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STRUCTURE AND PROPERTIES OF THE AK12M2MrH (AlSi12Cu2MgNi) PISTON ALLOY FORMED UNDER THE INFLUENCE OF A COMPLEX MODIFYING ADDITIVE OF FULLERENE-CONTAINING SOOT AND COPPER

The article presents the study results of the complex modification of eutectic silumin АК12М2МгН (AlSi12Cu2MgNi) with additives of fullerene-containing soot (FCS) and copper. It is shown that the effect on the alloy structure is caused by the introduction of carbon nanoparticles into the melt and is manifested in the dispersion of the structural phases and their uniform distribution in the casting volume. At the same time, the use of dispersed copper powder provides wetting of aluminum carbon particles with the melt and additional alloying of the melt. The formation of a dispersed structure leads to an increase in the mechanical and tribotechnical characteristics of the alloy: an increase in the ultimate strength (by 1.3–1.6 times) with a simultaneous increase in the relative elongation by up to 3 times, a significant decrease in the coefficient of friction (by 1.1–1.7 times) and the intensity of wear. The lowest coefficient of friction and high wear resistance are achieved at small fractions of FCS (0.05–0.1 wt.%) and the copper content in the modifier is not more than 0.5 wt.%.

Keywords: *fullerene-containing soot, copper powder, modification, aluminum-silicon alloys, structure, ultimate strength, wear resistance, coefficient of friction*

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Introduction. Currently, there is a steady increase in requirements for the quality of cast products, which is largely determined by the properties of the alloys used. In recent years, the production and consumption of alloys of “aluminum — silicon” (silumins) systems have increased which serve as the basis for most cast aluminum compositions, which are widely used in modern mechanical engineering. Due to the structural features of cast aluminum-silicon alloys, such as grained brittle inclusions of silicon, large particles of iron-containing phases, dendrites of solid solution, the strength characteristics of silumins are relatively low, and especially ductility is low. Their modification remains the most effective factor determining their favorable structure formation [1–3].

Recently, ultradispersed powders of chemical compounds, which play the role of additional crystallization centers, have been actively used as modifiers of foundry alloys. Thus, in [4], comparative data on the use of Al_2O_3 as a modifier with a particle size of 25 μm and 40 nm are presented. According to the data, the powder was introduced into the melt at a temperature of 750 ºC by mixing for 7 minutes. The authors show that in both cases there is an increase in strength, but the nanoscale modifier has a more effective impact on the structure and properties of the LM25 (AlSi8) alloy. Therefore, nanoscale powder modifiers are of considerable interest. In [5], the authors present data on the modification of the A356 (AlSi7) alloy by nanoscale particles of tungsten carbide (WC, 40–70 nm), titanium carbide (TiC, 20 nm) or silicon carbide (SiC, 60 nm). According to the results, the introduction of 0.03 wt.% TiC provides dispersion of the alloy structure, which is accompanied by an increase in its hardness and strength with a simultaneous increase in viscosity by 20–50 %. Similar results were obtained by modifying the A356 (Al356) alloy with nanoscale WC and SiC particles.

A number of studies indicate that the complex modification with carbide-corundum powder provides a more intense effect on the structure and properties of the alloy. Thus, it was shown in [6] that when 1 wt.%. SiC (40 nm) is introduced together with 1 wt.% Al_2O_3 (20 nm) there is an increase in the ultimate strength, stiffness and viscosity by 48, 45 and 51 %, respectively, in comparison with the initial alloy, and by 25, 22 and 40 %, respectively, in comparison with the single-component modification of 2 wt.% SiC [6]. The works [7–10] propose the use of composite nanostructured powders BN-AlN-AlB_2 [7, 8], TiC-α-Al_2O_3 [9], and SiC-α-Al_2O_3 [10] synthesized by gas-phase deposition in a reducing atmosphere of ammonia, hydrogen, and volatile forms of chloride compounds. It is shown that the introduction of these nanostructured modifiers together with aluminum micropowder leads to the dispersion of the silumin structure, and, as a result, a decrease in the friction coefficient and an increase in their wear resistance. However, the widespread use of nanoscale particles of high-melting compounds as modifiers of cast aluminum alloys is hindered by the low level of wetting properties of nitrides, carbides and oxides by molten aluminum, as a result of which additional processing of the modifier or the addition of auxiliary substances to its composition is required.

