

DESIGNING OF HIGH-RHENIUM SINGLE CRYSTAL NI-BASE SUPERALLOY FOR GAS TURBINE BLADES

E.N. Kablov, N.V. Petrushin

All-Russian Scientific- Research Institute of Aviation Materials (VIAM), Radio str, 17, Moscow, 107005, Russia

Keywords: nickel-base single crystal superalloy, rhenium, alloy designing, phase stability, lattice misfit, stress-rupture, ductility

ABSTRACT

Chemical and phase composition for new high-rhenium single crystal Ni-base superalloy is computer designed. The microstructure, liquation coefficients and misfit-factor of alloy single crystals as cast condition and after thermal treatment are studied. High-temperature creep tests within the temperature range of 900-1100 °C for 1000 hours for single crystals of an alloy with orientation $\langle 001 \rangle$ are carried out and evolution of their microstructure is studied at creeping. The alloy contained 9 % Re, has acceptable phase stability and a high stress rupture:

$$\sigma_{1000}^{900} = 450 \text{ MPa}, \sigma_{1000}^{1000} = 215 \text{ MPa}, \sigma_{1000}^{1100} = 95 \text{ MPa};$$

density 9089 kg/m³. It is shown, that formation of Re enriched lamellar precipitations of a new phase at prolonged high temperature action has a low influence on ductile properties of single crystals of the studied alloy.

INTRODUCTION

At present some generations of Ni-base alloys for single crystal gas turbine casting are developed [1 - 5]. The first generation Ni-base superalloys contain traditional alloying elements such as Al, Ti, Cr, Mo, W, Ta, Nb and Hf. The alloying element Re in number of 2-4% and 5-7% (by wt.) respectively is introduced in the compositions of the second and third generations Ni-base superalloys. Re contained Ni-base superalloys additionally alloyed with Ru in number of 2-6% are referred to the fourth and fifth generations.

It is quite natural, that the basic alloying elements are available in various combinations for different alloys conditionally referred to one of the specified generations. Each new generation of single crystal Ni-base superalloys allows increasing in temperature capability of the blades by 20-25°C. Positive influence of Re on high temperature strength of nickel-base alloys is caused by increase in solidus temperature of the alloy, high solubility of Re in Ni γ - solid solution and increase in the period of its crystal lattice, decrease in diffusion coefficient of alloying elements. However at prolonged action of high temperature topological close-packed phases (TCP) that can have negative influence on mechanical properties are formed in Re contained Ni-base superalloys. To stabilize phase composition and decrease the probability of TCP phase precipitation these alloys are alloyed with Ru [6 - 9]. It can be assumed that from the thermodynamic point of view in anyone balanced Re and Ru bearing nickel-base superalloys availability of TCP phases is probable. Not only a chemical composition but also the kinetic factor, namely diffusion of atoms of refractory elements in Ni solid solution that are part of TCP phase, determines the volume fraction of TCP phases in alloy

Structure. Re and Ru have low diffusion coefficient in Ni [10]. High price of Ru as alloying element refers to its disadvantage. The purpose of the article is designing and an experimental research of a new Re-bearing single crystal Ni-base alloy with extremely high stress rupture in the temperature range of 900-1100°C, satisfactory density and acceptable phase stability. To increase the efficiency of a new alloy development a method of computer design was used. The basis of this method are regressive models of "composition-property" type to calculation the phase composition and material properties (for example, γ' -phase volume fraction, chemical composition of γ - and γ' -phases, the periods of phase crystal lattices, phase transformation temperatures, stress rupture, thermophysical properties etc.) [11].

ALLOY DESIGNING

The searching of a composition for a new single crystal Ni-base superalloy was carried out on the basis of multicomponent system Ni-Al-Cr-Mo-W-Ta-Co-Re for which initial conditions of designing were formulated: Re concentration - 9...10 %, density of an alloy less than 9 g/cm³, a volume fraction γ' -phase 60...65 %, temperature of complete solution of γ' -phase in γ -solid solution (solvus γ') more than 1320°C, lack of freckles in the process of directional solidification of castings, "window" of thermal treatment more than 20°C, acceptable phase stability, etc. (total about 15 conditions).

The chosen alloying system does not contain in the composition such alloying elements as Ti, Nb, Hf widely used at development of Ni-base superalloys. It is basically caused by the following circumstances. First, Ti, Nb and Hf additives considerably decreasing in solidus temperature of Ni-base alloy raise homologous temperature and, hence, diffusion mobility of atoms of components in such alloy will be higher. Second, these alloying elements reduce temperature of eutectic $L \leftrightarrow \gamma + \gamma'$ (peritectic $L + \gamma \leftrightarrow \gamma'$) transformation and promote formation of nonequilibrium precipitations of eutectic (peritectic) phases at crystallization and thus make difficult complete homogenization of γ -solid solution without risk of fusion for axes dendrite spaces in single crystal casting. In the third, negative influence of Ti, Nb, Hf consists also in the fact that these elements, having significant solubility in γ' -phase, have unfavorable effect on dimensional misfit of the periods of crystal lattices for γ - and γ' - phases in Ni-base superalloy (misfit) and, hence, morphology of strengthening particles for γ' -phase.

The algorithm of computer search for a new alloy composition was consisted in the following. In the chosen alloying system the given concentration for each of 8 alloying elements was broken into three levels: minimal, average, maximal and made a design matrix of complete factorial experiment such as 2ⁿ type for eight variable factors.

