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Comprehensive Study of Surface Layers and Tribological Properties of Antifriction Alloys of the AI-Si-Cu-Sn + Fe System

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ABSTRACT: The paper studies new antifriction aluminum alloys. The effect of small iron additions on the structure and tribological properties of samples was investigated. The tests were carried out using the "drive-roller" scheme with a step-by-step change in pressure. When analyzing the tribological properties, the operating modes were studied both in lubrication and without it. Tests in lubrication simulated the operation of the friction unit under normal conditions. Tests without lubrication simulated an extreme operating mode and were used as an express method for assessing wear resistance. The alloys were studied in the cast state and after heat treatment, as well as before and after tribological tests. It was found that after heat treatment, the silicon and mica phases acquired a rounded shape, and the copper content decreased. An iron-containing phase was isolated, which, due to the addition of manganese, acquired a favorable "skeletal" shape. After testing and lubrication, rounded solid particles were found on the surface of the drive. These particles remain on the surface and roll in the lubricant, creating a kind of protective framework that promotes stable operation of the contact pair. In tests without lubrication (leading to a strong increase in temperature in the friction zone), solid particles, on the contrary, enhance surface destruction, playing the role of an abrasive and promoting scuffing. The study of the block section prepared after testing in lubrication made it possible to estimate the thickness of the near-surface layer (30-40 cm) and showed the redistribution of elements in this layer. It was found that tin is squeezed out of the volume and forms "tracks" in the near-surface layer, elongated along the friction direction. In tests without lubrication, tin melts and is homogenously redistributed in the near-surface layer. It is shown that in tests in lubrication, the resulting film of secondary structures is distributed over the surface in a thin uniform layer with protective properties. During testing without lubrication, the film is uneven in thickness, which contributes to the development of macrorelief and can lead to scoring. It is shown that alloys containing iron (up to 1%) have higher tribological characteristics in modes with and without lubrication.

KEYWORDS: antifriction alloys, tribological tests, wear resistance, surface and near-surface layers, microscopy

1. INTRODUCTION

Friction plays a vital role in the operation of various units and mechanisms. The study of friction processes and changes in surface structure during friction is of great interest. Many studies have been devoted to the issue of assessing the performance characteristics of materials during friction and the changes in the rubbing surfaces that occur during this process [I-4].

For example, it has been shown that the deformation of antifriction bearing materials leads to a change in the surface topography and the release of so-called soft phases that serve as a kind of lubricant [5, 6]. It has also been shown that destruction is a multi-brain process, the links of which are the transfer of material from one rubbing surface to another, a change in the surface topography, and scoring. All these processes manifest themselves and proceed differently under different conditions and for different materials [7, 8].

The choice of materials for the manufacture of friction surfaces is very important in the development of machines and mechanisms. In the development of bearing units, one of the trends is the transition from expensive bronzes to aluminum alloys [9, 10]. In turn, for these alloys, an urgent task is to improve and / or modify their properties, achieved by improving their composition and selecting optimal heat treatment modes [II, 12].

This paper examines the promising idea of using aluminum alloys with iron additives. Interest in such materials is due to the potential possibility of producing alloys from cheaper materials (scrap) and waste from our own production, which would reduce the cost of the resulting product. However, the inevitable consequence of this will be the appearance of iron in the composition of the final product. It is known that iron is often a harmful impurity, leading to deterioration of the plastic properties of the material. To neutralize this effect, manganese is added to aluminum alloys [13].

Previously [14] the authors showed that the addition of iron does not lead to a noticeable deterioration in the tribological properties of the alloys. In (14) the tests were carried out using an express method, which provides for an operating mode without lubrication. However, modeling real operating conditions, which usually occur in lubrication, is also of interest.

The objective of this study was to study samples of experimental iron-containing aluminum alloys: their tribological properties, structure and elemental composition of the surface layers, as well as their change during tribological tests in two operating modes - with and without lubrication.

The final goal of the work was to assess the possibility of using iron-containing aluminum alloys as antifriction materials.

2. MATERIALS AND RESEARCH METHODS

2.1. Materials

In this work, we studied the aluminum alloy AI-5% Si-4% Cu—6% Sn with additives of iron (about 1%) and other elements. To neutralize the harmful effects of iron, manganese (0.5%) was added to the alloy in small quantities. Alloys of similar composition but without iron additives were used to compare tribological properties. These alloys were selected during sclerometric tests [15], which showed that these alloys have good load-bearing capacity with low shear resistance. The chemical composition of all the studied alloys is presented in Table 1.

After production, the cast alloys were subjected to heat treatment - annealing at a temperature of 500 °C, followed by quenching in water and aging.

This heat treatment mode was selected based on previously obtained results (14).

