SEM and SPM investigations of the surface of antifriction aluminium alloys of the system Al-5%Si-4%Cu-6%Sn

O O Shcherbakova¹, T I Muravyeva¹, D L Zagorskiy¹², I V Shkalei¹, V A Lapitskaya³ and T A Kuznetsova³

¹Ishlinsky Institute for Problems in Mechanics RAS, 119526 Moscow, Russia
²Gubkin Russian State University of Oil and Gas, 119991 Moscow, Russia
³A.V. Luikov Heat and Mass Transfer Institute of NAS Belarus, 220072 Minsk, Belarus

shcherbakovaoo@mail.ru

Abstract. Two antifriction alloys based on the Al–5%Si–4%Cu–6%Sn system with the addition of bismuth, lead, and cadmium were investigated. The effect of these elements on the change in surface topography and the tribological properties of the samples was studied. Tribological tests simulating the work of the friction unit were carried out using “shoe-roller” scheme. After heat treatment in both alloys, the phase components acquired a globular shape. Mass transfer between the shoe and the roller led to the formation of a film of secondary structures of variable thickness on the roller surface. SEM and EBSD allowed observing a near-surface layer (50-100 µm thick) with a modified structure and grain shape. SPM made it possible to evaluate the difference in the mechanical properties of the surface layer at the nanoscale level. It was shown that the material was strengthened in the process of friction.

1. Introduction

The development of new antifriction materials is an important national economic task. In particular, it is of great importance for engineering to develop alloys used in friction mechanisms, for example, in plain bearings [1, 2]. The service life of such nodes depends on the correct, scientifically based selection of the composition of the alloys. For the manufacture of such bearings expensive bronze are usually used [3, 4]; therefore, the urgent task is to create cheaper materials with high performance properties. Alloys based on aluminium, possessing antifriction characteristics comparable to bronze, meet these requirements. It is known that aluminium alloys are promising materials used in various fields of mechanical engineering [5, 6].

Al–5%Si–4%Cu–6%Sn alloys were proposed as the optimal variant of antifriction materials for bearing assemblies [7, 8]. It is assumed that this alloy can be a basic composition. The addition of small amount of alloying fusible elements can effectively influence the structure and properties of the resulting alloy [8]. The idea of this work was the partial replacement of tin by bismuth, lead, and cadmium. It is known that the addition of cadmium to some aluminium alloys significantly improves their properties – for example, increases their hardness [9]. In this case, the use of cadmium seems promising (despite its toxicity). One of the aims of this work was to verify the effectiveness of cadmium alloying of the proposed system.

The change in the properties of the resulting alloys can be evaluated by various methods. Thus, tribological tests allow determining the main operational parameters (coefficient of friction, wear...
resistance, and abrasive resistance). The changes occurring on the contact surfaces and in the surface layers during friction were evaluated after these tests. These studies were carried out by a complex of different methods. So, scanning electron microscopy (SEM) studies of surfaces are carried out not only in secondary, but also in back-scattered electrons, which greatly increases the possibilities of research, allows more accurate studying the surface morphology and visualizing its phase composition. X-ray analysis is used for investigation of elemental composition. The SEM method is also used to estimate the near-surface layers on sample slices (cuts, sections).

The electron back-scattered diffraction (EBSD) method provides information on the surface crystal structure. As a rule, the electron diffraction patterns are processed and presented as an image of the orientation of the grains, which is very important for evaluating the properties of the surface.

Electron microscopic studies are often carried out in combination with the scanning probe microscopy (SPM) surface studies. The value of the SPM method consists both in obtaining the topographic images of the surface itself and in the possibility of using additional modes. Among the latter, the measurement of force curves can be noted.

The purpose of this work was to study the new antifriction alloys using a complex of the abovementioned methods.

2. Experimental

2.1. Samples

Two samples based on the Al–5%Si–4%Cu–4%Sn system were studied. Samples of the following composition were obtained: No. 1 – Al–5%Si–4%Cu–4%Sn–0.5%Bi–0.5%Pb–0.5%Cd and No. 2 – Al–5%Si–4%Cu–4%Sn–0.5%Bi–0.5%Pb.

The proposed composition of the alloy is determined by several factors. The alloy must have sufficient hardness, which provides by the basic composition of Al–5%Si–4%Cu. The addition of silicon improves casting properties. Also, the alloy must contain a sufficient number of low-melting elements, which is important for reducing friction and ensuring the resistance to tearing during dry friction or in lubrication conditions. It is known that under extreme conditions of friction on the contact surface, these elements are released, which protects the surface, performing the function of solid lubricant [10]. Previous studies have shown that the addition of Bi to the base alloy increases the fill rate of the form, and the addition of Cd increases the strength and hardness of the alloy [9]. Sample No. 1 differed from sample No. 2 by the presence of 0.5% cadmium.

