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МОДИФИКАЦИЯ СОСТАВА, СТРУКТУРЫ И МЕХАНИЧЕСКИХ СВОЙСТВ ПОВЕРХНОСТНОГО СЛОЯ СИСТЕМЫ (Ti, Cu)N/Al – 12 ат. % Si, ОБРАБОТАННОЙ КОМПРЕССИОННЫМИ ПЛАЗМЕННЫМИ ПОТОКАМИ

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Исследованы изменения состава, структуры и свойств поверхностного слоя системы (Ti, Cu)N/Al – 12 ат. % Si, обработанной компрессионными плазменными потоками. Использованы следующие методы исследования: растровая электронная микроскопия, энергодисперсионный микроанализ, рентгеноструктурный анализ, измерение микротвердости и коэффициента трения. В результате роста плотности энергии, поглощенной поверхностью образцов при плазменном воздействии, в диапазоне 26–40 Дж/см^2 наблюдаются увеличение степени растворения материала покрытия в поверхностном слое алюминиевого сплава и уменьшение в нем концентрации титана и меди. Снижение концентрации легирующих элементов связано с ростом толщины расплавленного слоя и интенсивности эрозии при плазменном воздействии. В диапазоне плотности поглощенной энергии 35–40 Дж/см^2 установлено формирование гомогенного поверхностного слоя толщиной 25–45 мкм с дисперсной структурой, состоящего из элементов подложки и покрытия и содержащего пересыщенный твердый раствор на основе алюминия и поверхностную пленку нитрида алюминия. Воздействие плазмы приводит к увеличению микротвердости поверхности на 25 % по сравнению с исходным сплавом. Наименьшее значение коэффициента трения наблюдается у образца, обработанного при плотности поглощенной энергии 30 Дж/см^2.

Ключевые слова: эвтектический силумин; нитридное покрытие; компрессионные плазменные потоки; сканирующая электронная микроскопия; рентгеноструктурный анализ; микротвердость; шероховатость; коэффициент трения.

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MODIFICATION OF COMPOSITION, STRUCTURE AND MECHANICAL PROPERTIES OF THE SURFACE LAYER OF (Ti, Cu)N/Al – 12 at. % Si ALLOY SYSTEM TREATED BY COMPRESSION PLASMA FLOWS

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Changes in composition, structure and properties of the surface layer of (Ti, Cu)N/Al – 12 at. % Si alloy treated by compression plasma flows were investigated. Scanning electron microscopy, energy dispersive analysis, X-ray diffraction, microhardness and friction coefficient measurements were used as investigation techniques. Increase of the energy density absorbed by the samples surface in the region of 26–40 J/cm^2 during plasma impact leads to the growth of coating material dissolution degree in the surface layer of aluminum alloy and diminishing of titanium and copper content in it. Decrease of alloying elements concentration is explained by the growth of melted layer thickness and erosion intensity during plasma impact. The formation of a surface homogeneous layer with dispersed structure and thickness of 25–45 μm, consisting of substrate and coating elements was observed in the region of the absorbed energy density 35–40 J/cm^2. A supersaturated solid solution based on aluminum is formed, as well as a surface film of aluminum nitride. Plasma impact leads to an increase in the microhardness of the surface up to 25 % compared to the initial alloy. A sample processed at an absorbed energy density of 30 J/cm^2 has the lowest friction coefficient.

Keywords: eutectic silumin; nitride coating; compression plasma flows; scanning electron microscopy; X-ray structure analysis; microhardness; roughness; friction coefficient.

Introduction

Silumins – aluminum and silicon alloys, have good casting properties, high corrosion resistance, low specific mass, low coefficient of thermal expansion along with the cheapness and the possibility of using recycling products in their fabrication. These alloys are used in such areas of industry as aviation, rocket and automotive production, manufacturing of household products for mass use. However, silumins have unsatisfactory tribological and strength characteristics, which hinders the use of these materials in appropriate applications [1; 2].

