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Nickel Alloys Structure and Properties Control by Deformation-Thermal Treatment in Solid State

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Abstract

Systematic studies of a large number of nickel- and iron-nickel-based alloys have been done to find out optimum conditions for hot and severe plastic deformation, ensuring the transformation of the initial coarse-grain structure into the ultra-fine-grain structure with given parameters within micrometer, submicron and nanometer range.

It has been shown that a controlled change in the parameters of the hardening phase, its relation to the matrix and modes of deformation-thermal treatment can control the processes of structure formation in precipitation-hardening nickel alloys due to a purposeful change in the existing mechanisms of deformation and recrystallization.

The findings were used as a basis for developing a single methodological approach to obtaining bulk and sheet semi-finished products of nickel alloys with ultra-fine-grain and nanocrystalline duplex structures. The idea of the method was to carry out an intensive deformation-thermal treatment with stepwise temperature decrease from 0.9–0,8 T_{pl} to 0.6–0.5 T_{pl}. As a result of such treatment stepwise refining of the initial coarse-grained structure up to nanocrystalline structural state was achieved. It has been found that in precipitation-hardening alloys (γ'; γ''+δ) with submicrocrystalline and nanocrystalline structures there occurs low-temperature superplasticity effect which manifests itself at temperatures 200–350 ºC lower than in materials with microduplex structure. Herein 1.5–2-fold decrease in the flow stress and 1–1.5-order increase in the deformation rate is achieved. The correlation between the phase composition and the type of hardening of heterophased nickel alloys with thermal stability of submicrocrystalline and nanocrystalline structures was revealed.

The efficiency of the use of semi-finished products with a prepared ultra-fine-grain structure for producing precise complex geometry parts with homogeneous structure and high complex of mechanical properties obtained by the methods of volumetric and local forming and further standard thermal treatment was proved. There was proposed a method of molding under superplastic conditions which allows producing parts such as aircraft engine disks from the ultra-fine-grain workpieces with a structure regulated by varying the radius, which provides a high complex of properties, optimized to the actual conditions of their use. Using precipitation hardening nickel alloy such as Inconel 718 as an example, it was shown that the formation of submicrocrystalline structure in workpieces enables realization of the effect of low temperature superplasticity in promising technological processes of pressure welding and pneumoforming.

Keywords

Deformation; microstructure; nanocrystalline; nickel alloys; phase; recrystallization; reeling; solid state welding; superplasticity.

Introduction

The development of plastic deformation and recrystallization in heat-resistant nickel alloys is to a great extent affected by the condition of hardening phases: their morphology and the type of their relation to a matrix [1–4]. From this it follows that by varying the state of a hardening phase, its parameters and modes of deformation-thermal treatment we can considerably control the processes of structure formation in nickel alloys and thereby obtain the required complex of both technological and service properties in them. It is known that in nickel alloys [1, 4] with prepared microcrystalline (**MC**) structure the effect of superplasticity (**SP**) occurs at sufficiently high homologous temperatures (0.8–0.85 T_{pl}) and low deformation rates (10^{-3} –10^{-4} s^{-1}), which limits the wide use of this promising technology in the processes of producing the parts from the abovementioned materials. The enhancement of technological possibilities of the SP effect owing to an increase in the deformation rate and a decrease in the temperature of its occurence is possible using materials with submicrocrystalline (**SMC**) and nanocrystalline (**NC**) structures [1, 5].

At present much attention is paid to the preparation of such structural states, the study of their physical and mechanical properties, as well as the development of effective methods for producing semi-finished products with SMC and NC structures, which is especially important with regard to hardly-deformed nickel alloys [1, 5]. However, the formation of SMC and NC structures in complex nickel alloys and their influence on the SP behavior of these materials have not been sufficiently studied yet. The prospects of applicability of low temperature SP effect in the development of new resource-saving technologies for producing complex-geometry essential parts are not clear either.

Thereby the purpose of the paper is to summarise the results of systematic studies aimed at establishing patterns of formation of MC, SMC and NC structures in nickel-based alloys with different phase composition and morphology of hardening phases during severe plastic deformation in a wide temperature range, to assess their influence on SP properties and develop scientifically justified technological recommendations to obtain semi-finished products and parts with regulated complex of technological and operational properties.

Materials and Methods of Research

In order to establish general laws of forming regulated structures during hot deformation, in particular, those of the microduplex type, there were conducted systematic studies on a wide range of deformable precipitation hardening nickel alloys EI437BU, EI698-ID, EP742-ID, EP962, EP975, Waspaloy manufactured according to the traditional technology as well as alloys Rene88, Astroloy, Rene95 and N18 manufactured by powder metallurgy. These alloys significantly differ by the number (14–55 %) of hardening γ'-phase isomorphic with the matrix (γ-phase, face-centered cubic) on the basis of intermetallic $Ni_3(Al,Ti)$ with FCC (face-centered cubic) lattice.

Detailed comparative studies have been conducted on the typical representatives of alloys with isomorphic (EP962, γ'-phase of cuboidal shape with FCC) and nonisomorphic (Inconel 718, Ni_3Nb with orthorhombic structure of δ-phase of platelet shape) precipitation of hardening phases. Furthermore, to assess the high-speed SP effect, there was also studied the PDU1alloy with combined hardening, the chemical composition of which is given in [6]. Methods of studying the structure and properties of the alloys are described in detail in [7–9]. To produce large-scale SP semi-finished products, a hydraulic press with a tonnage of 1600tf equipped with isothermal die unit UISHB-510 was used. The fabrication of complex-geometry parts by method of local forming under temperature-speed SP conditions was carried out on the modernized mills SRD-800 (reeling) and PNC-600 (rotary drawing).