In this regard, carbon nanostructured materials are of great interest as a modifying additive. Thus, in [11], the results of modifying the AlSi8Cu alloy with a recycled nanostructured diamond powder are presented. According to the results, the modification led to the grinding of the structural components, as well as to the creation of conditions for the dispersion of intermetallides during the subsequent heat treatment of the alloy, which provides an increase in the physical and mechanical properties. Similar data are given in [12], according to which the introduction of graphite in the amount of 4 wt.% in the structure of the AlSi8Cu4 alloy leads to an increase in hardness, whereas in [13] it is shown that the introduction of graphite into the aluminum melt provides an increase in wear resistance by 2–2.5 times. In [14], a method for modifying aluminum-silicon alloys is proposed and a significant effect of carbon nanotubes (CNT) on the structure of silumins is noted. However, due to the low wettability of CNT by aluminum melt, the potential of this carbon material is not fully realized in this case.

In [15, 16], an improved method for modifying aluminum-silicon alloys with carbon nanostructured materials is proposed. Thus, it is shown in [15] that the use of copper powder in the modifier, which has a high affinity for both aluminum melt and carbon nanotubes, increases the efficiency of CNT when modifying the structure of the AlSi12Cu2MgNi alloy, which provides an increase in mechanical and tribotechnical properties.

In addition to carbon nanotubes, such a structural form of nanocarbon as fullerene is of considerable interest as a silumin modifier. Numerous studies show that due to their unique properties, fullerenes significantly increase the properties of polymers, composites and lubricating oils. However, there is no information about the use of this form of carbon for modifying silumins.

Objective of the work: to study the structural and phase state and tribomechanical properties of the AlSi12Cu2MgNi piston alloy formed as a result of its modification by fullerene-containing soot introduced into the melt together with a dispersed copper powder.

Materials and methods of research. As the base material, silumin AlSi12Cu2MgNi was selected (Si-11-13; Cu-1.5-3; Mg-0.8-1.3; Mn-0.3-0.6; Fe-0.4; Zn-0.2; Ti-0.05-0.2; Al-rem., wt.%).

As a nanocarbon modifier, fullerene-containing soot obtained at the Ioffe Institute (Saint Petersburg) was used. The content of fullerenes in the soot is 7 % (75±5 C60, 25±5 C70, 1–3 higher fullerenes).

The modification was carried out by introducing a pressing containing FCS and dispersed electrolytic copper powder (CSP-1, [17]) into the melt of the matrix alloy AlSi12Cu2MgNi at a temperature of 720–740 °C by the alloying basket method. Then, blanks were cast into a metal casting mold heated to 400 °C, from which samples were subsequently cut out to study the structure and properties of the alloy.

The structure of the samples from the modified castings, as well as the structure of the friction surfaces, were studied by metallographic analysis using XJM300 and MIM-8 microscopes.

Tribotechnical tests were carried out on a universal tribometer MFT-5000 (Rtec Instruments, USA) according to the scheme of reciprocating movement of the sample relative to a fixed steel counterbody (ШХ15 (102Cr6) steel, [18]), which was a bearing roller with a diameter of 3 mm. The sliding speed of the sample relative to the end surface of the roller was 0.12 m/s. The test was carried out at pressures p of 10, 20, 30 and 40 MPa. The test duration at each pressure was 100–120 minutes. The tests were carried out under conditions of boundary friction in the medium of industrial oil of the И-40А (I-40A) brand [19]. The test results were used to determine the coefficients of friction (f), mass wear Δm, and wear intensity I_q ($I_q = \Delta m/L$, where L is the friction path). The mass loss Δm of the samples was determined by weighing them on the VLR-200 analytical balance (Zavod “Gosmetr”, Russia). The hardness of the samples was determined on the universal hardness tester 2137 ТУ (2137 TU, OOO “TOCHPRIBOR”, Russia) at a load of 49 N. The microhardness of the castings was determined on the PMT-3 device (AO “LOMO”, Russia) at a load on the identer of 0.198 N.

