The object of research in this work is the processes of forming the microstructure and mechanical properties of heat-resistant steels depending on their chemical composition.

The microstructure and high-temperature mechanical properties of austenitic heat-resistant chrome-nickel steels of the proposed chemical composition were investigated in the work. The microstructure study determined the grain size and topography of inclusions in steels. Based on the results of mechanical tests, a multifactorial experiment was planned, which made it possible to establish the relationship between the mechanical properties of steels with alloying elements selected as factors. The obtained regression equations were used to estimate the quantitative influence of each alloying element on the corresponding mechanical properties.

Analysis of the results of the experiment made it possible to determine the optimal chemical composition of steel for gas turbine engines used in the aerospace industry, as well as in the metallurgy of titanium production.

It is shown that the state of solid solution and heat-resistant niobium and molybdenum carbides (chromium carbides dissolve at a temperature of 950 °C) are an important factor that significantly affects the structure, mechanical, and service properties of heat-resistant steel. The austenite structure is provided by the required amount of nickel.

The resulting indicators of heat resistance of steels of different compositions, tested at temperatures of 850 °C, 950 °C, and 1050 °C, proved the superiority of steel with a higher content of carbon and chromium and a lower content of nickel.

The mechanism of steel strengthening and the influence of alloying with carbon, chromium, and nickel on it have been determined. The optimal chemical composition of heat-resistant steel on an iron-nickel basis for operation at a temperature of 950 °C has been established

Keywords: austenitic heat-resistant chrome-nickel steel, alloying elements, microstructure, carbides, mechanical properties, regression analysis

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DEVELOPING THE OPTIMAL CHEMICAL COMPOSITION OF HEAT-RESISTANT CR-NI STEEL FOR AEROSPACE EQUIPMENT

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1. Introduction

The range of assemblies and parts of gas turbine engines and equipment, which are used in the aerospace, metallurgical, and other industries under conditions of increased temperature and energy loads, is constantly increasing. At the same time, the mineral and raw material base for the production of refractory alloying components for the production of heat-resistant steels in Ukraine is significantly limited. Therefore, the issue of designing a material, first of all, for gas turbines, which would provide high technological and operational properties of aircraft engines in combination with economy, is one of the main ones in their design.

It is possible to consider the combination of C-Cr-Ni multi-component alloying systems with Mo-Nb-P3M microalloying of steel on an iron-nickel-chromium basis together with advanced technologies of its production as promising in solving such a pressing problem.

It is important to determine the influence of Fe-Cr-Ni steel matrix alloying elements on the mechanical and service properties of steels operating up to a temperature of 950 °C.

Therefore, developing an economically alloyed heat-resistant steel with increased mechanical properties at temperatures up to 950 °C is relevant and in demand for improving the operation of gas turbine engines and reactors for the magnethermal production of titanium.

2. Literature review and problem statement

Modeling the heat-resistant characteristics of the required steel, which does not undergo polymorphic transformations, is carried out by a combination of its hardening and subsequent aging [1]. During aging, carbide and intermetallic phases are released in the γ-matrix of steel. For the greatest strengthening of the metal, these compounds must be finely dispersed and evenly distributed throughout the body of the grain. In addition, grain boundaries should be strengthened by finely dispersed inclusions [2]. The microstructure of heat-resistant steels is usually austenite with carbides of chromium, such as $Me_{23}C_6$ [3] and other alloying carbide-forming elements.

Austenitic chrome-nickel steels are characterized by a combination of high corrosion resistance, plasticity, and economy.

AISI 316 steel (wt.%: $C \leq 0.8$; $Si \leq 0.75$; $Mn \leq 2.00$; Cr 16.0–18.0; Ni 10.0–14.0; Mo 2.0–3.0; $S \leq 0.03$; $P \leq 0.045$; Fe – the rest), considered in [4], belongs to the austenitic class. It is used for the manufacture of parts of mechanisms operating in a wide temperature range, as well as for welded structures operating in various aggressive environments (sea water, acids). But the questions related to the use of this steel at temperatures of 850–1050 °C remained unresolved. The reason for this is the insufficient amount of carbon, which causes the low strength of the steel at these temperatures.

