Reviews
Advanced structural materials, materials for extreme conditions

УДК 669.245 DOI: 10.17277/jamt.2023.01.pp.070-082

The influence of ultrafine-grained structure on solid-state weldability and formability of precipitation-hardening nickel-based superalloys

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Abstract: The formation of an ultrafine-grained (UFG) structure of a mixed type in the EK61 superalloy provides the manifestation of the low-temperature superplasticity effect. It can be successfully implemented in an innovative process of pressure welding (PW) to obtain solid-state joints (SSJ) in a combination of EK61 and EP975 superalloys characterized by different types of hardening phases. According to the energy-dispersive analysis resultsin the process of PW in vacuum under low-temperature and rate superplasticity conditions (T = 850 °C, $\dot{\varepsilon} = 10^{-4}$ s^{-1}) the interdiiffusion of alloying elements occurs between the joined EK61 and EP975 superalloys. As a result of the interdiiffusion, a narrow transition zone of diffusion interaction is formed, the width of which is 3 μm. The effect of heat treatment (HT) on the change in the phase composition in the SSJ and its strength in welded specimens in a combination of EK61//EP975 superalloys has been studied. It is shown that HT leads to expansion of the diffusion interaction zone by 4.6 times (3 to 14 μm). According to the results of mechanical tensile tests at room temperature, it was found that the strength of welded samples after PW reaches a value of 0.8 of the strength of the EK61 superalloy being joined, and after the following HT it remains at the same level. The influence of the strain on the microstructure and microhardness changes of sheet blanks from the EK61 superalloy with a UFG structure subjected to superplastic forming (SPF) into a cylindrical matrix under low-temperature superplasticity conditions has been studied.

Keywords: nickel-based superalloys; ultrafine-grained structure; superplasticity; pressure welding; solid-state joint.

For citation: Galieva EV, Klassman EYu, Valitov VA, Stepukhov EM, Gabbasov RR, Safiullin RV, Lutfullin RYa. The influence of ultrafine-grained structure on solid-state weldability and formability of precipitation-hardening nickel-based superalloys. *Journal of Advanced Materials and Technologies*. 2023;8(1):070-082. DOI:10.17277/jamt.2023.01.pp.070-082

Влияние ультрамелкозернистой структуры на свариваемость в твердофазном состоянии и формуемость дисперсионно-твердеющих суперсплавов на основе никеля

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Аннотация: Показано, что формирование в суперсплаве ЭК61 ультрамелкозернистой (УМЗ) структуры смешанного типа обеспечивает проявление эффекта низкотемпературной сверхпластичности, который может быть успешно реализован в инновационном технологическом процессе сварки давлением для получения твердофазных соединений (ТФС) в сочетании суперсплавов ЭК61 и ЭП975, характеризующихся различным типом упрочняющей фазы. По результатам энергодисперсионного анализа установлено, что в процессе сварки давлением в вакууме в температурно-скоростных условиях низкотемпературной сверхпластичности (T = 850 °C, $\dot{\varepsilon} = 10^{-4}$ с$^{-1}$) между соединяемыми сплавами ЭК61 и ЭП975 в результате протекания процессов взаимной диффузии легирующих элементов формируется узкая переходная зона диффузионного взаимодействия, ширина которой составляет 3 мкм. Изучено влияние термической обработки на изменение фазового состава в зоне ТФС и его прочность в сварных образцах в сочетании суперсплавов ЭК61//ЭП975. Показано, что термическая обработка приводит к расширению зоны диффузионного взаимодействия в 4,6 раза (от 3 до 14 мкм). По результатам механических испытаний на растяжение при комнатной температуре установлено, что прочность сварных образцов после сварки давлением достигает величины 0,8 от прочности соединяемого сплава ЭК61, а после термической обработки сохраняется на том же уровне. Исследовано влияние степени деформации на изменение микроструктуры и микротвердости листовых заготовок из сплава ЭК61 с УМЗ структурой, подвергнутых формовке в цилиндрическую матрицу в условиях низкотемпературной сверхпластичности Установлено, что после сверхпластической формовки (СПФ) в модельных отформованных образцах сохраняется однородная равноосная УМЗ структура дуплексного типа, параметры которой и значения микротвердости сохраняются на одном уровне после различных степеней деформации при формовке.

Ключевые слова: никелевые сплавы; ультрамелкозернистая структура; сверхпластичность; сварка давлением; твердофазное соединение.

Для цитирования: Galieva EV, Klassman EYu, Valitov VA, Stepukhov EM, Gabbasov RR, Safiullin RV, Lutfullin RYa. The influence of ultrafine-grained structure on solid-state weldability and formability of precipitation-hardening nickel-based superalloys. *Journal of Advanced Materials and Technologies*. 2023;8(1):070-082. DOI:10.17277/jamt.2023.01.pp.070-082

1. Introduction

Superalloys (heat-resistant nickel alloys) are widely used for the manufacture of various parts of aircraft gas turbine engines: compressor and turbine disks, rotor and nozzle blades, and other parts [1–4]. At present, for modern and promising aircraft engine building, the problems of increasing the energy efficiency of gas turbine engines and the efficiency of their production are very relevant. The first problem of increasing the energy efficiency of a gas turbine engine can be solved not only by creating new compositions of high-temperature materials with improved characteristics of heat-resistant properties [1–4], but also by developing and implementing new technical solutions related to the use in the design of a gas turbine engine bimetallic parts, such as “blisk” and “disk-shaft” [4–8]. Another problem of increasing the efficiency of production of parts, including bimetallic ones, for gas turbine engines can be solved through the development and implementation of new resource-saving technologies based on the use of the superplasticity effect [8–13].

