

CHAPTER 3

**REGULARITIES OF THE FORMATION OF STRUCTURE,
PHASE COMPOSITION AND TRIBOLOGICAL PROPERTIES
OF HEAT-RESISTANT CHROMIUM-NICKEL ALLOYS "NIKORIN"****ABSTRACT**

In this work, new solutions have been obtained for a scientific and practical problem, which consists in increasing the complex of tribological properties and heat resistance of chromium-nickel alloys for modern mechanical engineering. The composition of heat-resistant chromium-nickel alloys is investigated. The distribution of alloying elements between phases and structural components is studied, the dependence of the change in mechanical properties on the parameters of the structure and phase composition is established.

Calorimetric analysis of the alloys under study during heating/cooling is carried out. The crystallization/melting temperatures of the nickel-chromium alloy "Nikorin" were determined. The structure, phase composition, microhardness of the structural components of the heat-resistant chromium-nickel alloy "Nikorin" after quenching from various temperatures have been studied. The tribological properties of chromium-nickel alloys in the cast state are investigated.

KEYWORDS

Chromium-nickel alloys, structure, austenite, hardness, wear rate, wear resistance index.

Nowadays, chromium and nickel-based alloys – the most complex, have amazing properties of all alloys – are widely used to make parts that are exposed to the most heat. They are used at high homologous temperatures compared to any conventional alloy system.

The main group of such alloys is made up of heat-resistant alloys with high long-term strength and creep limits at a temperature of 700 °C and above. In terms of chemical and phase composition, heat-resistant high-chromium alloys are very diverse, since chromium and nickel have a large number of extremely soluble alloying elements that have a positive effect on their heat resistance. This can explain the significant possibilities and reserves for increasing the heat resistance of high-chromium alloys. As the content of chromium, titanium and aluminum in these alloys increases, their characteristics of long-term strength increase, since these elements

form reinforcing excess phases $\eta(\text{Ni}_3\text{Ti})$, $\gamma'(\text{Ni}_3\text{Al})$, $\text{Ni}_3(\text{Al}, \text{Ti})$, as well as chromium and titanium carbides, which strengthen the alloys. Complications of the chemical composition of high-chromium and chromium-nickel alloys with promising refractory alloying elements in the presence of a sufficient number of hardening phases further increase the heat resistance of high-chromium and chromium-nickel alloys.

From the point of view of the dependence of the technological properties of nickel on the chromium content in the solid solution, attention was drawn to the fact that as the chromium concentration increases, the plasticity of the alloys decreases. Alloys with chromium content above 30 % are quite tough, therefore their processing in a cold state becomes complicated, although the limiting solubility of chromium in nickel is 34–35 %. The combination of physicochemical, electrical and technological properties of chromium-nickel alloys has determined the areas of practical application of alloys of this system.

At present, heat-resistant chromium-nickel alloys have come to one of the first places in terms of their importance and are widely used in various fields of technology.

The experience of domestic and foreign industry and special studies indicate that the most promising structural materials for operation under friction at elevated temperatures and various loads are nickel-chromium alloys "Nikorin". These alloys are used for the manufacture of rolling tools (gauges, mandrels for hot tube rolling mills). The most outstanding properties of high-chromium irons are high wear resistivity (with a low temperature coefficient) and high-temperature strength. This determined a specific and fairly wide area of application of these alloys. They are used for the manufacture of rolls, armor plates, blades of shot blasting machines, grinding balls, rolling tools (gauges and roll mandrels) [1]. The stability of the "Nikorin" alloy mandrels significantly exceeds the stability of the high-chromium iron mandrels in the as-cast condition.

In this regard, the purpose of this work is to study the structure, phase composition and wear resistance of the nickel-chromium alloy "Nikorin".

3.1 A CONTEMPORARY VIEW ABOUT THE PATTERNS OF STRUCTURE FORMATION, PHASE COMPOSITION, MECHANICAL AND TRIBOLOGICAL PROPERTIES OF HIGH-CHROMIUM AND CHROMIUM-NICKEL ALLOYS

3.1.1 PATTERNS OF STRUCTURE FORMATION IN HEAT-RESISTANT HIGH-CHROMIUM AND CHROMIUM-NICKEL ALLOYS

3.1.1.1 FEATURES OF STRUCTURE FORMATION IN HIGH-CHROMIUM ALLOYS

Increasing the service life of machine parts that wear out quickly is the most important problem in modern mechanical engineering. The short service life of parts reduces the economic efficiency of many machines and industrial equipment and leads to irrecoverable loss of metal.

Modern high-chromium alloys are complex-alloyed multicomponent alloys, differing in structure and properties. They represent a separate group of industrial alloys, the hardening of which forms a carbide phase. It is this, in combination with a certain type of matrix, which determines the specific properties of high-chromium alloys and at the same time creates significant difficulties in the production and operation of these alloys.

High chromium alloys have a complex metal matrix structure. Depending on the chemical composition, mass, cooling rate of the ingots or the type of heat treatment, it consists of austenite, martensite and their decomposition products, the ratio of which can vary within wide limits. Until now, no clear position has been formed that determines the optimal phase composition and structure of the metal matrix, both in terms of wear-resisting properties and mechanical properties (impact hardness and strength). Some authors take the martensite matrix as the optimal one, which provides maximum wear-resisting properties [1–3]. Other authors [4, 5] give preference to metastable austenite. There is evidence that the ultimate wear-resisting properties can be obtained with a mixed microstructure – martensite and metastable austenite [6, 7].

A similar picture is observed in the analysis of literature sources, in which the effect of the matrix on the strength characteristics of high-chromium alloys was studied. According to various studies, it is believed that the best is the austenite-martensite base [6], purely austenitic [4], martensitic [8]. There is an opinion [9] that the structure of the metal matrix does not affect the strength of high-chromium alloys at all. Naturally, the complexity of the structure, inhomogeneity of the composition of the solid solution, various methods of obtaining and testing castings lead to ambiguous and contradictory conclusions about the dependence of properties on the type of structure of the metal base.

High chromium alloys are widely used in industry. Parts made of high-chromium alloys operating under abrasive and shock-abrasive wear conditions, both at room temperature and at elevated temperatures. Industrial high-chromium alloys are complex multicomponent systems that contain permanent impurities (phosphorus, sulfur, oxygen, hydrogen, nitrogen), as well as various alloying elements.

By changing the chemical composition, the type of modifying and alloying impurities, the conditions of hardening and cooling in the solid state, it is possible to regulate the physicomechanical and operational properties of wear-resistant high-chromium alloys over a wide range.

The wear resistance of high-chromium alloys is determined by the amount, size, morphology, microhardness of eutectic and secondary carbides and a metal base – the main structural components of these alloys [10].

In wear-resistant high-chromium alloys based on the Fe-C-Cr system, depending on the ratio of carbon and chromium, carbides of the $(\text{Fe}, \text{Cr})_3\text{C}$ type are formed; $(\text{Cr}, \text{Fe})_7\text{C}_3$; $(\text{Cr}, \text{Fe})_{23}\text{C}_6$. The carbide $(\text{Cr}, \text{Fe})_7\text{C}_3$, has the highest hardness, especially along the axis of its crystal lattice [11].

The high wear resistance of the solid phase (in particular, carbides in high-chromium alloys) can manifest itself only when this phase is firmly fixed in the matrix and the degree of plastic deformation of the matrix is low [11]. With weak fixation of even very hard carbides in a soft plastic (for example, ferrite or pearlite) matrix, the high potential stability of carbides during abrasive wear is not realized.

When choosing the alloy composition, first of all, one should take into account the value of the ratio of carbide-forming elements and carbon. This ratio should be such as to ensure that the required amount of carbides is obtained in the alloy. To obtain the most alloyed carbides, the ratio of the carbide-forming element content to the carbon content must reach certain (critical) values. This ratio for chromium is (at. %) $\text{Cr/C} \geq 4.5$ [12]. It is quite obvious that not every increase in the content of carbon or carbide-forming elements will unambiguously increase wear resistance. In complex-alloyed high-chromium alloys, the type of carbides depends on the affinity of the carbide-forming elements to carbon and their quantitative content. With a close atomic content, the leading element in the formation of carbides is the element that is most similar to carbon.

The experience of domestic and foreign industry and special studies indicate that high-chromium and chromium-nickel alloys are the most promising structural materials for operation under shock loads, as well as under friction conditions at room and elevated temperatures.

3.1.1.2 FORMATION OF THE STRUCTURE OF CHROMIUM-NICKEL ALLOYS

At present, nickel-based alloys are used as heat-resistant materials intended for operation at temperatures from 700 to 1100 °C. Heat-resistant nickel alloys, in terms of their importance, have come out on top and are widely used in various branches of technology. This is explained by the fact that heat-resistant nickel alloys successfully combine high heat resistance, scale resistance and manufacturability.

Alloying nickel with chromium (up to 50 %) increases its stability in oxidizing environments, for example, in boiling solutions of nitric and chromic acids.

The main advantage of nickel-chromium alloys (>20 % Cr) is their high corrosion resistance in nitric acid solutions in the presence of a fluorine ion and higher heat resistance at high temperatures.

Many works have been devoted to the study of the Ni-Cr phase diagram. By this time, the most acceptable diagram seems to be given by Taylor and Floyd, which is characterized by a long-focus transformation with the limiting solubility of the components. **Fig. 3.1** shows a refined phase diagram of the Ni-Cr eutectic type system [13], according to which the eutectic crystallizes at 1345 °C and 49 % Cr. On the nickel side, there is a wide region of a γ -solid solution with an fcc lattice (structure of the A1 type, $a=0.358\text{--}0.359$ nm). The solubility of chromium in nickel at 1345, 1100, 600, and 20 °C is 47, 43, 35 and 30 %, respectively. The chromium-based alpha solid solution (structure of type A2, $a=0.288$ nm) has a bcc lattice and exists in a much smaller concentration range than the γ -solid solution.

For a long time, there remained controversial questions about the existence in the Ni-Cr system of a eutectoid transformation due to high-temperature allotropic transformation in chromium, as well as about the possibility of the formation of the σ -phase. For the first time, Blum and Grant, on the basis of X-ray and microscopic studies, concluded that eutectoid transformation occurs in the Ni-Cr system at a temperature of about 1180 °C and 35 % Ni. The assumption about

the possibility of the formation of the σ -phase in the Ni-Cr system was first expressed by A. Sully on the basis of theoretical considerations

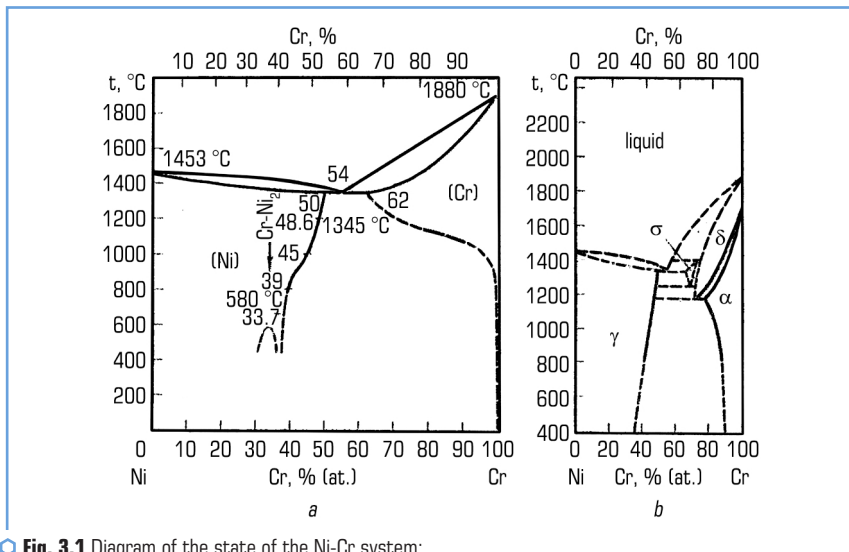


Fig. 3.1 Diagram of the state of the Ni-Cr system: *a* – according to the data [13]; *b* – according to the data [14]

Yukawa, Haida and others [14] investigated by X-ray analysis particles of Ni-Cr alloys with a size of 10–100 nm, obtained by evaporation of alloys in argon under pressure and proposed a state diagram of Ni-Cr, in which, in addition to α - and γ -solid solutions due to the mutual solubility of nickel and chromium, the established limits for the existence of the σ -phase as intermetallic compounds, and the δ -phase (high-temperature allotropic modification of chromium), as shown in **Fig. 3.1, b**. In this case, the σ -phase has a β – U type structure (space group D_{6h}^8 , $a=0.881$ – 0.882 nm). The structure of the δ -phase is assigned to type A15, $a=0.458$ nm.

