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Superplastic forming of EK61 nickel-based superalloy with ultrafine-grained structure

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The formation of an ultrafine-grained structure in the heat-resistant nickel-based EK61 superalloy makes it possible to realize the effect of low-temperature superplasticity. This is important for the development of resource-saving technologies for forming hollow products of complex configuration. The study of mechanical properties of the nickel-based EK61 superalloy with ultrafine-grained structure under conditions of uniaxial tension and compression at temperatures of 650–850°C and strain rates of 10^{-3}–10^{-4} s^{-1} was carried out. The process of superplastic forming (SPF) under conditions of low-temperature superplasticity of ultrafine-grained sheet blanks from the EK61 superalloy using cylindrical matrix has been studied. It was found that after SPF, a homogeneous equiaxed ultrafine-grained duplex structure is preserved in the samples.

Keywords: wrought heat-resistant nickel-based superalloy, multiple isothermal forging, low-temperature superplasticity, mechanical properties, superplastic forming.

1. Introduction

Heat-resistant nickel-based superalloys are a unique class of materials capable of operating at elevated temperatures and in aggressive environments due to their complex chemical composition. Therefore, superalloys are widely used for the manufacture of various parts of aircraft gas turbine engines, as well as rocket engines [1–6]. The complex chemical composition of modern superalloys, which ensures the achievement of the required characteristics of heat-resistant properties due to solid-solution hardening and the precipitation of intragranular coherent particles of strengthening phases, for example, intermetallic particles based on $Ni_3(Al, Ti)$, has led to a significant decrease in their technological plasticity and an increase in laboriousness during processing. At the same time, the fabrication of hollow thin-walled structures for gas turbine and rocket engines from Ni-based superalloys is a very actual scientific and technical problem. To manufacture such structures from heat-resistant superalloys by hot sheet stamping at temperatures above 1000°C is very difficult task. In this regard, superplastic forming under low-temperature superplasticity conditions seems to be a promising method of processing at temperatures not exceeding 900°C.

It is known [6–9] that the most effective way to increase the technological plasticity of superalloys is the formation of an ultrafine-grained structure in bulk semi-finished products. This is a necessary condition for realizing the effect of structural superplasticity in technological processes for manufacturing parts of complex shape from such superalloys, for example, by the method of superplastic forming.

The implementation of an efficient technology for manufacturing parts of complex geometry is possible by forming an initial ultrafine-grained structure in workpiece that provides high plasticity and low flow stresses during deformation processing [6,7,10–20]. This is important especially in relation to hard-to-deform superalloys [8,16,21,22]. Deformation of materials with nano- and ultrafine-grained structures creates an opportunity for shaping at the temperatures of manifestation of both traditional and low-temperature superplasticity superalloys based on titanium [12,14,20,23–27] and nickel [16,21,22]. The multiple isothermal forging method turned out to be very effective for obtaining a homogeneous ultrafine-grained structure in bulk large-sized semi-finished products from superalloys based on titanium and nickel [6,7,26].

Using of low-temperature superplasticity for the manufacture of parts having complex geometry from heat-resistant hard-to-deform nickel-based superalloys is particularly important. For instance, in [21,22], using the Inconel 718 as an example, it was shown that the use of sheet semi-finished products with an ultrafine-grained structure (about 0.3 μm) makes it possible to reduce the level of flow stress by a factor of 1.5 compared to the fine-grained state (about 6.0 μm). Due to the effect of low-temperature superplasticity, it is possible to reduce the temperature of the superplastic process of sheet blanks down to 900–850°C.

The purpose of this work is to study the evolution of the structure and mechanical properties in the EK61 superalloy having ultrafine-grained structure at low processing temperatures, as well as to evaluate the formability of its sheet blanks under low-temperature superplasticity conditions.

2. Materials and methods

The material under investigation was wrought heat-resistant EK61 nickel-based superalloy, which is the closest analogue of the Inconel 718 in terms of chemical and phase compositions. The chemical composition of EK61 is 58Ni-16.6Cr-15Fe-3.9Mo-5Nb-Al-Ti-Cu-V [28–30]. Russian wrought EK61 superalloy is a representative of heat-resistant superalloys hardened by the γ''-phase [4,6,28]. The deformation-heat treatment was carried out to obtain an ultrafine-grained structure of a duplex type in the EK61 superalloy. Such processing was performed using the well-known [7,9,18,30] method of multiple isothermal forging developed at IMSP RAS [7,18].

