

## SUPRAPLASTIC DEFORMATION OF ALUMINUM-LITHIUM ALLOYS 1450 AND 1460, ALLOYED WITH ZIRCONIUM AND SCANDIUM

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The article presents the results of research directed to the determination of mechanisms of an ultrafine-grained structure formation in aluminum-lithium alloys 1450 and 1460, which include dispersed particles of zirconium and scandium aluminides, which are effective stabilizers of the microstructure, as well as for the determination of the features of structural changes during the course supraplastic deformation.

### INTRODUCTION

Aluminum and its alloys as constructional materials are used in industry for a little over 100 years and are currently the most common materials after steel [1]. They are an indispensable constructional material not only in the aircraft industry, but also in the nuclear industry. In particular, seamless tubes for reactors are produced from aluminum alloys, as well as centrifuges for uranium enrichment. This was facilitated by a number of favorable properties of aluminum alloys, which are of particular importance in many areas of technology.

Aluminum-lithium alloys are characterized by a high degree of alloying, the complexity of phase transformations during heating and cooling during homogenization, hot deformation, and intermediate heat treatments during cold deformation and residual thermal processing [5, 6].

It is known that for the production of complex shaped parts from aluminum-lithium alloys the method of superplastic forming is used [2–5]. One of the necessary conditions for its implementation is the presence in them of an ultrafine-grained structure that is resistant to coarsening at high homologous temperatures [2–8].

To ensure the stability of the ultrafine-grained structure during deformation of aluminum alloys at high homologous temperatures, they are alloyed with zirconium and scandium [2–11].

These elements are included in the composition of multicomponent aluminum alloys because they effectively show their modifying and anti-recrystallization properties. It was revealed that during the crystallization of multicomponent aluminum alloys, zirconium and scandium, which are in a solid solution based on aluminum, ensure the grinding of its grains formed in the ingot. Alloying of aluminum-lithium alloys by zirconium and scandium leads to a significant increase in their strength due to the formation of a non-recrystallized structure and the dispersion of particles

(about 50 nm in size) intermetallic phases, which include zirconium and scandium. It has been revealed that dispersed particles of zirconium aluminide  $ZrAl_3$ , scandium aluminide  $ScAl_3$ , or of the composite phase  $Al_3(Sc_xZr_{1-x})$  coherent with the matrix are effective stabilizers of the microstructure [2–11], since they effectively prevent static recrystallization during heating of alloy samples to high homologous temperatures. The purpose of the work is to reveal the mechanisms of fine-grained structure formation in samples of industrial multicomponent aluminum alloys 1450 and 1460 and to establish the features of its changes during their superplastic deformation.

### 1. MATERIAL AND EXPERIMENTAL

Samples for mechanical tests and structural studies were made from sheet semi-finished products of the following industrial multicomponent aluminum-lithium alloys: alloy 1450 (Al-2.64 wt.% Cu-2.2 wt.% Li-0.12 wt.% Zr, small impurities of Ti, Fe, Si, Na, Be); alloy 1460 (Al-2.64 wt.% Cu-2.2 wt.% Li-0.12 wt.% Zr-0.12 wt.% Sc, small impurities of Ti, Fe, Si, Na) [5, 6].

