Investigation of the influence of heat treatment of aluminum alloy on its structure and properties

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Abstract. The paper considers the issue of improving the quality of aluminum alloys of the D16 type, based on the data performed at the Department of “Physical and applied materials science” within the framework of the contract on samples of the Belokalitvensky metallurgical combine. The influence of the composition and technology on the structure and mechanical properties was studied. A recommendation for optimizing the composition and structure was developed and based on the analysis of phase diagrams, the microstructures of the alloy were justified.

1. Introduction
This paper examines aluminum alloy D16 which is one of the most popular duralumin alloys in the shipbuilding, aviation and space industries. It has such characteristics as stable structure; high strength characteristics; 3 times lighter weight than steel products; good machinability on lathes and milling machines, second only to some other aluminum alloys. This will be considered in more detail based on the experimental part. Duralumin D16 related to aluminum alloys of the Al-Cu-Mg system, alloyed with manganese. Most of it is aluminum — up to 94.7%, the rest is accounted for by copper, magnesium and other impurities (Table 1). Manganese increases the corrosion resistance of the alloy and improves its mechanical properties, although it does not form common hardening phases with aluminum, only dispersed particles. [2].

Table 1. Chemical composition of D16 alloy according to GOST 4784-97 in bulk %

| Alloy grade | Si  | Fe  | Cu  | Mn  | Mg  | Cr  | Zn  | Ti  | Ni  | Other elements | Al  |
|-------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|----------------|-----|
| D16 (1160)  | 0,50| 0,50| 3,8-4,9| 0,3-0,9| 1,2-1,8| 0,10| 0,25| 0,15| —  | Ti+Zr≤0,2       | The rest |

D16 is a structural heat-strengthened and naturally-aged alloy in the billet, which is used in various areas of the national economy. It is also used for the manufacture of power elements of structures in aircraft: parts of the skin, frame, ribs, control rods, spars, etc. It is also used in the automotive industry for the manufacture of bodies, pipes and other fairly strong parts. D16 is used for manufacturing rivets with high shear strength [3].

In connection with the above, the purpose of this work was an examination of the effect of heat treatment on the microstructure and mechanical properties of aluminum alloy D16.
2. Material and experimental procedure
As mentioned above, aluminum alloy D16 was chosen as the research material. Experimental studies of the structure and mechanical properties were conducted in the laboratories of the Don State Technical University at the Department of Physical and Applied Materials Science.

The study used the original billet made of aluminum alloy D16. Two identical samples № 4 and № 5 (with the end mark “124”) in a non-heat-treated state (figure 1) was taken, cut from a D16 alloy ingot (the composition is shown in Table 1).

3. Results and discussion
The conditions for working with the sample should be set as follows. The holding time at the homogenization temperature should be from 4 to 18 hours. It is important to track the detection of inclusions. The structure of duralumin in the annealed state is an α-solid solution of copper substitution in aluminum, along the grain boundaries of which fine-dispersed CuAl2 or other secretions are located.

Six control samples were made, each of which was continuously kept in the oven for 4, 6, 8, 12, 16 and 18 hours without extraction. Samples were loaded into the furnace at the same time. The experiment was carried out in a chamber furnace SNOL 6.7/1300 at a temperature of 485±5.0°C. The temperature was chosen based on the Al-Cu diagram (Figure 2). Heating to lower temperatures does not provide maximum mechanical properties while heating to higher temperatures causes burnout, i.e. oxidation and partial melting of the alloy along the grain boundary, which sharply reduces the strength and plasticity. Therefore, the temperature value was chosen correctly. Temperature control was carried out not only by a temperature indicator of the furnace but also by a verified chromel-alumel thermocouple.

After the specified time intervals, one of the samples was removed from the furnace, cooled in air, and underwent the necessary sample preparation operations to study the structure, chemical and phase composition.

The change in the structure of samples depending on the holding time in the furnace is shown in Figure 3 (ZEISS optical metallography, etching with reagent № 3, the composition of which looks like h3po4-75ml, h2so4-15ml and hno3-10 ml). It is important to clarify that the etching lasted 3-5 minutes.

The main characteristic of all presented microphotographs is that all phase selections are located along the grain boundaries. The grain size does not change with increasing exposure in the oven and is 40...70 microns (there are also larger crystallites). Figure 3 shows photos with different degrees of optical contrast, so the samples with an exposure time of 6, 12 and 16 hours (2, 4 and 5 rows of double photos) all phase selections are identical, and the remaining samples (4, 8 and 18 hours of exposure) these selections seem to belong to different phases. The analysis of the chemical and phase composition will be presented later.
According to the visual estimates of Figure 3, it is quite difficult to draw even qualitative conclusions about the phase formation kinetics as the sample holding time in the furnace increases.

**Figure 2. Phase Diagram of Al-Cu**

![Phase Diagram of Al-Cu](image)

**Figure 3. Micrographs of Al-Cu**

(a) 100 μm  
(b) 20 μm  
(c) 300 μm  
(d) 20 μm
As you can see in Figure 3, as a result of heating, the main amount of CuAl₂ or other particles (the reducing phases will be described later) is dissolved in an α-solid solution. The D16 structure itself consists of a fine grain whose shape and size are symmetrical. If you compare the microstructures, you can see the difference in the density of grains throughout the entire volume of the alloy and the presence of etching pits. This is just what the picture shows.

