RESEARCH OF OBTAINING OF COMPOSITE MATERIALS BASED ON ALUMINUM MATRIX

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Abstract

In this work was studied the processing of aluminum alloys (silumins) by ligatures containing various carbon modifications (in the form of microcrystalline graphite, nanocarbon additives in the form of fullerenes, fullerene soot, fullerene niello) when casting-deformation technology of the manufacturing of products. In work were studied the elemental and phase composition, structural state and mechanical and tribological properties of the initial components of the charge in the Al-C and Al-Si-C after its mechanical activation alloys after severe plastic deformation (extrusion) of the charge and cast aluminum workpieces after processing by ligatures. Gradually studied the processes of structure formation of alloys in the system Al-C and Al-Si-C when they are received and thermomechanical loading. This of particular interest is the formation of superhard carbon phases in ligatures, where instead of microcrystalline graphite was used nanocarbon additives. Using spectroscopy of combined light scattering was revealed that these amorphous phase, similar to glassy carbon. In ligatures identified also carbides of aluminum Al_4C_3 and/or silicon SiC (during annealing (800° C, 30 minutes) to 10-12% carbides). Such structural state of alloys obtained when the activation of the charge (mechanical activation in dispersive devices and at intensive plastic deformation) determines the prospects of their use as additives providing not only dispersed hardening, but also the modification of the alloy when creating composites characterized by high anti-friction, plastic and strength properties.

Keywords: fullerene niello, fullerene soot, casting, deformation, Al alloy

Introduction

In today's world is scheduled trend toward increasing research in the field of development and wide using of metal-matrix composite materials. The cheapest and most reliable are materials based on aluminum alloys subjected to modification and reinforcement by dispersed refractory particles. Among the distinctive properties of such materials are high-friction and strength properties, heat resistance, hardness, low density, which provide substantial weight reduction products and designs, reduce material consumption of the product while increasing reliability and increasing their lifetime.

The need to create new composite aluminum material (CAM) and technologies of their production was explained by obtaining of competitive products and the situation of the gradual depletion of natural raw materials. In this case, the components of the CAM must be available and cheap. From this perspective, the composition of the surface of the earth's crust contains up to 50% SiO_2, 30% Al_2O_3 and 10% Fe. For this reason mankind in recent years, more intensively develops the production of Al_2O_3 for production of aluminum and materials based on it.

The use of graphite in antifriction alloys based on aluminum gave new impetus to the development and implementation of technologies aimed at the replacement of scarce and expensive bronzes used in friction pairs. Despite the progress achieved in this field, it is necessary to note the main disadvantages of such materials: high gas saturation of alloy, low strength and wear resistance of the resulting products, and impossibility of using this method for obtaining products of complex design.

Therefore, the goal of the present research is to improve the wear resistance of articles made of silumin and their strength by grinding the material structure and its stabilization during subsequent plastic deformation and heat treatment.

In accordance with the research objective in this work was evaluated the possibility of replacing expensive fullerene by cheaper fullerene material. To obtain the developed composites were used casting-deformation technology (technology-in-suit), comprising mixing the powder components of the charge and conduct of mechanical activation of the mixture, extruding the charge with obtaining ligatures and obtaining composite materials based on aluminum matrix during casting.

The samples were prepared from aluminum powders with a particle size of the main fraction is 5-100 µm or crushed chips alloy AK9 and number of nanocarbon materials in the ratio of Al - 10 mass. % C in the original mixture.

As the carbon materials used:

- fullerene-containing soot, production of Institute Ioffe, St. Petersburg;
- fullerenes C_{60}, production of Institute Ioffe, St. Petersburg;
- fullerene niello, production of Institute Ioffe, St. Petersburg;
- carbon microparticles of size 3, 4, 9 µm, the production of ASBURY GRAPHITE MILLS, INC., USA.

To obtain the developed composite material as the base used alloy AL 25.

The melt was prepared in an induction furnace ISV 0,004. Ligatures containing 10 wt.% carbon was introduced into the melt AC at a temperature of 750-780°C, while the melting alloys amounted to 3-5 min. Number of input ligatures in aluminum melt was calculated from the condition 1 wt.% carbon composite. The temperature was controlled by a multichannel detector RMT 39D, connected to the PC.