As is known [1–4], the achievement of the required level of heat resistance in complexly alloyed nickel-based superalloys is achieved by separating coherent particles of strengthening phases, such as Ni_3Nb or $Ni_3(Al, Ti)$. It should be noted that the presence in the coarse-grained matrix (γ-phase) of superalloys of a significant amount (the volume fraction of γ′-phase is 55–60 %) of dispersed coherent particles of the strengthening $Ni_3(Al, Ti)$ phase leads to a sharp decrease in their technological plasticity. Therefore, for the manufacture of parts from superalloys, it is advisable to use the promising technology of superplastic deformation [9, 10]. Its implementation can be achieved by obtaining in semi-finished products from superalloys a fine-grained (1–10 μm) or ultrafine-grained (UFG) structure of a duplex type (with a grain and phase size of less than 1 μm), which is formed in the process of preliminary thermomechanical treatment (TMT) of such materials [9–11, 14–22].

It follows from the analysis of the scientific literature that various methods are used to obtain solid-state joints (SSJ) from metals and alloys, as well as nickel-based superalloys. In [23], using the example of Inconel 718 alloy, the efficiency of using inertial friction welding in welding with Inconel 718, Incoloy909, U720LI, Rene88DT alloys was shown. However, according to the results of the research, the authors showed that in the SSJ zones there are areas where the dissolution of the δ-phase and the γ′′-phase particles occurred. The width of such a zone reaches 500 μm, which leads to a decrease in hardness. When assessing the weldability of the EP742 alloy by the method of linear friction welding, the authors of [24] showed that macrodefects in the form of lack of penetration and discontinuities in the zone of the welded joint are not observed. At the same time, there are many pores along grain boundaries and in triple junctions, as well as chains of coarse carbide precipitates, leading to a decrease in the strength of the welded joint and contributes to the initiation and development of cracks during welding.

The studies [25–27] were devoted to the effect of rotational friction welding and heat treatment on the properties of wrought nickel-based superalloys. Thus, it was shown in [25] that a high level of properties of welded joints of the VZh172 alloy with the EK79 alloy (0.80–0.96 of the strength of a less strong alloy) is achieved during welding both with subsequent complete heat treatment and in the heat-strengthened state. The work [27] is devoted to the study of weldability of homogeneous and dissimilar combinations of semi-finished products of high-temperature nickel alloy VZh159 by rotational friction welding.

The data presented in [28] are of interest. This paper provides an overview of the weldability of a wide range of nickel-based superalloys and aluminides. In another work [29], the features of the formation of diffusion bonding of heat-resistant alloys with each other and with structural steels, the issues of manufacturing technology for rotors of small-sized gas turbine engines and reinforcement of shroud shelves of aircraft engine blades are considered.

Of particular interest is the patented diffusion welding technology [30], which can be used to fabricate bimetallic structures consisting of a combination of stainless steel + titanium alloy or titanium alloy + nickel alloy materials in almost many industries. A feature of this method is that the welded bimetallic structures are made of materials that form intermetallic phases between themselves. An intermediate layer in the form of a porous tape of ultrafine metal powder (UFMP) was placed between the materials to be welded. Diffusion welding was carried out at a temperature of 0.85–0.9 of the temperature of formation of intermetallic phases of the metal of the intermediate layer with one of the welded materials and between the welded materials. The intermediate layer of UFMP minimizes the formation of intermetallic compounds, which avoids embrittlement of the diffusion joint while maintaining its tensile strength. In another method of diffusion welding [31], for the manufacture of parts from powder heat-resistant nickel alloys, after assembling the elements for welding, they are evacuated and heated to a temperature not exceeding the solvus temperature by more than 10 °C. A welding force of 1.5–2.5 kg·mm^{-2} was applied with a holding time of 1.5–2 hours. Then the welding force was removed and the holding time was carried out for 2 hours.

The disadvantages of many well-known widely used methods, such as diffusion welding [6, 7, 28–33], are the duration of the process and high homologous temperatures, which in many cases are close to pre-melting temperatures, at which the process of obtaining SSJ is carried out. The methods of inertial, linear and rotational friction welding [23–27] are characterized by intense deformation heating up to the melting of the materials being joined, which has a negative effect on the structure of the SSJ zone and its strength.

One of the most promising methods for producing bimetallic joints from hard-to-deform superalloys is pressure welding (PW) using superplastic deformation, which makes it possible to obtain SSJ from various alloys [8–10, 12–13, 34–43]. At the same time, a sufficient condition for obtaining a high-quality joint is the manifestation of the superplasticity effect in at least one of the materials being joined [8, 12, 34, 36–43]. The use of welded structures not only saves metal, but also reduces the weight of the resulting parts. However, there is a problem of joining dissimilar alloys, since brittle inclusions may appear in the weld zone [12, 13]. In this regard, much attention is paid to research aimed at obtaining high-quality permanent joints from superalloys, as well as the development of innovative methods for manufacturing bimetallic parts with the required set of operational properties.

