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Low-temperature superplastic deformation of the EK79 nickel-based superalloy with the mixed ultrafine-grained microstructure

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Abstract: One of the most effective ways to increase the processing plasticity of advanced superalloys (heat-resistant nickel-based alloys) is the formation of an ultrafine-grained (UFG) microstructure in bulk semi-finished products. Such a microstructure is a necessary condition for the manifestation of the structural superplasticity effect in the technological processes of manufacturing products from such superalloys. One of the most promising methods for producing UFG microstructures is thermomechanical treatment (TMT) according to the multiple isothermal forging scheme. It has been shown that the EK79 superalloy after TMT, with a gradual decrease in the processing temperature from 0.88 to 0.62 Ts (where Ts is the strengthening phase dissolution temperature) leads to the transformation of the initial micro-duplex fine-grained microstructure into a mixed UFG microstructure. Such a mixed UFG microstructure consists of: 1) relatively coarse (inherited from the fine-grain microstructure) particles – γ'-phase with a size of 3.0±0.8 μm; 2) γ-grains, and incoherent γ'-phase particles with a size of 0.3–0.5 μm; 3) strengthening coherent intragranular γ'-phase particles with a size of 0.05–0.1 μm, released upon cooling from the TMT temperature to room temperature. During uniaxial compression tests, the EK79 superalloy with such microstructure, demonstrates low-temperature superplasticity in the temperature range of 800–1000 °C. It has been found that an increase in the deformation temperature up to 1000 °C, leads to the increase of γ-phase grains to micron size. The maintenance of superplastic properties in the presence of relatively coarse incoherent particles in the microstructure of the second phase (γ'-phase) is apparently related to the fact that the deformation is localised in the UFG component.

Keywords: heat-resistant nickel-based superalloy; EK79; strengthening phase; microduplex microstructure; ultrafine-grained microstructure; low-temperature superplasticity; thermomechanical treatment; uniaxial compression.

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INTRODUCTION

Nickel-based superalloys are used for production of gas turbines in the aerospace and energy generating industries, due to their good mechanical properties, such as high temperature strength, creep resistance and fatigue life capability, and corrosion resistance [1–3]. Such characteristics of mechanical properties are achieved through complex alloying of modern superalloys, having a complex chemical composition, including more than 10 alloying elements [1; 3; 4]. In these alloys, the required values of performance (service) characteristics are achieved both due to effective solid-solution strengthening, with refractory alloying elements, and due to the release of a plastic matrix γ-phase of coherent particles inside the grains, for example, a strengthening γ'-phase based on the $Ni_3(Al,Ti)$ intermetallide [1; 5; 6].

The desire of the developers of superalloys to achieve maximum heat-resistant characteristics by complicating, the chemical composition and increasing the volume fraction of the strengthening γ'-phase, led to a sharp decrease in their technological plasticity, and an increase in the complexity of their deformation processing [5–7]. For example, complex alloyed superalloys such as EK79 and EP975, the volume fraction of the strengthening γ'-phase of which reaches 40 and 55 %, respectively, have low technological plasticity. This is due to the fact that in these alloys, the strengthening γ'-phase is released from the supersaturated solid solution of the matrix (γ-phase) almost instantly, in the form of nanosized coherent particles of spherical or cuboid shape [5; 7]. The release of such particles occurs both during thermal and thermomechanical treatment. In the latter case, nanosized γ'-phase particles are released primarily in the near-surface layers of a hot blank, the outer surface of which is intensively cooled during its transfer from a high-temperature furnace to a press die and then during subsequent deformation. This leads to a sharp decrease in technological plasticity, and as a consequence, to the formation of a network of small cracks on the side surface of a deformable superalloy blank. During further hot stamping, this can lead to the growth of microcracks, up to the destruction of the deformed blank.

