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THE INFLUENCE OF MAGNESIUM ON PHASE TRANSFORMATIONS AND STRUCTURAL CHANGES PERFORMED DURING SUPERPLASTIC DEFORMATION OF THE ALLOY 01420T

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It is determined that the concentration of magnesium in the grain boundaries of the specimens of alloy 01420T is increased compared to its average concentration in the alloy and in the middle of the grain. Increased concentration of magnesium in the surface regions of the grains leads to their partial melting during deformation at high homologous temperatures. As a result, viscous liquid phase, which promotes implementation of grain boundary sliding - the primary mechanism of superplastic deformation appears on grain boundaries. The determining role of magnesium in the formation and development of fibrous structures is shown.

INTRODUCTION

For constructional materials which are used in the aircraft industry and also in atomic engineering and transport, the important characteristics are the density and the modulus of elasticity. Therefore multicomponent constructional aluminum alloys containing as alloying elements lithium, magnesium and other elements, are being increasingly used in engineering because they have a low density and high modulus of elasticity compared to other aluminum alloys [1-5]. One such alloy is the alloy 01420T of the system Al-Mg-Li-Zr, which have a high corrosion resistance, good weldability, high modulus of elasticity and sufficient static strength [1-5].

It is used to manufacture the welded fuselage stringers for passenger and military aircrafts, as well as for manufacturing of welded large cryogenic tanks for space and aviation applications. This alloy as well as other aluminum-lithium alloys can be used as a perspective constructional material for applications of nuclear engineering [6].

In this regard, investigations aimed on exploring the dependences of the mechanical properties of the alloy 01420T and its modifications from their phase composition and structural state and, in particular, the influence on them on the manifestation of structural superplasticity (SSP), are relevant.

High-temperature structural superplasticity (HTSSP) of 01420T alloy was studied in [7].

The aim of this study was to determine the influence of magnesium on phase transformations and structural changes occuring during superplastic deformation (SPD) of the alloy 01420T under high-temperature structural superplasticity.

1. MATERIALS AND METHODS

As a material for research was selected alloy 01420T (5,0 … 6,0% Mg; 1,9 … 2,3% Li; 0,09 … 0,15% Zr; 0,1 … 0,3% Si; ≤ 0,3% Fe; 0,1% Ti; 0,3% Mn; 0,005% Na; base Al, wt.%) [1-5].

Mechanical tests of the specimens of alloy 01420T are conducted in air in creep regime at a constant flow stress in accordance with the procedure described in details in [8]. Grain structure, morphology of cavities and of fibrous structures presented in specimens is tested using light microscopy (MIM-6, equipped with a digital camera Pro-MicroScan) and scanning electron microscopy (JEOL JSM-840), and standard techniques of quantitative metallography [9].

Energy dispersive X-ray microanalysis of local areas of grains and of fibrous structures is performed using scanning electron microscope JEOL JSM-840, equipped with the special device for conducting the X-ray energy dispersive microanalysis EDC-1.

Preparation of thin sections was performed as follows. After grinding of specimens to 1/3 of their thickness their surfaces were exposed to mechanical polishing. Finish treatment of thin sections surface was performed using a diamond grit paste 1/0.

To identify the grain boundaries the universal etchant of such composition was used: 17 ml HNO_3, 5 ml HF, 78 ml H_2O.

The average grain size $\bar{d}$ was determined from microphotograph by method of random secants [9].

Calculations of the mean grain size $\bar{d}$, the average longitudinal grain size $\bar{d}_{\parallel}$ (relative to the axis of elongation) and the average perpendicular grain size $\bar{d}_{\perp}$ (relative to the axis of elongation) was performed on data obtained from 100 measurements.

This provided relative error in the determination of the average grain size not more than 20%.

The magnitude $\bar{d}$ of the average grain size was calculated by the formula

$$\bar{d} = \sqrt[3]{\bar{d}_{\parallel} \cdot \bar{d}_{\perp}^{2}}, \qquad (1)$$

where $\bar{d}_{\parallel}$, $d_{\perp}$ – average grain sizes in parallel and perpendicular directions to the elongation axis, respectively.