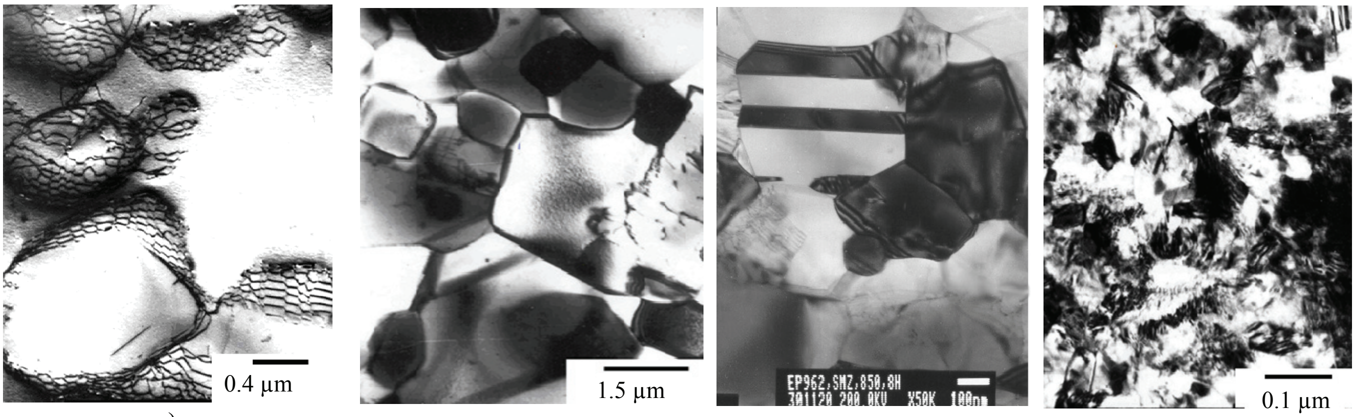
Results and Discussion

Formation of Microcrystalline Structure

An alloy with isomorphic hardening γ' – phase (EP962) precipitates. The results of systematic studies of microstructural changes in the EP962 alloy under heating, annealing of cold-worked material and hot deformation cited in [9] made it possible to identify patterns of formation of microduplex structure under deformation-thermal treatment in two-phase ($\gamma + '\gamma$)-region. In this study it was shown that the most favorable conditions for the development of continuous dynamic recrystallization in a two-phase ($\gamma + \gamma'$)-region are created in case of pre-heterogenization of initial coarse-grained (80 μm) structure, as a result of which a coagulated γ'-phase of cuboidal shape with a size of 0,5mkm precipitates inside the grains. It has partially coherent γ/γ' interphase boundaries in which misfit dislocations are observed (Fig. 1, *a*). Moreover, even long annealing in the two-phase ($\gamma + \gamma'$)-region for 50–100 hours does not lead to further transformation of partially coherent interphase boundaries γ/γ' into incoherent ones, although the size of the particles is enlarged to the size of 1–3 μm. The study of evolution of the microstructure during hot deformation of the EP962 alloy has allowed to identify the main deformation and recrystallization processes that lead to the transformation of a coarse-grained microstructure into a microduplex one (Fig. 1, *b*).

It was found that at any studied strain rate of the material in a pre-heterogenized condition a microduplex-type structure is formed, the parameters and recrystallized volume fraction of which are determined by temperature and strain-rate conditions.

It was found out that new grain γ/γ and interphase γ/γ', γ'/γ' boundaries with predominantly high-angle misorientations form by means of interaction between lattice dislocations and first of all misfit dislocations at interphase boundaries γ/γ'. As a result of continuous formation and accumulation of dislocations at the



a) *b*) *c*) *d*)

Fig. 1. Microstructure of the EP962 alloy after deformation-thermal treatment:
a – CG structure, after annealing in $\gamma + \gamma'$ – region, 100 hours; *b* – MC structure; *c* – SMC structure; *d* – NC structure

partially coherent phase boundary, which is an effective barrier to dislocation motion, there occurs a disturbance of coherence up to its complete loss, which leads to an increase in the distortion of the matrix near the particles and the development of multiple slip, which causes dynamic polygonization process at the initial stage of deformation to form a substructure, wherein subgrain size is determined by the distance between the particles of γ'-phase.

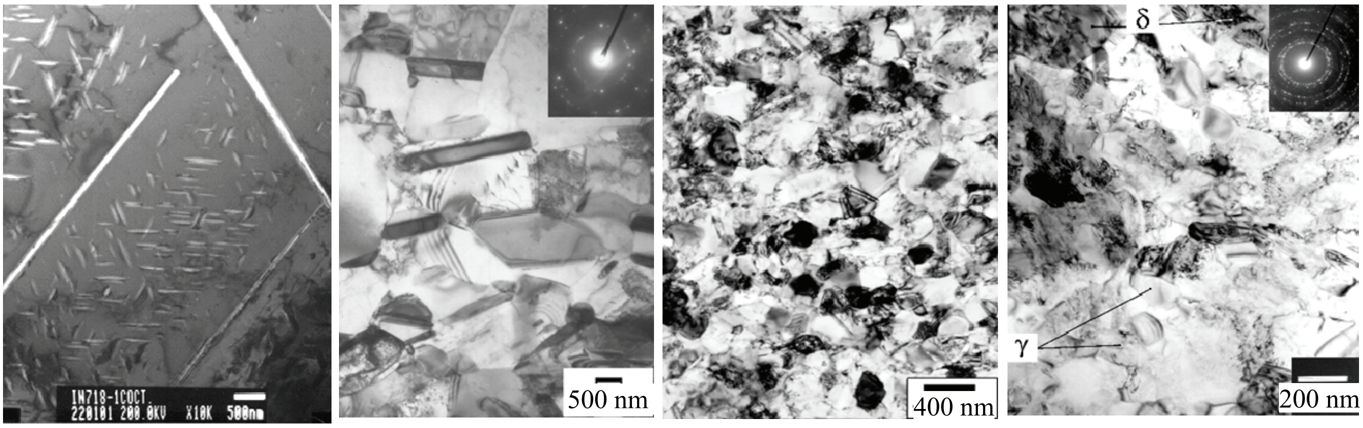
The occurence of "bridges" (sub-boundaries) interconnecting adjacent particles activates diffusion mass transfer. It results in the acceleration of the process of dissolution of smaller particles and coagulation of larger ones, thus creating conditions favorable for the development of a mechanism of coalescence of subgrains, leading to an increase in their size and disorientation angle between them. During deformation partially coherent particles gradually transform into incoherent γ'-phase grains of globular shape separated from the matrix by arbitrary high-angle interphase boundaries. While γ'-phase particles enlarge and initial partially coherent interphase boundaries transform into incoherent ones in the course of developing dynamic recrystallization the disorientation angle between the grains increases and they gradually transform into the grains with high-angle boundaries of general type.