Then an estimation of balanced chemical composition for all 2ⁿ variants of alloy was carried out that was determined by parameter ΔE [12]:

$$\left\{ \begin{array}{l} \Delta E = \bar{E}_{\text{alloy}} - \bar{E}_0 \\ \bar{E}_0 = 0,036\bar{A}_{\text{alloy}} + 6,28 \end{array} \right\} \quad (1)$$

where $\bar{A}_{\text{alloy}} = \sum_{i=1}^n A_i C_i$: average atomic mass of the alloy,

$\bar{E}_{\text{alloy}} = \sum_{i=1}^n E_i C_i$: average number of valence electrons in the

alloy components, A_i , E_i and C_i : accordingly atomic mass, number of valence electrons and atomic fraction of i-component in the alloy; n : number of components, including the alloy base without taking into account carbon, boron microadditions, rare-earth elements (La, Ce, Y, etc.) and impurity.

Parameter \bar{E}_0 for (1) is obtained by a method of regressive analysis of experimental data on phase structure for large group of Ni-base superalloys with various chemical compositions. For coordinates $\bar{E}_{\text{alloy}} - \bar{A}_{\text{alloy}}$ parameter \bar{E}_0 determines phase boundary of stable state for γ/γ' structure.

Usually commercial Ni-base superalloys have \bar{E}_{alloy} parameter value differed from \bar{E}_0 in value $\pm \Delta E$ named by alloying disbalance for the alloy. Value and sign ΔE determines the probability of phase transformation course with formation of undesirable phase of various type in γ/γ' structure of the alloy. In alloys with large negative alloying disbalance ($\Delta E < 0$) the probability of carbide formation such as M_6C or TCP phases is great; alloys where $\Delta E > 0$ have tendency to formation of phases such as Ni_3Ti (DO_{24}) and Ni_3Nb (A_3), and also eutectic (peritectic) phases on basis of Ni_3Al ($L1_2$); at $\Delta E = 0$ the alloy is considered as balanced on the chemical composition. The chemical compound of the alloy was considered balanced (i.e. the alloy had phase structure γ/γ') if $-0.04 \leq \Delta E \leq 0$ condition was fulfilled. Then, the balanced compositions of the alloy were estimated by New PHACOMP (Md) method [13].

With these purpose average values of parameter \bar{Md} for alloy γ/γ' and γ -matrix was determined by formula [13]:

$$\bar{Md} = \sum_{i=1}^n C_i (Md)_i \quad (2)$$

where C_i : atomic fraction of i-element in γ -solid solution or in the alloy, $(Md)_i$: tabular values of parameter (Md) for this element, n : number of elements, including an alloy base.

For the subsequent studies those alloy compositions were chosen where a phase stability condition was fulfilled: $\bar{Md}(\gamma/\gamma') < 0.975$ and $\bar{Md}(\gamma) < 0.907$.

For the chosen phase stable compositions of the alloy physical and chemical, structure-phase and high temperature strength characteristics were determined.

At that the major factor which determined a choice of the most perspective alloy composition among phase-stable compositions, was misfit. Parameter $\Delta a = (a_{\gamma'} - a_{\gamma})/a_{\gamma}$, (here a_{γ} and $a_{\gamma'}$: the periods of crystal lattices for γ - and γ' -phases) determined misfit. For a new alloy the misfit value should be positive ($a_{\gamma'} > a_{\gamma}$) and, at least, 2 times more, in comparison with this value for second and third generation single crystal Ni-base superalloy.

Other factors on the basis of which perspective alloy composition was chosen were temperature of solvus γ' -phase (T_{solvus}), temperature of eutectic transformation (incipient melting) (T_{eut}), number of γ' -phase in the alloy at temperature $\sim 850^\circ\text{C}$ (F_0) and density of the alloy (d) etc. (total about 15 conditions). Values of the above characteristics were determined by the following models [11]:

$$a_{\gamma'} = \{3.5219 + 0.00221(\text{Al}) + 0.00122(\text{Cr}) + 0.00412(\text{Mo}) + 0.00435(\text{W}) + 0.00693(\text{Ta}) + 0.00059(\text{Co}) + 0.00595(\text{Nb}) + 0.00302(\text{Ti}) + 0.00142(\text{V}) + 0.00382(\text{Re}) + 0.00303(\text{Ru}) + 0.01559(\text{Hf})\} 10^{-10}, [\text{m}] \quad (3)$$

$$a_{\gamma} = \{3.5691 + 0.00014(\text{Cr}) + 0.00097(\text{Mo}) + 0.00151(\text{W}) + 0.00398(\text{Ta}) - 0.00002(\text{Co}) + 0.00275(\text{Nb}) + 0.00149(\text{Ti}) - 0.00189(\text{V}) - 0.00504(\text{Re}) + 0.00083(\text{Ru}) + 0.01339(\text{Hf})\} 10^{-10}, [\text{m}] \quad (4)$$

$$F_0 = 14.7 + 0.54(\text{Al}) + 1.07(\text{Cr}) - 1.27(\text{Mo}) + 0.23(\text{W}) + 4.96(\text{Ta}) + 0.31(\text{Co}) + 2.54(\text{Nb}) + 3.81(\text{Ti}) - 0.75(\text{Re}) - 3.11(\text{V}) + 1.87(\text{Hf}) - 1.91(\text{C}) + 0.17(\text{Al})^2 - 0.08(\text{Cr})^2 [\%] \quad (5)$$

$$T_{\text{eut}} = 1358 - 0.18(\text{Al}) - 2.45(\text{Cr}) - 10.02(\text{Mo}) - 6.24(\text{W}) + 0.84(\text{Ta}) - 1.11(\text{Co}) - 9.55(\text{Nb}) - 10.34(\text{Ti}) - 5.41(\text{Re}) - 4.01(\text{V}) - 87.87(\text{Hf}) - 23.32(\text{C}), [^\circ\text{C}] \quad (6)$$

$$d = 0.144 \bar{A}_{\text{alloy}} \quad (7)$$

In formulas (3)-(7) symbols of chemical elements designate their concentration (atomic %) in phases and the alloy; \bar{A}_{alloy} : average atomic mass of alloy.