Deformation-heat treatment of the EK61 superalloy using the scheme of multiple isothermal forging was carried out in the temperature range of $(0.71-0.93)T_{\delta}$ (T_{δ} is the temperature of complete dissolution of the δ-phase). The multiple isothermal forging was carried out using an isothermal die block mounted on a hydraulic press with a force of 6.3 MN. Such processing was performed at the conditions established earlier in the temperature range of 950–750°C with a step-by-step decreasing of the temperature [9,11,16,28]. The total compression strain for forged billet was $e \approx 11$.

The study of the superplastic properties of the EK61 superalloy with an ultrafine-grained structure in uniaxial tensile tests were carried out using an Instron-5982 testing machine. The experiments were carried out on flat samples with a working part length of 10 mm, a thickness of 2 mm, and a width of 3 mm. Mechanical tests in uniaxial compression were carried out on cylindrical samples with a diameter of 10 mm and a height of 15 mm. The compression tests were carried out using a Schenck RMS-100 universal dynamometer with a load of up to 100 kN. The microstructure was studied by Mira 3LMH (TESCAN) scanning and JEM-2000EX transmission electron microscopes.

Studies of superplastic formability were carried out according to the original method [22] using special equipment with a cylindrical matrix 30 mm in diameter (Fig. 1a). The figure shows an intermediate stage of forming. The second sheet is auxiliary, that has a hole for the passage is of the working gas for forming in its center there the sheet under study (the hole is shown by the red arrow, Fig. 1a). It is necessary to seal the workpiece under study. A fitting is welded to the auxiliary sheet. The investigated sheet is welded to the auxiliary sheet along the perimeter by electro-contact welding. Figure 1b shows a sample before forming with a fitting and an auxiliary sheet (red arrows show the welded joint of the auxiliary sheet with the test one, Fig. 1b).

The superplastic forming of polished sheets in size of 40×40 mm and the thickness of 0.7 mm with an ultrafine-grained structure made of EK61 superalloy was carried out at a temperature of 850°C with a strain rate of $3\cdot10^{-3}$ s^{-1}. The depth H of the matrix for the experimental forming of hollow cylindrical specimens was 5, 10 and 15 mm.

After superplastic forming the thicknesses of the sample in the cross section and its microstructure were studied. According to the recommendations given in [17], the value of the accumulated strain at a random point of the samples after forming (1) was determined by the formula:

$$e = (1.000 - 1.1555)\cdot \ln (S_0 / S), \quad (1)$$

where S_0 and S are the thicknesses of the initial workpiece and the model sample at the considered point, respectively.

For the convenience of comparing the obtained data with known experiments on uniaxial tension, the equivalent strain (2) was determined:

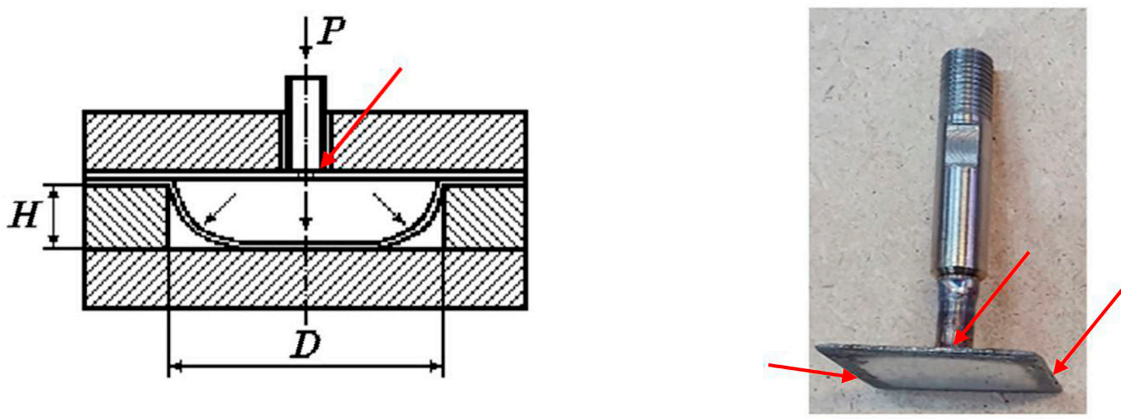
$$\varepsilon = [\exp(e) - 1]\cdot 100\% \quad (2)$$

3. Results and discussion

3.1. Ultrafine-grained structure of EK61 superalloy

In the initial state, the superalloy under investigation was a hot-deformed billet of 80 mm in diameter with an initial coarse-grained structure of γ-phase matrix with a grain size of 62 μm. In such coarse grains of matrix, the coherent nanosized (≈40 nm) particles of the strengthening γ''-phase were precipitated [28].