A promising way to improve the properties of the surface layer of materials is exposure to laser, electron, ion and plasma flows, including compression plasma flows (CPF) [1; 3]. Modern magnetoplasma installations allow the formation of plasma flows with a discharge duration of the order of hundreds of microseconds while maintaining high plasma parameters, that leads to the formation of modified layers with a thickness of tens of micrometers and high performance characteristics [1; 4]. Plasma treatment of materials with preliminary deposited coatings allows alloying the surface layer of the material with coating elements, to synthesise hardening phases, to disperse its structure, which, as a rule, lead to an improvement in mechanical and tribological characteristics [1; 4].

The treatment of the coating – substrate system by high-energy particle beams also allows increasing the adhesion of the coating and significantly affecting the tribological properties. Earlier it was shown that the impact of high-current electron beam on the system (Ti, Cu)N – eutectic silumin leads to partial fusion of the hard coating into the silumin surface layer and, as a consequence, providing increase of coating wear resistance and decrease of friction coefficient [1]. Investigation of composition, structure and mechanical properties of (Ti, Cu)N/Al – 12 at. % Si alloy surface layer treated by CPF with different energy absorbed by the surface layer is the objective of this work.

Materials and research methods

Deposition of (Ti, Cu)N coating on silumin alloy substrate (12 wt. % Si, 0.7 wt. % Fe, 0.5 wt. % Mn, 0.6 wt. % Cu, 0.3 wt. % Zn, 0.6 wt. % Ni, 0.6 wt. % Mg; Al – balance) was carried out by vacuum-arc technique using Ti – Cu cathode. The thickness of the coating (Ti, Cu)N was ~1 μm. Concentration of Ti and Cu in the coating was about 49 and 1 at. %, respectively. The samples were processed by three CPF pulses with a duration of 100 μs at the distance 8–14 cm between the sample and the cathode, which led to a change in the energy density absorbed by the surface layer of the sample in the range of 26–40 J/cm^2 per pulse. Nitrogen was used as the plasma generating gas, when the pressure in the vacuum chamber was 400 Pa.

X-ray diffraction analysis was carried out on a diffractometer Ultima IV (*Rigaku*, Japan) using monochromatised copper radiation (λ = 0.154 178 nm; K_{α_1}). The analysis of the surface morphology was performed using a scanning electron microscope LEO-1455VP (*Carl Zeiss*, Germany) having an accelerating voltage of 20 kV. The elemental composition of the samples was determined by energy dispersive X-ray microanalysis using an detector (*Oxford Instruments*, United Kingdom) coupled to a scanning electron microscope (relative error of concentration is more or equal 10 %). Microhardness was measured by the Vickers method on a 402MVD installation (*Wilson Instruments*, United Kingdom) with a load of 0.1 N. The surface roughness was measured using a profilometer MarSurf SD26 (Germany). Tribological tests were carried out on a tribometer TAU-1M at a load of 0.2 N under dry friction conditions, the indenter was made of VK8 alloy.

Results and discussion

The interaction of compression plasma flows with the coating – substrate system is accompanied by melting of the coating and the surface layer of substrate material (when the required surface temperature is reached, provided by the treatment modes), erosion of the surface layer, liquid-phase mixing of the melt as a result of the development of hydrodynamic instabilities and subsequent crystallisation under ultrafast cooling conditions [1; 4]. An increase in the absorbed energy density leads to an increase in the lifetime of the melt and consequently more homogeneous distribution of elements in it due to the increase of convective flows action duration. The change in the energy density absorbed by the surface due to the large difference in the melting temperatures of the coating (Ti, Cu)N and the substrate (Al – Si) allows to choose the treatment regime in which the substrate is mainly melting. In this regime, one should expect the effect described in [1] – fusion of the hard coating into the substrate and improvement of tribological characteristics of the material.