The alloy composition (hereinafter N1[®] alloy) was chosen for experimental studying. The chemical composition (wt. %) is given in table I; in table II the values of some characteristics of this alloy received in the designing are given.

Table I. Chemical composition for designed alloy

Ni	Al	Cr	Mo	W	Ta	Co	Re
base	5.75	2.5	2	1.3	8.8	11	9

RESULTS AND DISCUSSION OF EXPERIMENTS

OBTAINING OF SINGLE CRYSTALS

Experimental studying of designed alloy was carried out on single crystal samples with the axial orientation close to crystallographic direction $\langle 001 \rangle$. Single crystals obtained by LMC method (Liquid Metal Cooling) with refractory seeds in UVNK-9 furnace for directional solidification. To form an optimum microstructure of single crystals for the alloy with homogeneous distribution of cubical particles γ' -phases in γ -matrix the heat treatment including step homogenization in the range of temperatures 1285-1320 $^\circ\text{C}$ for 26 hours and double-stage ageing at temperatures 1130 $^\circ\text{C}/4$ hr and 870 $^\circ\text{C}/48$ hr was carried out.

MICROSTRUCTURE AND SEGREGATION COEFFICIENT

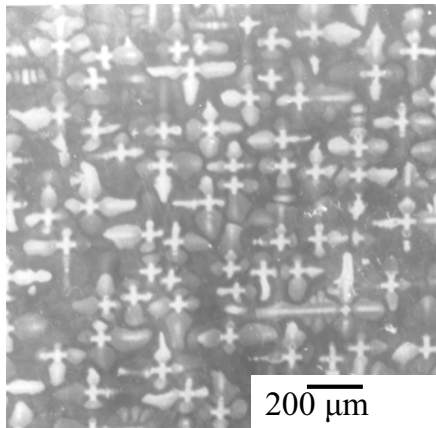
The obtained single crystals of high-rhenium Ni-base superalloy N1 had well-marked dendrite structure (Fig. 1a) after the directional solidification.

Nonequilibrium eutectic $\gamma + \gamma'$ (Fig. 1b) which volume fraction makes 5-7 % lies in interdendrite spaces. The size and morphology of γ' -phase particles essentially differ in dendrite

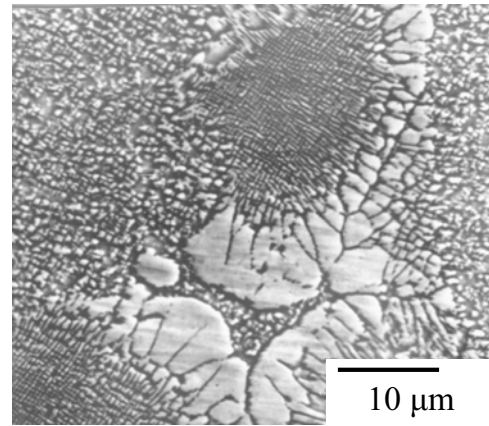
Table II. Parameters of structure, properties and characteristics of designed alloy

		Designed	Experimental
Physical and chemical characteristics:	$d, \text{g/cm}^3$	9.064	9.089
	$T_{\text{solvus}}, ^\circ\text{C}$	1325	1328
	$T_{\text{eut}}, ^\circ\text{C}$	1346	1330
	$T_s, ^\circ\text{C}$	1369	1359
	$T_L, ^\circ\text{C}$	1447	1436
Structure-phase characteristics:	$F_0, \%$	65.7	65.3
	$F_{\text{eut}}, \%$	5.5	7
	$\Delta a (20^\circ\text{C}), \%$	0.31	0.23
Phase stability parameters:	$(Md)_{\text{alloy}}$	0.973	-
	$(Md)_\gamma$	0.881	-
	ΔE	-0.038	-
Stress rupture:	$\sigma_{100}^{1000}, \text{MPa}$	321	330
	$\sigma_{1000}^{1000}, \text{MPa}$	209	215

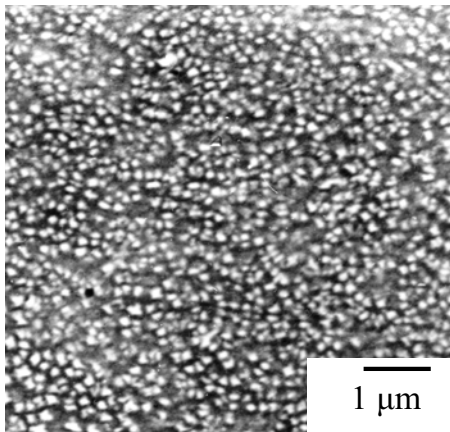
Note: d – density; T_{solvus} – solvus γ' ; T_{eut} – melting temperature of nonequilibrium eutectic ($\gamma+\gamma'$); T_s – solidus temperature; T_L – liquidus temperature; F_0 – number of dispersed γ' -phase; F_{eut} – number of nonequilibrium eutectic ($\gamma+\gamma'$); $\sigma_{100}^{1000}, \sigma_{1000}^{1000}$ – accordingly 100- and 1000-hours stress rupture at temperature 1000°C for single crystals with orientation $\langle 001 \rangle$.



