Features of Alloying and Heat Treatment of High-Strength Casting Aluminium−Copper Alloys

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There are studied the structure formation of the high-strength casting Cu–Al alloys. Providing high level of properties for such alloys is connected by maintaining the necessary Cu content and alloying special strengthening additives. The most effective additives are unfortunately toxic Cd or expensive Ag. It is proposed to remove such components from the composition of alloys. To provide the structure dispersion and an increasing of properties, other alloying and modifying components, in particular, Mn and Zr, should be used. There are developed the technological bases for obtaining master alloys Al–Mn and Al–Zr with applying the energy of electromagnetic fields and magnetodynamic equipment. Modes for adding made master alloys into experimental liquid high-strength casting Cu–Al alloy are worked. As result, it is provided the substantiate refining of the cast alloy structure. To provide further structural transformations and to increase the properties of the experimental alloy, as well as due to a change in its chemical composition, the modes of its heat treatment are clarified. The proposed mode is similar to T6 and includes two-stages' homogenization (annealing) under quenching in water and subsequent artificial ageing. The further development of research consists in determining

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the new alloying components for such alloys. These components should make a strengthening effect on the structure, promote properties' increasing, and, at the same time, are non-toxic and relatively cheap.

Key words: high-strength casting alloys, Al–Cu, magnetohydrodynamic processing, master alloys, refining, structure, heat treatment.

Досліджено структуроутворення високоміцних ливарних стопів Cu–Al. Забезпечення високого рівня властивостей таких стопів пов'язано з підтримкою необхідного вмісту Cu та леґувальними спеціяльними зміцнювальними добавками. Найефективнішими добавками є, на жаль, токсичний Cd або дорогий Ag. Такі компоненти пропонується вилучити зі складу стопів. Для забезпечення дисперґування структури та підвищення властивостей потрібно використовувати інші леґувальні та модифікувальні компоненти, зокрема Mn і Zr. Розроблено технологічні основи одержання ліґатур Al–Mn і Al–Zr із застосуванням енергії електромагнетних полів і магнетодинамічного обладнання. Розроблено режими додавання виготовлених ліґатур у експериментальний рідкий високоміцний ливарний стоп Cu–Al. В результаті забезпечується істотне модифікування структури литого стопу. Для забезпечення подальших структурних перетворень і підвищення властивостей дослідного стопу, а також за рахунок зміни його хемічного складу уточнено режими його термічного оброблення. Запропонований режим аналогічний Т6 і включає двостадійну гомогенізацію (відпал) із загартуванням у воді та подальшим штучним старінням. Подальший розвиток досліджень полягає у визначенні нових леґувальних компонентів для таких стопів. Ці компоненти мають надавати зміцнювальну дію на структуру, сприяти підвищенню властивостей і при цьому бути нетоксичними та порівняно дешевими.

Ключові слова: високоміцні ливарні стопи, Al–Cu, магнетогідродинамічне оброблення, ліґатура, модифікування, структура, термічне оброблення.

1. INTRODUCTION

As known, high-strength casting Al–Cu alloys are dispersion-hardening alloys. Their operational properties are determined mainly by the structure, morphology, volume fraction, and surface energy of the strengthening particles formed at aging of the supersaturated solid solution [1–4]. The basis of the structure of such alloys is α-solid solution of Cu in Al and a eutectic consisting of Al and $CuAl_2$. The decomposition of the supersaturated solid solution during the aging leads to the formation of a strengthening nanoscale metastable θ'-phase. At the same time, for the majority of such industrial alloys, the optimal Cu content is considered to be in the range of 4.5–5% by weight [5–11]. This is due to the fact that decreasing Cu content in the alloy below 4% by weight leads to a decrease in its strength at room tempera-

tures. Excessively high Cu content (more than 5.5% by weight) provokes excessive release of the $CuAl_2$ phase, which embrittles the alloy not only at 20°C, but also at elevated temperatures due to the development of diffusion ductility, which sharply reduces its heat resistance.

Additional alloying and microalloying of the Al–Cu alloys with elements with a low diffusion coefficient in aluminium helps to reduce the rate of development of diffusion processes, increases the stability of the finely dispersed θ′-phase at room and high temperatures and the stability of the fine structure at elevated temperatures, which leads to an increase in the tensile strength [1–4].

