The paper presents the investigation of the structure of a composite material based on aluminum modified by schungite carbon additives after thermal processing under high pressure conditions. It is shown that adding schungite filler makes aluminum grain growth slow and increases the microhardness of the composite material. In addition, the formation of the aluminum coating on the schungite carbon particles improves its interaction with the melted material and makes for the retention of the filler in the matrix. Process flow schemes for formation of Al-schungite composite material containing schungite within the range of 1.5-5 wt % are worked out. For a quantitative description of the fracture structure of the made aluminum composites, an original method of multifractal parameterization of structures is used.

Keywords: schungite carbon, modification, high pressure, thermal treatment, composite material

Introduction

A topical problem of today is to develop aluminum-based alloys with a high level of physical-and-mechanical and service properties, characterized by fine-grained structure. Conventional methods for improving physical and mechanical properties of composite materials (CM) and alloys used in the machine-building industry, reached their potential to a considerable extent. It is possible to improve properties of the materials through task-oriented formation of nanostructures in them, for example, by adding nanoscale modifying additives into the matrix [1]. Various ceramic nanoparticles as well as schungite carbon can be used as such additives. Schungite carbon is known to be effectively used as a modifying technologically active ingredient in the production of composite materials, including aluminum alloys [2]. For example, the impact of modified schungites on the structure and operational characteristics of the CM are evaluated in [3]. It has been shown that at the combination of components in the liquid phase, a complex use of discrete ceramic particles of silicon carbide and carbon in the form of graphite particles, fullerenes, or nanotubes produced by modifying processing of schungite rocks allowed to increase the wear resistance by 2-4 times and to reduce friction coefficients of the developed CM by (30-40) % to use them in their tribounits of various technical applications.

1. Problem Statement

To improve the interaction of the added modifiers with aluminum alloy, its uniform distribution in the matrix various methods are used. For example, in [4-6] the authors proposed and developed a fundamentally new approach to formation of reinforcing nanostructural multifunctional composite refractory ceramic fillers, effectively influencing the structure of aluminum alloys and their physical and mechanical and operational characteristics.
The developed principles of reinforcing fillers formation are in the purposeful modification of original micro- and nano-powders by reactionary active elements, as a result of which in-situ on the surface of the filler refractory compounds in nanodispersed state are formed. These compounds are in chemical affinity to the aluminum matrix. In addition, the primary ceramic micro- and nano-powders make for uniform distribution of the nanofillers in the composite.

On the other hand, the conditions in which the composite is formed, have a great impact on the structure and properties of both the matrix and the ceramic filler. In [7, 8] the authors studied the effect of high static pressure and modification on submicro- and micro-structural changes in schungite carbon. It was shown that at high-temperature annealing, followed by thermobaric processing under high pressure and temperature conditions, there is a transformation and fusion of the original schungite carbon globules resulting in crystallite size growth. The best combination of structural characteristics and strength properties are provided by integrated processing of schungite rock, including annealing in a reducing medium and thermobaric sintering under a pressure of 1.5 GPa. Thus, the use of modified nanostructural filler (schungite) in combination with a thermobaric treatment of CM under high pressure conditions significantly affect the structural characteristics of the aluminum alloy at micro-, meso- and macro-levels.

The work objective was to study the structure of the aluminum alloy, modified by the addition of schungite carbon, after thermal treatment under high pressure conditions.

2. The experiment routine

As a base of primary materials alloy, AP-4 aluminum powder was used, to which a schungite powder containing carbon up to 40% was added as a filler. In order to heighten the chemical affinity with the aluminum matrix, a thermochemical modification of the schungite filler had been previously carried out, that was its high-temperature treatment (annealing) at a temperature of 950°C in aluminum-containing compounds (aluminum chlorides) medium. Then, preparation of the reactionary charge on the of aluminum and schungite powders base was carried out by their mixing. 2 types of powder charges were prepared: aluminum-schungite and aluminum-schungite coated by aluminum (schungite+Al).

Schungite at the initial state without aluminum coating was added to aluminum powder in the amount of 5 wt %. The modified schungite was added to aluminum powder in the amount of 1.5 and 5 wt %. Then the powders were shaken in a mixing vessel for 2 hours. From the prepared powder, sample weights of 1g were weighed, which were tilted out into a special moulded container made of lithographic stone. The thermobaric treatment of the reactionary charges in containers was carried out in a high pressure apparatus called "anvil with a hole" under pressures of 1.5 h·Pa to 2.5 h·Pa and at temperatures of 800°C and 1100°C for 30 seconds. Heating of the samples was carried out by passing a direct electric current through the charge at quasi-hydrostatic compression under high pressure. On the resulting compact samples microscopic sections were made, which were then examined using optical microscopy. The material fracture surface was examined using AFM. Microhardness measurements were carried out by «Micromet-II» microhardness tester at indentation load of 50g.