Superalloys such as Inconel 718, and its Russian analogue EK61 (KhN58MBYuD), in which strengthening is achieved through the release of nanosized γ"-phase (Ni_3Nb) particles, are more technologically advanced [8]. Expansion of technological capabilities when processing hard-to-deform superalloys (heat-resistant nickel alloys) is possible due to the superplasticity (SP) effect, and can be achieved as a result of the formation of an ultrafine-grained (UFG) or nanocrystalline (NC) microstructure in these materials [9–11]. The methodological approach patented in [9] consisting in carrying out thermomechanical treatment (TMT), with a step-by-step decrease in temperature, allows ensuring a step-by-step transformation of the initial coarse-grained microstructure into a fine-grained microduplex microstructure. With a subsequent decrease in the processing temperature, the microstructure is refined to the UFG state and further up to the nano-crystalline state. This methodological approach turned out to be very effective when processing other alloys, based on aluminium, magnesium, titanium, and even intermetallic compounds based on the latter [10]. It should also be noted that in the paper [10], the principles of the method of multi-axial isothermal forging (MIF) are formulated, which makes it possible to obtain homogeneous bulk nanostructured semi-finished products of metals and alloys, including heat-resistant and intermetallic ones.

One of the most promising methods for microstructure refinement in superalloy is thermomechanical treatment [11–13], during which the use of a scheme (MIF) is effective [10; 14]. Thus, in the previous work [15], using the example of the EP975 superalloy with the same type of strengthening phase, it was shown that TMT with a gradual decrease in the processing temperature, leads to the production of a mixed UFG microstructure, and during uniaxial tensile tests, an superalloy with such a microstructure demonstrates maximum superplasticity characteristics (δ=1320 %; m=0.5) achieved at a temperature of 1000 °C and a strain rate of $\acute{\varepsilon}=10^{-3}$ s^{-1}.

To evaluate the characteristics of superplastic properties, the method of isothermal deformation, according to the uniaxial tension scheme is traditionally used [16–18]. In the case of using superplastic deformation in traditional technological processes (stamping, forging), which are carried out mainly according to the scheme of uniaxial compression, when producing a complex-profile part, a complex stress-strain state will arise in it, characterised by the action of both tensile and compressive stresses [15; 19; 20]. In particular, during traditional stamping of a part, compressive stresses will predominantly act in its central zone, and tensile stresses will act on the periphery in the tangential direction [21; 22]. Therefore, when using the effect of superplasticity in practice in the technological process of manufacturing parts from a specific hard-to-deform superalloy, it is important both to determine the optimal modes for producing UFG microstructure semi-finished products from the selected material and to identify the features of microstructural changes during subsequent deformation according to the uniaxial compression scheme.

The purpose of this work is to study the influence of thermomechanical treatment on the formation of a mixed ultrafine-grained microstructure in the EK79 superalloy, as well as to evaluate the mechanical properties of such a microstructure, when tested according to a uniaxial compression scheme.

METHODS

The studies were carried out on the EK79 heat-resistant nickel superalloy. In this superalloy, strengthening is achieved due to the precipitation of intragranular coherent particles of the γ'-phase, based on the $Ni_3(Al,Ti)$ intermetallic compound. The chemical composition of the studied EK79 superalloy is presented in Table 1, and corresponds to GOST 5632-2014. For the EK79 superalloy, the well-known Utimet 520 superalloy is the closest in chemical composition.

The original material was a deformed blank with a diameter of 400 mm and a thickness of 40 mm, with a homogeneous fine-grained microduplex microstructure, from which samples measuring 40×50×70 mm^3 were cut. To obtain the UFG microstructure, TMT of the samples was carried out using the MIF scheme developed by the Institute for Metals Superplasticity Problems of the Russian Academy of Sciences [15]. TMT was carried out on a hydraulic press equipped with an isothermal stamping block, with a force of 6.3 MN, in the temperature range of (0.88–0.62) Tγ' (Tγ' is the γ'-phase dissolution temperature). The strain rate was $\acute{\varepsilon}=10^{-2}$–$10^{-3}$ s^{-1}.

The microstructure was studied using a TESCAN MIRA 3 LMH scanning electron microscope, and a JEM-2000EX transmission electron microscope. To carry out electron backscatter diffraction (EBSD) analysis at various structural levels, several EBSD maps were obtained, with a scanning step from 0.06 to 5 μm, depending on the structural state. Due to the peculiarity of the EBSD method, all low-angle grain boundaries with misorientation less than 2°

Table 1. *Chemical composition of the EK79 heat-resistant nickel-based superalloy*
Таблица 1. *Химический состав жаропрочного никелевого сплава ЭК79*

Superalloy	**Component content, wt. %**												
	C	**Cr**	**Co**	**V**	**W**	**Mo**	**Nb**	**Al**	**Ti**	**B**	**Si**	**Mn**	**La**
EK79	0.06	11	14	0.5	2.5	4.5	2.7	3	2.6	≤0.01	≤0.30	≤0.04	≤0.08

were excluded from consideration. Compression tests were carried out on a Schenck RMS-100 universal dynamometer. For mechanical uniaxial compression tests, cylindrical samples with a diameter of 10 mm, and a height of 15 mm were used.