The most effective alloying additives are Cd and Ag. However, due to its extreme toxicity and danger to human health, the use of Cd in the production of alloys, as well as the operation of products made of such materials, is currently prohibited in almost all developed countries of the world, and the use of Ag significantly increases the cost of the alloy [12, 13].

Therefore, from a practical and economic point of view, it is more promising to use already known, more readily available and cheaper components of the alloy, primarily low-soluble transition elements (Mn, Zr, Ti) [2, 3, 14–18]. They form refractory intermetallides, which affect the nature of liquations in the alloy and prevent the migration of grain boundaries during the homogenization process [3]. At the same time, Zr performs not only a modifying function (as part of the intermetallide Al_3Zr), but also acts as a microalloying additive that is part of the solid solution, *i.e.*, it helps to improve both the strength and plastic properties of the alloy [3, 8, 9, 15–18]. Moreover, zirconium improves the casting and technological properties of Al–Cu alloys in the solid−liquid state and in the pre-crystallization period, in particular, by reducing the tendency of the alloy to form hot cracks [15].

In the course of earlier studies [19], the specialists of the Physico-Technological Institute of Metals and Alloys of the National Academy of Sciences of Ukraine, taking into account the trends in the use of high-strength casting Al–Cu alloys, modern environmental and economic requirements for their production [7–9], based on the use of energy of electromagnetic fields, proposed solutions to the main problems of technological processes for the preparation and casting of such alloys, in particular, as follow.

• Complex refining and magnetohydrodynamic (MHD) processing of the experimental alloy in liquid state in casting magnetodynamic installation made it possible to ensure a sufficiently high level of basic mechanical properties. Thus, the tensile strength of the experimental Al–Cu alloy was on average 80%, and the elongation was 2.5 times higher than the values required by the standards for such alloys containing strengthening additives—toxic Cd, which is prohibited for use in almost all industrial developed countries of the world, or expensive

Ag [19].

• At the same time, the level of the main technological properties of the experimental alloy—fluidity and propensity to form hot cracks—was ensured in accordance with the standards. This indicates the possibility of manufacturing from such alloys not only ingots and cast billets, but also thin-walled parts of complex configuration, obtained by various casting methods.

Priority areas for further research were also formulated, which are intended to ensure the development of the proposed solutions:

• to further increase the strength properties, it is necessary to consider the possibility of using strengthening additives—alloying (microalloying) and modifying components that can ensure the formation of the necessary alloy structure and achieve a high set of properties;

• it is necessary to study the peculiarities of introducing components into the initial melt (both in pure form and master alloys), including under conditions of electromagnetic influences;

• in connection with a possible change in the chemical composition of high-strength alloys, special attention should be paid to the issues of clarifying the modes of heat treatment of cast products in order to ensure the maximum strengthening effect.

2. EXPERIMENT PROCEDURE

2.1. Obtaining Experimental Master Alloys

It is known that the macro- and microstructure of modified alloys strongly depends on the microstructure of master alloys & ligature modifiers [15, 18]. In order to avoid the influence on the quality of experimental alloys of the used master alloys (having different chemical composition, obtained by different methods of smelting from different charge materials from different manufacturers, *etc*.), it was decided to manufacture the main master alloys (Al–Mn and Al–Zr) independently.

The Al–Mn master alloy was prepared in the magnetodynamic casting installation MDN-6A for aluminium alloys [19]. Al of technical purity and metallic Mn were used as the initial charge materials. The Mn content in the experimental master alloy was chosen for the following reasons:

• to eliminate the saturation of the melt with H_2 and its accelerated oxidation, excessive overheating of Al (more than 10% of its melting point, which is about 660°C) should not be allowed when Mn is added;

• since the Mn content in the most popular high-strength casting Al–Cu alloys is in the range of 0.35–0.80% by weight [10, 11], the Mn content of the master alloy should ensure that such values are relatively easy to achieve with relatively low weight and convenient for insert-

ing ligature portions into the alloy.

Therefore, it was decided to ensure that the Mn content in the experimental master alloy was about 4% by weight. In this case, according to the state diagram of Al–Mn [3, 4], the liquid ligature temperature will be no more than 710°C, which is about 50°C (or 7–8%) higher than the melting point of Al. In order to ensure the required residual content of Mn in the final alloy, it is necessary to add 0.12–0.25 kg of such master alloy per 1 kg of initial melt.