The results of EBSD analysis of the changes in the grain boundary misorientations in hot-deformed samples (1100 °C, 10^{-4} s^{-1}) of the EP962 alloy during hot plastic deformation showed the following. During hot deformation the fragmentation of initial coarse grains into microregions, disoriented relative to each other occurs. At the initial stage of deformation (10–30 %), the average misorientation angle between submicroregions is 2.4–3.0° and with further increase in the extenet of deformation 75 % it increases up to high-angle > 10–15 %. The share of the low-angle grain boundaries decreases from 55 to 43.5 %, and that of high-angle boundaries, on the contrary, increases from 39.4 to 46.8 %.

Thus, based on the results of comprehensive studies of the microstructure of hot-deformed samples it was found that the principal mechanism of conversion of grained structure both in a single-phase nichrome [10], which is the basis (γ-phase) for most nickel-based alloys, and in the matrix (γ-phase) of the EP962 alloy (only in the case of pre-heterogenized state) is the development of multiple slip and formation of a stable subgrain structure at small hot deformation, which with increasing extent of deformation up to 70–80 % and higher transforms into a grained structure. At the same time the main role of the coagulated partially coherent γ'-phase precipitates present in the structure, is that they contribute to the development of multiple slip, leading to the formation of a stable subgrain structure whose parameters are determined by the interparticle distance. As a result of structural changes taking place in the hot deformation in a two-phase (γ+γ)-region, a stable structure of microduplex type is formed, which is very important for the manifestation of the SP effect.

Metallographic analysis confirmed that an increase in the rate of deformation as well as fractional deformation with postdeformation annealing contributes to the production of the recrystallized microstructure in the bulk of the material [7, 8, 11].

An alloy with nonisomorphic hardening δ-phase (Inconel 718) precipitates. The comparative analysis of this alloy to the EP962 alloy revealed both general patterns of structure formation under hot deformation and the differences that are apparently due to the influence of chemical and phase composition of alloys,



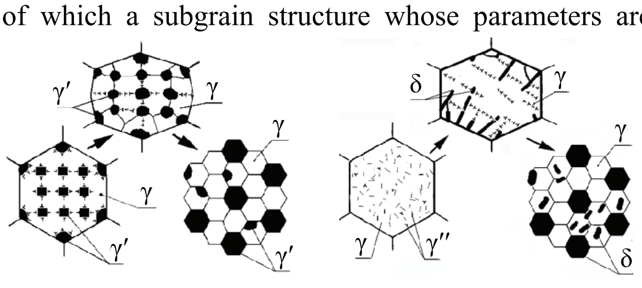
a) *b*) *c*) *d*)

Fig. 2. Microstructure of the Inconel 718 alloy after deformation-thermal treatment:
a – grained structure, heterogenizing annealing in γ + δ – region; *b* – MC structure; *c* – SMC structure; *d* – NC structure

which manifests itself through the difference in morphology of hardening phases [12, 13] as well as due to the peculiarities of their precipitation during heat treatment and deformation (Fig. 2).

The differences in the formation of microduplex structure under hot deformation in the Inconel 718 and EP 962 alloys are mainly due to the different nature of hardening δ and γ′ phases, their morphology and precipitation kinetics. In particular, while in Inconel 718 alloy with nonisomorphic δ-phase the decomposition of the solid solution is preceded by quite a long incubation period 10–15 min, in the EP962 alloy isomorphic γ′-phase under cooling below the solvus line precipitates almost instantaneously. Furthermore, a higher homologous temperature of hardening phase dissolution of the EP962 alloy causes an increase in temperature region of the dynamic recrystallization 1100 ºC as compared with Inconel 718 wherein such recrystallization occurs at lower homologous temperatures due to the lower temperature of dissolution of δ-phase 990 ºC.

What is common to the alloys being studied is that the transformation of a coarse-grain structure into a MC of a microduplex type occurs during continuous dynamic recrystallization in the course of which a subgrain structure whose parameters are



a) *b*)

Fig. 3. Schemes of transformation of the initial coarse-grain structure into a MC structure of a microduplex type under hot deformation:
a – EP 962, 1100 ºC, 10^{-4}–10^{-5}s^{-1}; *b* – Inconel 718, 925 ºC, 10^{-4}s^{-1}

determined by the distance between the coagulated precipitates of a hardening phase, transforms into a grain structure with high-angle boundaries of an arbitrary type with the growth in strain. At the same time coherent or partially coherent precipitates of the second phase (γ′-phase in the EP962 alloy, or δ-phase in the Inconel 718 alloy) are transformed into noncoherent grain-particles randomly oriented relative to the matrix grains. Schematically, the process of transformation of a coarse-grained structure into a MC of a microduplex type in the EP 962 and Inconel 718 alloys is shown in Fig. 3.