RESULTS

Formation of a mixed UFG microstructure in the EK79 superalloy during TMT

The initial microstructure of the EK79 superalloy, with an average γ-phase grain size of 8–9 μm and large incoherent particles, – strengthening γ'-phase grains with a size of 3.0±0.8 μm is shown in Fig. 1. Using scanning and transmission electron microscopy, it was found that the superalloy contains dispersed (0.2–0.3 μm) coherent γ'-phase particles inside the γ-phase grains, which are usually released upon cooling from the stamping temperature to room temperature (Fig. 1 a, 1 b). According to EBSD analysis, it was found that in the initial fine-grained microstructure, all grains have different orientations in space, and the microstructure itself is homogeneous (Fig. 1 c).

Low-temperature TMT in the temperature range of (0.88–0.62) Tγ', using the MIF scheme, led to the formation of a mixed UFG microstructure from an UFG component, which is a mixture of incoherent γ'-phase particles, and γ-phase grains with a size of 0.3–0.5 μm. The volume fraction of the UFG component exceeds 80 %. Moreover, in the UFG microstructure, individual coarse particles – globular-shape γ'-phase grains with a size of 3.0±0.8 μm are relatively evenly distributed (Fig. 1 d, 1 f). These coarse particles are well identified on the results of EBSD analysis (Fig. 1 f). Probably, they were formed earlier at the high-temperature TMT stage, during which the initial fine-grained duplex microstructure was formed. The proportion of relatively large γ'-phase precipitates in the EK79 superalloy is 10 %. When cooling from the processing temperature, coherent nanosized γ'-phase particles with a size of 0.05–0.1 μm are revealed in the body of the γ-phase grains.

It should be noted that in the EK79 superalloy under study, the matrix γ-phase and the strengthening γ'-phase have the same type of crystal lattice – a face-centred cubic lattice, and the lattice mismatch parameters are very small (less than 1 %). Therefore, the applied EBSD analysis method, does not allow distinguishing between phases and perceives (represents them) on EBSD maps as one phase. Based on the results of the analysis of such maps (Fig. 1 c, 1 f), it is clear that all small γ-phase grains and incoherent γ'-phase particles-grains less than 1 μm in size are separated by high-angle grain boundaries (γ/γ), with a grain-boundary angle of more than 15°, and interphase (γ/γ') boundaries, and are also coloured in different colours indicating their different crystallographic orientations. Noteworthy is the fact that in large (“inherited” from the microduplex microstructure) γ'-phase particles, a colour gradient is revealed, i. e., the colour within one grain changes contrast, and in the place where the contrast change occurs, subboundaries exhibiting an increased proportion of low-angle boundaries (LABs) are revealed (Fig. 1 f). Apparently, during low-temperature TMT, local deformation of coarse γ'-phase particles occurs, along individual crystallographic planes, which leads to a change in the shape of coarse particles: the initial round shape takes on an irregular contour, in the form of individual protrusions. Probably, simultaneously with the recrystallisation process, which resulted in the UFG microstructure formation (EBSD maps show recrystallised small grains, many of which are free from dislocations, which is confirmed by transmission electron microscopy data), deformation of coarse incoherent γ'-phase particles-grains is observed. It has been found that the share of LABs in the EK79 superalloy, with a mixed UFG microstructure is 25 %.

In contrast to coarse γ'-phase particles, in the UFG component (0.3–0.5 μm in size), during deformation, the retention of the equiaxial shape of the γ-phase grains, and incoherent γ'-phase particles, as well as a lower fraction of LABs, is observed. This apparently indicates the partial development of superplastic deformation mechanisms at the final stage of low-temperature TMT, in particular the main mechanism – grain boundary sliding.