In the production of the Al–Mn master alloy, the main task was to ensure the rapid dissolution of manganese in aluminium with a slight overheating over the liquidus temperature, as well as the uniform distribution of Mn in Al. The magnetodynamic installation MDN-6A has wide range of capabilities to create various modes of melt circulation in its crucible and channels [19, 20]. To control the process of dissolution and stirring, it was necessary to develop a special device installed on the central mouth of the W-shaped induction channel of the magnetodynamic installation MDN-6A (Fig. 1).

For example, at the channel mouth, a graphite insert is installed under the melt level, inside which ceramic foam filter is installed, and Mn is placed on its surface in the form of pieces of 10–20 mm in size. It then passes through the filter, enters the central branch of the MDN-6A channel, exits through the lateral mouths of the W-shaped channel,
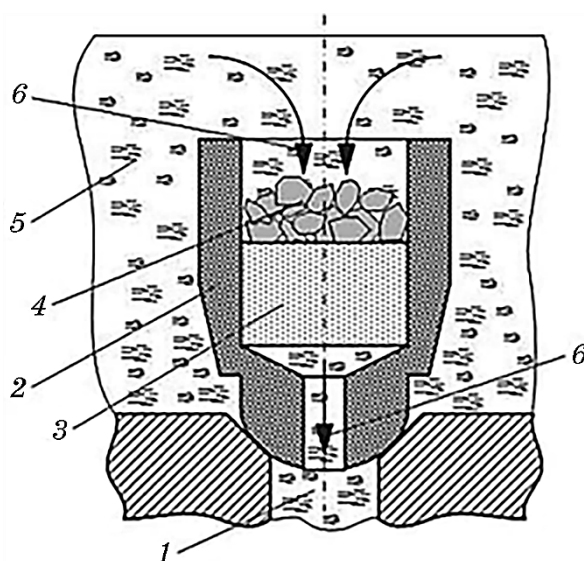

Fig. 1. Diagram of the introduction of metal Mn into Al when obtaining the Al–Mn master alloy: the mouth of the channel central branch (*1*), graphite body (*2*), ceramic foam filter (*3*), manganese pieces (fraction 10–20 mm) (*4*), aluminium melt (*5*), direction of melt movement (*6*).

mixes with the bath melt and passes through the device again. In this way, the melt is repeatedly circulated through the graphite insert with Mn along the 'liquid metal bath–channel' circuit. After complete dissolution of Mn, the master alloy was poured into ingots and further used for the preparation of experimental alloys.

As for the Al–Zr master alloy, in order to provide a strengthening effect on the structure of the processed alloy, it must contain zirconium in a solid solution with aluminium, and to modify effectively the structure, it must contain a sufficient amount of dispersed particles of Al_3Zr intermetallic compound. At the same time, the residual content of Zr in the Al–Cu alloys ranges from 0.10 to 0.40% by weight [8–11]. In industrial master alloys, the zirconium content reaches 85% by weight, but under normal conditions of melting and crystallization, the resulting intermetallides acquire a needle-like shape and a length of up to 150 μm (Fig. 2) [20]. In this form, they are not able to exert any effective modifying effect on the structure. In addition, the ligature oxidizes strongly at a temperature of about 400°C.

Therefore, in order to ensure intensive effect on liquid state of the Al–Zr master alloy, its preparation was carried out in a special crucible with a capacity of about 2 kg of aluminium melt. The crucible is equipped with a resistance-heating element (nichrome coil) with a maximum heating power of up to 2 kW. It is mounted on the pole of an electromagnetic stirrer to produce a master alloy under intense electromagnetic exposure (Fig. 3).

The preparation of the Al-master alloy was carried out as follows. In a crucible (see Fig. 3), technical purity Al was melted, overheated, and
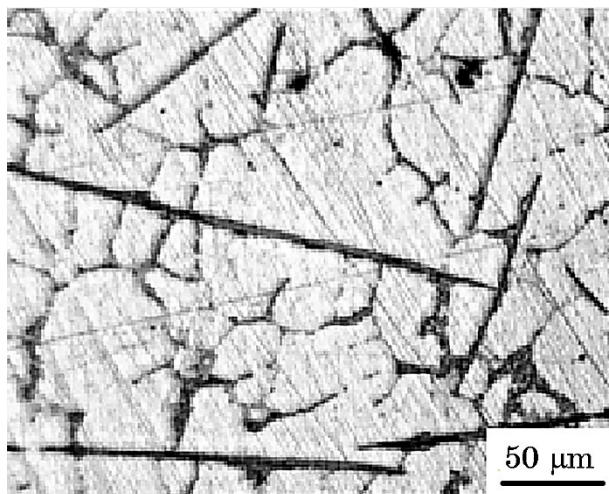