Investigation of the kinetics of phase transformations occurring during heating in the specimens of alloy

01420T, was conducted using the instrument «Derivatograph Q-1500».

2. RESULTS AND DISCUSSION

Found that the original grain structure of the investigated heat-treated alloy 01420T (Fig. 1) is ultra fine. The average grain size is $\overline{d} = (5 \pm 1)\ \mu m$.

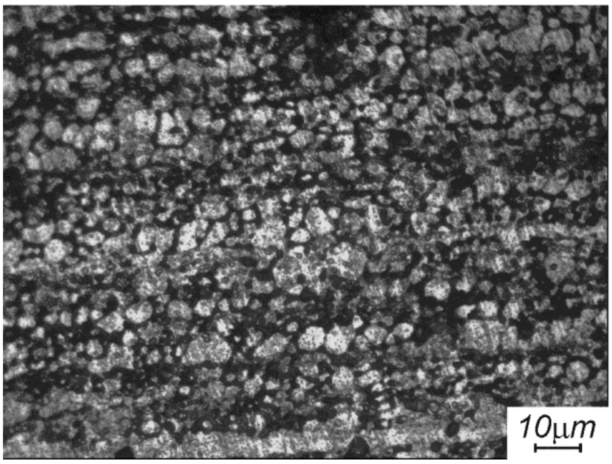

Fig. 1. The initial structure of the specimens of alloy 01420T. Light microscopy

As it is seen from Fig. 1, on the boundaries of some grains the areas of increased etching are observed. It is the evidence, as mentioned in [10], of the presence of magnesium segregations, which are solved in aluminum-based solid solution and also are the composites of S_1-phase (Al_2LiMg).

Figure 2,a shows a typical view of an area of the working part of the alloy 01420T specimen prepared for mechanical testing. Numbers 1 and 2 marked the points at which using energy dispersive x-ray microprobe, concentrations of aluminum and magnesium were determined. On figure 2,b and 2,c the energy spectra of the characteristic X-rays obtained in the study of this section of the working part of the specimen are presented.

The concentration of magnesium in point 1, located on the grain boundary is 8.64% and the concentration at point 2 is 4.36%. As can be seen, the concentration of magnesium from the grain boundary is increased as compared with its average concentration in the alloy and in the middle of the grain.

Mechanical tests of specimens of the alloy 01420T were performed under T = 520 °C, flow stress σ = 5.5 MPa and strain rate $\dot{\varepsilon} = 5{,}8 \cdot 10^{-5} s^{-1}$. These conditions, as it was shown in [7], are the optimal temperature – strain rate conditions of performance of high-temperature structural superplasticity for this alloy.

Fig. 3,a shows a general view of the specimen of the alloy 01420T, superplastically deformed to fracture under the optimal conditions compared to the initial specimen. It is seen that the superplastic deformation of the specimen of the alloy 01420T was carried out with high stability.

This is evidenced by the absence of a macroscopic necking in the destroyed specimen.