10Cr17Ni13Mo3Ti steel is also widely distributed (wt.%: $C \leq 0.10$; $Si \leq 0.80$; $Mn \leq 2.00$; Cr 16.0–18.0; Ni 12.0–14.0; Ti $5 \cdot C$ –0.70; Mo 3.0–4.0; $S \leq 0.020$; $P \leq 0.035$; Fe – the rest) [5] of the austenitic class. It is used for the manufacture of parts that work under conditions of high temperatures and an aggressive environment of the chemical industry and the production of spongy titanium. But due to the excess content of molybdenum, the appearance of the σ-phase is possible, which causes brittleness.

The chemical composition closest to the steel studied in work [6] is steel 10Cr17Ni13Mo2Ti (wt.%: $C \leq 0.10$; $Si \leq 0.80$; $Mn \leq 2.00$; Cr 16.0–18.0; Ni 12.0–14.0; Ti $5 \cdot C$–0.70; Mo 2.0–3.0; $S \leq 0.020$; $P \leq 0.035$; Fe – the rest) of the austenitic class. It is intended for the manufacture of products that work in highly aggressive environments, designed for long service life at a temperature of 600 °C. However, this steel is not suitable for work at temperatures of 850–1050 °C, due to insufficient heat resistance and the presence of δ-ferrite.

In work [7], the authors investigated the possibility of creating retorts [8] for magnethermal production of spongy titanium based on bimetal. The basis of the bimetal was steel, which was used with plating layers of ferritic and austenitic steels. It has been established that this steel is suitable for use under difficult conditions at high temperatures.

Solving the considered problems can be achieved by creating an austenitic chrome-nickel steel with an experimentally selected chemical composition, which will have high operational characteristics in the temperature range of 850–1050 °C.

3. The aim and objectives of the study

The purpose of this work is to determine the chemical composition of economical heat-resistant austenitic chrome-nickel steel, most suitable for use at temperatures up to 950 °C. This will make it possible to create the most suitable heat-resistant steel for use in aerospace engineering and in the production of spongy titanium at temperatures up to 950 °C.

To achieve the goal, the following tasks were set:

– to determine the mechanical properties of the studied steels in the temperature range of 850...1050 °C;

– to perform a regression analysis and obtain dependences for calculating the value of heat resistance and plasticity for different chemical compositions of chrome-nickel-based steel, microalloyed Rare-earth element;

– to produce experimental samples of selected steels and to study their microstructure after long-term exposure at operating temperature.

4. The study materials and methods

The object of our research is the processes of forming the microstructure and mechanical properties of heat-resistant steels depending on their chemical composition. The main hypothesis of the research assumes that optimization of the chemical composition of steel will make it possible to choose a steel in which a fine dispersed structure is formed at a temperature of 950 °C. It is assumed that thanks to this, the obtained steel will be suitable for the manufacture of gas turbine engines used in the aerospace industry, as well as in metallurgy for the production of titanium.

The research materials were austenitic chrome-nickel steels of different chemical composition, microalloyed with Mo and Nb (Table 1). Steels were smelted under laboratory conditions in an induction furnace with a capacity of 50 kg with a main lining. Castings were rolled into 40×80×100 mm pieces, from which 25-, 20-, and 16-mm thick samples were obtained by hot rolling.

Table 1

Chemical composition of experimental steels, %

Steel	C	Si	Cr	Mn	Ni	Cu	Nb	Mo	Fe
11Cr13Ni13MoNb	0.11	1.24	12.9	1.7	13.1	0.28	0.45	0.24	70
11Cr13Ni16MoNb	0.11	1.32	13.6	1.9	15.9	0.28	0.31	0.24	66.3
14Cr16Ni17MoNb	0.14	1.21	16.3	1.5	17.6	0.1	0.38	0.11	62.86
14Cr16Ni13MoNb	0.14	1.18	16.05	1.6	13.7	0.16	0.39	0.11	66.82

The obtained samples were subjected to heat treatment: they were heated to a temperature of 1050 °C, cooled in water, followed by holding in a vacuum at a temperature of 850 °C for 8 hours. After heat treatment, the test steels were subjected to grinding and polishing.