The analysis of the surface morphology (fig. 1, *a* and *b*) showed that the impact of CPF with an absorbed energy density of 26 J/cm^2 on the surface of the samples already leads to partial fusion of the coating into the substrate material. When the absorbed energy density increases to 30 J/cm^2, no coating material is observed on the surface (fig. 1, *c*). One can see that plasma impact leads to the formation of cracks and pores at the surface of the samples (fig. 1, *b* and *c*).

Besides that the findings showed that the surface of the treated samples was covered by a discontinuous nitride film (fig. 2). The formation of nitrides on the surface of materials can be caused by the interaction of atoms in the residual atmosphere of the vacuum chamber with surface atoms at the stage of the surface layer cooling [5].

Energy dispersion microanalysis carried out on the surface of the samples in the layer with a thickness of ~1 μm showed that the concentration of coating elements, in particular Ti, decreases with the growth of absorbed energy density (table 1). An increase in the thickness of the melt and the redistribution of the coating elements into deeper layers leading to a decrease in the average concentration of elements over the whole layer can be one of the reasons of this effect [4].

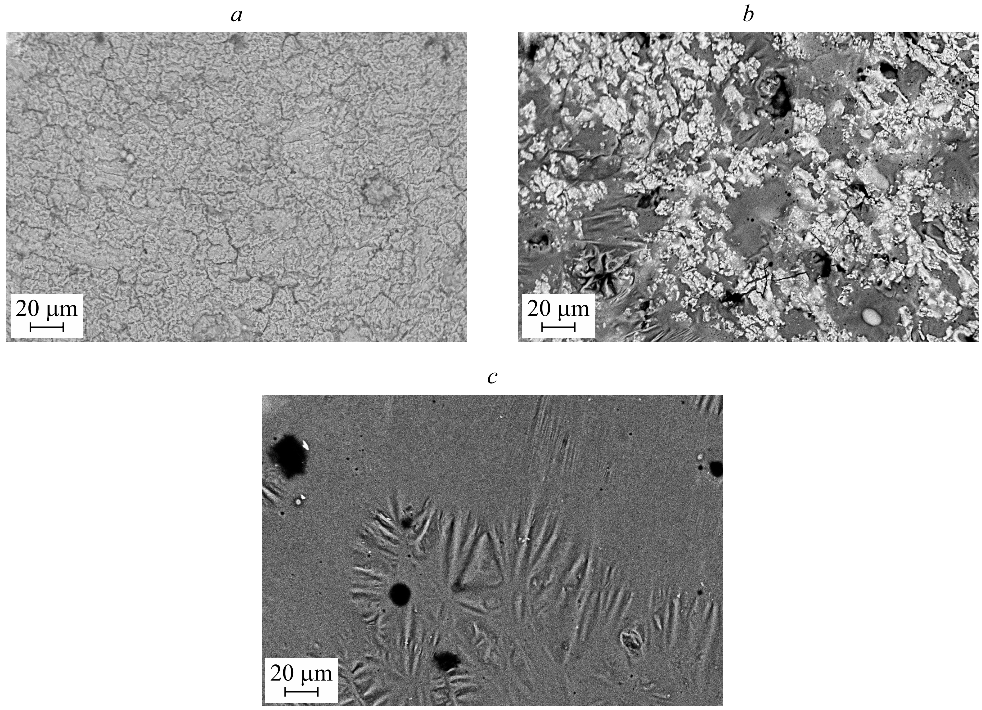
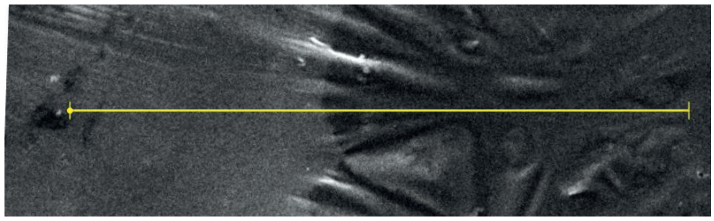