Formation of SMC and NC Structures

Many years' research conducted in the IMSP RAS served as a basis for the development of the universal methodological approach to the production of bulk and sheet of semi-finished nickel alloys with SMC and NC structures. Its essence is to carry out intensive deformation-thermal treatment (**IDTT**) using mainly a scheme of comprehensive isothermal forging (or forging followed by rolling) and stepwise decrease in the processing temperature from 0.9–0.8 T_{pl} to 0.6–0.5 T_{pl} [12 ,13].

The developed methodological approach is implemented in a number of ways the scientific novelty of which is protected by patents of the Russian Federation № 2041284, 2119842, 2269,585 as well as by European patent EP № 0 909 339 B1. The proposed treatment of precipitation-hardening alloys results in stepwise structure refinement: a coarse-crystalline (**CC**) structure of a macroduplex type is transformed into a MC structure of a microduplex type, and then at a lower processing temperature MC structure is transformed into a SMC structure and further into a NC one (Fig. 1, *c*, *d*) that is into the structures of "submicroduplex" and "nanoduplex" types, respectively [13].

Schematically, the process of structure formation under IDTT leading to stepwise refinement of the initial coarse-grain structure of the matrix (macroduplex) type into a more fine-grain structure of a duplex type, up to a NC structure in heat-resistant nickel-based alloys (**HNA**), hardened by isomorphic γ′-phase is shown in Fig. 4.

During stepwise structure refinement from CC to MC and further to the size of SMC, according to the data in [13], the level of flow stress significantly reduces for Inconel 718 alloy. For example, at a temperature of 0,8*ts* a decrease in the grain size from 40 µm CC to 1–3 µm MC leads to a 3-fold decrease in the level of flow stress (Fig. 5). It should be noted that the Inconel 718 alloy with MC structure at the above temperature exhibits high values of technological plasticity because 0,8*ts* temperature 800 ºC corresponds to the lower temperature threshold of SP for this structural state.

It is obvious that for further structure refinement up to SMC and NC sizes IDTT should be carried out at lower homological temperatures (0.67–0.57 T_{pl}). So after IDTT at 700 ºC bulk workpieces with SMC structure were obtained. Their study showed that the lowest temperature threshold of low-temperature SP effect is 650 ºC.

This effect is known [5] to be caused by the fact that a decrease in the grain size up to submicron sizes results in a significant increase in the diffusion coefficient by approximately 1.5 orders of magnitude. At the same time the increased length of grain boundaries leads to an increase in the contribution of grain boundary diffusion controlling grain boundary sliding which is the dominant mechanism of low-temperature SP. This circumstance seems to be very essential since it enables the development of recrystallization processes in SMC material, including continuous dynamic recrystallization at lower homological temperatures at which these processes are not possible in the material with the initial CC structure.

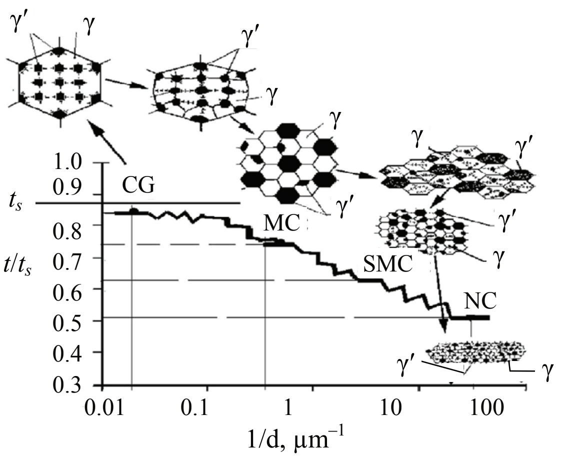


Fig. 4. Scheme of microstructure refinement in HNA hardened by isomorphic γ′-phase under IDTT

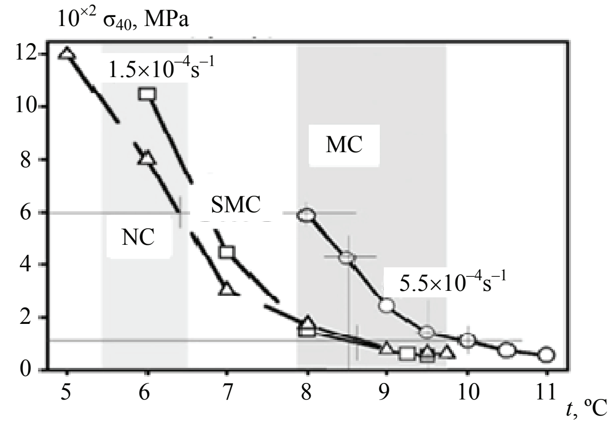


Fig. 5. Temperature fields of MC, SMC and NC structures formation in the Inconel 718 alloy and dependence of flow stress σ_{40} on temperature and the type of the initial state:
o – coarse-grain (40 µm); □ – microcrystalline (1–3 µm);
Δ – submicrocrystalline (0.3 µm)

It was found experimentally that by means of IDTT in the Inconel 718 alloy there can be also formed a NC structure (0.08 µm) in a bulk semi-finished product (minimum size 10×10×50 mm^3) at a low homological temperature of 0.57 T_{pl} (550 ºC) which corresponds to temperature range appropriate for cold deformation of the initial coarse-grain state [13]. Schematically the staging of structure formation under IDTT leading to gradual refinement of the initial coarse-grain structure of a matrix (macroduplex) type up to a nanoduplex state in an alloy, hardened by precipitating isomorphic γ′-phase is shown in Fig. 2.

Thus, as a result of purposeful control of IDTT modes in nickel alloys with different phase composition and different types of reinforcement there can be obtained various microstructure states, the parameters of which are regulated by the need to provide the required technological characteristics of the SP in a semi-finished product taking into account the peculiarities of a finished part design and the demands imposed on performance properties.