Experiment results and their discussion. *Structure of the AlSi12Cu2MgNi alloy modified with fullerene-containing soot introduced into the melt together with copper.* The microstructure of the AlSi12Cu2MgNi piston alloy in the initial cast state has a dendritic

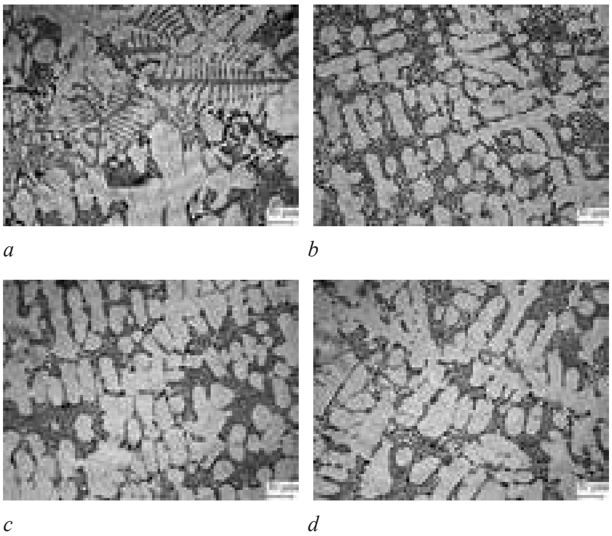


Figure 1 — Microstructure of the AlSi12Cu2MgNi alloy before (*a*) and after modification with a complex additive (*b–d*):
a — AlSi12Cu2MgNi; *b* — Q_{fcs} = 0.05 wt.%, Q_{Cu} = 0.25 mass.%; *c* — Q_{fcs} = 0.1 wt.%, Q_{Cu} = 0.5 wt.%; *d* — Q_{fcs} = 0.4 wt.%, Q_{Cu} = 2 wt.%

structure formed by the branches of the α-solid solution and the needle eutectic α+Si. In the body of dendrites, copper- and iron-containing intermetallic phases are mainly needle-shaped and skeletal, the size of which reaches 200 µm (Figure 1 *a*).

Modification of the melt with fullerene-containing soot, introduced together with copper, leads to a noticeable dispersion of its structural components. So, with the introduction of 0.05 wt.% FCS and 0.25 wt.% Cu there is a decrease in the grain size of the α-solid solution to 40–60 µm, dispersion of eutectic silicon particles (up to 6–10 µm), as well as a uniform distribution of eutectic in the casting structure (Figure 1 *b*). The morphology of intermetallic inclusions also changes, which are formed globularly or hieroglyphically with a particle size of up to 25 µm and are located mainly in the grain body of the α-solid solution, breaking it into smaller zones.

A more detailed analysis of the structure revealed the presence of three types of intermetallic inclusions: copper-, magnesium- and iron-containing inclusions (Figure 2, Table 1).

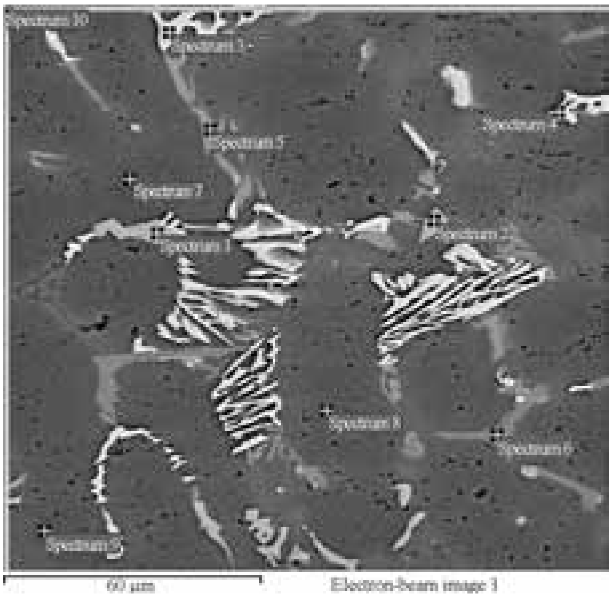


Figure 2 — SEM-image of the microstructure of a sample made of AlSi12Cu2MgNi alloy modified by 0.05 wt.% FCS with 0.25 wt.% copper