The mechanical properties of the experimental steels were tested on tensile samples at temperatures of 850 °C, 950 °C, and 1050 °C on a tensile testing machine of the HMS-20 type.

To construct regression dependences of the mechanical properties of steels on temperature and the content of components, the experiment planning procedure was applied [9, 10].

The following mechanical properties were selected as optimization parameters in the planning of the experiment: strength limit, yield limit, relative elongation, and relative narrowing.

Factors acting on the studied steels are alloying components – carbon, chromium, and nickel in selected percentage ratios (Table 2). The remaining alloying elements (Si, Mn, Cu, Nb, Mo) are not included in the optimization of the chemical composition of steels, as they are added in microdoses and do not affect the final mechanical properties. Niobium and molybdenum carbides are refractory and poorly soluble, so they do not take part in dispersion hardening processes, but they are mainly distributed along grain boundaries, thereby strengthening them.

Table 2

Research factors

Characteristic	Factors		
	C, % wt.	Cr, % wt.	Ni, % wt.
Code	X_1	X_2	X_3
Basic level	0.12	15	15
Variation interval	0.02	2	2
Lower level	0.10	13	13
Upper level	0.14	17	17

To solve the problem, it is necessary to derive a mathematical model of the study, that is, an equation connecting each optimization parameter with all factors. In order to save on research, a half experiment was used, that is, with the number of factors equal to three, the number of experiments will be equal to $2^{3-1}=4$. The planning matrix of the research experiment is given in Table 3, where x_i are alloying elements, y_i are mechanical properties.

According to the matrix, regression equations were constructed (Table 4). They make it possible to quantitatively assess the influence of each of the alloying elements on the optimization parameters, that is, the mechanical properties. The free term of the equation is the average value of the optimization parameter.

Table 3

Experiment planning matrix

No.	x_0	x_1	x_2	x_3	x_1x_3	x_2x_3	$x_1x_2x_3$	y_i
1	+	–	–	–	+	+	–	y_1
2	+	–	–	+	–	–	+	y_2
3	+	+	+	+	+	+	+	y_3
4	+	+	+	–	–	–	–	y_4

Table 4

Regression equations

Mechanical properties	T, °C	Equation
σ_U, MPa	850	$y_1=167.5+11X_1+11X_2-0.65X_3+1.15X_1X_3+1.15X_2X_3-0.65X_1X_2X_3$
	950	$y_2=111.25+8.75X_1+8.75X_2-4.25X_3-5.75X_1X_3-5.75X_2X_3-4.25X_1X_2X_3$
	1050	$y_3=64.625+1.625X_1+1.625X_2+1.125X_3+1.125X_1X_3+1.125X_2X_3+1.125X_1X_2X_3$
$\sigma_{0.2}$, MPa	850	$y_1=142.15+6X_1+6X_2+6.5X_3+2.65X_1X_3+2.65X_2X_3+6.5X_1X_2X_3$
	950	$y_2=84.5+22.5X_1+22.5X_2-10X_3-10X_1X_2X_3$
	1050	$y_3=26.75-0.25X_1-0.25X_2+2.75X_3-0.25X_1X_3-0.25X_2X_3+2.75X_1X_2X_3$
δ, %	850	$y_1=58.335-2.85X_1-2.85X_2+1.635X_3-0.15X_1X_3-0.15X_2X_3+1.635X_1X_2X_3$
	950	$y_2=57.175-9.975X_1-9.975X_2+9.925X_3-1.625X_1X_3-1.625X_2X_3+9.225X_1X_2X_3$
	1050	$y_3=60.525-3.15X_1-3.15X_2+7.075X_3-2.2X_1X_3-2.2X_2X_3+7.075X_1X_2X_3$
ψ, %	850	$y_1=81.97-5.995X_1-5.995X_2+0.205X_3-0.57X_1X_3-0.57X_2X_3+0.205X_1X_2X_3$
	950	$y_2=79.1-14.525X_1-14.525X_2+1.225X_3-1.05X_1X_3-1.05X_2X_3+1.225X_1X_2X_3$
	1050	$y_3=86.65-11.675X_1-11.675X_2-5.8X_3-4.825X_1X_3-4.825X_2X_3+5.8X_1X_2X_3$

The resulting regression equations were decoded using the zero level factors and their variation intervals.