The technology of obtaining ligatures included: mechanical activation processing of source materials in the planetary mill, the compacting in rigid molds and hot extrusion. Mechanically activated powders were compactional in tablets at P=450 MPa. Next, the tablets were extrudible at a temperature of 450-500°C With a drawing ratio ≥ 10 and received a ligature in the form of rods.

Mechanical activation treatment was carried out for 30-40 minutes at frequency of rotation of the Central shaft 400-600 rpm and the ratio of the mass of grinding bodies to the mass download of 20:1.

1. The results of research of the initial components of the charge

1.1 Aluminium powder

The samples were prepared from powders of aluminum with a particle size of the main fraction is 5-100 µm. Topographic images of the original powder of aluminum is shown in figure 1.

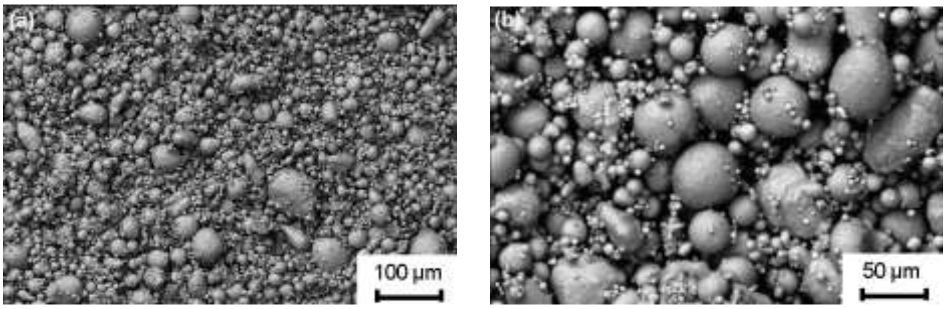


Fig. 1 Topographic images of the original aluminum powder

1.2 Fullerene soot

The results of the study of powder of fullerene soot in a scanning electron microscope is shown in figure 2. The powder consists of the dispersed soot particles and large particles of fullerenes.

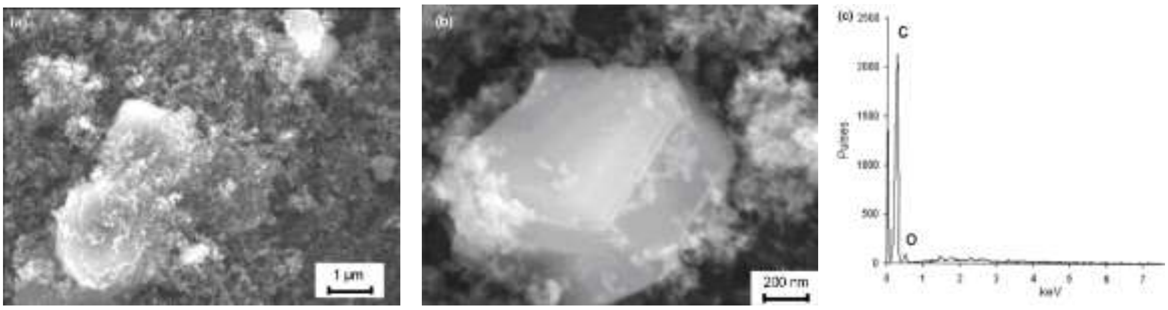


Fig. 2 Fine structure and diagram of content items fullerene soot

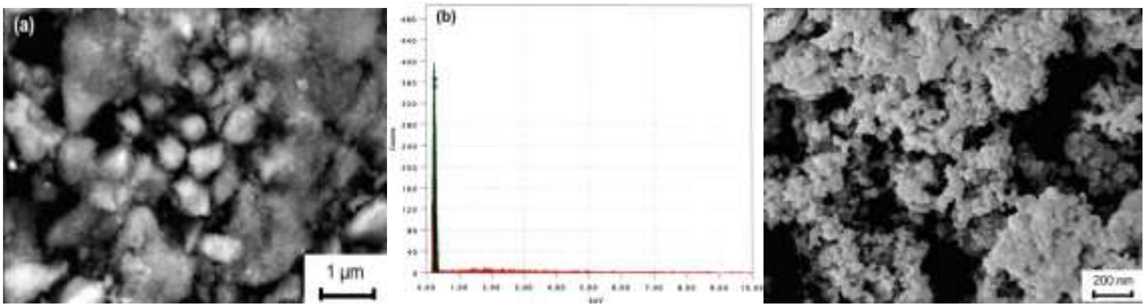
The results of the research phase and elemental compositions show that the investigated fullerene soot basically consists of amorphous carbon, contains ≈ 8% of fullerenes and contains also small amounts of oxygen, no impurities.