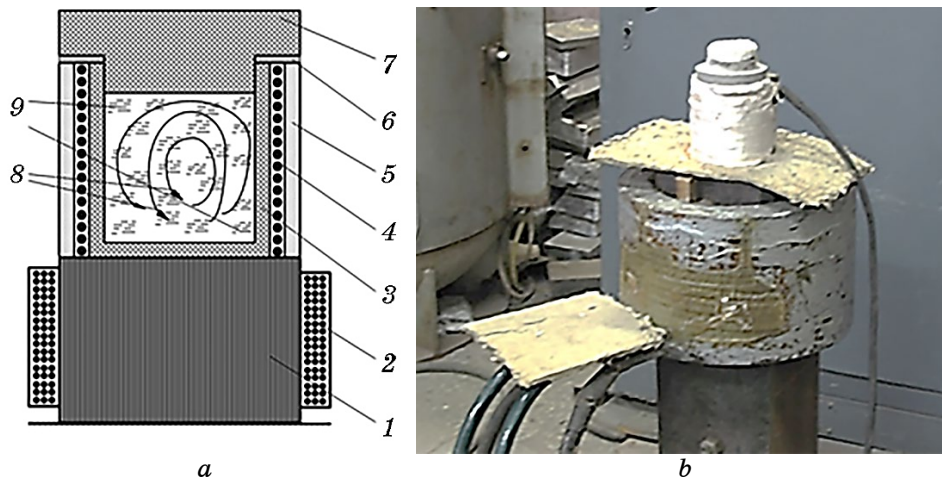Fig. 2. Microstructure of standard (Al–85% wt. Zr) master alloy [18].

Fig. 3. Diagram (*a*) and appearance (*b*) of the Al–Zr master alloy device: electromagnetic core (*1*), EMF winding (*2*), crucible (*3*), heating electro-resistance element (*4*), thermal insulation (*5*), sealing gasket (*6*), cover (*7*), direction of melt mixing (*8*), aluminium melt (*9*).

then an **(Al–85% wt. Zr)** master alloy was added. The choice of this fraction is due to the fact that the finer material is poorly immersed in liquid Al, floats on its surface and burns. Next, the electromagnetic stirrer was switched on and the intensity of melt stirring was substantially increased. After the complete dissolution of the solid pieces, the resulting melt was poured into metal moulds heated to a temperature of 200°C. Heating of the moulds is necessary to obtain a cast surface without irregularities. At the same time, the cooling rate of the cast billets was no more than 50 K/s. Thus, batches of master alloys with a Zr content of 3%, 5% and 6% by weight, respectively, were obtained.

2.2. Features of the Heat Treatment for High-Strength Casting Al–Cu Alloys

At present, the main methods of obtaining castings for machine-building purposes from high-strength casting Al–Cu alloys are die casting and sand moulds, while additional physical effects (centrifugal forces, electromagnetic fields, ultrasound, vibration, vacuuming of the mould itself, *etc.*) can be used to improve the filling of the mould, affect the casting structure during crystallization, *etc.* [8, 9, 21, 22].

After obtaining castings, depending on the required level of properties, they must be subjected to heat treatment according to various modes: T4—homogenization for hardening; T5—homogenization for hardening and short-term (incomplete) artificial ageing;

T6—homogenization for hardening and complete artificial aging; T7—homogenization for q hardening and stabilizing tempering [2, 10, 11, 18, 22]. The goals of such heat treatment are to eliminate intracrystalline liquation that occurs during dendritic crystallization, to control the processes of dissolution of components and separation of phases, to disperse the structure, *etc.* [23].

Standardized homogenization temperature values within (527–547°C) are based on the following considerations. The upper limit shall not exceed the melting point of the eutectic of 547°C to prevent contact reflow, and the lower temperature shall be above the temperature of 527°C, at which the ultimate solubility of Cu in a solid solution of aluminium is 5% by weight.

However, taking into account the changes in the chemical composition of the experimental high-strength casting Al–Cu alloy in comparison with standard alloys, the issue of refinement, development and possible adjustment of heat treatment modes becomes important and requires separate studies.