Another feature of the Ni-Cr system is that in a wide range of chromium concentrations (from 19.0 to 36 % (at)), the γ -solid solution is ordered with the formation of a Ni_2Cr superstructure [15, 16]. Transition temperature order – disorder alloys from 25.0; 29.2; 33.3 (stoichiometric composition) and 36.6 % (at.) Cr, respectively 540, 580, 590, 585 °C [15–17].

Ye. S. Vintaykin and H. H. Urushadze [18–20] proposed a peritectoid type of state diagram at low temperatures. At about 580 °C, an ordered phase (Ni_2Cr), a disordered γ -solid solution, and an α -phase can be in equilibrium.

The study of the structure of the ordered phase, carried out by the methods of X-ray diffraction, neutron diffraction [16, 17], and electron microscopy [21], showed that it belongs to the Pt_2Mo type. This structure is characterized by a layered arrangement of atoms:

parallel to the $\{110\}$ initial fcc lattice, two layers of nickel atoms and a layer of chromium atoms alternate. The unit cell of an ordered Ni_2Cr alloy is orthorhombic (space group $D_{2h}^{25} = \text{Immm}$), contains 4 nickel atoms and 2 chromium atoms. The unit cell parameters of the ordered phase are determined by the relations:

$$a = 3a_{\text{fcc}}/\sqrt{2}; \quad b = a_{\text{fcc}}/\sqrt{2}; \quad c = a_{\text{fcc}}. \quad (3.1)$$

The Curie point of nickel-chromium alloys decreases with increasing chromium content and reaches room temperature at about 8 at. % Cr. Therefore, under normal conditions, none of the industrial alloys has magnetic properties.

3.1.2 STRUCTURAL TRANSFORMATIONS IN CHROMIUM-NICKEL ALLOYS

In nickel alloys with 30–50 % Cr, which are of interest as a basis for heat-resistant materials characterized by high stability at elevated temperatures, the following structural transformations are possible:

- 1) the formation of intermetallic γ' -phases from a fcc lattice of the $\text{Ni}_3(\text{Al}, \text{Ti})$ type;
- 2) the order of the γ -solid solution with the formation of the Ni_2Cr superstructure in alloys with 30–33 % Cr, or 33–36 % (at) at temperatures below 580 °C;
- 3) precipitation of the α -phase from the γ -solid solution in alloys with >39 % Cr at 600–900 °C;
- 4) carbide formation is due to the temperature dependence of the solubility of carbon in the γ -solid solution. The main carbide is Me_{23}C_6 which precipitates mainly along the grain boundaries in the temperature range 600–1000 °C with short holding times.

3.1.2.1 FORMATION OF γ' -PHASE

The amount and nature of the distribution of the γ' -phase in heat-resistant chromium-nickel alloys depend on alloying, cooling rates during solidification of the casting and heat treatment [17].

The γ' -phase is a unique intermetallic phase. When interacting with dislocations, it contributes to the strengthening of the antiphase limits of the γ - γ' -alloy. By the way, the strength of the γ' -phase increases with increasing temperature. In addition, the hereditary plasticity of the γ' -phase prevents strong embrittlement, in contrast to strengthening by phases that have a higher hardness, for example, carbides. Also, its action is directly opposite to strong embrittlement during the formation of brittle σ - or Laves phases. The γ' -phase was first observed in the form of spheroidal particles, then cubic; later it was found that their shape is associated with the magnitude of the mismatch between the lattice parameters of the matrix and precipitates.

According to the observations of Hagel and Beatti, γ' -particles have a spherical shape with a lattice mismatch of 0–0.2 %, cubic – with a lattice mismatch of 0.5–1.0 % and a lamellar shape with values of more than 1.25 % [22].

3.1.2.2 ORDERING BY Ni_2Cr TYPE

Alloys containing up to 39 % Cr are single-phase. However, in the range of chromium concentrations of 30–33 %, there is an ordered Ni_2Cr phase. The ordering temperature in an alloy of stoichiometric composition Ni + 30.5 % Cr is 590 °C. In the quenched state, the structure of an alloy of stoichiometric composition is characterized by short-range order in the arrangement of atoms.

For the development of ordering, substantial hypothermia (~90 °C) is required. However, even at 500 °C, which corresponds to the maximum speed of improvement, the process is sluggish: to achieve an equilibrium degree of the order of Ni_2Cr at 500 °C, it takes ~500 h ordering in the Ni_2Cr system is a first-order phase transition and proceeds according to a homogeneous mechanism [23–25].

The development of the ordering process according to the Ni_2Cr type leads to a significant increase in the strength properties [24] and to the electrosupport [25].

3.1.2.3 FORMATION OF α -PHASE

In accordance with the equilibrium phase diagram of Ni-Cr [26], alloys with 30–40 % Cr (0.03 % C and 0.1 % Si) in the state of quenching from 1100 °C in water have the structure of a γ -solid solution with a grain size No. 5–6 and hardness ~210 HB. In the structure of alloys containing more than 43 % Cr, along with the γ -solid solution after quenching from 1100 °C, the α -phase is present. The amount of the latter increases with increasing chromium content. In an alloy with 50 % Cr, the α -phase appears in the form of relatively large particles with a size of 20–70 and 1–5 microns, elongated along the rolling direction, and also in the form of separate globular precipitates less than 1 micron. The grain size in these alloys during quenching decreases approximately to No. 7 and the hardness increases to 290 HB [24].

Alloys containing more than 39 % Cr are subject to precipitation hardening. Tempering at 600–900 °C causes the decomposition of the γ -solid solution with the release of the α -phase, as well as carbides of the Me_{23}C_6 type.

The morphology, size, distribution and quantity of the α -phase are determined primarily by the content of the main element of chromium in the alloy and the temperature-time regime of tempering; as well as the presence of additional alloying elements in the alloy (Mn, Fe, Ti, Al, C, etc.).

The type of precipitation of the α -phase in Ni – 40–50 % Cr alloys is classified as cellular or discontinuous.

In alloys with 40 % Cr, boundary cellular precipitates of the α -phase (colonies of "pearlite-like" structure) are observed in the structure after 1:00 tempering at 700–800 °C, which practically does not affect the change in the hardness of the quenched alloy (**Fig. 3.1, b**). Tempering at 600–900 °C of alloys with 45, 50 and 55 % Cr causes intense decomposition of the nickel-chromium solid solution, which is accompanied by abundant precipitation of the α -phase throughout the volume [24, 15, 16]. At the same time, in an alloy with 45 % Cr, after 700–800 °C tempering, there is mainly a lamellar α -phase, the size of the plates and the distance between them increase with an increase in the tempering temperature. In an alloy with 50 % Cr, a predominantly globular α -phase is precipitated, which grows larger with increasing temperature. The separation of the α -phase from the supersaturated solid solution leads to an increase in the hardness of the alloy (**Fig. 3.2**).

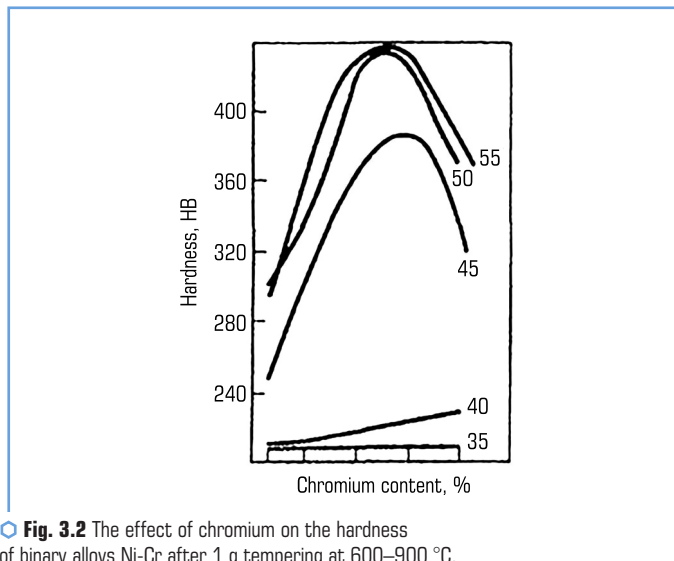


Fig. 3.2 The effect of chromium on the hardness of binary alloys Ni-Cr after 1 g tempering at 600–900 °C. Quenching from 1100 °C in water (figures in Cr % curves) Source [16]

3.1.2.4 CARBIDE FORMING

Depending on alloying and heat treatment, carbide phases of various types are formed in heat-resistant alloys. Most of the carbon is bound in the primary low-soluble titanium carbide of the TiC type, and in the presence of nitrogen – in titanium carbonitrides Ti(CN). Titanium carbides form a eutectic with nickel, which has a lower melting point, which is the reason for their distribution in the middendritic volumes.

The distribution of titanium carbides and nitrides in the cast structure of heat-resistant chromium-nickel alloys depends on the crystallization conditions (pouring temperature and cooling rate). When pouring from high temperatures and slow cooling, carbides and carbonitrides are coarser and, standing out along the grain boundaries, form a network (frame) when rapidly cooling, they are precipitated as smaller formations. On thin sections of titanium carbide or carbonitrides, they look like cubic or polyhedral pink inclusions.

In addition to titanium carbides and carbonitrides, carbon in cast nickel alloys forms double carbides. These carbides are precipitated into carbides of the Cr_{23}C_6 type predominantly along the crystal boundaries in the form of a mesh or rim, which negatively affects the ductility of the alloys. The shape of the distribution of double carbides depends on the heat treatment mode: the higher the hardening temperature, the coarser the precipitation [26, 27].

3.1.3 CARBIDES IN HEAT-RESISTANT CHROMIUM-NICKEL ALLOYS

The role of carbides in heat-resistant alloys is complex and dynamic. Nowadays, most researchers come to the conclusion that carbides actually have an important and beneficial effect on the long-term strength at high temperatures. In addition, it is clear that the morphology of carbides has an effect on ductility. Also, carbides can affect the chemical stability of the matrix through the bonding of electrons interacting with carbon.

In alloyed heat-resistant nickel alloys, such carbides are formed as: MeC , Me_{23}C_6 , Cr_7C_3 and Me_6C . Their type and stability are determined by alloying – the content of carbon and strong carbide-forming elements (tantalum, hafnium, niobium, titanium, etc.), as well as the concentration and ratio in the alloy of other, less strong carbide-forming agents – chromium, tungsten, molybdenum. The morphology of carbides, as well as their size, mainly depend on the conditions of crystallization and alloying.

MeC carbides are formed in heat-resistant alloys at temperatures slightly below their solidification temperature. They exist in the form of individual particles, unevenly distributed in the alloy along the boundaries and within the grains [28, 29]. These carbides have fcc lattice and are the most durable and fairly stable phases. In carbide reactions in alloys, they serve as the main source of carbon, since they are rich in this element in comparison with other carbides. They are stable up to about 1300 °C [28]. They are formed from a simple combination of carbon and reactive or refractory metals and are characterized by a classic formula like TiC or TaC . In order of decreasing their stability, they are arranged in the series of TAC , NbC , TiC and VC . The composition of MeC carbides in real alloys includes tungsten and molybdenum, as well as nickel and chromium in smaller amounts, while the binding forces in MeC carbides weaken and decomposition reactions can occur, leading to the formation of other types of carbides.

Carbides Me_6C are formed in nickel heat-resistant alloys at temperatures of 815–980 °C. They are similar to carbide Me_{23}C_6 , but are usually formed with a high content of refractory metal.

These carbides are formed in alloys with a high content of tungsten and molybdenum and are precipitated both along the grain boundaries and within them. Carbides of the Me_6C type have a complex cubic structure. Me_6C carbides are formed when chromium is replaced by molybdenum or tungsten in other carbides. In contrast to the more stable carbide $Me_{23}C_6$, their compositions can vary within wide limits. Since Me_6C carbides are stable at temperatures higher than those of $Me_{23}C_6$ carbide, their precipitation along the grain boundaries is more preferable and can be used to control the grain size.

The chromium content of more than 8–12 % already leads to the formation in the structure of chromium-nickel alloys of the primary trigonal carbide, which has the formula Cr_7C_3 . Carbides of this type are located in chromium-nickel alloys in the form of irregularly shaped particles. Usually, in alloys with a complex composition, Cr_7C_3 carbides are unstable and can transform into $Me_{23}C_6$ during aging. With a less complex chemical composition, Cr_7C_3 carbides strengthen the structure of heat-resistant alloys.

In the Ni-Cr system, which contains more than 30 % Cr, the main equilibrium carbide phase is $Cr_{23}C_6$, which has a complex fcc lattice (of the D_{34} type) with a cube edge $a=1.060$ nm. The $Me_{23}C_6$ unit cell contains 92 metal atoms and 24 carbon atoms and consists of 8 octants, in each of which the vertices are alternately surrounded by cuboctahedra (48h-position) and simple cubes (32f-position). In addition, metal atoms can also occupy the centers of the cuboctahedron (4a-position of the cell vertex, considered) and one of the most spacious center of each octant (8c-positions). Carbide $Cr_{23}C_6$ is characterized by a very narrow homogeneity region, due to which, in terms of the ratio of metal and carbon atoms, it almost always represents a phase of stoichiometric composition [28]. Compound $Me_{23}C_6$ can be a carbide of one element, for example, chromium ($Cr_{23}C_6$); however, according to the principle of chemical indifference [29], chromium atoms can be replaced by atoms of other elements (Fe, Ni, Mn). In pure $Cr_{23}C_6$, iron can replace up to 30 at. % Cr, while occupying predominantly cubic f-positions [30].