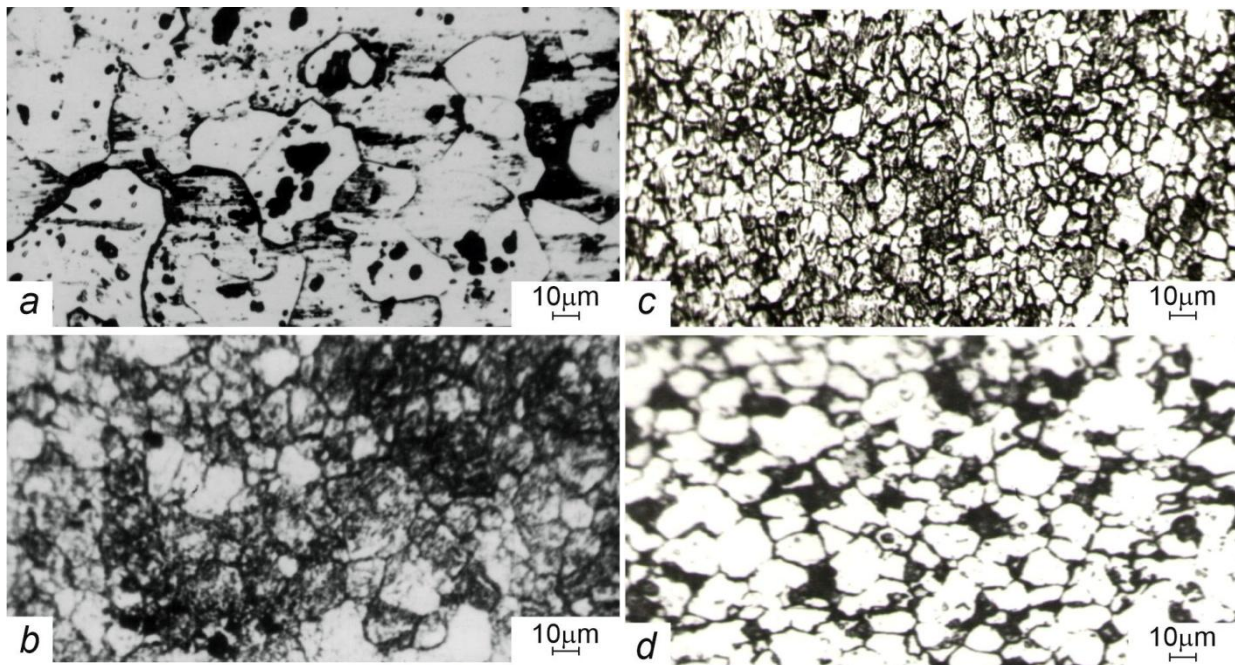
Mechanical tests of the samples, as in works [12–17], were performed in air in the creep mode with constant applied flow stress. They were performed under those temperature-strain rate conditions in which alloys exhibit the effect of structural superplasticity [12–17]. The microstructure of the samples was studied using a MIM 6 light microscope equipped with a Pro-Micro Scan digital camera. In order to detect grain boundaries on the surface of the working part of the samples, along with chemical etching, deformation relief was used, which is formed during superplastic deformation due to grain boundary sliding. Fractographic studies were performed using a JEOL JSM-840 scanning electron microscope.

### 2. RESULTS AND DISCUSSION

It was revealed that the initial microstructure of samples of alloy 1450, made from sheet industrial semi-finished product in the delivery state, is non-

recrystallized and multi-grained (Fig. 1,a). The grains contain a large number of clusters consisting of particles of intermetallic phases. They are concentrated both in the grain body and on the grain boundaries. According to the works [5, 19–21], in which the phase composition of alloy 1450 was investigated, in its samples in the delivery state, depending on the conditions under which

they underwent preliminary thermomechanical processing, intermetallics enriched by Fe and Si atoms were detected, dispersoids  $\text{Al}_3\text{Zr}$  also were detected and particles of intermetallic phases  $\text{Al}_3\text{Li}$ ,  $\text{Al}_2\text{Cu}$ ,  $\text{Al}_2\text{CuLi}$ ,  $\text{Al}_6\text{CuLi}_3$ . These phases strengthen the matrix - aluminum based solid solution.



*Fig. 1. Characteristic types of microstructures of alloy 1450: in samples in the initial state (a); in samples superplastically deformed at temperature  $T = 773\text{ K}$  and flow stress  $\sigma = 3.5\text{ MPa}$  to the degree of relative deformation  $\varepsilon_{rel}$  25% (b), 100% (c), 500% (d). The sample strain direction is horizontal*

It is known that large particles of the  $\text{Al}_2\text{Cu}$ ,  $\text{Al}_2\text{CuLi}$ ,  $\text{Al}_6\text{CuLi}_3$  phases, as well as particles of the phases containing Fe and Si atoms, are localized mainly along the grain boundaries, while dispersed particles of the  $\text{Al}_3\text{Zr}$ ,  $\text{Al}_3\text{Li}$ ,  $\text{Al}_2\text{CuLi}$  phases are localized both in the body and on the grain boundaries of the matrix [5, 19–21].