2.3. Research Methods

The studies were carried out using metallographic, x-ray, calorimetric, resistometric and durometric analyses. Phase transformations in alloys were evaluated using durometric, resistometric and calorimetric methods. Vickers hardness was measured with TP-2 at a load of 5 kg. Calorimetric analysis was carried out differential scanning calorimetry (DSC) method using NETZSCH 404 F1 Pegasus®, in dynamic mode at heating rate of 20 K/s in temperature range of 20–780°C. Temperature intervals of aging were determined by changes in the reduced temperature coefficient of electrical resistance $\alpha_T = R^{-1}dR/dT$ at continuous heating at a rate of 3 K/min in the temperature range of 20–600 °C.

Metallographic studies were carried out on the Neophot-2 optical microscope. For etching thin sections, Keller's reagent was used: HNO_3—2.5 ml, HCl—1.5 ml, HF—1 ml, H_2O—95 ml. The phase composition and structural state of the alloys were studied by x-ray diffraction analysis on the DRON-4 standard diffractometer with CuK_α-radiation.

3. RESULTS AND DISCUSSION

3.1. Structure of Produced Al–Zr Master Alloy

Metallographic studies of the obtained master alloy showed the following. In its structure, Al_3Zr intermetallides are observed, which have a
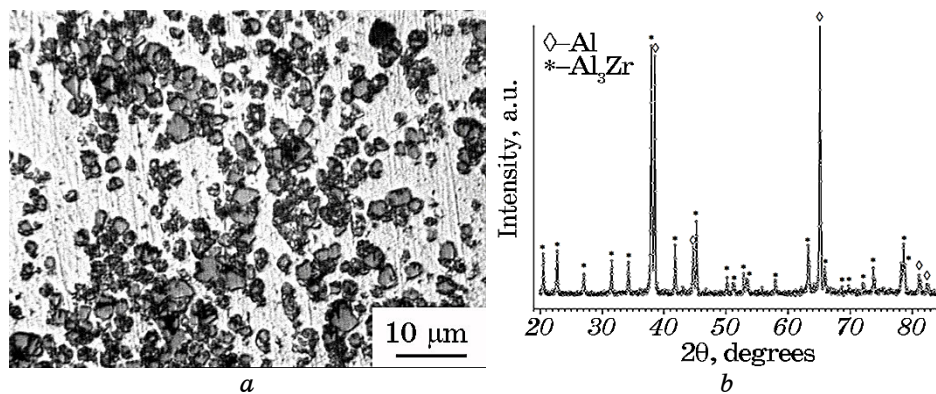
Fig. 4. Microstructure (*a*) and x-ray diagram (*b*) of the Al–5% wt. Zr master alloy.

compact shape and dimensions of predominantly 2–5 µm (Fig. 4, *a*), in contrast to needle-like intermetallides of standard master alloys up to 150 µm long (see Fig. 2). In general, the inclusions of intermetallides are evenly distributed over the volume of the sample. The diffractogram (Fig. 4, *b*) shows the presence of two phases—Al (64%) and Al$_3$Zr (36%). No peaks of other phases (*e.g.*, Zr, Zr$_3$Al, *etc.*) were detected.

It is important to emphasize that the resulting intermetallic compound is not a metastable phase, which is known to have a strong modifying effect. However, the stable phase obtained in the experiments should also be a good modifier, since it has the form of compact polyhedron with a predominant size of up to 5 µm. It is known from the theory of crystallization [5, 7] that coherent crystallization of an alloy containing a modifying particle in the form of a polyhedron occurs as follows. The addition of alloy atoms occurs first at the vertices of the polyhedron, then at its edges, and then at its faces. Since a single particle of intermetallide has many of these geometric elements, each of them creates many centres of crystallization (on average, their number is equal to the sum of the number of their vertices, edges, and faces).

