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Superplastic deformation of high strength aluminum alloy 1933 after hot working

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The structural state of the specimens of industrial alloy 1933, passed previous hot working processing, is investigated. It is found, that the microstructure has considerable anisotropy. The grains, elongated in the rolling direction, dominate in it. The specific proportion of low-angle grain boundaries and high-angle grain boundaries in specimens after hot working is determined. The optimal conditions of superplastic deformation in specimens past previous hot working are determined.
Keywords: superplasticity, hot working, grain boundaries, structural anisotropy.

Исследовано структурное состояние образцов промышленного сплава 1933, прошедшего предварительную термомеханическую обработку. Установлено, что микроструктура обладает значительной анизотропией. Зерна, удлиненные в направлении прокатки, преобладают в ней. Определена удельная доля малоугловых и большеугловых границ зерен в образцах после термомеханической обработки. Определены оптимальные условия проявления сверхпластической деформации в образцах прошедших предварительную термомеханическую обработку.
Ключевые слова: сверхпластичность, термомеханическая обработка, границы зерен, структурная анизотропия.

Досліджено структурний стан зразків промислового сплаву 1933 що пройшов попередню термомеханічну обробку. Встановлено, що мікроструктура має значну анізотропією. Зерна, видовжені в напрямку прокатки, переважають в ній. Визначено питому частка малокутових і багатокутових меж зерен в зразках після термомеханічної обробки. Визначено оптимальні умови прояву надпластичної деформації в зразках, що пройшли попередню термомеханічну обробку.
Ключові слова: надпластичність, термомеханічна обробка, межі зерен, структурна анізотропія.

Introduction

In [1-5], it was found that the forging high strength alloy 1933 of the system Al-Zn-Mg-Cu-Zr with the original bimodal grain structure showed the effect of high-temperature structural superplasticity (HTSP) in a solid-liquid state. The initial structure of industrial semi-finished alloy 1933 consisted of large poligonized unequiaxed grains that surrounded areas, consisting of ultrafine equiaxed grains. In [5] the specific proportion of grain boundaries in the initial misorientation of various 1933 alloy specimens, prepared for mechanical testing was determined. It was found that the specific proportion of low-angle boundary (LAB) grains for the tested area was 65.5% and the specific proportion of high-angle boundary (HAB) grains was 35.5%. It was shown [1-5] that the grain boundary sliding (GBS) occurs in the specimens during superplastic deformation (SPD). It occurs not only on the boundaries of ultra-fine grains, but also on the boundaries of coarse poligonized grains, oriented parallel to the stretch axis. On the basis of generalization of the results obtained in the work, and taking into account the data available in the literature, the analysis of development of the deformation and accommodative mechanisms of SPD of this alloy is conducted [5].

Since 1933 alloy is increasingly used in aircraft construction, in particular for the manufacture of load-bearing aircraft fuselage [6], it was necessary to continue research aimed at improving of its superplastic characteristics. These improvements, in particular, could be achieved if it were possible to create a uniform ultrafine structure in the original alloy specimens. As it has been shown in [7], the implementation of static recrystallization does not provide the formation of homogeneous nonpoligonized grain structure and does not eliminate bimodality in the alloy 1933 of Al-Zn-Mg-Cu-Zr system, it was necessary to conduct studies aimed at using of other methods of forming of ultrafine homogeneous structure in the specimens of high-strength aluminum alloy, by selecting them from the set of those techniques that are traditionally used for its creation in multicomponent aluminum alloys [8-11]. The aim of research, the results of which are described in this article was to determine the effect of pre-thermomechanical processing, carried out as a result of cold rolling of pre-annealed industrial semi-finished alloy 1933 specimens on their structural condition, as well as improving of performance of superplasticity of the alloy specimens by creating in them firstly fibrous structure, and then uniform ultra-fine microstructure instead of bimodal structure.

Materials and methods of the experiment

Investigated in the paper alloy 1933 has such a chemical composition (1,6 – 2,2% Mg; 0,8 – 1,2% Cu; 0,1% Mn; 0,66 – 0,15% Fe; 0,1% Si; 6,35 – 7,2% Zn; 0,03 – 0,06% Ti; 0,05% Cr; 0,10 – 0,18% Zr; 0,0001 – 0,02% Be; base Al, % wt.) [5]. The main alloying elements in the alloy are magnesium, zinc and copper. They play a fundamental role in the partial melting of superplastic aluminum alloys and as a result, creating small amounts of viscous liquid phase at the grain boundaries during deformation [12-13].