Superplastic deformation of EK79 superalloy with a UFG microstructure under uniaxial compression

The results of mechanical tests, for uniaxial compression, of the EK79 superalloy with a UFG microstructure are shown in Fig. 2. It can be observed that even at the initial stage of deformation (5–10 %) under conditions of relatively low temperatures, flow stress peaks are not detected. With increasing degree of deformation, a weak monotonic flow stress growth is observed. A more intense increase in the flow stress values at deformation degrees of more than 40 %, is apparently determined by an increase in contact friction on the end surfaces, between the deformed blank and the strikers.

Microstructural studies of samples after testing according to the uniaxial compression scheme

Microstructural studies of samples after testing under the uniaxial compression scheme are presented in Fig. 3.

Analysis of microstructural changes after deformation of the EK79 superalloy with a pre-prepared UFG microstructure indicates that, during low-temperature superplastic deformation, retention of the mixed UFG microstructure is observed. Deformed grains are free of dislocations, and twins are also detected in many γ-phase grains. At the same time, the equiaxial shape of the grains is retained, which indicates the development of the main mechanism of superplastic deformation – grain boundary sliding (Fig. 3).

Microstructural analysis showed that in the EK79 superalloy, as a result of deformation according to the uniaxial compression scheme at a temperature of 950 °C, the superalloy microstructure is stable. An increase in the deformation temperature to 1000 °C leads to the coarsening of the γ-phase grains, which is associated with the partial dissolution of smaller (less than 1 μm) γ'-phase particles, i. e., the microstructure is transformed into a fine-grained duplex microstructure. The EBSD maps (Fig. 3) show that the microstructure is characterised by a uniformly equiaxial grain shape. Maintaining equiaxiality is a sign that deformation occurs under superplastic conditions.

Various areas of the samples were studied: those in which intensive development of deformation occurs, and