3.2. Specification of Heat Treatment Modes for the Experimental Alloy

The previously obtained Al–Cu alloy [19] was used in further experiments. At the initial stage, in order to avoid metal losses and excessive energy consumption, as well as to test the efficiency of various options for MHD-processing, a scheme for small portions of the alloy was used (Fig. 3). In the alloy, which does not contain harmful (Cd) and expensive (Ag) strengthening additives, Zr was added with previously manufactured Al–Zr master alloy in modes without MHD-effects and with their application. As a result, an experimental high-strength casting
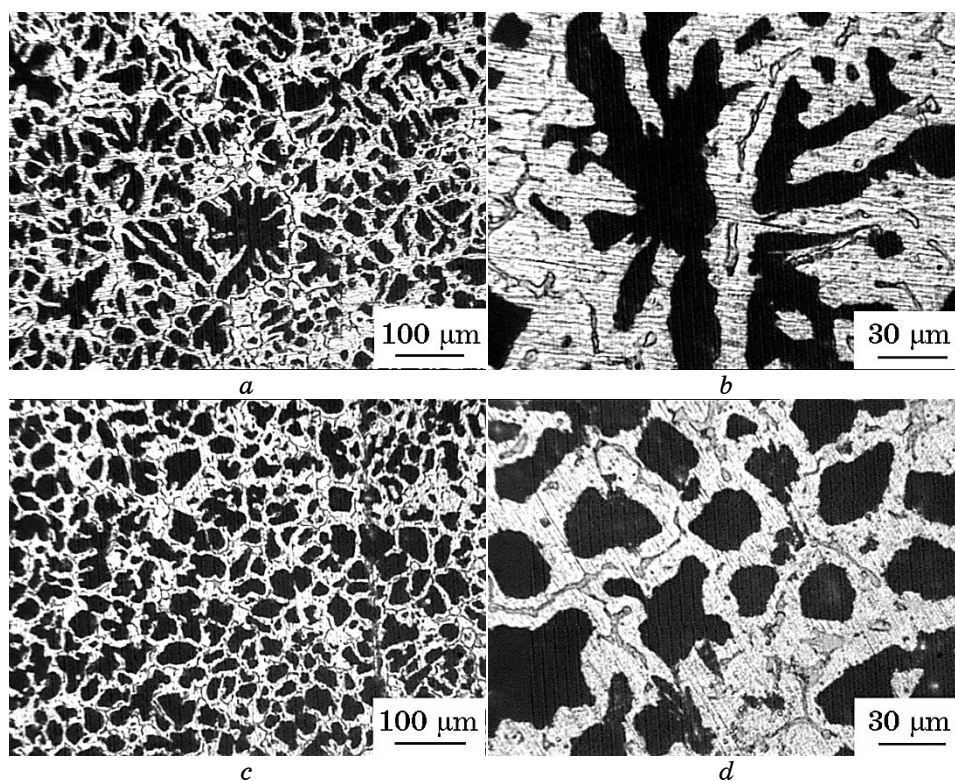
Fig. 5. Structure of the experimental high-strength casting Al–Cu alloy (in cast state): without external actions (*a*, *b*); under MHD-processing (*c*, *d*).

Al–Cu alloy was obtained with the following chemical composition, % by weight: Al—base; Cu—4.65; Mn—0.43; Ti—0.19; Zr—0.17; Si—0.18; Fe—0.06; Mg—0.01; Ni—<0.01; Zn—<0.01.

The microstructure of the alloy is shown in Fig. 5. It is absolutely identical to the structure of the initial casting alloy Al–Cu of a similar chemical composition (but without Zr) obtained in the magnetodynamic casting installation MDN-6A using electromagnetic actions and MHD-effects [19].

For example, in the absence of processing, the alloy in the cast state has a dendritic structure, and after MHD-processing, the dendrites acquire an almost globular cellular structure. This unequivocally testifies to the effectiveness of such an impact on the liquid alloy in order to control its structure and properties in the solid state, confirms the correctness of theoretical prerequisites and technical solutions, as well as the technological reproducibility of the results.