Fig. 1. Morphology of the surface of the initial coated sample (*a*) and samples treated with CPF at 26 J/cm^2 (*b*), 30 J/cm^2 (*c*)

a



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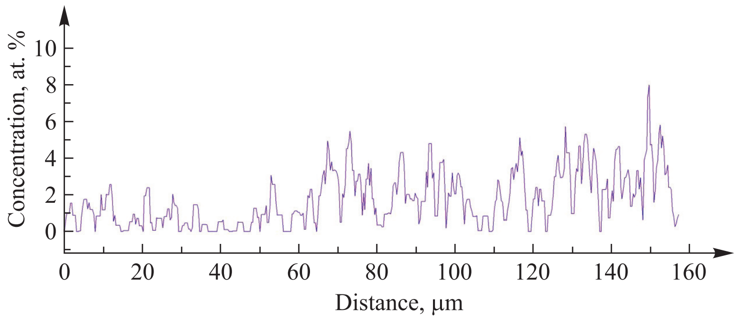

Fig. 2. Surface morphology (*a*) and nitrogen distribution along the line (*b*) for a sample treated with CPF at 30 J/cm^2

Table 1

Dependence of the titanium concentration in the surface layer on the absorbed energy density

Density of energy absorbed by surface layer, J/cm^2	26	30	35	40
Titanium concentration in the analysed layer, at. %	8.2	1.2	1.0	0.1

Surface erosion during plasma impact also contributes to a decrease in the coating elements concentration in the surface layer. It was found that the mass removed from the surface of the samples as a result of plasma treatment was changed in the range of 1.4–172.0 μg/mm^2 depending on the absorbed energy density in the range 26–40 J/cm^2. Plasma impact with samples surface leads to melting of the surface layer. Traces of the melt are observed on the lateral surface of the samples, which suggests that the main mechanism of erosion is the hydrodynamic motion of the melt from the center to the edges of the sample. The plasma stream interacts with the target and spreads over the surface in radial directions that provides the melt ejection out of the sample [6–8]. The plasma flow also causes the development of various hydrodynamic instabilities at the plasma-melt boundary and the formation of droplets, which can be ejected from the melt [9]. Evaporation and boiling of the material can also be causes of mass loss [6; 7].

Concentration of silumin alloying elements (Cu, Ni, Mg, Fe) increases at the surface with the growth of absorbed energy density (table 2). Such behaviour is related to the melting of alloy intermetallides at the surface layer with a thickness of several tens of micrometers as well as the redistribution doped elements in alloy over the entire thickness of the melt leading to an increase in the average concentration of elements in the layer analysed by the energy dispersion detector. Some increase of copper concentration could be associated with fragmentation of (Ti, Cu)N coating in the melted surface layer during plasma treatment.

Table 2

The concentration of elements in Al – 12 at. % Si sample and (Ti, Cu)N/Al – 12 at. % Si sample after CPF treatment at 30 J/cm^2, at. %

Sample	Chemical element			
	Cu	Ni	Mg	Fe
Al – 12 at. % Si alloy sample	0.3	0.3	0.5	0.2
(Ti, Cu)N/Al – 12 at. % Si sample treated by CPF	0.7	0.5	0.6	0.3

Analysis of the cross-section of the Al – 12 at. % Si alloy initial sample showed the presence of aluminum grains, Al – Si eutectic, intermetallides formed by alloying elements and primary silicon crystals with sizes from units to tens of micrometers. During coating deposition part of the titanium atoms diffused 3–4 μm deep into the substrate due to the heating of the sample surface by accelerated titanium ions (fig. 3).

