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Low-temperature superplastic deformation of EK61 and EP975 wrought nickel-based superalloys with an ultrafine-grained structure

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It is shown that thermomechanical treatment (TMT) of the EK61 superalloy with a strain rate ($\dot{\varepsilon}$) of 10^{-3} s^{-1} and a gradual decrease in the treatment temperature from $(0.92-0.65)T_{\delta}$ leads to the transformation of the initial coarse-grained structure into an ultrafine-grained (UFG) mixed type structure. Such a mixed UFG microstructure consists of γ-phase grains and δ-phase particles 0.3 μm in size and relatively large particles of the δ-phase up to 2 μm in size. The EK61 superalloy having such a microstructure exhibits low-temperature superplasticity in the temperature range of 700–900°C, the maximum characteristics of which are achieved at 850°C and $\dot{\varepsilon}=10^{-3}$ s^{-1}. Low-temperature TMT in the temperature range of $(0.84-0.8)T_{\gamma'}$ also leads to the formation of an UFG structure in the EP975 superalloy. The UFG microstructure of the EP975 superalloy consists of γ-phase grains and γ'-phase particles 0.5 μm in size. And there are relatively large particles of the γ'-phase up to 3.5 μm in size of a globular-shaped form. The EP975 superalloy with such a structure exhibits low-temperature superplasticity in the temperature range of 900–1000°C, the maximum characteristics of which are achieved at 1000°C and $\dot{\varepsilon}=10^{-3}$ s^{-1}.

Keywords: superalloy, low-temperature superplasticity, mechanical properties, deformation.

1. Introduction

Currently, materials scientists are actively working on the development and wider application of severe plastic deformation methods [1–6]. Such methods are aimed at obtaining an ultrafine-grained (UFG) microstructure and nanostructural states in bulk and sheet semi-finished products from various metals and alloys, as well as from high-temperature nickel-based superalloys.

It should be noted that modern heat-resistant superalloys have a complex chemical composition including more than 10 alloying elements [7–11]. In addition, superalloys differ significantly in morphology and kinetics of hardening phase precipitates [8–10]. It is obvious that the deformation behavior of superalloys, the nature of the recrystallization processes occurring both under dynamic and static conditions, significantly depend on the physical and mechanical characteristics of the hardening phase (incubation period of its precipitation, morphology, type of bond with the matrix), as well as processing conditions. Thus, in superalloys of the EP975 type the strengthening γ'-phase (Ni_3Al) precipitates almost instantly upon cooling in a spherical or cuboid form [7,12]. EP975 superalloy, used for the manufacture of parts of gas turbine engines, has low technological plasticity. This is probably largely due to the extremely short incubation period for the isolation of the strengthening γ'-phase, which has the same face-centered lattice as the matrix (γ-phase) and a very small lattice mismatch parameter of the γ and γ'-phases. Therefore, the formation of a fine-grained structure of the microduplex type in massive workpieces from the EP975 superalloy is one of the necessary conditions for subsequent deformation under superplasticity conditions [1,7,8]. At the same time, in the EK61 superalloy, which is a close analogue of the foreign Inconel 718 superalloy in terms of chemical and phase composition, during TMT, the δ-phase (Ni_3Nb) is released. The incubation period of δ-phase is 5–10 min [8,13,14]. Apparently, the long incubation period for the precipitation of the hardening phase in the Inconel 718 and EK61 superalloys ensures high technological plasticity of these superalloys during TMT in two phases of δ+γ region. For these superalloys, the use of the multiple isothermal forging (MIF) scheme is very effective [1,2,6,15–17]. There are literature data devoted to investigation of the UFG microstructure formation in nickel-based superalloys [1,15–27]. In [15,25–27], a method was proposed to obtain bulk semi-finished products from nickel-based heterophase superalloys with a fine-grained and UFG duplex-type structure. This one provides to be effective in the TMT of various precipitation hardening nickel superalloys, for example, EP962, EP742, EP741NP, Waspaloy, Astralloy [1,6,15,21,27], as well as in the processing of a new Re-containing SDZhS-15 superalloy [26]. As a result of such processing of superalloys, a stage-by-stage refinement of the structure can be achieved. At the first high-temperature stage of TMT, carried out at high temperatures $(0.95-0.92)T_s$, where T_s is the temperature of the dissolution of the second phase, for example, γ'-phase), in bulk workpieces from hard-to-deform superalloys, the initial coarse-grained structure is transformed into a structure of the microduplex type with a grain and phase size of 1–10 μm [1,15–17]. At the second low-temperature stage of TMT, due to a significant reduction in the treatment temperature to $(0.84-0.65)T_s$, the microduplex structure can be transformed into a UFG structure of the duplex type [18–24]. A necessary condition for the realization of superplasticity in superalloys is the formation of a fine-grained or UFG structure of the duplex type in workpieces. The expansion of the technological possibilities of the effect of superplasticity in the processing of deformable heat-resistant superalloys is possible by increasing the rate of deformation and a beautiful temperature up to $(0.8-0.6)T_s$.

