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Influence of Refractory Elements on Phase–Structural Stability of Heat-Resistant Corrosion-Resistant Alloys for Gas Turbine Blades

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One of the ways to improve the performance of gas-turbine engine blades is improvement of the alloying complexes of heat-resistant nickel alloys used for casting. The influences of refractory metals on the macro- and microstructure and the properties of alloys of this type are analysed. It is proposed to introduce rhenium and tantalum into a heat-resistant corrosion-resistant nickel alloy for the production of working blades of gas-turbine engines. The paper presents the results of the study of the influence of chemical composition on the phase–structural components of heat-resistant alloy. Phase-transition temperatures for a new heat-resistant corrosion-resistant nickel alloy containing rhenium and tantalum have been established: liquidus temperature is of $\cong 1370^\circ\text{C}$, solidus temperature is of 1320°C . Studies of the microstructure of the obtained samples in the cast state, after heating and cooling in calorimetric studies, make it possible to confirm that the phase–structural state corresponds to the alloy of this type, it consists of γ -solid solution including γ' -phase and carbides. Rhenium is mainly part of the γ -solid solution, and tantalum strengthens the cell boundaries. When analysing the structures of the studied samples after heating in a calorimeter to 1250°C ,

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topologically close-packed phases in the alloy containing rhenium and tantalum are not formed. Comparative studies have shown that the introduction of rhenium and tantalum to alloy improves its thermophysical properties, namely, liquidus, solidus and complete-dissolution temperatures, by about 50°C higher than for the standard heat-resistant nickel alloy CM88Y.

Key words: heat-resistant alloy, phase–structural stability, liquidus, solidus, turbine blade, gas-turbine engine.

Одним із шляхів поліпшення експлуатаційних характеристик лопаток газотурбінних двигунів є вдосконалення леґувальних комплексів жароміцних ніклевих стопів, що використовуються для їх лиття. Проаналізовано вплив тяжкотопких металів на макро- і мікроструктуру та властивості стопів такого типу. Запропоновано введення Ренію та Танталу у жароміцний корозійностійкий ніклевий стоп для одержання робочих лопаток газотурбінних двигунів. У роботі представлено результати дослідження впливу хемічного складу на фазово-структурні складові жароміцного стопу. Встановлено температури фазових переходів для нового жароміцного корозійностійкого нікелевого стопу, що додатково містить Реній і Тантал. Дослідження мікроструктури одержаних зразків у литому стані, після нагріву й охолодження (калориметричні дослідження) дають можливість підтвердити, що фазово-структурний стан відповідає вимогам до стопу розглянутого типу, тобто складається з γ -твердого розчину з включенням γ' -фази та карбідами. Під час аналізу структур досліджених зразків після нагріву в калориметрі до 1250°C топологічно щільно паковані фази у стопі, що містить Реній і Тантал, не утворювалися. Проведені порівняльні дослідження показали, що введення у стоп тяжкотопких металів сприяє підвищенню його теплофізичних властивостей, а саме, температур ліквідус, солідус та повного розчинення γ' -фази, приблизно на 50°C вище, ніж для стандартного жароміцного нікелевого стопу CM88Y.

Ключові слова: жароміцний корозійностійкий ніклевий стоп, фазово-структурна стабільність, ліквідус, солідус, лопатка турбіни, газотурбінний двигун.

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1. INTRODUCTION

It is known that heat-resistant nickel alloys are used for the production of turbine blades for gas turbine engines. The urgency of developing new heat-resistant corrosion-resistant alloys is due to the need to increase the service life of the turbine blades, increase the gas temperature before entering the turbine and save fuel. Simultaneously with the increase in operating temperature in the turbines, it is necessary to ensure the reliability of the blades, reduce the cost of repairing the engine and the production of spare parts. In accordance with the requirements of the design documentation for modern power and marine gas turbine engines, it is necessary to ensure their reliable operation at

the gas inlet temperature at the turbine inlet 1150–1200°C, which will increase power compared to previously developed engines. To increase heat resistance and reduce high-temperature corrosion of the blades, chromium is injected from 12 to 20% of the mass in the alloys. The increased chromium content promotes the formation of carbide phases in the alloy, which reduce the ductility of the cast metal [1–7].

Heat-resistant alloys based on nickel, which are alloyed with rhenium, tantalum, and ruthenium, are created. The use of these elements for alloying modern alloys allows to reduce the concentration of chromium and to increase the strength without reducing the level of corrosion resistance of the blades.

Scientific and practical developments in recent years of world centres engaged in materials science of heat-resistant nickel alloys for gas turbines, aircraft engines have considered materials for blades with low chromium content [8–10]. In this case, the most promising heat-resistant alloys based on nickel (superalloys) for gas turbine blades are alloys doped with rhenium [11–13]. The authors of proceedings [12–14] showed that rhenium is more effective than Ta, W, Mo and others alloying elements and has a high solubility in the γ -phase while the distribution coefficient of the alloying elements between the γ' - and γ -phases is $K/K_s = 0.1$.

The predominant dissolution of rhenium in the austenitic matrix of the alloy reduces the rate of diffusion processes that control the softening of the metal under conditions of high-temperature creep, thereby inhibiting the rate of coagulation and dissolution of the γ' -phase. This helps to maintain long-term strength by increasing the high-temperature stability of the solid solution. It is empirically proved that increasing the concentration of alloying elements with large atomic radii has a more significant effect on the change in the lattice parameter of the γ -phase than the γ' -phase, and since rhenium and tantalum have an atomic radius larger than nickel (ratio for rhenium—1.063), they have a positive effect on the mismatch between the periods of the crystal lattices of the γ - and γ' -phases—the so-called misfit $\Delta a_{\gamma-\gamma'} = (a_{\gamma} - a_{\gamma'})/a_{\gamma}$. This is a determining factor in the long-term strength of poly- and single crystals of heat-resistant alloys at high temperatures. Rhenium has a low diffusion coefficient in nickel and therefore it inhibits the coagulation processes of the strengthening γ' -phase, thereby, increasing its thermal stability. In this case, Re partially displaces tantalum and aluminium from the solid solution, which are mainly in the γ' -phase, as a result of which it effectively strengthens it [15, 16].

