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The Annealing Under 1350 °C of Magnetron Sputtered Coatings on Base AlN-TiB_2($TiSi_2$)

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The magnetron sputtered coatings on base AlN-TiB_2($TiSi_2$) were investigated in this paper. The element composition, structural-phase composition, morphology and mechanical properties were investigated before and after annealing of coatings under 1350 °C. The concentration of elements in the coating were changed after annealing under 900 °C and further annealing under 1350 °C (especially after annealing under 1350 °C).The hardness of as-deposited coatings was 15 GPa, but after the annealing under 1350 °C the value of hardness was increased up to (22÷23.5) GPa. The value of the index viscoplastic was 0,07. The amorphous like structure and high damping properties of the AlN-TiB_2($TiSi_2$) coating makes promising the use of these coatings as a contacting layer in multilayer wear resistant coatings, and as diffusion barriers in the form of independent elements.

Keywords: Magnetron Sputtering, Structural-Phase Composition, Nanostructure, Annealing.

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1. INTRODUCTION

It well known [1-3] that it is possible to control the structure and substructure of produced materials by changing of ion-plasma deposited particles energy, which participate in formation of films. From the other side, it is possible to block the growth of grains of base phase by addition of one or several elements to a base material and thereby to form coatings with nanocrystalline or amorphous-like structure. It is necessary to learn how to operate by grain size and crystallographic orientations of grains in the growing film in order to form nanocrystallyne films and coatings. It can be achieved by several ways [4]: by changing of energy of sputtered particles of growing condensate; by penetration of addition elements into base material which bounds grains size growth; by deposition of multilayer films with nanoscale thickness; by formation of nanocomposite coatings. It can be achieved by several ways: by changing of energy of sputtered particles of growing condensate; by penetration of addition elements into base material which bounds grains size growth; by deposition of multilayer films with nanoscale thickness; by formation of nanocomposite coatings.

The basic parameters which can be used to control film structure are substrate temperature T_s; the energy Ebi which is transferred to growing film by bombarded ions and fast neutrals, as well as a quantity and type of addition elements [5, 6]. However, the other factors play a great role in formation of nanostructured films: intermiscibility or nonmiscibility of film components; ability of components to form solid solution or intermetallic compounds; enthalpy of alloy formation ΔH_f (positive or negative).

The structure of a film depends greatly on different factors and their reciprocal combinations [7, 8]. That is why investigation of phase composition, structure, physical-mechanical properties of multicomponent coatings which are composed of non-metallic matrix and nanostructured metallic components based on nitrides, carbides, borides and silicides have great scientific interest.

The main aim of this work is to carry out comprehensive analysis of structure, phase composition, surface morphology and physical-mechanical properties of coatings which are obtained by magnetron sputtering of multicomponent target AlN-TiB_2-$TiSi_2$.

2. EXPERIMENTAL DETAILS

Polished steel (Fe, 18 %-Ni, 12 %- Cr, 10 %- Ti) and monocrystalline silicon cylinders of 20 mm diameter and 3 mm highness were used as substrate materials.

The high-temperature AlN-TiB_2 composite systems with addition of $TiSi_2$ were used as sputtered material. The coatings investigated in this paper were deposited via the magnetron sputtering in Ar atmosphere.

Coating thickness, state of the coating-substrate interface were characterized using fracture fractography obtained by scanning electron microscope Quanta 600 and JEOL-7000F (Japan) operating at acceleration voltage 20 kV. The chemical composition and morphology were investigated using scanning electron microscopy (SEM) with microanalysis (EDS- energy disperse X-ray spectroscopy).

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The X-ray diffraction (XRD) data was recorded in Bruker - 8 Advance and DRON-4m apparatus using a Cu-Kα radiation.

Additionally, the analysis of surface topography was carried out by atomic force microscope NT-MDT (Russia). Si-cantilevers NSG10/W2C with hard current-conducting coating W2C of the 30 nm thickness were used.

The mechanical characteristics of the coatings layers were measured by a Nanoindentor G200 (MTS Systems Corp., USA) tester with a trihedral Berkovich pyramid with the tip radius at the apex 20 nm.

3. RESULTS

Surface topography investigations of specimens with coating were carried out by scanning electronic microscope Qunta 600 D. Fig. 1a represents SEM images of surface topography.

