Multi-cycle alloying of silumin surface layer with titanium: structure, properties

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Abstract. It is suggested a combined method of silumin surface treatment, including deposition of a metal film and the subsequent irradiation by an intensive pulsed electron beam in the mode of surface layer melting. The formation of the multilayered multiphase surface volume having submicro- and nanocrystalline structure is revealed. It is shown that the sizes of crystallites of the modified layer rise at increase of the distance from the treated surface.

1. Introduction

At present aluminum-silicon alloys (silumin) with silicon concentration in the range of 4-22 wt. % remain the leader in many industries (car manufacturing, aircraft construction, the river and marine transport, civil engineering and medicine, etc.). Generally, it is connected with good foundry properties, low weight, high specific strength, wear resistance and corrosion resistance. Despite it, continuously increasing requirements to the equipment demand the corresponding improvement of quality of device and mechanism details. The last is directly connected with formed phase and structural state of material which is characterized by the chemical composition and conditions of thermal / thermomechanical treatment of material [1, 2].

Usually the modification of a structural and phase state for working surface layer, but not for the bulk material of a detail or product is economic and technically reasonable [3]. One of perspective ways of realization of such approach is the use of the concentrated energy flows (continuous and pulsed electron beams, powerful ion beams, plasma flows, continuous and pulsed laser beams, etc.) [4–7]. The equipment based on generation of low-energy high-intensity pulsed electron beams with micro- and submillisecond duration are developed and actively used in Institute of high current electronics SB RAS (IHCE SB RAS) within the solution of materials science problems [8]. At interaction of an intensive pulsed electron beam with solid state (1) the high-speed heating of a surface layer of material leading to its melting and evaporation; (2) the course of hydrodynamic processes in the melted layer, which promote the active mixture of chemical elements of material; (3) the superfast cooling of surface layer due to heat removal in the bulk of the treated material, which leads to repeated refinement of structure (up to a nanodimensional state), take place. That positively affects various properties of surface layer and product in general [3].

It is obvious that the complex treatment has more essential effect on the structure and the properties of material. It includes the irradiation of the material surface by an intensive pulsed electron beam, the deposition of metal films and hard ceramic coatings, the formation of the layers enriched with atoms of gas elements in a certain sequence. These opportunities of metals and alloys treatment are realized
on the COMPLEX installation (development of IHCE SB RAS). It allows to carry out the initial heating of specimens to the required temperature, cleaning and activation of the treated surface in argon plasma; arc plasma-assisted deposition of metal (with the use of argon plasma) films or ceramic hard and superhard coatings; electron-beam treatment by an intensive pulsed electron beam; saturation of a surface layer of modified material by gas elements (nitrogen, carbon, oxygen) in an uniform vacuum volume [9].

The purpose of the present work is the approbation of a complex method for treatment of a silumin surface which combines the deposition of a metal film and the subsequent irradiation by an intensive pulsed electron beam in the mode of melting of surface layer.

2. Material and methods

The Al-Si alloy (Al-12%Si: Russian AK12 grade) was chosen for researches. It belongs to eutectic silumin and has the following chemical composition (wt. %): Al - (10-13) Si - to 1.5 Fe - to 0.5Mn - to 0.1Ti - to 0.6Cu - to 0.1Zr - to 0.1Mg – to 0.3Zn [10]. The specimens for researches had the form of plates with the sizes of 20×20×8 mm³. Before modification (further in the text it will be "an initial state") the alloy was in cast state (gravity die casting). The experiments were carried on the COMPLEX installation developed and created in IHCE SB RAS [9]. Silumin surface layer allying was carried out with the titanium. The thickness of titanium film in each cycle of "deposition/irradiation" was 0.5 μm. Quantity of cycles was 10.

The research of silumin structure in an initial state and after modifying was carried out by the methods of the X-ray diffraction analysis (XRD 6000 diffractometer), scanning (SEM 515 Philips device) and transmission diffraction (JEM-2100F device) electron microscopies. The research objects for the transmission electron microscope were made by the methods of ion thinning of the plates which are cut out from a massive sample perpendicularly to the processed surface. Elemental composition of specimens was determined by methods of the micro X-ray analysis. The properties of the modified layer characterized by wear resistance (TRIBOtechnic device; the condition of dry friction at the room temperature; the counter body is a ball of ShH15 with a diameter of 6 mm, radius of a wear track is 2 mm, the linear speed of 2.5 cm/s, load on the indenter of 5 N, laps quantity of 8000). The wear resistance of the material surface layer was calculated after profilometry test with the formed track.

3. Results and discussion

The structure of Al-12%Si alloy in a cast state is presented by eutectic grains, the grains of solid solution based on aluminum, the inclusions of intermetallic compound and silicon which has the lamellar form and a form of "hieroglyphs" (figure 1, a).

![Figure 1. The structure of Al-12%Si surface in a cast state (a) and after multi-cycle treatment (b, c).](image-url)
The multi-cycle complex treatment of the silumin surface combines formation of the "Ti film / Al-12%Si substrate" system the subsequent irradiation by an intensive pulsed electron beam in a single vacuum cycle. Electron-beam treatment is followed by the dissolution of silicon and intermetallic inclusions (figure 1, b) and the formation of structure with crystallite sizes in the range of 0.4-0.7 μm (figure 1, c).