In [41], a method was developed for obtaining SSJ intermetallic alloys of the VKNA type based on the Ni_3Al intermetallic compound with a single-crystal structure with wrought alloys of the EP975 type with an initial fine-grained structure of the microduplex type, under conditions of high-temperature (T = 1125–1175 °C) superplasticity. The co-authors of this patent in [8, 36–41] showed that SSJ between EP975//VKNA-25 alloys can be obtained in a wide temperature-speed range of superplasticity (1075–1175 °C). It has been established that the strength of welded samples obtained by PW at temperatures of 1125 and 1175 °C (Vacuum $P = 5\cdot10^{-2}$ Pa) corresponds to the strength of the VKNA-25 intermetallic alloy. All samples tested at room temperature failed along the VKNA-25 alloy being joined.

In a recently published study [43], it was shown that the pressure welding method is an effective method for obtaining SSJ superalloys of the EK61 and EP975 types with various types of hardening phase. It was found that in the process of pressure welding at a temperature of 925 °C, the UFG structure is transformed into a fine-grained structure with a grain size of 1.5 μm. This temperature corresponds to the upper temperature limit of manifestation of superplasticity of the EK61 alloy with UFG structure. It is of interest to assess the possibility of obtaining SSJ at a much lower temperature of 850 °C, at which the EK61 alloy with a mixed-type UFG structure demonstrates the maximum characteristics of low-temperature superplasticity [44, 45]. However, such data are not available in the scientific literature.

The study of formability is an important element for determining the process characteristics of sheet materials with an ultrafine grain structure. As is known [46–62], in order to assess the formability of superplastic materials, most researchers conduct experiments using the methods of molding into special cylindrical or conical matrices. In the case of forming into a cylindrical matrix, either spherical or cylindrical specimens can be obtained. Moreover, the formed material is deformed in a wide range of strain rates. The main task of such experiments is to establish the optimal modes of superplastic forming (SPF), which can later be used to develop a technology for obtaining real thin-walled parts.

The use of low-temperature superplasticity is also important for the fabrication of parts of complex geometry from hard-to-deform superalloys by the SPF method. Thus, in [34], using the example of the Inconel 718 alloy, it was shown that the use of sheet semi-finished products with an UFG structure (~ 0.3 μm) makes it possible to reduce the flow stress level by a factor of 1.5 compared to the fine-grained state (~ 6.0 μm). Due to the implementation of the effect of low-temperature joint venture, it is possible to reduce the temperature of the SPF process of sheet blanks made of Inconel 718 alloy to 900 °C.

The aim of this study is to generalize the new results and those previously obtained to evaluate the effectiveness of the effect of deformation under conditions of low-temperature superplasticity of the EK61 superalloy on its formability, as well as on the formation of SSJ with the EP975 superalloy with a different type of hardening phase.

2. Materials and Methods

2.1. Characteristics of the developed alloys

The wrought heterophase nickel-based superalloys EK61(KhN58MBYuD) and EP975 (KhN59KVYuMBT), which differ in chemical and phase composition, and the type of hardening phase, were chosen as materials for the study. Table 1 shows the chemical composition of the studied superalloys, which fully complies with TU 14-1-50-45-91 (for the EK61 alloy) and Russian Standard 5632-2014 (for the EP975 alloy). The EK61 alloy is characterized by a high content of niobium (5%) and a low content of aluminum and titanium. The EP975 alloy, on the contrary, has a high total content of aluminum and titanium (7.2 %) and a reduced amount of niobium. These differences in the content of such alloying elements determine significant differences in the phase composition, type of the hardening phase, its morphology, and precipitation kinetics in the investigated EK61 and EP975 alloys.

In the EK61 alloy, as well as in its foreign analogue, the Inconel 718 alloy [2], hardening is achieved due to the precipitation inside the grains of the γ-phase (a solid nickel-based solution with an FCC lattice) of the strengthening γ′′-phase based on the Ni_3Nb intermetallic compound. The strengthening γ′′-phase is not isomorphic to the matrix (γ-phase, FCC), since it has a different type of crystal lattice – a body-centered tetragonal (bct.) lattice. It is released inside the grains of the γ-phase in the form of nanosized coherent disk-shaped particles with an ordered structure of the DO22 type. According to the data of [2], the γ′′-phase is metastable because, during long-term aging at an elevated temperature (more than 650 °C), it transforms into a δ-phase (Ni_3Nb) of a lamellar form with an orthorhombic lattice. During deformation-heat treatment of the plates, the δ-phases undergo fragmentation with subsequent spheroidization of the fragments [21]. In the initial state, the EK61 alloy was billets 80 mm in diameter and 90 mm high, cut from a hot-deformed rod 80 mm in diameter with an initial coarse-grained structure: the average grain size of the γ-phase is ~ 62 μm, inside which coherent nanosized (~ 40 nm) particles are homogeneously isolated metastable strengthening γ′′-phase based on Ni_3Nb intermetallic compound.