It is important to note, that in according to EDX analysis results, significant changes of elements concentration was observed after annealing at 900 °C and 1350 °C (Fig. 1b and Tab. 1).

Table 1 – Results of energy dispersive analysis of AlN-TiB_2-$TiSi_2$ coating before and after annealing under 900°C and 1350°C.

Element	At. %		
	Annealing temperature		
	As-deposited	900 C	1350°C
B	30.25	33.92	5,45
C	4.92	2.93	15,44
N	21.80	16.67	20,33
O	8.23	20.20	7,5
Al	14.18	9.80	1,7
Si	11.26	11.52	47,8
Ar	2.03	0.17	–
Ti	3.32	4.64	1,57
Mo	–	0.16	–
Re	–	–	0,22
Sum	100.00	100.00	100.00

Fig. 2a shows AFM image of as-deposited coating surface where grain boundary and nanotopography appears. Profile of AFM image and statistical analysis of heights distribution provides additional and more precise information about relief of the coatings. The statistical analysis showed that most of asperities had a heights in the range 15-20 nm. The lateral dimensions of asperities at the bottom were ~200 nm, and the width of asperities at the half of height were approximately 70 nm. It was established that roughness of the surface was varied from (30-35) nm to (70-90) nm after high temperature annealing.

At the numerous papers, which are devoted to investigation of results of energy influence on surface of condensed matter [9, 10] the appearance of defect-deformation instability have been shown. It causes the realization of critical conditions for development of synergetic effect which leads to development of surface structures of topography.

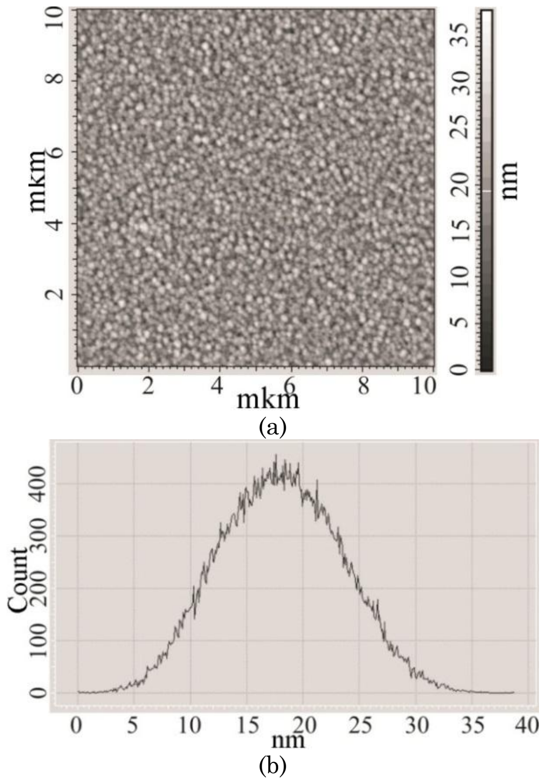
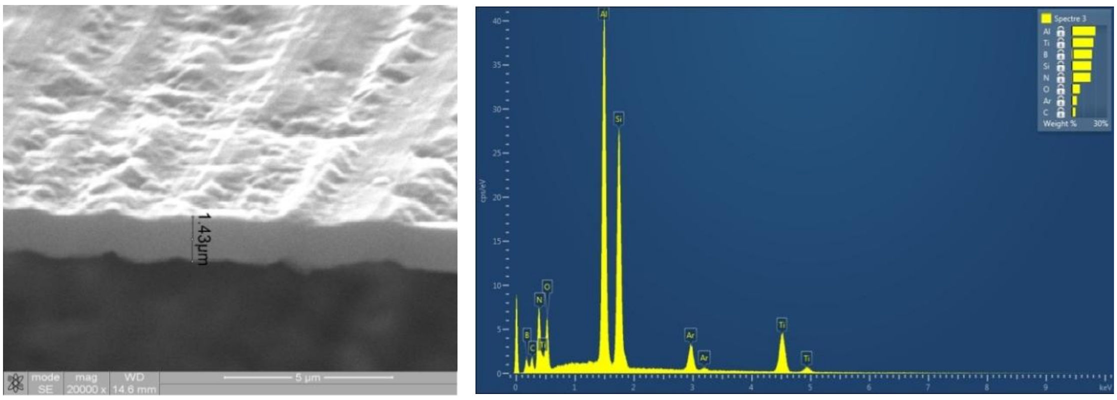


Fig. 2 – Profile of AFM image of AlN-TiB_2-$TiSi_2$ coating (a) and statistical analysis of heights distribution (b)



(a) (b)

Fig. 1 – Surface topography of AlN-TiB_2-$TiSi_2$ coating: a) fracture fractography; b) the EDS data.