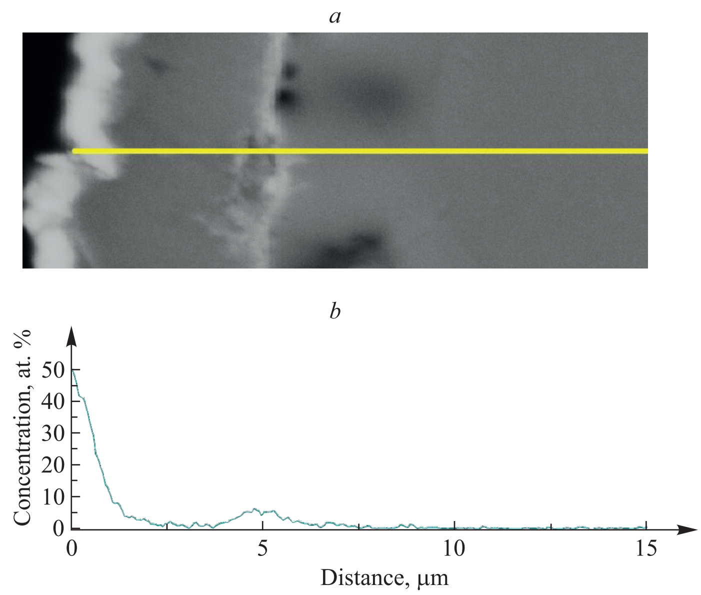

Fig. 3. Cross-section morphology (*a*) and the distribution of titanium (*b*) along the depth of the (Ti, Cu)N/Al – 12 at. % Si alloy sample

After CPF impact on (Ti, Cu)N/Al – 12 at. % Si alloy system the formation of a mixed layer is observed (fig. 4, *a* and *b*), the thickness of which depends on the density of the absorbed energy. The thickness of a mixed layer was 19 and 45 μm after CPF treatment at 26 and 40 J/cm^2, respectively. Mixed layer has dispersed cellular structure, which is caused by high cooling rates of the melt. The bulk of the mixed layer contains separate inclusions of undissolved primary silicon crystals up to several micrometers in size (fig. 4, *c*) and intermetallides containing aluminum, copper, nickel, magnesium and iron atoms. Dendritic structures are observed at the boundaries between the melted and non-melted layers formed during crystallisation on undissolved inclusions of silicon and intermetallides (fig. 5). These inclusions play role of the heterogeneous crystallisation centers at the cooling stage of the surface layer.

The X-ray diffraction analysis showed that the analysed layer of the coated sample contains aluminum, silicon, titanium nitride and titanium phases (fig. 5). No copper diffraction lines are seen on diffraction patterns. Due to the low concentration of copper detected by energy dispersive microanalysis one can suggest that copper atoms can take part in formation of δ-(Ti, Cu)N solid solution. Besides that copper can form fine inclusions in the coating, which are indistinguishable by X-ray diffraction analysis. CPF treatment leads to a decrease in the intensity of TiN and Ti diffraction peaks indicating a decrease in the volume fraction of these phases in the analysed layer. This effect increases with the growth of the absorbed energy density that correlates with the results of scanning electron microscopy.

Diffraction patterns show a broadening and decrease in the intensity of silicon lines with an increase in the density of absorbed energy. Such behaviour is caused by crystallisation of the melted surface layer of samples in conditions of high cooling rate, which leads to dispersion of silicon crystallites. In addition, silicon atoms can take part in formation of supersaturated aluminum-based solid solution. The latter is confirmed by the shift of aluminum lines on X-ray diffraction patterns towards large angles.

The lattice parameters of aluminum-based solid solution and its dependence on the absorbed energy density were determined by analysing Al (311) diffraction line (fig. 6). The decrease in the lattice parameter after treatment at 26 and 30 J/cm^2 is associated with the penetration of silicon atoms with a smaller radius into the aluminum crystalline lattice. A further increase in the lattice parameter can be caused by the integration of alloying elements with a bigger atomic radius into the solid solution due to intermetallides dissolution in the melt under plasma impact.

Diffraction patterns of plasma-treated samples also show aluminum nitride diffraction peak of low intensity that correlates with the results of scanning electron microscopy (see fig. 2).

