



Development of Aero-Space Structural Ni₃Al-based Alloys for Service at Temperature above 1000⁰C in Air without Protection Coating

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Summary:

The principles of alloying are developed for alloys based on the γ' phase Ni₃Al and realized for the design of a high-temperature alloy VKNA-1V destined for a wide range of "hot" GTE articles (e.g., flaps, nozzle vanes, turbine rotor blades, elements of flame tubes, and other complex thin-wall articles) produced by vacuum investment casting. Owing to a fortunate combination of the selected boron-free alloying system (Ni-Al-Cr-W-Mo-Zr-C), the presence of a ductile structure constituent such as nickel-based γ solid solution (~10 wt%) and directed columnar or single crystal structure the alloy is characterized by high ductility at room (EI=14-35 %), middle and high temperatures (EI=18-31 % at 673-1473K), by a melting temperature (solidus) as high as $T_m = 1613\text{K}$, a density of at most 7930 kg/m^3 , high short term and long term strength at temperatures 1273-1573K ($\sigma_{100}=110 \text{ MPa}$ at 1373 K). Alloy has a high oxidation resistance at temperatures up to 1573 K and is resistant to stress corrosion and general atmospheric corrosion. New VKNA-1V Ni₃Al-based alloy with equiaxed grained, directional solidification (DS), or single-crystal structures can be produced by conventional cast processes used for investment casting of nickel superalloys, including the process of high-gradient DS. Compared to nickel analogs, the alloy is relatively cheap and do not need in protective coating up to 1573K in air.

Keywords:

Structure alloy, γ Ni₃Al, principles of alloying, mechanical properties, oxidation resistance, direct solidification, single crystal structure

1. Introduction:

Alloys based on γ phase Ni_3Al (γ is the main strengthening phase of the $(\gamma+\gamma')$ nickel superalloys) are attractive as structural materials for the extensive production of hot gas turbine engines (GTE) articles such as noncooled turbine blades, combustion chamber parts, nozzle vanes, flaps, and other complex-shape thin-wall products intended for working temperatures exceeding those of nickel superalloys (1350K) (1-4). Compared to nickel superalloys, the Ni_3Al alloys are lighter and more oxidation-resistant owing to an increased aluminum content (14-24 at.%), retain an ordered fcc structure ($L1_2$ type) up to the melting temperature ($\sim 1670\text{K}$). Ni_3Al is characterized by a potential resource of low-temperature ductility (owing to more than five independent slip systems) (1-5). An additional advantage of Ni_3Al is that the semiproducts and articles of complex shape may be produced from Ni_3Al alloys using a well-developed technology of the production and treatment of nickel superalloys.

2. Reasoning of the Selection of the Ni_3Al -Based Alloy Compositions:

The well known present-day Ni_3Al -based alloys type of IC 221M, 396M, IC-6 et. al. contain 0,005-0,03 wt% (0,02-0,16 at.%) boron because, according to the current concepts, the microalloying with boron allows one to elevate the Ni_3Al RT ductility, increases cohesion and facilitates the transmission of gliding through the grain boundaries (5,6). In our opinion, a disadvantage of the Ni_3Al -based alloys with boron is a probability of a decrease in the solidus temperature because of the formation of boron-containing eutectic and a tendency to hot brittleness (a decrease in ductility at 673-1123K in air and in humid atmospheres) because of the oxygen penetration into the grain boundaries enriched with boron. Therefore, the design of Ni_3Al -based alloys in our works was aimed at a virtually complete exclusion of boron (7-9). We designed Ni_3Al -based alloys using only the formation of a ductile structure constituent such as the Ni-based γ solid solution to increase the low-temperature ductility and fracture toughness. Chromium providing the formation of self-healing chromium-oxide film is introduced in many γ - Ni_3Al alloys as an additional protection against hot brittleness (1,3-5,7-11).