The samples were subjected to heat treatment, which consisted in heating to 500°C followed by quenching in water and aging (at a temperature of 175°C for three hours). This mode of maintenance was previously selected for alloys of similar composition in Ref. [9].

Sample preparation for further studies (SEM, EBSD, and SPM) consisted in the preparation of thin sections (initial surface and sections after testing) and their polishing.

2.2. Investigation of hardness

Surface hardness of the samples was measured. The Brinell measurements were carried out on thin sections (cuts) of the samples in the cast state and after heat treatment. The hardness was determined by the standard method on the Nemesis 9000 universal hardness tester with ball diameter 2.5 mm and load on the indenter about 600 N.

2.3. Tribological tests

Tribological tests of alloys were carried out on the T-05 tribometre according to the shoe (from the material under study) – roller (counterbody made from steel St45), with step-by-step load changes. This contact pair was studied according to the previously tested method [8] at the pressures of 0.5 and 1 MPa. After tribological tests, the surface and cuts (slides) of the sample were investigated (see 2.4). Perpendicular slices were prepared by cutting the sample after tests and polishing the surface of the cut.
2.4. Microscopy
In this work, a complex microscopic examination technique was used. Electron microscopic studies were performed on a Quanta-650 SEM (comp. FEI) with EDAX analytical equipment including EDS X-ray microanalyser and an EBSD electron back-scattered diffraction chamber (EBSD method). An accelerating voltage of up to 25 kV and two detection modes, secondary and back-scattered electrons, were used. Samples were examined before and after heat treatment, as well as before and after tribological tests. After tribological tests, the surface of the end sections was also studied.

![SEM images](image)

**Figure 1.** SEM image (secondary electrons) of the initial surface of the samples: (a) No. 1 in the cast state, (b) No. 1 in the heat-treated state; (c) No. 2 in the cast state, (d) No. 2 in the heat-treated state. The numbers indicate the various phases, determined by X-ray analysis: 1 – Al-Si; 2 – Al2Cu; 3 – Sn-Bi-Pb-Cd; 4 – Sn-Bi-Pb.

Nanoscale studies were performed using various methods of probe microscopy. SPM Dimension FastScan (Bruker). SPM studies of the alloys were carried out in the PeakForce Tapping QNM mode using standard silicon cantilevers of the NSC-11 type (MikroMasch) with a probe tip curvature radius of 29 nm and force constant of 99.72 N/m. SPM images of surfaces were obtained, and their local properties were investigated. At each point of the image, the probe approaches the surface of the sample with recording force curves. Obtaining and recording such curves is the basis of the PeakForce QNM mode, which automatically recalculates the values of mechanical properties (modulus of elasticity and adhesion), taking into account the characteristics of the probe used.
3. Results and discussion

3.1. The influence of heat treatment on the structure and elemental composition of the surface

For both samples, the structure of the initial surface of the samples and its change as a result of maintenance were investigated by the SEM method. Figure 1 shows the obtained micrographs of the surface of alloys 1 and 2 – in the cast state and after heat treatment.

The analysis of previous optical images (not presented here), Figure 1, and other SEM images shows high surface uniformity and the absence of macroscopic defects. It can be seen that despite some difference in composition (alloy No. 1 contains Cd), both alloys are similar in structure and contain similar phases. Earlier, for alloys of similar composition, it was shown that heat treatment led to spheroidization of individual phases and to partial dissolution of copper in the aluminium matrix [11]. In the present work, the SEM method also shows that heat treatment leads to similar changes. So, after maintenance, the elongated phase constituents of the alloy – silicon and low-melting phases – acquired a more compact rounded shape. By the nature of the change in the phase distribution on the surface, it can be seen that copper dissolves in the aluminium matrix.

3.2. Measurement of hardness

For both samples, the hardness values were determined. The measurement results are presented in Table 1 for cast alloys and after heat treatment. Cast alloys have hardness at the level of the base composition Al–5%Si–4%Cu, which is about 70 HB. After heat treatment, the alloys have a hardness of 125-130 HB, which is the same as in the alloy Al–5%Si–4%Cu–6%Sn. This indicator of hardness is not worse than that of the brand alloy AO3-7. The table shows that the values of hardness are almost the same for both samples.

