

# Optimization of aluminum and its alloys doping by ionic-beam-plasma coating

M Rygina<sup>1</sup>, O Krisina<sup>2</sup>, Yu Ivanov<sup>1,2</sup>, E Petrikova<sup>2</sup>, A Teresov<sup>2</sup>

<sup>1</sup>Tomsk Polytechnic University, 30, Lenina ave., Tomsk, 634050, Russia

<sup>2</sup>Institute of High Current Electronics, Siberian Branch of the Russian Academy of Sciences, 2/4, Akademicheskoye., Tomsk, 634055, Russia

E-mail: L-7755me@mail.ru

**Abstract.** The surface morphology, chemical composition, microstructure, nanohardness, and tribological properties of systems were investigated. The paper considers the methodology of film application using ionic-beam irradiation by means of the installation 'Solo' with different exposure modes. Irradiation modes which allow an increase in the microhardness of the material and a decrease in its wear rate are defined. Physical substantiation of this phenomenon is given.

## 1. Introduction

Alloying is the best way to improve characteristics of metals. However, the alloying process demands a large amount of dopant, but the application of plasma methods allows reducing this amount. In this method heating and cooling rates are sufficiently high for a rapid growth of a crystal. In addition to it, there is a method of ion implantation allowing one to introduce alloying atoms directly into the surface layers of solids [1]. Silicon and titanium are the main alloying elements. Silicon is one of the main additives in creation of cast alloys (silumin). Silumin usually contain from 5 to 14% of Si, i.e. a few percent higher or lower than the eutectic concentration Sr and Sb [2], V and Mo [3], W [4] are normally used as additives. The addition of these elements influences beneficially on the structure and mechanical properties of alloys.

We have revealed the presence of intermetallic compounds of aluminum and titanium (TiAl, Ti<sub>3</sub>Al, Al<sub>3</sub>Ti) in the alloys. The main disadvantage of these compounds is low plasticity and impact strength. This drawback can be reduced owing to a decrease of the grain sizes and a creation of a columnar structure.

The aim of our study is to choose an optimal mode of sample influence, to analyze the structures and strength properties of commercially pure aluminum of the A7 grade and silumin of the AK12 grade alloyed with silumin (25 wt. %) and titanium.

## 2. Materials

Samples of commercially pure aluminum of the A7 grade, silumin of the AK12 grade, and silumin (25 wt. % Si) were used as a material for study. Samples were subjected to electronic-ionic-plasma treatment on the 'SOLO' installation. At the first stage samples were evaporated with a thin silumin film ( $h = 0.5 \mu\text{m}$  and  $h = 1.0 \mu\text{m}$ ) by the method of sputtering of the silumin AK25 sample. After it, samples were irradiated by a high-intensity pulsed electron beam at different irradiation modes.

Another system is represented by the Ti film / Al substrate system, the thickness of the Ti film being 0.5  $\mu\text{m}$ . Thin (0.5  $\mu\text{m}$ ) Ti films were synthesized on a special vacuum installation 'TRIO' in the low-pressure arc plasma [4]. The film/substrate system was modified by the high-intense pulsed electron beam on the SOLO installation [5]. The electron beam energy density was 10 J/cm<sup>2</sup> and 15 J/cm<sup>2</sup>; the pulse repetition frequency was 0.3 Hz and a number of radiation pulses were varied in the range of 3...30.

The samples were characterized by microhardness (a PMT-3 device, the indenter load is 0.1N). The study of the wear rate of the film/substrate system was conducted in the disc-pin geometry by means of a