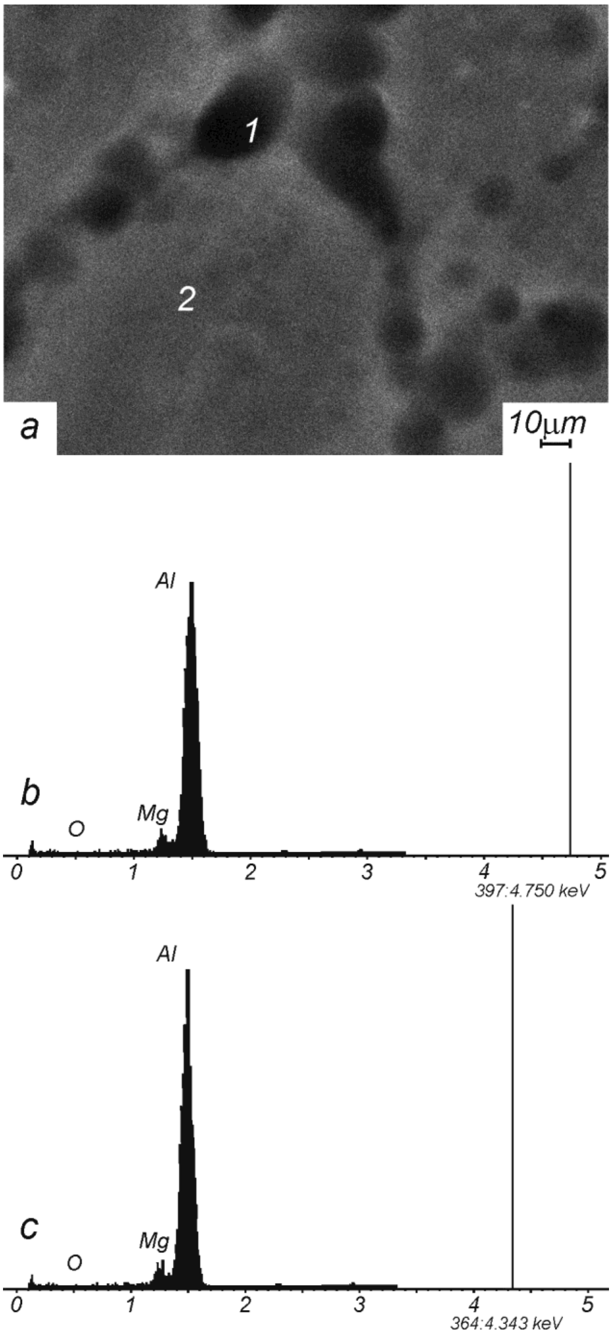

Fig. 2. a – typical view of an area of the working part of the alloy 01420T specimen prepared for mechanical testing (REM); energy spectra of the characteristic X-ray: b – spectrum resulting from point 1 at the grain boundary; c – spectrum resulting from point 2 in the middle of grain

As a result of metallographic studies it was observed that in the structure of the destroyed specimens (see Fig. 3,b and c) the isolated from each other grain boundary cavities of deformation origin were present. Cracks perpendicular to the tensile direction of the specimen, which formed through the merger of grain boundary cavities, is also present. The average grain size in the working part of the specimens of the alloy 01420T increases during superplastic deformation and to the time of their failure is $\overline{d} = (10 \pm 1)\ \mu m$.