The positive effect of rhenium on the heat resistance of heat-resistant nickel alloys is due to the increase in solidus temperature, its increased solubility in solid nickel solution and decrease in the diffusion coefficients of alloying elements.

In recent years, tantalum has been actively used for alloying heat-

resistant alloys, which leads to a significant increase in the strength characteristics of the alloy [17–19]. It belongs to the γ' -phase-forming elements and therefore significantly increases the heat resistance and heat resistance of alloys, as well as the dissolution temperature of the γ' -phase. Tantalum-doped alloys are also used for the manufacture of blades of stationary power gas turbines (GTU), for example, of type ZMI-3U, TsNK-21, TsNK-7, TsNK-8. In them, the gas temperature at the inlet to the turbine is from 750°C to 950°C, which is much lower than in aviation gas turbine engines.

Studies in recent years have shown that the joint introduction of tantalum and rhenium in the alloy provides a sharp decrease in the diffusion mobility of elements in the alloy, especially at high temperatures, significantly strengthens the solid solution and stabilizes the γ' -phase, in particular slows down the formation of large γ' -phase particles, due to coagulation and increases the dissolution temperature of small [19–21]. This also leads to an increase in heat resistance and thermal stability of heat-resistant nickel alloys [17]. However, it was found that the replacement of vanadium with tantalum in another alloy (ZhS32) also increases its heat resistance by about three times, compared with alloys ZhS-6U and ZhS-26U.

The high content of refractory metals in alloys of this type increases the risk of formation in them of fragile intermetallic topologically densely packed (MS) phases and, above all, σ -phases $(\text{Cr}, \text{Mo})_x(\text{Ni}, \text{Co})_y$, where x and y can change in ranging from 1 to 7 [2, 9, 12, 17]. The crystal structure of the σ -phase belongs to the tetragonal with a complex unit cell containing 30 atoms. Since the crystal lattices of the σ -phase and Me_{23}C_6 carbide are coherent, the σ -phase often originates on the secretions of Me_{23}C_6 carbide. This type of phase is formed in the form of plates and therefore dramatically reduces the resistance to destruction of alloys at high temperatures, adversely affecting ductility and durability.

The most important role in ensuring the long-term strength of heat-resistant alloys, their manufacturability during casting and heat treatment belongs to the temperatures of phase transformations. Temperatures of complete dissolution of the γ' -phase in γ -solid solution $T_{\text{c.d.}}$ (T_{solvus}), temperature solidus T_S and liquidus T_L are not only characteristics of thermal stability, but also determine, in the first approximation, the maximum temperature performance of the heat-resistant alloy [22–24].

Phase transition temperatures also determine some technological properties. In particular, the temperature interval of crystallization $\Delta T_{\text{cr}} = T_L - T_S$, which characterizes the tendency of alloys to form a directional columnar structure of castings during directional crystallization and bulk microporosity. The solvus and solidus temperatures determine the conditions for the formation of a homogeneous struc-

ture of γ' -phase particles in the matrix of a homogeneous γ -solid solution during heat treatment. The value of the 'heat treatment window', which is expressed by the temperature difference $T_S - T_{c.d.}$, should be large enough to carry out high-temperature homogenization without the risk of melting [25, 26].

Over the last decade, PTIMA of the N.A.S. of Ukraine has been studying the effect of refractory metals such as rhenium, tantalum, ruthenium on the structure and properties of heat-resistant corrosion-resistant alloys for gas turbine blades and created a new composition of heat-resistant corrosion-resistant alloy containing additional rhenium and tantalum. The purpose of this work is to study the temperatures of phase transformations, the study of phase-structural components of the experimental heat-resistant corrosion-resistant alloy for the blades of the first and second stage turbines of gas turbine engines for energy purposes. One of the ways to improve the performance of gas turbine engine blades is improvement the alloying complexes of heat-resistant nickel alloys used for casting. The influences of refractory metals on the macro- and microstructure and the properties of alloys of this type are analysed. It is proposed to introduce rhenium and tantalum into a heat-resistant corrosion-resistant nickel alloy for the production of working blades of gas turbine engines.

The paper presents the results of the study of the influence of chemical composition on the phase-structural components of heat-resistant alloy. Phase transition temperatures for a new heat-resistant corrosion-resistant nickel alloy containing rhenium and tantalum have been established: liquidus temperature of $\cong 1370^\circ\text{C}$, solidus temperature of $\cong 1320^\circ\text{C}$. Studies of the microstructure of the obtained samples in the cast state, after heating and cooling in calorimetric studies make it possible to confirm that the phase-structural state corresponds to the alloy of this type, it consists of γ -solid solution including γ' -phase and carbides. Rhenium is mainly part of the γ -solid solution, and tantalum strengthens the cell boundaries. When analysing the structures of the studied samples after heating in a calorimeter to 1250°C , topologically close-packed (TCP) phases in the alloy containing rhenium and tantalum were not formed.

Comparative studies have shown that the introduction of rhenium and tantalum alloy improves its thermophysical properties, namely liquidus, solidus and complete dissolution temperatures, by about 50°C higher than for the standard heat-resistant nickel alloy CM88Y.

2. EXPERIMENTAL/THEORETICAL DETAILS

Five samples of experimental alloy and known industrial alloy CM88Y [5, 27–29] were investigated. The chemical composition of the samples of experimental alloys is shown in Table 1.

TABLE 1. Chemical composition of nickel-based heat-resistant alloys.