Therefore, to make a more accurate assessment, a statistical analysis of the total area of phase secretions in the samples was performed using the computer program "KOI-structure analysis". Figure 4 shows the measurement data for the amount of dark grain boundary phase (% of the total image area) in the sample structure after 12 hours of exposure in the oven (Figure 3, h) using the KOI program method.

Three randomly selected optical microstructure images of each of the six control samples were analyzed using the given KOI method. The results of statistical processing have given a stable growth trend of phase grain boundary discharge, which is graphically shown in Figure 5. The growth rate of the grain boundary phase area in accordance with the experimental dependence of Figure 5 is approximately
0.7% per hour. This rate of growth is quite problematic to catch in the visual analysis of images (Figure 3). Moreover, the spread of the values of the area of phase allocations on a single microshliff, calculated in the KOI program, sometimes reached up to 40%. The difficulties of visual analysis are also related to the fact that the growth of the dark phase occurred only along the grain boundaries. In the studied samples, phase separation in the grain volume was not observed.

The elemental composition of inclusions in the test samples was studied by energy-dispersion analysis (EDAX) (performed using an energy-dispersion spectrometer based on the gas-free x-Mach detector of the company "Oxford instruments", mounted on a transmission electron microscope JEM-2100). Its results in the form of point probing (spectra) and a color map of the distribution of elements are shown in Figure 6 for a sample kept in a furnace during homogenization for 18 hours.

The results obtained for the remaining samples are not given since the morphology and composition of inclusions in them remained constant (only the volume fraction of inclusions changed during aging). In the dispersion-hardening (thermally strengthened) alloy D16, the following hardening phases may exist [1, 3]: CuAl₂, MgCuAl₂ and Cu₂Mn₃Al₁₀. According to Figure 6, there are two types of phases among the grain boundary secretions – copper-based and iron-based. Both of these phases are located in the intergrain space, adjacent to each other, but localized separately. This is clearly visible on the color map. Analysis of the obtained data allows to conclude that the composition of these phases is MgCuAl₂ and FeMnSi. Moreover, the dependence shown in Figure 5 on the growth of the grain boundary phase as it is aged in the furnace refers to the MgCuAl₂ phase, which "sprouts" along the grain boundaries due to the diffusion of alloying elements of the alloy from the grain volume. The number of FeMnSi phases does not depend on the duration of the homogenization process. Since Fe and Si are not part of the D16 alloy, the data obtained suggest that FeMnSi particles are exogenous inclusions (particles of flux, slag, modifier, lining, etc.) that were present in the melt.

Since FeMnSi particles are not isomorphic to the α - solid solution, they are displaced to the boundaries of growing crystallite-phase crystallites during crystallization. If the FeMnSi particles are isomorphic with MgCuAl₂, they can serve as a substrate for the crystallization and growth of this phase.
along the grain boundaries of the $\alpha$-solid solution during the homogenization process. This mechanism of growth of the MgCuAl$_2$ phase is fundamentally different from the mechanism of separation of dispersed particles of the CuAl$_2$ phase in the volume of grain $\alpha$-solid solution during aging. Thus, it is shown that the phase formation in the presented samples № 4-5 of the D16 alloy during homogenization occurs by forming the MgCuAl$_2$ phase by the mechanism of heterogeneous (on FeMnSi inclusions) grain boundary growth in accordance with the kinetics shown in Figure 5.

| The name of the spectrum | Mg  | Al  | Si  | Mn  | Fe  | Ni  | Cu  | The amount (% at) |
|-------------------------|-----|-----|-----|-----|-----|-----|-----|------------------|
| Spectrum 1              | 71,33 | 3,86 | 6,17 | 8,97 | 0,33 | 9,34 | 100  |
| Spectrum 2              | 73,92 | 5,1  | 6,65 | 9,01 | 0,24 | 5,07 | 100  |
| Spectrum 3              | 19,49 | 57,57 | 22,94 | 100  |
| Spectrum 4              | 8,12  | 66,41 | 0,83 | 0,22 | 24,43 | 100  |
| Spectrum 5              | 19,84 | 57,45 | 22,71 | 100  |
| Spectrum 6              | 13,46 | 68,92 | 0,08 | 0,25 | 17,29 | 100  |
| Spectrum 7              | 77,89 | 3,51 | 4,1  | 6,98 | 0,28 | 7,24 | 100  |
| Spectrum 8              | 6,81  | 64,6 | 0,14 | 0,32 | 28,14 | 100  |
| Spectrum 9              | 11,62 | 56,79 | 4,61 | 0,08 | 26,9  | 100  |
| Spectrum 10             | 8,76  | 60,06 | 31,18 | 100  |
Figure 6. Chemical composition of grain-boundary phase separations in the D16 alloy sample after 18 hours of exposure in the furnace, EDAX

4. Conclusions
Fairly wide and versatile information about the aluminum alloy D16-duralumin was obtained. Alloy requires exposure during the technological process as shown by the experiment the most optimal time is 4 hours or 8 hours, depending on what goal is pursued. One of the important conditions is the heating temperature at which no oxidation or melting of the surface should occur. There should also be no deformations as a result of mechanical processing. The experiment was successful which led to excellent results. The developed process which shows all the details D16 describes some regularities of changes in the pattern of the interaction structure, the possibility of determining the chemical composition of the grain boundary phase precipitates in the sample and the experimental dependence of the change % of the area of the micro section occupied grain-boundary phase, from the time of exposure.

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