2.2. Sample preparation

In this work, the alloy surface was examined before and after testing. To study the original surface, sections were prepared using the TegraPol-25 and TegraForce-5 (Struers) complex. To study the surface layer after testing on the Accutom-S programmable cutting machine (Struers), an oblique cut of the alloy sample (at an angle of 45°) was made, which was also subjected to grinding and polishing. The above-mentioned works were carried out according to standard methods.

Chemical composition of the studied alloys Таблe1

Marking	Concentration, wt.%								
	Al	Si	Cu	Sn	Pb	Bi	Fe	Mn	Impurities
№ 1	82.80	5.1	4.3	5.6	0.4	0.4	0.7	0.5	<0.2
№ 2	84.30	4.9	4.2	5.5	1.0	<0.01	–	–	<0.1
№ 3	91.10	4.8	3.9	–	–	–	–	–	<0.2
№ 4	89.30	4.6	4.1	<0.01	0.7	1.2	–	–	<0.1

2.3. Tribological tests

Tribological tests were carried out on a T-O5 tribometer using the "partial liner-shaft" (pad-roller) scheme: the shaft (counter-sample) material was 45 steel, the partial liner (pad) was made of the experimental alloys under study. This contact pair was tested using a previously developed technique [16], with the only difference being that for tests with lubrication, the counter-sample was partially immersed in the lubricating medium so that during rotation the lubricant was captured by the counter-sample and drawn into the contact zone. Alloy samples were tested both with lubrication (5W40 diesel lubricant) and without it (dry).

In both cases, the tests were carried out with a step-by-step change in pressure from 0.2 MPa for tests without lubrication and from 3.2 MPa for tests with lubrication. The pressure was increased stepwise until scoring was reached. 2.4. Surface microscopy

Scanning electron microscopy was the main method for surface examination: a FEI QUANTA 650 scanning electron microscope with an EDAX elemental analysis attachment was used. The image was obtained using two detectors - secondary and backscattered electrons.

An accelerating voltage of 25 kV was used. The measurements were carried out in vacuum, no special surface preparation was required. The microscope is also equipped with a backscattered electron diffraction chamber, which made it possible to obtain information about the grain structure of the surface and near-surface layers, stresses in crystallites and their orientation. Surface studies using scanning electron microscopy were carried out before and after tribological tests.

Scanning probe microscopy was used to study the surface at the micro- and nanolevel: a SmartSPM™™ scanning probe microscope (AIST-NT) was used. The taping mode was used (AIST-NT cantilevers, fpN10, beam stiffness 10-20 N/m, resonance frequency 200-300 kHz). The scanning field was 100 × 100 µm?. The studies were carried out on the original surface.

3. RESEARCH RESULTS AND THEIR DISCUSSION

3.1. Microscopic studies of the surface of the original samples

To study the original surface of the experimental alloy in the cast and heat-treated state, a comprehensive microscopic study technique was used [11]. This technique involves applying special markers (reference points) to the surface, which allows the same surface areas to be isolated and studied using different microscopy methods (electron and probe).

Fig. 1 shows images obtained by scanning electron and probe microscopy of the surfaces of thin sections of the original samples (cast and after heat treatment).

The conducted complex of microscopic studies made it possible to obtain more complete information about the surface topography of the alloy. The applied method of reference marks allowed us to correlate the data of scanning electron and atomic force microscopy and to identify individual phases. X-ray spectral analysis (scanning electron microscopy) made it possible to determine the elemental composition of individual phases, while the atomic force microscopy method was used to determine their morphology. It should be noted that the use of atomic force microscopy made it possible to identify phases that were practically invisible in the image obtained by the scanning electron microscopy method (the silicon phase in the aluminum matrix is very poorly visible due to the proximity of the atomic numbers). As was shown earlier [I1, 14], heat treatment changes the structure of the alloy. In the present work, a comprehensive analysis of the surface of the sections confirmed that after heat treatment the silicon and soft phases acquired a rounded shape, the copper content decreased (due to its partial dissolution in the aluminum matrix). Also, an iron-containing phase (AI, (Fe, Mn), Si,) with a specific "skeletal" shape (Fig. 1) was found in the experimental alloy. It is known that a "needle-shaped" phase is usually formed in an aluminum alloy with iron, which causes embrittlement of the alloy. In this case, the addition of manganese neutralizes the harmful effect of iron and promotes the formation of a "skeletal" phase, which does not worsen the properties of the alloy (13, 14). It was also found that heat treatment does not affect this phase.