	Heat-resistant alloy analog, weight %	Experimental heat-resistant alloys, weight %				
	CM88Y	1	2	3	4	5
C	0.07	0.05	0.05	0.05	0.04	0.05
Cr	15.8	12.75	12.75	12.75	12.30	12.75
Co	11.7	7.15	7.15	7.15	7.15	7.15
Mo	1.96	1.15	1.15	1.15	1.15	1.15
W	5.30	6.30	6.30	6.30	6.30	6.30
Al	3.00	3.25	3.25	3.25	3.25	3.25
Ti	4.60	2.20	3.60	2.05	2.05	2.05
Nb	0.15	0.30	0.30	0.30	0.30	0.30
Hf	0.3	≤ 0.1	≤ 0.1	≤ 0.1	≤ 0.1	≤ 0.1
Ta	–	2.75	2.75	3.30	2.75	3.30
Re	–	4.00	4.00	4.00	4.00	4.00
B	0.09	0.01	0.01	0.01	0.01	0.01

To obtain prototypes of the heat-resistant alloys, which were pre-melted from pure materials, that were melted by vacuum-induction remelting on a foundry unit UPPF-2 by known technology (pressure in the furnace of 1.2–2.5 Pa, pouring temperature in the mould of 1560–1580°C, mould temperature of 800°C).

Determination of the number of main components of alloys and impurities was performed by the chemical method according to standard methods, microdoping additives were controlled by chemical-spectral method with a relative error of $\pm 0.001\%$. To study the structure, phase and local chemical composition of the alloy used a scanning electron microscope 'Jeol JSM6490LV' (Japan) with an additional node for micro-x-ray analysis 'Oxford Jnea Drycool'.

Temperatures of phase transformations, T_s , T_L , $T_{c.d.}$ (complete dissolution of the γ' -phase), were determined by differential scanning calorimetric (DSC). The studies were performed on a high-precision synchronous thermal analyser Netzsh STA449C (Germany) under heating and cooling at a rate of 20°C/min samples of alloys with a size of 2.8×2.8×2.8 mm³. The accuracy of temperature measurement is of $\pm 1.5^\circ\text{C}$.

3. RESULTS AND DISCUSSION

3.1. Study of the Microstructure of Alloy Samples in the Cast State

The structure, phase composition of CM88Y alloys and alloy doped with rhenium and tantalum and their chemical composition depending on the

content of refractory elements are shown in Figs. 1, 2. It is seen that the alloys have a dendritic-cellular structure consisting of γ -solid solution strengthening γ' -phase and carbides. The structure of CM88Y contains carbides $Me(\text{Cr, Fe, W, Mo})_{23}\text{C}_6$ and carbides $Me\text{C}$ based on Ti and Nb.

When studying the structure of the experimental alloy, it is seen that eutectic carbide formations of different sizes and shapes are located at the boundaries of dendritic cells and in the axial space. Refractory chemical elements of the alloy (W, Re) are located in the axes of the dendrites, and Ta, Al, Nb, Ti—enrich the cell boundaries. Rhenium is absent at the boundaries of the dendritic cell. It is mainly part of the γ -phase, and tantalum is mainly doped with the γ' -phase (Fig. 3).

3.2. Determination of Temperature Conditions of Phase Transitions in Heat-Resistant Alloy

For five samples of the experimental alloy and the known industrial alloy CM88Y were determined the main temperatures of phase transformations: temperatures of solidus, liquidus, solvus (complete dissolution) ($T_{\text{c.d.}}$ of γ' -phases). The shift of the level of values of critical points (T_S, T_L) in the direction of increase, from temperature 1271°C,

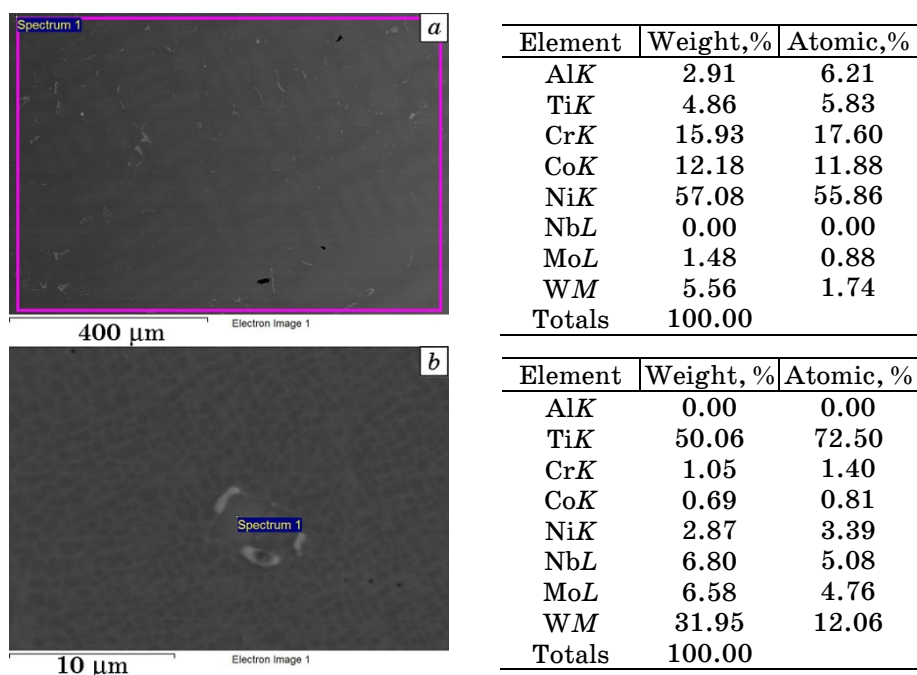


Fig. 1. Microstructures and chemical composition of heat-resistant alloy CM88Y phase: microstructure (a); carbide $Me\text{C}$ (b).