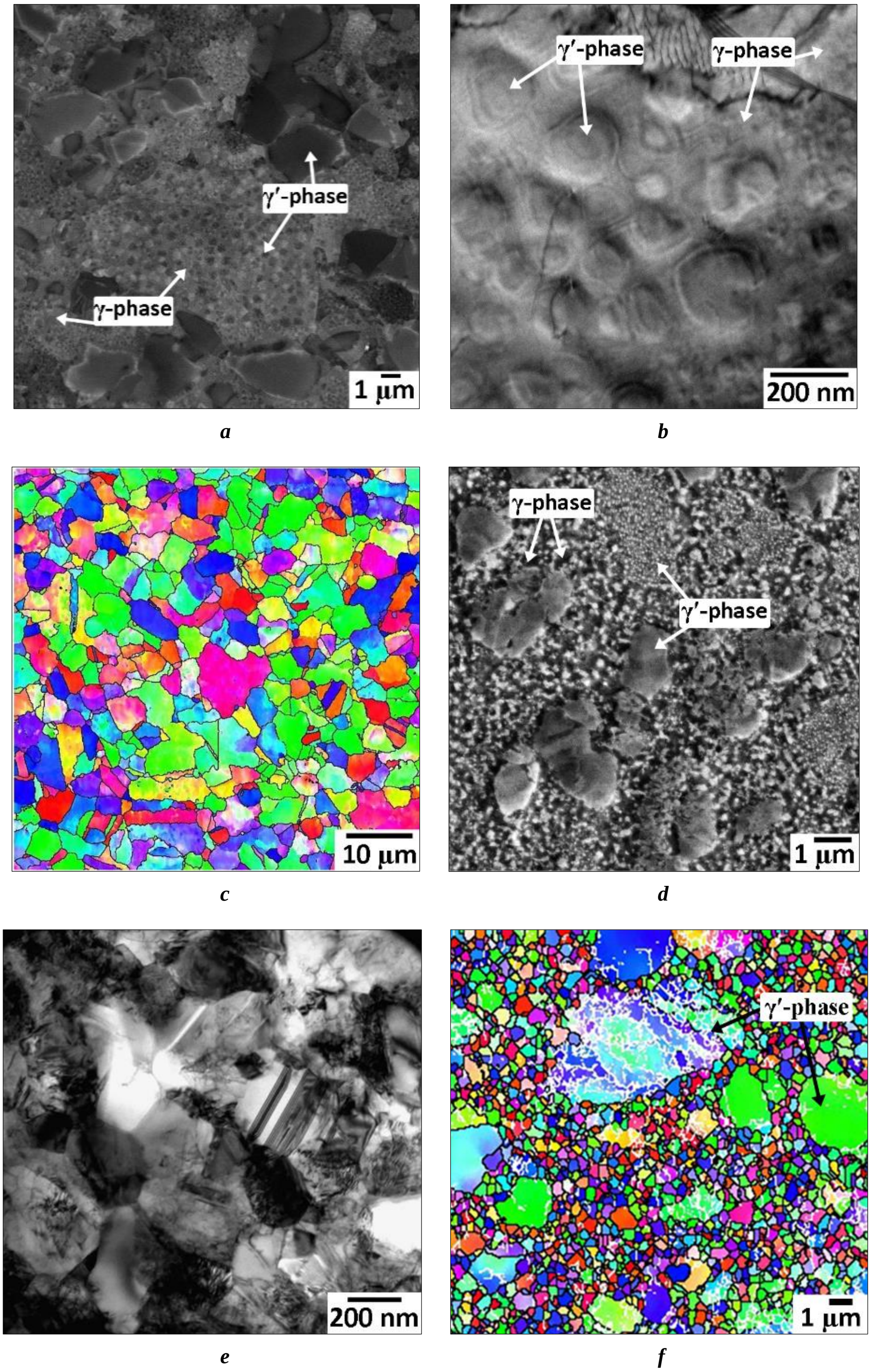


Fig. 1. *Microstructure of the EK79 superalloy:* ***a–c*** *– the initial state;* ***d–f*** *– after TMT*
Рис. 1. *Микроструктура сплава ЭК79:* ***a–c*** *– исходная;* ***d–f*** *– после ДТО*

those in which there is practically no deformation (Fig. 4). It has been found that in the zones of the samples involved in deformation (the centre of the sample), the increase in grain size is associated with both thermal influence, and stimulation by deformation, as a result of which the coarsening of grains occurs more intensively. In the stagnant zone during upset, where there is practically no deformation, grain growth is caused by the influence of high temperature, as a result of which partial γ'-phase dissolution occurs.

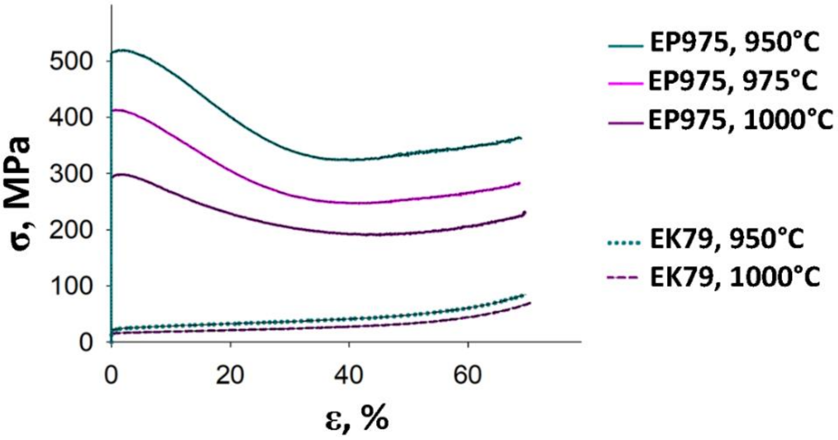
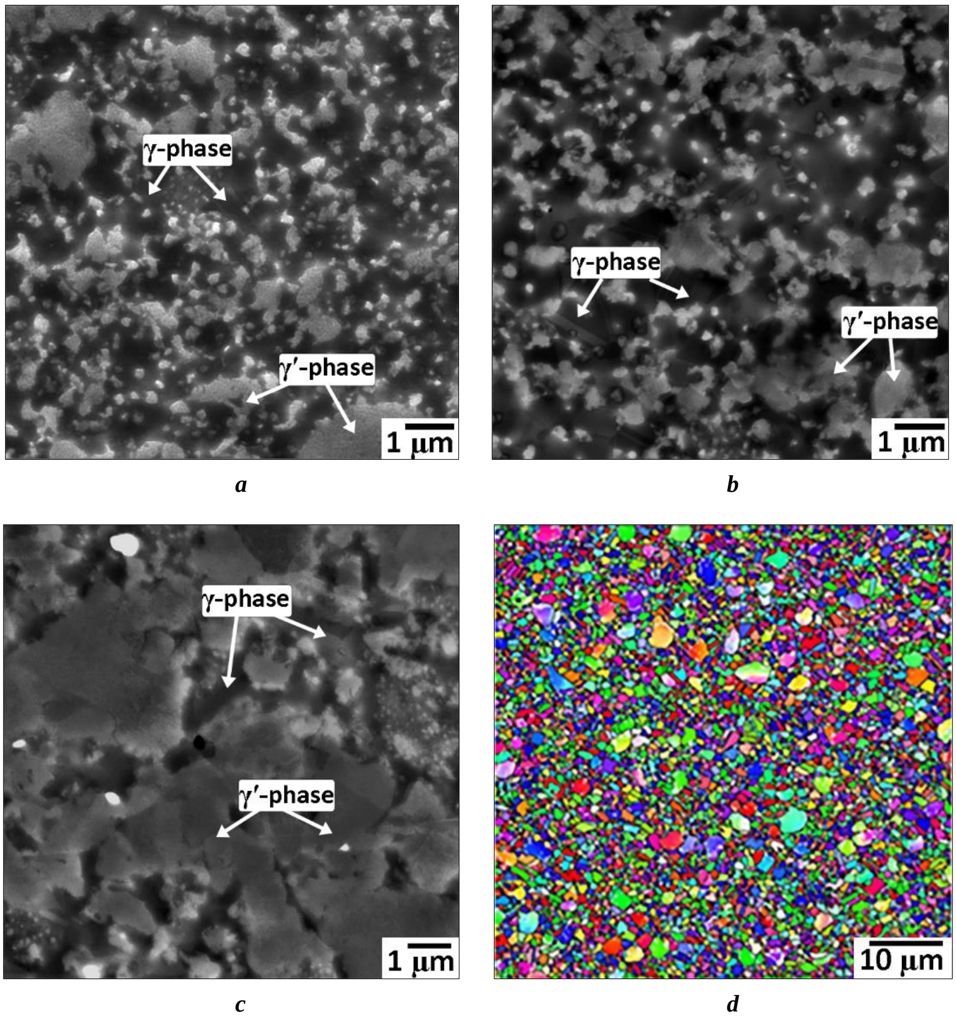