Strengthening of the Ni_3Al -based solid solution by alloying is determined by several factors that may be summarized as: the higher is the Ni_3Al lattice distortion caused by the difference in the atomic sizes and electron structures of alloying element (AE) and substituted metal (Ni or Al) and the higher is the AE content, the larger is the solid-solution strengthening of Ni_3Al at low and

medium temperatures (3,5). At temperatures $\geq 0.6T_m$, the improvements in strength, durability, and creep resistance of the both γ' alloys (as the nickel $\gamma+\gamma'$ superalloys) are substantially caused by the deceleration of diffusional processes due to the alloying with the most "slow" refractory metals (W, Mo, Ta, Nb, and Hf), whose contents are limited by their solubility, danger of the TCP-phase formation, and increasing density of the alloy. However, the solid-solution strengthening in any case is insufficient for the aluminide competitiveness with nickel superalloys in the temperature range of precipitation hardening and dispersion strengthening effect ($\gamma'+\gamma$) (2-5).

For the strengthening of heterophase Ni₃Al-based alloys, the particles of the nickel-based γ solid solution are generally selected from all possible second phases that may exist in equilibrium with γ' -Ni₃Al in multicomponent systems. Owing to the presence of the $\gamma+\gamma'$ eutectic in the multicomponent system Ni-Al-Cr-W(Mo)-Ti(Zr,Hf), that was a base for development of Ni₃Al -structural alloy the ductile γ particles may uniformly precipitate in the γ phase upon the solidification of the alloys approximately consisting of 90 vol % $\gamma'+10$ vol % γ . Owing to strong temperature dependence of the γ' solubility in the γ phase, this γ -phase may be additionally strengthened by fine secondary γ' -phase particles precipitating upon heat treatment or upon high temperature work of the material (dynamic dispersion strengthening). This allows us to rise strength of Ni₃Al alloys at 293 – 1273K.

The presence of metallographic and crystallographic textures and a decrease in the extension of transverse grain boundaries in the heterophase Ni₃Al-based alloys substantially increase the long-term strength, creep resistance, and durability of the alloys.

With allowance for the above reasons, we designed a Ni₃Al-based alloy VKNA-1V destined for cast shaped hot-circuit GTE articles having directional or single-crystal structure and operating under general climatic conditions at 1273 – 1473 K with short-term (≤ 10 h) overheatings to 1573 K (3,7-9,12,13).

3. Experimental procedure:

The casting technology of the VKNA-1V alloy articles including those with single-crystal structure is the same as that used for the current ZhS-type Russian nickel superalloys and requires the same equipment and accessory (ceramic molds, cores, etc.) (8). This is generally caused by the closeness of their heat-conductivity coefficients λ , specific heat capacity C, and linear expansion coefficient α in the entire temperature range between room temperature and the maximum operation temperature as well as by their

similar characteristics of linear shrinkage and by the absence of crack-formation ability upon casting in electrocorundum molds with damping layers. For example, the characteristics of the VKNA-1V SC alloy with a single-crystal (SC) structure in the temperature range between 293 and 1473 K change as follows: λ from 8.39 to 27.7 W/m²*K, C from 0.472 to 0.858 kJ/kg*K, $\alpha \cdot 10^6$ from 12.3 to 14.5 1/K. The linear shrinkage is 1,5-2%. The solidus temperature upon heating is ~1600 K, and the solidification range is 80 K (8). The basic chemical composition of VKNA – 1V (wt. %): 76.9 Ni; 8.6 Al; 5.7 Cr; 3.5 W 3.2 Mo; 1.6 Ti; 0.5 Zr(Hf).

The alloy contains the following impurities (wt. %) 0.005 S, 0.005 P, 0.001 Pb, 0.0005 Bi, 0.003 Sn, and 0.003 Sb. We studied the cast ingots directionally solidified (DS) at a rate of 20-40 mm/min with oriented columnar or SC structures with the <001>, <011>, and <111> crystallographic orientations. The deviations of these orientations did not exceed 10⁰. The cast ingots of 55 mm in diameter with equiaxed grains were extruded into rods of 16-18 mm.

The mechanical properties were determined by tension tests of the specimens with a length-to-diameter ratio of five at 293-1623 K by conventional methods. At least three specimens were taken for each point of short-term tests and ten specimens for each point of long-term strength.