The purpose of this work is to study the effect of TMT on the formation of a mixed-type UFG structure and its effect on the manifestation of the low-temperature superplasticity effect in nickel-based EK61 and EP975 superalloys with types of hardening phases.

2. Materials and methods

Wrought heat-resistant nickel-based EK61 and EP975 superalloys with types of hardening phases were chosen as materials for the study. In the EK61 hardening is achieved by separating coherent particles of the metastable γ''-phase based on the Ni_3Nb intermetallic phase. The noted phase is metastable; during long-term aging or TMT it is transformed into a thermally stable δ-phase. In the EP975 hardening is achieved due to the precipitation of coherent particles of the γ'-phase based on the $Ni_3(Al, Ti)$ intermetallic phase.

As the starting material from EK61 a billet 80 mm in diameter and 90 mm high, cut from a hot-deformed bar 80 mm in diameter with an initial coarse-grained structure was used. A billet $40\times50\times70$ mm^3 were used as the starting material from the EP975. From the last billet a sample with a diameter of 400 mm and a thickness of 40 mm with a uniform fine-grained structure were cut/from of the microduplex type was formed in the process of high-temperature deformation-heat treatment $(0.95-0.92)T_s$. To obtain the UFG structure in the superalloys under study, the TMT was carried out using the MIF scheme developed at the IMSP RAS [1,6]. The TMT was performed on a hydraulic press with a force of 6.3 MN, equipped with an isothermal stamp block in the temperature range: $(0.93-0.65)T_\delta$ (T_δ — dissolution temperature of δ-phase) for EK61, and in the temperature range $(0.84-0.8)T_{\gamma'}$ ($T_{\gamma'}$ — dissolution temperature of γ'-phase) for EP975. The strain rate was $\dot{\varepsilon}\approx10^{-2}-10^{-3}$ s^{-1}.

The microstructure of the EK61 and EP975 superalloys were studied by Mira 3LMH TESCAN scanning and JEM-2000EX transmission electron microscopy. Tensile tests were carried out using an Instron 5982 testing machine, and compression tests were carried out on a Schenck RMS-100 universal dynamometer. Both tensile tests and compression tests were carried out under isothermal conditions. The EK61 and EP975 superalloys having UFG microstructure have been subjected tensile and compression tests. To study superplastic properties, the flat samples with a working part length of 10 mm, a thickness of 2 mm, and a width of 3 mm were used. The cylindrical specimens with a diameter of 10 mm and a height of 15 mm were used for the mechanical uniaxial compression tests.