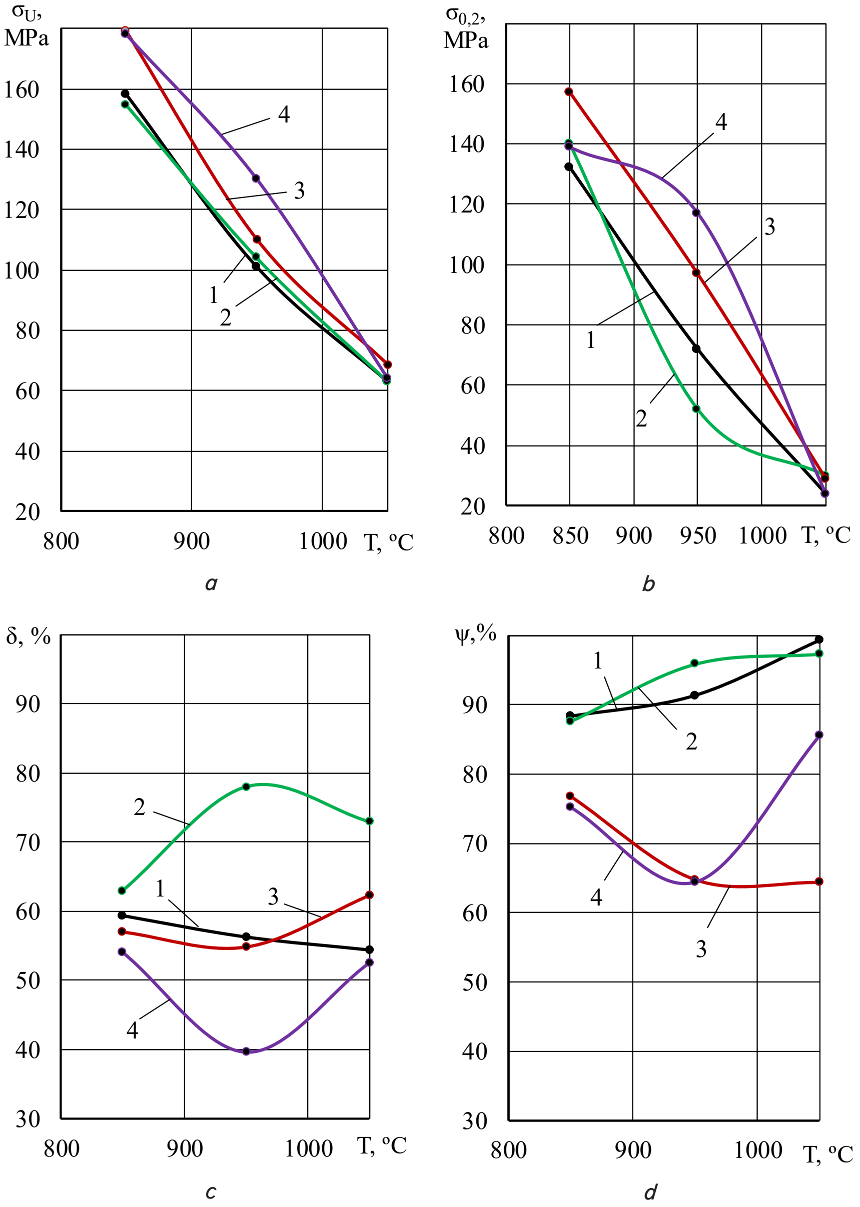
In order to study the microstructure of steels, their micro sections were made. A titanium herbalist was used for pickling the sections. The structure of steels was studied using a horizontal metallographic microscope MYM-8.

X-ray spectral method of analysis was used to determine the composition of special carbides in the steel structure.