1.3 Fullerene niello

The powder consists of particles of small size from 3-5 µm and larger particles, but these are only conglomerates (figure 3a).

Studies have shown that fullerene niello is 100% black carbon, or any other impurities not detected (figure 3b).

In works [1-3] have reported that fullerene niello is a black fine powder with a particle size of 40-50 nm, which follows from the data of scanning electron microscopy (figure 3c).



a)image of the area where was done EDX analysis; b) graph of Counts-keV; c) image of the particles in SEM

Fig. 3 Fine structure and results of EDX analysis of the powder of fullerene niello

1.4 Microcrystalline carbon

The results of the study of microcrystalline carbon powders of different dispersion showed that the carbon particles have both kind of plates, scales, typical for hexagonal crystal structure and micro-particles of spherical shape. Figure 4 shows the topographic images of the microcrystalline carbon.

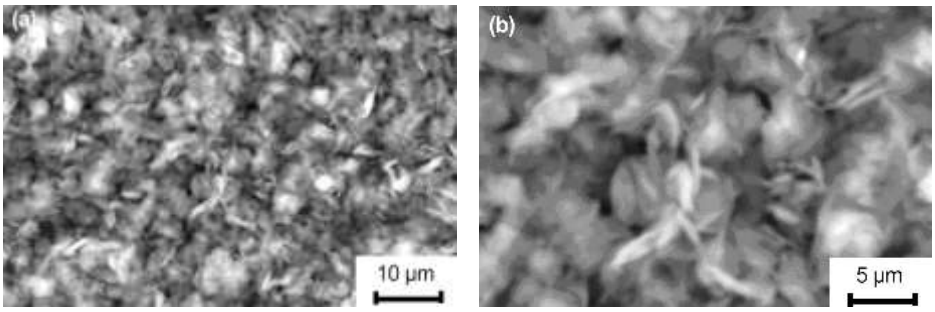


Fig. 4 Topographic images of microcrystalline powder of carbon

2. The results of studies of the charge after its mechanical activation

Studies have shown that in the charge of the system Al-C after mechanical activation there are processes of plastic deformation of the initial powder components and the observed changes of the elemental and phase compositions. Thus obtained topographic images of the charge powders with different carbon additives are similar. Figure 5 shows topographic images of powder charge Al + 10% fullerene soot.

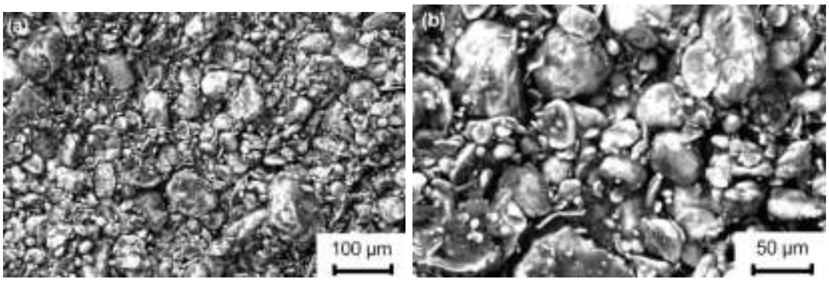
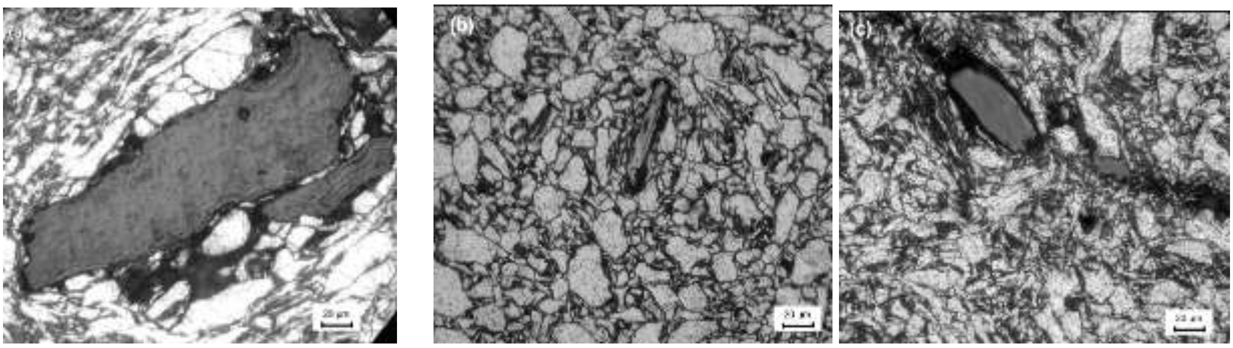