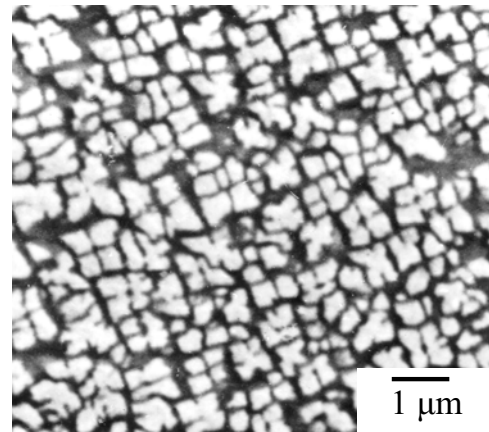
a)



b)



c)



d)

Figure 1. Microstructure of N1 alloy single crystal with orientation $\langle 001 \rangle$ (cross-section) after the directional solidification:

a – dendrite structure; b – eutectic $\gamma+\gamma'$ in interdendrite area;
c, d – γ' -phase particles in γ -matrix of dendrite (c) and in interdendrite area (d)

and interdendrite spaces (Fig. 1c, d). In the last ones the particles of γ' -phase are 3-5 times coarser than the particles in dendrite and have less strict facet. Dimensional and morphological heterogeneity of γ' -phase, presence of eutectic $\gamma+\gamma'$ is direct consequence of alloying elements microsegregation at the directional solidification of the alloy. For a quantitative estimation of chemical microheterogeneity segregation coefficients K_i were used. The segregation coefficients were determined by standard method:

$$K = \frac{C_{id}}{C_d} \text{ at } C_{id} > C_d \quad (8)$$

$$K = -\frac{C_d}{C_{id}} \text{ at } C_{id} < C_d \quad (9)$$

where C_{id} and C_d : local concentrations of alloying element in interdendrite areas and axes of dendrite respectively.

At such definition absolute value of the segregation coefficient is always >1 and is positive at straight segregation when the alloying element enriches interdendrite areas. In case of reverse segregation the alloying elements concentrate in dendrite axes and the factor has minus sign.

In table III values of alloying element concentrations in a material of axes areas and dendrite axes and designed by formulas (8) and (9) values of segregation coefficients in single crystals of N1 alloy after directional solidification are given.

One can see the most demonstrative elements with reverse segregation in cast alloy are rhenium $K_{Re} = -3.4$ and tungsten $K_W = -2.1$. Ta and Al has straight segregation $K_{Ta} = 2.0$ and $K_{Al} = 1.4$ respectively. The other alloying elements have minor segregation.

It is known, that chemical microheterogeneity of cast Ni-base superalloys predetermines temperature intervals of basic phase transformation at heating, which to γ' -phase solution in γ -solid solution, $\gamma+\gamma'$ eutectic melting and γ -solid solution melting are referred. Temperature values of the specified phase transformations for studied N1 alloy are given in table I. Based on these data the heat treatment mode for single crystals of the alloy, in particular homogenization temperature was chosen.

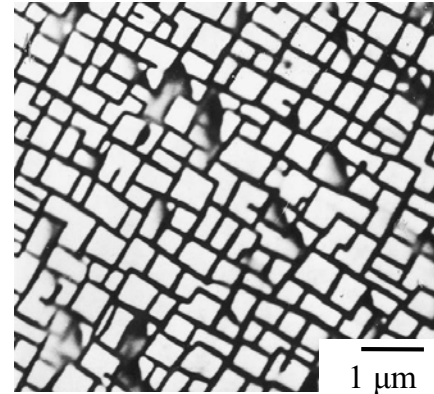
The N1 alloy single crystal microstructure after homogenization and double-stage ageing is given on Fig. 2.

At high-temperature homogenization of the alloy two processes proceed: in the beginning nonequilibrium eutectic $\gamma+\gamma'$ is solved and then diffusion balance of chemical composition in the limits of dendrite cells occurs.

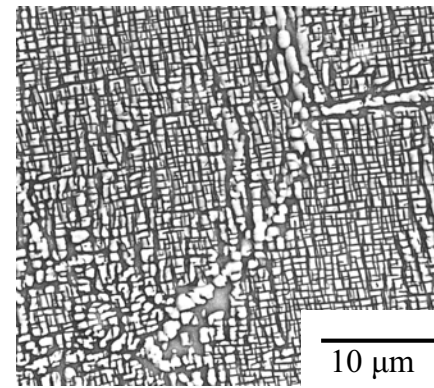
The formation of micropores in the size of up to 10 microns in

Phase irregular-shaped particles are formed on the subgrain boundaries between fragments of single crystal (Fig. 2b).