In another studied wrought alloy EP975, heat-resistant characteristics were observed after the final heat treatment, as a result of which there was a homogeneous precipitation inside large (50–150 μm)

Table 1. Chemical composition of EK61 and EP975 superalloys (heat-resistant nickel alloys) (wt. %)

Alloy	Al	Cr	Co/Fe	W/V	Mo	Ti	Nb	Cu	C	La
EK61	1.0	16.6	15.0 Fe	0.5 V	3.9	0.8	5.0	0.5	≤0.05	–
EP975	4.8	8.2	15.1 Co	10.2W	1.2	2.4	1.5	–	0.06	≤0.01

grains of the matrix (γ-phase, nickel-based solid solution) coherent (0.2–0.4 μm) particles of the strengthening γ′-phase based on $Ni_3(Al, Ti)$ intermetallic compound [1, 4]. In the EP975 alloy, both phases (grains of the γ-phase and dispersed particles of the γ′-phase) had the same type of face-centered cube crystal lattice. Billets from the EP975 alloy 40×50×70 mm^3 in sizewere used as the starting material; they were cut from forgings with a diameter of 400 mm and a thickness of 40 mm, in which a uniform fine-grained structure of the microduplex type was formed during high-temperature TMT: the average grain size of the γ-phase was 6-8 μm, along the boundaries and in the triple junctions of which large (2–3 μm) incoherent particles-grains of the γ′-phase were observed. In the microduplex structure, inside the grains of the γ-phase, there were dispersed (0.1–0.2 μm) coherent particles of the strengthening γ′-phase, which usually precipitate upon cooling from the forging temperature to room temperature. The volume fraction of the recrystallized microduplex structure exceeded 80 %.

2.2. Thermo-mechanical treatment of mixed-type UFG structure in EK61 alloy

To obtain a UFG structure of a mixed type in the EK61 alloy, TMT was carried out in the temperature range (0.93–0.65) T_δ (where T_δ is the dissolution temperature of the δ-phase) for the EK61 alloy) using the scheme of multiple isothermal forging (MIF), developed at IMSP RAS [9]. TMT of blanks was carried out with a gradual decrease in temperature in the above range on a hydraulic press with a force of 6.3 MN, equipped with an isothermal stamp block. At each temperature, at least 5-fold pressing was carried out with successive rotation of the deformation axis by 90°. The strain rate was $\dot{\varepsilon} \approx 10^{-2} - 10^{-3}$ s^{-1}.

Experiments on pressure welding under conditions of superplasticity of one of the materials being joined (EK61) were carried out on cylindrical specimens with dimensions: diameter d_0 = 15.7 mm for both alloys and height h_0 = 20 mm for the EK61 alloy and 15 mm for the EP975 alloy. Pressure welding was carried out on a Shenck Trebel type RMS100 testing machine equipped with a UVSD-1 high-temperature pressure welding unit developed at IMSP RAS (Fig. 1).

This setup includes a high-temperature furnace (with a working temperature of up to 1250 °C), strikers made of an intermetallic alloy of the VKNA-1B type, a vacuum pumping system including a fore-vacuum and diffusion pumps to ensure a vacuum in the container with samples at a level of $5 \cdot 10^{-2}$ Pa. This unit was developed earlier in the course of research led by Dr. V.A. Valitov on topics of RFBR projects No. 13-08-12200\15 (2014-2016) and RSF projects No. 18-19-00685 (2018-2020).

Pressure welding of cylindrical specimens in a combination of EK61//EP975 alloys was carried out under temperature and rate conditions of superplasticity of the EK61 alloy at a temperature of 850 °C and an initial strain rate of 10^{-4} s^{-1}. Samples of EK61//EP975 pairs were placed in a sealed container made of stainless steel, in which, during the entire process, the LED was passed through a gas outlet tube connected to a vacuum system. The strength of SSI EK61//EP975 at elevated (650 °C) and room temperatures was evaluated in comparison with the less high-temperature EK61 superalloy.

(*a*) (*b*) (*c*)

Fig. 1. The working zone of the furnace of the UVSD-1 installation for high-temperature pressure welding based on the RMS-100 testing machine from SCHENK (*a*, *b*) and samples before and after pressure welding in sealed containers (*c*)

2.3. Mechanical tests

Mechanical tests were carried out on an Instron 5982 testing machine. Superplastic formability was studied according to the original technique [30] using special equipment for SPF of sheet blanks into a cylindrical matrix with a diameter of 30 mm [60–62], the diagram of which is shown in Fig. 2.

To study the formability, the investigated sheet blank was hermetically welded with an auxiliary sheet along the contour. A fitting was welded to the auxiliary sheet, to which a pressure supply device is attached. The pressure of the working gas can vary in the range from 0 to 6 MPa. The welded package with the investigated sheet blank was installed in the tooling. The equipment was placed in an electrically heated oven and heated. The SPF of polished sheets 40×40 mm in size and 0.7 mm thick with an UFG structure made of the EK61 alloy was carried out according to the modes developed earlier in [45].

The SPF was carried out in the temperature-speed mode of low-temperature superplastic deformation of the EK61 alloy with a UFG structure of a mixed type: at a temperature of 850 °C with a deformation rate of $3 \cdot 10^{-3}$ s^{-1}. During heating, a vacuum (1.3–0.13 Pa) was created in the inner cavity of the blank to prevent oxidation of the surface under study. After reaching the required temperature, the working gas is fed into the internal cavity of the blank according to a special law – $P = f(\tau)$, where τ is the forming time [60, 61]. After the completion of the SPF process, the tooling was disassembled and formed cylinders were cut out. On the obtained samples, the deformation relief was examined, the thickness distribution was examined, and the equivalent tensile strain was found. When using interchangeable dies with a depth of 5, 10 and 15 mm, the equivalent deformation was from 10 to 800 %.