Fig. 5 Topographic images of powder charge Al + 10% fullerene soot after mechanical activation

3. The results of studies of the compositions of Al-C after extrusion of the charge

At this stage of research was conducted to study the structural state obtained by extrusion of ligatures. In the samples revealed unusual for Al-C alloys, superhard grey particles of various modifications (figure 6). Measurements of microhardness of the phases detected the effect of restoring the imprint of the indenter, indicating its very high elastic properties. Micro-x-ray spectral analysis EDX showed that this superhard phase is carbon (figure 7).



a) Al + 10% fullerene niello; b) Al + 10% fullerenes C_{60}; c) Al + 10% fullerene soot

Fig. 6 The microstructure of the samples ligatures Al-C

In the microstructure of several samples (especially in the series with fullerene niello) particles was observed gray phase with wavy surface (globular relief), without any traces of grinding and polishing (figure 6a), having a very high microhardness: the prints of the indenter on the image are not visible, fingerprints moving out from the particles, leaving the crosses chipped. This behavior of the phase in the measurement of microhardness indicates that their hardness close to the hardness of diamond.

This phase contains all manufactured samples with nanocarbon additives (figure 6). The analysis showed that the size, shape and number of extra hard pure carbon phase with high elasticity are various in ligatures of different compositions.

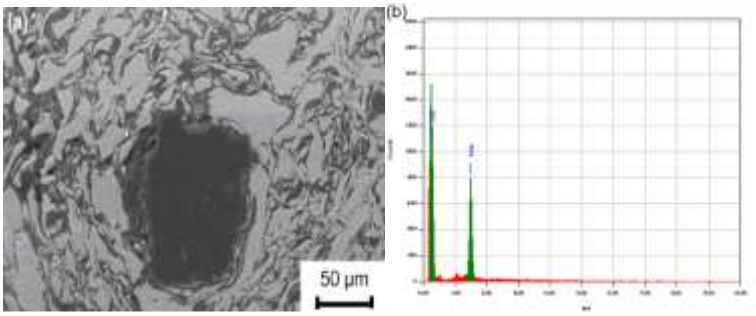
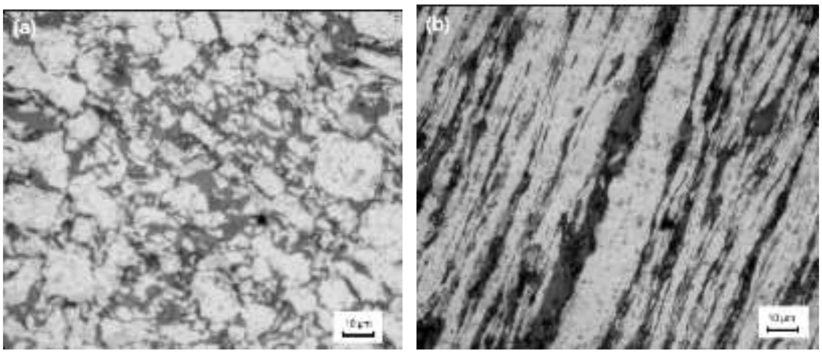


Fig. 7 Fine structure and results of micro-x-ray spectral

Table 1 Results of EDX analysis of ligatures Al + 10% fullerene niello

Element	(keV)	mass%	Error%	At%	K
C	0.277	85.64	0.89	93.05	70.5855
Al	1.486	14.36	0.49	6.95	29.4145
Total		100.00		100.00	

Analysis of the results of the study of the structural state of the samples of compositions of Al-microcrystalline carbon after extrusion of the charge showed a uniform distribution of the carbon component (black and grey inclusions) in an aluminum matrix (figure 8). While the small size of the carbon inclusions do not allow measure of microhardness, which makes it impossible to identify them as superhard carbon phases, which were obtained in the case of nanocarbon additives.



a) cross section; b) longitudinal section.