According to the data obtained, iron-containing inclusions crystallize in the form of polyhedra with a particle size of up to 20 µm, the composition of which is close to the compound $(\mathrm{Fe, Mn})_3\mathrm{Si}_2\mathrm{Al}_5$ (see Table 1, spectrum 1–2). Inclusions containing magnesium in their composition have the shape of needles, the length of which reaches 40–45 µm, and correspond to the compound $\mathrm{Al}_7\mathrm{Cu}_3\mathrm{Mg}_6$ (see Table 1, spectrum 5–6). The presence of silicon in the spectrum of the analyzed compound is due to the proximity of its particles in the studied area. Copper-containing inclusions have the form of colonies of needle-shaped and plate-shaped particles, the composition of which is close to the compound $(\mathrm{Cu, Ni})_2\mathrm{Al}_3$ (see Table 1, spectrum 3–4). It should be noted that iron- and copper-containing inclusions are mainly located in the eutectic area and at the interface, while needle-like compounds with magnesium are formed in the grain body of the α-solid solution, breaking the dendrites into smaller sub-grains.

Table 1 — Chemical composition of spectral points of a sample made of AlSi12Cu2MgNi alloy modified by 0.05 wt.% FCS with 0.25 wt.% Cu

Spectrum no.	C	Mg	Al	Si	Ca	Cr	Mn	Fe	Ni	Cu
1	32.81	—	37.88	5.68	—	1.10	6.54	10.15	3.05	2.78
2	31.32	1.58	39.23	5.56	—	1.16	5.82	8.79	3.18	3.37
3	26.58	—	27.93	—	—	—	—	—	11.00	34.49
4	25.87	—	33.34	0.36	0.14	—	—	0.30	11.80	28.18
5	30.68	21.06	16.77	18.25	—	—	—		0.83	12.40
6	29.37	12.42	35.34	15.03	—	—	—	1.84	5.00	1.00
7	33.02	—	65.66	0.84	—	—	—	—	—	0.47
8	32.84	—	65.81	0.88	—	—	—	—	—	0.46
9	31.66	—	67.20	0.66	—	—	—	—	—	0.49
10	—	1.31	83.44	9.37	—	—	0.28	—	1.74	3.86

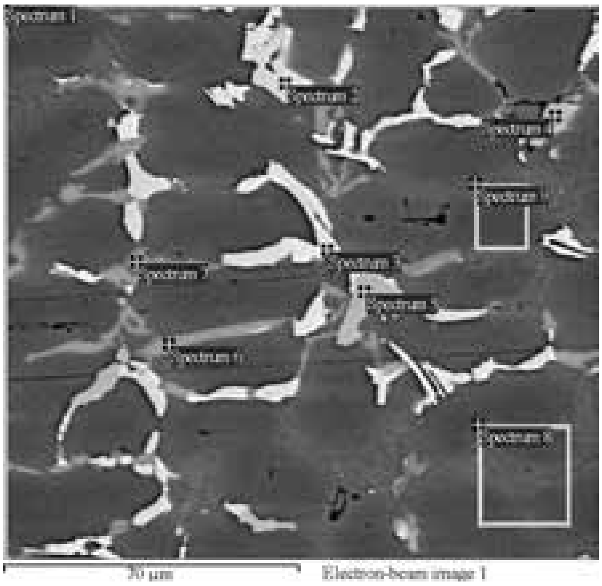


Figure 3 — Microstructure of a sample made of AlSi12Cu2MgNi alloy modified by 0.4 wt.% FCS with 2.0 wt.% Cu