It was revealed that the recrystallization annealing of alloy 1450 samples performed in air at different temperatures and heating rates did not lead to the development of static recrystallization in them. The purpose of recrystallization annealing is the formation of a homogeneous fine-grained structure in samples. Due to this, the ultra-fine-grained structure was formed directly during the superplastic deformation of alloy 1450 samples due to the occurrence of continuous dynamic recrystallization in them. It was revealed that the almost uniform microstructure with an average grain size  $\bar{d} = 3 \dots 6\ \mu\text{m}$  (see Fig. 1,b) was created in samples which were superplastically deformed in the creep mode at temperatures of 753, 763, 773, 778 K and flow stress  $\sigma = 2.0 \dots 8.0\text{ MPa}$  to degrees of relative deformation  $\varepsilon_{rel}$ , which were 20...50% (see Fig. 1, c).

As can be seen in Fig. 1,c,d during the subsequent stages of superplastic flow, the average grain size in samples of alloy 1450 increases.

It was revealed that in the alloy 1450 samples, which were deformed to failure under the optimal conditions,

the grains retain their equiaxed shape, and their average size is  $\bar{d} = 10 \dots 15\ \mu\text{m}$ .

It was revealed [13, 14] that the optimal conditions for the appearance of the effect of structural superplasticity by samples of alloy 1450, which have the above-described characteristics of the grain structure, in the investigated flow stress interval  $\sigma = 2.0 \dots 8.0\text{ MPa}$  are as follows: optimal temperature  $T = 773\text{ K}$ , optimal flow stress  $\sigma = 3.5\text{ MPa}$ , the average strain rate of true deformation  $\bar{\dot{\varepsilon}}_{true} = 3.5 \cdot 10^{-4}\text{ s}^{-1}$ . The maximum relative elongation to failure  $\delta$  of alloy 1450 samples, which were superplastically deformed to failure under these conditions, is 650% (Fig. 2,b). Fig. 2,b shows a sample superplastically deformed to failure by 650% at  $T = 773\text{ K}$  and  $\sigma = 3.5\text{ MPa}$  compared to the initial sample. It is seen that its failure was quasi-brittle at the macroscopic level and occurred without noticeable localization of deformation in the form of a neck.

Due to the study of surface fractograms of alloy 1450 samples, which were superplastically deformed before failure (Fig. 3) it was revealed that at the microscopic level their failure is mixed. This is evidenced, in particular, by the fact that on the fracture surface of the samples there are areas engaged by macrodiscontinuities, which were formed during the coalescence of grain-boundary cavities and the subsequent development of main cracks, bordering

those areas where the failure takes place according to the intercrystalline type. This is evidenced by the presence of a characteristic grain relief on the fracture surface, formed during the propagation of cracks along the intergrain boundaries. There are also areas on the fractures that were formed due to the localized plastic flow of individual ultrafine grains and their subsequent transcrystalline failure.