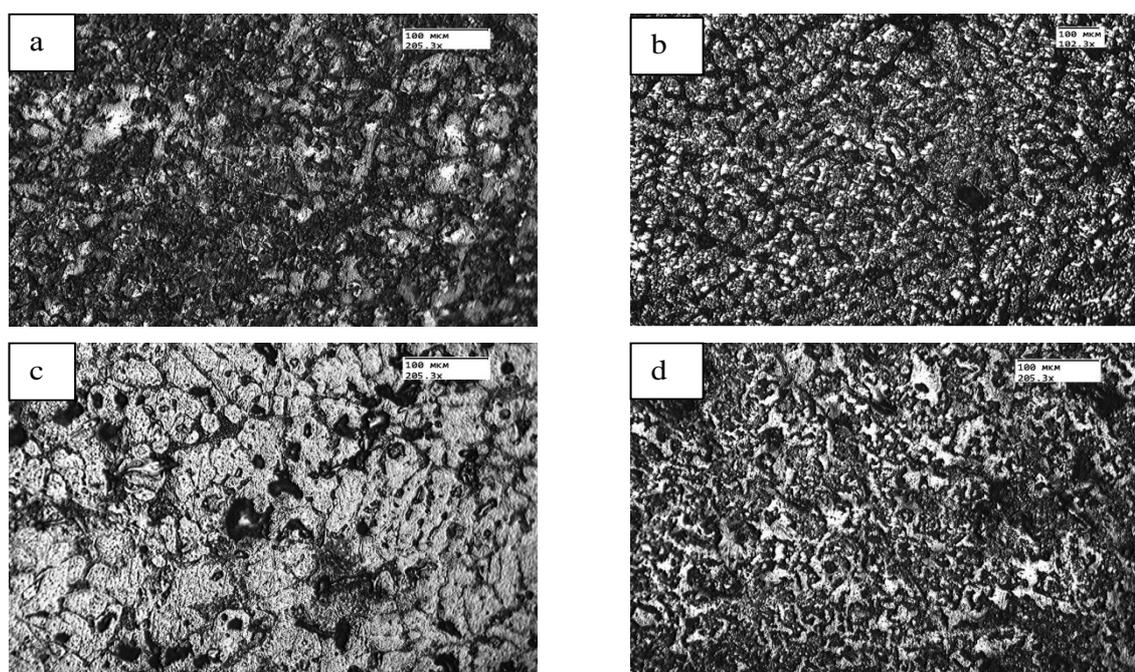
CSEM tribometer, Switzerland, at room temperature and air humidity. The wear volume of the material was estimated after conducting a profilometry of the formed track using a laser range optical profilometer 'MicroMeasure 3D Station' (Stil, France). The elemental and phase compositions of the modified surface were analyzed by means of an optical microscope (NEOFOT-32) and a scanning electron microscope (Philips SEM-515).

### 3. Research Results and Discussion

Let us analyze the results for the film (Al-25wt.% Si) / substrate (Al-12wt.% Si) system presented in Figure 1. The silicon inclusions in the thin near-surface layer have a globular shape, if the film thickness is 0.5  $\mu\text{m}$  (Figure 1a, b), and an island shape, if the film thickness is 1.0  $\mu\text{m}$  (Figure 1c, d).

**Table 1.** Dependence of the hardness on the mode exposure.

Processing mode	Hardness
Silumin (12 wt.%)	125 kg/mm <sup>2</sup>
$E_s = 15 \text{ J/cm}^2$ , $h = 0.5 \mu\text{m}$	84 kg/mm <sup>2</sup>
$E_s = 20 \text{ J/cm}^2$ , $h = 0.5 \mu\text{m}$	104 kg/mm <sup>2</sup>
$E_s = 15 \text{ J/cm}^2$ , $h = 1 \mu\text{m}$	88.7 kg/mm <sup>2</sup>
$E_s = 20 \text{ J/cm}^2$ , $h = 1 \mu\text{m}$	165 kg/mm <sup>2</sup>



**Figure 1.** The surface structure of samples of the film (Al-25wt.% Si) / substrate (Al-12wt.% Si) system irradiated by an electron beam pulse; a)  $E_s = 15 \text{ J/cm}^2$ ,  $h = 0.5 \mu\text{m}$ ; b)  $E_s = 20 \text{ J/cm}^2$ ,  $h = 0.5 \mu\text{m}$ ; c)  $E_s = 15 \text{ J/cm}^2$ ,  $h = 1 \mu\text{m}$ ; d)  $E_s = 20 \text{ J/cm}^2$ ,  $h = 1 \mu\text{m}$

**Table 2.** The wear resistance and friction coefficient of the surface layer of silumin (12 wt. %) modified by sputtered silumin and subsequent processing by the electron beam.