Fig. 8 The microstructure of the ligature sample Al + 10% microcrystalline carbon

4. The results of studies of aluminum alloys obtained by casting with the use of alloy AL 25 as the base and ligatures Al-C

At this stage of the research the was carried out study using light and scanning electron microscopy, x-ray diffraction and microprobe analyses, measurements of microhardness of the structural state of alloys obtained by casting with the use of alloy AL 25 as the base and ligatures Al-C, added to the melt at a rate of 10% of the total weight of the alloy.

The results of the study of the microstructure are shown in figures 9-13. The analysis of these studies showed that all composites of Al-C have modified structure of the metal substrate with distributed carbon structural component.

As it is seen from the figures, there are significant differences in the amount, structure and distribution of these carbon inclusions in the volume of the alloys obtained using different modifications of used carbon materials. In this case, all the samples of the composites obtained using microcrystalline carbon different dispersion have similar structure and distribution of carbon phases: the greatest, in comparison with the other samples, the number of carbon inclusions, mostly compact shape is close to spherical and low volume of the dispersed allocation (figure 10).

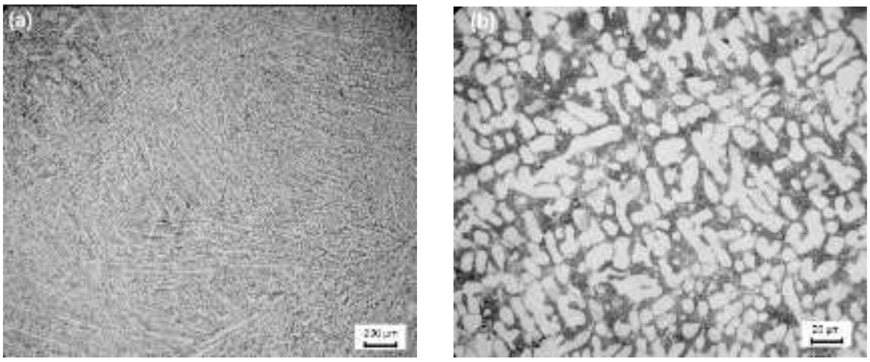


Fig. 9 The original microstructure of the alloy AL 25 after etching

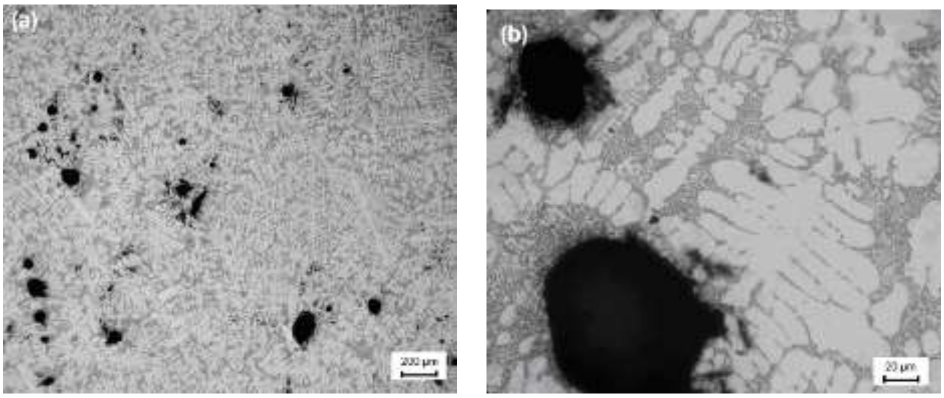


Fig. 10 The microstructure of the composite sample with microcrystalline carbon after etching