The above results were used as a basis for the development of a predicting technique according to a nomogram first presented by the author in [7], the specific thermo-mechanical modes of producing MC, SMC and NC structures with regulated parameters. The pseudo-binary diagram was expanded by including new results, which allowed us to cover almost the entire range of structural states, which can be obtained in precipitation-hardening nickel- and iron-nickel-based alloys, and recommend optimum temperatures for parts treatment by IDTT method followed by SP strain (Fig. 6).

The pseudobinary diagram shown in Fig. 6. summarizes the results of the studies about the influence of γ′-forming elements Al and Ti elements determining the amount of precipitating γ′-phases, on

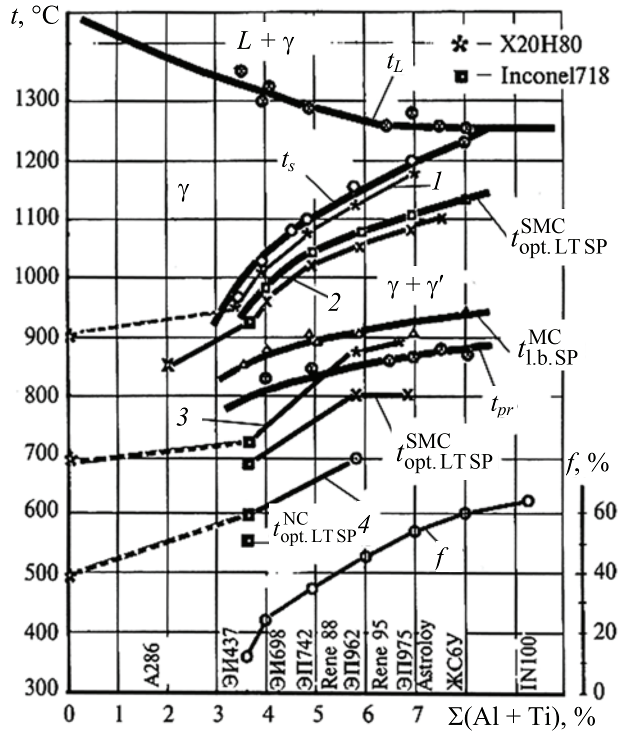


Fig. 6. The effect of γ′-forming elements Al and Ti on temperature ranges of obtaining MC, SMC and NC structures and SP deformation in nickel alloys:

t_{pr}, t_s – temperatures of dissolution start and complete dissolution of γ'-phase; t_L – melting temperature of γ – solid solution; *L* – liquid melt; γ – single-phase region of a solid solution of γ-phase; γ + γ' – two-phase region of γ and γ'-phases; $t^{MC}_{opt. SP}$, $t^{MC}_{l.b. SP}$ – optimal temperature and lower temperature boundary of SP occurrence in an alloy with MC structure; $t^{SMC}_{opt. LT SP}$, $t^{NC}_{opt. LT SP}$ – optimal temperature of low-temperature SP in alloys with SMC and NC structures; *f* – the volume fraction of hardening γ'-phase.

Curve *1* – treatment temperatures at which a microcrystalline structure of a matrix type is formed in alloys. Curves *2*, *3* and *4* are optimal temperatures at which IDTT leads to the formation of MC, SMC and NC structures of a duplex type: microduplex, submicoduplex and nanoduplex, respectively. Dashed curves designate the areas with the predicted temperature treatment modes

temperature modes of obtaining MC (microduplex), SMC (submicroduplex) and NC (nanoduplex) structures and SP deformation of nickel alloys.

The diagram shows the lines corresponding to the optimal (opt.) temperatures of SP effect, including low-temperature (**LT**) SP for a variety of structural states as well as average temperatures for each stage of deformation-thermal treatment, providing obtaining duplex structures with a regulated grain size: MC 3–5 μm or SMC 0.3–0.5 μm or NC 0.08 μm in bulk and sheet semi-finished products from nickel- and nickel- iron-based alloys (the Inconel 718 alloy is placed in accordance with δ-phase dissolution temperature).

The practice of using a developed nomogram and its improved version testifies to the effectiveness of predicting specific thermo-mechanical modes of producing the MC, SMC and NC structure states in heat-resistant alloys. When using the nomogram in most cases the studies are limited themselves to checking the selected treatment modes in practice based solely on the data of chemical composition or even the quantitative content of basic phase-forming elements (Al, Ti), dissolution temperature of basic hardening phase, and if necessary to a small correction of the selected modes. This allows you to manifold reduce the amount of research while developing treatment modes for new alloys and thereby significantly reduce material and labor expenditure.

Low-Temperature and High-Speed Superplasticity of Nickel Alloys

By varying the modes of deformation-thermal treatment in nickel alloys under investigation there can be formed MC structural states with approximately the same grain size of γ-phase [7, 8, 11]. However, the differences in phase composition, especially in the number of isomorphic γ′-phase, according to the results of mechanical tensile tests, have a significant impact on their SP properties of [7, 11, 14]. For example, after deformation at a speed of $1.33 \cdot 10^{-2}$ s^{-1} plasticity of low alloys did not exceed 190 %, and coefficient $m = 0.2$. At the same strain rate the samples of more doped alloys (35–45 % γ-phase) collapsed only after 400–500 % stretching, although the coefficient was close to 0.3. Maximum values of unit elongation ≥ 550 % and coefficient $m = 0.5$ at a strain rate of 1.33×10^{-2} s^{-1} were observed in the alloy containing 55 % γ′-phase. Note that this alloy is characterized by high values of unit elongation δ ≈ 200–250 % and coefficient of rate sensitivity $m = 0.3$–0.35 even at a strain rate of $1 \ldots 1.33 \cdot 10^{-1}$ s^{-1}. The latter testifies to the fact that the alloy, in which the ratio of γ and γ′-phases in microduplex structure is practically equal to 1:1, is also characterized by high-rate SP [14].