4. Results and Discussion:

4.1. Structure.

The following four types of structure were observed in the specimens obtained: (1) fine-grained structure (equiaxed polyhedral grains) typical of the deformed and recrystallized material; (2) coarse-grained equiaxed structure (equiaxed dendrites) typical of "equiaxed" ingots; (3) the structure consisting of elongate grains (columnar dendrites) typical of DS alloys; (4) single-crystal structure that is also typical of DS alloys.

Both polycrystalline alloys with equiaxed grains (Fig. 1a) and the DS alloys including single-crystals (Fig. 1b-d) contain primary γ' -phase light particles of an irregular shape ("lilies"). In some alloy modifications, these particles contain globular inclusions of refractory MC carbides; nonequilibrium β -NiAl globular inclusions can also present in the γ' lilies, confirming the occurrence of the peritectic transformation $L+\beta \leftrightarrow \gamma'$ in this high-alloy material (see also Fig.4). The primary lily like γ' phase particles in the cast material are form rows or chains in the solidification direction between the columnar dendrites. It is

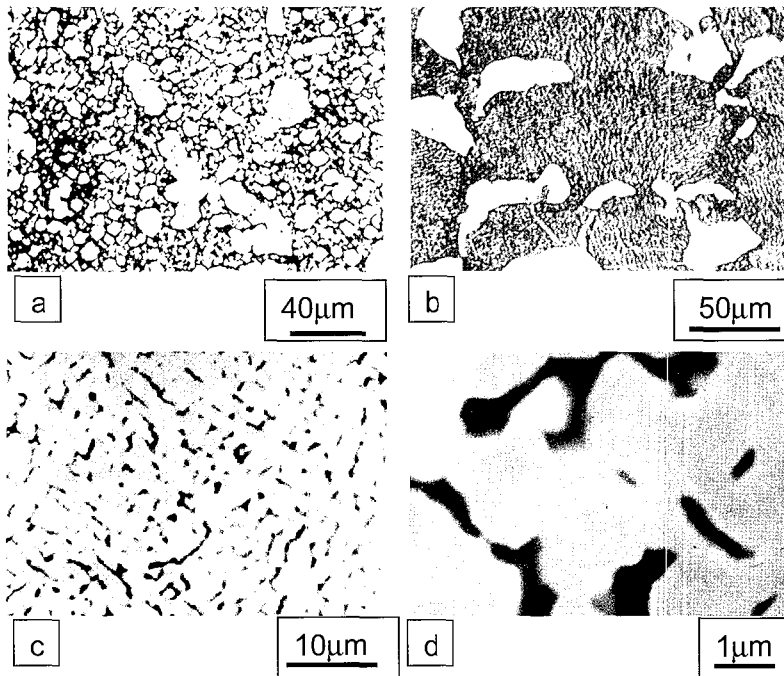


Fig. 1. Microstructure of the VKNA-1V (a) cast in vacuum into ceramic molds and (b), (c), (d) directionally solidified $\langle 111 \rangle$ single-crystal.

likely that in this multicomponent alloys the dendrites are of the eutectic ($L \leftrightarrow \gamma' + \gamma$) origin and are based on the γ' phase, in which isolated γ -phase inclusions $\leq 0.5\text{-}3 \mu\text{m}$ thick and $\leq 5\text{-}12 \mu\text{m}$ long are present (Fig. 1c,d). Some heat-treatment or operating conditions cause the decomposition the supersaturated γ solid solution. This results in the formation of fine ($0.1\text{-}0.5 \mu\text{m}$) secondary γ' precipitates inside the isolated γ -phase particles. Such γ structures with the secondary cubic γ'_{sec} precipitates are typical of the Ni-based superalloys (see also Fig. 4).