An increase in the proportion of the introduced composite additive by a factor of 2 (0.1 wt.% FCS + + 0.5 wt.% Cu) leads to the formation of a fine eutectic with a particle size of Si up to 8 µm, while the size of the α-Al dendrites increases slightly, while the intermetallic particles do not acquire visible changes (see Figure 1 *c*). Some deterioration of the structure parameters of the AlSi12Cu2MgNi alloy is associated with a low rate of assimilation of the modifying additive by the melt when using mechanical mixing technology, so when a larger amount of the modifier is introduced, the formation of agglomerates from carbon particles and their transition to a slag layer occurs.

A further increase in the mass fraction of the introduced additive to 0.4 wt.% FCS + 2.0 wt.% Cu leads to the grain enlargement of the α-Al solid solution, within which intermetallic compounds are formed (see Figure 1 *d*). The eutectic also undergoes changes, which is expressed in an increase in the size of Si particles up to 25 µm.

The observed turning point of the dependence of the size of the alloy structural components on the amount of FCS additive can be associated with the process of agglomeration of carbon nanoparticles, which is intensified with an increase in their concentration. When a large amount of modifying additive (more than 0.1 wt.% FCS) is mixed in a small volume of the melt, the concentration of carbon nanoparticles per unit volume increases, which, being at a small distance from each other and having a high surface energy, form clusters or agglomerates. As a result of agglomeration, the modification effect is reduced, while some of the agglomerates are pushed into the slag layer by the lifting force (the particle density is lower than the melt density).

According to the results of scanning electron microscopy, the introduction of an additive of FCS with copper in the amount of 2 wt.% leads to additional alloying, increasing the Cu content to 3–3.5 wt.%. An increase in the copper content causes a change in the morphology of such copper-containing inclusions as $(Cu, Ni)_2Al_3$ (Figure 3, Table 2, spectrum 2–3). In this case, the intermetallic particles are located along the grain boundaries, have an elongated shape with rounded boundaries. At the same time, the alloy structure retains a certain proportion of inclusions in the form of colonies (see Figure 3).

The structural phase analysis of samples from the modified alloy made it possible to establish that the copper, which is part of the additive, almost completely passes into the melt, carrying out its additional alloying. However, studies have shown that the process of dispersing the alloy structure is due to the introduction of carbon nanoparticles into the melt, since the use of only dispersed copper powder does not lead to the achievement of the obtained result. On this basis, it can be assumed that copper performs the function of a catalyst when introducing carbon particles. Presumably, when copper particles enter the melt, it dissolves in the boundary area of the aluminum alloy, which leads to the formation of a liquid layer saturated with copper. According to the Al-Cu state diagram, an increase in the copper content in the aluminum melt reduces its melting point and increases the wetting parameters. As a result, carbon particles fixed on the surface of copper are wetted with supersaturated aluminum melt and introduced into the melt. The structure of copper particles is dendritic (Figure 4), which contributes to the retention of

Table 2 — Chemical composition of spectral points of a sample made of AlSi12Cu2MgNi alloy modified by 0.4 wt.% FCS with 2.0 wt.% Cu

Spectrum no.	C	Mg	Al	Si	Cr	Mn	Fe	Ni	Cu
1	3.18	1.60	79.21	9.39	—	—	—	0.97	5.66
2	10.56	—	41.40	0.75	—	—	—	1.17	46.12
3	10.90	—	37.38	—	—	—	—	22.79	28.92
4	9.28	0.33	52.03	7.94	0.77	10.11	10.89	4.22	4.43
5	8.07	—	51.78	8.02	0.92	10.21	11.19	4.76	5.05
6	9.38	27.28	22.59	24.00	—	—	—	0.44	16.32
7	10.59	24.63	24.19	24.16	—	—	—	0.75	15.68
8	4.78	0.26	73.15	20.66	—	—	—	—	1.14
9	2.75	—	90.74	4.96	—	—	—	—	1.56