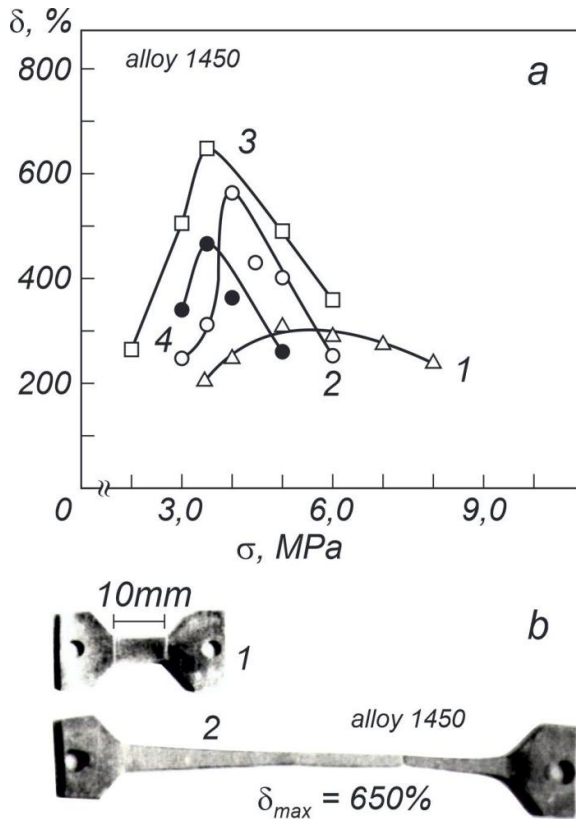


Fig. 2. Dependences of the relative elongation to failure  $\delta$  on the applied stress  $\sigma$  for samples of alloy 1450 superplastically deformed to failure (a) at  $T = 733$  (1), 753 (2), 773 (3), 793 K (4) and the sample deformed to failure by 650% at  $T = 773$  K and  $\sigma = 3.5$  MPa with the initial one (b)

Ultra-fine grain structure with an average grain size  $\bar{d} = 5 \mu\text{m}$  in sheet semi-finished products of alloy 1460 was formed in industrial conditions as a result of thermomechanical processing performed during their manufacture. This was probably achieved due to the presence of dispersed particles of  $\text{Al}_3\text{Zr}$ ,  $\text{Al}_3\text{Sc}$ , and  $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$  in the microstructure of alloy 1460 samples, which, as was established in works [5, 9, 21], are able to ensure the thermal stability of their grain structure at high homologous temperatures due to the fact that they are an effective obstacle to grain boundaries migration at elevated temperatures.

A typical view of the initial grain structure of alloy 1460 samples is shown in Fig. 4, a, b-d shows the typical views of microstructure of the working part of alloy 1460 samples superplastically deformed to failure at different temperatures and flow stresses.

It should be noted that during the superplastic deformation of alloy 1460 samples when they are deformed at test temperatures higher than 773 K, when coagulation of dispersoids  $\text{Al}_3\text{Zr}$ ,  $\text{Al}_3\text{Sc}$ , and  $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$  begins [5, 6, 9, 11, 21], and they cannot fully restrain the migration of high-angle boundaries, the average grain size in the working part of the samples begins to increase. However, the vast majority of grains, despite the high degrees of relative deformation of the samples, remain equiaxed (see Fig. 4, c, d).

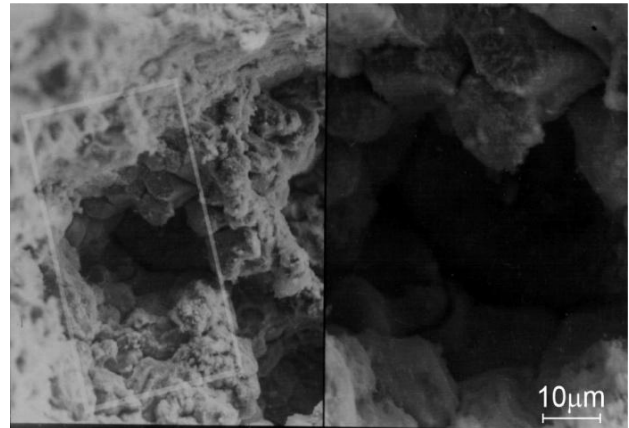


Fig. 3. Fracture fractogram of alloy 1450 sample deformed to failure under the optimal conditions. Scanning electron microscope

It was revealed [13, 16] that the optimal conditions for the appearance of the structural superplasticity effect by samples of alloy 1460, which have the above-described characteristics of the grain structure, are in the investigated stress range  $\sigma$  equal to 3.0...6.0 MPa and in the temperature range  $T = 753...853$  K are as follows:  $T = 793$  K, flow stress  $\sigma = 3.5$  MPa, average strain rate of true deformation  $\bar{\epsilon}_{true} = 3.5 \cdot 10^{-4} \text{ s}^{-1}$ .