It should be noted that SP behavior of a specific nickel alloy is largely determined by the initial grain size of the phases. Thus, in [14] it was showed that in general phenomenology of the SP effect and the nature of the existing deformation mechanisms for MC, SMC and NC structures are largely similar. At the same time, there are some peculiarities which are primarily due to SMC and especially NC disequilibrium as well as a greater length of interphase and grain boundaries [6, 14]. A number of studies [6, 14, 15] have shown that the formation of SMC structure with non-equilibrium grain boundaries and high levels of internal stress in nickel alloys contributes to a significant expansion of the SP temperature-rate interval. Thus, in precipitation-hardening alloys (γ'; $\gamma''+\delta$) with SMC structure the effect of low-temperature SP occurs at temperatures of 200–250 ºC lower than in the materials with MC structure. This is accompanied by 1.5–2-fold decrease in flow stress and an increase in strain rate by 1–1.5 order of magnitude [14]. A distinctive feature of microstructural changes in nickel alloys with SMC and NC structures occurring during SP deformation is the transformation of non-equilibrium boundaries into equilibrium ones, which is largely similar to the structural changes that occurred during annealing of the aforementioned structural states. These results indirectly confirm the mechanism of formation of SMC and NC structural states found out in the research. It is evident that under SP deformation of SMC and NC structures further development of recrystallization processes that have not completed in the course of preliminary IDTT takes place.

Grain and subgrain refining up to NC sizes provides maximum decrease in temperature at which manifestation of the effect of low-temperature SP is possible [13]. For example, the Inconel 718 alloy with NC structure exhibits the signs of SP even at a temperature of 600–575 ºC.

In the study of dispersion-hardened PDU-1alloy it was found that after preliminary IDTT the alloy exhibits the features of both high-rate and low-temperature SP [6]. High-rate SP at high homological temperatures (1200–1275 ºC, above the temperature of γ'-phase dissolution) results from high stability of SMC structure which is due to the presence of refractory particles of yttrium phase. As the temperature increases from 1100 to 1275 ºC SP optimum shifts to higher strain rates (by 0.5–1 order of magnitude). The low-temperature 1000–800 ºC SP is conditioned by the presence of a large number of γ'-phase grains with incoherent interphase boundaries of the general type in the structure.

Pore formation under SP deformation is observed in all alloys. Moreover, the places of preferred pore nucleation are carbide particles. In precipitation-hardening alloys (Inconel 718, EP962) coalescence of single microscopic pores occurs primarily in the longitudinal direction, while in the PDU-1alloy the formation of grain boundary microcracks as a result of pores merger occurs predominantly in the direction transversal to the axis of stretch of the samples. Obviously, because of the differences in pore formation plasticity indexes in the PDU-1alloy are significantly lower than in the precipitation hardening alloys [14–16]. Apparently this is due to the presence of refractory particles of yttrium phase at grain boundaries which accelerate the development of pore formation under SP strain especially at lateral borders.

Using the Superplasticity Effect in Processes of Producing Precise Nickel Alloy Parts

The implementation of high-performance technologies for the production of complex-geometry parts under SP is possible only if the workpieces have MC and SMC structures exhibiting high plasticity and low flow stress under SP strain [1, 4, 7].

Based on the research done, theoretical foundations have been developed and experimental-industrial testing of a integrated processing technology of heat-resistant nickel alloys has been carried out. The latter is protected by RF patent number 2119842, which includes the stage of producing large-sized SP semi-finished products with MC structure as well as the stages of the manufacture of parts such as gas turbine engine (**GTE**) disks by method of local shaping – rolling in SP mode – and final thermal treatment.

Numerous results of experimental-industrial testing of new technological processes of SP rolling and rotational drawing of parts such as GTE disks (EP742, EP962, Inconel 718, Rene 95, N18), as well as those of field tests (EP962) showed that their application provides a significant increase in metal utilization factor (1.5–2-fold), reduction of production costs and quality improvement. This ensures a high level of technology (**SP**) at the manufacturing stage of precision complex-geometry parts and high level of service properties that are optimized to the actual operating conditions in the finished parts made of heat-resistant nickel alloys.

The designed technique of IDTT proved to be very effective for obtaining both MC and SMC structures 0.3–0.5 µm in massive bulk (with a weight of 10 to 100 kg) and sheet semi-finished products (with a size of at least 40×200×0.8 mm^3) made of the Inconel 718 alloy [12, 17, 18]. Moreover, the maximum sizes of semi-finished products being manufactured are limited only to the capacities of the existing press-forging and rolling equipment which is suitable for implementing the developed method of IDTT in industrial environment and not just in isothermal one. It should be noted that with a decrease in IDTT temperature the labor input of manufacturing semi-finished SP products increases due to a considerable increase in power parameters of the process, the number of transitions. In this context a rational choice of IDTT modes to produce either an MC or SMC structural state is needed, which will be further used in a particular process of manufacturing parts under superplasticity conditions.

The developed methods to control structure formation of processes under intense and hot plastic deformation enable us to change structural states in a wide range in heat-resistant alloys and render them