In $Me_{23}C_6$ carbide, each carbon atom is surrounded by eight metal atoms located along the vertices of a quadrangular prism.

With insignificant decarburization, $Me_{23}C_6$ carbides can transform into bcc structures (for example, α -Cr) or into the σ -phase. The actual nature of the $Me_{23}C_6 \rightarrow \alpha$ -phase transition is determined by the structural relationship and diffusion rates.

The solubility of carbon in nickel alloys with 30–45 % Cr is small and decreases with increasing chromium content and decreasing heating temperature.

At 800 °C, the solubility of carbon in alloys with 35–45 % Cr decreases by more than an order of magnitude.

Carbides $Me_{23}C_6$ are formed during heat treatment or operation at 600–1000 °C. They stand out mainly along the grain boundaries, sometimes they can be observed along the twin line and on stacking faults.

The location and size of carbides are determined by the content of chromium, additional and impurity elements in the alloy, and by the heat treatment mode. Carbide $Me_{23}C_6$ dissolves at 1100 °C [27, 24].

In alloys with a chromium content of 30–39 % Cr after tempering at 600–900 °C for 10 min. Up to 100 hours, only particles of $Me_{23}C_6$ carbide are present at the grain boundaries, the size of which increases with increasing temperature and heating duration. In alloys containing more than 39 % Cr, along with precipitates of carbides, cellular precipitates of the α -phase based on Cr are formed. With an increase in the chromium content in the alloy (up to 50 %), the temperature-time interval of its appearance shifts towards lower temperatures and short-term exposures. So, if in an alloy with 40 % Cr after 10 hours of tempering at 600 °C, only $Me_{23}C_6$ carbide is present within the limits, then in an alloy with 50 % Cr after a similar tempering, the α -phase is also present along with carbide.

3.1.4 INFLUENCE OF ALLOYING ELEMENTS ON THE STRUCTURE AND PROPERTIES OF HIGH-CHROMIUM NICKEL ALLOYS

In terms of chemical and phase composition, heat-resistant nickel alloys are very diverse, since nickel has a greater number of extremely soluble alloying elements, which have a positive effect on its heat resistance. This can explain the significant possibilities and reserves for increasing the heat resistance of nickel alloys.

The choice of alloying elements and their optimal composition in heat-resistant nickel alloys must be approached with caution, given their effect on the various properties of the alloys. In particular, chromium, widely used for alloying nickel, which strengthens the nickel solid solution by reducing the rate of diffusion processes, must be added to heat-resistant nickel alloys in different amounts, since the alloys are intended for different operating temperatures.

Chromium is added to heat-resistant chromium-nickel alloys to increase oxidation resistance at high temperatures, and the more, the higher its content in the alloys.

An increase in the chromium content in chromium-nickel alloys strengthens the alloys of this system: the creep resistance at high temperatures approximately doubles.

In alloys with an austenitic matrix, chromium increases the binding energy of the atoms of the crystal lattice of the γ -solid solution, somewhat increases the heat-resistant properties and the recrystallization temperature of doped austenite.

In nickel heat-resistant alloys, chromium reduces the amount of the γ' -phase that is formed during decomposition, worsens the thermal stability of the γ' -phase and thereby negatively affects the durability and ductility. In addition, more than other elements, chromium extends the crystallization range of alloys, worsens their manufacturability. At the same time, a decrease in the chromium content leads to an inevitable deterioration in heat resistance.

Chromium forms with carbon a number of carbides ($Cr_{23}C_6$, Cr_7C_3), which, as a result of quenching and aging, precipitating in a γ -solid solution in a highly dispersive state, strengthen austenite alloys.

Chromium carbides have a relatively low thermal stability, and therefore the increase in the heat resistance of austenitic alloys due to only such carbides is insufficiently effective.

Manganese promotes the expansion of the γ -area of the nickel-chromium system, forming alloys with an austenitic structure. Manganese as iron, cobalt and copper forms a continuous solid solution with nickel at 1000 °C.

As an austenite-forming element, manganese is less effective than nickel, especially with high chromium content. Therefore, it is advisable to introduce small amounts of carbon or nitrogen together with manganese, which promotes the strong development of precipitation hardening processes [25].

Manganese reduces the critical cooling rate and increases quench susceptibility and cross-sectional improvement.

Manganese somewhat increases the solubility of chromium carbides (Cr_7C_3) in austenite, which also depends on the chromium content [26].

Aluminum. As the content of titanium and aluminum in Ni-Cr alloys increases, their characteristics of long-term high-temperature strength increase, since these elements form, reinforcing, excess phases. In this case, aluminum has a strong effect on the amount of γ' -phase in nickel-chromium-titanium alloys. With an increase in the amount of aluminum, the content of alloying elements in the γ' -phase increases sharply. For example, when the aluminum content is 0.6 %, the amount of γ' -phase is 5.1 %; at 1.7 % Al – 25.5 %; at 4.1 % Al – 42.5 %. According to the Ni-Al-Ti phase diagrams, aluminum has a significant effect on the solubility of titanium in nickel. So, at 1150 °C, the titanium solubility in nickel decreases from 13 to 8 % with an aluminum content of up to 5 %. At 750 °C, this phenomenon is even more pronounced. Titanium has a similar effect on the solubility of aluminum in nickel.

Aluminum, introduced into complex alloyed nickel-chromium heat-resistant alloys, causes a very strong effect on the increase of heat-resistant properties of these alloys, and the more, the higher its content. However, if the amount of aluminum is more than 3–4 %, then difficulties arise during forging, which limits the possibility of a stronger alloying with this element of hard-to-form heat-resistant alloys.

Titanium. The region of solid solutions rich in nickel at 1150 °C extends in the nickel-titanium binary system up to 13 % titanium, and in the nickel-chromium-titanium ternary system at 20 % Cr up to about 10 % Ti.

With decreasing temperature, the solubility of titanium in the binary and ternary systems drops sharply. So, at 750 °C in the Ni-Ti binary system the solubility of titanium is already 10 %, and in ternary alloys with 20 % Cr – about 4 %. Apparently, with a further decrease in temperature, the titanium solubility in the Ni – Cr solid solution drops even more sharply, and alloys with a titanium content of less than 4 % at room temperature may already be two-phase. The introduction of up to 3 % titanium into chromium-nickel alloys promotes the formation of highly dispersed intermetallic phases at moderate temperatures, thereby increasing the resistance of the alloy to plastic deformation and increasing the long-term strength at 800 °C to 15 kg/mm².

It was found that the characteristic temperature, long-term strength limits, and internal friction increase with the penetration of titanium into a chromium-nickel alloy. However, the composition of heat-resistant alloys, in addition to titanium, as a rule, includes aluminum [27].

Carbon. Despite the small amount of carbon in heat-resistant nickel alloys, it has a great influence on their structure and properties, as it forms not only hardly soluble titanium carbide, but also chromium carbides Cr_{23}C_6 , Cr_7C_3 , Me_6C .

In chromium-nickel alloys, part of the carbon is bound in primary titanium carbides TiC or carbonitrides (TiCN), and the other part in chromium carbonitrides of the CrCN type.

The introduction of carbon into chromium-nickel alloys promotes the stabilization of austenite in the $\gamma \rightarrow \alpha$ transformation ratio and thereby expands the possibilities of alloying the solid solution with ferrite-flammable elements while maintaining the austenite structure of Cr_{23}C_6 , Cr_7C_3 .

Carbon has a large and variable with temperature solubility in γ -solid solution compared to solubility in α -solid solution, providing austenitic alloys with the ability to precipitation hardening as a result of heat treatment and increasing their mechanical properties at room and elevated temperatures.

Strengthening of austenitic alloys with carbon occurs due to the formation of chromium carbides of the Me_{23}C_6 type. However, the great tendency of chromium carbides to coagulate does not allow obtaining a large strengthening effect at high temperatures, although the mechanical properties at room temperature are greatly enhanced.

The greatest strengthening effect at high temperatures is achieved as a result of complex alloying, when several alloying elements are simultaneously introduced into the solid solution.

Iron. The study of the effect of iron in nickel-based alloys made it possible to establish that in heat-resistant alloys of a simpler composition, it is possible to admit the content of 5–8 % iron in the EI437BU alloy, the introduction of 5 % iron practically does not change the heat resistance and technological properties.

The expansion of the boundaries of the iron content up to 5 % in alloys of the EI437 type allows using ferrochrome instead of metallic chromium in their smelting. Foreign nickel-based nimonic alloys 80A, 90.95 contain up to 5 % Fe, and nimonic alloys 100 and 105 – up to 10 % [31].

In complex-alloyed heat-resistant alloys, the introduction of iron somewhat worsens the heat-resistant properties and its content should not exceed 1–2 %.

Silicon, which is present in heat-resistant nickel alloys with intermetallic reinforcement, has a negative effect on long-term strength. The higher the aluminum and titanium content, the lower the silicon content should be.

According to some data, for a number of alloys there are limitations in technical conditions: the silicon content is allowed no more than 0.3–0.5 %.

The example of the EI787 alloy clearly shows the negative effect of silicon.

Vanadium. Vanadium is sometimes added to austenitic alloys in small amounts (up to 0.3 %) to improve technological characteristics.

The introduction of a large amount of vanadium into these alloys does not have a positive effect on the heat-resistant properties, which is associated with the weakening of the binding energy of atoms in the crystal lattice of the γ -solid solution. In austenitic alloys, vanadium is added

together with carbon, which greatly increases the tendency of the alloy to precipitation hardening. As a result of aging, the strength characteristics of the alloy increase both at room temperature and at elevated temperatures, the plastic properties decrease with an increase in the content of vanadium and carbon.

With the introduction of vanadium and carbon, the strengthening proceeds due to the intensive formation of highly dispersed vanadium carbides VC and chromium carbides Cr_{23}C_6 with some mutual solubility of vanadium in chromium carbide and chromium in vanadium carbide.

Excessive alloying of alloys with vanadium and carbon promotes the intensive formation of carbide particles during aging, which leads to sharp embrittlement of the material during operation at high temperatures. Vanadium increases the heat resistance of alloys as long as it retains a sufficient reserve of plasticity and phase precipitation occurs uniformly [32].

The addition of vanadium together with carbon in chromium-nickel or even better chromium-manganese alloys is very expedient and has a positive effect on the heat-resistant properties of the alloys. However, the addition of only vanadium with carbon does not ensure the fulfillment of the requirements currently imposed on high-temperature alloys.

The best results are obtained when vanadium and carbon are added simultaneously with refractory metals (Mo, W, Nb) in certain combinations. This is explained by the fact that vanadium and carbon impart to the γ -solid solution the ability to develop precipitation hardening processes, and refractory elements inhibit diffuse exchange at higher temperatures, making it difficult for the coagulation of dispersed phases and not strengthening of the alloy.

Molybdenum. The introduction of molybdenum into nickel alloys increases the recrystallization temperature of γ -solid solutions and inhibits their softening.

The activation energy of self-diffusion of iron or chromium increases with an increase in the amount of molybdenum added to the alloy.

In most alloys, alloying with molybdenum, along with an increase in heat resistance, gives an increase in plasticity during short-term and long-term tests.

Molybdenum has a much greater effect on the heat resistance of precipitation-hardening alloys with carbide or intermetallic strengthening.

The effectiveness of the effect of molybdenum on the heat-resistant properties of alloyed alloys increases when they are introduced into the alloy simultaneously with other elements, including elements that cause precipitation hardening processes. In this case, molybdenum inhibits the softening of the alloy at higher temperatures due to the fact that it complicates the diffuse exchange, recrystallization and coagulation of dispersed particles.

In carbide-reinforced nickel-base creep alloys, molybdenum also has a positive effect by increasing creep resistance. In a number of alloys, molybdenum forms phases of the Laves type (Fe_2Mo), which contributes to an even greater increase in heat resistance due to precipitation hardening processes.

3.1.5 HEAT RESISTANCE OF CHROMIUM-NICKEL ALLOYS

The phenomenon of creep of metals under load at high temperatures was known for many years, and the first studies of this phenomenon are about a century ago. At first, however, progress towards improving the properties of metals was rather slow. Serious attempts to develop new materials with higher creep resistance began only from the time when the operating temperatures of steam engines rose so much that ordinary low-alloy steels were unsuitable for the manufacture of parts of these machines [33].