To eliminate the intracrystalline liquation that occurs during dendritic crystallization, the alloy was subjected to high-temperature an-
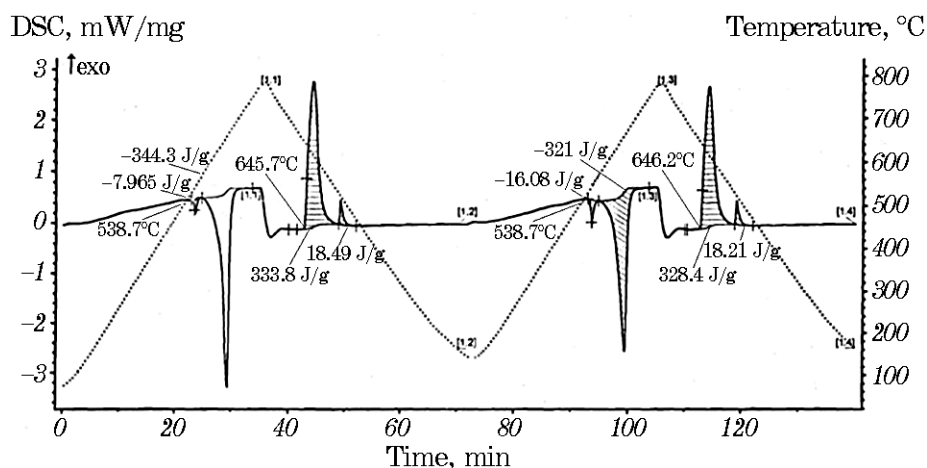
Fig. 6. DSC scans of experimental high-strength casting Al–Cu alloy registered during two heating and cooling cycles of the alloy in temperature range of 20–780°C.

nealing (homogenization for hardening). Studies of the structural state of the experimental alloy during homogenization under standard modes showed the presence of melting along the grain boundaries. In order to refine the homogenization modes using the differential scanning calorimetry (DSC) method, an analysis of changes in thermal effects over two periods of two years was carried out. Cycles of continuous heating and cooling of the alloy in the temperature range from 20°C to 780°C, and a low-temperature reflow peak at a temperature of 538.7°C was detected (Fig. 6).

As follows from metallographic studies (Fig. 7, *a*, *b*), after homogenization (annealing) for hardening according to the standard mode (535°C for 5 hours) the hereditary crystallization structure is preserved: the excess phase of Al_2Cu at the grain boundaries does not dissolve, the grain size does not increase, but melting is observed at the grain joints (black areas in Fig. 7, *b*). Transition to two-stage mode with an initial temperature increase to 545°C (Fig. 7, *c*) is accompanied by the dissolution of the Al_2Cu phase and the migration of grain boundaries. A new two-stage homogenization at low temperatures was proposed: annealing 510°C, 2 hours + 530°C, 10 hours, which made it possible to prevent melting along the grain boundaries (Fig. 7, *d*).

After homogenization, the samples were quenched into water. To characterize the degree of homogeneity of the solid solution and its ability to age in different annealing temperature ranges, the temperature coefficient of electrical resistance α_T at continuous heating at a rate of 3 K/min in the temperature range from 20°C to 500°C was measured. Resulting dependencies are given at Fig. 8, *a*.
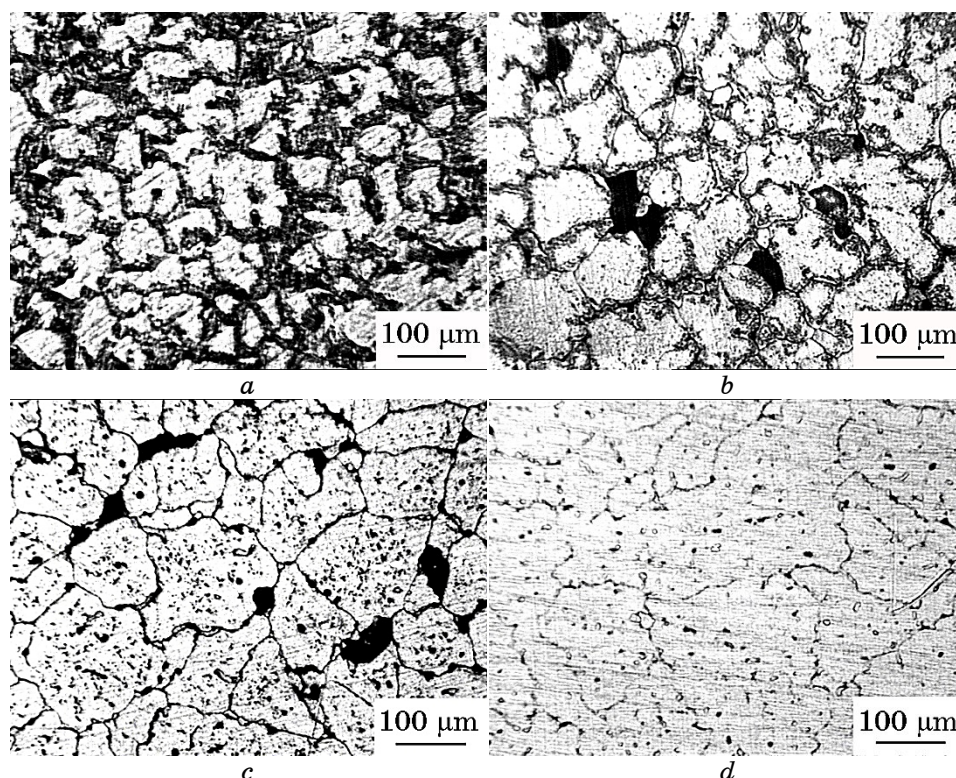
Fig. 7. Microstructure of experimental alloy: initial alloy (*a*); standard homogenization (annealing) at $T = 535°C$, 5 hours (*b*); two-stage standard homogenization (annealing) at 535°C, 5 hours + 545°C, 5 hours (*c*); proposed mode—two-stage homogenization (annealing) 510°C, 2 hours + 530°C, 10 hours (*d*).