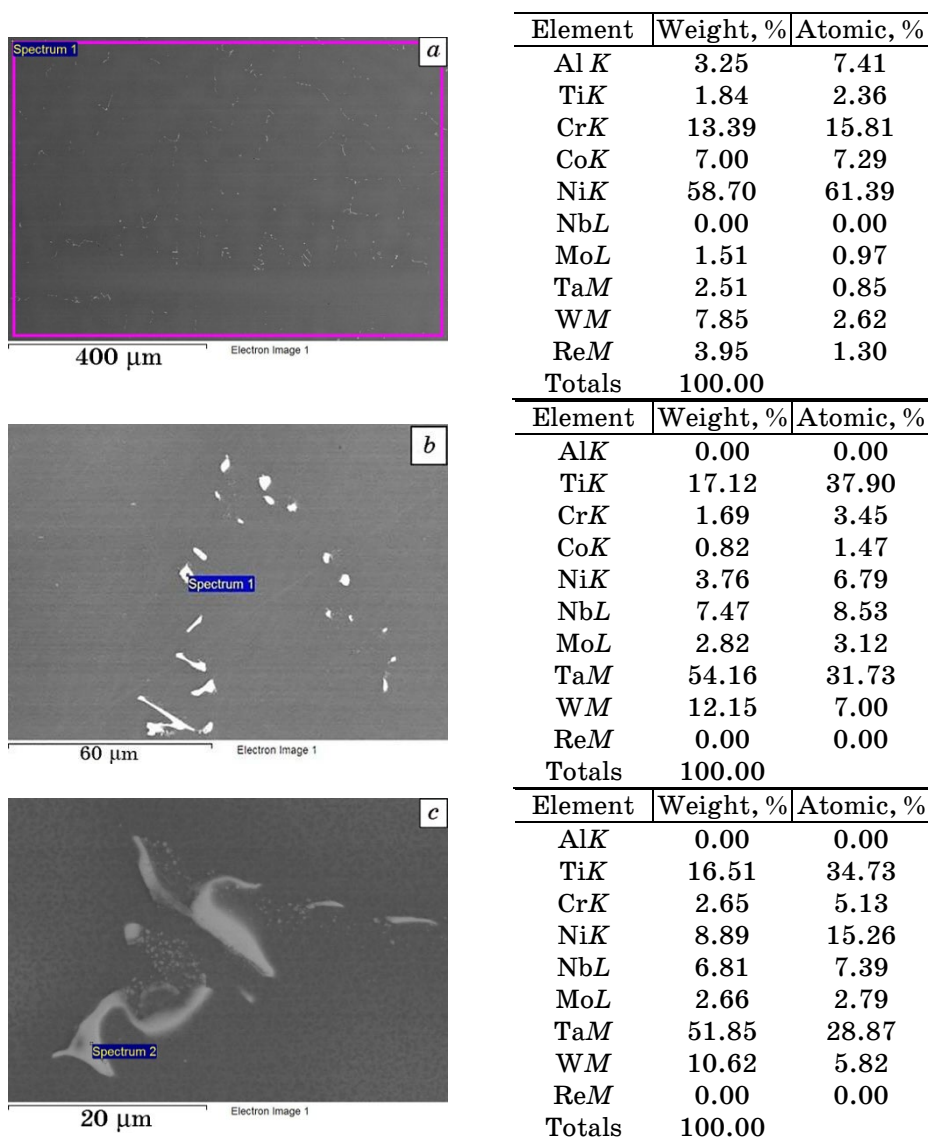


Fig. 2. Microstructures and chemical composition of experimental heat-resistant alloy phase: microstructure and Ti-based carbides (*a*), carbide *MeC* (*b*, *c*).

1332°C to 1326°C, 1385°C, respectively, at introduction of rhenium and tantalum (Table 2) is established.

This is a confirmation of the fact of the positive effect of these elements on the temperature threshold of dissolution of the reinforcing γ' -phase of $\text{Ni}_3(\text{Al}, \text{Ti})$ and determines the increase in the temperature lev-

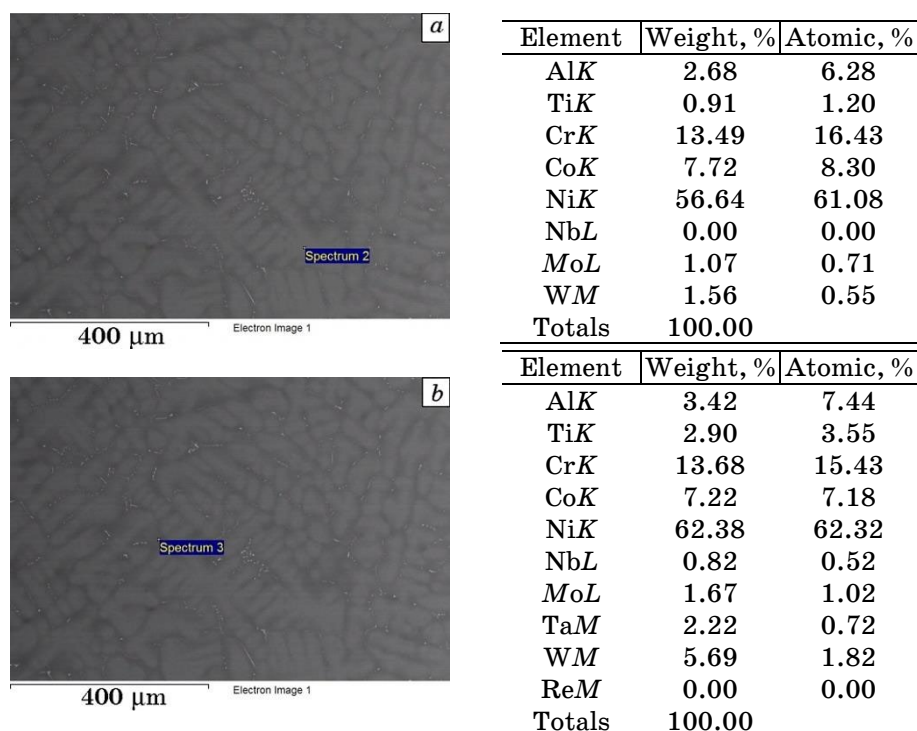


Fig. 3. Microstructure and chemical composition of the experimental alloy (sample 5) in the axes of the dendrite (*a*) and at the cell border (*b*).

el of the alloy containing rhenium and tantalum [17]. In comparison with temperatures for CM88Y, the data obtained for it are 50°C higher.