The maximum relative elongation of samples to failure that were superplastically deformed under these conditions is 1000% (Fig. 5, a, b).

Fig. 5, b shows a sample that was superplastically deformed to failure by 1000% at  $T = 793$  K and  $\sigma = 3.5$  MPa compared with the initial sample of alloy 1460. It can be seen that the superplastic flow of the sample was stable, and its failure occurred without noticeable localization of deformation in the form of a neck.

Conducting of metallographic studies made it possible to obtain experimental data on the influence of test temperature on grain growth in samples of alloy 1460.

Fig. 6 shows the dependence of the average grain size  $\bar{d}$ , as well as the average grain size  $\bar{d}_{\parallel}$  parallel to the strain direction of the sample and the average grain size  $\bar{d}_{\perp}$  perpendicular to the strain direction, on the test temperature for alloy 1460 samples superplastically deformed to failure.



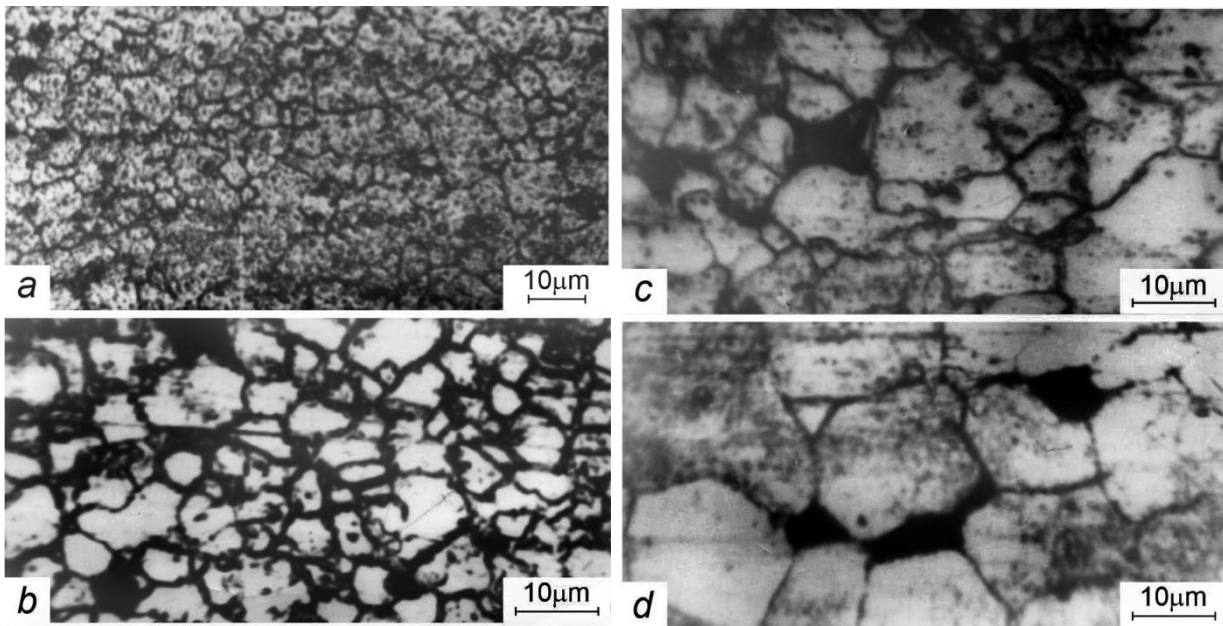


Fig. 4. Characteristic types of microstructures of alloy 1460: in samples in the initial state (a); in samples superplastically deformed at flow stress  $\sigma = 4.0$  MPa and  $T = 773$  K (b);  $\sigma = 3.5$  MPa,  $T = 793$  K (c);  $\sigma = 3.5$  MPa,  $T = 813$  K (c);  $\sigma = 3.5$  MPa,  $T = 823$  K (d). The sample strain direction is horizontal