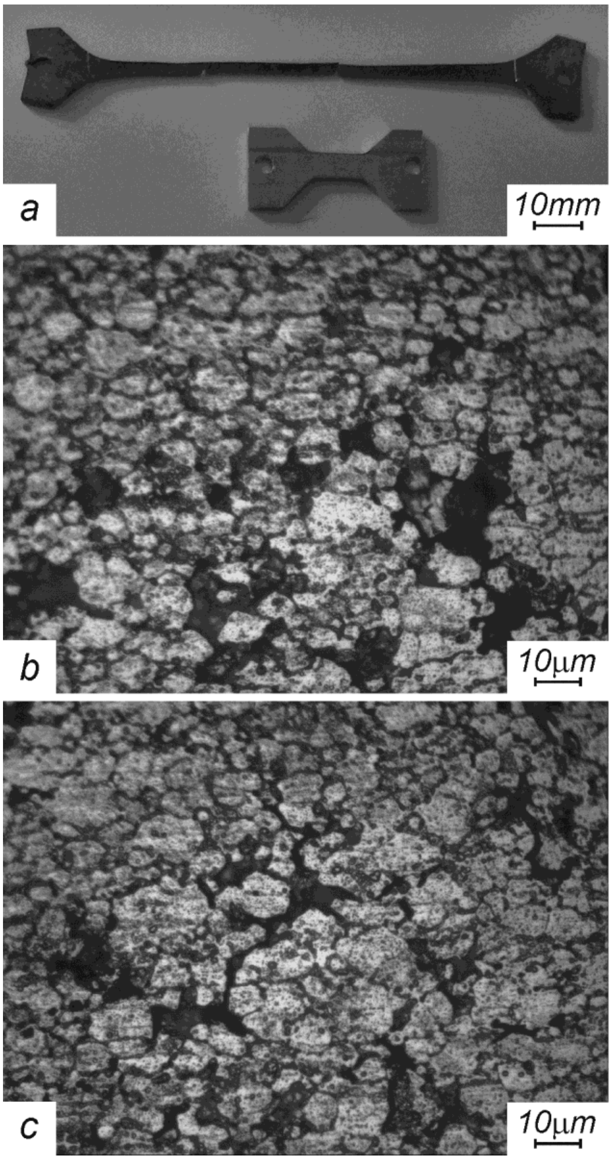

Fig. 3. a – general view of the specimen of alloy 01420T, superplastically deformed to failure in optimal conditions compared to the initial specimen; b and c – views of microstructure of the working part of the specimen deformed to failure in optimal conditions. Horizontal tensile direction. Light microscopy

The fact that the specimens of the alloy 01420T exhibit the effect of high-temperature structural superplasticity being in solid-liquid state as a result of partial melting of the alloy, in particular, indirect evidence of the following facts. In the near-surface cavities and cracks which formed and developed in the working part of the specimens during the superplastic deformation the specific fiber formations are present (Fig. 4,a and b). Also for the microstructure of deformed specimens the characteristic is the presence of so-called "tubular" cavities (see Fig. 4,c), which are formed as a result of flow (percolation) of the moving liquid phase at triple grain junctions.

Data on temperature conditions of manifestation of high-temperature structural superplasticity by the specimens of the alloy 01420T and the fact of the formation of fibrous structures during superplastic flow strongly suggest that the superplastic deformation of the specimens of this alloy at a temperature T = 520 °C develops in the presence of areas occupied by the liquid phase formed by the partial melting of the alloy on the grain boundaries.

