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Formation of nanoscale carbon structures in the surface layer of metals under the impact of high intensity ion beam

G.E. Remnev[a], V.V. Uglov[b,*], V.I. Shymanski[b], S.K. Pavlov[a], A.K. Kuleshov[b]

[a] High-Voltage Research Institute at Tomsk Polytechnic University, 2a Lenin Ave., Tomsk 634028, Russia
[b] Belarusian State University, 4 Nezavisimosti Ave., Minsk 220030, Belarus

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ABSTRACT

This work represents the results of phase composition and the mechanical properties of tungsten and titanium after high-intensity pulsed ion beam (HPIB) treatment. It was shown that nanoscale carbide particles are formed under the HPIB influence in the surface layers of metals. Raising the pulse number results in increase of volume fraction of the carbide phases. The microhardness is 1.5–2 times more than the initial value and wear resistance of the metals improves in response to alloying of tungsten and titanium with carbon atoms accompanied by the formation of carbides.

1. Introduction

The improvement of mechanical properties and wear resistance of construction materials is an important problem of the material science. A promising solution to the problem is treatment of materials by concentrated energy flows. Using of high power pulsed ion beams (HPIB) [1–7] demonstrates good results in alloying and modification of the materials surface layers. Depending on the treatment parameters, the HPIB impact gives rise to appearance of phase composition changes; disperse grain structure and formation of areas with higher defects density that improve the mechanical properties of the modified layer. The high-energy density (up to 100 J/cm^2) efficiently deposited in the ion range (0.1–10 μm) during a short pulse width (less than 1 μs) make the HPIB a unique powerful pulsed energy source to rapidly melt and strongly vaporize the near-surface layer of materials. The plasma plume formed by HPIB ablation expands away from the surface and generates strong shock stress in the irradiated materials. These non-equilibrium processes (thermal effects and subsequent dynamical ones) could cause significant changes in composition, microstructure and morphology the irradiated materials, affecting their mechanical properties [8].

Generally, there are three regions of energy density causing different effects [9]. Below ≈1 J/cm^2 heating of the target takes place, but no melting. The sub-surface microstructure is modified due to recrystallization process as well as ion implantation. Between 1 and 10 J/cm^2 melting of the subsurface and rapid solidification can occur. The implanted ions diffuse deeper inside the material. Above 10 J/cm^2 materials may evaporate from the surface. The cited values of energy density are approximate and can usually be distinguished for different materials. The melting regime of the HPIB influence was effectively used for ion-beam mixing of a substrate and film to enhance adhesion between them, and improve the mechanical properties of the surface [10,11].

In [12,13] it was shown that the HPIB impact on silicon target results in formation of silicon carbide and diamond nanoscale particles in the surface layer. The ratio of carbon atoms to silicon ones that is necessary to produce a strong stoichiometric composition was achieved by using a high number of pulses. The synthesis of nanoscale carbide particles within such materials as titanium or tungsten can provide sufficient improvement of their mechanical properties. Therefore, the possibility of synthesis of metal carbide nanoscale particles under short-pulsed implantation of carbon ions will be discussed in the present work.

* Corresponding author. Tel.: +375 17 209 51 34.
E-mail addresses: remnev@hvd.tpu.ru (G.E. Remnev), uglov@bsu.by (V.V. Uglov).

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Thus, the main goal of this work is the investigation of the structure and phase composition of titanium and tungsten subjected to HPIB impact with different pulse numbers.

2. Experimental

The experimental samples (1 cm × 1 cm and 3 mm thickness) were prepared from commercial pure (CP) titanium alloy and tungsten alloy without impurities. The samples were subjected to mechanical polishing before the ion implantation in order to remove the surface impurities.

The ion beam generation was carried out in the TEMP-4M source using magnetically self-insulated diode with the focusing geometry configuration [3,14] at accelerating voltage of 220 kV. The pulse duration of accelerating voltage was 110 ns. The residual pressure in the chamber was 10^{-2} Pa that was reached due to pumping by diffusion pump. It may cause the influence of the adsorbed atoms on the chemical composition of the surface layer. The metal samples were fixed on the surface of collimated Faraday's cylinder that was located at 130 mm from the ion diode and was in geometric focus of the plane strip diode. The density of ion current was changed from 20 to 100 A/cm^2. The ion beam consisted of 70–80% of carbon ions and 20–30% of hydrogen ions. Short-pulsed ion implantation was realized for the titanium as well as tungsten samples treatment, the energy density absorbed by the surface of the target was 0.4 J/cm^2 (for titanium samples) and 2.5 J/cm^2 (for tungsten samples). The chosen values of energy density provide the boundary non-melting and melting regimes. Number of pulses and the total dose of the implanted carbon ions on the target at the different modes are presented in Table 1.