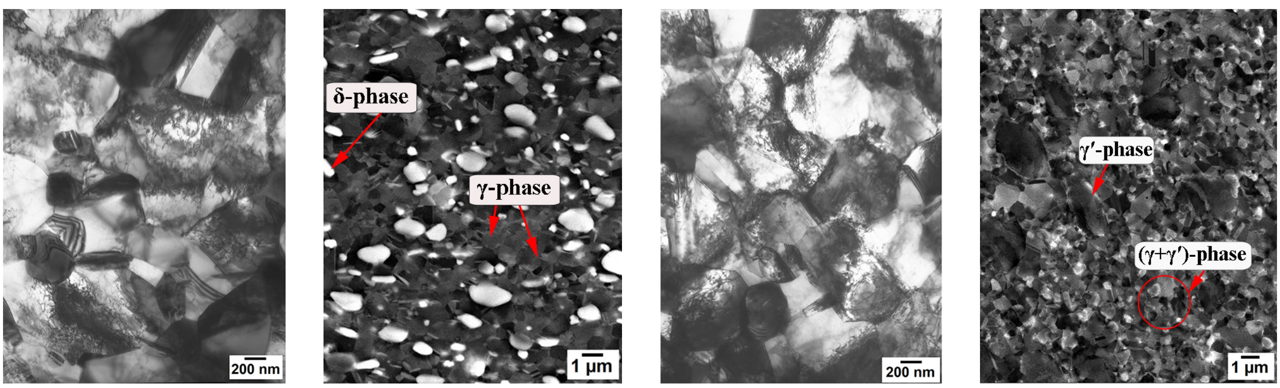
As known [1,6], one of the main factors indicating the manifestation of the effect of superplasticity in alloys is the coefficient of strain rate sensitivity *m*. It is assumed that at *m* close to 0.3 the material exhibits a superplastic property. The coefficient *m* was determined by the method of stepwise change in the strain rate at different temperatures according to the following formula: $m=\lg(\sigma_2/\sigma_1)/\lg(\dot{\varepsilon}_2/\dot{\varepsilon}_1)$.

3. Results and discussion

3.1. Formation of a mixed-type UFG structure in EK61 and EP975 superalloys after TMT

The initial structure of workpieces from the EK61 superalloy was a homogeneous coarse-grained one with an average grain size of the γ-phase of about 62 μm, inside which coherent nanosized (≈40 nm) particles of metastable strengthening (Ni_3Nb) were homogeneously precipitated [17]. The initial structure of the EP975 superalloy showed that it was a typical microduplex structure: the average grain size of the γ-phase was equal to 8 μm and that of the relatively large particles-grains of the strengthening γ'-phase was 3.2 μm, the volume fraction of the latter was $V_{\gamma'}=28\%$. Inside grains of the γ-phase, dispersed (0.4 μm) particles of the γ'-phase were observed, which usually precipitated upon cooling from the forging temperature to room temperature [28].

The TMT using the MIF scheme with a gradual decrease in the treatment temperature from $0.93T_\delta$ to $0.81T_\delta$ and a strain rate of 10^{-3} s^{-1} of EK61 superalloy provides a gradual transformation of the initial coarse-grained structure into a fine-grained microduplex type. It was possible to achieve additional refinement of the microduplex structure to the UFG state due to further low-temperature TMT carried out in the range of $(0.81-0.65)T_\delta$. In the entire volume of



a b c d

Fig. 1. The UFG structure of superalloys after MIF at strain rate $\dot{\varepsilon}=10^{-3}$ s^{-1}: EK61 (a, b), EP975 (c, d).

the deformed material, an UFG structure of a mixed type is formed, in which the UFG component is close in morphology to the nanoduplex type (Fig. 1a). The grain size of the γ-phase and particles of the δ-phase was ≈0.3 μm, and the volume fraction of the δ-phase was $V_\delta = 24 \pm 3\%$ (Fig. 1b). At the same time, along with the UFG component, individual relatively large particles of the δ-phase up to about 2 μm in size are observed in the superalloy structure. The volume fraction of such relatively large particles is $V_\delta = 5 \pm 1\%$. The noted particles are preserved and possible there are "hereditary", that is, previously formed at the stage formation of a microduplex structure.

The low-temperature TMT in the temperature range $(0.84-0.8)T_{\gamma'}$ using the MIF scheme also led to the formation of a UFG structure of a mixed type. Such a mixed type microstructure includes an UFG component of the duplex type with an average size of new precipitates of the γ'-phase and grains of the γ'-phase 0.5 μm (Fig. 1c, d) and relatively large globular-shaped particles-grains of the γ'-phase ≈3.5 μm in size are relatively evenly distributed. Such relatively large γ' particles were previously formed at the stage of microduplex structure formation in the process of high-temperature TMT. Thus, two types of particle sizes of the γ'-phase can be distinguished: relatively large ones — their volume fraction was $V_{\gamma'} = 13 \pm 3\%$; UFG component — precipitates of the γ'-phase, the size of the γ-phase is 0.5 μm, their volume fraction was $V_{\gamma'} = 30 \pm 3\%$ (Fig. 1d).