Segregation coefficients of Re and W reduced to -1.8 and -1.3 values respectively, and concentration of other alloying elements was completely balanced practically. Probably, it explains insignificant dimensional heterogeneity of γ' -phase in dendrite axes and interdendrite areas of the heat treated alloy single crystal.



a)



b)

Figure 2. Microstructure of N1 alloy single crystal after double-stage ageing:

a – shape and distribution of γ' -phase particles in matrix of γ -solid solution; b – decoration of low angle boundary with γ' -phase irregular-shaped particles

Table III. Local chemical composition and liquation factors in single crystals of N1 alloy after the directional solidification

Analysis site	Element content (wt. %) and segregation coefficients K_i							
	Cr	Mo	Al	W	Ta	Co	Ni	Re
Dendrite axes	2.7	1.7	3.9	1.5	5.3	11.5	57.7	17
Interdendrite area	2.2	1.4	5.4	0.7	10.5	10	65	5
K_i	-1.2	-1.2	1.4	-2.1	2	-1.2	-	-3.4

interdendrite areas is the effect of solving $\gamma+\gamma'$ eutectic. γ' -phase particles got the shape of quasi-cuboids and quasi-parallelepipeds at the subsequent double-stage ageing (Fig. 2a), the sizes of γ' -phase particles in interdendrite areas remain a little bit coarser than that of in dendrite axes, γ' -

STRUCTURE-PHASE CHARACTERISTICS

Results of X-ray single-crystal diffractometry method for the studied alloy after double-stage ageing are given on Fig. 3. In these experiments γ/γ' (222) Fe K_α – reflection and its division into γ -, γ' -phase singlet under program “Outset” is used]. Based on these results the periods of crystal lattices for γ' - and

γ -phases and misfit have been determined. The values are given in table 4.

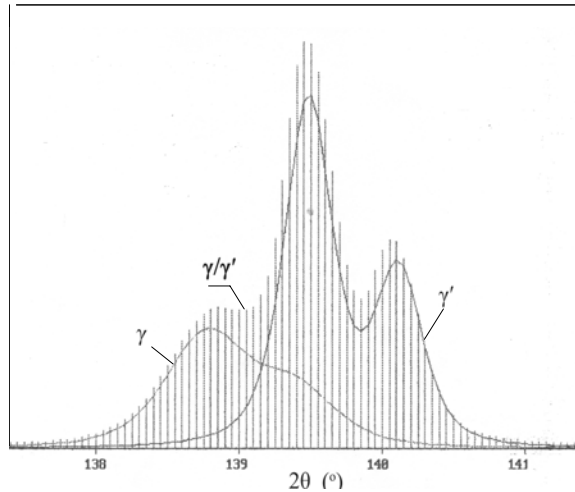


Figure 3. X-ray <001> single-crystal diffractometry for N1 alloy after double-stage ageing: total (222) $\text{FeK}\alpha$ - reflection γ/γ' and γ -, γ' - phase singlet

Table IV. Periods of crystal lattices for γ' - and γ -phases, misfit and number of γ' -phase in N1 alloy

a_{γ} , nanometer	$a_{\gamma'}$, nanometer	Δa , %	F_0 , %
0,35828	0,35745	0,23	65,3

Two conclusions based on data analysis given on Fig. 3 and in table IV follow. First, the studied alloy has high positive misfit ($a_{\gamma} > a_{\gamma'}$), equal to 0.23 % as heat treated condition. Apparently, primary solution of Re and Mo in γ -solid solution explains the fact. As atomic radiuses of Re and Mo are more than these of Ni and they concentrate basically in γ -phase, their interference results in increase in the period of a crystal lattice for this phase to a greater extent than that of γ' -phase, and, hence, causes increase in misfit. This circumstance favorably influences on high temperature strength of single crystals of Ni-base superalloys. Second, based on X-ray reflection intensity obtained separately from γ - and γ' -phases, to total γ/γ' reflex intensity ratio it is possible to estimate volume fractions of phases. It has been found that the volume fraction of γ' -phase in N1 alloy as heat treatment condition makes 65.3 %.

CREEP RUPTURE STRENGTH

Experimental definition of creep rupture strength for N1 alloy was carried out at temperatures of 900, 1000 and 1100°C (test period above 1000 hours) on single crystal specimens (total length 70 mm, designed length of a working part 25 mm at diameter 5 mm) with crystallographic orientation <001> (within the limits of the tolerance 10°).

Statistic result manipulation of creep rupture strength tests was carried out as per equation [14]:

$$\tau = \xi T^m \sigma^{-n} \exp\left(\frac{U_0 - \eta\sigma}{RT}\right) \quad (10)$$

where τ – creep-rupture life, hour; σ – stress, MPa; T – temperature, K; ξ , m , n , U_0 , η – coefficients determined based on results of stress rupture tests (U_0 and η – activation energy

And activation volume of stress rupture process respectively; $\ln \xi$ – value proportional to entropy member); R – universal gas constant.

Digital values of coefficients for equation (10) are given in table V.

Table V. Equation coefficients for stress rupture of N1 alloy

Temperature interval of tests, K	m	n	$\ln \xi$	U_0 , kJ/mol	η , J/(mol·MPa)
1173-1273	2	4	-29.85	475.7	48.5
1273-1373	2	3	-37.36	508.2	93.32

Stress rupture curves (average values) for temperatures of 900, 1000 and 1100°C shown on Fig. 4 (points show experimental data) were designed using the values of factors for the equation of stress rupture (10).

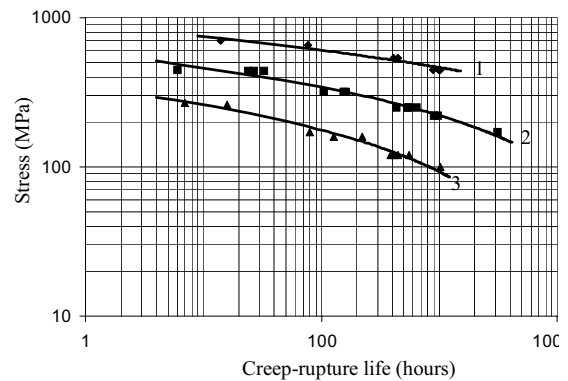


Figure 4. Stress rupture curves for N1 alloy single crystal with orientation <001> at temperatures of 900 °C (1), 1000 °C (2) and 1100 °C (3)

Average values of limits for stress rupture of N1 alloy (in comparison with characteristics of alloys EPM-102 and CMSX-10 are given in table V.

