

Structural and Phase Changes in the System Al-Si-Ti-B, Synthesized Using the Electron-Ion-Plasma Treatment Method

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Abstract. The results of surface modification of silumin samples of the eutectic composition using the combined method, which includes the irradiation with plasma formed during an electric explosion of the conductive material and the subsequent processing with a high-intensity pulsed electron beam, are presented. Formation of a multilayer multiphase submicron and nanoscale structure with high mechanical and tribological properties is established. The results of the structural phase analysis of the material are discussed from the standpoint of thermodynamics.

1. Introduction

Pulsed melting with the simultaneous saturation of surface layers of the material with doping elements and the subsequent crystallization and formation of strengthening phases, carried out by plasma formed during an electric explosion of the conductive material (electroexplosive doping), is one of the promising methods in the modification of the structure and properties of metals and alloys [1]. The formed layers have a metallurgical bond with the substrate having the adhesion at the level of cohesion and slightly change dimensions of the item. An additional increase in properties of the surface layer of the material and the item is generally possible during the subsequent heat treatment [2]. An effective method of heat treatment is high-intensity pulsed electron beams [3]. As compared to the widespread laser technology, the electron-beam technology has great potential in monitoring and control of the amount of the input energy, differs in the locality of energy distribution in the near-surface layer of the treated material and a high efficiency coefficient. Ultra-high rates ($10^6 \div 10^8$ K/s) of heating up to melting temperatures and the subsequent cooling of a thin near-surface layer ($10^{-7} \div 10^{-6}$ m) of the material, a very small time of exposure to high temperatures ($10^{-6} \div 10^{-3}$ s), formation of limiting temperature gradients (up to 10^8 K/m) that provide cooling of the near-surface layer due to heat transfer into the integrally cold volume of the material at a rate of up to 10^7 K/s, create conditions for formation of an amorphous and nanocrystalline structure in the near-surface layer [4].



The aim of the present work is to reveal evolution patterns of the structure and the phase composition of the alloy aluminum-silicon (silumin) subjected to electroexplosive doping and the subsequent irradiation with a high-intensity pulsed electron beam of a submillisecond exposure time.

2. Material and methods of research

The alloy Al–12.5% Si (eutectic silumin) was selected as a research material [5]. Electroexplosive doping of the silumin surface was carried out on the installation EVU 60/10 (Siberian State Industrial University, Novokuznetsk). The following mode of doping with plasma formed during an electric explosion of titanium foil with a sample of boron powder was used: the exposure time of plasma is $\sim 100 \mu\text{s}$, the absorbed power density on the axis of the jet is $\sim 8.2 \text{ GW/m}^2$, the pressure in the shock-compressed layer near the surface is $\sim 18.8 \text{ MPa}$; the thickness and the weight of titanium foil is about $\sim 20 \text{ microns}$, $\sim 90 \text{ mg}$, the weight of boron powder is $\sim 90 \text{ mg}$. The subsequent melting of the modified surface layer of silumin was carried out with a high-intensity pulsed electron beam on the installation "SOLO" (IHCE SB RAS) [6] under the mode: the electron energy is 18 keV , the energy density of the electron beam is $(15\div 30) \text{ J/cm}^2$, the pulse duration is $200 \mu\text{s}$, the number of pulses is 5, the pulse repetition rate is 0.3 s^{-1} ; the irradiation was performed in argon at a residual pressure of 0.02 Pa . The following took place at the given parameters of the electron beam: heating of the near-surface layer of silumin with a thickness of $\sim 30 \text{ microns}$ up to melting temperature, crystallization and quenching from the melt at rates up to 10^6 K/s . The study of the structure of the modified layer of silumin was carried out using methods of metallography, scanning and transmission diffraction electron microscopy, X-ray diffraction analysis.