5. Results of investigating the influence of alloying on the high-temperature mechanical properties and microstructure of steels

5. 1. Determination of the mechanical properties of the studied steels in the temperature range of 850...1050 °C

The results of tests of the mechanical properties of the studied steels are shown in Fig. 1.



a *b*

c *d*

Fig. 1. Mechanical properties of steels: 1 — 11Cr13Ni13MoNb; 2 — 11Cr13Ni16MoNb; 3 — 14Cr16Ni17MoNb; 4 — 14Cr16Ni13MoNb: *a* — yield strength; *b* — tensile strength; *c* — relative elongation; *d* — relative constriction

At 950 °C, the strength limit and yield limit of 14Cr16Ni13MoNb steel exceeded similar indicators of other alloys by 30 MPa and 70 MPa, respectively (Fig. 1, *a*, *b*). The plasticity of the studied steels also remains at a sufficiently high level (Fig. 1, *c*, *d*).

5. 2. Decoded regression equations of the influence of alloying elements on the mechanical properties of steel

After decoding the regression equations, the dependences of the mechanical properties of the test steels on the chemical composition and different test temperatures were built (Table 5).

Table 5

Regression equations of dependences of mechanical properties of steels on chemical composition and test temperatures

T, °C	Mechanical properties	Equation	R^2	F
850	σ_U, MPa	$\sigma_U=141.625-431.25C+5.5Cr-3.775Ni+28.75CNi$	0.989226	45.9
	$\sigma_{0.2}$, MPa	$\sigma_{0.2}=167.5-993.75C+3Cr-4.7Ni+66.25CNi$	0.917646	5.57
	δ, %	$\delta=60.6975+56.21C-1.425Cr+1.2675Ni-3.75CNi$	0.750816	6.027
	ψ, %	$\psi=151.0075-213.75C-2.9975Cr-1.6075Ni+14.25CNi$	0.991051	55.37
950	σ_U, MPa	$\sigma_U=-181.25+2156.25C+4.375Cr+15.125Ni-143.75CNi$	0.741067	1.43
	$\sigma_{0.2}$, MPa	$\sigma_{0.2}=-9.25+11.25Cr-5Ni$	0.741067	10.125
	δ, %	$\delta=-10.325+609.375C-4.9875Cr+9.4875Ni-40.625CNi$	0.985897	34.95
	ψ, %	$\psi=131.6+393.75C-7.2625Cr+3.7625Ni-26.25CNi$	0.994838	96.36
1050	σ_U, MPa	$\sigma_U=94.625-421.875C+0.8125Cr-2.8125Ni+28.125CNi$	0.755287	1.54
	$\sigma_{0.2}$, MPa	$\sigma_{0.2}=7.8875+0.9375C-0.125Cr+1.3825Ni-0.0625CNi$	0.99187	61
	δ, %	$\delta=-67.9125+825C-1.575Cr+10.1375Ni-55CNi$	0.92533	6.19
	ψ, %	$\psi=0.2375+1809.375C-5.8375Cr+11.575Ni-120.625CNi$	0.879516	3.65

Note: $F_{table}=119$.

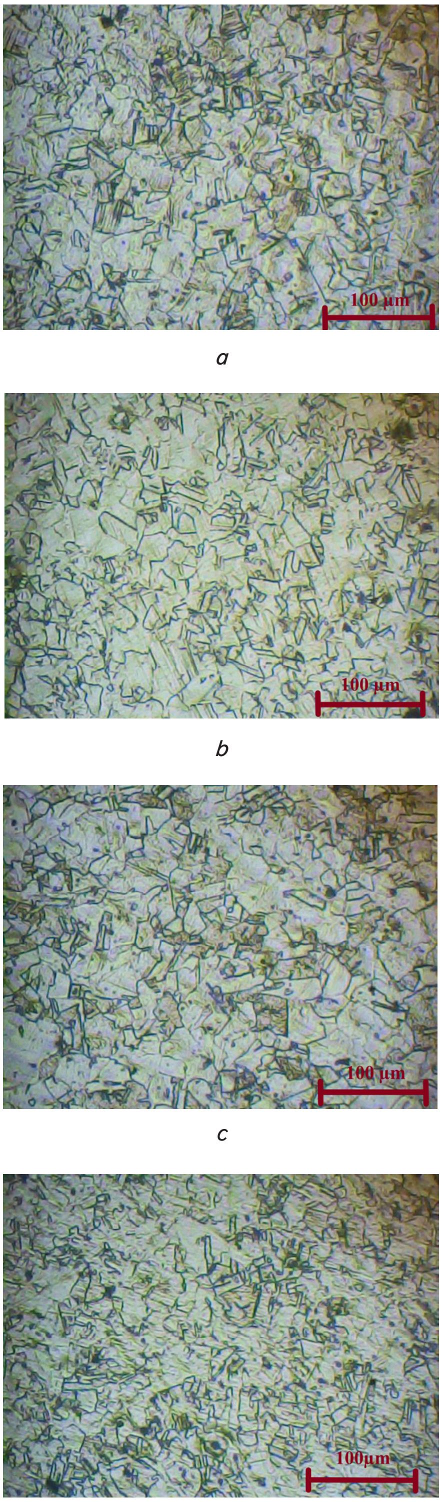
To assess the effect of elements on mechanical properties, it is necessary to substitute the values of the quantitative content of chromium, carbon, and nickel in the equation. At the same time, each resulting term indicates the quantitative impact of a particular element.