Figures 4–6 show the DSC curves and their derivatives during heating and cooling of an industrial alloy and two samples of alloys containing rhenium and tantalum (No. 1, No. 5). DSC curves show exothermic and endothermic peaks of reactions in samples during heating and cooling. At all dependences there is a peak in the temperature range from 570°C to 610°C, which is due to the processes of close ordering of chromium atoms in solid solution. As Cr in the alloy decreases, this peak shifts toward a higher temperature.

TABLE 2. Phase-transition temperatures of nickel-based heat-resistant alloys.

Temperature of phase transitions	Experimental heat-resistant alloy					CM88Y
	1	2	3	4	5	
$T_S, ^\circ\text{C}$	1313	1313	1326	1326	1331	1271
$T_L, ^\circ\text{C}$	1372	1372	1376	1376	1387	1332
$T_{\text{solvus}}, ^\circ\text{C}$	1158	1162	1174	1174	1172	1180

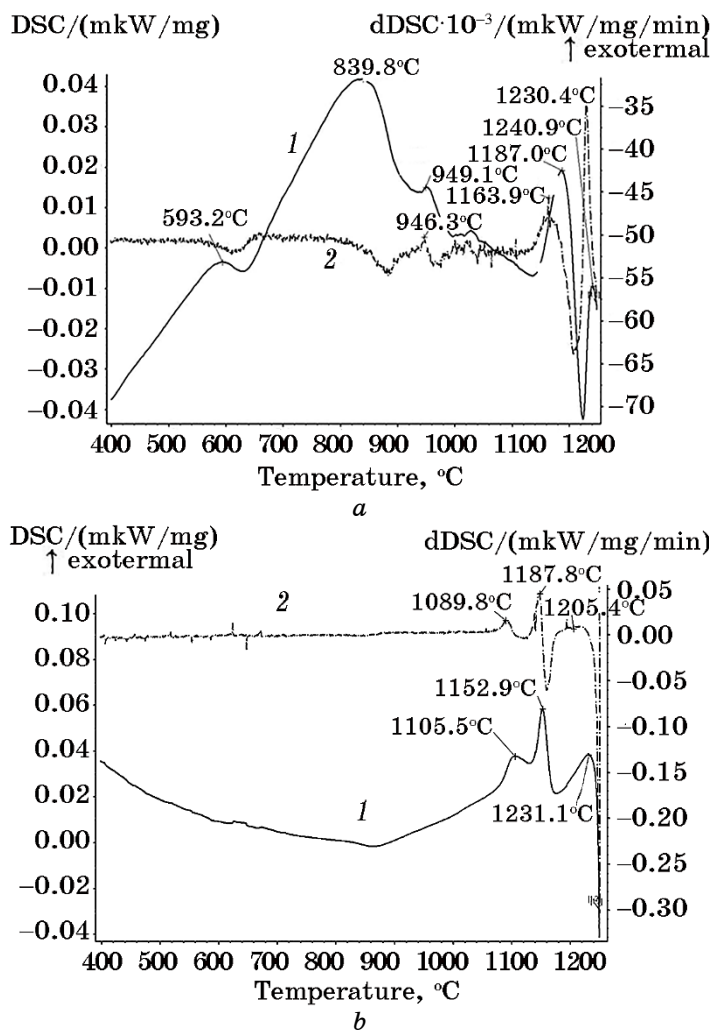


Fig. 4. Differential scanning calorimetry (DSC)—curves (1) and derivatives of them (2) alloy CM88Y: heating (a), cooling (b).

The intense peak on the DSC curve at 840°C (on a sample CM88Y) and on experimental samples—at 876°C (sample 1) and at 806°C (sample 5) during heating corresponds to the initial processes of dissolution of the γ' -phase in solid solution, and in the range up to 1100°C there is dissolution of γ' -particles having bimodal size distribution and coagulation process γ' -particles.

The study of thermal effects when heating the alloy CM88Y and alloys No. 1, No. 5 showed that the intense dissolution of the γ' -phase is carried out at $T = 1140^\circ\text{C}$ and complete dissolution of the γ' -phase in