Studies of silumin using transmission electron microscopy revealed the formation of a multilayer structure during multi-cycle processing. After ten cycles of processing such layers, nine were identified. The layers are visually distinguished by the size of the crystallites forming them (figure 2).

![Figure 2](image_url) Figure 2. The bright field image (a) and the image obtained in the characteristic X-ray radiation of titanium atoms (b) for silumin surface layer structure after multi-cycle treatment. Transmission electron microscopy.

The electron-microscopic microdiffraction analysis with the use of dark-field technique allowed to carry out a research of phase composition of the modified layer of silumin. From the results of surface layer analysis presented in figure 3 it was revealed the presence of aluminides of titanium with Al$_3$Ti structure (figure 3, c) and silicides of titanium with TiSi structure (figure 3, d) along with aluminum and silicon. Aluminides and silicides of the titanium form the volumes with various form divided by aluminum layers (figure 3, a). The sizes of such volumes change within 0.8-1.2 μm. The sizes of Al$_3$Ti particles are 40-60 nm; that of TiSi particles – 20-30 nm. The particles of silicon have the sizes of 10-20 nm and they are chaotically located in volume of surface layer (figure 3, d).

The results of the electron microscopic microdiffraction analysis of phase composition of a silumin modified layer, which is located in the depth of ≈20 μm, are given in figure 4. The researches revealed the presence of aluminum, silicon and aluminide of the titanium of Al$_3$Ti system. The results of researches presented in figure 3 and figure 4 show that there is the increase of the phases size at increase of distance from the surface of modification.

Tribological properties of the modified silumin characterized the wear resistance and coefficient of friction. It is shown that the wear resistance of the modified silumin exceeds that of initial material by 11.5 times; the friction coefficient of the modified layer is 1.6 times less than that of initial silumin.

4. Conclusions
The complex method for treatment of silumin surface, which combines in a single cycle formation of the "titanium film / Al-12%Si substrate" system and the subsequent irradiation by an intensive pulsed electron beam in the mode of surface layer melting, is suggested. The dissolution of primary
inclusions of silicon and intermetallic compound is revealed in the modified layer. The formation of the multilayered multiphase surface volume with submicro- and nanocrystalline structure is revealed. It is shown that the sizes of crystallites of the modified layer rise at the increase of the distance from treated surface. By the methods of the transmission electron diffraction microscopy it is established that modification of silumin surface layer is followed by repeated allocation of silicon particles, formation of particles of titanium aluminides and silicides with the nanocrystalline sizes. As a result of tribological tests it is revealed that the wear resistance of the modified silumin surface layer exceeds that of initial material by 11.5 times; the friction coefficient of the modified layer is 1.6 times less than that of initial silumin.

Developed complex method can be used for surface alloying of metals and alloys with the purpose of the increase the strength and tribological properties of the materials.

![Electron microscopic image of surface layer structure for Al-12%Si after multicycle treatment](image)

**Figure 3.** The electron microscopic image of surface layer structure for Al-12%Si after multicycle treatment: a – the bright field; b – the microdiffraction pattern; c–d – the dark fields obtained in reflexes of [200] Al,Ti (c), [122] TiSi (d), [101] Si (e). Arrows show the reflexes which correspond the dark fields images: 1 – (c); 2 – (d); 3 – (e).
Figure 4. The electron microscopic image of surface layer structure for Al-12%Si after multi-cycle treatment: a – the bright field; b – the microdiffraction pattern; c-d – the dark fields obtained in reflexes of [220]Si (c), [112]Al,Ti (d), [111]Al (e), [101]Si (f). Arrows show the reflexes which correspond the dark fields images: 1 – (c); 2 – (d); 3 – (e); 4 – (f). The analyzed layer is located on the distance of 20 μm from surface of modification.
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References
[1] Zolotarevskiy V S and Belov N A 2005 Physical metallurgy of cast aluminum alloys (Moscow: MISiS)
[2] Piston silumins: Training manual 2005 ed V K Afanasieva (Kemerovo: Poligraf)
[3] Rotshtein V, Ivanov Yu and Markov A 2006 Surface treatment of materials with low-energy, high-current electron beams Materials surface processing by directed energy techniques ed Y. Pauleau (Paris: Elsevier) chapter 6 pp 205–40
[4] Koval N N and Ivanov Yu F 2008 Izvestija Vuzov. Fizika 5 60–70
[5] Laskovnev A P, Ivanov Yu F, Petrikova E A et al. 2013 Modification of structure and properties of eutectic silumin by electron-ion-plasma treatment (Minsk: «Belarusskaja nauka»)
[6] Kadyrjanov K K, Komarov F F and Pogrebnjak A D 2005 Ion-beam and ion-plasma modification of materials (Moscow: Publishing house of MSU)
[7] Gribkov V A, Grigoriev F I and Kalin B A 2001 Perspective radiation-beam technology for material treatment (Moscow: Krugliy stol)
[8] Electron-ion-plasma modification of surface of non-ferrous metals and alloys 2016 ed N N Koval and Yu F Ivanov (Tomsk: NTL publishing house)
[9] Technology based on solid scientific research in the RSF project. Sibir 2017 ed S G Psahie, Yu P Sharkeev (Tomsk: NTL publishing house)
[10] Aluminum foundry alloys. Technical specifications. State Standard 1583-93 2000 (Minsk: Interstate Committee for Standardization, Metrology and Certification)