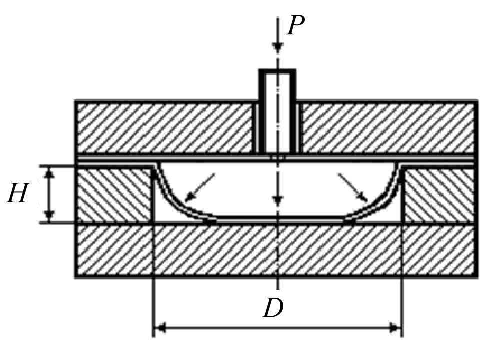


Fig. 2. Tooling scheme for SPF sheet blanks in cylindrical matrix: *P* – forming gas pressure; *H* – forming depth can be 5, 10, 15 mm; *D* – diameter of the cylindrical hole in the matrix (30 mm)

2.4. Analytical methods

Microstructural studies were carried out on a Mira 3LMH scanning electron microscope (TESCAN, Czech Republic). The fine structure was studied using a JEM-2000EX transmission electron microscope at an accelerating voltage of 160 kV. Energy dispersive analysis (EDA) was performed on a Vega 3SBH scanning electron microscope (TESCAN, Czech Republic). Microhardness measurements were carried out on an MHT-10 Microhardness Tester.

3. Results and Discussion

3.1. Certification of the microstructure of the investigated alloys

The microstructure of the EK61 alloy after TMT using the MAIF scheme is shown in Fig. 3. In the EK61 alloy (Fig. 3*a*, *b*), a mixed-type UFG structure is formed in the entire volume of the deformed material, in which the UFG component is close to the nanoduplex type in morphology and size.

The grain size of the γ-phase and the incoherent particles of the δ-phase was ~ 0.3–0.8 μm, the volume fraction of the δ-phase was $V_\delta \sim 24$ %. In this case, along with the UFG component, individual relatively large particles of the δ-phase (shown by red arrows)

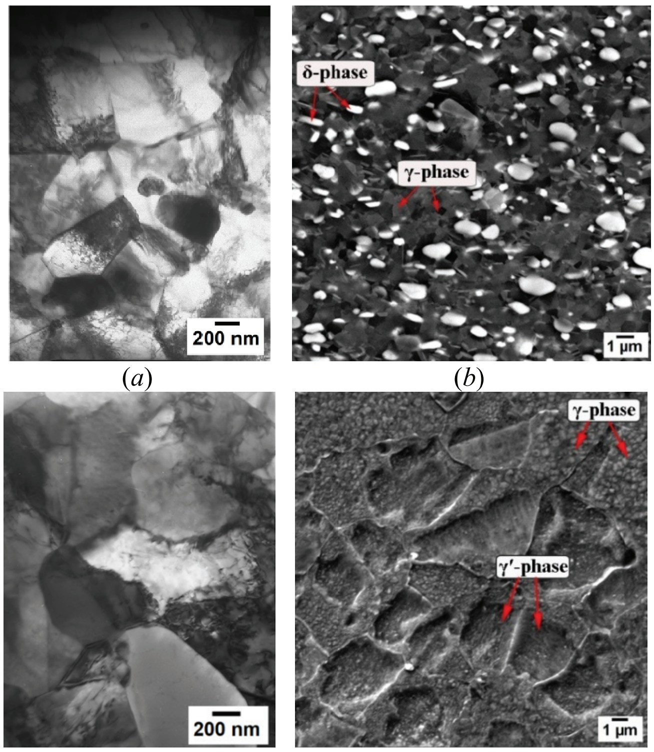


Fig. 3. The initial microstructure of the superalloys under investigation: *a*, *b* – EK61; *c*, *d* – EP975

up to ~ 2 μm in size are observed in the alloy structure, the fraction of which is $V_\delta \sim 5$ %. Relatively large particles are preserved and are "hereditary", i.e. previously formed at the stage of formation of the microduplex structure.

3.2. Pressure welding of nickel alloys in combination of alloys EK61//EP975 under conditions of low-temperature superplasticity of the EK61 alloy

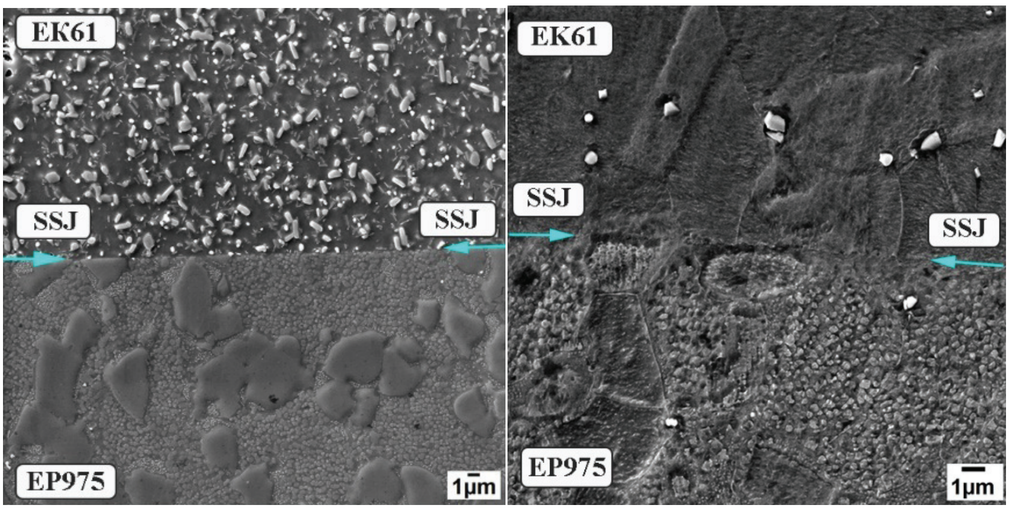
Figure 4 shows the microstructures of the alloys to be joined and the SSJ zone after PW (*a*) and subsequent heat treatment (*b*). In general, the entire deformation was localized in the less strong EK61 alloy, in which an UFG structure was formed before pressure welding, which ensured the implementation of the effect of low-temperature superplasticity during pressure welding.