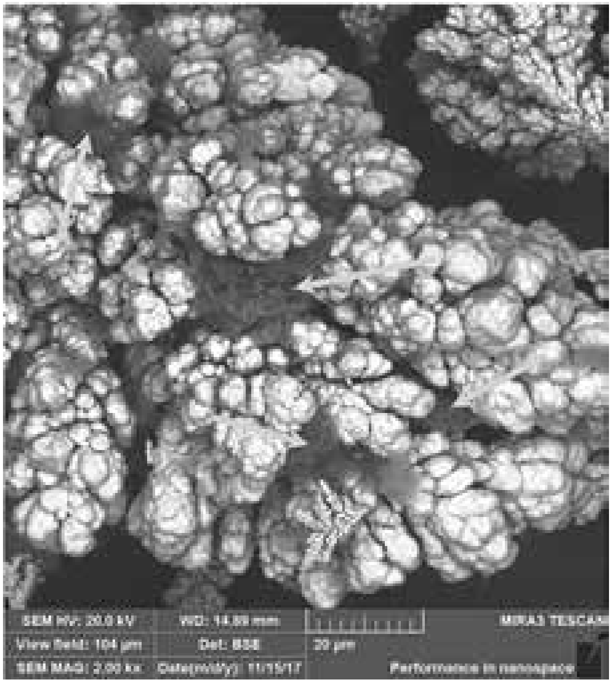


Figure 4 — Structure of copper particles with carbon particles fixed on the surface

carbon particles (shown by arrows) on the surface and their further transfer to the melt.

The results of the durometric analysis have shown that the introduction of a modifying additive in a small amount, 0.05 wt.% FCS with 0.25 wt.% Cu, leads to an increase in the hardness of the alloy (Table 3), while an increase in the microhardness of the structural components is observed with the introduction of 0.1 wt.% FCS and more. With the introduction of 0.4 wt.% FCS together with 2.0 wt.% Cu, the microhardness of the α-Al solid solution and eutectic increases by 57 and 234 MPa, respectively, which provides an increase in the hardness of the alloy by 230–240 MPa. It should be noted that the increase in hardness is also associated with a uniform distribution of iron-containing inclusions, the microhardness of which reaches 1.3–1.4 GPa.

The results of testing the mechanical characteristics revealed a tendency to the alloy hardness grow with an increase in the proportion of the introduced modifier and a decrease in the strength parameters. Thus, according to the results of the tensile testing of the samples, the modification of the alloy of 0.05 wt.% FCS with 0.25 wt.% Cu leads to an intensive increase in the values of the ultimate strength and percent elongation to 345 MPa and 7.4 %, respectively. With a further grow in the mass fraction of the modifying additive, the strength parameters decrease, which may be associated with the formation of carbon agglomerates.

Tribotechnical properties of the AlSi12Cu2MgNi alloy formed under the influence of a modifier containing FCS and copper. Tribotechnical tests of samples modified with the addition of FCS and copper showed that the dispersion of the structure and the alloy mechanical properties buildup lead to an increase in tribotechnical characteristics. The highest effect is achieved with the introduction of 0.1 wt.% FCS with 0.5 wt.% Cu. In the specified concentration of the additive, the friction coefficient is reduced by 1.1–1.3 times at a pressure of 10, 20 and 40 MPa and by 1.6–1.7 times at a pressure of 30 MPa (Figure 5). In this case, the highest wear resistance of the samples is also provided (during the test period, no mass loss is recorded in the entire pressure range). At the same time, the wear resistance of the steel counterbody is increased by 4 times (Figure 6).

The friction coefficient grow when modifying the alloy of 0.4 wt.% FCS together with 2.0 wt.% Cu, in comparison with the original alloy, may be due to its additional alloying with copper, which leads to the formation of a large number of copper-containing inclusions with high hardness in the structure.

Metallographic studies of the friction surfaces of the samples (Figure 7) revealed the presence of scratches and discoloration on the sample made of unmodified AlSi12Cu2MgNi alloy, while the surface of the sample modified by 0.1 wt.% FCS with 0.5 wt.% Cu has traces of more uniform wear without pronounced scratches and discoloration.