A distinctive feature of the UFG structure of the mixed type in comparison with the microduplex one is that the UFG state is characterized by large length of interphase and high-angle grain boundaries. This contributes to the activation of diffusion processes at low temperatures and the active mechanisms of superplastic deformation. Due to this, it becomes possible to implement the effect of low-temperature superplasticity in the studied precipitation-hardening nickel-based superalloys with different types of hardening phase (γ'; γ''+δ) with a UFG mixed-type structure. Superplasticity in the studied superalloys with a UFG structure manifests itself at temperatures 150–250°C lower, and a decrease in the flow stress level by a factor of 1.2–1.5 is achieved compared to materials with a microduplex structure. The efficiency of the TMT to formation UFG microstructure is more obvious in comparison with the initial microstructure of superalloys under study. The initial microstructure of superalloys under study is presented in Fig. S1 (supplementary material).

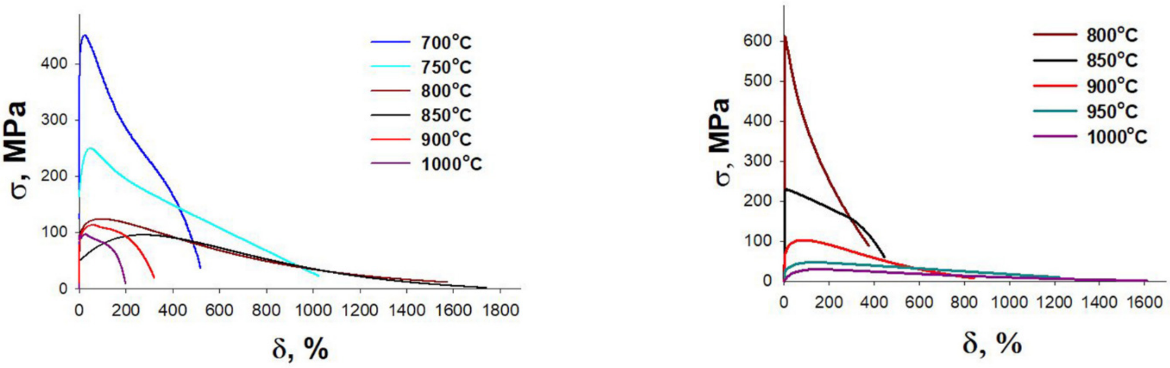
3.2. Superplasticity of EK61 and EP975 superalloys with UFG structure under conditions of uniaxial tension

The results of mechanical tensile tests of EK61 and EP975 superalloys are shown in Fig. 2.

It has been established that the effect of low-temperature superplasticity in the EK61 superalloy with UFG structure manifests itself at temperatures close to the aging temperature of 700-850°C (Fig. 2 a). The relative elongation is 510–1741%, and the coefficient of strain rate sensitivity *m* is 0.3–0.59. The formation of a UFG structure of a mixed type in the EP975 superalloy provides a decrease in the temperature of manifestation of superplasticity compared to the fine-grained state in which the temperature of the superplasticity was 1100–1125°C [1,7]. In superalloy with a UFG structure, the effect of low-temperature superplasticity manifests itself in the temperature range of 800–1000°C (Fig. 2b). The relative elongation is 375–1620%, and the coefficient of strain rate sensitivity *m* is 0.3–0.5.

It is interesting to note that results similar to those for the EP975 were obtained earlier in [21]. In this work in granular EP741NP superalloy it is shown that as a result of low-temperature TMT, a mixed-type UFG structure is also formed, which shows signs of low-temperature superplasticity in the temperature range of 900–1000°C, reaching the highest value ($\delta = 1320\%$) at $T = 1000$°C and $\dot{\varepsilon} = 10^{-3}$ s^{-1}. Thus, the formation of an UFG structure in the superalloy leads to a significant decrease in the superconducting temperature by 200–250°C as compared to the microcrystalline microduplex state [1,21].