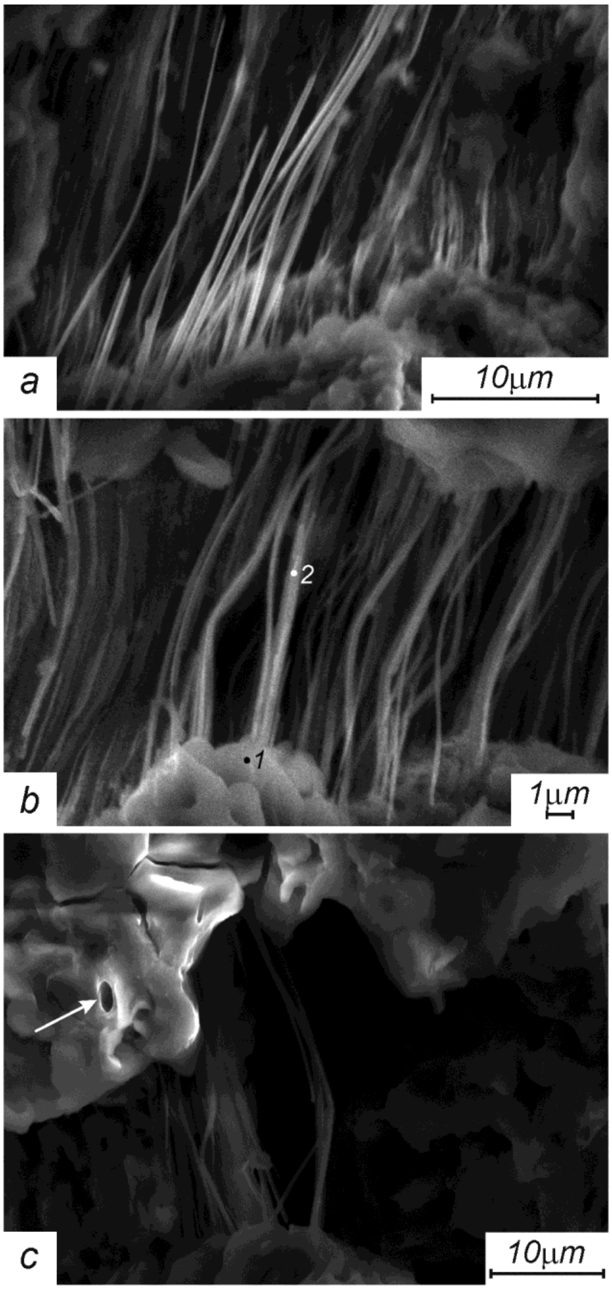

Fig. 4. a and b – types of fibrous structures in the working part of the specimens of alloy 01420T, deformed to failure in optimal conditions of high-temperature superplasticity. Numbers 1 and 2 are the locations in which the chemical composition of the fiber was determined; c – view of the oxidized surface of the grains, tubular cavity and oxidized fibrous structures in the working part of the specimen. The arrow points to the tubular cavity

We'll analyze the probable causes of partial melting of the alloy 01420T at high homologous temperatures. Fig. 5 shows a fragment of a differential thermal analysis curve that was obtained by heating of the specimen of the alloy 01420T from room temperature to 700 °C with rate of heating 10 °C/min.

It is seen that in temperature interval from 450 to 500 °C, which is near the temperature of mechanical testing of specimens of alloy 01420T (T = 520 °C) the phase transitions with heat absorption take place. This is the evidence of partial melting of alloy.

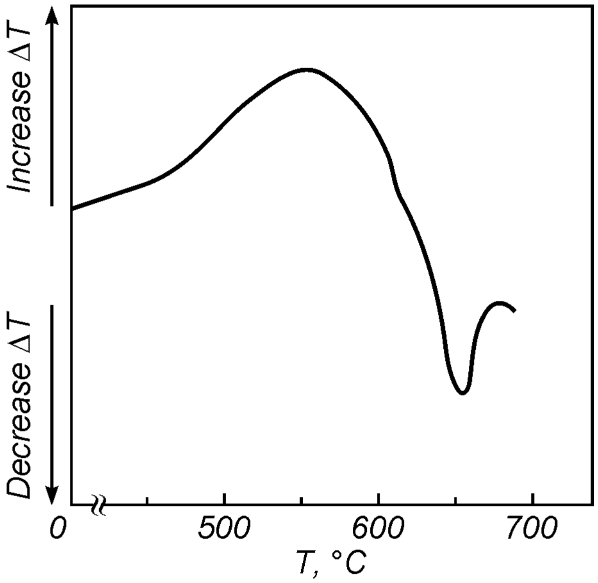

Fig. 5. Extract of differential thermal analysis curve of the specimen of the alloy 01420T in the temperature range 450…680 °C. Optimal temperature of high-temperature structural superplasticity of alloy 01420T T = 520 °C

Presumably, one of the most likely causes of the formation of liquid phase in the investigated alloy 01420T at high homologous temperatures can be the localized melting of those portions of grains and grain boundaries which contain a higher concentration of alloying elements (lithium and magnesium), lowering the melting point of the alloy. Partial melting of the alloy may take place due to inhomogeneities in the distribution of the alloying elements in microvolumes where their concentration is due to segregation at the grain boundaries or due to the intergranular phase separation is increased compared to its average concentration in the alloy [10, 11]. Since the test temperature for these microvolumes may be equal to the solidus temperature for these concentrations and even higher, then the samples are heated or directly in the course of superplastic deformation the partial melting of these regions can take place.

Analysis of the equilibrium Al-Mg-Li system diagram [1-4, 12, 13] suggests that three reversible peritectic transformations can take place in the specimens of alloy 01420T: under 488 °C: (L+LiAl) ⇔ (α_{Al}+$MgLiAl_2$); under 472 °C: (L+$MgLiAl_2$) ⇔ (α_{Al}+$Mg_{17}Al_{12}$); under 456 °C: (L+$Mg_{17}Al_{12}$) ⇔ (α_{Al}+Mg_2Al_3). These transformations can also be one of the probable causes of the liquid phase appearance at the test temperature.