At present, nickel-based alloys are used as heat-resistant materials designed to operate at temperatures from 700 to 1100 °C. They are used in gas turbines for engines of aircraft, ships, power generation systems, in the manufacture of rocket and space technology, in petrochemical equipment. Thus, in an aircraft gas turbine engine, more than 70 % of the mass is made up of heat-resistant alloys based on nickel, chromium-nickel and iron-nickel bases – these are discs, nozzle and rotor blades of turbines, combustion chambers, etc. The use of these alloys in the manufacture of stationary gas turbines has also sharply increased, since an increase in operating temperatures can significantly improve their technical and economic indicators.

The most difficult conditions are for turbine blades operating at temperatures of 850–1050 °C. They are subjected to significant tensile stresses as a result of centrifugal loads; these stresses cause creep of parts. The high-speed gas flow of high aggressiveness and the operating mode cause the occurrence of variable mechanical and thermal loads, which cause high-temperature and thermal fatigue, the active development of gas corrosion and erosion processes. The nozzle blades operate at temperatures up to 1150 °C and low stresses, and the disks – at temperatures of 600–800 °C and higher working loads (up to 500–600 MPa), therefore the heat-resistant alloys used for these groups of parts differ significantly in composition and properties.

Modern nickel heat-resistant alloys are essentially working to their limit, as operating temperatures often reach 0.8–0.85 solidus temperatures.

Therefore, the development of new technological processes, such as directional crystallization, growing parts in a monocrystallite state, the use of alloys reinforced with dispersed oxide particles, and the use of alloys reinforced with refractory wires, is of paramount importance in increasing the temperature level of modern heat-resistant alloys, their reliability and durability.

Modern high-temperature nickel-based alloys are very complex: they are included in 7–9 basic alloying elements and a large amount of impurities, their content should be controlled in the alloy.

Depending on the chemical and phase composition, structure, and basic properties, modern chromium-nickel alloys designed for operation at high temperatures can be divided into three large groups: heat-resistant, corrosion-resistant, and heat-resistant alloys [34].

Heat resistance is the ability of steels and alloys to withstand mechanical loads at high temperatures for a certain time.

Heat resistance is a structural and sensory property and, as such, is essentially determined by the structural features of the structure of the material and, first of all, its defectiveness –

by dislocations, their origin, movement, interaction with obstacles and boundaries and their disappearance. Point and spatial defects are also significant.

The main group of nickel alloys is made up of heat-resistant alloys with high long-term strength and creep limits at a temperature of 800 °C and above.

Nickel forms solid solutions with many elements, provides significant opportunities for achieving high heat resistance of alloys based on it. At 1000 °C, cobalt, iron, manganese and copper form unlimited solid solutions, and such refractory metals as chromium, tungsten, molybdenum, tantalum, niobium, vanadium – limited solid solutions with different regions of homogeneity. The solubility of elements such as titanium and aluminum at 1000 °C is 10 % and 7 %, respectively.

Pure nickel at 800 °C has $\sigma(100) = 40 \text{ Mn/m}^2$ (4 kgf/mm²). With the introduction of 20 % Cr into nickel, the long-term strength $\sigma(100)$ increases only to 50–55 Mn/m² (5–5.5 kgf/mm²), while the addition of titanium up to 2 % and 0.7 % aluminum abruptly increases $\sigma(100)$ to 150 Mn/m² (15 kgf/mm²).

It is important to note that the solubility of alloying elements in multicomponent nickel-based alloys can differ significantly in the direction of decreasing the values obtained in the study of binary nickel alloys – alloying element.

Most alloying elements, soluble in nickel or nichrome, are effective hardeners and increase the creep resistance of nickel-based alloys.

Despite the complexity of reports of heat-resistant nickel alloys, the following basic principles can be formulated that are used in their creation:

1) heat-resistant alloys are the main (matrix) solid solution on a chromium-nickel base, alloyed with cobalt and refractory elements-hardener, and the most effective effect is the heat resistance is the complex introduction of alloying elements. The content of these elements in the alloy depends on its purpose and economic feasibility;

2) titanium and aluminum, sometimes niobium, are necessarily present in the alloys, which mainly provide the formation of the main hardening phase of these alloys (γ' -phase);

3) are present in alloys chromium and aluminum provide resistance to gas corrosion due to the formation of protective films rich in oxides Cr₂O₃ and Al₂O₃;

4) carbon is contained in alloys in small amounts, forming carbides and carbonitrides. The kinetics of precipitation and morphology of carbide phases during crystallization, heat treatment, and during operation also largely determines the heat resistance of nickel-based alloys.

Thus, the high-temperature strength of nickel-based alloys is associated with the optimization of their composition according to the ratio of alloying elements included in the matrix γ -solid solution and strengthening intermetallic and carbonitride phases.

Operating temperature and operating stresses are decisive factors in the development of high-temperature nickel-chromium alloys. If the magnitude of stresses depends on the design of the part, then the level of operating temperatures determines the efficiency and main technical characteristics of the product.

With an increase in temperature, the strength of interatomic bonds decreases, the processes of diffusion and disintegration intensify. The mechanism and rate of gas corrosion, the mechanism of plastic deformation and destruction change depending on the temperature.

The operating mode of the chromium-nickel alloy – the nature of temperature and load changes over time – has a great influence on the heat resistance. In the presence of thermal changes, materials are exposed to thermal fatigue, which can sharply (several times) increase the creep rate or lead to the destruction of the part even in the absence of mechanical loads due to only thermal stresses (thermal fatigue). The action of alternating stresses, causing the development of high-temperature fatigue, can also strongly affect the heat resistance and heat resistance.

The composition of the gas environment can also significantly affect the heat resistance and heat resistance of alloys. The presence of aggressive components in the environment (for example, compounds containing sulfur, vanadium, halogens, alkali metals) causes the formation of low-melting or volatile compounds, destroys protective oxide films, and promotes the development of local types of gas corrosion. In addition, in many cases, the gas environment affects the alloy not in stationary conditions, but dynamically, that is, high-speed gas flows act on the surface of the parts, the speed of which can be hundreds and thousands of meters per second. Such operating conditions are typical, for example, for gas turbine blades, skin parts for high-speed aircraft and rockets. Under the influence of high-speed gas flows, creep processes are enhanced, as a result of which the operational resistance of the parts decreases.

Therefore, when determining the suitability of an alloy for operation in a loaded state at high temperatures, it is necessary to take into account not only the results of standard tests for creep and long-term strength, but also the possible change in these characteristics under operating conditions. This requires complex tests to be carried out as closely as possible to the work of the part in real conditions.

3.1.6 MODERN TRENDS IN THE DEVELOPMENT AND USE OF HIGH-TEMPERATURE NICKEL-CHROMIUM ALLOYS "NIKORIN" FOR THE MANUFACTURE OF MANDRELS FOR TUBE ROLLING MILLS

The mandrel longitudinal rolling states used in tube rolling mills have significantly less variety than the helical rolling states. This is primarily due to the fact that they all perform one function: in these states, the walls are rolled out and a rough pipe is obtained from the sleeve. In addition, only double-roll stands have a mandrel state for longitudinal rolling, which also reduces the type of states.

All states can be divided into two groups. The first group includes states of a linear type, with one or two working stands. Rolling in these states is carried out on a short cone-shaped mandrel fixed at the end of a long rod. Longitudinal rolling is based on the deformation of the metal by rolls located parallel in one plane and rotating in different directions; the metal rolling axis is perpendicular to the major roll axes.

The second group of states – multi-stand continuous states, in which rolling is carried out on a long mandrel (floating or held). Floating mandrel rolling is much more widespread. In our time, they began to use a held mandrel, which moves during the deformation of the pipe at a given speed. Floating mandrels of great length (18–20 m) with a diameter of more than 180–200 mm are quite heavy, difficult to transport, require the use of bulky equipment; great difficulties are caused by the manufacture of such mandrels. This is why, for large continuous states, retained mandrels are used, which are about half the length. Rolling on a retained mandrel increases the accuracy of pipes and is therefore also used for rolling smaller pipes [35].

The continuous mill carries out the rolling process in the most progressive and promising way. These units are characterized by the continuity and flow of production, a high degree of mechanization and automation, the best quality of pipes compared to other production methods. Tube-rolling units with an unrepeatable state allow to increase the rolling speed with a short duration of auxiliary operations and large metal deformations, using thick-walled sleeves for this purpose, increases the productivity of the entire TPA complex. The advantage of continuous rolling is also the possibility of obtaining thin-walled pipes of greater length and higher quality (without risks, scratches, with less transverse and longitudinal ripening). Due to the large length of the pipes, tension-reduction states are more efficiently used.

The states of the first group can be single-cast (automatic-state) or double-cast (tandem mill) with the distance between the stands more than the length of the pipe is rolled. Sometimes the two-state is called semicontinuous. Single-stand automatic states carry out rolling in two (and sometimes three) passes in the same caliber and for the task for each subsequent pass the pipe is turned on the front (input) table with the help of reverse feed friction rollers.

Automatic states can have multi-groove rolls (with a number of caliber up to 11–12) or single-groove rolls. In the first case, as one gauge wears out, they switch to rolling in another. Otherwise wear of the groove means the need to replace the rolls. Single-gauge stands are lighter and transshipment is usually carried out by replacing the stands: instead of a stand with worn rolls, a stand with new rolls is installed, assembled aside on a special stand. A very important advantage of single-gauge stands is their increased rigidity, which ensures higher accuracy of pipes with wall thickness. In recent years, more preference has been given to automatic states with single-gauge rolls [36].

Mandrels for rolling states (mandrels) are used for hot rolling of seamless pipes, when rolling a liner into a rough pipe by thinning its wall and increasing its length in pilgrim, rolling and automatic states. The mandrel in this case controls its own diameter and the inner diameter of the pipe. Due to the abrasive and abrasive action of the material of the rolling sleeves, the mandrels lose their material, which leads to a decrease in their diameter and a decrease in the inner diameter of the resulting pipes and a decrease in the thickness of their walls (**Fig. 3.3**).

The resistance of the mandrel to abrasion is measured by the number of rolling cycles, the mandrel withstands without changing the diameter and is determined by the material from which

the mandrel is made. In this case, the mandrel must meet two requirements at once: high wear resistance, in order to avoid its rapid failure, and low brittleness, to prevent the fracture of the shank due to the stress arising in the mandrel during rolling. Economically, the manufacture of mandrels is limited by the cost of materials for their manufacture. Technologically – the availability and manufacturability of the materials used [37].

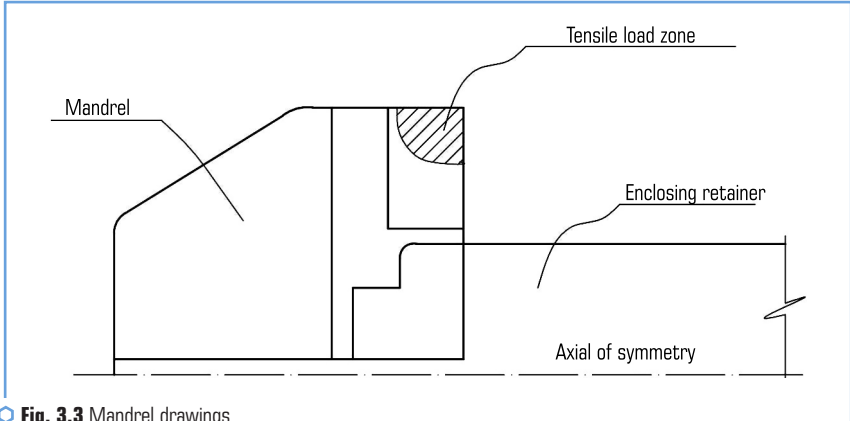


Fig. 3.3 Mandrel drawings
Source [37]

In this work, the choice of material for the manufacture of mandrels is substantiated, which meets all these requirements.

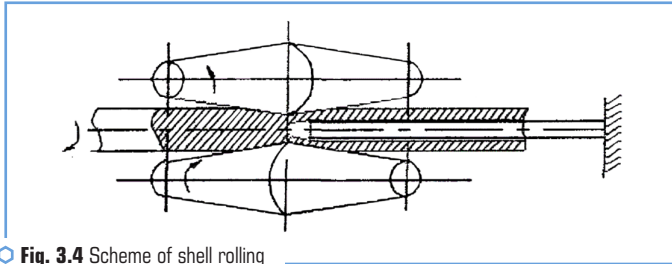
To do this, it is necessary to solve a number of tasks: to analyze the working conditions and determine the requirements for the mandrel material; to characterize the materials suitable for the manufacture of mandrels, to determine the chemical composition of the mandrel materials; determine the formation of the structure and properties of mandrels; justify the choice of material and select the modes of heat treatment of the mandrels.