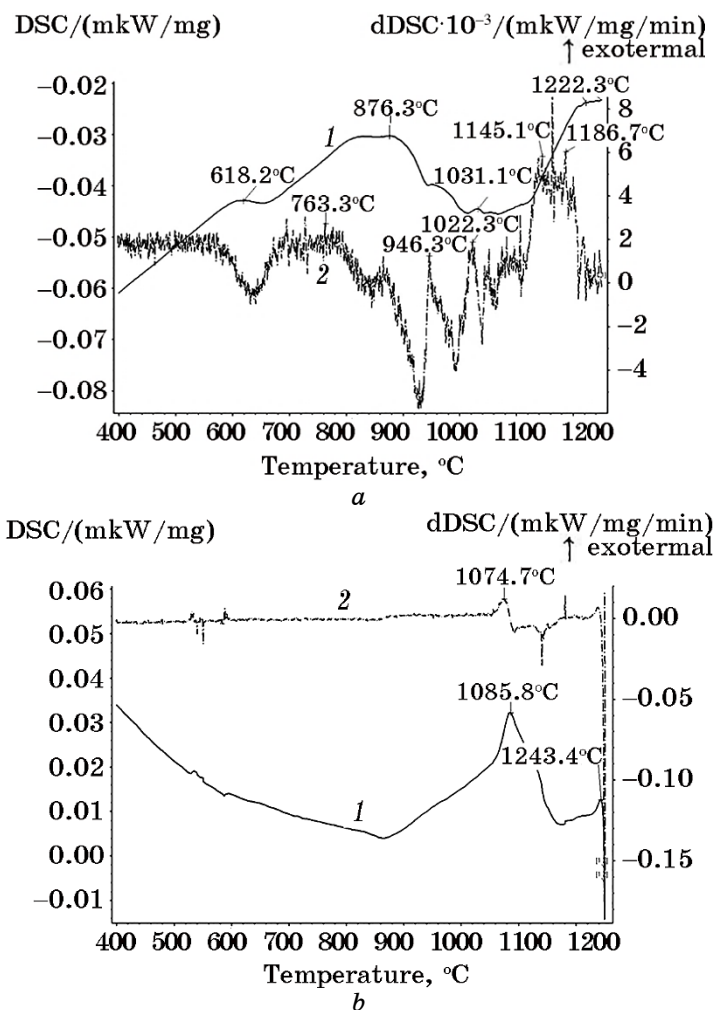


Fig. 5. Differential scanning calorimetry (DSC)—curves (1) and derivatives of them (2) experimental heat-resistant alloy (sample 1): heating (a), cooling (b).

solid solution occurs at $T = 1190^{\circ}\text{C}$, 1220°C and 1240°C , respectively. The maximum dissolution rate of the γ' -phase in solid solution for alloys CM88Y and No. 1, No. 5 corresponds to temperatures of 1165°C , 1165°C , 1164°C , respectively.

In the process of cooling the alloy samples from a temperature of 1250°C , an exothermic reaction is observed on the DSC curves, which indicates the beginning of the decay of the γ -solid solution with the formation of γ' -phase particles. The maximum of intense heat release during the decay of the γ -phase corresponds to alloys and temperatures of 1106°C , 1086°C , 1107°C , respectively. Also on the DSC curves,

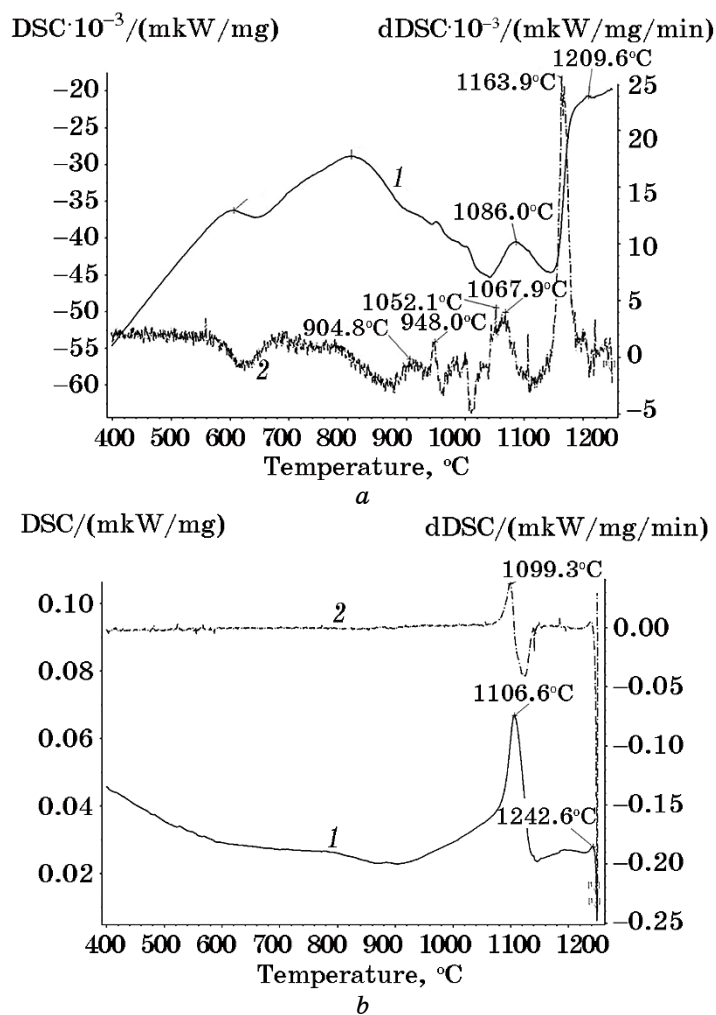


Fig. 6. Differential scanning calorimetry (DSC)—curves (1) and derivatives of them (2) experimental heat-resistant alloy (sample 5): heating (a), cooling (b).

there are small peaks that are associated with the release of carbides MeC and $Me_{23}C_6$. The temperature of the phase transitions in the alloys was determined from DSC curves and their derivatives—differential curves (dDSC) (Table 3).

3.3. Microstructure and Chemical Composition of the Phases of the Studied Samples of Heat-Resistant Alloys in the Annealed State

After heating to a temperature of 1250°C, the structure of the alloys

TABLE 3. Temperature of phase transitions of nickel-based heat-resistant alloys.