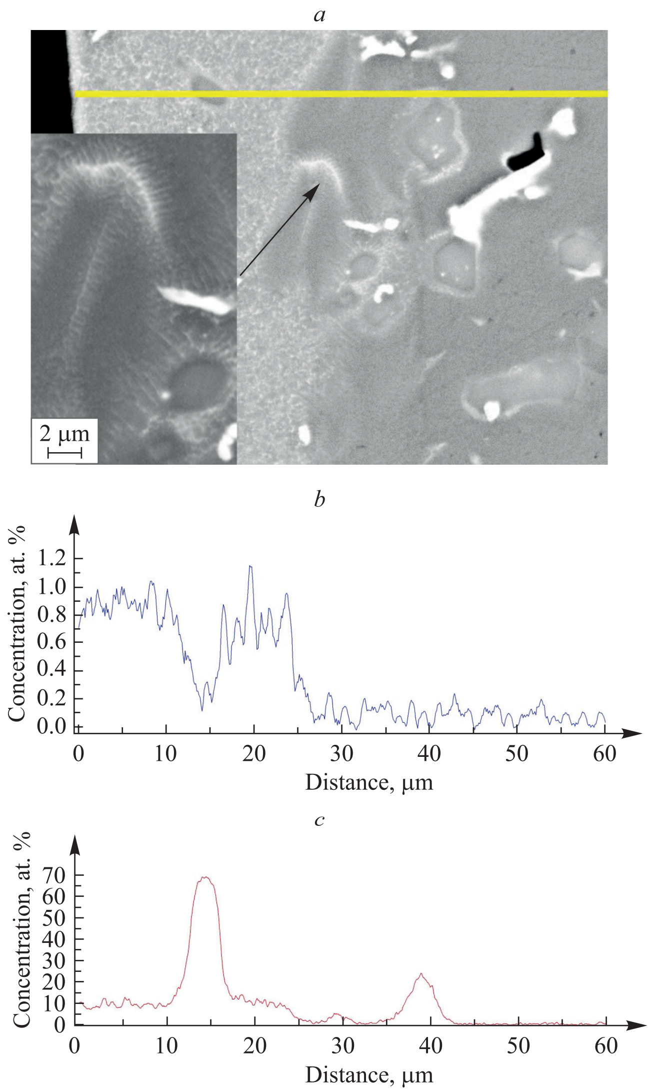

Fig. 4. Cross-section morphology (*a*), distribution of titanium (*b*) and silicon (*c*) along the depth of the surface layer in a sample treated with CPF at 35 J/cm^2