Today, pipes are used to transport solid (in the form of slurry), liquid and gaseous materials in the event that transportation of these materials by other means is unprofitable. Tubes are made today using the helical rolling method discovered by the Mannesmann brothers in 1885. In this method, the ingot, heated to 900 °C, is carried away by rolls rotating in one direction, it starts to rotate around its axis and between the rolls is pierced with mandrels (**Fig. 3.4**).

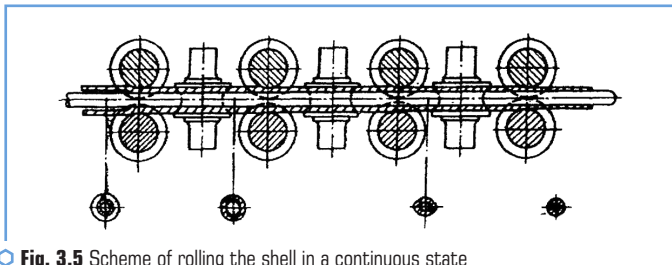
The shell obtained in this case is the initial product for further rolling into a rough tube in rolling states. In this case, the shell decreases its thickness and increases its length, turning into a rough tube. This operation is done on automaton states, continuous rolling states and pilgrim states (**Fig. 3.5–3.7**).

When rolling the shell, the inner diameter of the pipe is controlled by the diameter of the mandrel, which is in the pipe during the entire rolling process until it is removed.

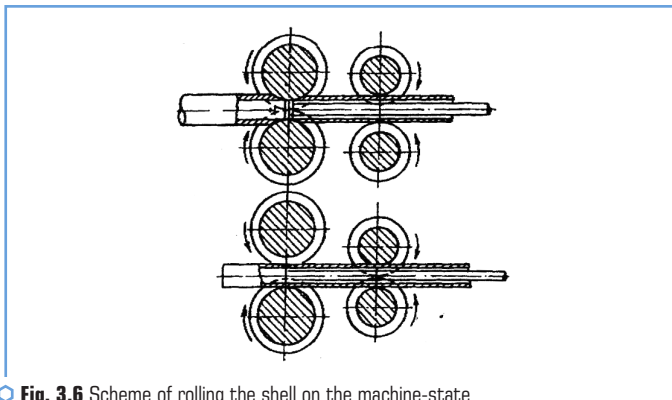
During rolling, due to prolonged contact of the mandrel with the workpiece, the mandrel surface heats up to 700 °C, so the mandrel material must withstand heating up to 700 °C without losing its properties. Also, the mandrel material must resist thermal cycling and abrasion, as well as have high surface hardness, resistance to impacts occurring at the beginning of rolling and during the removal of the mandrel.



○ Fig. 3.4 Scheme of shell rolling
Source [37]



○ Fig. 3.5 Scheme of rolling the shell in a continuous state
Source [36]



○ Fig. 3.6 Scheme of rolling the shell on the machine-state
Source [36]

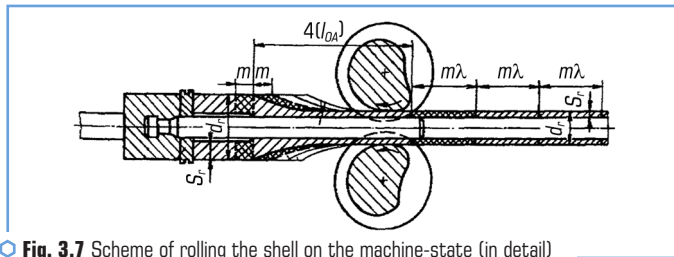


Fig. 3.7 Scheme of rolling the shell on the machine-state (in detail)
Source [36]

Thus, the limiting requirements for a steel mandrel are:

- surface hardness less than 24 HRC;
- heat resistance not less than 700 °C;
- impact strength KCU not less than 90 J/cm²;
- resistance to wear.

There is no normative documentation (technological instructions and technical conditions) that regulates the properties of mandrels made of cast iron and nickel-chromium alloy "Nikorin", which leads to the urgent need to develop requirements for the properties of alloys for mandrels.

3.1.7 STATEMENT OF THE RESEARCH PROBLEM

The object of this paper is to research:

- patterns of formation of the structure of the nickel-chromium alloy "Nikorin" in the initial cast state;
- influence of the structure, phase composition on the properties of the nickel-chromium alloy "Nikorin" in the cast state;
- temperature ranges of phase transformations of the nickel-chromium alloy "Nikorin";
- structure, phase composition, microhardness of phases and structural components of prototypes of the nickel-chromium alloy "Nikorin" after quenching from different temperatures;
- wear resistance of chromium-nickel alloys under friction conditions at elevated temperatures and various loads.

3.2 MATERIAL AND METHODOLOGY OF RESEARCH

Research alloys melted in laboratory conditions were used to carry out studies of phase and structural transformations in nickel-chromium alloys "Nikorin". The chemical composition of the research alloys is shown in **Table 3.1**.

● **Table 3.1** The chemical composition of the prototypes of the nickel-chromium alloy "Nikorin"

Alloy name	Content of alloying elements, %							
	C	Cr	Ni	Mn	Ti	Si	Al	Fe
Chromium-nickel alloy "Nikorin" No. 1	1.8	34.0	57.0	0.2	0.6	0.7	2.5	2.3
Chromium-nickel alloy "Nikorin" No. 2	1.5	36.0	57.2	0.3	0.7	0.6	1.9	1.7
Chromium-nickel alloy "Nikorin" No. 3	2.6	36.0	57.2	0.3	0.7	0.8	2.0	2.2

During melting, the melt was in a quartz tube in an experimental induction furnace. To obtain the required cooling rates during crystallization, a quartz tube with a melt was placed in a Tamman furnace and cooled at a given rate. As a result, samples were obtained that had the content of the main alloying elements within the grade composition for the "Nikorin" chromium-nickel alloy.

The microstructure of the samples of the nickel-chromium alloy "Nikorin" was developed in the Grosbeck reagent followed by thermal digestion. The study of the microstructure was carried out using an optical microscope "Neophot-21".

The determination of the number of phases and structural components in the structure of the research alloys was carried out by the method of A. A. Hlaholev at x400 magnification [38].

The phase identification in the nickel-chromium alloy "Nikorin" was carried out by X-ray structural analysis on a DRON-3M diffractometer in FeK α – radiation. To determine the lattice parameter, the profile of the diffraction maxima (111) g, (002) g, (113) g was recorded along three mutually perpendicular sides of the sample, each side being recorded five times at a rate of (1/8) °/min. The lattice parameter was calculated from the position of the center of mass of the above diffractometric maxima. The results obtained were subjected to statistical processing according to the standard method.

The amount of austenite was determined from the ratio of the integral intensities of the lines (001) and (111) [39–41].

The distribution of alloying elements between the phases and structural components of the nickel-chromium alloy "Nikorin" in the initial state was studied using a JSM-840 electron microscope with a "Link-860/500" microanalysis system ("Link Analytical", England). The studies were carried out in the mode of secondary electrons (SEI) and backscattered electrons (BEI). The beam current was $I = 10^{-7} \dots 10^{-9}$ A. Voltage $U = 20$ kV. The analysis was $t = 100$ s. The beam diameter was 1...1.5 μm . The analysis was performed using the ZAF4/FLS software. Pure samples for each investigated element were chosen as standards (purity was 99.99 %). Standard samples from Link Analytical were used.

Basic information on the nature and mechanism of phase transformations during hardening was obtained on the basis of differential thermal and quenching microstructural analysis. The regularities of the formation of the structure of the Nikorin alloy were studied by quenching-microstructural analysis on a specially designed installation [13]. Quenching temperatures were selected based on the Ni-Cr diagram [14, 42], as well as in accordance with the heating/cooling

thermogram of the research alloys. This setup allows to systematically record the high-temperature state of the sample with high accuracy by quenching. It was based on a vacuum furnace with a quenching tank (**Fig. 3.8**) of the following design.

The main supporting element of the installation is the furnace body. It has a cylindrical shape and is welded from a sheet of steel X18H10T with a thickness of 3 mm. Flanges made of the same steel are welded to the ends, allowing for quick disassembly and assembly of the furnace. One of the flanges is simultaneously a supporting one for screens, a heater and current drives, assembled into a single block. A sample holder assembly is attached to the second flange (**Fig. 3.9**), it is a crucible made of refractory material (3), rigidly fastened to a molybdenum tube (8).



Fig. 3.8 General view of the installation for hardening-microstructural analysis
Source [42]

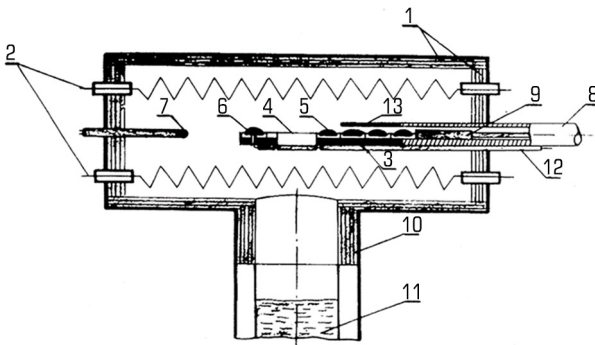


Fig. 3.9 Diagram of the working space of the furnace for hardening-microstructural analysis
Source [42]

The design of the holder allows 4 samples to be installed into the furnace at once on flat alundum substrates (5) and sequentially dumped into the quenching bath (11) through a special hole (4). In the working space of the furnace, limited by a coaxial shield (1), it includes the following elements: a pusher (9), with the help of which the sample (5) is dropped through the hole (4) into the shaft (10) into the quenching bath (11) molybdenum standard (6) with a hot junction of the thermocouple BP 5/20 (7), which controls the operation of the temperature setpoint of the heater power supply system; screen (13), which prevents intensive heating of the samples not participating in the experiment.

The samples were hardened in a 10 % solution in sodium chloride, which is constantly cooled by running water. The cooling rate of the sample during quenching was 20 deg/s at a speed of its movement in the bath of about 3 m/s. The heat loss of a sample with a diameter of 5 mm on the way from the working space of the furnace to the meniscus of the quenching liquid does not exceed 2 °C.

Determination of the temperature ranges of transformations was carried out by thermal analysis on a VD-4 calorimeter with heating at a rate of 5 deg/min.

The microhardness of the structural components of chromium-nickel alloys was determined using a PMT-3 device in accordance with GOST 9450-76 at a load of 50 g and an increase of $\times 485$, as an average of 50 measurements. The measurement accuracy of the diagonals of the indentation is 0.3 % of the smallest division of the micrometer scale. The hardness of the samples was determined by the Rockwell method according to the standard method.

The temperature dependence of the hardness of the chromium-nickel alloy Nikorin was studied using a Vickers device. A diamond pyramid was used as an indenter; the exposure of the indenter under a load of 50 g was 10 s. The hardness measurements were carried out at a heating rate of 5 °C/min with a slit of 100 °C. The hardness, which was measured from the indentation of the indenter, characterizes the value of the material resistance [43].

Tribotechnical tests for wear resistance under friction conditions at elevated temperatures and various loads were carried out on a modernized friction device 2070 CMT 1, according to the "shaft-plane" scheme. The counterbody was Art. 45. **Fig. 3.10** shows the appearance of the tribological contact at a load of 1000 N and a sliding speed of 2 m/s [44].

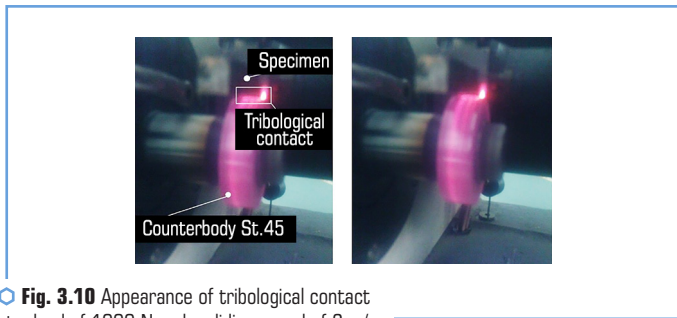


Fig. 3.10 Appearance of tribological contact at a load of 1000 N and a sliding speed of 2 m/s
Source [44]

3.3 INVESTIGATION OF THE REGULARITIES OF THE STRUCTURE FORMATION, PHASE COMPOSITION, PROPERTIES AND DISTRIBUTION OF ALLOYING ELEMENTS BETWEEN THE PHASES AND STRUCTURAL COMPONENTS OF NICKEL-CHROMIUM ALLOYS "NIKORIN" IN THE AS-CAST STATE

The work investigated the structure, phase composition, hardness, microhardness of the structural components of the samples of the nickel-chromium alloy "Nikorin" intended for the manufacture of mandrels for rolling pipes [45, 46].