3.2. Tribological tests

To study the effect of iron additives to aluminum alloys on their tribological characteristics, comparative tests of alloys with with iron (No. 1) and without it (No. 2-4) for wear according to the "shaft - partial liner" scheme. The tests were carried out at a constant sliding speed with a stepwise increasing load in two modes: with lubrication and without it. Work in lubrication is a standard operating mode for such materials in friction units. Work without lubrication (express method) was used to reduce the duration of the tests. This also made it possible to study the behavior of the alloy in an extreme operating mode (it is known that these materials, according to their parameters, belong to the so-called "self-lubricating")

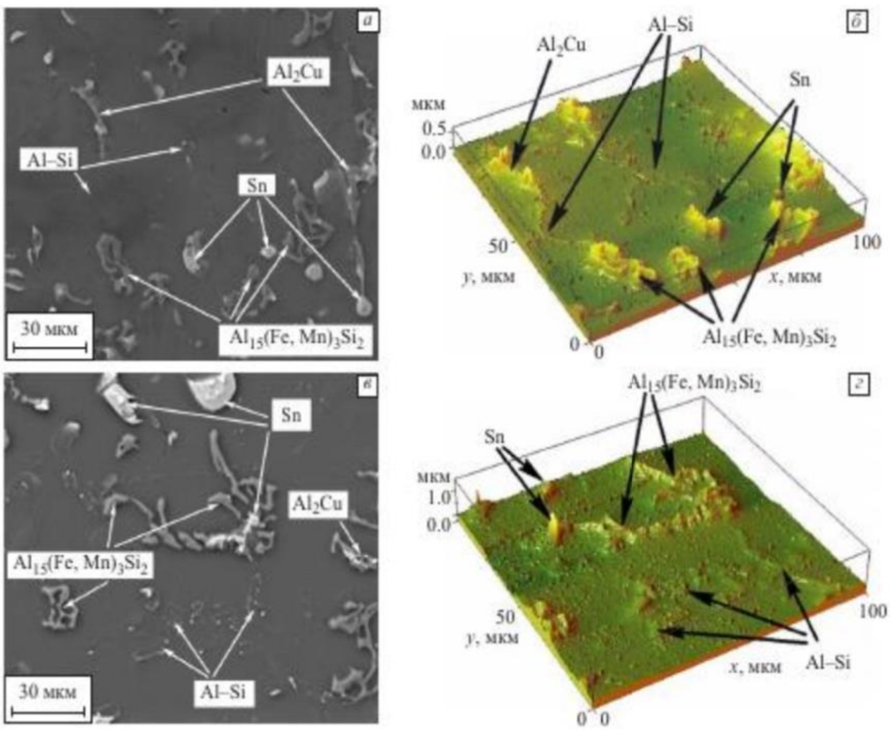


Fig. 1. Surface image of experimental alloy Lº 1 sections: scanning electron microscopy, lithos state (a); scanning probe microscopy (3D), as-cast state (b); scanning electron microscopy, heat-treated state («); scanning probe microscopy (3D), heat-treated state (2)

As a result of processing the experimental data, dependences of wear intensity on contact pressure were obtained for two cases — CO lubrication and without lubrication (Fig. 2).

For tests without lubrication, it is evident that wear occurs unevenly. For each alloy, a critical pressure can be identified, after which scoring occurs (see also Table 2). According to this parameter, alloy No. 1 with an iron content turned out to be the best, which withstood a contact pressure of 2-3 MPa. At pressures up to critical level, all alloys showed a similar dependence of wear intensity / on pressure P: l = 1.8-108 p0.7

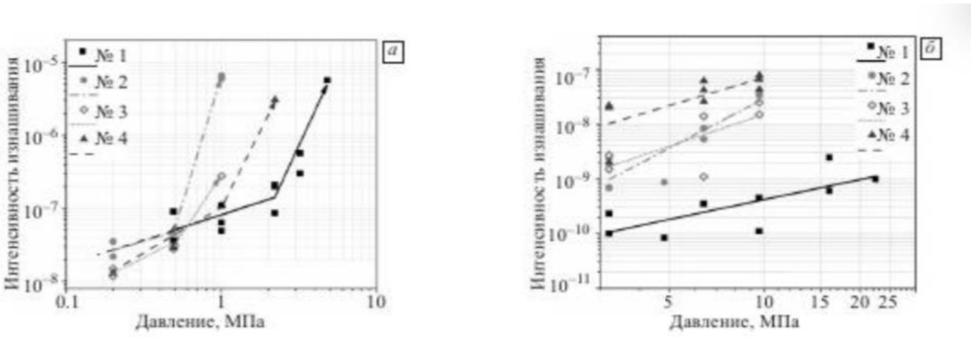


Fig. 2. Dependence of wear intensity 1 of the studied alloys on contact pressure P in modes without lubrication (a) and with lubrication (b)

For tests with lubrication, critical pressures were also determined and wear equations were obtained (dependences of wear intensity pressure). The results obtained are presented in Table 3.

