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VIII ВСЕРОССИЙСКАЯ НАУЧНО-ПРАКТИЧЕСКАЯ КОНФЕРЕНЦИЯ С МЕЖДУНАРОДНЫМ УЧАСТИЕМ, ПОСВЯЩЕННАЯ 50-ЛЕТИЮ ОСНОВАНИЯ ИНСТИТУТА ХИМИИ НЕФТИ

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PHYSICAL-MECHANICAL PROPERTIES AND MULTILAYER HIERARCHICALLY ORGANIZED STRUCTURE OF Ti-Ta-BASED SURFACE ALLOY SYNTHESIZED ON THE NiTi-SUBSTRATE BY HIGH-CURRENT PULSED ELECTRON BEAM

[1]D'yachenko F.A., [1]Meisner S.N., [2]Yakovlev E.V., [1, 3]Semin V.O., [1, 3]Meisner L.L.
[1]Institute of Strength Physics and Materials Science SB RAS, Tomsk, Russia
[2]Institute of High-Current Electronics SB RAS, Tomsk, Russia
[3]National research Tomsk state university, Tomsk, Russia

Due to the functional properties of shape memory effect and superelasticity (SME-SE), nickel titanium alloys are used for manufacturing of miniature devices (*actuators*) for microelectromechanical systems (MEMs) in fields of high tech medicine [1]. It is known, that the surface state and its physical-mechanical properties have an important influence on the bulk functional properties of NiTi alloys in miniature devices (from ~1 up to ~100 μm). The most effective methods of surface modification of metal materials are electron beam and ion-plasma treatments. Combination of these methods makes it possible to create on the surface a multilayer hierarchically organized structure that has a high strength and elastic-plastic characteristics. Such structure can be formed by cycles of low-energy high-current pulsed electron beam (LEHCPEB) melting of film-substrate system [2]. This structure is called surface alloy, provide high cohesive force to substrate and improved physical-mechanical properties, unlike conventional physical and chemical deposited films/coatings [3].

In this study, using the LEHCPEB-treatment for formation Ti-Ta surface alloy on the NiTi-substrate, we investigated formed multilayer structure and its physical-mechanical properties.

The substrate material was commercial NiTi alloy (MATEK-SMA, Russia) produced as rolled sheets by vacuum induction melting (VIM). The chemical composition of the alloy is: Ti–55.08 Ni–0.051 C–0.03 O–0.002 N (wt %). The specimens were spark cut (EDM) to dimensions of 10 × 10 × 1 mm, chemically cleaned (acid solution: 3 part HNO_3 + 1 part HF), electrolytically polished (acid solution: 3 part CH_3COOH + 1 part $HClO_4$) and washed in an ultrasonic bath with a distilled water (duration t = 15 min).

The Ti-Ta-based surface alloy was formed on the modified automatic setup "RITM-SP" (Microsplav, Russia) allowing pretreatment by a microsecond LEHCPEB, Ti-Ta film deposition by magnetron sputtering, and electron beam melting in a single vacuum cycle [2]. To avoid cratering and local separation of the Ti-Ta film during pulsed melting the NiTi-substrate was preliminary irradiated with a LEHCPEB at a pulse duration τ = 2-2.5 μs, beam energy density E_s = 3.4 ± 0.7 J/cm^2, and number of pulses n = 32. Thereafter, the NiTi-substrate was alternately positioned by a manipulator along the magnetron sputter axis for deposition of $Ti_{70}Ta_{30}$ (at %) film of ~ 50 nm thickness and along the electron beam axis for pulsed melting of the Ti-Ta-film and NiTi-substrate at regime: E_s = 2 ± 0.2 J/cm^2, and n = 5. The number of deposition-melting cycles was N = 20 so that the melted films were in total no thicker than ~1 μm.

The microstructure in depth of the Ti-Ta-based surface alloy on the NiTi-substrate was examined on a JEM 2100 transmission electron microscope (JEOL, Japan) at an accelerating voltage of 200 kV. Thin foils were prepared from plates ~0.3 mm thick by ion thinning on an EM 09100IS ion slicer (JEOL, Japan). The physical-mechanical properties [4-6] (dynamic microhardness – H_{IT}, Young's modulus - E_{IT}, plasticity characteristic – δ_H, and depth inelastic recovery ratio – η) of the NiTi specimens were examined by instrumented nanoindentation on a Nano Hardness Tester (CSM, Switzerland) with a 4-faceted Vickers pyramid. The load on the indenter increased stepwise from 5 mN to 300 mN.

As a result of surface modification NiTi alloy has unique multilayer hierarchically organized structure that correlates with physical-mechanical properties of the alloy. The highest elasticity, lowest plasticity, and high hardness occur in submicrocrystalline upper layer I of ~200 nm thickness ($H_{IT} \approx$ 8 GPa, $E_{IT} \approx$ 95 GPa, $\delta_H \approx$ 40 %, η ≈ 50 %), which contains Ti-Ta-based α"-martensite and structurally unstable high-temperature β-phase. Amorphous sublayer II of ~400 nm thickness

(H_{IT} decreases from ~7 GPa to ~5 GPa, E_{IT} decreases from ~95 GPa to ~80 GPa, δ_H increases from ~45 % to ~57 %, and η decreases from ~40 % to ~30 %) provides a gradual transition from elastic layer I to dispersedly hardened sublayer III. Composite sublayers III, IV, and V, which are close in thickness and contain grains of average size > 20 nm and < 20 nm, provide a gradual transition of the physical-mechanical characteristics to the close strength values of the NiTi-substrate ($H_{IT} \approx 3$ GPa, $E_{IT} \approx 45$ GPa, $\delta_H \approx 60$ %, $\eta \approx 30$ %).

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