	Phase transformation temperature, °C	heat-resistant alloys		
		CM88Y	experimental	
			No. 1	No. 5
Heating	Strengthening atoms Cr on γ -phase	593	618	606
	Dissolution of $Me_{23}C_6$ carbides in an alloys preheated to 950°C	950	950	950
	The beginning of intense dissolution γ' -phase	1040	1031	1086
	Maximum dissolution rate γ' -phase	1140	1140	1150
	Complete dissolution of the γ' -phase	1165	1165	1164
	Melting of nonequilibrium eutectic ($\gamma + \gamma'$)	1190	1224	1236
	Melting of nonequilibrium eutectic ($\gamma + \gamma'$)	1231	1243	1243
Cooling	Formation of $Me_{23}C_6$ carbides	1153	1180	1180
	The beginning of the formation of the γ' -phase	1140	1160	1150
	The maximum amount of γ' -phase formed during the decay of γ -solid solution	1106	1008	1106
	Carbide formation interval $Me_{23}C_6$	1050–900	1060–900	1060–900

has not changed. For the CM88Y alloy, it consists of a matrix ($\gamma + \gamma'$) component with carbides MeC based on Ti, Nb and carbides of the $Me(Cr, Fe, W, Mo)_{23}C_6$ (Fig. 7).

In samples of other alloys, carbides based on Ta are formed, and Re is not part of carbides (Figs. 8, 9).

In the analysis of the structures of the investigated samples after heating in the calorimeter to 1250°C, TCP-phases in the alloy containing rhenium and tantalum were not formed.

Doping of alloys with tantalum and rhenium, as well as reduction of chromium in them significantly increase the thermal stability of the γ' -phase. At the same time, $T_{c.d.}$ for γ' -phase increases by $\cong 50^\circ\text{C}$ to the value of 1220°C. In addition, the dissolution temperature of the nonequilibrium ($\gamma + \gamma'$) eutectic (T_{eut}) increases to 1245°C, due to the low partition coefficient ($K_{Re/\gamma'} = 0.1$) for Re in the γ -solid solution, and Ta— γ' -phase. As a result, the coagulation of the reinforcing particles of the γ' -phase proceeds slowly, respectively, and less intense is the weakening of the alloy.

In the structure of CM88Y there are carbides $Me_{23}C_6$ based on Cr, W and MeC based on Ti.

It can be seen in Figs. 7–9 that the alloys have a dendritic-cellular structure consisting of a γ -solid solution, a strengthening γ' -phase and carbides.

Thus, from the photo of the structure of alloys doped with rhenium and tantalum, it can be seen that carbide formations of different sizes

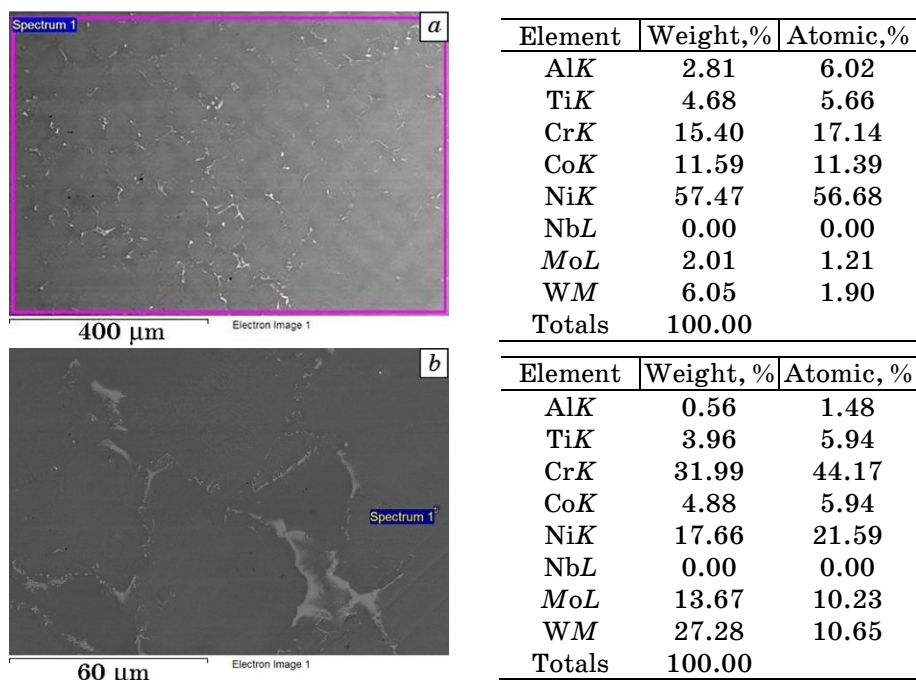


Fig. 7. Microstructure and chemical composition of heat-resistant alloy CM88Y phases after heating to temperature 1250°C: ($\gamma + \gamma'$)-phase + carbides (*a*), carbide $Me_{23}C_6$ (*b*).

and shapes are located at the boundaries of dendritic cells and in the axial space. Carbides are formed on the basis of a strong carbide-forming element—tantalum.

Refractory chemical elements of the alloy (W, Re) are located in the axes of the dendrites, and Ta, Al, Nb, Ti—enrich the cell boundaries. Rhenium is absent at the boundaries of the dendritic cell. It is mainly part of the γ -phase.

As a result of analysis of experimental data and literature sources [29–31] on temperatures and kinetics of phase transformations, the following mode of heat treatment of the experimental alloy was chosen: homogenization 1220°C, holding 4 hours, cooling with argon at a rate of 60–80°C/min; 1050°C, 6 hours, cooling in vacuum at a residual pressure of 0.133–0.00133 Pa; 870°C, 20 hours, cooling to room temperature in a dynamic vacuum for at least 80 minutes.