Table V. Stress rupture for Re-bearing single crystal Ni-base superalloys

Alloy	T , °C	σ_{100}	σ_{500}	σ_{1000}
		MPa		
N1 (9 % Re)	900	585	490	450
	1000	330	250	215
	1100	165	115	95
EPM-102* (6 % Re, 3 % Ru)	900	503	420	385
	1000	325	235	200
	1100	148	-	-
CMSX-10* (6% Re)	900	530	430	400
	1000	290	215	185
	1100	150	-	-

*We got values of stress rupture for alloys EPM-102 and CMSX-10 by processing particular value of rupture life at various temperatures and stress taken from papers [7, 15].

These results testify to advantage of N1 alloy in comparison with third and fourth generations single crystal nickel-base superalloys EPM-102 and CMSX-10 throughout temperature-time range.

EVOLUTION OF A MICROSTRUCTURE AT CREEPING

Test results of structure studies for materials ruptured by high temperature creep test of single crystal specimens from N1 alloy are given on Fig. 5 and 6. So-called raft-microstructure typical for single crystals of Ni-base superalloys with the orientation $\langle 001 \rangle$ subjected to constant tension at high temperatures was observed in the alloy structure at all test modes.

During a transient stage of high-temperature creeping the initial cubical particles of γ' -phase coagulate specifically by means of joining in plates which wide facets are perpendicular to axes of tension applying. Sections of these plates as consistently alternating layers γ - and γ' -phases (accordingly dark and light layers on Fig. 5a) will be projected on vertical facets (100) and (010). Such microstructure remains its regularity throughout stationary stage of specimen creeping. The regular raft-microstructure shows significant resistance to plastic creep strain as it interferes with crossing of γ' -phase plates by dislocations that move in layers of γ -solid solution. In this sense any regularity disturbance of raft-microstructure will result in softening alloy and decreasing in stress rupture. The greatest changes in alloy microstructure during creeping are observed in the material bulk close to ruptured zone of the specimen.

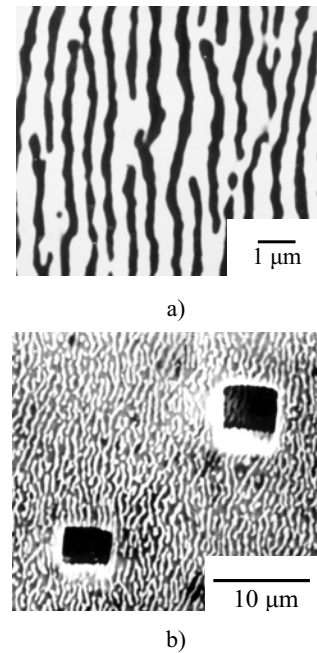


Figure 5. Raft-microstructure (a) and deformation pores (b) formed at high temperature creep for single crystal of N1 superalloy: 1000°C, $\tau=1361$ hr