Critical pressure and wear equation during wear tests of the studied alloys without lubrication Table2

Marking	Critical pressureMP	Wear equation
№ 1	2–3	$I = 1.8 \cdot 10^{-8} p^{0.7}$
№ 2	0.5	
№ 3	0.5	
№ 4	1.0	

Critical pressure and wear equation during wear tests of the studied alloys with lubrication Table3

Marking	Critical pressureM	Wear equation
№ 1	23	$I_1 = 2.6 \cdot 10^{-11} p^{1.2}$
№ 2	10	$I_2 = 3.1 \cdot 10^{-11} p^{3.0}$
№ 3	10	$I_3 = 1.8 \cdot 10^{-10} p^{1.9}$
№ 4	10	$I_4 = 1.4 \cdot 10^{-9} p^{1.7}$

It is shown that during tests with lubrication the wear process of alloys proceeds more uniformly, but at pressures above the critical value, scoring also forms. It is evident that for alloy No. 1 the critical pressure is 2 times higher than for the other alloys. In addition, this alloy showed a wear rate that is 1-2 orders of magnitude lower than the others and a smaller dependence on pressure.

Based on the results obtained, it can be concluded that alloy No. I (containing iron) surpassed alloys No. Nº 2-4 in all tribological parameters both in normal operating mode (i.e. in a lubricating environment) and in extreme mode (in the absence of lubrication).

3.3. Study of the contact pair after tribological tests with lubrication

After tribological tests with lubrication, studies of the shoe and roller surfaces were carried out. The studies have shown that significant changes have occurred on the surfaces of the contact pair tested in the lubricant. The scanning electron microscopy images of the surfaces of the contact pair (tested at a pressure of 20 MPa) are shown in Fig. 3. It is easy to see the formation of stripes, the direction of which coincides with the direction of friction: these stripes are more pronounced on the roller surface than on the surface of the shoe. Many rounded particles are visible on the surface of the shoe. Perhaps, their shape is associated with "rolling" during friction in a liquid oil medium. Of interest is the presence of elements and their distribution over the surface. Fig. 4 shows the mapping of the area shown in Fig. 3 by the main alloying components of the alloy. A comparison of the scanning electron microscopy images and the mapping results showed that the detected rounded particles contain a lot of silicon and copper (in the form of the eutectic compound Al-Si and the compound Al, Cu, respectively). Obviously, these particles have increased hardness. On the surface of the roller (Fig. 3, b) a film of secondary structures was found, the formation of which is due to the process of mass transfer of chemical elements from the surface of the shoe in the contact zone. It is evident that during tests in the lubricant the film of secondary structures protecting the surface of the roller from wear is distributed uniformly in a thin layer.

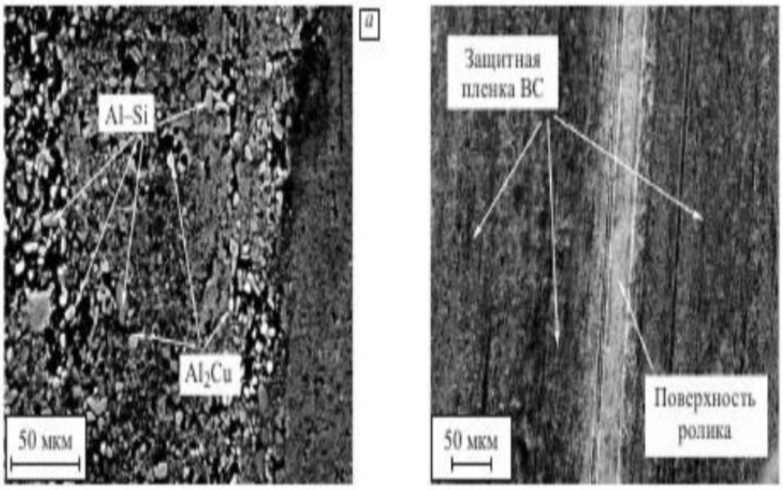


Fig. 3. Surface of contact pair after testing with lubricant at 20 MPa pressure, shoe (force No. 1) (a), roller (b). Scanning electron using secondary electron detector. BS – secondary structures.