4. CONCLUSION

As a result of the conducted research, the temperatures of phase trans-

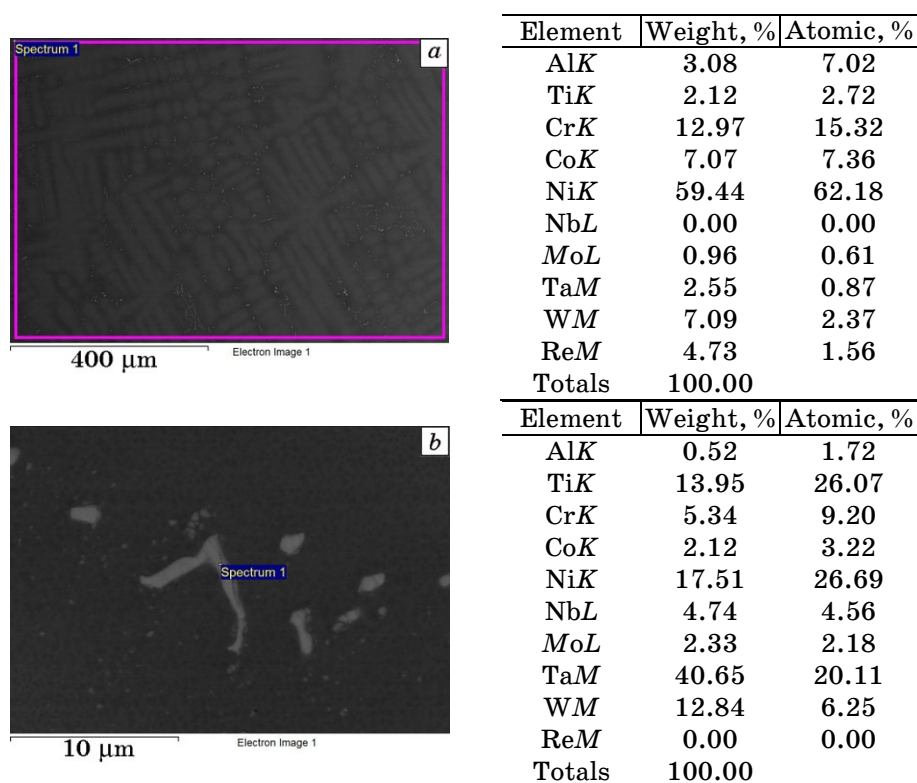


Fig. 8. Microstructure and chemical composition of experimental heat-resistant alloy phases (sample 1) after heating to temperature 1250°C: ($\gamma + \gamma'$)-phase + carbides (a), carbide $Me_{23}C_6$ (b).

formations were established: solidus, liquidus, beginning ($T_{b,\gamma'}$) and complete dissolution ($T_{c,d,\gamma'}$) of the γ' -phase in γ -solid solution, melting of nonequilibrium ($\gamma + \gamma'$) eutectic (T_{eut}) and dissolution of carbides. In the analysis of the structures of the studied samples after heating and cooling to 1250°C, the TCP phase was not observed.

Doping of alloys with rhenium and tantalum (the proposed alloy), as well as the reduction of chromium in them significantly increase the thermal stability of the γ' -phase: $T_{c,d,\gamma'}$ increases by $\cong 50^\circ\text{C}$ to 1220°C. At the same time, the melting temperature of the nonequilibrium ($\gamma + \gamma'$) eutectic (T_{eut}) increases to 1245°C.

Rhenium is mainly doping the γ -solid solution, and tantalum the γ' -phase. As a result, the coagulation of the reinforcing particles of the γ' -phase proceeds slowly, respectively, and the alloy is softened less intensively.

For a new heat-resistant Nickel alloy doped with Ta and Re, the homogenization temperature is of $\cong 1220^\circ\text{C}$, and the heat-treatment in-

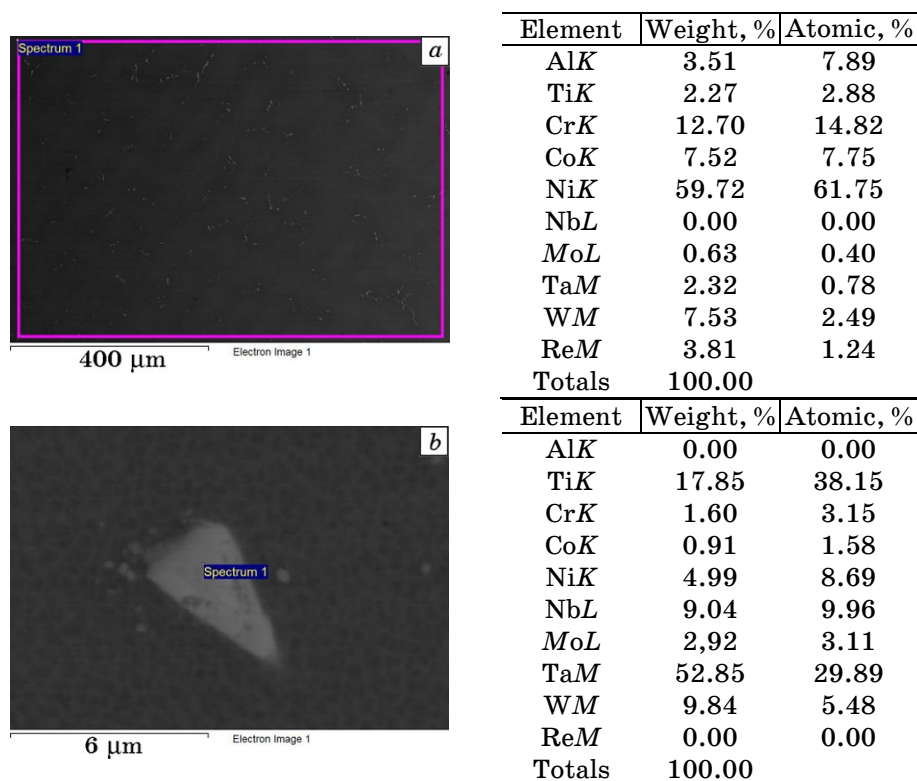


Fig. 9. Microstructure and chemical composition of experimental heat-resistant alloy phases (sample 5) after heating to temperature 1250°C: ($\gamma + \gamma'$)-phase + carbides (a); carbide MeC (b).

terval ($\Delta T = T_{eut\gamma'} - T_{c.d.\gamma'}$) is of $\cong 15^\circ\text{C}$, respectively.

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