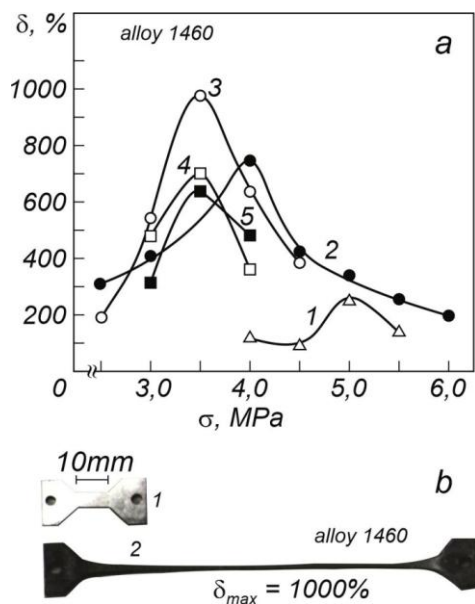


Fig. 5. Dependences of the relative elongation to failure  $\delta$  on the applied stress  $\sigma$  for samples of alloy 1460 superplastically deformed to failure (a) at  $T = 753$  (1), 773 (2), 793 (3), 813 K (4) and the sample deformed to failure by 1000% at  $T = 793$  K and  $\sigma = 3.5$  MPa with the initial one (b)

It is seen that when the test temperature increases to  $T = 823$  K,  $\bar{d}$  increases three times compared to its initial value. The largest grain size  $\bar{d} \approx 15 \mu\text{m}$  is in samples that were deformed to failure at  $T = 823$  K and  $\sigma = 3.5$  MPa. Smaller values of  $\bar{d}$ ,  $\bar{d}_{\parallel}$ ,  $\bar{d}_{\perp}$  are in samples deformed at test temperatures lower and higher than 823 K are probably related to the fact that the duration of their deformation and, therefore, the duration of

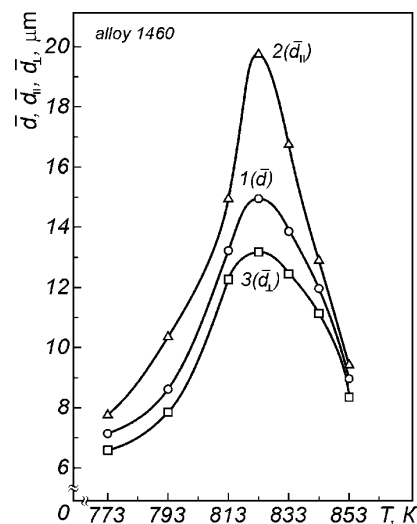


Fig. 6. Dependences of the change in the average grain size  $\bar{d}$  (curve 1), as well as the average grain size in the direction parallel to the strain direction of the sample  $\bar{d}_{\parallel}$  (curve 2) and the average grain size in the direction perpendicular to the strain direction  $\bar{d}_{\perp}$  (curve 3) on the test temperature for samples of alloy 1460, superplastically deformed to failure

dynamic recrystallization of their grain structure, was significantly shorter than in samples deformed at 823 K.

The failure of alloy 1460 samples, which showed superplasticity, takes place after significant accumulation of cavities in their working part. Fractographic studies of fractures showed that in samples that showed maximum plasticity, it is quasi-brittle. Failure takes place by the mixed type without noticeable narrowing in the neck.

It was revealed that there are isolated grain-boundary cavities and macrodiscontinuities elongated along the strain direction, as well as magistral cracks (Fig. 7) on the fracture surface of the samples together with the areas occupied by ultrafine grains that separated from each other as a result of intergranular fracture due to the development of cracks along the grain boundaries.