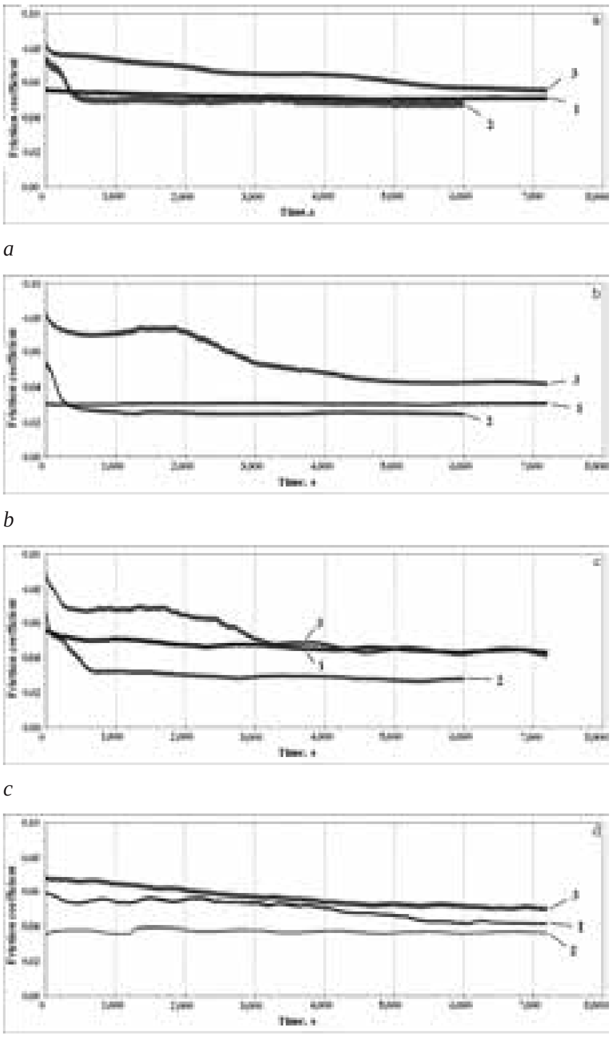
Increase in the mass fraction of the modifying additive to 0.4 wt.% FCS and 2.0 wt.% Cu leads to a decrease in the wear resistance of the sample and the steel counterbody by 2–3 times in comparison with the sample containing 0.1 wt.% FCS with 0.5 wt.% Cu (see Figure 6). This may be due to the formation in the latter case of an alloy structure with intermetallic inclusions of hieroglyphic shape, as well as the appearance of large eutectic silicon particles with a size of up to 15 μm (Figure 1 *d*). The formation of such a structure also affects the value of the friction coefficient, which has increased values over the entire test range of 10–40 MPa (see Figure 5).

Conclusion. The structural and phase changes in the AlSi12Cu2MgNi alloy are studied which occur

Table 3 — Mechanical properties of the AlSi12Cu2MgNi alloy after modification with a composite additive of FCS with copper

Sample	Q_{FCS}, %	Q_{Cu}, %	HV, MPa	σ_B, MPa	δ, %	H_μ, MPa	
						α-Al	eutectic
AlSi12Cu2MgNi	—	—	883	213	2.5	617	849
1	0.05	0.25	997	345	7.4	609	850
2	0.1	0.5	1,040	316	3.8	668	838
3	0.4	2.0	1,120	285	3.4	674	1,083

when the alloy is modified with a composite additive that includes fullerene-containing soot and copper. It is shown that the use of a copper micropowder with a developed particle surface ensures the introduction of carbon nanoparticles into the melt due to a local increase in the wetting parameters of the aluminum melt. It is established that the introduction of FCS with copper by mechanical mixing provides dispersion of the alloy structure and increases its tribomechanical characteristics. The ultimate strength grows by 1.6 times with an increase in ductility up to 3 times. The highest tribotechnical characteristics are achieved when the alloy is modified with the addition of 0.1 wt.% FCS with 0.5 wt.% Cu. In this case, the coefficient of friction is reduced to 1.7 times compared to the original alloy with a multiple increase in wear resistance.