As it is known [1], in nickel-based superalloys of the EK61 and Inconel 718 types, the metastable strengthening γ''-phase with body-centered tetragonal crystal lattice in the process of prolonged annealing at temperatures above 650°C transforms into the orthorhombic lattice of the δ-phase. At the same time, a distinctive feature of these γ''- and δ-phases is that the incubation period of their precipitation is more than 10 minutes. Apparently, this circumstance is a significant



a b

Fig. 1. (Color online) Scheme of the superplastic forming (a) and general view of the samples before the superplastic forming (b).

factor that ensures the high technological plasticity of Inconel 718 and EK61 superalloys during their deformation-heat treatment.

The ultrafine-grained structure of the mixed type (Fig. 2 a), obtained as a result of deformation-heat treatment with a stepwise decrease of the temperature in the processing, is similar in morphology and size to the nanoduplex type, which was obtained in [9,11]. The grain size of the γ-phase and particles of the δ-phase was 0.3 ± 0.1 μm (Fig. 2 b). At the same time, along with the ultrafine-grained component, individual relatively large particles of the δ-phase up to 2.1 ± 0.5 μm in size are observed, which are preserved and are indicated us "hereditary", i.e., previously formed on the first stage of microduplex structure. The total volume fraction of small and relatively large particles of the δ-phase is 29 ± 3%. Similar results were obtained in the Inconel 718 superalloy [21]. In Inconel 718, a nanocrystalline structure was formed by deformation-heat treatment, which is stable up to 700°C. But there are no relatively large particles of the δ-phase, there are only γ- and δ-phases up to 0.3 – 0.5 μm.

3.2. Superplasticity of EK61 superalloy with ultrafine-grained structure

According to the results of mechanical tensile tests (Fig. 3 a), it was found that the best superplastic properties of ultrafine-grained EK61 superalloy were in the temperature range of 675 – 850°C and strain rates of 10^{-3}–10^{-4} s^{-1}. Deformation of specimens under these strain-rate conditions makes it possible to achieve high values of relative elongation, up to 1741%. In this case, the rate sensitivity coefficient of the flow stress on the strain rate *m* is 0.39 – 0.59.

An analysis of the mechanical properties during uniaxial compression tests (Fig. 3 b) showed that an increase in the deformation temperature up to 850°C and above led to a significant decrease in the flow stress level, approximately by a factor of 8. The maximum value of the strain ε about 68% is observed in the temperature range T = 800 – 850°C and strain rates $\dot{\varepsilon} = 10^{-3} - 5\cdot10^{-4}$ s^{-1}. The statistical measurement error was 7%.

It should be noted that the formation of the ultrafine-grained structure with an average grain size of γ- and δ-phases of about 0.3 ± 0.1 μm leads to a significant decrease in the temperature of the superplasticity, by 200 – 250°C, compared to the fine-grained state [6]. At low temperatures (675 – 750°C), the ultrafine-grained structure is stable. With an increase in temperature to 800 – 850°C, partial dissolution of the δ-phase is observed, which, apparently, leads to coarsening of the grains of the γ-phase. An increase in temperature up to 850°C reduces the volume fraction of the δ-phase down to 15%, and, as a result, increases the average grain size of the γ-phase to about 0.8 ± 0.2 μm (Fig. 4 a). At the same time, as follows from the analysis of the structure shown in Fig. 4 b, in the process of superplastic deformation, the dynamic recrystallization is likely to develop, leading to the growth of grains of the γ-phase up to sizes of 1.5 – 2 μm. The variation coefficient for particles of the δ-phase from the head in the direction of the fracture zone of the sample varies