In our work, the influence of energy particles, which are the part of multicomponent coating, on evolution of surface topography formation is characterized by processes of phase composition changing of near-surface layers, recrystallization and appearing of areas with reduced stresses.

Fig. 3 shows two diffraction spectra of substrate with coating (1) and substrate without coating (2), and also the final spectrum obtained by subtraction from first spectrum the second spectrum, which corresponds to diffraction spectrum of coating.

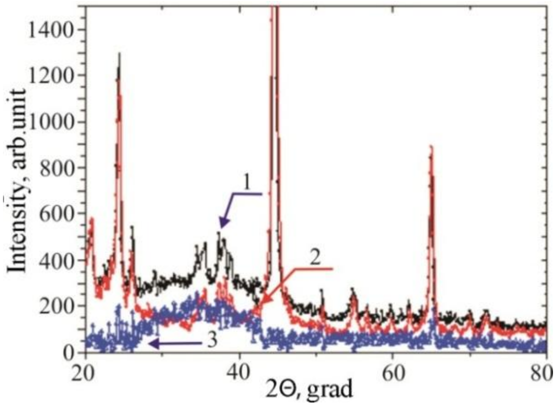


Fig. 3 – Diffraction spectra taken for the coatings of AlN-TiB_2-$TiSi_2$ system: 1-«coating- steel substrate»; 2-«steel substrate»; 3 – resultant spectrum.

After obtaining the resultant diffraction spectrum It was identified that the spectrum has no sharp diffraction maximum from the crystalline component, and also it has «halo-like» character.

The halo-like resultant curve (fig. 4), arrows indicate the maximums of short-range ordering) indicates strong disordering of coating crystallites that corresponds to dimensions of the ordered area about 2 nm with the defined correlation length of the ordering 0.259 nm according to position of first maximum.

The hardness of as-deposited coatings was 15 GPa, but after the annealing under 1350°C the value of hardness was increased up to (22 ÷ 23.5) GPa (fig. 5). The elastic modulus of as-deposited coating was equal to E = 206 GPa and almost it was not changed and equals to E = 218 ± 6 GPa. The value of the viscoplastic index was 0.07.

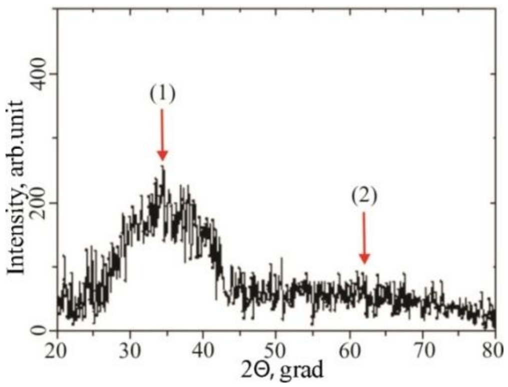
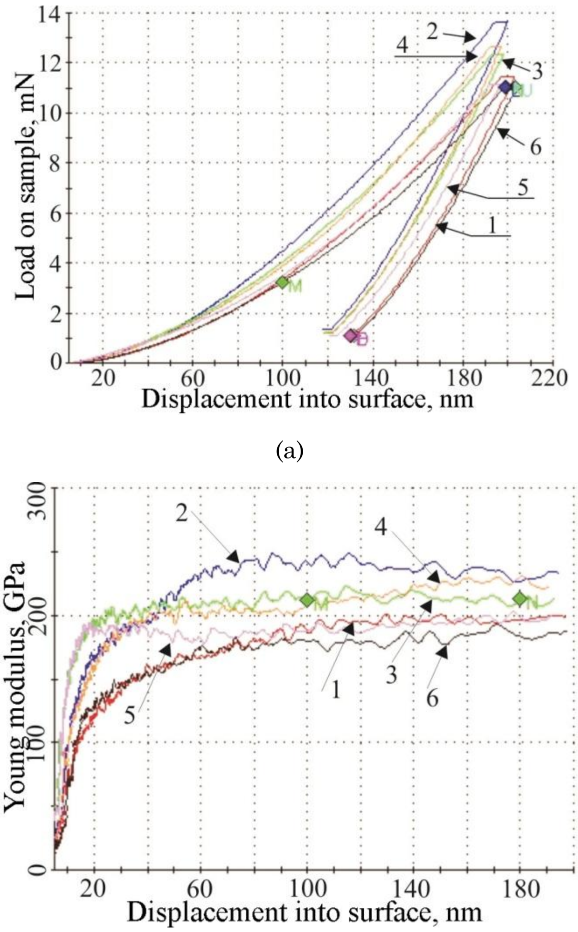