Analysis of the microstructure of samples (No. 1, 2, 3) of the heat-resistant chromium-nickel alloy "Nikorin" in the initial cast state (**Fig. 3.11–3.13**) indicates that an almost homogeneous structure is formed in these alloys over the section of the casting. Analysis of the microstructure of samples (No. 1, 2, 3) of the heat-resistant chromium-nickel alloy "Nikorin" in the initial cast state (**Fig. 3.11–3.13**) indicates that an almost homogeneous structure is formed in these alloys over the section of the casting. In the structure of three samples of the high-temperature nickel-chromium alloy "Nikorin", chromium carbide Cr_7C_3 is observed both in the longitudinal and in the cross section. In these alloys, due to the high carbon content, crumbling of the Cr_7C_3 carbide is observed.

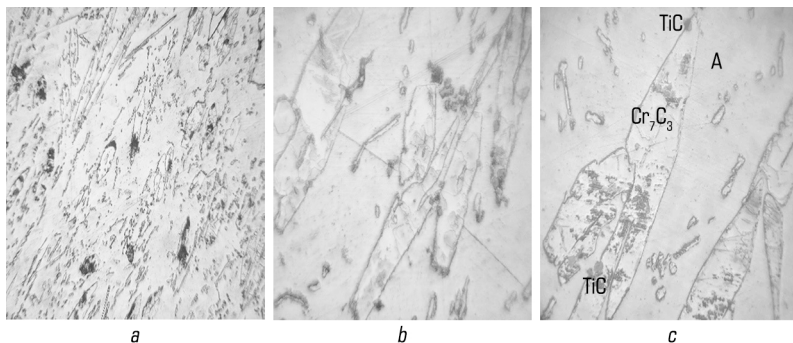
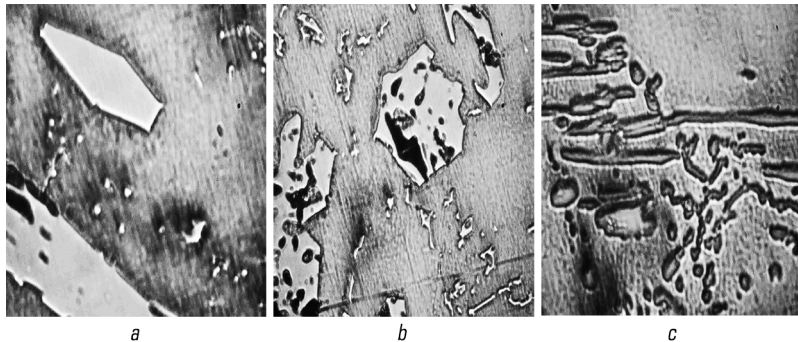


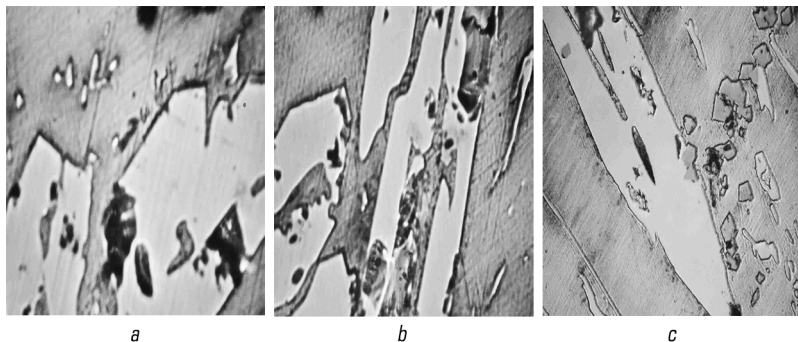
Fig. 3.11 Microstructure of heat-resistant chromium-nickel alloy "Nikorin" (alloy No. 1): *a* – casting surface $\times 150$; *b* – surface of casting $\times 600$; *c* – central part of casting $\times 600$
Source [44–46]

Since alloy 3 contains more carbon (2.62 % C), this phenomenon is much more pronounced in it than in alloys 1 and 2. Due to the fact that there is a sufficiently high carbon content in the alloys, the Cr_7C_3 chromium carbide becomes brittle and during operation (or the manufacture of a microsection), such a carbide crumbles unevenly throughout the carbide volume. Therefore, the higher the carbon content in the alloy, the less stable the carbide becomes. And since the carbide component strengthens chromium-nickel alloys, it can be concluded that parts from such an alloy will be less stable during operation. Another type of TiC carbides (**Fig. 3.11, c, 3.13, c**) is located

unevenly both in the volume of Cr_7C_3 carbides and in the austenitic matrix of the casting. Titanium carbides have a regular cubic shape.

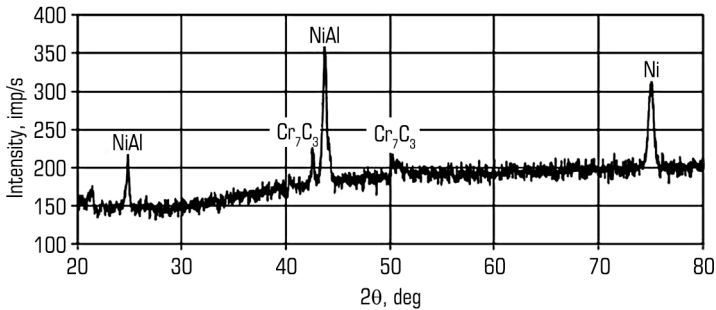


○ **Fig. 3.12** Microstructure of heat-resistant chromium-nickel alloy "Nikorin" (alloy No. 2): *a* – longitudinal section of Cr_7C_3 carbides; *b* – cross section of Cr_7C_3 ; *c* – coarsely differentiated austenite-carbide eutectic based on Cr_7C_3 chromium carbide
Source [44–46]



○ **Fig. 3.13** Microstructure of nickel-chromium alloy "Nikorin" (alloy No. 3): *a* – cross-section of Cr_7C_3 ; *b* – longitudinal section of Cr_7C_3 carbides; *c* – carbide *t* and, which grows together with chromium carbide Cr_7C_3
Source [44–46]

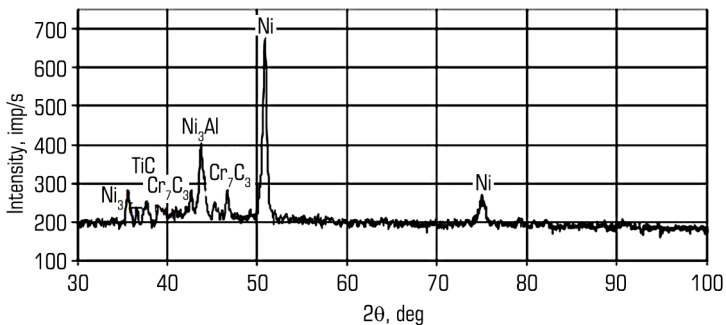
The phase composition of research alloys was studied using X-ray diffraction analysis (**Fig. 3.14–3.16**). **Fig. 3.14** shows the diffraction patterns of the nickel-chromium alloy "Nikorin" (alloy No. 1). In the chromium-nickel alloy, X-ray diffraction analysis has revealed excess and eutectic carbide Cr_7C_3 , as well as high-nickel austenite, as evidenced by the high intensity of its line (111).



○ **Fig. 3.14** Scheme of the diffractogram of the prototype (alloy No. 1) of the nickel-chromium alloy "Nikorin"
Source [44–46]

Fig. 3.15, 3.16 show diffraction patterns of samples of alloys 2 and 3 of the nickel-chromium alloy "Nikorin". In this alloy, X-ray diffraction analysis revealed excess and eutectic carbide Cr_7C_3 , as well as high-nickel austenite, as evidenced by the high intensity of its line (111). In addition, titanium carbide TiC is present in test alloys 2 and 3.

Additionally, carbide analysis was carried out on samples of research alloys No. 2, 3 (**Fig. 3.17, 3.18**), which, in addition to the above-mentioned carbides, made it possible to reveal the σ -phase (FeCr) in the structure of the heat-resistant chromium-nickel alloy. The presence of this phase is undesirable, since it reduces the stability of the alloys during operation. This is due to the fact that the σ -phase has a high hardness, however, it is very fragile.



○ **Fig. 3.15** Scheme of the diffractogram of the prototype (alloy No. 2) of the nickel-chromium alloy "Nikorin"
Source [44–46]

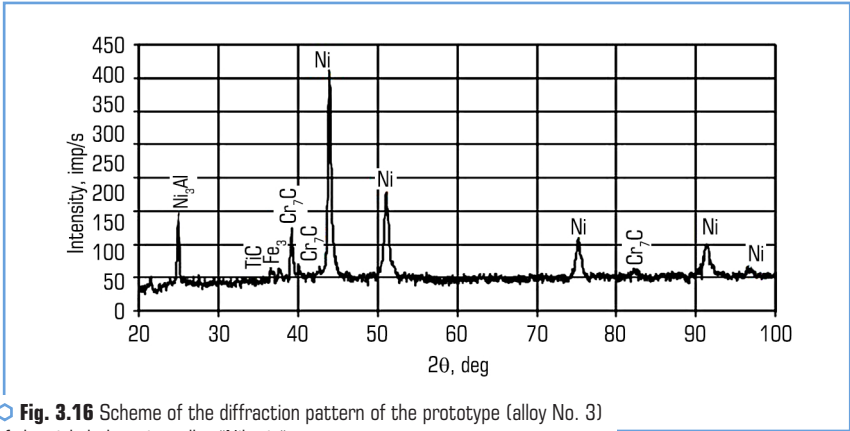


Fig. 3.16 Scheme of the diffraction pattern of the prototype (alloy No. 3) of the nickel-chromium alloy "Nikorin"
Source [44–46]

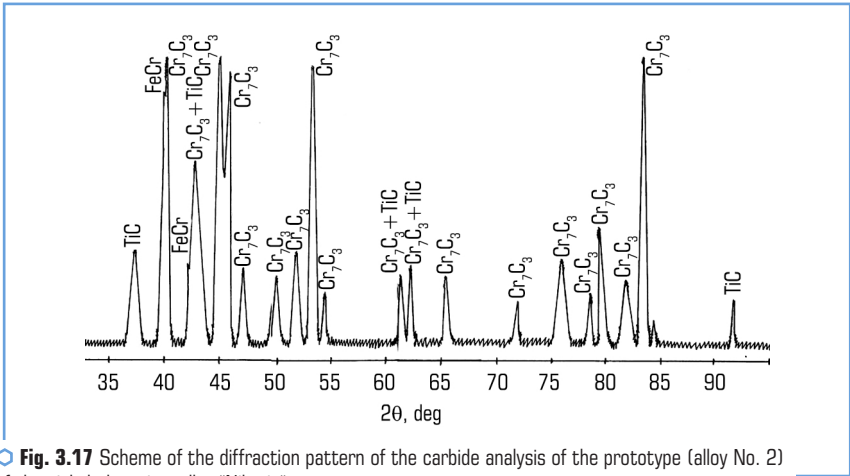
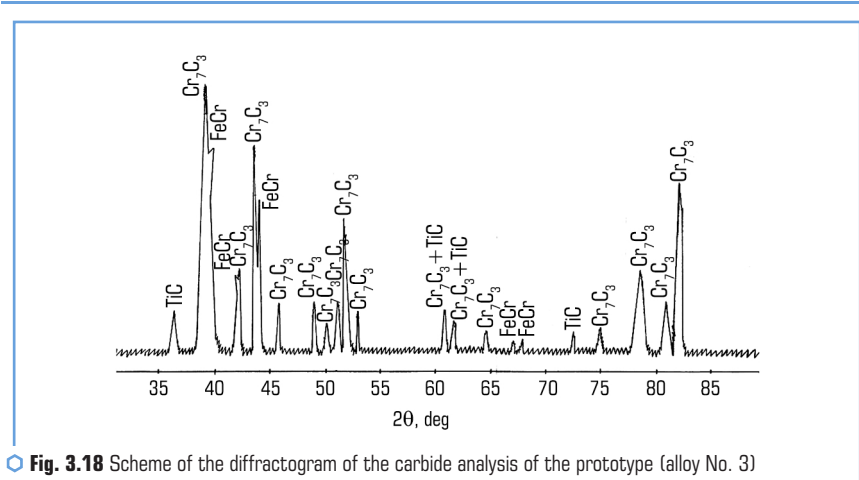


Fig. 3.17 Scheme of the diffraction pattern of the carbide analysis of the prototype (alloy No. 2) of the nickel-chromium alloy "Nikorin"
Source [44–46]

Table 3.2 shows the quantitative data of X-ray structural analysis of prototypes of the nickel-chromium alloy "Nikorin" in the cast state.

Analysis of the data in **Table 3.2** [44–46] indicates that the matrix of the chromium-nickel alloy consists of high-nickel austenite. The hardness of a nickel-chromium alloy "Nikorin" is 47 HRC units.

Table 3.3 shows the measurement data for the microhardness of the matrix and Cr₇C₃ carbide in all research chromium-nickel alloys.