According to the results of the research, it was found that the UFG structure of the EK61 alloy during pressure welding at a temperature of $T = 850$ °C is thermally stable, individual large particles of the δ-phase are preserved in the structure (Fig. 4*a*). After welding and heat treatment, it was found that the UFG structure of the EK61 alloy is transformed into a CG structure (Fig. 4*b*). The microstructure of the EP975 alloy is thermally stable, no significant microstructural changes occurred during the heat treatment (Fig. 4*b*).

This is evidenced by the preservation of the type and parameters of the fine-grained structure of the microduplex type, which is identical to the initial state of the microstructure of the EP975 alloy before pressure welding (Fig. 3*d*). It is important to note that intragranular dispersed particles of the γ-phase, no larger than 0.4 μm in size, present in the microduplex structure, are retained at the studied pressure welding temperature of 850 °C and, accordingly, can affect the formation of SSJ of opposite alloys in the EK61//EP975 combination.

Figure 5 shows the results of the energy-dispersive analysis of the SSJ zone of alloys in the EK61//EP975 combination after pressure welding and heat treatment, where the dashed lines indicate the boundaries of the diffusion zone. It is important to note that the processes of mutual diffusion of alloying elements are affected by the difference in the chemical composition of the alloys being joined, as well as the difference in the atomic radius (r_{at}) of the alloying elements. Nickel EP975 alloy is enriched with Co ($r_{at} = 0.125$ nm), W ($r_{at} = 0.140$ nm), in comparison with EK61 alloy, and depleted in Fe ($r_{at} = 0.127$ nm) and Cr ($r_{at} = 0.128$ nm). Therefore, apparently, the diffusion of Co ($r_{at} = 0.125$ nm) from the nickel alloy EP975 into the EK61 alloy proceeds actively. In this case, counter diffusion of iron and chromium is observed.

Thus, a detailed study of the results of energy-dispersive analysis indicates that, during pressure welding of two wrought superalloys EP975 and EK61 with a different type of hardening phase, the formation of SSJ occurs by diffusion with the formation of a γ-solid Ni-based solution. The chemical composition of the solid solution in the SSJ zone is intermediate between the chemical compositions of the joined alloys. As a result of pressure welding at a temperature of 850 °C, a diffusion interaction zone is formed, the width of which is approximately 3 μm.



(*a*) (*b*)

Fig. 4. Microstructure of SSJ zone: *a* – after PW; *b* – after PW and following HT

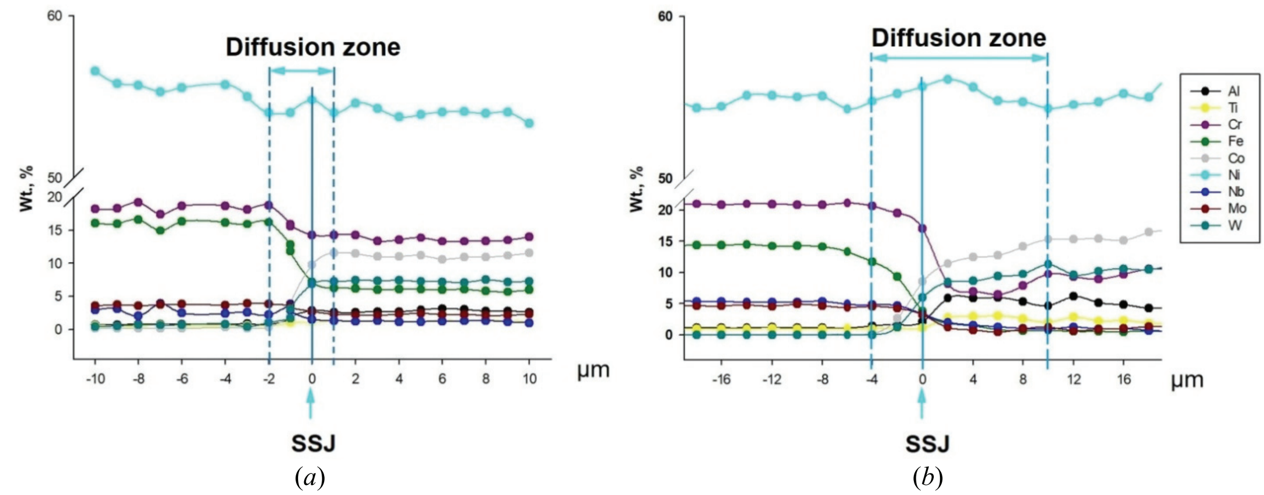


Fig. 5. Distribution of alloying elements in welded samples in a combination of EK61//EP975 superalloys after PW (*a*), as well as following HT (*b*)

The results of the microhardness study of welded samples in the combination of EK61//EP975 alloys after pressure welding and heat treatment are shown in Fig. 6.