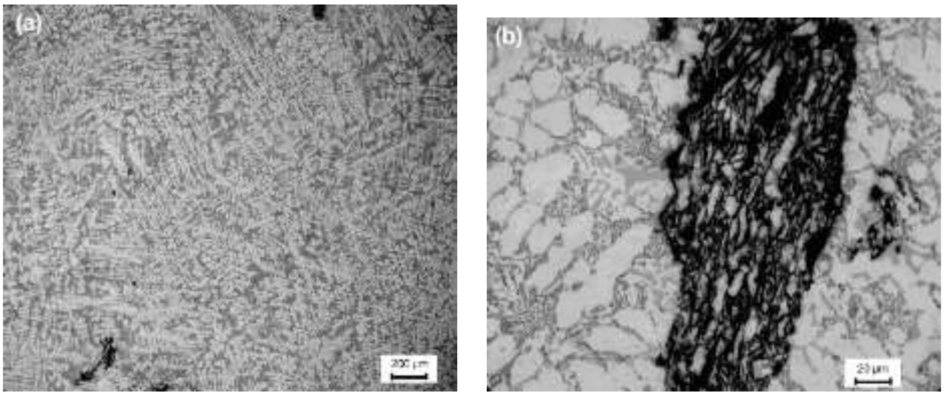


Fig. 11 The microstructure of the composite sample obtained using C_{60} after etching

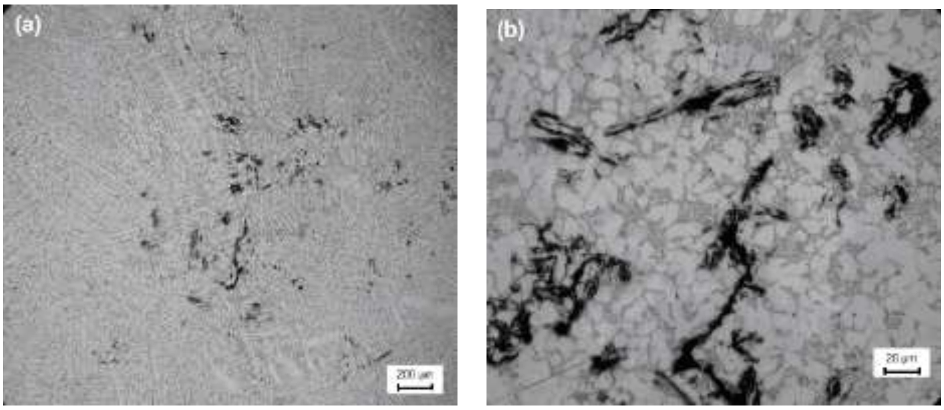


Fig. 12 The microstructure of the composite sample obtained with the use of fullerene soot after etching

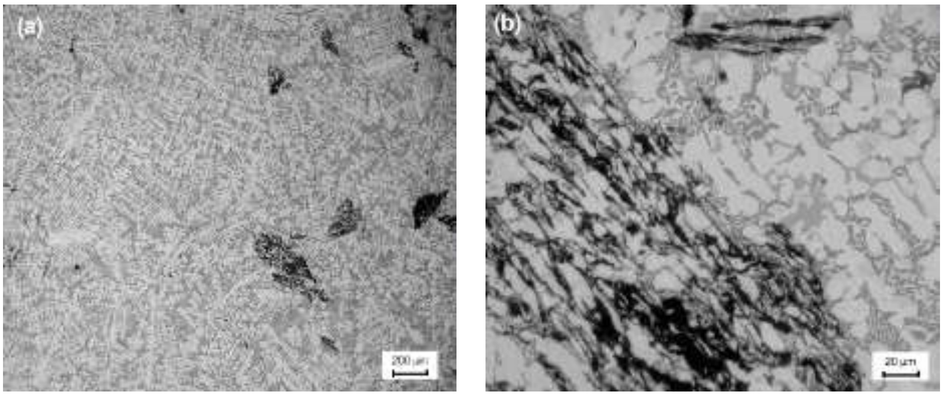


Fig. 13 The microstructure of the composite sample obtained with the use of fullerene niello after etching