After homogenization (at 510°C, 2 hours + 530°C, 10 hours), a minimum appears on the curve, which indicates supersaturation of the Cu matrix. The parameters of the peak, the temperature of the minimum, its width and amplitude characterize the kinetics of the release of phases of the Al_2Cu type during dynamic aging in the process of continuous heating.

For the selection of isothermal aging modes, isochrones were constructed in the temperature range of 100–230°C for 5 and 10 hours of aging (Fig. 8, *b*). The ascending branch characterizes coalescence processes, which are accompanied by a decrease in the density of Al_2Cu particles and, accordingly, a drop in strength.

Based on the analysis of the above data, the following heat treatment mode was proposed for further studies of experimental high-strength casting Al–Cu alloys: two-stage homogenization (annealing)
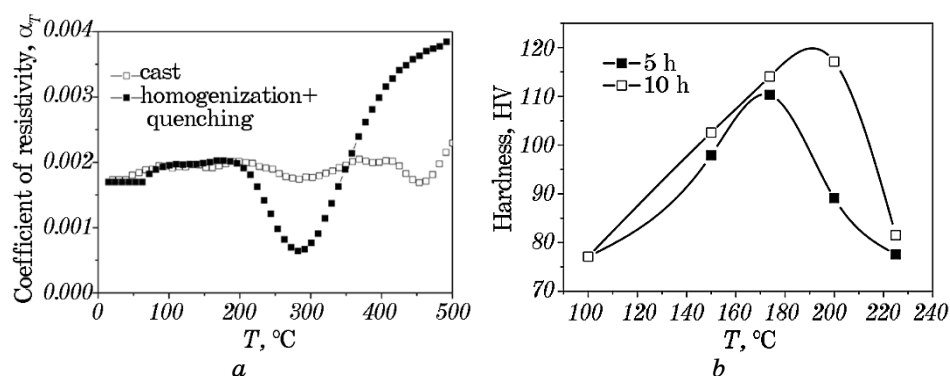
Fig. 8. Change in the temperature coefficient of electrical resistance of experimental high-strength alloy in cast and hardened states after homogenization (*a*) and hardness at isochronal ageing (*b*).

for hardening (510°C, 2 hours + 530°C, 10 hours), hardening into water from the homogenization temperature and subsequent aging at 190°C for 10 hours.

4. CONCLUSIONS

Thus, in the development of the initiated research, the technological foundations for the preparation of master alloys for the production of high-strength casting Al–Cu alloys, which do not contain harmful (Cd) and expensive (Ag) additives, were developed.

Due to some changes in the phase composition of such alloys, the modes of their heat treatment have been specified.

The purpose of further research is to select strengthening additives instead of Cd and Ag, which will significantly increase the strength properties of such alloys. It should be based on an approach that meets the following requirements: 1) the dispersed structure of the alloy already in the cast state; 2) high volume fraction of aluminides; 3) tendency to spheroidize (without coarsening) the structure during annealing.

Among the possible strengthening additives, based on world experience, the most interesting are transition metals, primarily rare earth metals (Sc, Y, La and lanthanides), as well as elements that are included along with Cd in the so-called 'triad' of low-melting and low-soluble elements in Al, namely Sn and In, which are able to modify the processes of nucleation and coalescence of the strengthening metastable θ'-phase.

The work was carried out within the framework of projects of competitive research programs of the National Academy of Sciences of

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