Figure 3 shows the microstructure of the EK61 after superplasticity tests. A characteristic feature of the superplastic deformation is the preservation of the equiaxed shape of the γ-phase grains. At the same time, it should be noted that at significant degrees of deformation ($\delta \geq 500\%$), precipitates of δ-phase particles are observed, which are elongated mainly in the direction of tensile deformation. The formation of new thin plates of the δ-phase was also found, which are mainly existed along the grain boundaries of the matrix γ-phase. In the structure, in the working part of the deformed sample, single rounded and/or elongated pores with a size of no less than 0.1 μm are found. Near the fracture zone, the pores line up in chains about 10–20 μm long, and thus, apparently, accelerate the process of sample fracture.



a b

Fig. 2. (Color online) Results of mechanical tensile tests of UFG superalloys at strain rate $\dot{\varepsilon} = 10^{-3}$ s^{-1}: EK61 (a), EP975 (b).

At low temperatures (700 – 750°C), the UFG structure is stable. With an increase in temperature to 800 – 850°C, a partial dissolution of the δ-phase is observed, which, apparently, leads to coarsening of the grains of the γ-phase. An increase in temperature to 850°C reduces the volume fraction of the δ-phase to 15% and, as a result, increases the average grain size of the γ-phase up to about 0.8 μm (Fig. 3 a). Simultaneously, in the process of superplastic deformation, the process of dynamic collective recrystallization is likely to develop, leading to the growth of grains of the γ-phase up to sizes of 1.5 – 2 μm (Fig. 3 b).

Figure 4 shows the microstructure of the EP975 after superplasticity tests. According to the results of microstructure investigation after tensile testing, it was found that in the EP975 at deformation temperatures of 800 – 850°C, the microstructure remains stable.

An increase in the deformation temperature from 900°C to 950°C leads to coarsening of grains by about 2 times. With an increasing temperature up to 1000°C, the grain size increases to 3.5 μm in the grip zone (Fig. 4 a) and up to 5.2 μm in the working part of the sample (Fig. 4 b). Such difference is associated with partial dissolution of the γ'-phase at a temperature of 1000°C, which is 50°C higher than the aging temperature of the EP975 superalloy. In the working part of the samples at the steady state stage of superplastic deformation, single rounded pores no larger than 0.1 μm in size are found.

3.3. Superplastic deformation of EK61 and EP975 superalloys with an UFG structure under uniaxial compression

The temperature range for the uniaxial compression testing for the EK61 superalloy 700 – 900°C. An analysis of the mechanical properties for the EK61 superalloy showed that an increase in the deformation temperature up to 800°C and above leads to a significant decrease in the flow stress level (Fig. 5 a). The temperature range for the uniaxial compression testing for EP975 was chosen based on the results of tensile tests at which the maximum characteristics were achieved. The compression tests of EP975 with UFG structure were carried out at in the temperature range of 950 –1000°C with a strain rate of 10^{-3} s^{-1}. From the analysis of the results of mechanical tests for uniaxial compression ($\varepsilon = 60 – 70\%$) of the EP975 a typical for superplastic deformation dependence of the flow stress on the deformation was observed (Fig. 5 b). A similar picture of the change in mechanical properties upon superplastic deformation was observed in other nickel-based superalloys [1, 6, 16].

Figure 6 shows the microstructure after deformation both EK61 (Fig. 6 a) and EP975 (Fig. 6 b). After deformation of EK61 superalloy in the temperature range of 700 – 900°C, the following changes in the microstructure parameters were observed. With an increase in temperature in the indicated range, due to the partial dissolution of the second phase, a