4.2. Mechanical Properties

The maximum RT strength values (UTS=1300-1520 MPa at El=17-37%) are characteristic of the alloy specimens with the deformed, recrystallized, and single-crystal structures. The minimum values of the RT relative elongation (El=7-10% after hot pressing at UTS=570-670 MPa) are characteristic of the alloy cast in ceramic molds and having equiaxed grain structure. The

formation of directional columnar or single-crystal macrostructure with a minimum extension of transverse grain boundaries or without such boundaries at all increases the high-temperature service life by an orders of magnitude at: $\tau = 0.2 - 4$ h of $\sigma = 40$ MPa for to the material in the recrystallized and, especially, deformed states, to $\tau = 12 - 26$ h of $\sigma = 50$ MPa for the material with the equiaxed structure and to $\tau = 130 - 150$ h of $\sigma = 100$ MPa for material with directional columnar or single-crystal macrostructure. It is seen that the optimal combination of RT short-term strength and ductility and the long-term strength at 1173-1373 K are characteristics of the alloy specimens with the single-crystal or dendritic columnar structures. For this reason, we used only the alloys with these two types of structures for the further studies.

The mechanical properties of the of the DS alloy VKNA-1V with the dendritic columnar structure and the $\langle 111 \rangle$ single-crystal structure were determined by short-term tests in air at 20-1473 K (Fig. 2).

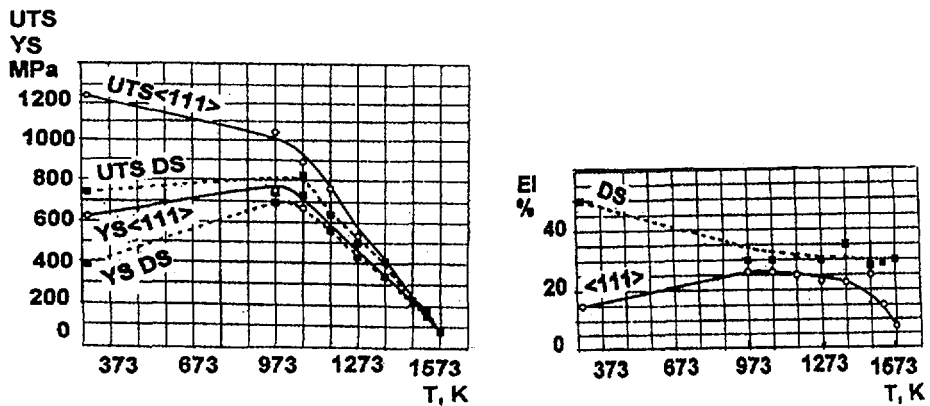


Fig. 2. Ultimate tensile strength, yield strength and relative elongation of the DS VKNA-1V alloy with single-crystal $\langle 111 \rangle$ and oriented columnar dendritic structures (DS) as a function of temperature.

Note that the ductility characteristics of both DS materials are weakly affected by increasing temperature, and the medium-temperature ($\sim 873 - 973$ K) ductility dip (inevitable for the boron-containing alloys) is virtually absent. The data on the long-term strength of the VKNA-1V alloy with the columnar dendritic and single-crystal structures based on 10, 100, and 500 h

given in Fig. 3. As it is seen from Fig. 2 and 3, the advantages of the specimens with the single-crystal structure over those with the columnar dendritic structure at temperatures above 1473 K ($>0.9 T_m$) are not so apparent as at medium temperatures. In any case, the data obtained indicate that the alloy is applicable for up to 500-1000 h at temperatures up to 1473 K and can undergo short-term (up to 10-100 h) small loads (10-13 MPa) at 1523-1573 K.

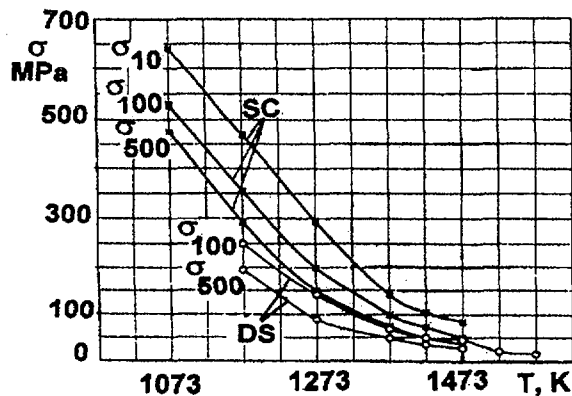


Fig. 3. Long-term strength based on 10, 100 and 500 h of the DS VKNA-1V alloy with single-crystal (SC) and oriented columnar dendritic (DS) structure as a function of temperature.