3. Results and discussion

The combined treatment of silumin surface, which includes the irradiation with plasma formed during an electrical explosion of the conductive material and the subsequent irradiation with a high-intensity pulsed electron beam, is accompanied by a multiple increase in the strength and tribological properties of the near-surface layer of the material. Analyzing the results obtained during tests it is possible to note the following. First, the combined treatment, which includes doping with plasma of an electric explosion of titanium foil with a sample of boron powder and the subsequent irradiation with an electron beam, is accompanied by hardening of the surface layer of silumin up to the value 7.5 times greater than the microhardness value of the sample core. Secondly, the combined treatment results in formation of extended hardened layers, the thickness of which reaches $200 \mu\text{s}$. Thirdly, the microhardness and the thickness of the hardened layer of silumin depend on the mode of the subsequent irradiation with an electron beam. Namely, an increase in the energy density of the electron beam (at constant values of other parameters of the beam) leads, as a rule, to an increase in the microhardness and the thickness of the hardened layer.

High level of hardness of the surface layer of silumin formed during the combined treatment, which includes electroexplosive doping and the subsequent irradiation with an electron beam, leads to a significant decrease in the friction coefficient of the material. During an electroexplosive doping of silumin with plasma formed during an electric explosion of titanium foil with a sample of boron powder and the subsequent irradiation with an electron beam with parameters of 25 J/cm^2 , 200 ms , 5 pulses, 0.3 s^{-1} a decrease in the friction coefficient in 5-6 times is observed (counterbody – solid alloy VK8, the load on the indenter is 20 g). It shall be noted that at the given treatment mode the sample of silumin was characterized by an extended modified layer with a high, as compared to the initial material, microhardness level.

Thus, the carried out studies show that the combined treatment, which includes doping of the surface layer of silumin with plasma formed during an electric explosion of the conductive material and the subsequent irradiation with a high-intensity electron beam, allows to modify silumin in the volume with a thickness of up to 200 microns ; surface layers are characterized by high level of microhardness and low values of the friction coefficient. It shall be expected that hardening mechanisms of the surface layer of silumin, implemented using the given treatment method, are: solid

solution (formation of aluminum-based solid solutions), dispersion (formation of nanoscale particles of second phases), grain boundary (refinement of the grain structure), and deformation (formation of the defect substructure).

It is obvious that the main of the above mentioned hardening mechanisms in the investigated alloy is dispersion, caused by formation of nanoscale particles of the second phase. Fig. 1 presents isothermal sections of ternary systems Al–Si–B [8], Al–Ti–Si [9], Al–Ti–B [10], Ti–Si–B [11] demonstrating many phases with an ability to form in the investigated alloy under equilibrium conditions. An important point for the considered alloy is the fact that, on the one hand, borides and silicides of transition metals, in regards to a range of physical-and-mechanical properties, are attributed to metal compounds with high electrical conductivity having temperature dependences of the thermal electromotive force in pairs with metals, similar to metal pairs [12, 13]. On the other hand, silicides and borides of titanium cannot be attributed to compounds of the implementation phase type since they do not satisfy the Hagg's condition and considerably exceed the Hagg's critical ratio of 0.59. This, for example, leads to the fact that in binary systems Ti–Si the substitution with silicon atoms is accompanied by formation of metal structures at a ratio $(R_{Si}/R_{Me}) > 0.9$. The content of silicon in silicides can reach values of about 50 at%. At higher contents of silicon, the substitution of metal atoms with silicon leads to formation of complex crystalline structures and to an increase in the role of the covalent bond among silicon atoms [14] (Figure 1).