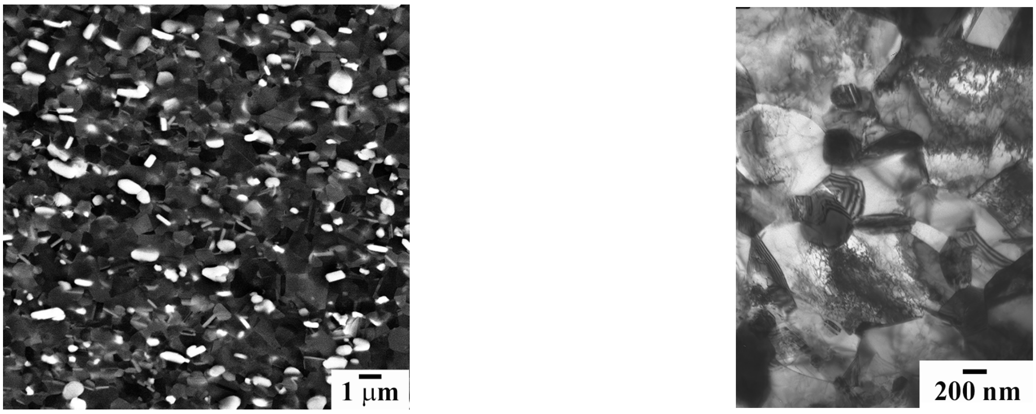


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Fig. 2. Ultrafine-grained mixed type structure of the EK61 superalloy.

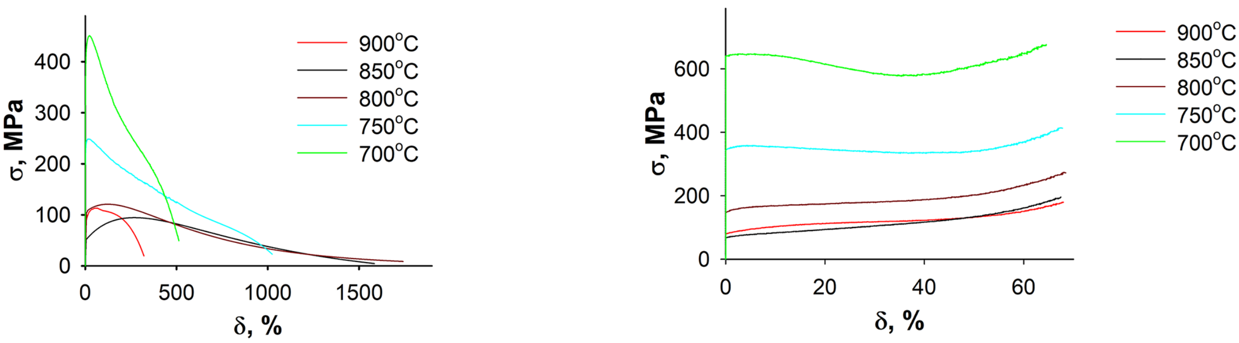

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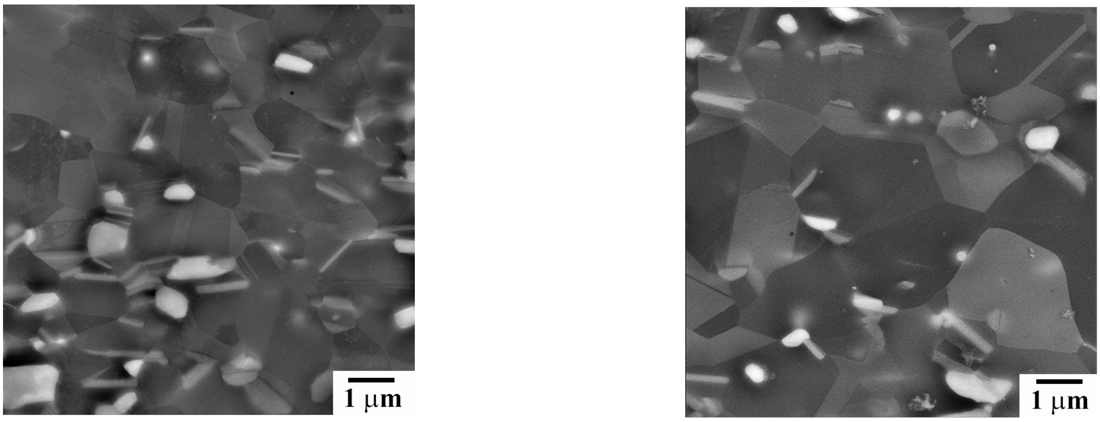
Fig. 3. (Color online) Results of mechanical testing of ultrafine-grained EK61 superalloy at strain rate $\dot{\varepsilon} = 10^{-3}$ s^{-1}: uniaxial tension (a), compression (b).

from 1.95 to 1.71, while the volume fraction of particles in the head is 20%, and in the base deformed zone is 15% (Fig. 4).