On the other hand, the samples of the composites obtained using nanocarbon materials (fullerenes, fullerene soot and niello) also have similar structure and distribution of carbon phases: essential smaller, compared with samples obtained using microcrystalline carbon, amount of carbon dispersed inclusions, distributed individual colonies in a grid (figures 11-13). During overheating of the melt at 120-180° C above the liquidus temperature there is a complete absorption of the ligature and uniform distribution of components throughout the volume of the melt. This interval overheating of the melt also contributes to the solubility of the gases, which reduces the gas-saturation of the alloy. Overheating of the melt at a higher temperature, for example at 200° C, leading to exposure of dispersed particles of carbonaceous material on the surface of the melt, additional energy and reduced performance. Overheating at lower temperature, for example 100° C, does not ensure complete dissolution of the ligature and optimal homogeneity of the melt, which affects the properties of the workpiece. Thus, the obtained results define basic possibility of introducing in the structure of the aluminum alloy ultrafine carbonaceous raw material of used modifications and dispersion. The data, obtained in the measurement of microhardness of all samples of composites Al-C, show low hardness, hardness characteristic of graphite, carbon inclusions. In the structure was not observed carbon inclusions with high hardness obtained in the ligatures after extrusion.

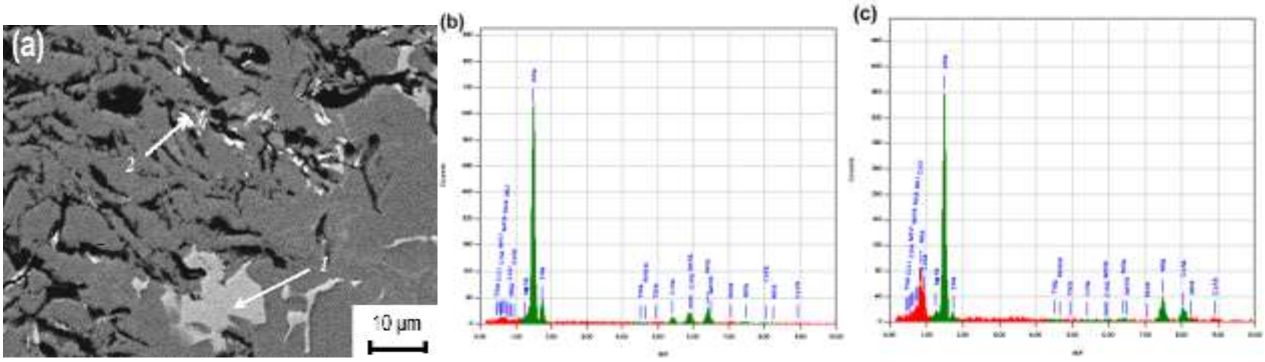
As shown above, all composites of system Al-C have modified structure of the metal substrate with a dispersion-distributed inclusions of intermetallic compounds (figures 9-13). According to the results of microchemical analysis identified 2 types of intermetallic compounds of different composition: intermetallic compounds with a high content of Fe and Mn (figure 14a) and intermetallic compounds with a high content of Cu and Ni (figure 14b). As showed results of studies of microhardness, intermetallic compounds characterized by significantly higher microhardness compared to the basis. Determining the true values of microhardness of intermetallic compounds in research was difficult due to their small size inclusions. However, were obtained hardness values at the level of 7000-8000 MPa (figure 15a), with hardness of the basics - 1000-1300 MPa (figure 15b). Thus, based on the above it can be concluded that the dispersed distribution of the above-mentioned intermetallic compounds has a hardening effect on the structure of composites Al-C.

Table 2 Results of EDX analysis at zone 1

Element	(keV)	mass%	Error%	At%	K
Mg	1.253	0.29	0.12	0.38	0.2305
Al	1.486	59.94	0.11	71.18	56.7050
Si	1.739	9.66	0.20	11.02	5.6332
Cr	5.411	4.18	0.26	2.57	5.2930
Mn	5.894	9.83	0.31	5.73	12.0902
Fe	6.398	13.36	0.32	7.67	16.7313
Ni	7.471	1.50	0.50	0.82	1.8505
Cu	8.040	1.24	0.65	0.63	1.4663
Total		100.00		100.00	