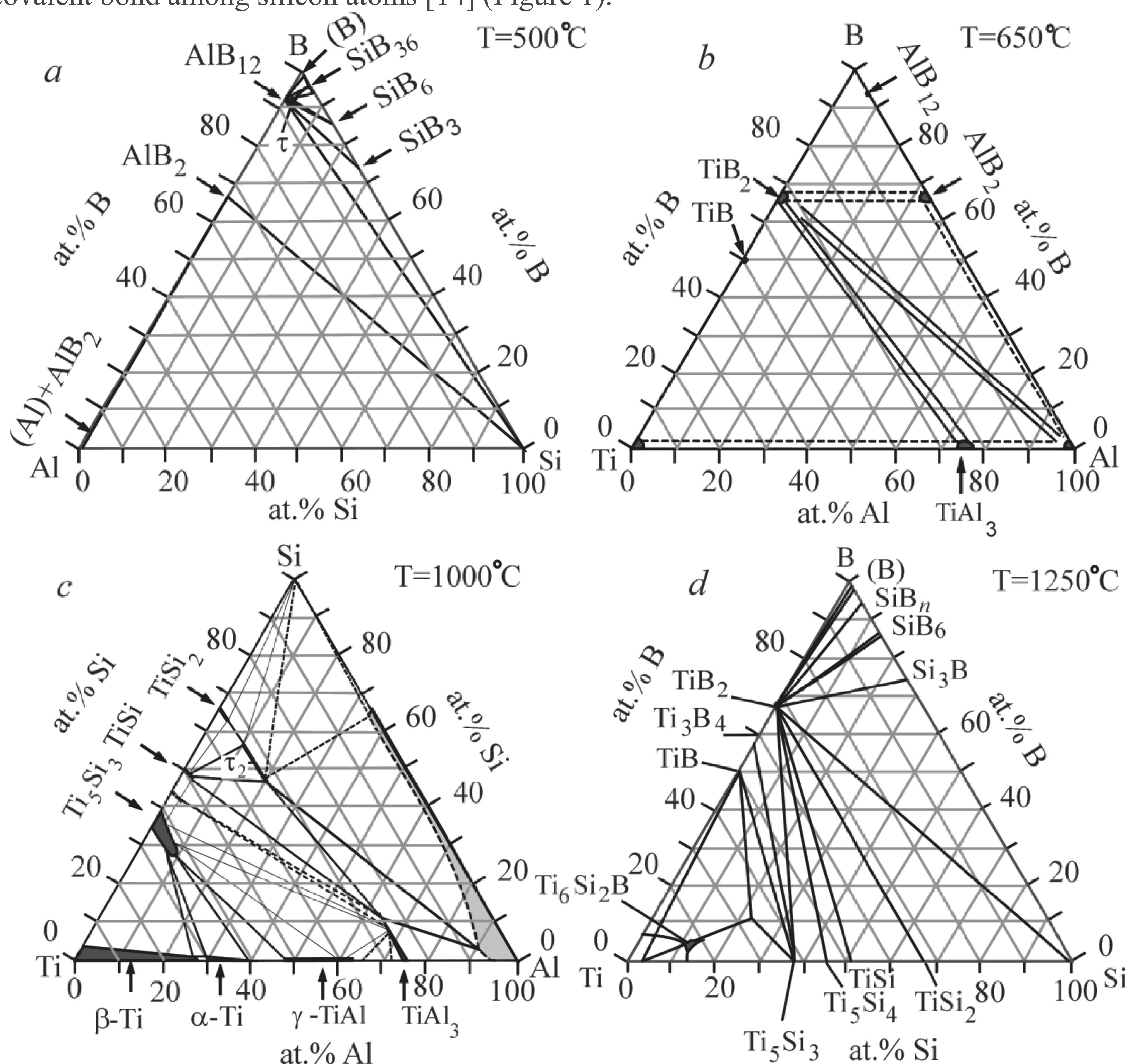


Figure 1. Isothermal sections of ternary systems, where *a* is Al–Si–B at 500 °C [8]; *b* is Al–Ti–Si at 1000 °C [9]; *c* is Al–Ti–B at 650 °C [10]; *d* is Ti–Si–B at 1250 °C [11].

Treatment of silumin surface with plasma formed during an electric explosion of a conductive material leads to formation of the doped layer with a thickness of tens of micrometers and a high level of roughness, containing a large amount of microcraters, cracks, particles and/or fragments of the exploded material, characterized by a high level of heterogeneity of the surface layer by alloying elements (Figure 2a).