● **Fig. 3.18** Scheme of the diffractogram of the carbide analysis of the prototype (alloy No. 3) of the nickel-chromium alloy "Nikorin"
 Source [44–46]

● **Table 3.2** Quantitative data of X-ray structural analysis of prototypes of the nickel-chromium alloy "Nikorin"

Test chromium-nickel alloys	a_γ by (022) γ %	γ , in the matrix	HRC
Alloy No. 1	3.59	100	47
Alloy No. 2	3.59	100	47
Alloy No. 3	3.59	100	47

Source [44–46]

● **Table 3.3** The value of the microhardness of the matrix and eutectic carbide in research chromium-nickel alloys

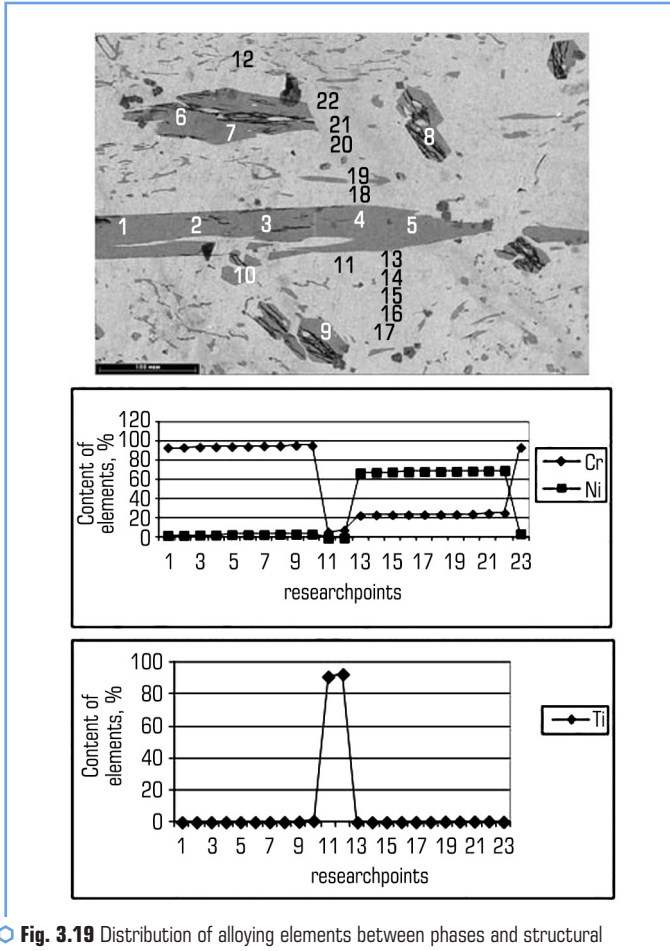
Test chromium-nickel alloys	Microhardness of structural components, MPa	
	Matrix	Carbide
Alloy No. 1	2769	18921
Alloy No. 2	2852	18921
Alloy No. 3	2680	18921

Source [44–46]

The alloys differ from each other in the content of carbon and iron, therefore the microhardness of the matrix of chromium-nickel alloys No. 1, No. 2 and No. 3 is not the same. Due to the high carbon content, crumbling of Cr_7C_3 carbide is observed in these samples, since alloys 1 and 3 contain more carbon, this phenomenon is more pronounced in them than in alloy 2, which explains

the decrease in the microhardness of the matrix. The microhardness of Cr_7C_3 carbide in all alloys remains unchanged.

The distribution of alloying elements between phases and structural components in research chromium-nickel alloys in the cast state was studied using local X-ray spectral analysis. The research results are shown in **Fig. 3.19–3.21** and in **Tables 3.4–3.6**. Quantitative local X-ray spectral analysis was carried out point by point. The content of alloying elements in the matrix and carbide was analyzed [46–48].



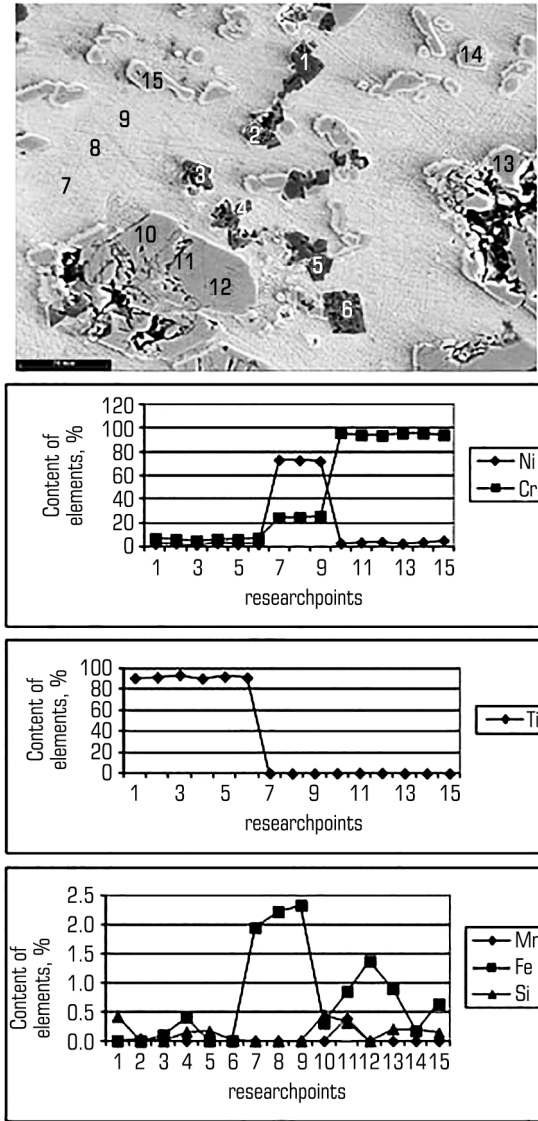


Fig. 3.20 Distribution of alloying elements between phases and structural components in the nickel-chromium alloy "Nikorin" (alloy No. 2)
Source [46–48]

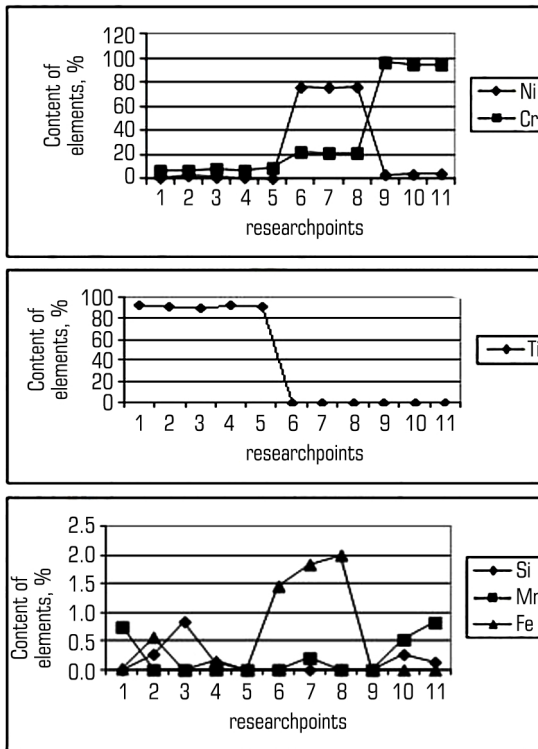
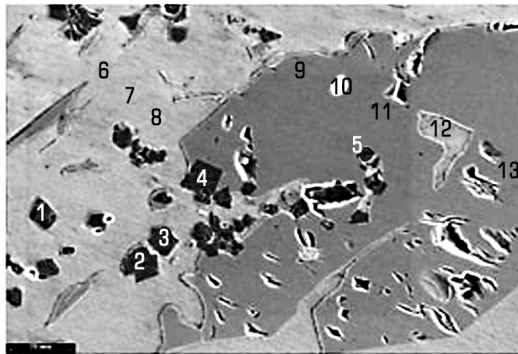


Fig. 3.21 Distribution of alloying elements between phases and structural components in the nickel-chromium alloy "Nikorin" (alloy No. 3)
Source [46–48]

● **Table 3.4** Distribution of alloying elements between phases and structural components in the chrome-nickel alloy "Nikorin" (alloy No. 1)

Structural components	Content of alloying elements in structural components, in %						
	Cr	Ni	Fe	Ti	Si	Mn	Σ alloying elements, %
Matrix	24.66	68.72	2.10	0.21	0.60	0.30	27.87
Carbide Cr ₇ C ₃	94.47	3.77	0.70	0.16	0.15	0.65	5.43
TiC carbide	7.51	0.24	0.13	91.81	0.16	–	8.04

Source [46–48]

● **Table 3.5** Distribution of alloying elements between phases and structural components in the chrome-nickel alloy "Nikorin" (alloy No. 2)

Structural components	Content of alloying elements in structural components, in %						
	Cr	Ni	Fe	Ti	Si	Mn	Σ alloying elements, %
Matrix	24.80	7.90	2.18	0.09	–	–	24.89
Carbide Cr ₇ C ₃	95.32	3.55	0.71	0.18	0.22	0.07	4.73
TiC carbide	6.08	2.34	0.09	91.36	0.13	0.07	8.71

Source [46–48]

● **Table 3.6** Distribution of alloying elements between phases and structural components in the chrome-nickel alloy "Nikorin" (alloy No. 3)

Structural components	Content of alloying elements in structural components, in %						
	Cr	Ni	Fe	Ti	Si	Mn	Σ alloying elements, %
Matrix	21.90	79.13	1.77	0.31	–	0.07	24.04

Source [46–48]

Fig. 3.19 and **Table 3.4** show data on the distribution of alloying elements between phases and structural components in the Nikora chromium-nickel alloy (alloy No. 1). The content of chromium and other alloying elements in the Cr₇C₃ carbide, which is formed in the nickel-chromium alloy "Nikorin", has been determined. The Cr₇C₃ carbide in the chromium-nickel alloy contains 94.5 % Cr, other alloying elements are present in small amounts. The chromium-nickel alloy matrix contains 68.7 % Ni and 24.6 % Cr and is a nickel-based solid solution with a face-centered cubic lattice.

In **Fig. 3.20, 3.21** and **Tables 3.5, 3.6** show data on the distribution of alloying elements between the phases and structural components in the samples of the high-temperature nickel-chromium alloy "Nikorin" (alloys No. 2 and No. 3).

Analysis of the obtained data on the distribution of alloying elements between the phases and structural components (**Fig. 3.20, 3.21, Tables 3.5, 3.6**) shows that the structure of the alloy

is homogeneous. The content of chromium and other alloying elements in the Cr_7C_3 carbide, which is formed in the nickel-chromium alloy "Nikorin", the matrix and titanium carbide TiC, has been determined. The Cr_7C_3 carbide in the prototype alloy No. 2 contains 95.32 % Cr, and in the alloy No. 3 – 95.35 % Cr, all other alloying elements are present in the carbide in small amounts. The matrix of the nickel-chromium alloy "Nikorin" sample No. 2 contains 72.9 % Ni and 24.8 % Cr, and sample No. 3 – 79.13 % Ni and 21.9 % Cr and is a solid solution based on nickel with an fcc lattice. Titanium carbide TiC in sample No. 2 contains 91.36 % Ti, and in sample No. 3 – 90.55 % Ti.

The data obtained indicate that the matrix of the research alloy No. 2 contains 3 % more chromium in comparison with the matrix of the alloy No. 3. The carbides Cr_7C_3 and TiC of alloy No. 2 of iron, silicon and manganese also contain more than alloy No. 3. In the matrix of chromium-nickel alloy No. 3, the content of nickel, iron, titanium is 7 % higher compared to alloy No. 2, the content of iron and titanium in sample No. 3 is slightly higher than in sample No. 2. It should be noted that the prototypes differ only in the content of alloying elements in the matrix, the degree of alloying of Cr_7C_3 and TiC carbides is the same.

Thus, the data obtained indicate that, over the section of the casting, heat-resistant chromium-nickel alloys have a homogeneous structure with a uniform distribution of alloying elements.

3.4 CALORIMETRIC ANALYSIS OF RESEARCH CHROMIUM-NICKEL ALLOYS

Phase transformations in metallic systems are accompanied by the release or absorption of heat, and some of them occur both in the forward and reverse directions (reverse), while others proceed in only one direction (irreversible). The first include the following phase transformations: melting-crystallization, polymorphic transformations, formation and decomposition of chemical compounds. Irreversible processes should include the reactions of transition from unstable, metastable compounds to stable (stable) ones, for example, the decomposition of solid solutions, the transition from an amorphous state to a crystalline state. The thermal effects of irreversible processes appear only on the heating curves and, as a rule, such reactions are exothermic [49, 50].

If no transformations occur during heating in the sample, then the differential record on the thermogram is fixed as a straight line parallel to the temperature axis. Thermal effects of phase transformations are reflected in the thermogram by sharp deviations of the differential curve. However, the onset of the effect is characterized by a sharp kink in the differential curve only for substances with high thermal conductivity. For substances with low thermal conductivity, the beginning of the deviation of the differential curve is always more or less smoothly rounded [50].