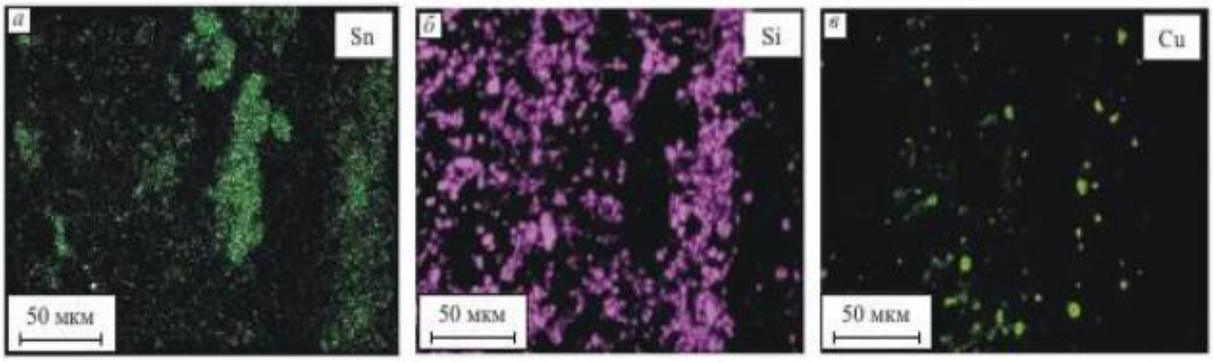


Fig. 4. Mapping shoe surface (alloy 1) by main alloying elements after tribological tests with lubricant Sn (a), Si (b), Cu (c)

For a more complete study of the processes occurring during operation in friction mode with lubricant, an oblique section of the shoe was prepared after testing, which allowed us to study the surface layers of the alloy sample.

The combined use of two detectors (secondary and backscattered electrons) allowed us to obtain a more complete picture of the presence of phases, their shape and distribution by depth (Fig. 5). In the obtained images, we can quite clearly distinguish the surface layer, the thickness of which is 30-40 μm. (Note that in work (14), where the tests were conducted in the dry friction mode at pressures an order of magnitude lower, the thickness of the modified layer was approximately 2-3 times greater. Obviously, this difference is determined by the difference in friction modes.) It is evident that the structure of this layer differs significantly from the structure in the volume. Phase components ordered in the direction of friction ("tracks") are traced. X-ray spectral analysis and mapping were carried out to determine the elemental composition on the section. The results of mapping for the main alloying components of the alloy are shown in Fig. 6.

Based on the data presented, it is possible to determine the composition of the "tracks", which are tin phases. Obviously, during friction, tin comes out of the volume to the surface. This is due to the fact that during friction in the near-surface layer, deformation of grains and dendritic cells of the aluminum matrix occurs, leading to the extrusion of the low-melting tin phase onto the surface of the pad. When the pressure on the rough contact surfaces increases, micro-seizure occurs, leading to the mass transfer process (tin, together with other elements, is transferred to the counterbody). This process leads to the formation of a protective film of secondary structures. Also, on the cut of the pad, it is possible to determine the distribution of refractory phases, which, during friction, are concentrated on the surface in the form of rounded particles. These particles create an additional framework that prevents further destruction of the soft aluminum matrix. The use of the backscattered electron diffraction method allowed us to obtain additional results that demonstrate the process of deformation of aluminum matrix grains in the near-surface

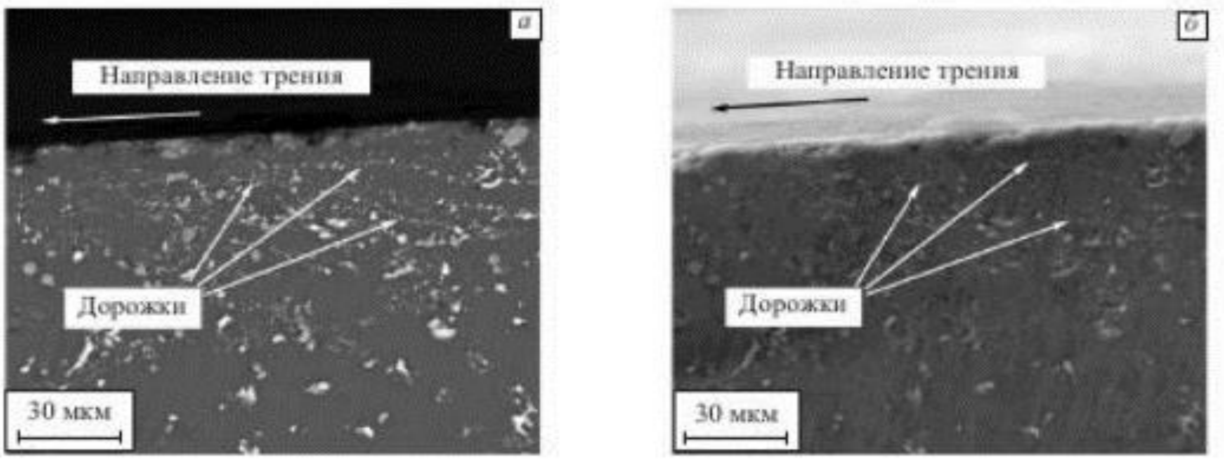


Fig. 5. Surface of the oblique cut of the shoe (alloy No. 1) after tribological tests with lubrication. Scanning electron microscopy in backscattered electrons (a), in secondary electrons (b)