Fig. 4 – The resultant diffraction pattern for AlN-TiB_2-$TiSi_2$ coating.



(a)

(b)

Fig. 5 – Load on sample vs displacement into surface (a) and Young modulus vs displacement into surface (b).

4. CONCLUSIONS

It was established that after deposition of ion-plasma coatings by magnetron sputtering of the AlN-TiB_2-$TiSi_2$ target, amorphous-like structure was formed. The high dispersity is explained by high concentration of high amorphizing element – bor.

The statistical analysis showed that the most of asperities had peaks in the range 15-20 nm. The lateral dimensions of asperities at the bottom were ~ 200 nm, and the width of asperities at the half of height were approximately 70 nm. The roughness of the surface was enhanced after high temperature annealing.

The value of harness has been enhanced from H = 15.3 GPa to H = (22-23.5) GPa after annealing under 1350°C.

It is provided high damping properties of AlN-TiB_2-$TiSi_2$ coatings, and amorphous-like structure makes promising the using of this coatings either like diffusion barrier or like self-contained element or like contacting layer in the multilayer wear-resistant coatings.

REFERENCES

1. A. Cavaleiro, J.Th.M. De *Hosson Nanostructure Science and Technology. Nanostructured Coatings*. (New York: Springer Science+Business Media: 2011).
2. A.D. Pogrebnjak, A.G. Ponomarev, A.P. Shpak, Yu.A. Kunitskii, *Phys. Usp.* **55**, 270 (2012).
3. A.D. Pogrebnjak, A.P. Shpak, N.A. Azarenkov, V.M. Beresnev, *Phys. Usp.* **52**, 29 (2009).
4. N.A. Azarenkov, O.V. Sobol, A.D. Pogrebnjak, V.M. Beresnev, S.V. Lytovchenko, O.N. Ivanov, *Materialovedenie neravnovesnogo sostoyania modificirovanoy poverhnosti* (Sumy: Sumy State University: 2012) [in Russian].
5. A.D. Pogrebnjak, V.M. Beresnev in "*Nanocomposites-New Trends and Developments*" (Ed. F. Ebrahimi) (Rijeka: InTech: 2012).
6. J. Musil, *Surf. Coat. Tehnol.* **125**, 322 (2000).
7. A.D. Pogrebnjak, A.P. Shpak, V.M. Beresnev, D.A. Kolesnikov, Yu.A. Kunitsky et al., *J. Nanosci. and Nanotechno.* **12** No 12, 9213 (2012).
8. A.V. Khomenko, *Phys. Lett. A* **329**, Iss.1–2, 140 (2004).
9. A.V. Larkin, A.K. Fedotov, J.A. Fedotova, T.N. Koltunowicz, P. Zhukowski, *Mater. Sci.-Poland* **30**, Iss. 2, 75 (2012).
10. A.D. Pogrebnjak, V.M. Beresnev, M.V. Il'yashenko, D.A. Kolesnikov, A.P. Shypylenko, et al., *Ceram. Eng. Sci. Proc.* **32**, 69 (2011).
11. A.D. Pogrebnjak, V.M. Beresnev, A.A. Demianenko, V.S. Baidak, F.F. Komarov, et al., *Phys. Solid State* **54**, 1882 (2012).