Cold rolling of the pre-annealed for 2 hours at a temperature of 500°C specimens was carried out on the mill BMC-64-2ВУ2С so that the accumulated amount of deformation was e = 1.3.

In the initial state the hardness of semi-finished alloy 1933 was HV – 126 HV, after annealing it was equal to 56, and after the cold rolling HV - 126.

Mechanical tests of the alloy specimens, prepared from industrial intermediates, held in air by stretching them in creep mode at constant flow stress according to the procedure described in details in [14].

Grain structure, cavity morphology and morphology of fibrous in specimens were studied using light microscopy (MIM-6 with the digital camera Pro-MicroScan) and scanning electron microscopy (JEOL JSM-840), and standard techniques of quantitative metallography [15].

To determine the proportions of ultrafine and coarse grains, the grain boundary misorientation angles and to make quantitative assessment of their content in the alloy 1933 the electron backscatter diffraction analysis (EBSD) was used [16]. Investigations were performed using a scanning electron microscope JEOL JSM-6490LV, equipped with energy dispersive spectrometry INCA Penta FETx3 and by detector of backscattered electrons Nordlys S. Analysis of the obtained structures was performed according to the procedure described in [16], using software HKL Channel 5, which is included in the set of technical documents to the microscope.

The surface of the working part of the specimens was subjected to grinding and to mechanical polishing. Surface finishing of sections for metallographic investigations was carried out using a diamond paste grit 1/0.

Specimens which were used for EBSD analysis were subjected to electropolishing. It was performed in a solution of such composition: 40 wt. % H_2SO_4, 45 wt. % H_3PO_4, 3 wt. % CrO_3, 11 wt. % H_2O [16]. Mode of operation: operating temperature 60-80°C, the anode current density, the voltage of 15-18 V, exposure – a few minutes.

To reveal the grain boundaries during the metallographic studies was used universal chemical etchant of such a composition: 17 ml HNO_3, 5 ml HF, 78 mL H_2O.

In addition to the chemical etching for revealing of grain boundaries of the working part surface of the test specimens of alloy as the initial one, and superplastically deformed to a certain degree of deformation, strain relief method was used.

To determine the contribution of grain boundary sliding (GBS) in the overall deformation and for studying of the kinetics of its development at different stages of SPD used a method of marker scratches. On the polished specimens using a diamond paste of dispersion 3 microns, the marker risks parallel to the axis of their subsequent direction of stretching were applied. Mechanical testing was carried out using specimens in a HTSP in the creep mode, which led to the formation on the surface of polished specimens of a deformation relief as a result of a small (3-5%) deformation. The views of it were studied.

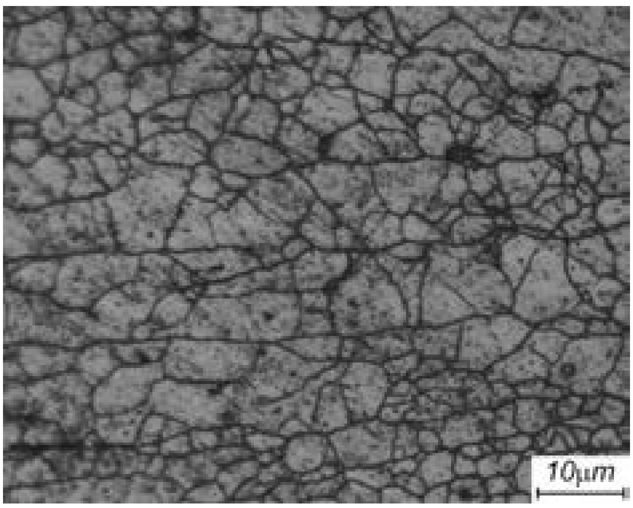

Fig.1. Typical view of the initial microstructure of the specimen of alloy 1933 in the initial state. Light microscopy.

The average grain size ⟨*d*⟩ was determined by light microscopy photomicrographs with use of random secant method [15].