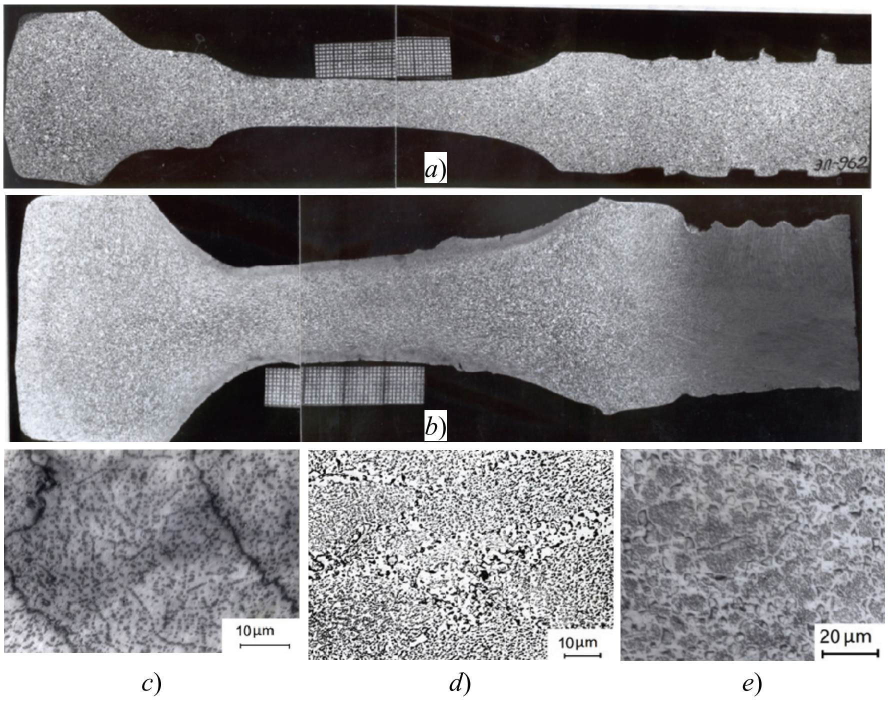


Fig. 7. Macro (*a*, *b*) and microstructure of rolled discs made of EP962:
a – coarse-grain structure being uniform over the entire cross-section of the disc: reeling at a temperature of 1050 ºC plus heat treatment by heating up to a temperature equal to $t_{pr\cdot\gamma'}$ and aging; *b* – regulated changes in the macro and microstructure over the cross-section of a rolled disc; *c* – a coarse-grain structure with winding grain boundaries in the rim; *d* – a structure of the "necklace" type in the web; *e* – a fine-grain structure in the hub; rolling at 1100 ºC plus heat treatment in (γ + γ′) region

the required technological and operational properties. For instance, parts like disks with a homogeneous or regulated by changing the cross section structure can be made. By the example of the wrought alloy EP 962 [19] it was shown that hot deformation in temperature and high-speed SP mode can create in heat-resistant nickel-based alloys (**HNA**) such microstructural states which after the final heat treatment provide them with the required combination of mechanical properties at low and high temperatures that are optimized taking into account actual conditions of parts operation. Fig. 7 shows macro- and microstructures of different types formed in discs made of EP962 alloy as a result of rolling in two different ways: in SP state (7*a*) and in speed SP mode, while varying the temperature and the structure (7*b*).

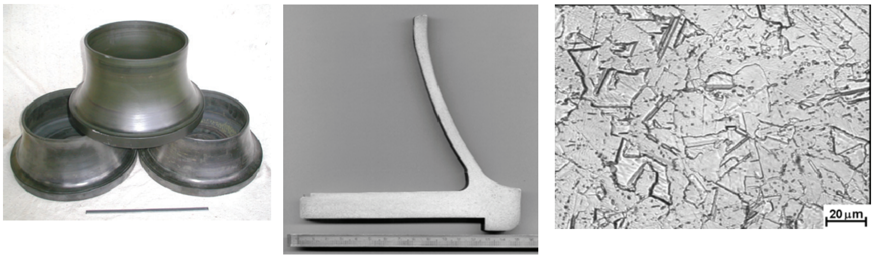
After having been rolled, discs were subjected to final heat treatment. In the first disc rolled in SP state, after heat treatment a homogeneous coarse-grain structure around cross-section was obtained. In the second disc a heterogeneous structure was formed. Since the greatest power load is experienced by a hub, a microduplex structure with $\sigma_b \approx 1650$ MPa is kept in it, given that during operation this part of the disk is heated up to a relatively small temperature ≤ 500 ºC at which a microduplex structure demonstrates long-time properties.

The web and the rim were rolled under conditions which provide the formation of a "necklace" type structure and a coarse-grain structure with winding grain boundaries, respectively. These types of structures ensure optimal properties of an alloy in the temperature-force conditions of the operation of these parts of the disc. In general, the mechanical properties in both disks conform to the specifications, but in the disc with a regulated changing structure they were significantly higher (Table 1).

Table 1

Mechanical properties of nickel alloys after SP deformation and heat treatment

Microstructure type	Room temperature				Heat-resistance at 650 °C	
	σ_b, MPa	$\sigma_{0.2}$, MPa	δ%	ψ %	Stress, MPa	Time before rupture, hour
EP962 alloy						
SMC (d_γ = 0.3μm) *After SP deformation*	1633	1422	9	27.4	–	–
Microduplex (d_γ = 5.5 μm) *After SP deformation*	1650	1176	24	22.4	1000	74
Fine-grain (d_γ = 30 μm)	1590	1090	14.5	15.5	1000	140
Coarse-grain (d_γ = 120 μm)	1500	1020	22.5	17.0	1000	284
"Necklace"	1630	1255	21.9	18.2	1000 1050	444 188
Coarse-grain structure with winding grain boundaries	1562	1222	20.7	15.0	1050 1080	243 102
Inconel 718 alloy						
SMC (d_γ = 0.3 μm) *After SP deformation*	1560	1300	5.1	11.0	–	–
Fine-grain (d_γ = 30 μm)	1503	1174	17.5	31.5	710	42.6



a) *b*) *c*)

Fig. 8. A general view of (*a*), macro- and microstructure (*b*, *c*) of parts made of the Inconel 718 alloy manufactured by rotary drawing under superplasticity

For the process of manufacturing by the local forming method (rolling or rotary drawing) of SP complex-geometry parts such as disks the most optimal workpieces are those with a prepared MC structure [8, 11]. For example, in a large-size workpiece made of the Inconel 718 alloy with the initial coarse-grain structure ∅(200–250)×400 mm the obtaining of MC structure was combined with the obtaining of a simple form stamp for subsequent rotary drawing. Rolling (rotary drawing) was carried out at a temperature of two-phase ($\gamma + \delta$)-region, while in the rolled section of the part of a "flange" type a homogeneous MC structure was formed (Fig. 8).