It is obvious that deficiencies do not allow the direct use of the electroexplosive doping method for treatment of silumin items. In the present work, as mentioned above, the additional treatment of the modified surface of silumin was carried out by an intense pulsed electron beam. The electron-beam treatment of samples subjected to electroexplosive doping leads to a significant change in the structure and the elemental and phase composition of the modified layer. Most notably, this change takes place in the morphology and the roughness level of the alloyed surface (Figure 2b).

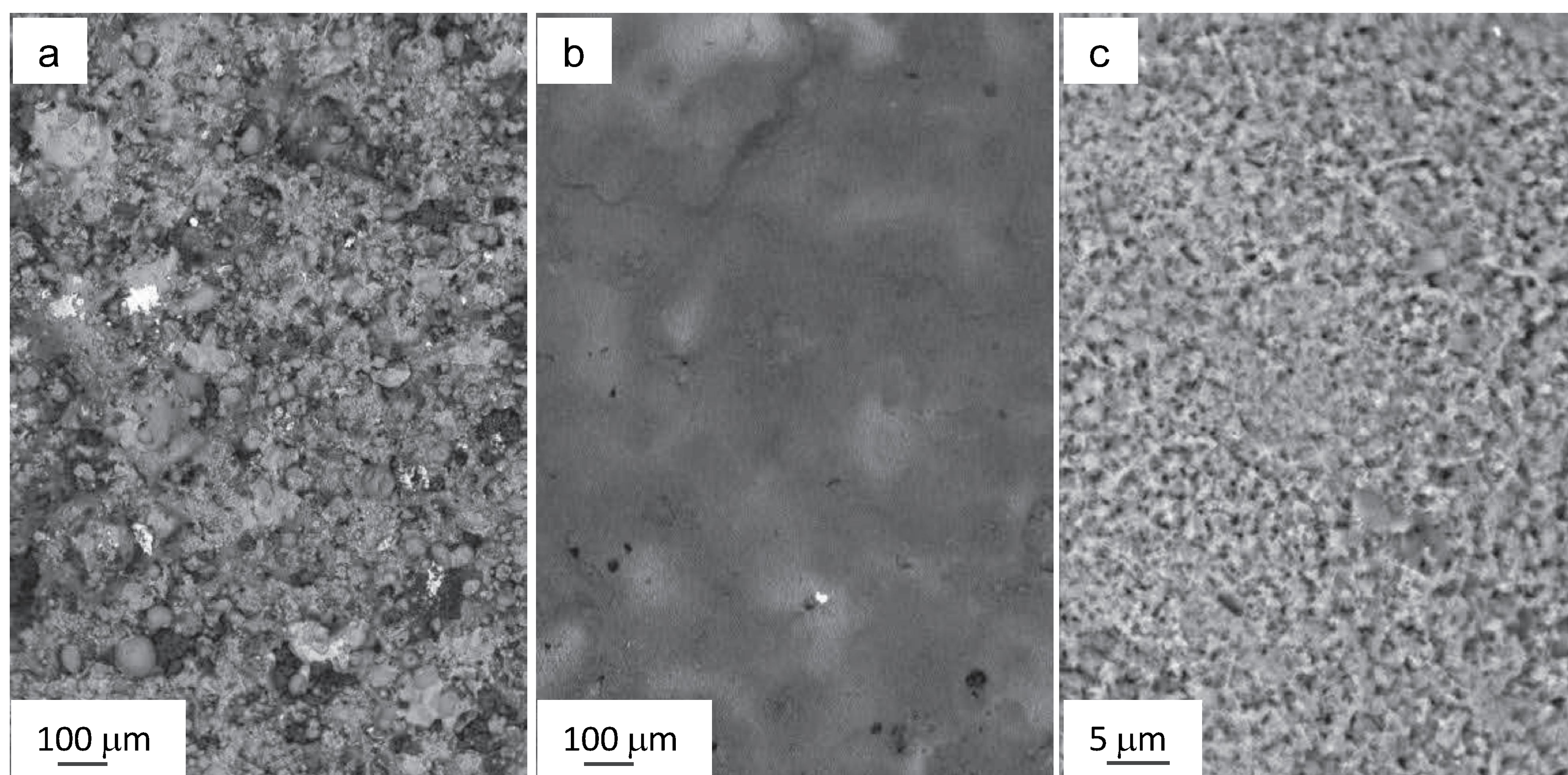


Figure 2. The structure of silumin modification surface, where *a* is with plasma formed during an electric explosion of titanium foil with a sample of boron carbide powder; *b*, *c* is after the additional irradiation with an intense pulsed electron beam with the parameters of 30 J/cm^2 , $200 \mu\text{s}$, 5 pulses.

Melting of the surface layer, which occurs when the energy density of the electron beam exceeds 20 J/cm^2 , and the subsequent high speed crystallization of the surface layer leads to formation of a structure, the crystallite size of which vary in the range of tens and hundreds of nanometers (Figure 2c).

Treatment of the doped layer of silumin with an electron beam is accompanied by an alignment of its thickness, a decrease in the number of microcracks and micropores (Figures 3a, 3b), a significant grinding of silumin layer adjacent to the modified surface layer (Figures 3c, 3d).

The studies of the phase composition of the surface layer of silumin subjected to a complex treatment, that combines doping with plasma formed during an electric explosion of titanium foil with a sample of boron powder and the subsequent irradiation with an intense electron beam, were carried out using methods of X-ray analysis.

By analyzing the X-rays shown in Figure 4 it can be noted that this doping method (electroexplosive doping and the subsequent electron-beam treatment) leads to formation of the following additional phases in silumin layer (along with Al and Si): Ti; Al_2Ti ; AlTi_3 ; Al_3Ti ; TiB.

The state of the defect substructure of phases of the surface layer of silumin subjected to electroexplosive doping and the subsequent electron-beam treatment has been analyzed using methods of diffraction electron microscopy by studying thin foils. First, the dispersion effect (up to submicron and nanoscale values) of the structure of main phases of silumin (aluminum and silicon), which agrees well with the results obtained using methods of scanning electron microscopy, shown in Figure 2 and Figure 3. The dimensions of phases formed during doping also vary in a wide range: from nano up to

submicron. In addition to phases detected using methods of X-ray analysis, nanoscale particles of titanium silicide TiSi have been revealed in the volume and at boundaries of aluminum grains, as well as at interphase boundaries aluminum/silicon.