Results and discussion

Figure 1 shows a micrograph of a typical view of the initial microstructure of the specimen of alloy 1933, obtained using optical microscopy techniques. The microstructure of the alloy is bimodal. It consists of areas containing a large number of recrystallized ultrafine grains with ⟨d⟩ = 7 ± 1 μm, divided by high-angle grain boundaries and contain some coarse elongated poligonized grains with ⟨d⟩ = 50 ± 1 μm, divided by low-angle grain boundaries [5].

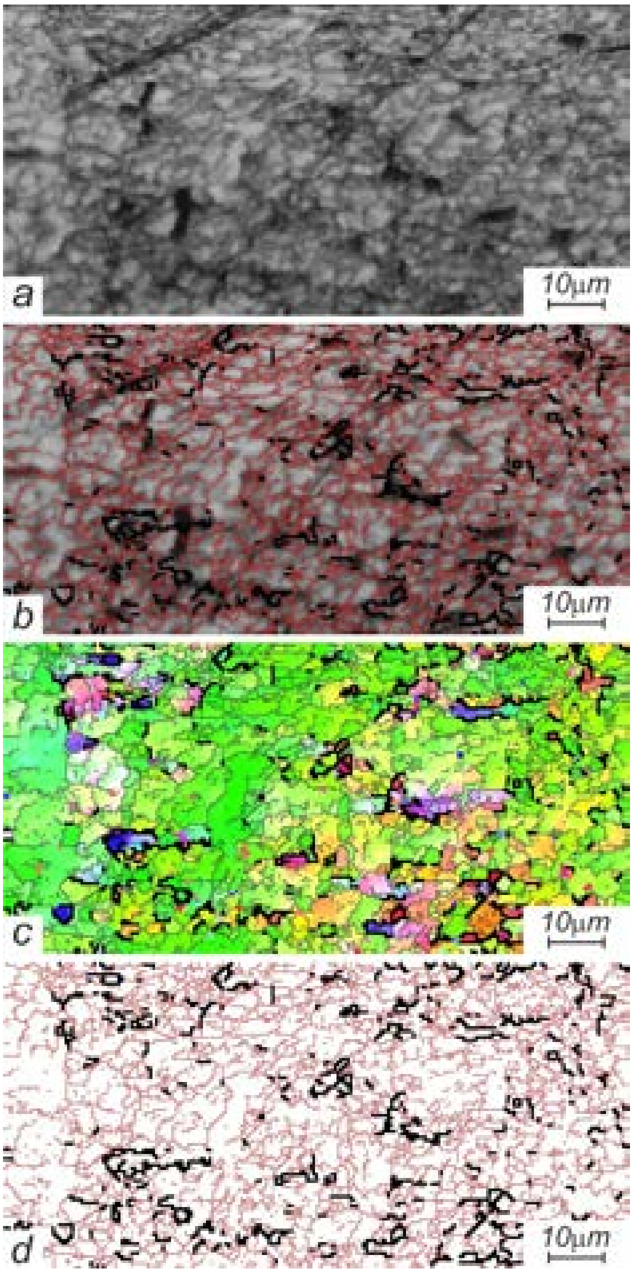

Fig.2. Cards of EBSD analysis: a – chosen fragment, b – card obtained as a result of combination of card of contrasts and of card of grain boundary misorientation; c, d – cards of grain boundary angles orientation.

As indicated in [7] a small amount of nonequilibrium eutectic constituents (Quasi-binary, ternary and quaternary eutectics), consisting of a mixture of crystals of a solid solution based on aluminum (α_{Al}) and intermetallic particles of T-phase, η-phase and S-phase are present in almost all industrial semifinished alloy 1933 which passed thermomechanical processing. It is characteristic for alloys of Al-Zn-Mg-Cu-Zr [6].

It is found that the stitch inclusions of intermetallic phases are mainly localized at the grain boundaries, which are parallel to the rolling direction. In grains the inclusions, which are scopes of mentioned above particles and the particles of ultrafine β'-phase (Al_3Zr) are present in grains. β'-phase (Al_3Zr) effectively inhibits the growth of the grains in multi superplastic aluminum alloys at high homologous temperatures [8-10]. After hot working of the alloy 1933, performed in this study, the analysis of microstructure was conducted using EBSD techniques. Figure 2, a shows a fragment of the working part of specimen that has been used for EBSD analysis of the structural state of the alloy in the investigated part of the surface of cold-rolled alloy specimen of 1933. It is seen that the structure of the alloy is homogeneous submicrocrystalline. On fig.2, b there is EBSD card, obtained as a result of combination of chosen for investigations fragment and of card of grain boundary misorientation. On fig.2, c, d cards of grain boundary angles orientation are shown. These cards were used for determination of the specific proportion of low-angle grain boundaries (LAB) and high-angle grain boundaries (HAB) for the investigated fragment of surface of specimen of alloy 1933.