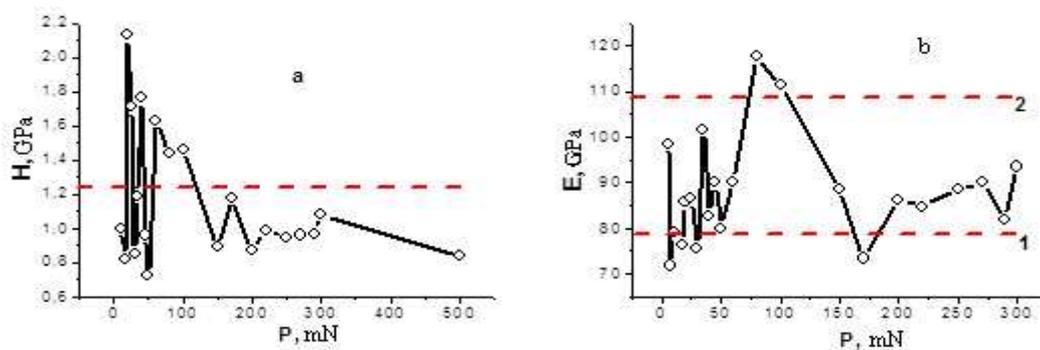
Mode	$V, 10^{-6}$	$V_0/V$	$\langle \mu \rangle$	$\langle \mu_0 \rangle / \langle \mu \rangle$
Silumin (12wt.%)	24322 mm <sup>3</sup> /(H·m)		0.612	
15 J/cm <sup>2</sup> , 0.5 $\mu\text{m}$ , Al-Si	18630 mm <sup>3</sup> /(H·m)	1.31	0.648	0.94
15 J/cm <sup>2</sup> , 1 $\mu\text{m}$ , Al-Si	22228 mm <sup>3</sup> /(H·m)	1.10	0.632	0.97
20 J/cm <sup>2</sup> , 0.5 $\mu\text{m}$ , Al-Si	19416 mm <sup>3</sup> /(H·m)	1.25	0.633	0.97

20 J/cm<sup>2</sup>, 1 μm, Al-Si21124 mm<sup>3</sup>/(H·m)

1.15

0.628

0.97

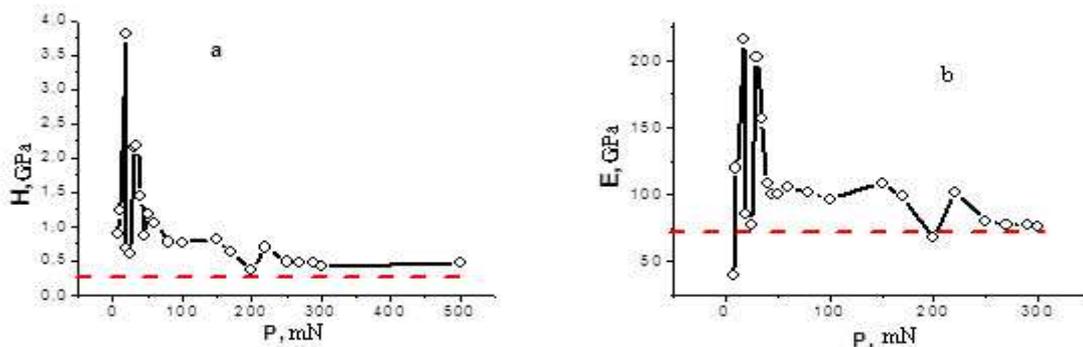


**Figure 2.** Dependence of hardness  $H$  and Young's modulus  $E$  on the indenter load (a nanoindentation method) of the surface layer of the AK12 grade silumin modified by silumin sputtering (the layer thickness is 0.5 μm) and subsequent treatment with an electron beam (in the mode of 15 J/cm<sup>2</sup>, 150 ms, 5 imp., 0.3 Hz). Dash-dotted lines indicate the characteristics of the original AK12 grade silumin (12wt.%). In figure b: 1 –silumin Young's modulus 2 –silicon Young's modulus

The data on microhardness for another film (25 wt.% of silumin)/substrate (Al) system are represented in Table 3.

**Table 3.** Dependence of hardness on the mode exposure.

Processing mode	Hardness
Mode of processing	250 MPa
Pure Al	425 MPa
$E_s = 15 \text{ J/cm}^2, h = 0.5 \mu\text{m}$	370 MPa
$E_s = 20 \text{ J/cm}^2, h = 0.5 \mu\text{m}$	548 MPa
$E_s = 15 \text{ J/cm}^2, h = 1 \mu\text{m}$	470 MPa



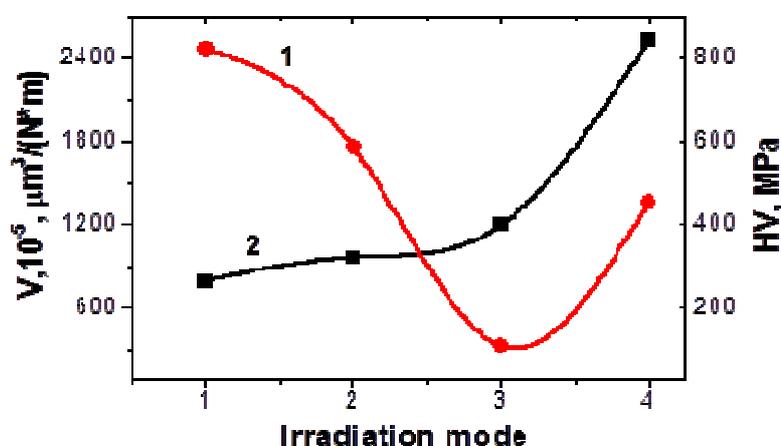
**Figure 3.** Dependence of hardness  $H$  and Young's modulus  $E$  on the indenter load (a nanoindentation method) of the surface layer of aluminum modified by the method of silumin sputtering (layer thickness is 1 μm) and subsequent treatment with an electron beam in the mode of 20 J/cm<sup>2</sup>, 150 ms, 5 imp., 0.3 Hz. Dashed-dotted lines indicate the characteristics of the original A7 grade aluminum.