It is determined that the fiber structures are present only in the working part of the specimens. They are found in the surface of grain boundary cavities and cracks, opened in the course of grain boundary sliding during the superplastic deformation in the degree of deformation of specimens 20…100%. Study of the types of fibrous structures allowed to make such generalizations about their basic morphological characteristics. In the cavities and cracks the fibers are oriented predominantly parallel to the tensile direction of the specimens. They are fixed at both ends of their inner surfaces formed during grain boundary sliding in the separation of grains from each other along the boundaries approximately perpendicular to the tensile direction. The number of fibers in cavities is different. As single fibers or fiber bundles are observed. It was determined that adjacent fibers in bundles are curve on one and the same areas. The fibers found in fractured specimens of alloy 01420T superplastically deformed to several hundred per cent, have a cylindrical form. Basically, they are uniform in thickness, but they have a thickening of the base. Typically, the average diameter of the fibers is 1…2 μm. The length of most of them is comparable with linear dimension of cracks and cavities in the tensile direction of fractured specimen and reaches 100 μm, and sometimes exceeds this value.

Fig. 6 shows as an example the spectra of characteristic X-ray energies, indicating the presence of atoms of Mg, O and Al in the fibers, which were formed during superplastic deformation of the specimen of alloy 01420T.

They were obtained by energy dispersive X-ray microanalysis of the chemical composition of the material, from which the fiber is, in the vicinity of the fixing of fiber to cavity wall (the base of fibers) and its middle (homogeneous) parts, respectively. Thus, for the fiber which is presented in Fig. 4,b found that the concentration of Mg in these areas is increased compared to its average concentration in the alloy and is at the point indicated by numeral 1 (see Fig. 4,b) of the base of fiber is 10.66 wt.%, and at a point indicated by number 2 in the middle portion of the fiber is 6.0 wt.%. This result agrees with the chemical composition of the fibers obtained in [14-18] under the investigation of structural state of specimens of another aluminum alloys, alloyed by magnesium, which performed high-temperature superplasticity in solid-liquid state.

It is known [19], that the influence of the presence of molten metal on the mechanical properties of the solid metal can manifest itself in different ways. In some cases, there is a catastrophic brittleness and a sharp decline in strength, and in the other – the plasticizing effect of the material. An important role in these processes play spreading, penetration (percolation) and migration of molten material on the surface of the grain boundaries, grain boundary cavities and cracks. It can be assumed that a substantial influence on the kinetics of these processes, in the case of high-temperature structural superplasticity of the alloy 01420T exert such factors: viscosity of the liquid-solid phase on the basis of the melt of a aluminum-based solid solution, the presence of oxide films on the surface of the melt, on the surface of the grains, cavities and microcracks and the presence in the melt of dispersed particles, which are fragments of oxide films. One can assume that for specimens of alloy 01420T which showed high-temperature structural superplasticity, increased viscosity of the liquid-solid phase formed by partial melting of the alloy is characteristic. This is primarily due to the fact that oxidation of lithium and magnesium, which are on the surface thereof, leads to the formation of brittle oxide films that fall into the melt. Furthermore except oxides in crystallizing melt crystals of α-based solid solution of aluminum, which are not fully solved when heated, including fused intermetallic particles that contain magnesium and (or) lithium and also dissolved at the temperature, dispersoids Al_3Zr may also be present.