Figure 3 shows the dependence of the relative amounts of different grain boundaries from misorientation

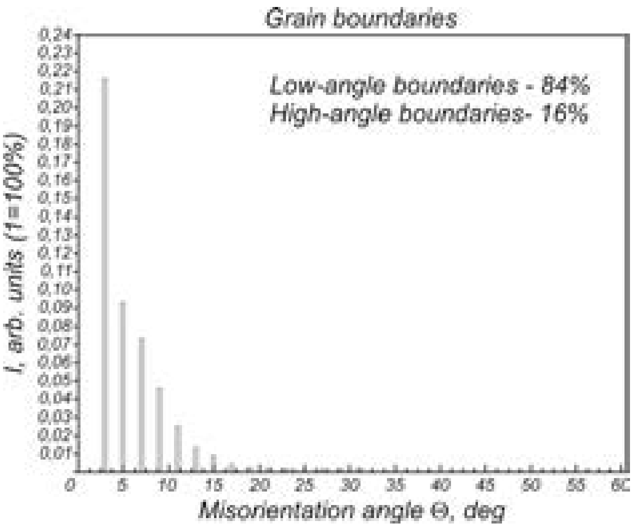

Fig.3. Relative amounts of the grain boundaries of the different misorientation from misorientation angle for the tested area of the specimen' surface after hot working.

angle for the tested area of the specimens' surface of alloy 1933 after hot working. This quantitative distribution of the disorientation angles of the grain boundaries was constructed as a result of accounting of all certified grain boundaries present in the tested specimen of alloy 1933.

When building this relationship was usually to attribute to the LAB grains those grain boundaries, which have misorientation angle below 10°, and to attribute to the to HAB - those grain boundaries, which are misoriented above 10° [16]. It is found that the specific proportion of LAB grains for the tested area is 84.0% and the specific proportion of HABs grains is 16.0%.

As a result of the mechanical tests performed in the creep mode at a constant strain stress it is found that specimens of the alloy 1933, subjected to preliminary hot working exhibit the effect of high-temperature structural superplasticity (HTSP). The optimal conditions of its performance is such: temperature T = 500°C, flow stress σ = 4.5 MPa. The maximum relative elongation of specimens to failure δ = 410%. Strain rate is $2{,}2 \cdot 10^{-3}$ s^{-1}.

Elongation to failure and the true strain rate in the specimens with a homogeneous fine-grained structure, previously passed thermomechanical treatment, is higher than that for the initial 1933 alloy specimens with bimodal structure [1-5].

Fig.4. General view of the specimen of alloy 1933, previously passed hot working, deformed to failure in mode of superplasticity in optimal conditions to 410% in comparison with the initial.

Fig.4 shows the general view of the specimen of alloy 1933, previously passed hot working, deformed to failure in mode of superplasticity in optimal conditions to 410% in comparison with the initial one. It's seen that the superplastic deformation of the specimen performed homogeneously and steadily on the macroscopic level. This is proved by the fact that the specimen's failure was quasibrittle without forming a distinct neck.