5. 3. Determination of the structure of steels after a long exposure at operating temperature

After a metallographic study, microstructures of steels were obtained (Fig. 2, 3).

Steels 11Cr13Ni13MB and 11Cr13Ni16MoNb have a similar microstructure but the first steel has larger grains than the second. Steel 14Cr16Ni13MoNb has much smaller grains compared to steel 14Cr16Ni17MoNb.

Steel 14Cr16Ni13MoNb has a finer and more even-grained structure than steel 11Cr13Ni13MoNb. In addition, the grains of steel 14Cr16Ni13MoNb have a more rounded shape.



a

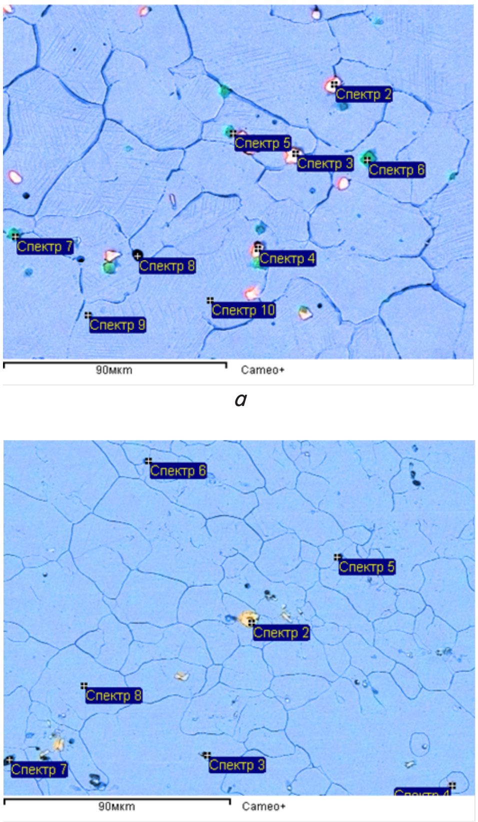
b

c

d

Fig. 2. Microstructures of steels at 950 °C, ×300: *a* – 11Cr13Ni13MoNb; *b* – 11Cr13Ni16MoNb; *c* – 14Cr16Ni17MoNb; *d* – 14Cr16Ni13MoNb

Decoding of spectra from excess phases in Fig. 3 showed that they correspond to a solid solution and niobium carbides.



a

b

Fig. 3. Microstructures of steels: *a* – 11Cr13Ni13MoNb; *b* – 14Cr16Ni13MoNb

6. Discussion of results of the effect of alloying on the microstructure and mechanical properties of steels

Our research results are explained by the fact that the four experimental steels have different chemical compositions (Table 1). The influence of each alloying element on the mechanical properties (Fig. 1) and the microstructure of each steel (Fig. 2, 3) are quantitatively indicated by the coefficients of the regression equations (Table 4).