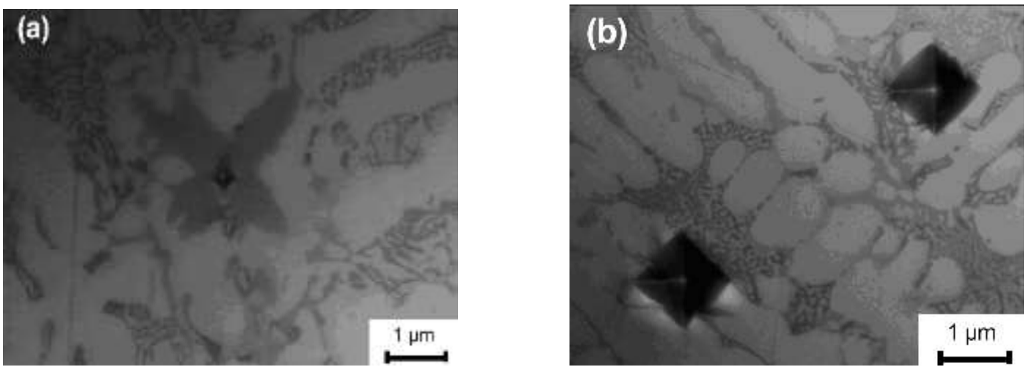
Table 3 Results of EDX analysis at zone 2

Element	(keV)	mass%	Error%	At%	K
Mg	1.253	1.62	0.20	2.37	0.9491
Al	1.486	53.24	0.17	70.01	38.9922
Si	1.739	1.75	0.25	2.21	0.9872
Cr K	5.411	0.23	0.29	0.16	0.3208
Mn	5.894	0.09	0.33	0.06	0.1277
Fe	6.398	1.33	0.32	0.84	1.9762
Ni	7.471	22.92	0.54	13.85	31.7825
Cu	8.040	18.81	0.70	10.50	24.8643
Total		100.00		100.00	



a) image of area where was done EDX analysis; b) results of micro-x-ray spectral analysis EDX at zone 1; c) results of micro-x-ray spectral analysis EDX at zone 2.

Fig. 14 Fine structure and results of micro-x-ray spectral analysis EDX of the composite sample obtained using fullerene niello



a) Hm=8310 MPa; b) Hm=1080 MPa, Hm=1200 MPa

Fig. 15 Microstructure of the alloy obtained using ultrafine raw materials, with imprints of the indenter

Conclusions

In this work was studied the processing of aluminum alloys (silumins) by ligatures containing various carbon modifications (in the form of microcrystalline graphite, nanocarbon additives in the form of fullerenes, fullerene soot, fullerene niello) when casting-deformation technology of the manufacturing of products, developed by scientists of the Republic of Kazakhstan and the Republic of Belarus in the framework of the state budget-funded theme on the program "Grant financing of scientific research in the Republic of Kazakhstan for 2013-2015".In work were studied the elemental and phase composition, structural state and mechanical and tribological properties of the initial components of the charge in the Al-C and Al-Si-C after its mechanical activation alloys after severe plastic deformation (extrusion) of the charge and cast aluminum workpieces after processing by ligatures.

Gradually studied the processes of structure formation of alloys in the system Al-C and Al-Si-C when they are received and thermomechanical loading. This of particular interest is the formation of superhard carbon phases in ligatures, where instead of microcrystalline graphite was used nanocarbon additives. Using spectroscopy of combined light scattering was revealed that these amorphous phase, similar to glassy carbon. In ligatures identified also carbides of aluminum Al_4C_3 and/or silicon SiC (during annealing (800° C, 30 minutes) to 10-12% carbides). Such structural state of alloys obtained when the activation of the charge (mechanical activation in dispersive devices and at intensive plastic deformation) determines the prospects of their use as additives providing not only dispersed hardening, but also the modification of the alloy when creating composites characterized by high anti-friction, plastic and strength properties.

The research results did not reveal fundamental differences in the structure formation of aluminum composites produced using expensive fullerenes, in comparison with the composites produced using cheap nanocarbon materials (fullerene soot, fullerene niello).

References

[1] Gusev A.I. *Nanomaterials, nanostructures, nanotechnology* (PHYSMATLIT, Moscow, 2009) – 416 p (in Russian).
[2] Lezhnev S, Kuis D, Volochko A, Shegidevich A, Svidunovich N, et al. (2015) Cast Aluminum Composite Obtained by Using Ultrafine Carbon Raw Materials. J Material Sci Eng 4:166. doi: 10.4172/2169-0022.1000166
[3] Violetta Andreyachshenko et al., 2015, Advanced Materials Research, 1095, 29.