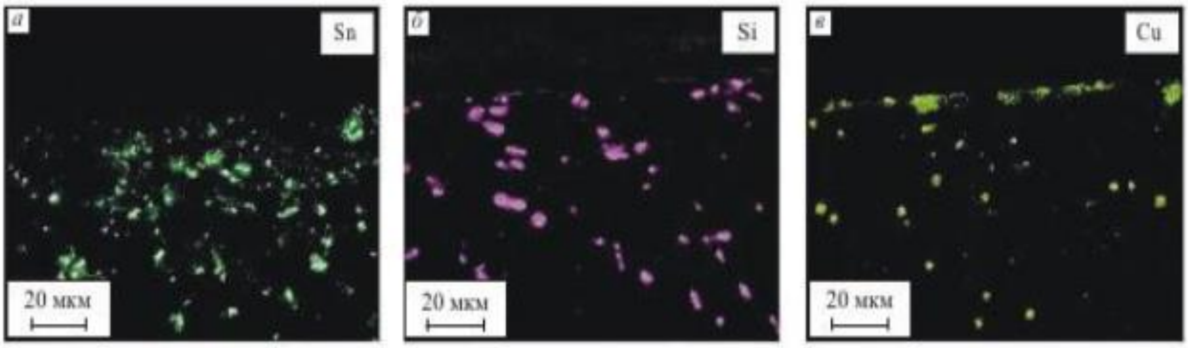


Fig. 6. Mapping of the surface of the oblique cut of the shoe (alloy No. 1) after tribological tests with lubrication by the main alloying elements Sn (a), Si (b), Cu (c)

layer after tribotesting (Fig. 7). Analysis of the backscattered electron diffraction patterns (Fig. 7, 6) showed that in the near-surface region it is not possible to identify the grains (black, indicated by the arrow), apparently as a result of their strong deformation. In the region further from the surface (100-150 µm) the grains (gray) are quite clearly visible, but it is not possible to determine their orientation - apparently due to their strong distortion.

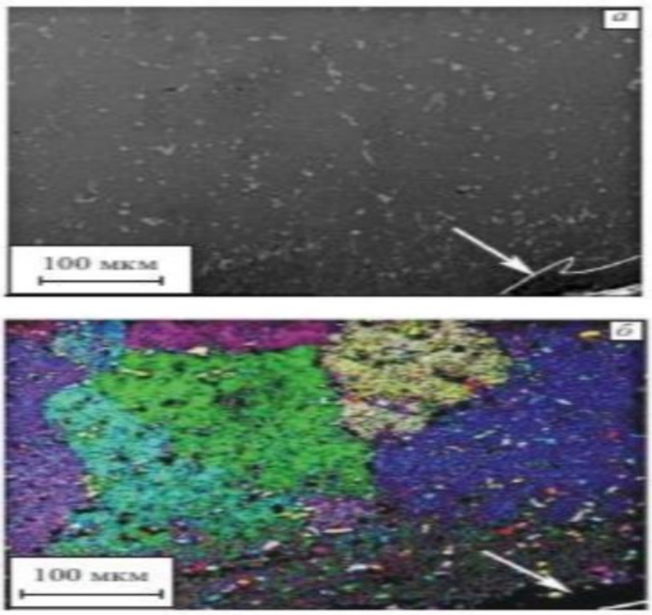


Fig. 7. Electron microscopic image of the surface of the oblique cut of the shoe (alloy No. 1) after tribological tests with lubrication: in secondary electrons (a); backscattered electron diffraction map (b)

At the same time, it is clear that in the bulk layers of the alloy, the grains have different orientations and are not distorted. Dendritic cells, which are sections of individual branches of the dendrites, are usually clearly visible inside the Al grains in a light microscope.

Thus, it is shown that a combination of various electron microscopic methods allows a detailed study of the processes occurring on the surface and in the near-surface layer of the material during friction.

3.4. Study of the contact pair after tribological tests without lubrication

After conducting tribological tests without lubrication, the changes that occurred in the contact zone were also studied. In Fig. 8 shows the images of the surface of the contact pair (a shoe made of an experimental alloy and a steel roller) after testing at a pressure of 2 MPa. For a detailed study of the processes occurring during operation in the friction mode without lubrication, an oblique section of the shoe was also prepared after the tests, which made it possible to study the changes occurring in the material in depth (Fig. 9). As can be seen from Fig. 8, a noticeable change in the topography and composition occurred on the surfaces of the shoe and roller. Under extreme conditions (without lubrication), a large friction force leads to an increase in the maximum tangential stresses of the pattern and, as a consequence, the formation of periodic microcracks on its surface, propagating inward in the direction of the maximum tangential stresses [17]. Under the influence of the friction force, these cracks open, forming a scaly surface structure (Fig. 8, a, indicated by arrows) with the subsequent separation of wear particles, which have a flat shape. However, due to the inhomogeneous structure of the alloy with the presence of hard particles, the development of some cracks is delayed. Fig. 9, a shows an enlarged fragment of a longitudinal section of the tested block.