The study of the microstructure of samples from the EK61 superalloy with a preliminarily prepared ultrafine-grained structure, deformed under low-temperature superplasticity conditions, indicates that after deformation, according to both the uniaxial tension scheme (Fig. 4) and the uniaxial compression scheme (Fig. 5), the equiaxed grain shape is preserved. In this case, the ultrafine-grained structure is relatively stable, which is probably determined by the presence of incoherent grain particles of the δ phase.

3.3. Superplastic forming of EK61 superalloy having ultrafine-grained structure

The most important results are presented in this section. The temperature condition of $T=850$°C for superplastic forming of EK61 was determined based on the previously mentioned results. According to the results of the experiments, it was found that in all cases the formation of sheet samples from the EK61 superalloy with an ultrafine-grained structure took place without destruction. The model samples had a regular cylindrical shape and were characterized by good filling of the matrix cavity (Fig. 6).



a b

Fig. 4. Microstructure of EK61 with ultrafine-grained structure of mixed type after uniaxial tension of flat sample at $T=850$°C, $\dot{\varepsilon}=10^{-3}$ s^{-1}: head zone (a), base deformed zone (b).

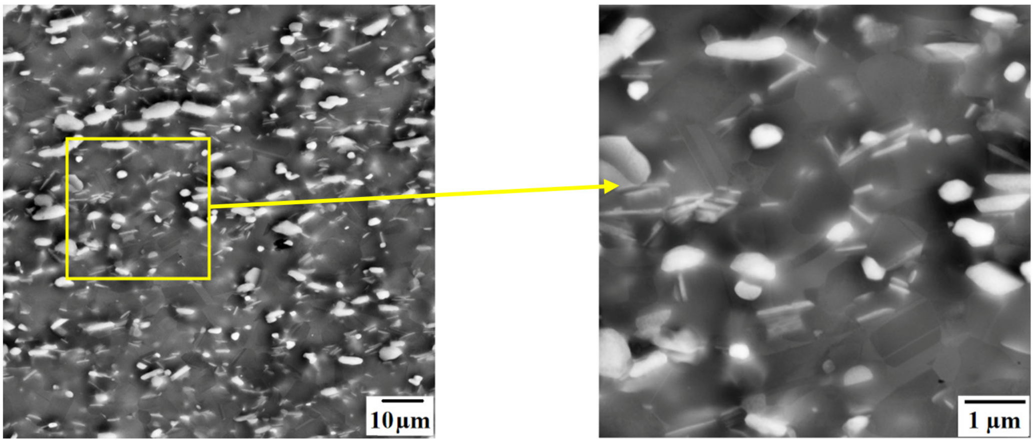


Fig. 5. Microstructure of the EK61 with ultrafine-grained structure of mixed type after deformation according to the scheme of uniaxial compression at $T=850$°C, $\dot{\varepsilon}=10^{-3}$ s^{-1}.

a b c d

Fig. 6. (Color online) Samples after superplastic forming at $T=850$°C to the depth: $H=5$ mm (a); $H=10$ mm (b); $H=15$ mm (c, d).

Tables 1 and 2 show the results of a study of the thickness and equivalent strain over the cross section of the molded specimens. It was found that with an increase in the forming depth from 5 to 15 mm, a decrease in the thickness of the side wall from 0.4 to 0.22 mm is observed (Table 1). Similar changes in wall thickness of hollow samples were found in the center of the base and in the angular zone. According to the results of the experiments, it was found that the maximum strain is observed in the angular (radial) zones of the samples. Maximum strain is no 380% for samples formed to a maximum depth of 15 mm (Table 2) in angular zone.

It should be noted that for the first time a possibility of superplastic forming of nickel-based superalloy was demonstrated in [16]. The authors of [16] got hollow cylindrical model samples fabricated at a temperature of 900°C from Inconel 718, a heat-resistant nickel-based superalloy with an UFG structure. A technological feasibility of lowering the temperature of superplastic forming of UFG sheet blanks from the EK61 superalloy down to 850°C is of real interest for the development of breakthrough technologies for shaping hard-to-deform nickel-based superalloys.

Microstructural analysis of the material of hollow samples after superplastic forming (Fig. 7) showed that the structure was duplex in all cases, the equiaxial shape of the γ-phase grains was preserved. Thus, at the deformation temperature, the grain size of the γ-phase was about 1.0 ± 0.2 μm, while the ultrafine-grained matrix structure of the duplex type remains equiaxed. With an increase in the value of the strain during superplastic forming, some coarsening of grains and a change in the phase composition are observed (Fig. 7): at a depth of $H = 5$ mm, the grain size of the γ-phase is about 1.7 ± 0.3 μm; at $H = 10$ mm, about 2.0 ± 0.3 μm; and at $H = 15$ mm about 2.4 ± 0.3 μm, and the volume fraction of the δ-phase is about 20 ± 4%.