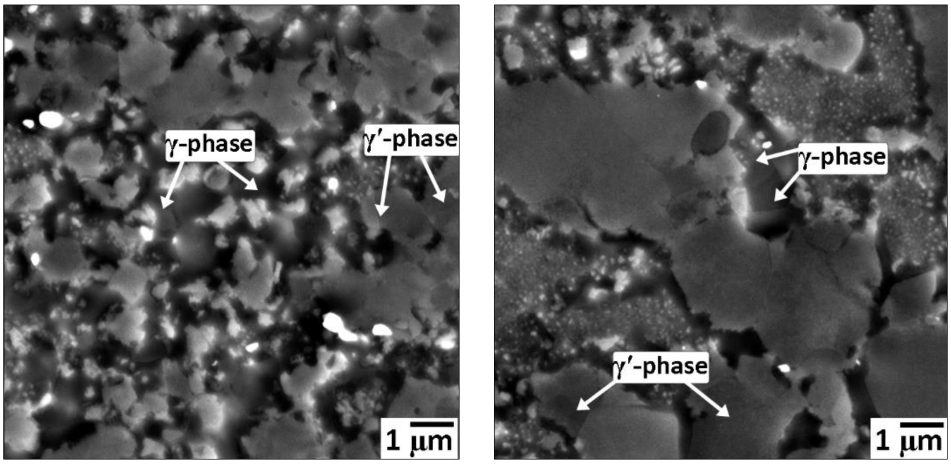
Fig. 2. *Mechanical properties of EP975 and EK79 nickel-based superalloys tested at a rate of* $\acute{\varepsilon}=10^{-3}$ s^{-1} *(EP975 [Repr. from: 15, p. 83])*
Рис. 2. *Механические свойства образцов из никелевых сплавов ЭП975 и ЭК79, испытанных при скорости* $\acute{\varepsilon}=10^{-3}$ c^{-1} *(ЭП975 [Привод. по: 15, с. 83])*