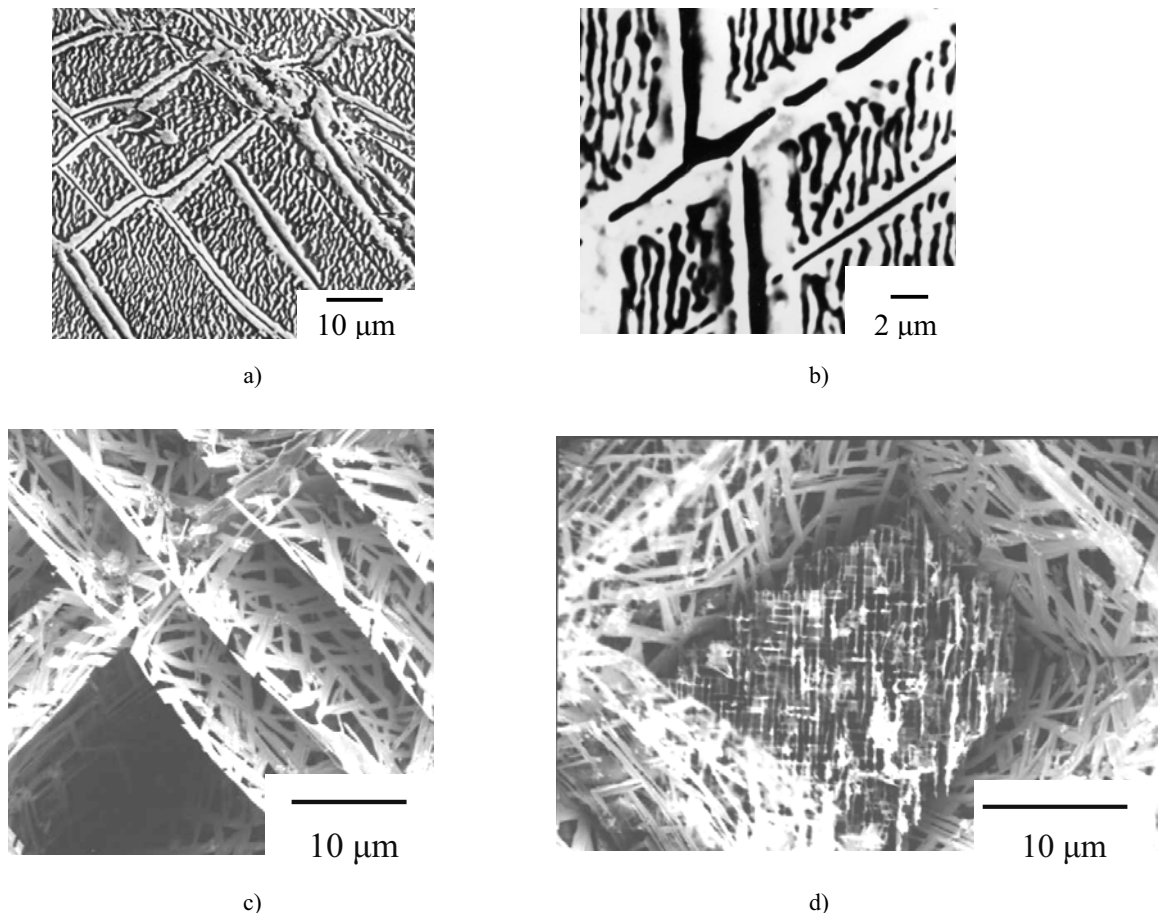


Fig.6. High rhenium FCC phase in single crystals of N1 superalloy formed at prolonged creep rupture at 1100°C ($\tau=559$ h) (a) and 1000°C ($\tau=2591$ h) (b...d): b, c, d – after deep chemical etching of specimen surface (G.I. Morozova)

Here the raft-microstructure undergoes topological inversion, i.e. γ' -phase plates jointed during creeping form one-coherent matrix and earlier one-coherent matrix γ -solid solution forms layers isolated from each other. It is determined that micropores with crystallographic facet (deformation pores) are available in the material of ruptured single crystals (Fig. 5b) which number and size increases as approaching ruptured zone of specimen.

After creeping at temperatures of 1000 °C for ~2600 hours and 1100 °C for ~500 hours lamellar precipitations of a new phase (dark thin plates in light envelope of γ' -phase) (Fig. 6a, b), penetrating practically throughout specimen material bulk are found in the alloy structure. X-ray structure analysis of the alloy with such microstructure has shown that the lamellar phase has face-centered cubic lattice (structural type A1) with the period of 0,361 nanometers whereas the periods of lattices for matrix γ' - and γ - phases are within the limits of 0,358-0,359 nanometers; Re content in lamellar phase makes 20 % (atomic). One can see on Fig. 6b where the surface microstructure of the single crystal specimen after deep chemical etching is shown, high rhenium FCC phase in the alloy structure at prolonged high temperature tests got a “woven” structure.

Let's consider influence high rhenium FCC phase on mechanical properties of N1 alloy. Adverse influence of classical TCP phases on mechanical properties of Ni-base superalloys, in particular, resulting in premature initiation of microcracks and embrittlement is known. However in case of single crystals for high rhenium N1 alloy it has been found experimentally that availability of lamellar precipitations of a new phase has low influence on its ductile characteristics. Results of studies of mechanical properties and microstructure N1 alloy in various conditions: after double-stage ageing and prolonged annealing at 1000°C given in tables VI...VIII and on Fig.7 testify to this fact.

Table VI. Stress rupture and ductility of N1 alloy after double-stage ageing

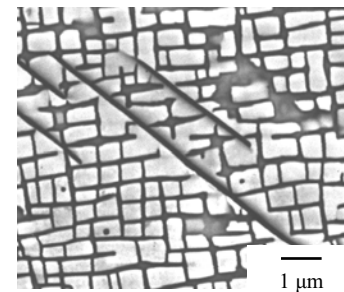
T, °C	Tension, MPa	Creep-rupture life, hour	Stress rupture ductility, %
1000	320	154.5	27
1000	250	593	22
1000	200	1361	17
1000	170	3119	20
1000	130	2591*	-
1100	170	112.5	19
1100	120	475	23

*Specimen is taken off from tests.

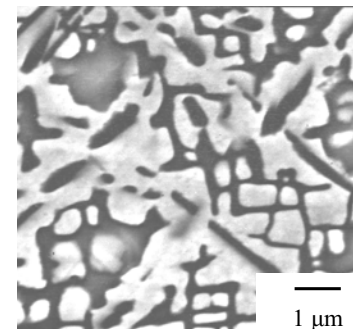
Table VII. Tensile properties (20 °C) for N1 alloy after double-stage ageing and annealing at 1000 °C

Heat treatment	Conventional yield strength, MPa	Ultimate strength, MPa	Relative elongation, %
Double-stage ageing	945	1190	21
Annealing: 500 hours	910	1170	19

The analysis of the experimental data obtained shows that despite of formation in N1 alloy material of a plenty lamellar face-centered cubic phase, value of ductility of alloy single crystals at a short-term tension, their service life and stress rupture ductility at creep test are kept at high enough level (tables VI...VIII). Hence, lamellar high rhenium phase of structural type A1 does not result in embrittlement of single crystals of N1 Ni-base superalloy. However, as one can see in table 8 that at stress rupture test service life of the alloy after annealing at 1000 °C for 500 hours is considerably less, than that of after double-stage ageing (table VI). Comparison of evolution of phase structure and γ/γ' -microstructure in single crystals of alloy during prolonged high-temperature annealing (Fig. 7) with results of stress rupture tests for these single crystals (table VIII) has shown that the reason of reduction of their service life is the roughening and degradation of particles of the basic strengthening of γ' -phase.