At 850 °C and 950 °C, the values of the influence coefficients of carbon and chromium in the equations for the strength and yield limits have the same and largest values in each of the equations. This indicates that it is these elements that provide the strengthening effect of the solid solution. Nickel has a smaller (than carbon and chromium) and negative effect on the strength limit at 850 °C and 950 °C. The effect of nickel on the yield strength at 950 °C is negative due to the increase in nickel diffusion.

At a temperature of 1050 °C, the coefficients of the yield strength regression equation for carbon and chromium decrease and become negative because their carbides dissolve. The coefficient for nickel becomes the largest in the equation and is positive; this is explained by the fact that nickel stabilizes the austenitic structure of steel. This indicates a gradual change in the mechanism of influence on heat resistance from carbide to solid solution.

Consider the equation for plastic properties at temperatures of 850...1050 °C. The influence coefficients of carbon and chromium in each of the equations have a negative sign because they contribute to the reduction of plasticity. Nickel has a positive multiplier, thereby increasing plastic properties.

Steels 11Cr13Ni13MoNb and 11Cr13Ni16MoNb have a similar structural state (Fig. 2, *a*, *b*). At a temperature of 950 °C, steel 14Cr16Ni13MoNb has a fine-grained structure, while steel 14Cr16Ni17MoNb is characterized by a larger grain size (Fig. 2, *c*, *d*). In addition, steel 14Cr16Ni13MoNb has grains of a more rounded shape than steel 11Cr13Ni13MoNb (Fig. 3, *a*, *b*), which contributes to lower brittleness.

Thus, the carbon content in steel should be at the level of 0.14 %, it contributes to the stabilization of austenite and expands the possibility of alloying the solid solution with ferrite-forming elements while preserving the austenite structure.

The chromium content should be at the level of 16 %, which will ensure the necessary mechanism of dispersion hardening of steel.

The nickel content of 13 % ensures the austenitic structure of the steel, regardless of the introduction of ferrite-forming elements into the solid solution. Increasing the nickel content to 17 % reduces the solubility of carbon in austenite, which leads to a deterioration of the yield strength of steel.

Heat resistance is preserved not only due to the strengthening of the matrix with chromium carbides ($Me_{23}C_6$), but also partially due to heat-resistant carbides of niobium and molybdenum.

Microalloying of steel with niobium (in the range of 0.20–0.40 wt. %) and molybdenum (in the range of 0.17–0.40 wt. %) leads to the formation of MoC and NbC carbides, which prevent the excessive formation of $Cr_{23}C_6$ carbides.

The selected limits of the content of carbon, chromium, and nickel make it possible to obtain the most optimal structural state of heat-resistant steel – 14Cr16Ni13MoNb and ensure its high operational properties in the temperature range of 850–1050 °C.

Unlike steel [5], which has a yield strength of 68 MPa at 950 °C, steel 14Cr16Ni13MoNb has a better value – 117 MPa. This becomes possible thanks to the increased carbon content – from 0.10 to 0.14 %.

The obtained solutions make it possible to create steel for the aerospace industry and magnetothermal production of spongy titanium with increased service characteristics.

The limitations of the study are that steel retains its high mechanical properties only in the temperature range of 850–1050 °C. With a further increase in temperature, it will not satisfy the required heat resistance indicators.

The disadvantages are the difficulty of ensuring uniform distribution of chemical elements in steel in industrial conditions.

The development of the research consists in the development of the necessary mode of thermo-mechanical processing of steel for further improvement of its service properties.

7. Conclusions

1. Steel 14Cr16Ni13MoNb has the best mechanical properties at temperatures of 950 °C due to the rational composition of C-Cr-Ni components.
2. Regression equations confirmed that carbon and chromium have the greatest positive effect on the heat resistance of steels, while nickel provides the austenitic structure of steel.
3. At a temperature of 950 °C, the heat resistance of 14Cr16Cr13MoNb steel is positively affected by alloying of

the solid solution with carbon and chromium and partially heat-resistant special carbides MoC and NbC.

Conflicts of interest

The authors declare that they have no conflicts of interest in relation to the current study, including financial, personal, authorship, or any other, that could affect the study and the results reported in this paper.

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Data availability

The data will be provided upon reasonable request.

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