After heat treatment, the microhardness of the EP975 alloy being joined far from the SSJ zone had the same values as after pressure welding. The microhardness of the less heat-resistant EK61 alloy after heat treatment increased to the level of the microhardness of the EP975 alloy. The microhardness values that were determined in the SSJ zone were not entirely correct. This is due to the fact that the diameter of the indenter print when measuring microhardness was approximately 20 μm, although the width of the diffusion interaction zone did not exceed 14 μm even after heat treatment. Therefore, the indenter print includes not only the SSJ zone, but also the adjacent areas of the EK61 and EP975 materials being joined, which undoubtedly affects the obtained microhardness value in this area of the welded sample.

The results of mechanical testing of welded blanks according to the uniaxial tension scheme are presented in Table 2.

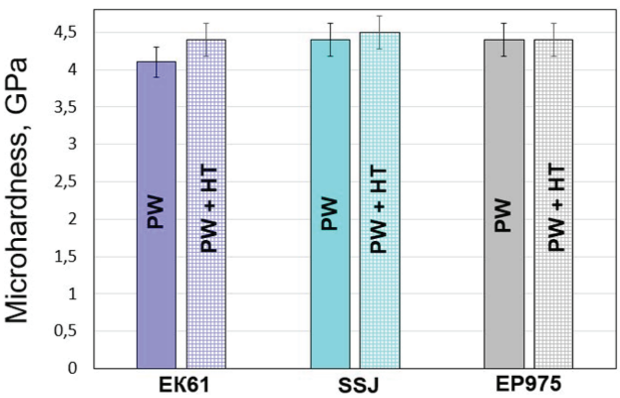


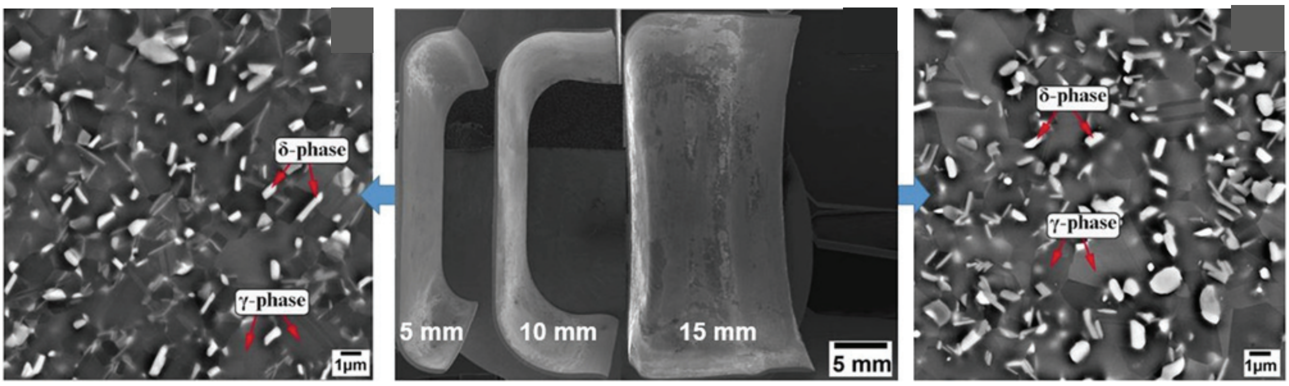
Fig. 6. Microhardness in welded samples in combination of EK61//EP975 superalloys after PW and PW + HT

The analysis of the tested samples showed that the fracture in the welded specimens occurred along the SSJ zone. It has been established that the strength in samples obtained by welding at $T = 850$ °C at room temperature is 0.8 of the strength of the EK61 alloy (Table 2). Tests at a temperature of 650 °C showed that the joint strength is 0.35 of the strength of the EK61 alloy (Table 2).

A comparative analysis of the above results with the data obtained by the authors earlier and published in [43] is of interest. In this paper, pressure welding of EK61 and EP975 alloys was carried out at a temperature of 925 °C, which is 75 °C higher than the temperature of pressure welding of the same alloys in similar structural states. The lower pressure welding temperature of 850 °C, which was used in this work, affected not only the width of the SSJ zone, but also the strength of the welded samples. A decrease in pressure welding temperature from 925 to 850 °C led to a significant decrease in the width of the diffusion interaction zone from 20 μm to 3 μm, which did not exceed 14 μm even after heat treatment. At the same time, the strength of the welded samples at room temperature (1170 MPa) turned out to be significantly higher than that of the samples after pressure welding at 925 °C (908 MPa). Although the test results at elevated temperature ($T = 650$ °C) showed the opposite result. The strength (455 MPa) of welded samples obtained by pressure welding at 850 °C turned out to be lower than after PW at 925 °C (664 MPa). At the same time, it should be noted that the level of SSJ strength achieved in both cases may be quite sufficient for obtaining real designs of bimetallic parts from opposite superalloys using SSJ in the form of a conical surface. It is assumed that during the operation of such bimetallic parts, the SSJ will be subjected to the action mainly of compressive stresses, rather than tensile stresses.