The effect of crystallographic orientation on the mechanical properties of the VKNA-1V single crystals at room temperature is given in Table 1. The yield strength and ultimate tensile strength of the $\langle 111 \rangle$ single crystal are higher than those of the $\langle 100 \rangle$ and $\langle 110 \rangle$ single crystals. This effect is smaller at 1073-1373 K. An anomalous temperature dependence of the yield strength is retained regardless of the crystallographic orientation. The single crystals with the $\langle 001 \rangle$ and $\langle 011 \rangle$ orientations exhibit a decrease in ductility at ~ 1073 K. A sharp increase in ductility of the $\langle 011 \rangle$ single crystals at high temperatures of 1273-1373 K undesirable for structural materials. The $\langle 111 \rangle$ single crystals are characterized by the minimum temperature dependence of ductility in the range 293 - 1373 K.

For a rough estimation of ability to strain hardening, we may use the ratio of the difference between ultimate tensile strength and yield strength to the total relative elongation (Table 1): the ability to strain hardening maximum for the $\langle 111 \rangle$ single crystals, which thus demonstrate a higher serviceability under

the conditions of extreme loading. The long-term tests on the bases of 100 and 500 h exhibit that the strength of the <111> single crystals is higher than that of the <001> and, especially, <011> single crystals (Table 1).

Tabl 1. Mechanical properties of the VKNA-1V single-crystals of different crystallographic orientations.

Crystal orientation	T, K	YS, MPa	UTS, MPa	δ , %	$\frac{UTS - YS}{\delta}$	σ_{10} , MPa	σ_{100} , MPa	σ_{500} , MPa
					MPa/%			
<001>	293	330	550	55	4.00	—	—	—
	1073	760	840	14	5.70	570	480	430
	1273	540	550	44	0.22	270	150	94
	1373	430	440	31	0.32	140	90	62
<011>	293	400	670	29	9.30	—	—	—
	1073	1000	1030	12	2.30	580	500	430
	1273	520	540	49	0.40	240	130	83
	1373	360	410	54	0.78	120	72	50
<111>	293	620	1350	14	52.00	—	—	—
	773	800	1450	22	29.50	—	—	—
	1073	670	890	26	8.50	630	530	470
	1273	430	520	30	3.00	290	200	150
	1373	340	410	22	3.18	140	100	70

4.3. Effect of Long-Term Heat Treatment on the Structure and Properties of the Cast VKNA-1V Alloy

We studied the effects of the annealing of single-crystal VKNA-1V samples at 1543 K for 5 and 30 h (air and water cooling) on the structure and mechanical properties and also the stability of structure and mechanical properties of the single-crystal specimens after long-term (250 and 500 h) annealings at 1523 K under the conditions simulating the possible service conditions including overheatings. It shown that neither long-term annealings at 1523 K in air nor rapid cooling (water quenching from 1523 K) do not virtually affect the mechanical properties of the alloy: as-cast and heat-treated samples have UTS=1300-1400 MPa, EI=11-18%. These data display a high thermal stability of the single-crystal structure with the <111> orientation (Fig. 1).

However, the structure of the material substantially changes upon tests at high temperatures in a stressed state. This is evident from the comparison

between the $\langle 111 \rangle$ single-crystal microstructures before (Fig. 1) and after tests at 1373 K at $\sigma=100$ MPa for 257 h (Fig. 4). Already after the resource depletion by 50%, the boundaries of primary coarse γ' -phase precipitates ("lilies") observed between the dendrites in cast single-crystal specimens