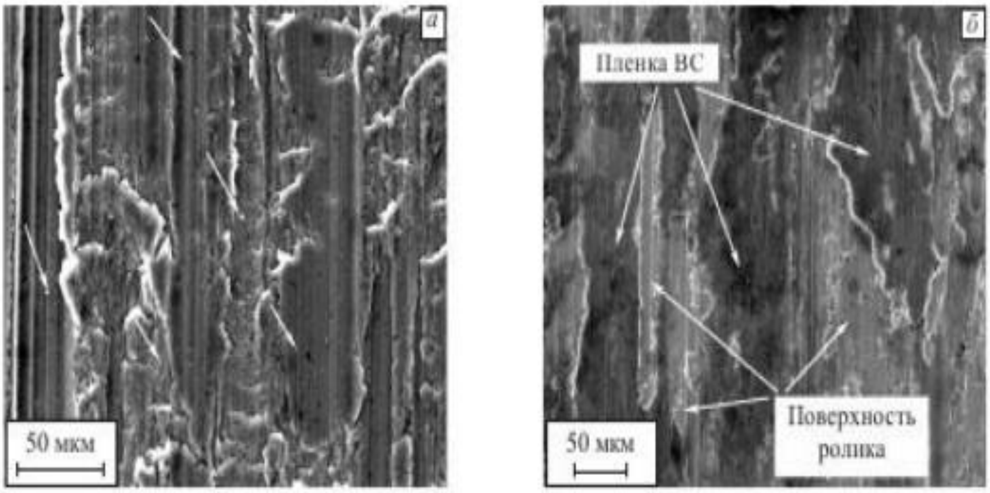
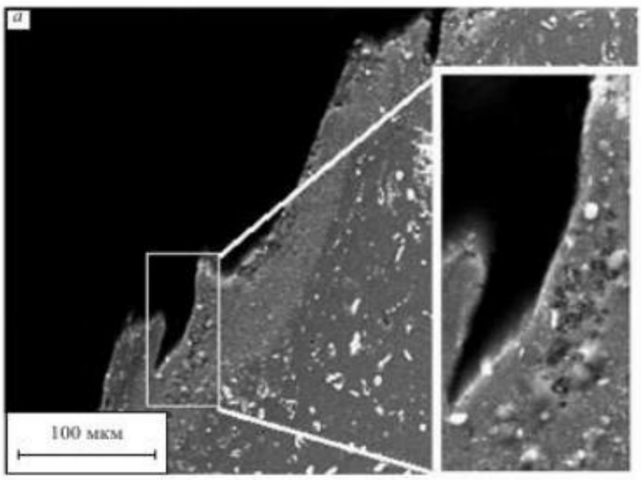


Fig. 8. Contact pair after tribological tests without lubrication at a pressure of 2 MPa: shoe (alloy No. 1) (a), roller (b).

Scanning electron microscopy in secondary electrons. It is evident that the crack propagation ends at a solid inclusion (Al, Cu). Thus, the alloy structure prevents the development of cracks during friction in extreme conditions, thereby providing greater wear resistance and load capacity, which is confirmed by the results of tribological tests (see Fig. 2, a). In tests with lubrication, in contrast to tests without lubrication, a lower friction force creates lower shear stresses, which are insufficient for the loss of shear stability of the material.