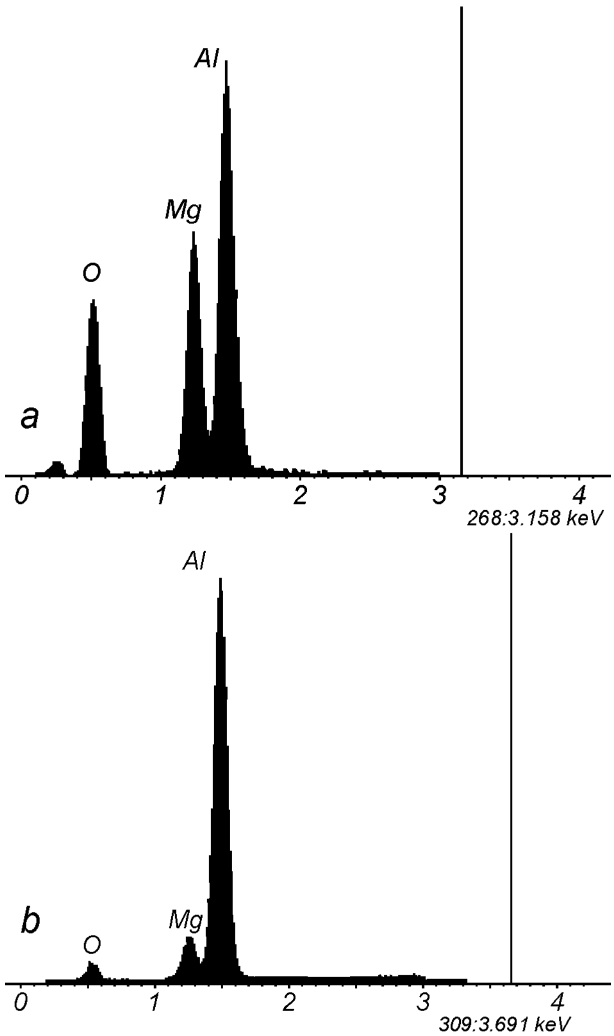

Fig. 6. Energy spectra of the characteristic X-ray that is received from the fiber sections formed during superplastic deformation of the alloy of the specimen 01420T: a - from fiber section, which is located near the place of fixing the fiber to the cavity wall (at the base of the fiber (Fig.4b, point 1), b - the portion of fiber which is located in the middle portion of fiber (Fig. 4,b, point 2)

During superplastic deformation of specimens of alloy 01420T, which performs mainly through the development of grain boundary sliding under the influence of local normal stress and shear stress, it is difficult to predict what shape will be the inclusions of the liquid phase, localized at the grain boundaries.

Apparently, the melting grain boundary material in specimens of the alloy 01420T is in a small amount of isolated liquid inclusions in some triple grain junctions, as well as a thin liquid layer at some grain boundaries. This is supported by the analysis of the distribution of the cavities and type of fibrous structures and the presence of oxide films on the surface of grains. The oxide film as described in [20], are not conducive to rapid spreading of the liquid-solid phase from the melt of aluminum-based solid solution on the grain surface, the cavities and cracks. One can assume that superplastic deformation of specimens of alloy 01420T will be provided by the cooperative development of deformation and accommodative processes occurring simultaneously in solid and liquid phases. Self-consistent effect of these deformation and accommodative processes provides manifestation of the effect of high-temperature structural superplasticity, stable on a macroscopic level within the samples and achieve their considerable elongations to failure.

Found [21], if the aluminum alloy contains in a small amount such elements as Cu, Si, Zn, Mn, Fe, then the oxide film formed on the surface of aluminum alloys with these elements during their oxidation in air in the liquid state, is similar in composition and structure of the oxide film which is formed on the molten pure aluminum. As is well known [21], it is continuous, stable and consists of aluminum oxide crystals Al_2O_3. Meanwhile, when the content of Mg in the aluminum alloy is more than 1.5 wt.%, as shown in [21], the oxide film is composed of a mixture of oxides: magnesia MgO and $MgAl_2O_4$ spinel, magnesial spinel or magnesia only MgO. This film is friable [22, 23]. It can be assumed that the presence of magnesium atoms in the melt based on aluminum is a major reason that during the superplastic deformation of friable oxide film in localized areas of the working part of the specimen is no formation of continuous "receptacle", which is formed by partial melting of the liquid phase. Since the oxide film on the surface of the liquid-phase inclusions is unstable, its presence will not interfere with formability of liquid-solid inclusions, asking when it is elongated by the mechanism of viscous flow during the opening of grain boundary cavities in the grain boundary sliding. The fine fragments which formed as a result of failure of oxide films during the superplastic deformation will fall into the melt that leads to its conversion into slurry.