a ***b***

c ***d***

Fig. 3. *Microstructure of the EK79 superalloy with the mixed-type UFG microstructure after superplastic deformation according to the uniaxial compression scheme at* $\acute{\varepsilon}=10^{-3}$ s^{-1}*and temperatures of:* ***a*** *– 900 °C;* ***b*** *– 950 °C;* ***c, d*** *– 1000 °C*
Рис. 3. *Микроструктура суперсплава ЭК79 с УМЗ структурой смешанного типа после сверхпластического деформирования по схеме одноосного сжатия при* $\acute{\varepsilon}=10^{-3}$ c^{-1} *и температуре:* ***a*** *– 900 °C;* ***b*** *– 950 °C;* ***c, d*** *– 1000 °C*



a ***b***

Fig. 4. *Microstructure of the EK79 superalloy with the mixed-type UFG microstructure after superplastic deformation according to the uniaxial compression scheme at* $\dot{\varepsilon}=10^{-3}$ *s*$^{-1}$ *and T=1000 °C:* ***a*** *– central part of the specimen;* ***b*** *– stagnant zone of the specimen*
Рис. 4. *Микроструктура сплава ЭК79 с УМЗ структурой смешанного типа после сверхпластического деформирования по схеме одноосного сжатия при* $\dot{\varepsilon}=10^{-3}$ *с*$^{-1}$ *и T=1000 °C:* ***a*** *– центральная часть образца;* ***b*** *– застойная зона образца*

DISCUSSION

Analysis of the results obtained indicates that in the process of low-temperature TMT in forgings made of EK79 superalloy, a mixed UFG microstructure is formed, in which three types of the γ'-phase particle sizes, can be distinguished: 1) relatively coarse (size of 3.0±0.8 μm) – obviously “inherited” from the original fine-grained microduplex microstructure; 2) UFG component – γ-phase grains and incoherent γ'-phase particles with a size of 0.3–0.5 μm; 3) strengthening intragranular γ'-phase particles with a size of 0.05–0.1 μm, released upon cooling, from the TMT temperature to room temperature.

The formation of a mixed UFG microstructure in the EK79 superalloy, during low-temperature TMT, is apparently determined by the following circumstances. The processing temperature is quite low, and the deformation degree, and the time during which the UFG microstructure is formed, are insufficient to ensure the development of recrystallisation in the γ'-phase particles, that are larger and stronger than the matrix γ-phase and were inherited from the duplex microstructure. Therefore, in the process of TMT according to the selected modes, the recrystallisation development occurs mainly in small grains of the plastic γ-phase, with an initial microduplex microstructure, in which coagulated γ'-phase particles were additionally distinguished.

A comparative analysis of the mechanical properties (Fig. 2) of the EP975 superalloy given in the paper [15], and the EK79 superalloy studied in this work, showed that after deformation, according to the uniaxial compression scheme, there is a significant difference in the level of flow stresses, and in the dependence of the flow stress on the degree of deformation. In the EP975 superalloy, at the initial stage of deformation (2–5 %), a peak in flow stress and a subsequent decrease are observed. This type of dependence may indicate the development of dynamic recrystallisation processes during superplastic deformation, and as a consequence, may lead to the formation of a more fine-grained microstructure. At the same time, such a peak is not observed in the EK79 superalloy in the studied temperature-rate strain range. Under the same temperature rate strain conditions, the level of flow stress in the more doped and more heat-resistant EP975 superalloy is almost an order of magnitude higher.

According to the known ideas about the UFG microstructure formation in the superalloy presented in [7; 12], at each stage, microstructure refinement is achieved step by step: by transforming the initial coarse-grained microstructure into a fine-grained duplex one at the high-temperature TMT stage, and then into an UFG microstructure at the subsequent low-temperature TMT stage. In this case, the initial fine-grained microduplex microstructure in the superalloy, under study, should have been transformed into a completely homogeneous UFG microstructure. However, in this work, as noted above, a mixed UFG microstructure was formed in the EK79 superalloy. It is obvious that during the TMT process, recrystallisation occurred predominantly in the more plastic γ-phase.

Compared to the EP975 superalloy [15], blanks made from the more plastic and less heat-resistant EK79 superalloy, were subjected to more intense TMT (the temperature range was wider than in the EP975 superalloy, and the temperature of TMT end was 150 °C lower). This fact probably determines the formation of a more fine-grained microstructure in the EK79 superalloy, since more intense TMT at lower temperatures leads to the formation of new recrystallisation centres in the form of fragments and subgrains with LABs.