a)



b)

Figure 7. N1 superalloy microstructure after annealing at temperature 1000°C: a – 200 h; b – 500 h

Table VIII. Stress rupture and ductility for N1 alloy after annealing at temperature 1000°C

T, °C	Tension, MPa	Annealing 200 h		Annealing 500 h	
		Stress-rupture life, h	Stress rupture ductility, %	Stress-rupture life, h	Stress rupture ductility, %
900	580	100	20.5	56	22
1000	320	106.5	26	17	26.5
1100	170	95	16	-	-

CONCLUSIONS

1. On the basis of phase structure designing, physical and chemical and structure - phase characteristics of nickel-base alloys for system Ni-Al-Cr-Mo-W-Ta-Co-Re we designed the new single crystal superalloy containing 9 % Re. To improve phase stability the total contents of refractory (Re, Mo, Ta, W) and γ' -formed (Al, Ta) elements in the alloy was balanced.
2. Developed high rhenium VJZM-1 alloy has advantage on stress rupture in the field of working temperatures and service life over third and fourth generations CMSX-10 and EPM-102 single crystal Ni-base superalloys.
3. At prolonged heating or creeping in temperature range of 1000-1100 °C lamellar precipitations of face-centered cubic phase enriched with Re and having the period of crystal lattice equal to 0,361 nanometers are formed. It has been founded that this phase does not negative influence on stress rupture ductility of the alloy single crystals.

ACKNOWLEDGEMENTS

Authors thank dr. G.I. Morozova, dr. A.I. Samoylov and dr. V.V. Gerasimov for their help and fruitful discussion during fulfillment of the work.

REFERENCES

1. G.L. Erickson and K. Harris, "DS and SX superalloys for industrial gas turbines", Materials for Advanced Engineering: Proc. of Conf. Part II, Liege (Belgium), 3-4 Oct. 1994, 1055-1074.
2. G.L. Erickson, "The development and application of CMSX-10", in Superalloys 1996, ed. R.D. Kissinger et al. Publ. of the Minerals, Metals & Materials Society. Seven Springs Mountain Resort, Champion (Pennsylvania), 1996, 35-43.
3. P. Caron and T. Khan, Third generation superalloys for single crystal blades", High Temp. Mater. for Power Engineering: Proc. Conf. Part II, Liege (Belgium). Germany: Forschungszentrum Jülich GmbH, 1998, 897-912.
4. H. Hino, Yoshioka Y., Nagata K. et al., "Design of high Re containing single crystal superalloys for industrial gas turbines. Material for Advanced Power Engineering", High Temperature Materials for Power Engineering 1998: Proc. of Conf. Part II, Liege (Belgium). Germany: Forschungszentrum, Jülich GmbH, 1998, 1129-1137.
5. E.N. Kablov and N.V. Petrushin, "Physicochemical and Technological Features of Creating Metal-Based High-Superalloys", *Pure Appl. Chem.*, 76, N.9 (2004), 1679-1689.
6. P. Caron, "High γ' solvus new generation nickel-based superalloys for single crystal turbine blade applications", in Superalloys 2000, ed. T.M. Pollock et al., Champion (Pennsylvania): Publ. of the Minerals, Metals & Materials Society. Seven Springs Mountain Resort, 2000, 737-746.
7. S. Walston, A. Cetel, R. MacKay et al., "Joint development of a fourth generation single crystal superalloy", in Superalloy 2004, ed. K.A. Green et al., Champion (Pennsylvania): Publ. of the Minerals, Metals & Materials Society. Seven Springs Mountain Resort, 2004, 15-24.
8. Y. Koizumi, T. Kobayashi, T. Yokokawa et al., "Development of next-generation Ni-base single crystal superalloys", in Superalloys 2004, ed. K.A. Green et al., Champion (Pennsylvania): Publ. of the Minerals, Metals & Materials Society. Seven Springs Mountain Resort, 2004, 35-43.
9. K.S. O'Hara, W.S. Walston, E.W. Ross and Darolia R., "Nickel base superalloy and article", US Patent 5482789, January 9, 1996.
10. Chong Long Fu, Roger Reed, Anderson Janotti and Maja Kremer, "On the diffusion of alloying elements in the nickel-base superalloys", in Superalloys 2004, ed. K.A. Green et al. Champion (Pennsylvania): Publ. of the Minerals, Metals & Materials Society. Seven Springs Mountain Resort, 2004, 867-876.
11. E.N. Kablov and N.V. Petrushin, "Computer method of designing casting nickel-base superalloys", in Casting Superalloys: Kishkin's Effect (Moscow, Nauka), 2006, 172-184.
12. G.I. Morozova, "Formation of the chemical composition of the γ'/γ matrix of multicomponent nickel alloys", *Dokl. Akad. Nauk SSSR*, 320, N6 (1991), 1413-1416.
13. N. Yukawa, M. Morinaga, H. Ezaki and Y. Murata, "Alloy design of superalloys by the d-electrons concept", High Temp. Alloys for Gas Turbines and Other Applications. Proc. of Conf. Liege (Belgium, 6-9 October 1986). Dordrecht: C.R.M., 1986, 935-944.
14. E.N. Kablov and E.R. Golubovsky, High-Temperature Strength of Nickel Alloys, (Moscow, Mashinostroenie) 1998, 464.
15. G.L. Erickson, "Single crystal nickel-based superalloy – CMSX-10", US Pat. 5366696. Nov. 22, 1994.