3.3. The effect of tribological tests on the structure and elemental composition of the surface

When testing for wear, it was found that both alloys had a score at a pressure of 1.0 MPa, while alloy No. 2 had a higher wear resistance. Figure 2 shows the SEM images of the surface of contact pairs (shoe and rollers for two alloys) after tribological tests at a pressure of 1 MPa. To identify the phases and assess their distribution on the surface of the roller after testing (Fig. 2f), the distribution of chemical elements (ElementOverlay) is presented. In Table 2, the elemental composition of the surfaces of the shoe at different pressures (0.5 and 1 MPa) is shown.

Studies have shown that the topography and chemical composition of the surface after testing at various pressures are different from the parameters of initial surface. Thus, at minimum pressures, iron was found on the surface of a shoe made of an alloy with a lower content of low-melting elements (Table 2). This is due to the fact that, in view of the small amount of lubricating component, the solid phase components of the alloy act as an abrasive, causing mass transfer of iron from the steel surface.

Table 1. Hardness of the samples.

| Alloy | Cast | Heat-Treated |
|------|------|--------------|
| No.1 (Al–5%Si–4%Cu–4%Sn–0.5%Bi–0.5%Pb–0.5% Cd) | 73 ± 0.9 | 129 ± 1.6 |
| No.2 (Al–5%Si–4%Cu–4%Sn–0.5%Bi–0.5%Pb) | 71 ± 1.5 | 125 ± 1.2 |

Table 2. The chemical composition of the surfaces of the shoe (alloys No. 1 and No. 2) after testing.

| Pressure | Shoe-alloy | Concentration of elements (% mass) |
|----------|------------|-----------------------------------|
|          |            | O   | Al   | Si   | Cd   | Sn   | Fe | Cu | Pb | Bi |
| 0.5 MPa  | No. 1      | 0.59 | 85.36 | 6.98 | 0.32 | 2.74 | 0.16 | 3.42 | 0.29 | 0.14 |
|          | No. 2      | 16.76 | 65.54 | 4.89 | -    | 2.64 | 6.53 | 3.09 | 0.35 | 0.20 |
| 1.0 MPa  | No. 1      | 15.27 | 72.95 | 5.76 | 0.35 | 2.36 | 0.21 | 2.66 | 0.25 | 0.19 |
|          | No. 2      | 11.19 | 75.90 | 6.49 | -    | 2.61 | 0.21 | 2.97 | 0.43 | 0.20 |
Figure 2. SEM image (secondary electrons) of the “shoe-roller” contact pair after tribological tests at a pressure of 1 MPa: (a) shoe from alloy No. 1, (b) roller after tests of alloy No. 1, (c) mapping the surface of the roller after testing alloy No. 1 (blue – iron, red – aluminium); (d) shoe from alloy No. 2, (e) roller after testing alloy No. 2, (f) mapping of the roller surface after testing alloy No. 2 (blue – iron, red – aluminium). (b) and (c), as well as (e) and (f) correspond to the same areas.

Figure 3. SEM image (back-scattered electrons) of the surface of an oblique cut of the shoes after tribological tests at a maximum pressure of 1 MPa: (a) alloy No. 1, (b) alloy No. 2.

of the counterbody. At the same time, mass transfer of the shoe material also occurs on the surface of the roller, resulting in the formation of a film of secondary structures (blue phase in the mapping in Figure 2c,f). With increasing pressure, an unevenly applied film of secondary structures thickens, which leads to the development of macrorelief and may contribute to a score.
3.4. The effect of tribological tests on the change in the surface layers – study by SEM

For a more complete study of the processes occurring in the contact zone under friction, sections of the shoes were prepared after the tests, which made it possible to investigate the surface layers of the alloys. Figure 3 shows the SEM images of slices (sections, cuts) of the No. 1 and No. 2 shoes after tribological tests at a maximum pressure of 1 MPa.

When studying sections of all samples, it can be seen that in the near-surface region the structure is noticeably different from the structure in volume. Thus, phase components traced in the direction of friction (“tracks”) are traced on it. This is due to the fact that in the process of friction in the surface layers, the grains and dendritic cells of the aluminium matrix are deformed, leading to a change in the geometry of these components. The resulting images show that the differences between two alloys are small. The images allow us to estimate the thickness of the deformed layer: for both alloys, this is 20-30 μm (for pressures of 0.5 MPa) and 50-80 μm (for pressures of 1 MPa).

