantation with $2\cdot10^{17}cm^{-2}$ dose (*a*) and subsequent thermal annealing in vacuum at 550 °C temperature during 2 hours (*b*)

The hardness H and the elastic modulus E were determined using nanohardness measuring device (Nanoindenter II) according to the method of Oliver-Pharr [5] and Berkovich indentation. *Table 4* present measurement results for nanohardness and elastic modulus of VT-6 samples before and after W and Mo ion implantation (with $2\cdot10^{17}$ cm^{-2} dose).

The initial sample hardness decreased a little with the print depth from 50 to 150 nm. This is a usual scale effect (indentation size effect). The hardness of implanted layer was higher a little, especially at 50 nm depth. Annealing after implantation resulted in a sharp increase of the surface layer hardness, and in comparison with initial material the depth decrease of the hardness occurred more intensively. This was an effect of lower situated non-hardened material. As it is seen from calculation results, the ion implantation resulted in increased hardness (almost by 100%), especially at 50 nm depth. And at 150 nm such increase amounted only about 50 %. The elastic modulus increased also by 45% for an indentation depth 50nm, little by little decreasing at 100 and 150 nm.

Table 4 – Results of hardness and elastic modulus for VT-6, GPa

Cast	Depth					
	50 nm		100 nm		150 nm	
	H	E	H	E	H	E
Before,	5,8 ± 0,9	123 ± 14	5.6 ± 0.8	124 ± 21	5.0 ± 0.5	141 ± 10
implantation	6,8 ± 0,3	127 ± 5	5.9 ± 0.4	120 ± 5	5.2 ± 0.5	115 ± 8
After annealing	10,7 ± 1,4	164 ± 25	9.7 ± 0.8	145 ± 9	8.5 ± 0.6	140 ± 7

CONCLUSION

In this work it was demonstrated that the double implantation of Mo and W ions into titanium alloys VT-6 with $5 \cdot 10^{17}$ cm^{-2} resulted in formation of concentration profiles with high element concentration at the maximum. Thermal annealing samples at 550 °C temperature during 2 hours resulted in the decreased peak Mo and W concentration and smearing of the element profiles.

Nanohardness measurements demonstrated that maximum hardness change was observed at about 50 nm depth, at 150 nm its value being essentially lower. After thermal annealing an elastic recovery of a print depth in unloading was a little higher than for initial sample, which indicated that the hardness growth was accompanied by a weaker elastic modulus increase.

Thermal annealing after Mo and W ion implantation resulted in a sharp hardness increase of a near surface layer. The hardness decrease with a depth was more significant in comparison with initial samples, i.e. it seemed to be explained by influence of a lower layer (a nonhardened material).

Results of a phase analysis performed for samples of titanium alloys before and after Mo and W ion implantation demonstrated that the VT-6 alloy was composed of: α-Ti, β-Ti, Al_2Ti, Al_3Ti. All the changes occurring after annealing of the samples by two types of ions seem to be related to Al_3Ti phase (i.e. the fully separated peak (111) Al_3Ti). Simultaneously, in the region (001) and (100) α-Ti an increased emission intensity was observed, which was conditioned by an additional line (111) Al_3Ti.

W and Mo ion implantation with $5 \cdot 10^{17}$ cm^{-2} dose resulted in increased hardness – almost by 100% at 50 nm depth and its decrease at 150nm. An elastic modulus of VT-6 alloy after implantation increased also at low indentation depths (50 nm) to 50% and decreased with indentation depth increasing.

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