a

b

c

d

Figure 5 — Change in the friction coefficient of samples from the test time at different pressures: *a* — 10 MPa; *b* — 20 MPa; *c* — 30 MPa; *d* — 40 MPa; curve 1 — AlSi12Cu2MgNi; curve 2 — 0.1%FCS+0.5%Cu; curve 3 — 0.4%FCS+2%Cu

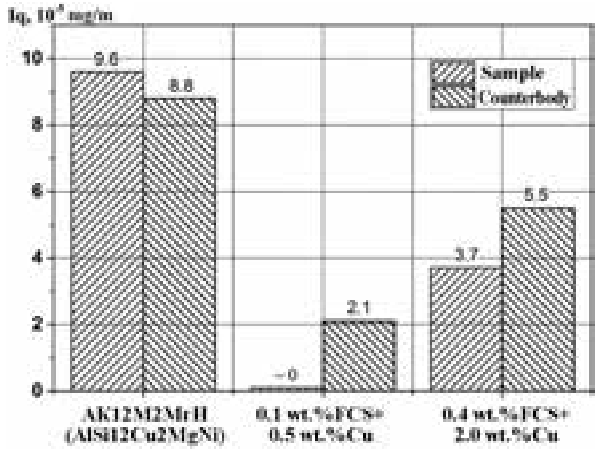
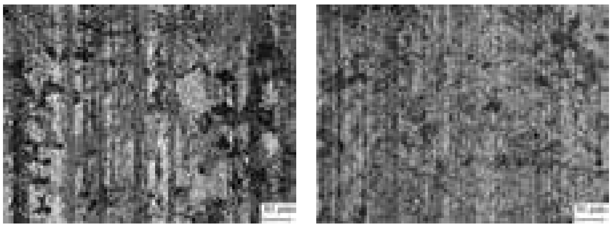


Figure 6 — Wear rate of the sample and counterbody before and after modification of the AlSi12Cu2MgNi alloy with the addition of FCS with copper



a *b*

Figure 7 — Microstructure of the friction surfaces of samples made of AlSi12Cu2MgNi alloy before (*a*) and after (*b*) modification with the addition of 0.1 wt.% FCS + 0.5 wt.% Cu

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СТРУКТУРА И СВОЙСТВА ПОРШНЕВОГО СПЛАВА АК12М2МгН, СФОРМИРОВАННЫЕ ПОД ВОЗДЕЙСТВИЕМ КОМПЛЕКСНОЙ МОДИФИЦИРУЮЩЕЙ ДОБАВКИ ФУЛЛЕРЕНСОДЕРЖАЩЕЙ САЖИ И МЕДИ

В статье представлены результаты исследования комплексного модифицирования эвтектического силумина АК12М2МгН добавками фуллеренсодержащей сажи (ФСС) и меди. Показано, что воздействие на структуру сплава обусловлено введением углеродных наночастиц в расплав и проявляется в диспергировании структурных фаз и их равномерном распределении в объеме отливки. При этом использование дисперсного порошка меди обеспечивает смачивание частиц углерода алюминиевым расплавом и дополнительное легирование расплава. Формирование дисперсной структуры приводит к повышению механических и триботехнических характеристик сплава: повышению предела прочности (в 1,3–1,6 раза) с одновременным увеличением относительного удлинения до 3 раз, существенному снижению коэффициента трения (в 1,1–1,7 раза) и интенсивности изнашивания. Наиболее низкий коэффициент трения и высокая износостойкость достигаются при малых долях ФСС (0,05–0,1 масс.%) и содержании меди в модификаторе не более 0,5 масс.%.

Ключевые слова: *фуллеренсодержащая сажа, порошок меди, модифицирование, алюминий-кремниевые сплавы, структура, предел прочности, износостойкость, коэффициент трения*

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