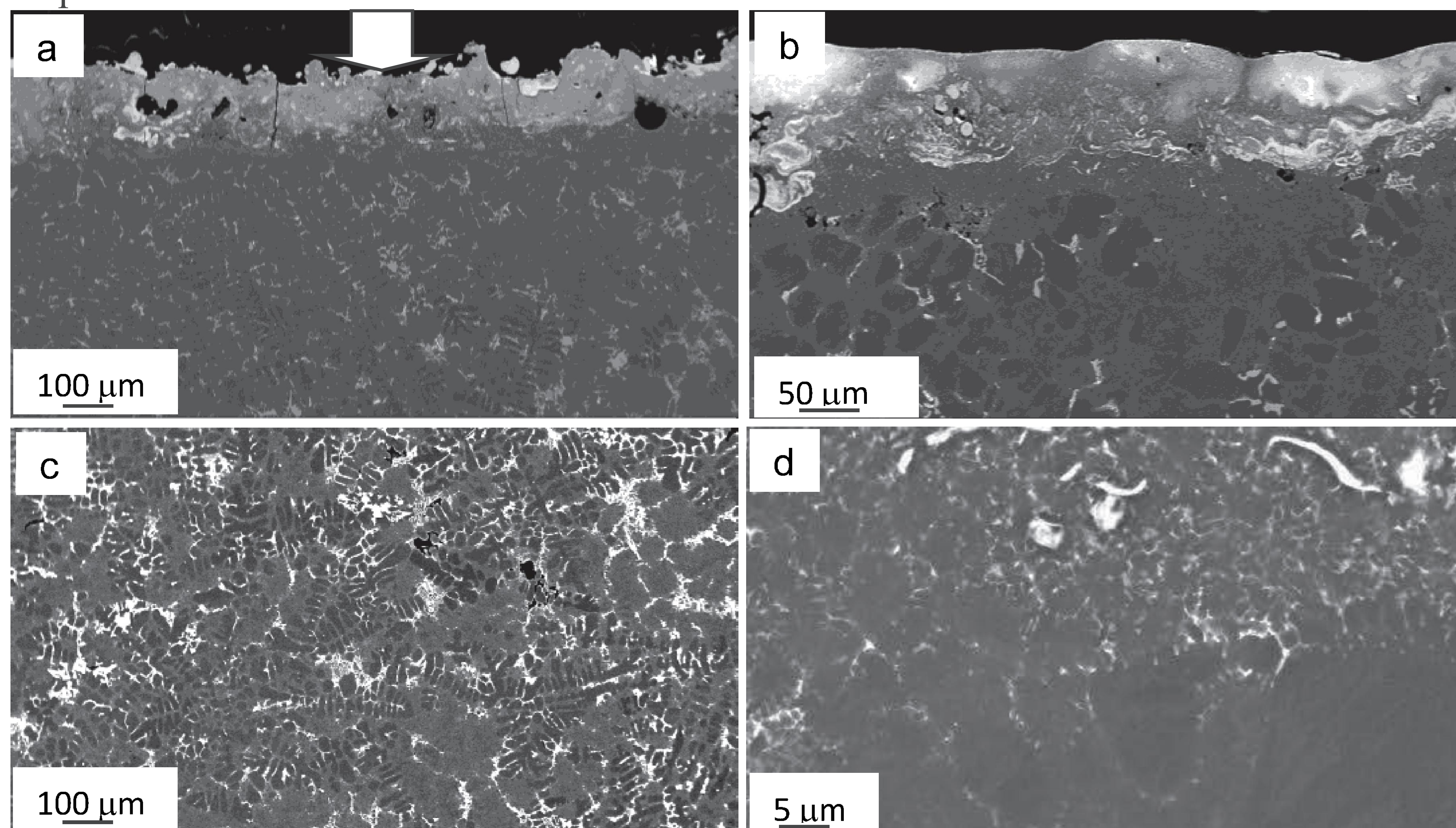


Figure 3. The structure of silumin cross-section is in the initial state (*c*); the structure after doping with plasma formed during an electric explosion of titanium foil with a sample of boron carbide powder (*b*) and after the additional irradiation with an intense pulsed electron beam with the parameters of 30 J/cm^2 , $200 \mu\text{s}$, 5 pulses. (*a*, *d*). In (*a*) the arrow indicates the modified surface.