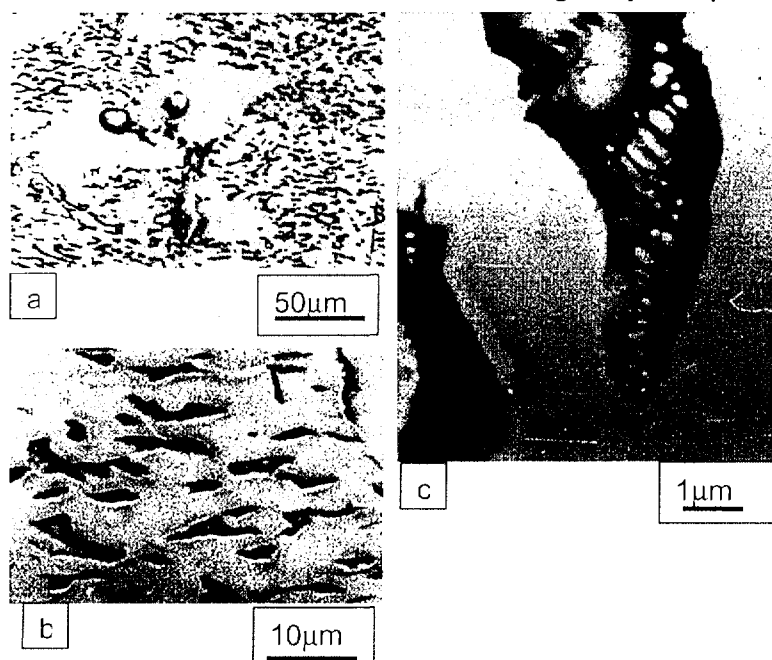


Fig. 4. Microstructure of the VKNA-1V alloy with the $\langle 111 \rangle$ single-crystal structure after test for long-term strength at 1373 K ($\sigma = 100$ MPa, $\tau = 257$ h).

become more diffused because of the equalization of the composition of the primary and eutectic γ' -phase precipitates (Fig. 4a). Inside the γ' -dendrites, the isolated γ -solid solution inclusions of eutectic origin containing the secondary fine γ' -phase (γ'_{sec}) particles become coarser (the γ -particles reach 2-4 μm in transverse size and ~ 10 -15 μm in length) and are oriented along the loading direction (Fig. 4b). The secondary fine γ' -phase precipitates inside the γ -phase particles also become coarser (Fig. 4c).

We emphasize the difference between the Ni_3Al VKNA-1V alloy and conventional nickel superalloys in strengthening mechanisms.

The $(\gamma + \gamma'_{\text{sec}})$ structure, providing the extremely high strength of the well-known present-day Ni-based superalloys of ZhS (Russia) or CMSX2, Rene 4

and PWA1480 (USA) types at the temperatures of age hardening (up to 1273 K), losses stability at higher temperatures because of the coarsening of fine γ'_{sec} particles, their dissolution in the matrix, and destruction of in their coherency with the matrix. The VKNA-1V alloy ranks below the Ni-based superalloys in strength up to 1173 – 1273 K (the operating temperatures of blades and discs of gas-turbine engines are 993 – 1173 K) because in VKNA-1V the strengthening γ -phase particles in the γ' matrix are too coarse and occupy at most 10 vol %; in its own turn, their strengthening by the secondary precipitates γ'_{sec} does not substantially contribute the alloy strength as is in the case of the Ni superalloys. However the VKNA-1V alloy surpasses the nickel superalloys in strength at temperatures above $0.8 T_m$ and up to subsolidus temperatures (1573 K) due to the high thermal stability of the structure consisting of ($\gamma' + \gamma$) eutectic with an excess of the primary γ' Ni₃Al inclusions. This structure provides a constancy of the phase composition and the stability of sizes of structure constituents, whereas alloying of both solid solutions (γ' matrix and γ particles) with refractory elements (W, Mo, and Hf) decelerating diffusion processes in the grains and at interphase boundaries allows one to retain the high-strength state of the γ' Ni₃Al alloy up to subsolidus temperatures, at which the age hardening mechanism is ineffective.

4.4. Corrosion Resistance of the VKNA-1V Alloy

The tests in air under general environmental conditions were performed by two methods. We determined the gain in weight for 100 h at the expense of the formation of Al₂O₃ based oxide film at the surface (10,0 and 22,5 g/m² at 1373 and 1473 K respectively) and the loss in weight of the sample for 100 h after cleaning its surface and removal of the damaged layer (including a dense protective coating) formed upon the above tests (12-20 and 24-25 g/m² at 1473 and 1573 K respectively). The equality of the weight gain and loss displays the strength of the alloyed Al₂O₃-NiO protective oxide film and the surface stability of the alloy upon oxidation in air flow. Owing to the absence of boride segregates or phases at grain boundaries, the alloy does not tend to corrosion under a stress of 0.8 of the bending yield strength and to intercrystalline corrosion including “hot” cracking and hot brittleness at medium temperatures. The high corrosion resistance of the alloy allows one to use it for critical coating-free parts of GTE and plants.