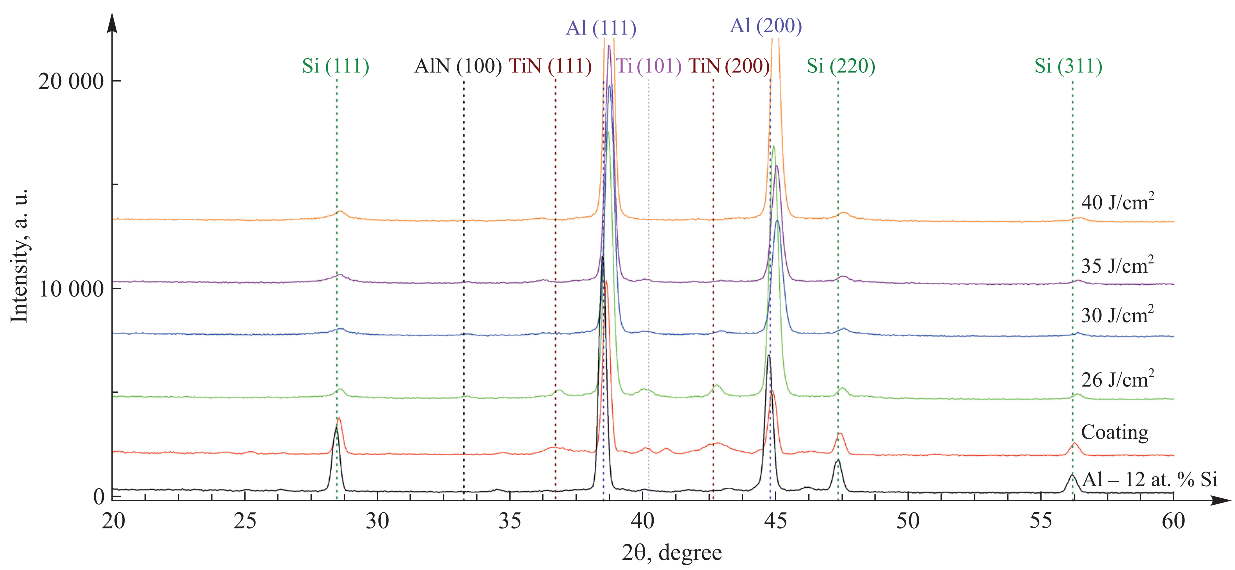

Fig. 5. Diffraction patterns of Al – 12 at. % Si alloy, (Ti, Cu)N/Al – 12 at. % Si alloy samples and (Ti, Cu)N/Al – 12 at. % Si alloy samples treated at different absorbed energy density

Investigations carried out earlier showed that CPF impact on the titanium – silumin system led to the formation of (Al, Si)$_3$Ti intermetallides as well [1]. However (Al, Si)$_3$Ti phase was not found in current experiment. This phenomenon can be explained by higher value of binding energy of the TiN compound. Thus CPF impact on (Ti, Cu)N/Al – 12 at. % Si alloy system leads mainly to fragmentation of coating instead of it dissolution in the melt. As a result formation of a composite surface layer containing dispersed (Ti, Cu)N inclusions inside Al – 12 at. % Si alloy structure can be expected. This supposition is confirmed by XRD data in fig. 5 where δ-TiN based phase is observed in the analysed layer in the whole range of treatment regimes.

A changes in the surface layer structure of investigated samples leads to an improving of their mechanical and tribological characteristics. The microhardness of the surface layer increased from (1.2 ± 0.1) GPa for the initial Al – 12 at. % Si alloy to (1.3 ± 0.2) GPa after treatment at 26 J/cm^2 and to (1.5 ± 0.1) GPa after treatment at 40 J/cm^2. The formation of aluminum nitride and a supersaturated aluminum-based solid solution enforced with (Ti, Cu)N inclusions, structure refinement are the possible reasons for the surface layer microhardness increase.

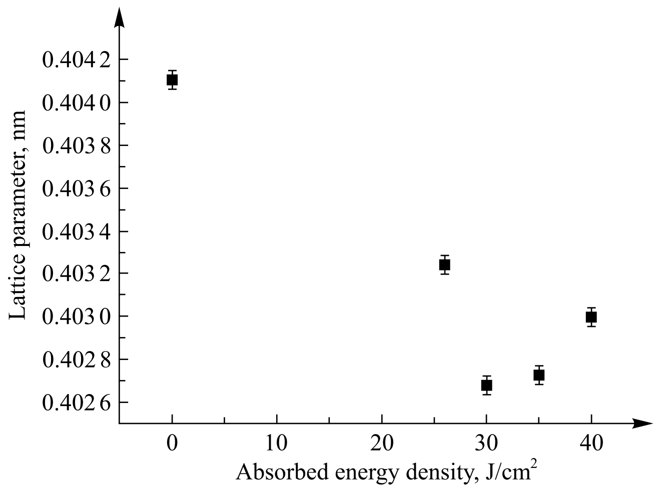

Fig. 6. Dependence of the aluminum-based solid solution lattice parameter on the absorbed energy density

The dependences of the friction coefficient on the indenter siding distance before and after plasma treatment with different absorbed energy density are shown in fig. 7. Minimal value of friction coefficient is observed for the sample treated at 30 J/cm^2 (except of the area corresponding to the beginning of the sliding distance). Maximum value of friction coefficient is observed after treatment at 40 J/cm^2.