A significant volume fraction of the UFG component (≥80 %), obviously plays a decisive role in the implementation of the effect of low-temperature superplasticity in the EK79 superalloy with a mixed UFG microstructure. As is known [11; 16], alloys with a UFG microstructure are characterised by a large proportion of grain boundaries, which leads to activation of the main mechanism of superplastic deformation – grain boundary sliding. Moreover, an increase in the extension of grain boundaries promotes the activation of another mechanism of superplastic deformation – diffusion creep. Therefore, the presence in the UFG microstructure of a small amount of relatively coarse γ'-phase particles-grains, apparently, does not have a significant influence on the manifestation of the effect of low-temperature superplasticity in the material under study.

CONCLUSIONS

1. Low-temperature thermomechanical treatment of the EK79 heat-resistant nickel superalloy leads, to the transformation of a fine-grained duplex microstructure into an ultrafine-grained mixed microstructure.

2. The ultrafine-grained mixed microstructure in the EK79 superalloy consists of γ-phase grains and γ'-phase incoherent particles of 0.3–0.5 μm in size, along with which there are relatively coarse γ'-phase particles of up to 3.8 μm in size. In this case, the share of coarse particles is about 10 %, and the share of the UFG component exceeds 80 %, which plays a decisive role in the implementation of the low-temperature superplasticity effect.

3. The EK79 superalloy with a mixed UFG microstructure has thermal stability at deformation temperatures not higher than 950 °C, which provides the necessary conditions for the implementation of the low-temperature superplasticity effect in the superalloy under study even at temperatures of 800–850 °C corresponding to the ageing temperature range. The analysis of microstructural changes in the samples deformed under uniaxial compression showed that the equiaxial shape of γ-phase grains, and incoherent γ'-phase particles less than 1 μm in size is retained. The latter indicates the development of the main mechanism of superplastic deformation – grain boundary sliding.

4. An increase in the deformation temperature to 1000 °C leads to the UFG microstructure transformation into a fine-grained duplex microstructure.

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Низкотемпературная сверхпластическая деформация никелевого сплава ЭК79 с ультрамелкозернистой структурой смешанного типа

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Аннотация: Одним из наиболее эффективных способов повышения технологической пластичности современных суперсплавов – жаропрочных никелевых сплавов – является формирование в объемных полуфабрикатах ультрамелкозернистой (УМЗ) структуры, которая является необходимым условием для реализации эффекта структурной сверхпластичности в технологических процессах изготовления изделий из таких сплавов. Одним из наиболее перспективных методов получения УМЗ структуры является деформационно-термическая обработка (ДТО) по схеме всесторонней изотермической ковки. Показано, что ДТО сплава ЭК79 с постепенным снижением температуры обработки с 0,88 до 0,62 Ts (где Ts – температура растворения упрочняющей фазы) приводит к трансформации исходной мелкозернистой структуры типа микродуплекс в УМЗ структуру смешанного типа. Такая смешанная УМЗ микроструктура состоит из: 1) относительно крупных (наследственных от мелкозернистой структуры) частиц γ'-фазы размером 3,0±0,8 мкм; 2) зерна γ-фазы и некогерентных частиц γ'-фазы размером 0,3–0,5 мкм; 3) упрочняющих когерентных внутризеренных частиц γ'-фазы размером 0,05–0,1 мкм, выделяющихся при охлаждении с температуры ДТО до комнатной температуры. Сплав ЭК79, имеющий такую микроструктуру, при испытаниях на одноосное сжатие демонстрирует низкотемпературную сверхпластичность в диапазоне температур 800–1000 °C. Установлено, что повышение температуры деформации до 1000 °C приводит к укрупнению зерен γ-фазы до микронного размера. Сохранение сверхпластических свойств при наличии в структуре сравнительно крупных некогерентных частиц второй фазы (γ'-фазы), по-видимому, связано с тем, что деформация локализована в УМЗ компоненте.

Ключевые слова: жаропрочный никелевый сплав; ЭК79; упрочняющая фаза; микродуплексная структура; ультрамелкозернистая структура; низкотемпературная сверхпластичность; деформационно-термическая обработка; одноосное сжатие.

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