Table 1. Changes in the wall thickness of hollow samples made by superplastic forming.

The depth, mm	Thickness, mm		
	angular zone	center zone	side wall zone
5	0.30	0.38	0.40
10	0.25	0.28	0.33
15	0.08	0.17	0.22

Table 2. Equivalent strain in different zones of hollow specimens.

The depth, mm	Equivalent strain, %		
	angular zone	center zone	side wall zone
5	149.2	93.1	82.8
10	203.3	168.4	111.0
15	380.8	359.5	248.0

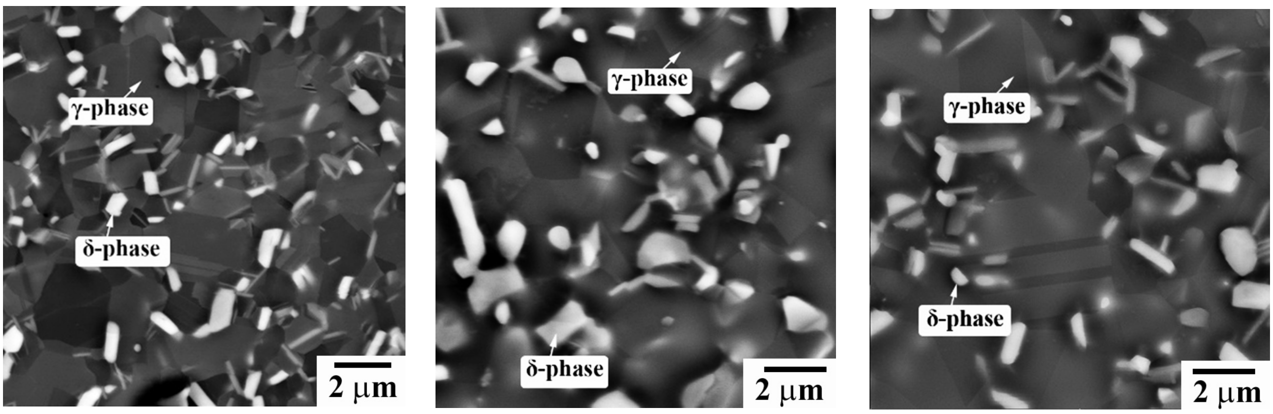
Thus, the analysis of the obtained data shows that sheet blanks from the EK61 superalloy with a preliminarily prepared ultrafine-grained structure have good superplastic formability and, therefore, can be in demand for the manufacture of various types of complex-profile thin-walled hollow billets. One of the examples of such parts is spherical pressure vessel obtained by the superplastic forming method. Compared with the known results of studies previously carried out on the Inconel 718 superalloy [16, 21, 22], the possibility to additionally lower the superplastic forming temperature by 50°C was demonstrated. Thus, with an increase of the strain during superplastic forming, a coarsening of grains from 0.3 to 2.4 μm and a change in the phase composition of the superalloy under study are observed. A comparative analysis of the obtained results with the known data [16, 21, 22] indicates that the superplastic behavior of EK61 and Inconel 718 having ultrafine-grained structure is very similar.

4. Conclusions

1. Formation the ultrafine-grained structure with an average grain size of γ- and δ-phases of about 0.3 ± 0.1 μm leads to implementation of low-temperature superplasticity in EK61 superalloy in the temperature range of 675 – 850°C. The maximum superplasticity characteristics are achieved at 850°C.

2. The superplastic forming process of sheet from the EK61 superalloy with ultrafine-grained structure using a cylindrical matrix was experimentally studied under the temperature-rate conditions at 850°C of the superplasticity. It is shown that the macrodeformation of model hollow samples during superplastic forming is relatively homogeneous and uniform.

The obtained data give evidence to the high superplastic properties of the EK61 superalloy with an ultrafine-grained structure which can be used in innovative technological processes of low-temperature superplastic forming in order to form various types of hollow parts of complex configuration for aerospace technology.



a b c

Fig. 7. Microstructure of the EK61 after superplastic forming of hollow samples: $H = 5$ mm (a); $H = 10$ mm (b); $H = 15$ mm (c).

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