Table 2. Mechanical properties of welded joints and EK61 and EP975 superalloys

Condition	σ_b, MPa	$\sigma_{0.2}$, MPa	δ, %	T_{tests}, °C
(SSJ) PW, T = 850 °C	1170	1070	19	20
(SSJ) PW + HT, T = 850 °C	1140	970	20	
EK61, the initial state UFG	1490	1030	33	
EP975, the initial state FG	1690	1130	40	
(SSJ) PW, T = 850 °C	455	360	20	650
(SSJ) PW + HT, T = 850 °C	1110	780	2	
EK61, the initial state UFG	1300	870	55	
EP975, the initial state FG	1460	990	22	



(*a*) (*b*) (*c*)

Fig. 7. Samples after superplastic forming at T = 850 °C to the different depth:
a – microstructure (forming H = 5 mm); *b* – general view of the samples after SPF;
c – microstructure (forming H = 15 mm)

3.3. Superplastic forming of nickel alloy EK61 with UFG structure

According to the results of experiments described in detail in [62], as well as those carried out in this work, it is shown that low-temperature superplastic deformation of the EK61 UFG alloy by a mixed-type structure can be successfully implemented under various deformation schemes (compression, tension), including the formation of sheet samples. Implementation of the effect of low-temperature superplasticity of the EK61 alloy has good formability without cracking even at low temperature T = 850 °C and strain rate $\dot{\varepsilon} = 3\cdot10^{-3}$ s^{-1}. The formed model samples had a regular cylindrical shape [62] and were characterized by good filling of the die cavity (Fig. 7*b*).

The microstructural analysis of the material of hollow samples after the completion of the SPF (Fig. 7*a*, *c*) showed that the structure is duplex in all cases, the equiaxiality of the grains of the γ-phase was preserved. With an increase in the degree of deformation during SPF, some coarsening of the grains and a change in the phase composition were observed: at a forming depth of H = 5 mm, the grain size of the γ-phase was ~ 1.72 μm (Fig. 7*a*), at H = 10 mm ~ 2.0 μm, and at H = 15 mm ~ 2.4 μm (Fig. 7*c*), and the volume fraction of the δ-phase was ~ 20 %.

Due to the increase in the grain size of the γ-phase from 0.3–0.8 to ~1.7 – 2.4 μm during the SPF process, a decrease in the microhardness of the material was observed (Table 3).

It is interesting to note that the values of microhardness after SPF were significantly lower than in a similar material after pressure welding (Fig. 4). Apparently, this is due to a significant difference in the stress-strain state that occurs in the compression scheme implemented during pressure welding compared to the tension scheme observed in SPF.

Table 3. Microhardness of EK61 superalloy before and after of superplastic forming

Sample condition (forming depth *H*), mm	Microhardness, GPa
The initial state (before forming)	4.2
5	3.4
10	3.5
15	3.6

In the latter case, the most favorable conditions were observed for the realization of the effect of superplastic deformation and softening of the deformed material.

Compared with the results of a study [34] previously carried out on the foreign alloy Inconel 718, which is the closest analogue of the domestic alloy EK61 in terms of chemical and phase composition, the latter succeeded in additionally lowering the SPF temperature by 50°C.

Thus, the results of the study showed that sheet blanks from the EK61 alloy with a preliminarily prepared UFG structure had good superplastic formability and, therefore, can be in demand for the manufacture of various complex-profile thin-walled products by the SPF method.

4. Conclusion

As a result of the experimental studies, it was found that pressure welding under conditions of low-temperature superplasticity is an effective method for obtaining SSJ from EK61 and EP975 nickel alloys with various types of hardening phase. It is shown that in the process of pressure welding at a temperature of 850ºC between the alloys being joined, as a result of the processes of mutual diffusion of alloying elements, a transition zone of diffusion interaction is formed, the width of which is ~3 μm. The strength of the SSJ compound EK61//EP975 at room temperature reaches 0.8 of the strength of the EK61 alloy. Macrodeformation of model hollow specimens during SPF proceeds relatively uniformly and uniformly. With an increase in the degree of deformation (holding time) during SPF, a certain coarsening of grains from ~0.3 to ~2.4 μm is observed, which is consistent with a decrease in the microhardness values of the samples under study. The obtained data testify to the high superplastic properties of the EK61 alloy with the UFG structure and make it possible to recommend it for use in innovative technological processes of pressure welding to obtain SSJ from superalloys, as well as low-temperature SPF for shaping various types of hollow products of complex configuration for aerospace technology. The results obtained can be used to optimize scientific and technical solutions related to the development of innovative deformation technologies based on the use of the superplasticity effect to obtain, for example, bimetallic parts of a disk-shaft gas turbine engine, and are also relevant in the creation of resource-saving technologies for forming hollow products of complex configuration.

5. Funding

The research work on pressure welding EK61//EP975 was supported financially by the Grants Council of the President of the Russian Federation (Presidential Scholarship for Young Researchers and Postgraduates SP-4002.2022.1).

The SPF experiments were carried out within the framework of the State Assignment of the IMSP RAS No. 122011900474-5.

6. Acknowledgements

Electron microscopic studies were carried out at the collective service center of the IMSP RAS "Structural and physical-mechanical studies of materials".

7. Conflict of interests

The authors declare no conflict of interest.

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Received 20 February 2023; Accepted 13 April 2023; Published 26 May 2023