The wear resistance and friction coefficients of the surface of samples are given in Table 4. It has been established that high wear resistance belongs to sample treated in the mode of 20 J/cm<sup>2</sup> and the film thickness of 0.5 μm. The lowest friction coefficient is acquired by the material in the mode of 20 J/cm<sup>2</sup> and the film thickness of 1 μm.

**Table 4.** The wear resistance and friction coefficients of the surface layer of commercially pure A7 grade aluminum modified by siluminsputtering and subsequent processing by the electron beam (150 ms, 5 imp., 0.3 Hz.)

Mode	$V, 10^{-6}, \text{mm}^3/(\text{H}\cdot\text{m})$	$V_0/V$	$\langle\mu\rangle$
Pure Al	7592	-	0.483
15 J/cm <sup>2</sup> , 0.5 μm	6080	1.25	0.385
15 J/cm <sup>2</sup> , 1 μm	6566	1.16	0.4
20 J/cm <sup>2</sup> , 0.5 μm	5466	1.39	0.353
20 J/cm <sup>2</sup> , 1 μm	4790	1,59	0.311

The research results of the hardness of the thirdTi film/Al substrate system have shown that hardness increases more than three times during the electron beam irradiation under the mode of 15 J/cm<sup>2</sup>, 30 pulses, 50 μs, and 0.3 Hz (Figure 4, curve 2).



**Figure 4.** The wear rate (curve 1) and the surface microhardness (curve 2) of the film (Ti)/substrate (Al) system after different modes of treatment: 1 – substrate (commercially pure A7 aluminum); 2...4 the film/substrate systems irradiated with the electron beam with parameters of 10 J/cm<sup>2</sup>, 50 μs, 10 pulses (lower index 2); with parameters of 15 J/cm<sup>2</sup>, 50 μs, 3 pulses (lower index 3); and with parameters of 15 J/cm<sup>2</sup>, 50 μs, 30 pulses (lower index 4)

The best resulting properties are attained during the electron beam irradiation of the film/substrate system with parameters of 15 J/cm<sup>2</sup>, 3 pulses, 50 μs, and 0.3 Hz (Figure 4, curve 1); the wear rate of the modified layer is more than 7.5 times lower compared to the substrate.

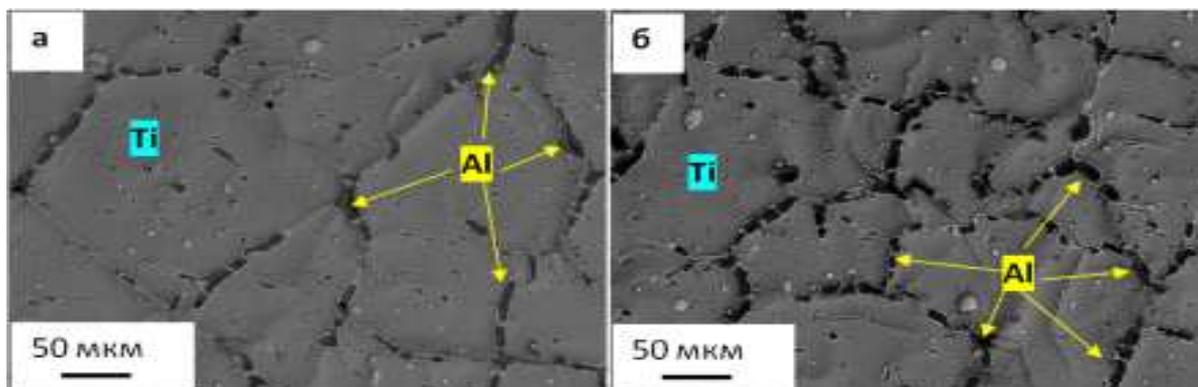
The surface alloy, while displaying high hardness, reveals a decline in wear resistance, which is apparently due to structural features of the formed film/substrate system. This is due to the appearance of intermetallic compounds in the structure.