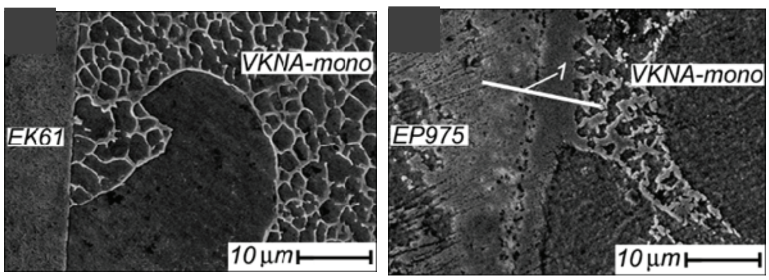
The study of the structure and properties of rolled parts has shown [20] that after standard heat treatment quite a fine-grain 12–16 μm structure providing an increased level of short-term strength and plastic properties at room and elevated temperatures remains in disks. Heat-resistant properties at 650 ºC of samples cut from the disk also completely meet technical requirements.

When sheet semi-finished products with MC and SMC structures are produced from hard-to-deform nickel alloys it will be preferable to use a process flow diagram according to which at the first stage a massive semi-finished product with MC structure is produced. Then, at the second stage a sheet is rolled to a desired thickness by isothermal or quasiisothermal rolling (in the heat insulating package) with simultaneous forming a required SMC structure in the latter. Basic principles of the new cost-effective technology for producing semi-finished sheet products with MC and SMC structures of Inconel 718 alloy are protected by RF patent number 2269585.

Solid-Phase Welding of Heat-Resistant and Intermetallic Nickel-Based Alloys

A systematic study of the influence of different structural states (CG, MC and SMC) on weldability in solid state was carried out using the Inconel 718 alloy as an example [16]. It was experimentally found in [21, 22] that forming a SMC structure in workpieces enables implementation of SP forming processes and pressure welding in the low-temperature SP state. A qualitative porous solid state compound was obtained at temperatures of 150–200 ºC lower than temperature ranges of known processes. In addition, the principle possibility of producing solid-phase compounds from the samples made of the same material but with different structures such as CG-MC, or CG-SMC or MC-SMC was shown. Moreover, it was found that lower temperature of obtaining solid-phase compound is determined by SP characteristics of a sample having more fine-grain structure. Similar results [23, 24] have been obtained with the Russian EK61 alloy which is a close analogue to Inconel 718 alloy.

Interesting results have been obtained in a recently published paper [25] obtained in collaboration with scientists from the VIAM and IMET RAS. In this study it was shown that the most effective method for producing permanent alloy connections on the basis of Ni3Al with highly-resistant nickel alloys such as EP975 is a new solid phase pressure welding process under high-temperature superplasticity. Solid-phase compounds (**SPC**) of heterophase monocrystals of alloys on basis of Ni3Al with deformable nickel alloys EK61 and EP975 have been produced. The characteristic features of gradient SPC structures obtained under low and high temperature superplasticity at homologous temperatures of ~ 0.6 and 0.9 Tpl were revealed, as well as the nature and direction of diffusion processes at the interface of a single crystal of intermetallic alloy with a deformable polycrystalline alloy. The study of gradient SPC structures (Fig. 9, 10) showed that under pressure welding the deformation is localized in disk alloys having UFC structure (duplex $\gamma + \gamma'$ for EP 975 and $\gamma + \delta$ for EK61) as a result of the preceding thermoplastic processing and showing superplasticity.



a) *b*)

Fig. 9. Specific microstructures of SPC pairs VKNA-mono//EK61 (*a*) and VKNA-mono//EP975 (*b*) (1 is a line of micro-*X* ray structural analysis)

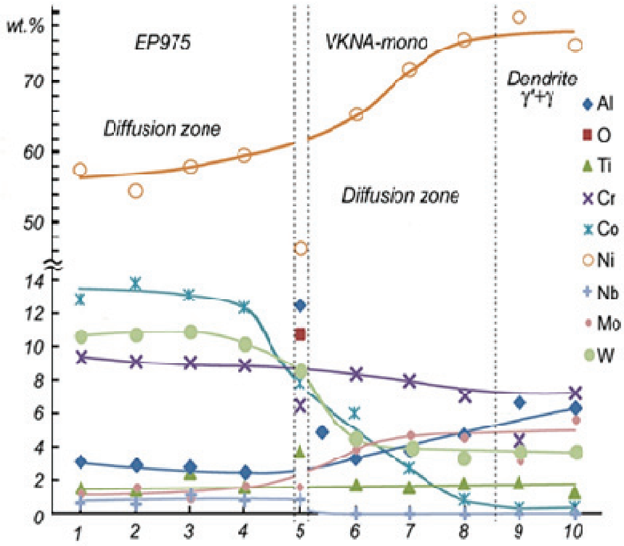


Fig. 10. The distribution of components in SPC along the line shown in Fig. 9, *b*

Conclusion

This paper summarizes the results of long-term (over 25 years) systematic reserach of a wide range of nickel- and iron-nickel-based alloys with different types of hardening related to the study of patterns of structure formation under plastic deformation in a broad temperature and strain-rate range. It analyzed the nature of the existing mechanisms of plastic deformation and recrystallization, and their relationship with the processes of dissolution and precipitation of the second phases. On the basis of the analysis the first attempt was made to develop a methodological approach to the control of the processes of structure formation under deformation-thermal treatment by means of integrated regulation of of hardening phases condition, processing modes, including modes of SP deformation and the final heat treatment. The practical application of the developed methodological approach enables to obtain in semi-finished products and parts regulated structural states which provide the required complex of technological and operational properties.

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