4.5. Comparison of the Properties of the VKNA Ni₃Al Based Alloy with those of Other High-Temperature Alloys

The VKNA-1V alloy surpasses the known Russian nickel-based aviation alloys ZhS-type with DS oriented or, especially, equiaxed grain structures in 100-h strength and 100-h specific strength. At 1373 K for VKNA-1V with DS

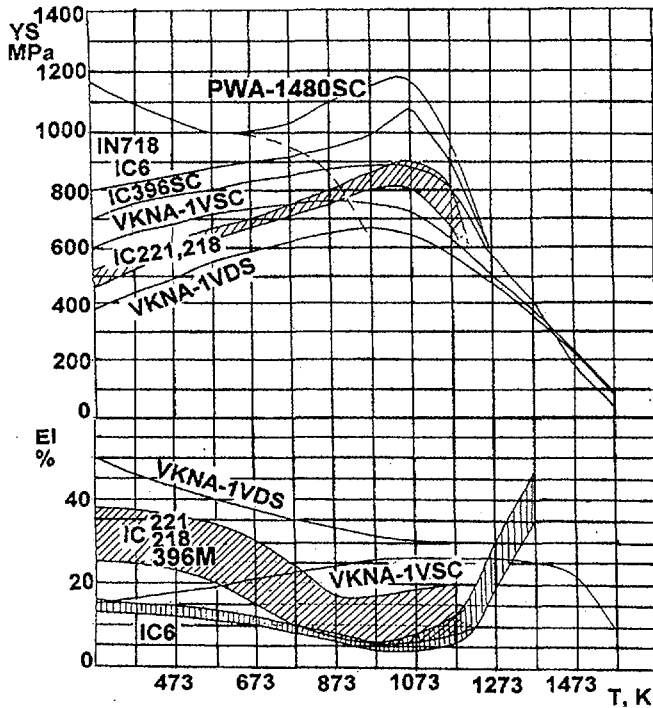


Fig. 5. Comparison of the properties of the most advanced high-temperature alloys based on Ni and Ni₃Al: temperature dependences of yield strength (YS) and relative elongation (EI).

and $\langle 111 \rangle$ SC structure $\sigma_{100}=90$ and 110 MPa; $\sigma_{100}/\rho=1,13$ and 1,26 km respectively versus $\sigma_{100} = 60$ and 90 MPa; $\sigma_{100}/\rho = 0,6$ and 1,05 km for ZhS6U and ZhS26 alloys with DS structure respectively despite VKNA-1V lower total content of expensive refractory metals decelerating the development of diffusional processes has 7.2 wt% W+Mo+Hf vs. 14.6 and

15.7 wt% W+Mo+Nb+Ta+Re in the ZhS alloys. In these properties, the VKNA-1V alloy ranks below only single-crystal high-alloy nickel superalloy ZhS40 (at 1373 K $\sigma_{100}=145$ MPa; $\sigma_{100}/\rho=1,64$ km). However, the density of the latter alloy is higher than that of the VKNA-1V alloy and its content of heavy refractory "slow" AE is higher than that of the VKNA-1V alloy by a factor of 2.8 (20.4 wt% W+Mo+Nb+Ta+Re). For this reason, the ZhS40 alloy is much more expensive than the VKNA-1V alloy. An undeniable advantage of the VKNA-1V alloy over nickel alloys is the ability of the articles produced of it to operate in air without protective coatings, whereas the all above Ni-superalloys ZhS-type and PWA, Rene, CMSX-type need protective coatings. The comparison of the data on yield strength of the Ni- and Ni₃Al-based high-temperature alloys in the temperature range between 293 and 1473 K shows (Fig. 5) that, in the medium temperature range (up to ~1273 K), the VKNA-1V