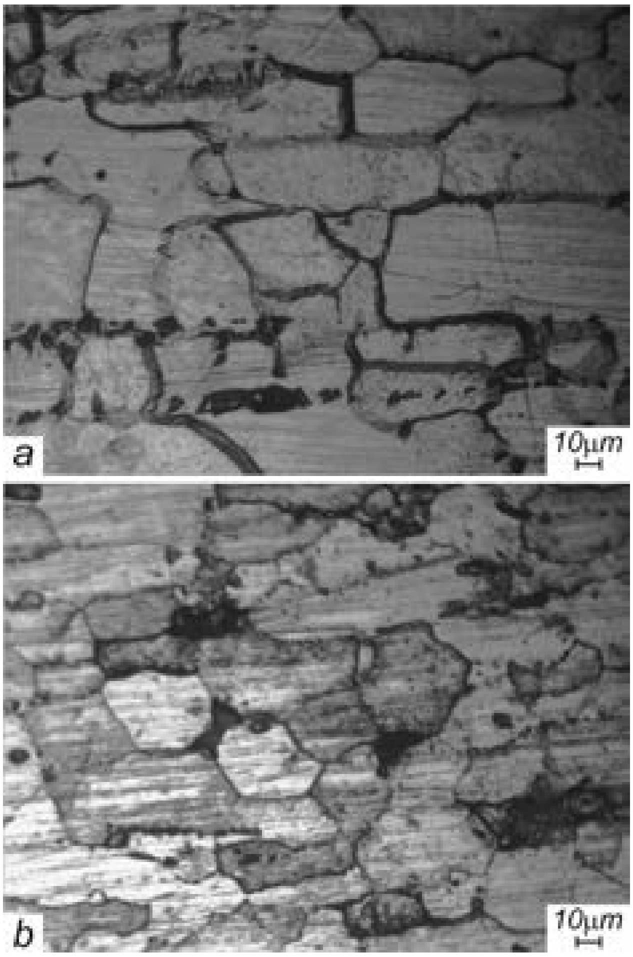

Fig.5. The typical view of the microstructure of the working part of specimen: a - heated to a test temperature T = 500°C, b - superplastically deformed to failure under the optimal conditions. Light microscopy.

Fig.5 shows the typical view of the microstructure of the working part of specimen heated to a test temperature T = 500°C and superplastically deformed to failure under the optimal conditions. Their analysis showed that as a result of a preliminary thermomechanical processing of semifinished alloy 1933 bimodality was eliminated and a uniform ultrafine grain structure having an average size d = 15 μm formed. It made possible to increase the phenomenological parameters characterizing the superplastic properties of the alloy.

It can be assumed that a certain amount of intermetallic phases localized at the grain boundaries, is dissolved in the aluminum-based solid solution during heating of the specimen to a test temperature and directly during the superplastic deformation. Not had time to dissolve intermetallic and non-equilibrium components to which they belong, as well as the border edge grains in which, as shown in [4] in a solid solution based on aluminum contains increased compared to the nominal alloy composition concentration of zinc atoms and magnesium act as centers of partial melting of the alloy. As a result, plots occupied