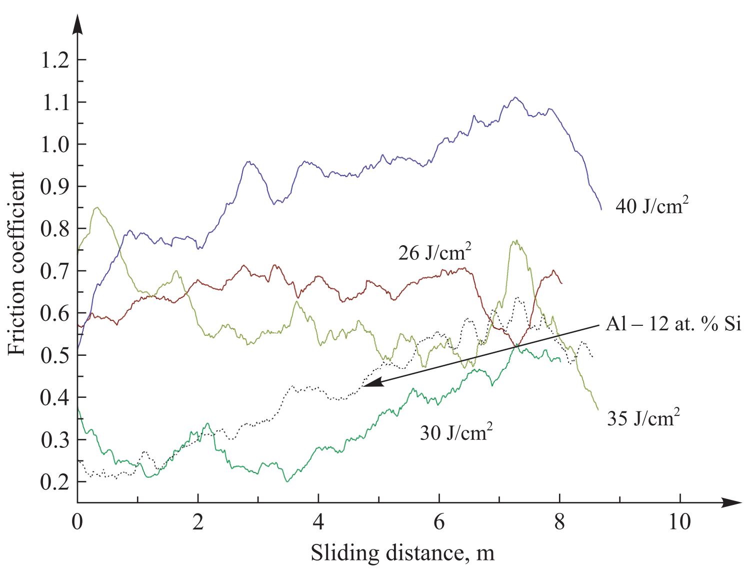

Fig. 7. Dependence of the friction coefficient on the indenter sliding distance for the sample of Al – 12 at. % Si alloy and samples of (Ti, Cu)N/Al – 12 at. % Si treated by CPF with different absorbed energy density

One can see that there is no direct correlation between the microhardness of the surface layer and the friction coefficient. At the same time, the friction coefficient value is depended on many factors including strength characteristics, roughness, etc. Therefore, an additional analysis of the surface roughness was conducted (table 3).

Table 3

The R_a value (arithmetic average roughness) of the Al – 12 at. % Si alloy and samples of (Ti, Cu)N/Al – 12 at. % Si alloy processed by CPF with different absorbed energy density, μm

Al – 12 at. % Si alloy sample	(Ti, Cu)N/Al – 12 at. % Si alloy sample treated by CPF			
	at 26 J/cm^2	at 30 J/cm^2	at 35 J/cm^2	at 40 J/cm^2
4.3	6.2	4.0	3.3	6.2

As can be seen from the results obtained, the lowest roughness is observed for samples treated with CPF in the range of energy density 30–35 J/cm^2 that could effect on the friction coefficient behaviour.

Conclusions

The findings showed that treatment of (Ti, Cu)N/Al – 12 at. % Si alloy system by compression plasma flows with an energy density absorbed by the surface in the range of 26–40 J/cm^2 leads to formation of silumin surface layer with a thickness of 19–45 μm containing (Ti, Cu)N inclusions. The concentration of titanium in the surface layer decreases from 8.2 to 0.1 at. % with the growth of energy density that is explained by an increase of the thickness of the melted layer and growth of the intensity of surface erosion during plasma impact. An AlN film is formed at the surface of the material as a result of the interaction of the surface atoms with the residual atmosphere in the vacuum chamber.

The mixed layer is characterised by a finely dispersed cellular structure. Partial dissolution of silicon and intermetallic compounds crystallites occurs in this layer, which leads to the incorporation of silicon atoms and alloying elements into the aluminum crystal lattice. The lattice parameter of an aluminum-based solid solution shows a non-monotonic dependence on the absorbed energy density.

CPF treatment with an absorbed energy density of 40 J/cm^2 leads to an increase in microhardness from 1.2 GPa (Al – 12 at. % Si alloy) to 1.5 GPa. Minimal value of friction coefficient (lower than that of Al – 12 at. % Si alloy) is observed for the sample treated at 30 J/cm^2.

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