For a quantitative description of the fracture structure of the made aluminum composites, the experimenters used an original method of multifractal parameterization of structures [9].

The most informative multifractal characteristics are generalized entropies (dimensions) Dq Renyi. They make it possible to estimate the thermodynamic conditions of formation of the structures under study, as well as effective quantitative characteristics of their uniformity f_q and regularity Δq [9, 10]. When being compared, larger D_q values correspond to more non-equilibrium conditions of the structures formation, greater f_q values correspond to more uniform distribution of unite elements of the considered structure in Euclidean space, covering the structure, and an
increase in $\Delta q$ for the series of structures under study indicates that their periodic component becomes more common.

3. The investigation results

The schungite powder after the thermochemical treatment appears as conglomerations in size of up to 100 $\mu$m, which consist of polyhedral aggregates in size of 0.5-1 $\mu$m [7]. The aluminium is distributed over the powder surface rather evenly, without forming clusters on the surface of schungite particles.

![Fig.1. The outside view of the schungite carbon powder after modification by aluminum: a - is a general view of the powder; b - is the image of schungite+Al grains](image)

The structural studies of CM made by adding 5 wt % of schungite filler without thermochemical treatment showed that the schungite in the material is in an aggregated state, weakly bound to the matrix and is easily scaled off when preparing microscopic sections.

As a result of studies of the surface of microscopic CM sections made by adding 5 wt % of schungite filler coated by aluminum (schungite+Al), it was found that their structure is quite heterogeneous. The schungite carbon particles gathered in conglomerates from a few to several tens of microns, Fig. 2 a, b.

The bond of the schungite inclusions with the matrix of CM formed at a lower temperature is rather weak, as well as in the case of the uncoated schungite. With a rise in treatment temperature, the filler retention strength in the aluminum matrix slightly grows, that makes itself evident in its lesser scaling off. Decrease in the schungite concentration to 1.5 wt % results in significant size reduction of the filler inclusions ranging from several tenths of microns to 10-15 microns. In this case the schungite powder distribution in the matrix is more uniform, Fig. 2 b.

With rise in heating temperature, the amount of large (5-10 $\mu$m in size) schungite carbon inclusions in the matrix decreased significantly, Figure 2 d. The material structure became more homogeneous. In addition, as metallographic analysis showed, the aluminum grain sizes in the case of treatment in high temperature area did not substantially grow as compared with the samples formed at lower temperature (10-30) $\mu$m in size.

This fact can be explained by the impact of high pressure and relatively short duration of heat treatment as well as by modifying effect of the filler on the composite structure. It was also found that with rise in temperature, the microhardness of CM increased for each of the sample group and exceeded the microhardness of the base material made without adding schungite by (1.5-2) times. The results of microhardness measurements are presented in Table 1.
Table 1 - Dependence of aluminum – (shungite + Al) CM microhardness on the filler concentration and sintering temperature under a pressure of 1.5 GPa

<table>
<thead>
<tr>
<th>MEASUREMENT NUMBER</th>
<th>MICROHARDNESS, 5 WT % SCHUNGITE + AL, KGF/MM²</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>AT 800°C TEMPERATURE</td>
</tr>
<tr>
<td>1</td>
<td>33,5</td>
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<tr>
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<tr>
<td>5</td>
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</tr>
</tbody>
</table>

Conclusion

Thus, modification of schungite carbon by aluminum makes for intensification of the chemical affinity and filler retention strength in the aluminum matrix. Reduction in the filler concentration in the reaction charge from 5 to 1.5 wt %, as well as increase in the thermal treatment temperature results in a significant reduction in the size of schungite carbon inclusions, meanwhile its distribution in the composite becomes more regular.
The grain size of the aluminum matrix of the CM made at different temperatures is within the range of 10-30 microns. It can be explained by the effect of high pressure and short duration of thermal action, as well as by the modifying impact of the filler on the composite structure. Besides, with rise in the temperature, the CM microhardness grows on average by 10% for samples with different concentration of schungite carbon and exceeds by 1.5-2 times the microhardness of the material made without the addition of schungite.

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