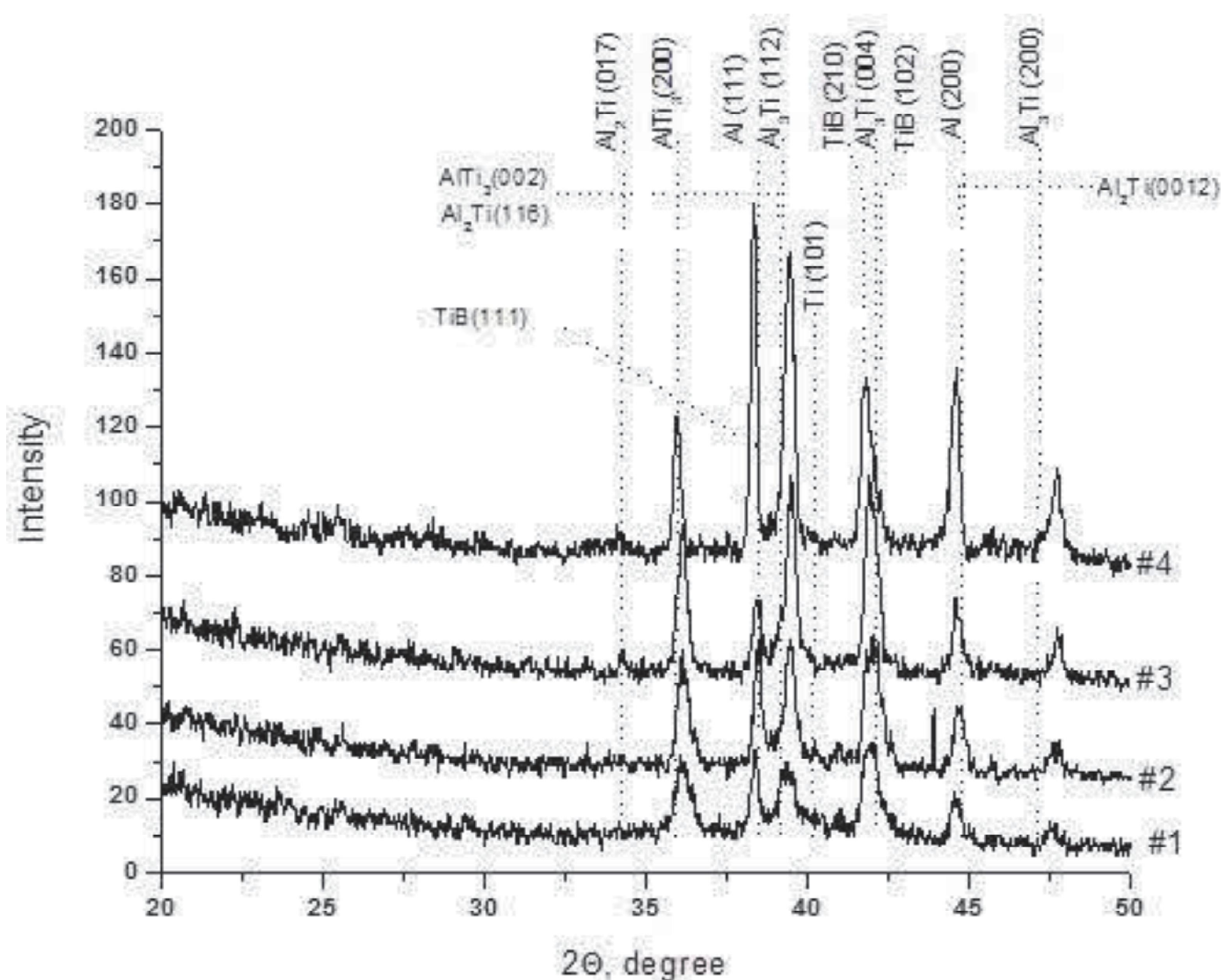


Figure 4. Sections of radiographs of the silumin sample subjected to doping with plasma formed during an electric explosion of titanium foil with a sample of boron powder (1); samples subjected to an additional treatment with an intense pulsed electron beam at various electron beam energy densities ($200 \mu\text{s}$, 5 pulses, 0.3 c^{-1}): 20 J/cm^2 (2); 25 J/cm^2 (3); 30 J/cm^2 (4).

4. Conclusion

The studies on the combined electron-ion-plasma treatment of silumin surface, which include doping with plasma formed during an electric explosion of titanium foil with a sample of boron powder and the irradiation with a high-intensity pulsed electron beam, have been presented. Formation of a multiphase submicron and a nanoscale structure of the surface layer, characterized by a significant increase in the strength and tribological properties, has been revealed.

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