It can be assumed that the viscosity of the material from which the fibrous structures formed during superplastic deformation of specimens of investigated alloy is closely related to the presence of a significant amount of solid inclusions of fine loose fragments captured magnesium and aluminum oxide (magnesia spinel MgO and $MgAl_2O_4$) in melt [22, 23], and also formed and growing crystals of the solid solution based on aluminum. Since magnesium, dissolved in liquid aluminum, also increases the melt viscosity [20, 24, 25], its presence in the surface layer of fibers and also the presence of oxides particles leads to the formation of a liquid or of a liquid-solid suspension, different from its internal by the higher viscosity.

Thus, it can be argued that the presence of a viscous liquid-solid material in the form of aluminum-based solid solution, richen by magnesium atoms and saturated by its oxides, is a favorable condition for the development of fibrous structures involved in disclosing of grain boundary cavities during grain boundary sliding.

CONCLUSIONS

1. The microstructure and chemical composition of different areas of the grain in the working part of the alloy 01420T specimens prepared for mechanical testing are investigated. It is determined that the concentration of magnesium in the grain boundaries is increased compared to its average concentration in the alloy and compared to concentration in the middle of the grain.

2. In near surface cavities and cracks which formed and developed in the working part of the specimens of alloy 01420T during superplastic deformation fibrous structures are observed. This indirectly indicates that, during the superplastic deformation of the alloy specimens were in a solid-liquid state.

3. The phase transformations which take place in the alloy 01420T during heating it to a test temperature, are investigated, the probable causes of partial melting of the alloy 01420T at high homologous temperatures, which leads to the formation of a liquid phase are analyzed.

4. The chemical composition of the material from which the fibers consist is determined. Suggested that the formation and development of fibrous structures during superplastic flow of specimens of alloy 01420T takes place by the mechanism of viscous flow of liquid mixture of solid phases located at the grain boundaries. It is related to the disclosure of grain boundary cavities during the intensive development of grain boundary sliding.

5. The influence of magnesium and the influence of magnesium and aluminum oxides, formed from oxidation of the specimens of alloy 01420T at high homologous temperatures, on the nature of the viscous flow of liquid-solid phase based on the melt aluminum-based solid solution is analized.

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ВЛИЯНИЕ МАГНИЯ НА ФАЗОВЫЕ ПРЕВРАЩЕНИЯ И СТРУКТУРНЫЕ ИЗМЕНЕНИЯ, ОСУЩЕСТВЛЯЮЩИЕСЯ В ХОДЕ СВЕРХПЛАСТИЧЕСКОЙ ДЕФОРМАЦИИ СПЛАВА 01420Т

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Установлено, что концентрация магния на границах зерен образцов сплава 01420Т повышена в сравнении с его средней концентрацией в сплаве и в средней части зерна. Повышенная концентрация магния в приповерхностных областях зерен приводит к их оплавлению при деформировании при высоких гомологических температурах. В результате на границах зерен появляется вязкая жидкая фаза, способствующая осуществлению зернограничного проскальзывания – основного механизма сверхпластической деформации. Показана определяющая роль магния в процессе зарождения и развития волокнистых образований.

ВПЛИВ МАГНІЮ НА ФАЗОВІ ПЕРЕТВОРЕННЯ І СТРУКТУРНІ ЗМІНИ, ЩО ЗДІЙСНЮЮТЬСЯ ПРИ НАДПЛАСТИЧНІЙ ДЕФОРМАЦІЇ СПЛАВУ 01420Т

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Встановлено, що концентрація магнію на межах зерен зразків сплаву 01420Т підвищена в порівнянні з його середньою концентрацією в сплаві і в середній частині зерна. Підвищена концентрація магнію в приповерхневих областях зерен призводить до їх оплавлення при деформуванні при високих гомологічних температурах. У результаті на границях зерен з'являється в'язка рідка фаза, що сприяє здійсненню зернограничного проковзування - основного механізму надпластичної деформації. Показана визначальна роль магнію в процесі зародження і розвитку волокнистих утворень.