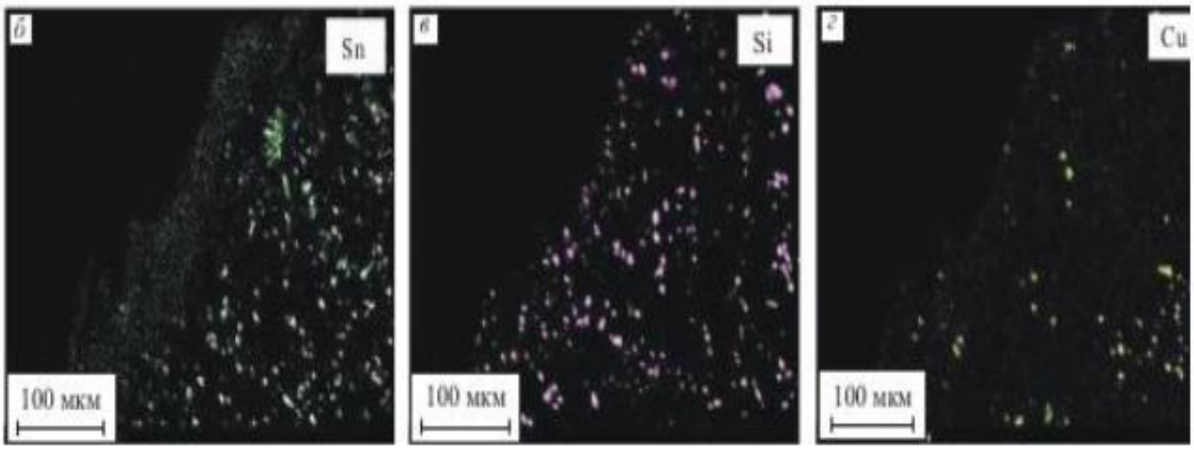


Fig.9. Surfaces of the oblique cut of the shoe (alloy No. 1) after tribological tests without lubrication: Scanning electron microscopy (a), mapping by the main alloying elements: Sn (b), Si (c), Cu (a)

It is known that a change in conditions in the contact zone (in particular, an increase in pressure and temperature) can cause elastic-plastic deformations of the surface and near-surface layers of the rubbing bodies [18].

In this case, a complex of mechanical and physicochemical processes occurs on the contact surfaces, during which the shoe material is transferred to the steel surface of the roller, promoting the formation of a film of secondary structures. Under extreme operating conditions of the friction unit, a further increase in pressure and temperature in the contact zone leads to plastic flow of the shoe material and more intense mass transfer. These processes contribute to the formation of adhesions and the development of relief on the roller (Fig. 8, b).

Scanning electron microscopy of the roller surface with elemental analysis (Fig. 8, b) revealed the presence of longitudinal stripes and areas that differ in color and chemical composition from the roller material. Thus, it was established that friction of two homogeneous materials occurs in the contact zone ("aluminum transferred to the roller" — aluminum shoe). The adhesion of homogeneous materials is higher, therefore, a macro-relief develops, which entails deformation and partial destruction of the shoe material (in Fig. 8, a, the formation of longitudinal grooves and cavities was detected). During friction, the structure of the surface layer material and its properties change. This is due to plastic deformation in the contact zone, when mechanical energy is converted into thermal energy

[1]. The conducted studies of the oblique cut of the shoe (Fig. 9) showed that the soft phases in the surface layers were homogeneously redistributed (mixed) (Fig. 9, b). It is obvious that this occurs due to a significant increase in temperature in the contact zone. At the same time, refractory phases (eutectic compound Al-Si and AI, Cu) remained in the near-surface layers without changing their configuration (Fig. 9, c, 2).

In the present work, during testing in lubrication, a process of squeezing of soft phases to the surface is observed (occurring due to deformation of grains (Fig. 7, b) and dendritic cells), which cannot be traced during dry testing.

4. Conclusions

The work shows that the addition of iron leads to an increase in wear resistance (during testing both in lubrication and without lubrication).

During tribological testing in the lubricated mode, scuffing was observed for all the studied alloys at much higher pressures than without lubrication. At the same time, for the iron-containing alloy, the critical pressure (after which scuffing occurs) is also significantly higher than for the other alloys. This is explained by the fact that the iron-containing phases are solid inclusions and, together with the Al, Cu and Al-Si phases, create an additional framework that prevents further destruction of the soft aluminum matrix. This alloy showed a wear rate 1-2 orders of magnitude lower than the others and a lower dependence on pressure.

During tribological tests in the mode without lubrication, scoring occurs for all alloys with an increase in pressure.

However, for the iron-containing alloy, this occurs at higher pressures (approximately 2 times greater than for other alloys) due to the inhomogeneous structure containing solid inclusions that prevent cracks from propagating from the surface deep into the material.

A characteristic relief develops on the surface of contact pairs during tribological tests.

In the near-surface layers of the alloys under study (pads) after testing in lubrication, a change in the arrangement of elements is observed - primarily, the extrusion of tin to the surface. Tests without lubrication lead to a homogeneous redistribution of this element. During friction, due to mechanical and physicochemical processes occurring on the surface of contact pairs, mass transfer occurs, which promotes the formation of a film of secondary structures. The film of secondary structures on the roller surface can play a different role under friction conditions in lubrication and without lubrication. In the first case, it plays a protective role, while during friction without lubrication (with the development of macrorelief), it can promote the formation of scoring.

Thus, the work reveals and explains the sufficiently high tribological properties of iron-containing aluminum alloys. The alloy can operate both in lubrication and in critical friction modes. Thus, the possibility and prospects of using secondary iron-containing raw materials (scrap) in the production of antifriction aluminum materials are shown.

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