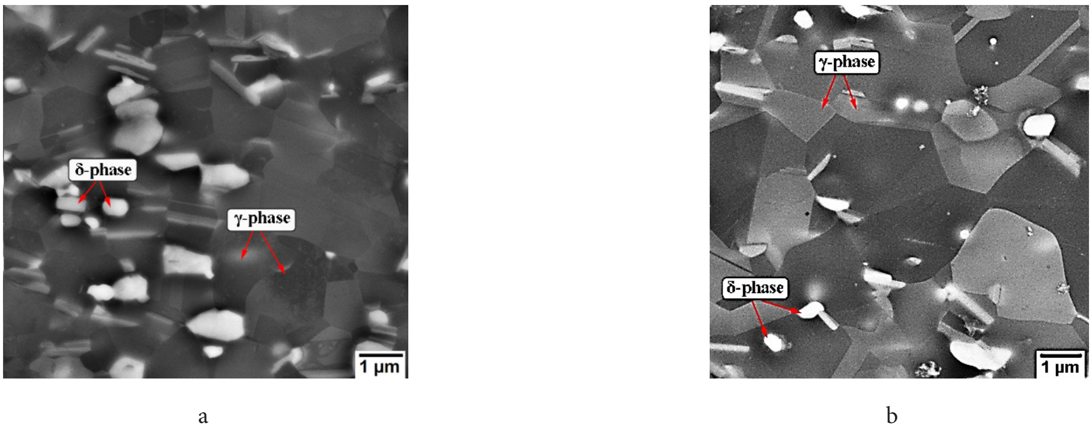


Fig. 3. Microstructure of the EK61 with UFG structure of mixed type after superplastic deformation according to the scheme of uniaxial tension at $T = 850$°C, $\dot{\varepsilon} = 10^{-3}$ s^{-1}: grip zone (a), gauge section (b).

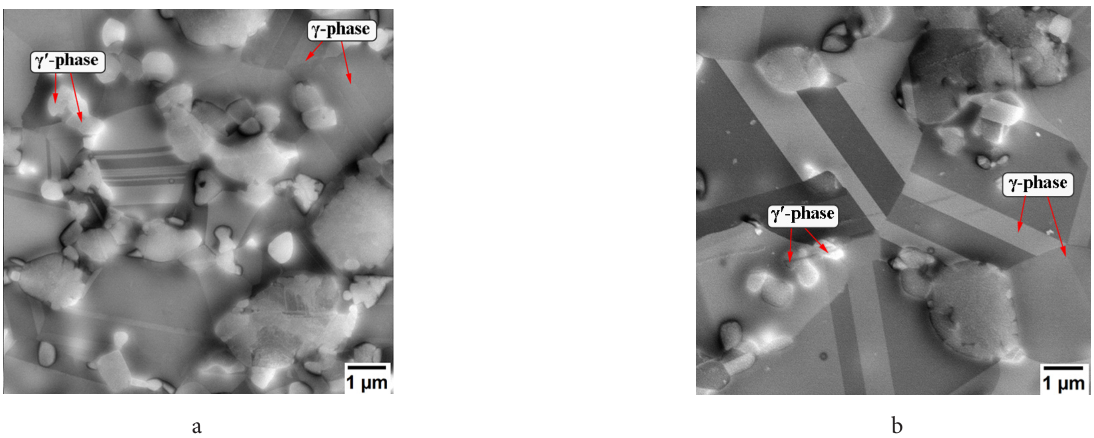
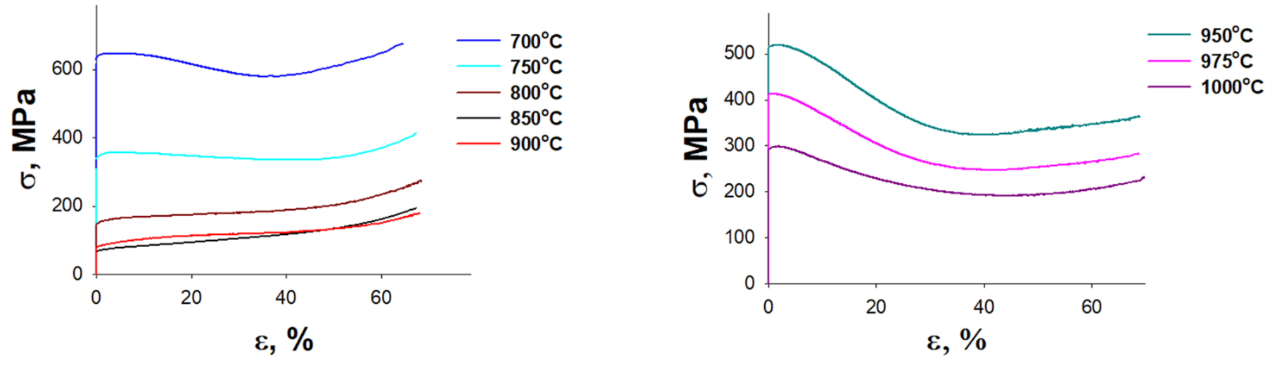
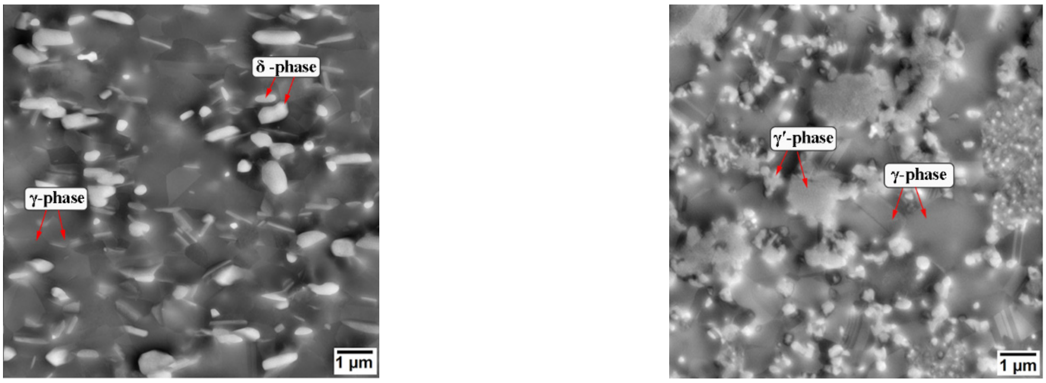