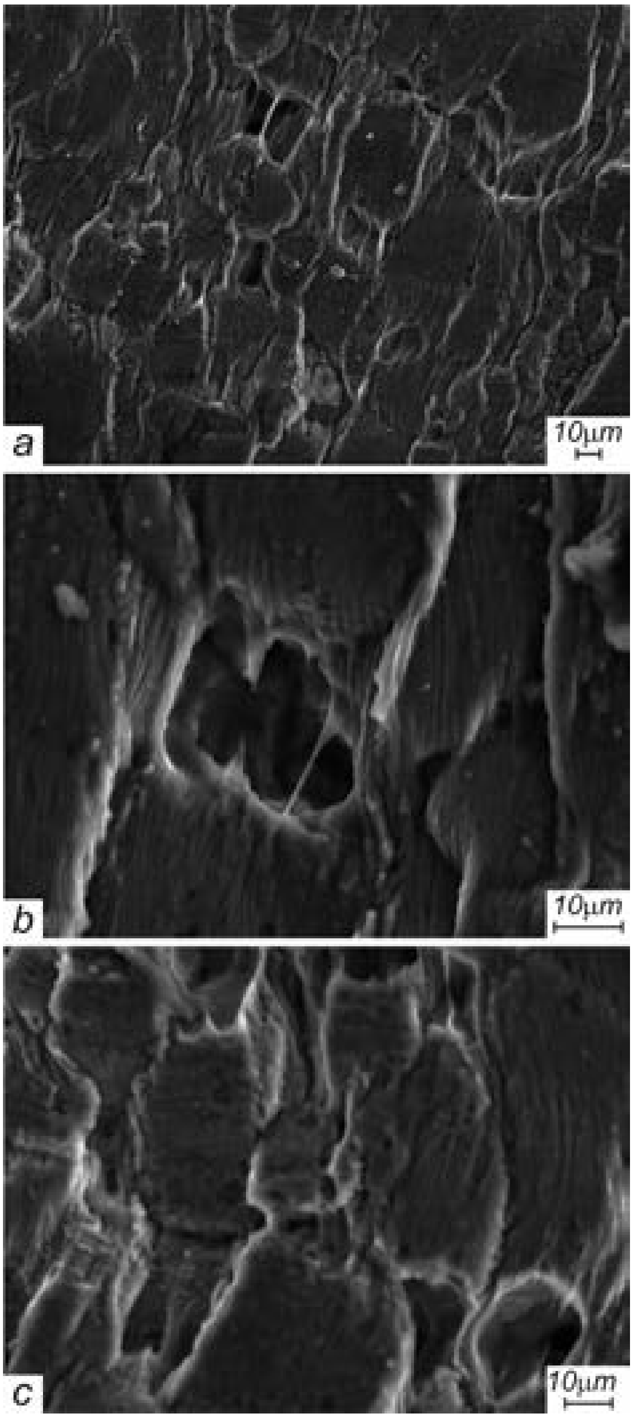

Fig.6. Typical views of deformation relief formed on the surface of the working part of specimens of the alloy 1933 superplastically deformed to fracture under the optimal conditions. Scanning electron microscopy.

by a metastable liquid phase, necessary for active and facilitated the development of HTSP accommodative processes [17-23] are formed on the grain boundaries.

As a result of detailed studies of specific views of strain relief formed on the surface of the working part of the alloy 1933 specimens superplastically deformed to failure performed using scanning electron microscopy, fibrous structures were found (see. fig.6). They formed and developed during the superplastic deformation in near-surface grain boundary pores and microcracks of grain.

It is considered [17-22] that the formation and development of such structures is an indirect confirmation of the fact that the 1933 alloy during superplastic deformation is in the solid-liquid state due to its partial melting. The study of specific types of strain relief found that the ends of long fibers (see. Fig.6, a, b) connect the surface of grain boundary cavities and cracks formed in the course of GBS in the separation of grains from each other along the boundaries, approximately perpendicular to the strain direction, and short fibers form a fringe on the edges of the grains (see. Fig.6, c).

The number of fibers found in the near-surface grain boundary cavities is different. Apparently, it depends on the amount of metastable liquid phase localized at the grain boundaries perpendicular to the strain direction of the specimen.

On the surface of the fibers and grains to which they are connected, friable oxide film are detected. This suggests that during SPD in specimens of alloy 1933 at a test temperature T = 500°C, in its working part the dynamic oxidation of the surface of the specimen and the inclusions of a metastable liquid phase, which in a small amount are present at the grain boundaries and in the boundary edges of slipping grains intensively occured. The dynamic oxidation led to the formation of oxides of Al_2O_3, MgO and magnesia spinel $MgAl_2O_4$, consisting of the oxides, which are the most common for multicomponent aluminum alloys doped with Mg [23]. Saturation of metastable liquid phase by particles of magnesium and aluminum oxides is apparently led to the formation of liquid-solid material with higher viscosity [24], viscous flow of which occured due to the disclosure of grain boundary cavities during the development of GBS in the SPD of specimens of alloy 1933. It, in its turn, led to the formation and development of the fibrous structures according to the mechanism described in [3].

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Conclusions

1. The structural state of the specimens of industrial alloy 1933, passed previous hot working, is investigated. It is found, that the microstructure has considerable anisotropy. The grains, elongated in the rolling direction dominate in it.

2. The specific proportion of grain boundaries of different misorientation in specimens of alloy 1933 passed hot working is determined. It is found that the specific proportion of the low-angle grain boundaries is 84% and the specific proportion of high-angle grain boundaries is 16%.

3. It is shown that a uniform equiaxed grain structure with an average grain size of d = 15 microns in specimens of alloy 1933 previously past thermomechanical processing, forms in the early stages of superplastic deformation.

4. It is found that the specimens of alloy 1933 previously past thermomechanical processing exhibit the effect of high temperature structural superplasticity. Optimal conditions for its manifestation are: temperature T = 500°C, flow stress σ = 4,5 MPa. The maximum relative elongation of the specimens to failure δ, superplastically deformed under T = 500°C, σ = 4,5 MPa under the true strain rate $2{,}2\cdot10^{-3}$ s^{-1} reached 410%.

5. In the process of superplastic deformation of the specimens of alloy 1933 with an ultrafine grain boundary structure the fibrous structures form and develop in the surface cavities and intergranular cracks as a result of their disclosure during the development of grain boundary sliding.

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