The concentration profiles of carbon and metals (titanium and tungsten) were measured by Auger electron spectroscopy (AES) using PHI-660 (Perkin Elmer) spectrometer. The experiments were carried out by means of sputtering of the samples surface by Ar^+ ions with kinetic energy 3 keV. The surface morphology was analyzed by means of scanning electron microscopy (SEM) in the mode of backscattered and secondary electrons registration using the LEO 1455 VP microscope. Phase composition of the implanted layer in the materials was determined by X-ray diffraction in Bragg-Brentano geometry with $Cu_{K\alpha}$ radiation (λ = 0.154178 nm). Structure and phase composition of the transverse section of the surface layer were investigated by means of transmission electron microscopy (TEM) technique using the Jeol JEM 2100-F microscope. A silver foil was used as a substrate for preparation the samples to TEM investigation.

Mechanical properties of the treated materials were characterized in terms of microhardness and tribological properties. The microhardness was measured with MVD402 Wolpert Wilson Instruments using Knoop diamond indenter. The applied load of the indenter being 25 g during the test, the indenter penetration depth did not exceed 1 μm that was comparable to the implanted depth. The tribological parameter, such as a friction coefficient, was determined by means of sliding test in "pin-on-plane" system with the 4 mm/s indenter speed. The tracks after tribological test were investigated by optical microscopy in order to reveal the main wear mechanisms.

Table 1
Modes of ion implantation by HPIB.

Material	Energy density (J/cm^2)	Number of pulses	Dose (ions/cm^{-2})
Ti	0.4	100	1.2×10^{15}
		300	3.6×10^{15}
		500	6×10^{15}
W	2.5	50	3.1×10^{15}
		150	9.4×10^{15}
		250	1.6×10^{16}

3. Results and discussion

The HPIB impact on tungsten and titanium samples results in alloying of the surface layers with carbon atoms, their concentration being determined by the number of the exposure pulses. The presence of carbon in the surface layer of the metals is confirmed by the elemental and phase composition analyses.

According to the AES results the near surface layers of the tungsten and titanium contain carbon atoms (Fig. 1). After 50 pulses of the HPIB influence the maximum carbon concentration achieved on the surface is 90 at.%. The average sputtering rate of tungsten by Ar^+ ions being equal to 100 nm/min, the thickness of the carbon containing layer can be estimated as 300–400 nm depth. The typical carbon distribution profile indicates the main diffusion mechanisms of mass transfer in the implanted layer. Calculations with SRIM/TRIM code showed the maximum penetration depth of the carbon ions (220 keV) up to 200 nm. So, there are additional mechanisms of mass transfer in the subsurface layer.

The kinetic energy of the ion beam partly transforms to the heat energy of the target during the HPIB exposure. The energy density absorbed by the surface layer is enough for its melting, as is confirmed by the SEM results (Fig. 2). The calculations of the space–time temperature evolution in the samples irradiated by HPIB are presented in Figs. 3 and 4. The numerical solution of the heat equation was obtained using finite difference method for the explicit scheme in accordance with the model described in [15,16]. The melting depth is about 320 nm and the maximum lifetime of the molten layer is 115 ns according to the calculations.

The intense heating of the tungsten surface layer under the ion beam influence increases the role of thermal diffusion associated with high (10^9 K/m) temperature gradient [17]. The pulsed mode of the HPIB impact results in formation of internal mechanical stress in the surface layer. The stress comes out from high temperature gradient and shock wave propagation in the material under the HPIB influence [18]. A set of cracks on the tungsten surface was revealed by SEM under 50 pulses HPIB impact (Fig. 2a). The propagation of the cracks toward the deeper layers of the sample results in fragmentary lamination of the areas where the internal mechanical stress exceeds the yield strength. The depth of the delaminated part of the tungsten does not exceed 10 μm (Fig. 2a) and the mechanical stress developed along this layer reaches at least 760 MPa (the

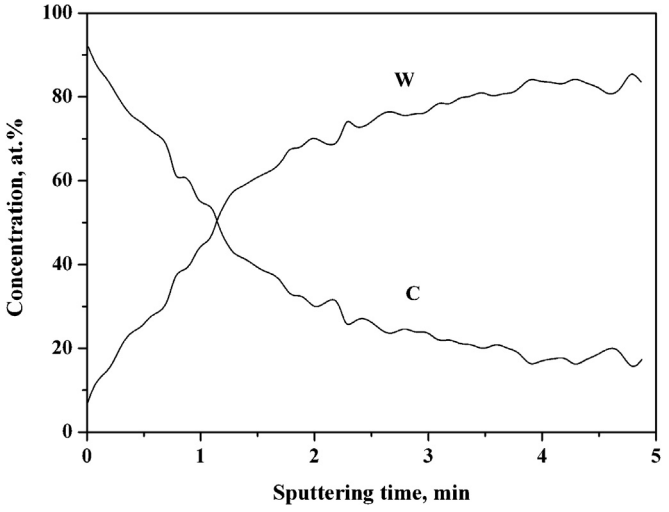

Fig. 1. Concentration profiles (W and C) obtained from the tungsten samples after 50 pulses of the HPIB influence (AES results).

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