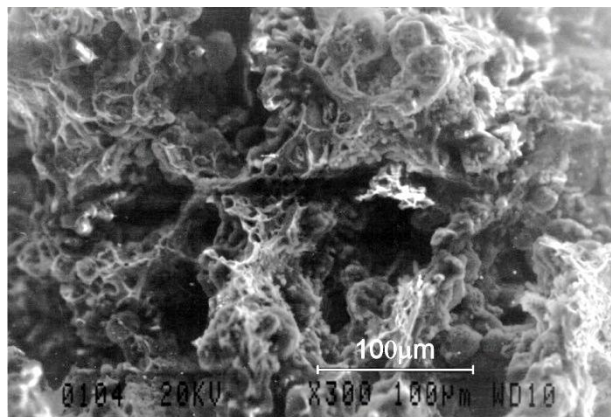


Fig. 7. Fracture fractogram of alloy 1460 sample deformed to failure under the optimal conditions. Scanning electron microscope

## CONCLUSIONS

1. It is found that the presence of dispersed particles of zirconium and scandium aluminides in the studied aluminum alloys 1450 and 1460, as effective microstructure stabilizers, ensures the creation in their samples of an ultrafine-grained structure stable to coarsening during superplastic flow, which occurs at high homologous temperatures.

2. It is found that the recrystallization annealing of alloy 1450 samples, performed in air at different temperatures, does not lead to the formation of a homogeneous fine-grained structure in them. It is shown that almost homogeneous microstructure with an average grain size  $\bar{d} = 3 \dots 6 \mu\text{m}$  is created in them due to the implementation of continuous dynamic recrystallization, which occurs in the presence of dispersed particles of zirconium aluminide  $\text{ZrAl}_3$  in the composition of the alloy, during superplastic deformation at temperatures of 753, 763, 773, 778 K and flow stresses  $\sigma = 2.0 \dots 8.0 \text{ MPa}$ , up to degrees of relative deformation  $\varepsilon_{\text{rel}}$ , which are 20...50%.

3. It was found that during the subsequent stages of superplastic flow, the average grain size in samples of alloy 1450 increases. It is shown that in the samples of this alloy, which were deformed to failure under the optimal conditions, the grains retain equiaxed shape, and their average size is 10...15  $\mu\text{m}$ .

4. It is found that the ultra-fine grain structure of the working parts of alloy 1460 samples with an average grain size  $\bar{d} = 5 \mu\text{m}$ , which was formed during the manufacture of sheet industrial semi-finished product due to the presence of dispersed particles of zirconium and scandium aluminides in their composition, grows during their superplastic deformation, which is performed in the temperature range  $T = 753 \dots 853 \text{ K}$  and at flow stresses  $\sigma = 3.0 \dots 6.0 \text{ MPa}$ . In samples of

alloy 1460 that were deformed to failure by 1000% at  $T = 823 \text{ K}$  and  $\sigma = 3.5 \text{ MPa}$ , the average grain size  $\bar{d} \approx 15 \mu\text{m}$ .

5. The failure of superplastically deformed ultrafine-grained samples of alloys 1450 and 1460 at the macroscopic level is quasi-brittle. It occurs without noticeable localization of deformation in the form of a neck. At the microscopic level, their failure is mixed.

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## НАДПЛАСТИЧНА ДЕФОРМАЦІЯ АЛЮМІНІЄВО-ЛІТІЄВИХ СПЛАВІВ 1450 ТА 1460, ЛЕГОВАНИХ ЦИРКОНІЄМ ТА СКАНДІЄМ

*А.В. Пойда, В.П. Пойда*

Викладені результати досліджень, спрямованих на встановлення механізмів формування ультрадрібно-зернистої структури у зразках алюмінієво-літєвих сплавів 1450 та 1460, що мають у своєму складі дисперсні частинки алюмінідів цирконію та скандію, які є ефективними стабілізаторами мікроструктури, а також на встановлення особливостей її зміни у ході надпластичної деформації.