Fig. 4. Microstructure of the EP975 superalloy with an UFG structure of the mixed type after superplastic deformation according to the scheme of uniaxial tension at $\dot{\varepsilon} = 10^{-3}$ s^{-1} and $T = 1000$°C: grip zone (a); gauge section (b).



a b

Fig. 5. (Color online) Results of mechanical tests for compression of UFG superalloys at strain rate $\dot{\varepsilon}=10^{-3}$ s^{-1}: EK61 (a), EP975 (b).



a b

Fig. 6. Microstructure of the superalloy with UFG structure after superplastic deformation according to the scheme of uniaxial compression at (stagnant zone): EK61, $T=850$°C and $\dot{\varepsilon}=10^{-3}$ s^{-1} (a), EP975, $T=1000$°C and $\dot{\varepsilon}=10^{-3}$ s^{-1} (b).

slight growth of the grains of the matrix γ-phase occurs up to a size not exceeding 1 μm (Fig. 6a).

An analysis of the microstructure of EP975 superalloy after deformation at a temperature of 950°C showed that the structure is stable. An increase in temperature up to 1000°C leads to coarsening of the γ-phase grains up to 2 μm (Fig. 6b). This occurs due to exposure to high temperatures and partial dissolution of smaller (less than 1 μm) particles of the γ'-phase. The size of large precipitates of the γ'-phase was 3.2±0.8 μm. In fact, at the indicated temperature of deformation, the UFG structure began to transform into a fine-grained microstructure.

A comparative analysis of the obtained results with the data of other studies [1,6,16,21,22] showed that a similar picture is observed both in EK61 and EP975 superalloys with different types of hardening phases. Under conditions of low-temperature TMT, a mixed type ultrafine-grained structure is formed. However, the formation of such a mixed structure in the entire volume of a high-temperature nickel-based superalloy, regardless of the type of strengthening phase, is a sufficient condition for ensuring the phenomenon of low-temperature superplasticity.

4. Conclusion

1. The deformation-heat treatment of heat-resistant EK61 and EP975 nickel-based superalloys with different types of hardening phase leads to the formation of an ultrafine-grained structure of the mixed type.

2. The UFG structure of the EK61 superalloy consists of grains of the γ-phase and particles of the δ-phase both having size of 0.3 μm, along with which there are relatively large particles of the δ-phase up to 2 μm in size. The UFG structure of the EP975 superalloy consists of grains of the γ-phase and particles of the γ'-phase both having size of 0.5 μm, along with which there are relatively large particles of the γ'-phase up to 3.2 μm in size.

3. Both the EK61 and EP975 superalloys having the mixed type ultrafine-grained structure demonstrates the maximum characteristics of the superplasticity are achieved at a temperature of 850 and 1000°C and a strain rate of $\dot{\varepsilon}=10^{-3}$ s^{-1} respectively.

Supplementary material. *The online version of this paper contains supplementary material available free of charge at the journal's website (lettersonmaterials.com).*

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