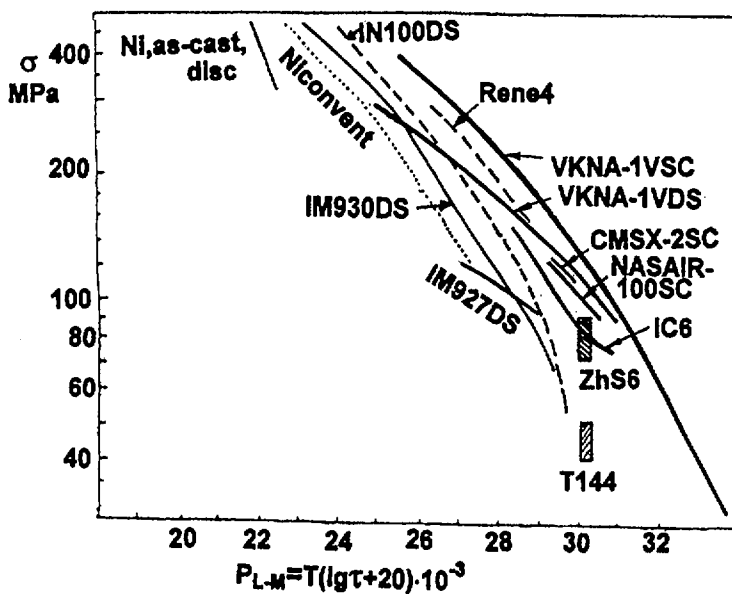


Fig. 6. Long-term strength of perspective materials based on intermetallics and conventional alloys. P_{L-M} is the Larson-Miller parameter, $C = 20$ (2-4,9-14).

alloy ranks somewhat below the most known Ni- and Ni₃Al-based alloys (1-4, 10-14). However, at temperatures above 1273 K, the advantage of the VKNA-1V alloy with columnar dendritic structure and, especially, single-crystal structure are rather pronounced.

We compared the best industrial nickel superalloys and the Ni₃Al-based alloys in long-term strength using the Larson-Miller curves because the temperature ranges, loads, and durations in all cases are different (Fig. 6).

The stable high characteristics of long-term strength and service life of the VKNA-1V alloy are provided by the optimally selected combination of the alloy component without boron, the optimal combination of the γ' and γ phases and the controlled structure formed upon the alloy preparation and retaining a high thermal stability.

New γ Ni₃Al+ γ ductile and high-strength alloy can be used as the best matrix material for composition materials (CM) having high heat resistance and excellent high-temperature strength, are reinforced by single-crystal sapphire continuous fibres (15). Such CM is prepared by the impregnation of sapphire fibres by the molten matrix materials under pressure.

Conclusions:

1. The alloying principles for Ni₃Al were formulated and realized for the design of the VKNA-1V alloy, which is characterized by good formability, low-temperature ductility, and high-temperature short and long term strength at temperatures exceeding the operating temperatures of current nickel superalloys, and is relatively cheap. The alloy does not need any protection against oxidation, has no tendency to hot brittleness and corrosion cracking.
2. The alloy composition is balanced so that it is based on a high-alloy γ + γ eutectic with a small excess of the γ' primary crystals. A high low-temperature ductility is provided by up to 10 vol % of ductile γ solid solution precipitates. This allows us to exclude alloying with boron and thus to avoid stress corrosion cracking and medium-temperature decrease in ductility. Chromium and zirconium improves medium-temperature toughness and strength, small amounts of "slow" refractory elements (W, Mo and Hf) decelerates softening that is induced by diffusional processes at temperatures above 0.8 T_m.
3. The strength of the alloy in various temperature ranges is caused by simultaneous or successive realization of several strengthening modes: solid-solution strengthening of the γ and γ' phases; disperse strengthening

by the γ -phase particles present in the γ' matrix (in the dendrites of eutectic origin); precipitation hardening by the fine γ' -phase precipitates in the γ particles.

4. The alloy is perspective material for heat resistant high-strength CM reinforced single-crystal sapphire fibres.

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