3.5. The effect of tribological tests on changes in the surface layers – study by EBSD

The use of the method of EBSD allowed obtaining additional results that demonstrate the process of deformation of the grains of the aluminium matrix in the surface layer after tribo-testing. Figure 4 shows, as an example, the results of this study for alloy No. 2 after testing at a maximum pressure of 1.0 MPa (the results of all the studied samples have a similar picture).

The images obtained can be used to estimate the average grain size in a volume of 20-80 μm. The colour of the grains in the resulting image characterizes their orientation; it can be seen that there are grains with different orientations in the volume. At the same time, analysis of EBSD paintings showed that it was not possible to identify grains and determine their orientation in a narrow (several microns) surface area and in a wider near-surface area. The reasons for this, obviously, is a strong distortion of the grains in these areas as a result of deformation during friction. At the same time, undistorted grains are clearly visible in the bulk layers of the alloy. The results obtained allowed estimating the thickness of the deformed near-surface layer, which was 50-100 μm (grey colour – indicated by an arrow – a homogeneous area, on which the grain structure is not detected). These results are in good agreement with the electron microscopy data.

3.6. Probe microscopy

The sections (cuts) obtained after tribological tests were studied by probe microscopy. Figure 5a,b shows the SPM images of the morphology of the cut-off surface of sample No. 2 after tribological tests (a – in the near-surface layer, b – in the volume). Figure 5c,d shows the corresponding images of the distribution map of elastic modulus.

![Figure 4](image_url)  
**Figure 4.** The EBSD map of the cut surface of the block (alloy No. 2) after tribological tests – areas with different colour intensities correspond to different grains. Arrow indicates narrow, deformed, near-surface area.
Corresponding to the data of Figure 5c,d, as well as intermediate areas, the numerical values of the modulus of elasticity are presented in Figure 6. Figures 5 and 6 demonstrate that the modulus of elasticity in the surface layer of the alloy is significantly higher than in the volume – 2.7 and 0.6 GPa, respectively (these values are relative and give a qualitative assessment of changes in parameters). This can be explained by the hardening of the alloy material in the process of friction. These measurements are relative and may differ significantly about the values obtained by nanoindentation. At the same time, they convincingly show surface hardening due to friction.

Figure 5. SPM image of the cut surface of the sample (shoe) from alloy No. 2 after tribological tests at a pressure of 1.0 MPa: (a) the morphology of the surface layer (the distance from the friction boundary is up to 20 μm), (b) the volume morphology (the distance from the friction boundary is 240-300 μm), (c) distribution map of the elasticity modulus of the surface layer, (d) distribution map of elastic modulus in volume.

Figure 6. The value of the elastic modulus at the shear block of alloy No. 2 at different distances from the friction surface boundary after tribological tests 1.0 MPa.
In general, a comparison of the results obtained for the two alloys shows that the addition of cadmium practically does not change the parameters under study. Note that earlier the effect of cadmium was found for alloys of the Al–Si–Cu system. In the present work, in the presence of other elements (Sn, Bi, and Pb), cadmium did not significantly affect the properties.

4. Conclusion
It was found that after heat treatment in both alloys, the phase components acquired a globular shape. So, after heat treatment, the elongated phase components of the alloy – silicon and low-melting points – acquired a more compact, rounded shape. It was shown that in the studied samples, copper in the process of heat treatment dissolved in an aluminium solid solution.

Analysis of the SEM image of the shoe showed that in the process of friction, the deformation of the grains, the extraction of the soft phase to the surface, and mass transfer occurred. In the study of the surfaces of the contact pair after tribological tests, it was established that the solid phase components of the alloy acted as an abrasive, contributing to wear and mass transfer. So, there is a transfer of iron from the steel surface of the counterbody to the sample (shoe) under investigation.

The study of sections of the shoe using SEM and EBSD allowed concluding about the formation of a surface layer (30–100 µm). In this layer, low-melting elements extract on the contact surface due to the deformation of the matrix grains. On the roller surface, mass transfer of the shoe material occurs, leading to the formation of a film of secondary structures. With increasing pressure, this unevenly formed film thickens, which leads to the development of macrolief and contributes to the score.

According to SPM studies, the value of the elastic modulus in the surface layer of the alloys is much higher than that in the volume. This can be explained by hardening the alloy material in the process of friction.

Hardness, tribological properties, and surface structure for both studied alloys are almost the same. It can be concluded that the addition of bismuth and lead contributes to the partial replacement of expensive tin. At the same time, the addition of cadmium does not change the operational parameters, i.e. the use of cadmium together with bismuth and lead for doping these alloys is impractical.

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