Intermetallic compounds of aluminum and titanium (TiAl, Ti<sub>3</sub>Al, Al<sub>3</sub>Ti) feature a unique combination of properties: high values of specific strength, heat resistance, thermal stability, elastic modulus, creep resistance, and anomalous temperature dependence on the mechanical properties [1].

Typical images of the surface structure of the film / substrate system irradiated with the intense electron beam are shown in Figure 5. When irradiating with the electron beam with the energy density of  $E_s = 10 \text{ J/cm}^2$  (10 imp.) and  $15 \text{ J/cm}^2$  (3 imp.), the Ti film deposited on the aluminum surface is preserved. However, at the same time, the Ti film is fragmented with micro-cracks (Figure 5a, b). The sizes of fragments under  $E_s = 15 \text{ J/cm}^2$  are 3...4 times less than those in the sample irradiated under  $E_s = 10 \text{ J/cm}^2$ . A microcracks volume with cross-sectional dimensions of ~10 microns is filled with the melt of aluminum.

The crystal lattice parameter of aluminum, when  $E_s = 10 \text{ J/cm}^2$ , is  $a = (0.4041 \pm 0.0001) \text{ nm}$ , which corresponds to the lattice parameter of pure aluminum. In addition to the diffraction maximum of aluminum and titanium, the X-ray patterns show the diffraction maximums of the Al<sub>3</sub>Ti phase. The volume

fraction of the intermetallic is ~20 %, titanium – 28 %. Irradiation under  $E_S = 15 \text{ J/cm}^2$  (3 imp.) leads to an increase in the lattice parameter of aluminum up to  $a = (0.4050 \pm 0.0001) \text{ nm}$ , which indicates the formation of alloy Al-Ti.



**Figure 5.** The surface structure of the film/substrate system irradiated with an electron beam under: *a*–  $10 \text{ J/cm}^2$ , 10 imp., *b*–  $15 \text{ J/cm}^2$ , 3 imp.

The volume fraction of the Al<sub>3</sub>Ti intermetallic compound reduces to ~3 %, the volume fraction of titanium remains virtually constant (~30 %).

#### 4. Conclusion

The maximum microhardness is achieved by means of influence on the surface of the silumin (25 wt. %) film/substrate (Al) system and the energy density of the electron beam of  $E_S = 15 \text{ J/cm}^2$ . An increase of the energy density up to  $20 \text{ J/cm}^2$  (regardless of the deposited film thickness) leads to a decrease of the microhardness of the irradiated surface. A percentage of silicon in the surface changes in a similar way. The hardness of the modified layer depends on the percentage of silicon in it. A better wear resistance was shown by a sample irradiated with an electron beam under  $E_S = 20 \text{ J/cm}^2$ ,  $h = 0.5 \text{ μm}$ . The minimum friction coefficient was detected in the sample under  $E_S = 20 \text{ J/cm}^2$ ,  $h = 1 \text{ μm}$ .

The maximum microhardness is identified in the silumin (25 wt. %) film / silumin (12 wt. %) substrate system irradiated by the electron beam with the energy density of  $E_S = 20 \text{ J/cm}^2$ . When using a substrate, wear resistance reduces irrespective of the irradiation modes and the layer thickness for 12 % silumin.

The Ti film/Al substrate system subjected to high-energy impact under  $15 \text{ J/cm}^2$ , 3 pulses,  $50 \text{ μs}$ ,  $0.3 \text{ Hz}$  demonstrates a 7.5 time increase in the wear rate. The irradiation under  $15 \text{ J/cm}^2$ , 30 pulses,  $50 \text{ μs}$ ,  $0.3 \text{ Hz}$  provides a more than 3 time increase of the system microhardness of the material compared to the substrate.

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#### References

- [1] Mashkov Yu, Poleschenko KN, Povoroznyuk C H and Orlov PV 2000 *Friction and modification of materials tribosystems* (M: Nauka) p 280
- [2] Szymczak T, Gumienny G and Pacyniak T 2015 *Archives of Foundry Engineering* **15**(1) 105–108
- [3] Szymczak T, Gumienny G and Pacyniak T 2015 *Archives of Foundry Engineering* **15**(4) 81–86
- [4] Szymczak T, Gumienny G, Pacyniak T and Walas K 2015 *Archives of Foundry Engineering* **15**(3) 61–66
- [5] Grinberg BA and Ivanov MA 2002 *Intermetallic Ni<sub>3</sub>Al and TiAl compounds: microstructure and deformation behavior* (UrB RAS: Ekaterinburg)