Changes in the course of the curve correspond to the start and end temperatures of the conversion. As a rule, the nature of the effect can be determined by the nature of the peaks in the thermogram. However, it should be noted that a large number of thermograms of various substances with a large number of effects, the nature of which has not been deciphered, has been

published so far [50]. One of the factors that make it difficult to decipher thermograms is the superposition of effects on each other. If both merging effects are accompanied, for example, by heat absorption, then one unusually large effect takes place on the thermogram. If, however, two heat effects, opposite in sign, merge, then they superimpose one on the other, mutually reduce the size of the corresponding deviations on the differential curve. The overlapping of peaks of various effects can be deciphered after additional research using other methods.

Considering the above, let's consider the thermograms of the nickel-chromium alloy "Nikorin".

Fig. 3.22 shows the heating and cooling thermograms of the nickel-chromium alloy "Nikorin". It can be seen from the presented curve (**Fig. 3.22, a**) that a horizontal section is observed on the thermogram of heating to a temperature of 490 °C, which indicates the absence of phase transformations, apparently due to the high stability of the initial structure. Heating to a temperature of 490 °C indicates the presence of a stable austenitic structure in this alloy.

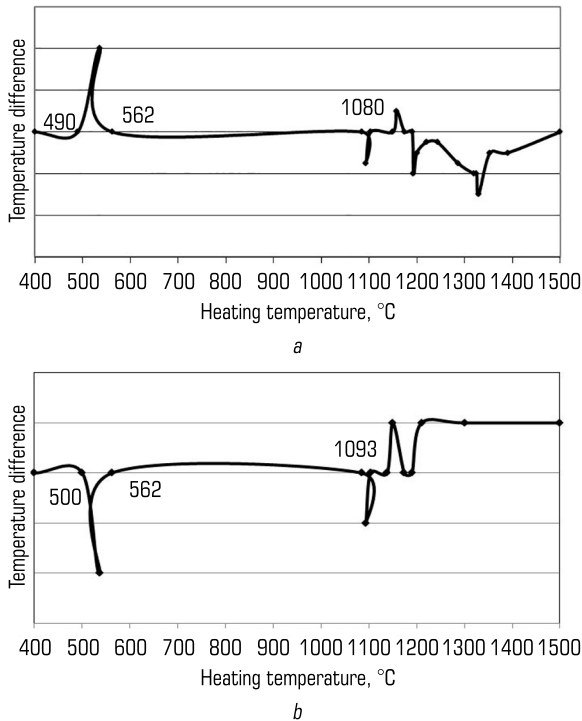


Fig. 3.22 Thermogram of the nickel-chromium alloy "Nikorin":
a – heating; *b* – cooling

In the temperature range 490–562 °C, the ordered phase CrNi₂ is likely to dissolve in the nickel austenite. Upon further heating to a temperature of 1085 °C, the structure of the alloy under study retains its stability, because, according to the thermogram, there are no thermal effects in this gap. On the cooling thermogram, the thermal effects are much less and they are not as intense as during heating.

Table 3.7 shows the temperatures of the onset of crystallization and melting of the high-temperature alloy "Nikorin", determined by thermal analysis during heating and cooling. The melting range of the Nikorin alloy is 104 °C, and the crystallization range is 93 °C. When heated from room temperature to 1286 °C, only one thermal effect is observed in the temperature range 490–562 °C, probably dissolution of the ordered CrNi₂ phase in the nickel matrices.

● **Table 3.7** Temperatures of the onset of crystallization/melting of samples of chromium-nickel alloy "Nikorin" during heating/cooling

Research alloy	Crystallization temperature, °C		Melting point, °C	
	Heat	Cooling	Heat	Cooling
"Nikorin"	1390	1373	1286	1280

3.5 STRUCTURE, PHASE COMPOSITION, MICROHARDNESS OF PHASES AND STRUCTURAL COMPONENTS OF PROTOTYPES OF NICKEL-CHROMIUM ALLOY "NIKORIN" AFTER QUENCHING FROM DIFFERENT TEMPERATURES

The quenching microstructural research method, developed by the staff of the "Bunin School" and widely used to study the mechanism and kinetics of phase transformations in alloys of the Fe-C system, has been successfully applied to alloys of other systems, and makes it possible to obtain comprehensive information about the features of phase transformations and patterns of structure formation in these systems, in particular, in the Ni-Cr system, which is the basis of high-temperature and heat-resistant alloys "Nikorin".

"Nikorin" alloys are intended for the manufacture of rolling tools (calibers and mandrels for rolling pipes) and must have increased strength properties at high temperatures and be resistant to oxidation. Alloying nickel with chromium results in a strong increase in oxidation resistance at high temperatures.

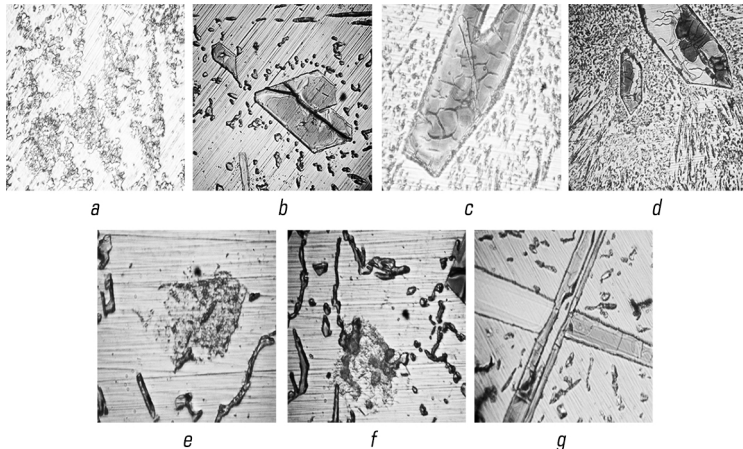
The critical minimum amount of chromium required for a significant increase in the heat resistance of nickel and complex nickel alloys is 20–25 %. The maximum heat resistance in alloys of the Ni-Cr system is observed at 40 % chromium [51].

Alloys of the Ni-Cr system are alloyed with effective reinforcements – aluminum and titanium. These elements in nickel alloys contribute to the formation of a stable dispersed intermetallic compound Ni₃Al(Ti), which causes coherent strengthening of the matrix, hinders the sliding of the

metal under load at high temperatures, and thus increases the strength of the alloy. It has been established that it is the formation of this phase that increases the strength of a number of industrial Ni-Cr alloys at high temperatures [26, 52].

An analysis of the microstructures of the chromium-nickel alloy samples indicates that, depending on the quenching temperature, a different structure and phase composition are formed in the research alloy. As a result of quenching the nickel-chromium alloy "Nikorin" from a liquid state from a temperature of 1390 °C, a quenched liquid with a clearly pronounced cellular structure and well-developed dendrites of the primary γ -Ni solid solution are recorded (**Fig. 3.23, a**).

Quenching from a temperature of 1373 °C indicates the development of the crystallization process, which does not end at this temperature. Austenite dendrites grow together and form a matrix in which chromium carbides Cr_7C_3 are present, as well as near the carbides – quasi-eutectic regions of a quenched liquid (**Fig. 3.23, b, c, d**). When quenched from a temperature of 1280 °C, crystallization also does not have time to complete. A rim of eutectic austenite is formed around coarse crystals of Cr_7C_3 carbides, which indicates the initial stage of eutectic crystallization: $P \rightarrow \text{Cr}_7\text{C}_3 + \gamma$ (**Fig. 3.23, c**). Sections of the quenched liquid solidify in the form of a quasi-eutectic.



○ **Fig. 3.23** Microstructure of samples of chromium-nickel alloy "Nikorin" after quenching from different temperatures ($\times 1000$): *a* – hardening temperature 1390 °C; *b* – hardening temperature 1373 °C; *c* – hardening temperature 1280 °C; *d* – hardening temperature 1170 °C; *e* – hardening temperature 1150 °C; *f* – hardening temperature 1100 °C; *g* – hardening temperature 1100 °C ($\times 500$)
Source [53, 54]

In the samples quenched from a temperature of 1150–1100 °C, together with the carbides Cr_7C_3 , TiC and austenite, the structure contains an intermetallic phase (probably σ is the FeCr phase). The presence of this phase is undesirable, since it reduces the stability of the alloys during

operation under conditions of tensile stresses. This is due to the fact that the σ -phase has a high hardness, however, at the same time, it is very brittle. The presence of the σ -phase is recorded in the structure of the metallurgical tooling parts, obtained by casting from the "Nikorin" alloy [55].

It is known [56–58] that in high-chromium and chromium-nickel alloys during crystallization, peritectic-eutectic transformations are realized:

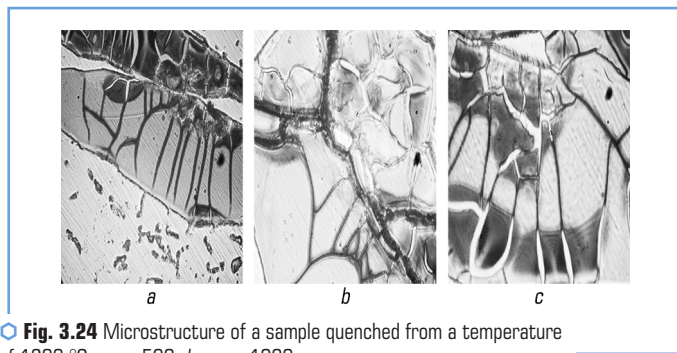


The study of quenched samples confirms the implementation of the peritectic transformation ($P + \gamma \rightarrow Cr_7C_3 + \gamma_{zal} + P_{zal}$) in the nickel-chromium alloy "Nikorin" (Fig. 3.24).

The peritectic transformation leads to a change in the shape and morphology of the primary carbide crystals of Cr_7C_3 as a result of the diffuse interaction of the liquid and γ -Ni austenite crystals; dark channels are formed in the middle of the carbide crystals, through which the liquid enters during the peritectic transformation to the γ/Cr_7C_3 recrystallization front. Regions of retained austenite (white) are observed in the center and periphery of the carbide crystals. Crystallization is completed upon cooling below 1190 °C. In the sample quenched from a temperature of 1085 °C, crystallization is completely completed, as evidenced by the microstructure represented by a matrix and a large amount of Cr_7C_3 carbides. The structure contains dispersed needle-shaped carbides along with large ones (Fig. 3.25, a).

In a specimen quenched from a temperature of 526 °C (Fig. 3.25, b), the structure is represented by an austenite matrix and carbides of various shapes and sizes.

Fig. 3.26–3.28 shows the sections of the diffraction patterns of the samples of the nickel-chromium alloy "Nikorin" after quenching from different temperatures. X-ray diffraction analysis of samples quenched at different temperatures revealed excess and eutectic Cr_7C_3 carbide, high-nickel austenite, as well as the Ni_3Al phase.



○ Fig. 3.24 Microstructure of a sample quenched from a temperature of 1280 °C: a – $\times 500$; b, c – $\times 1000$
Source [53, 54]

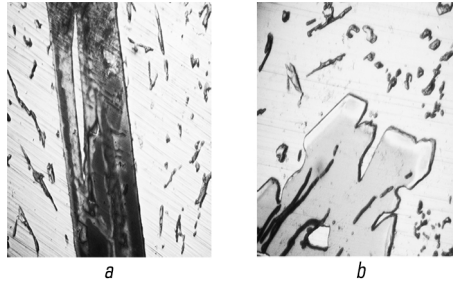


Fig. 3.25 Microstructure of samples, chromium-nickel alloy "Nikorin" after quenching at different temperatures: *a* – hardening temperature 1085 °C; *b* – hardening temperature 526 °C
Source [53, 54]

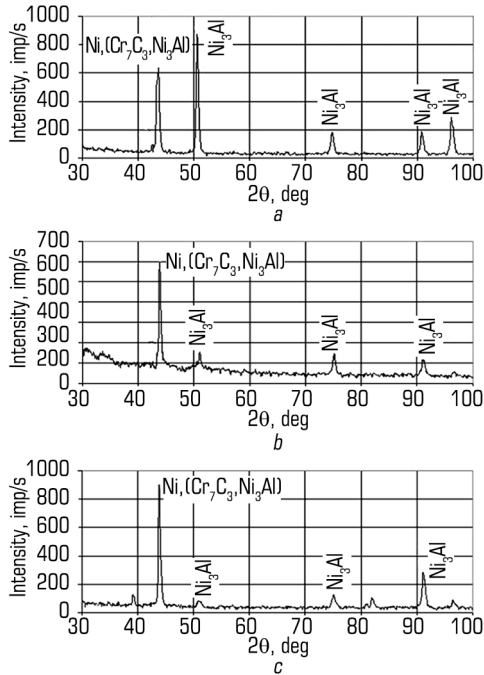
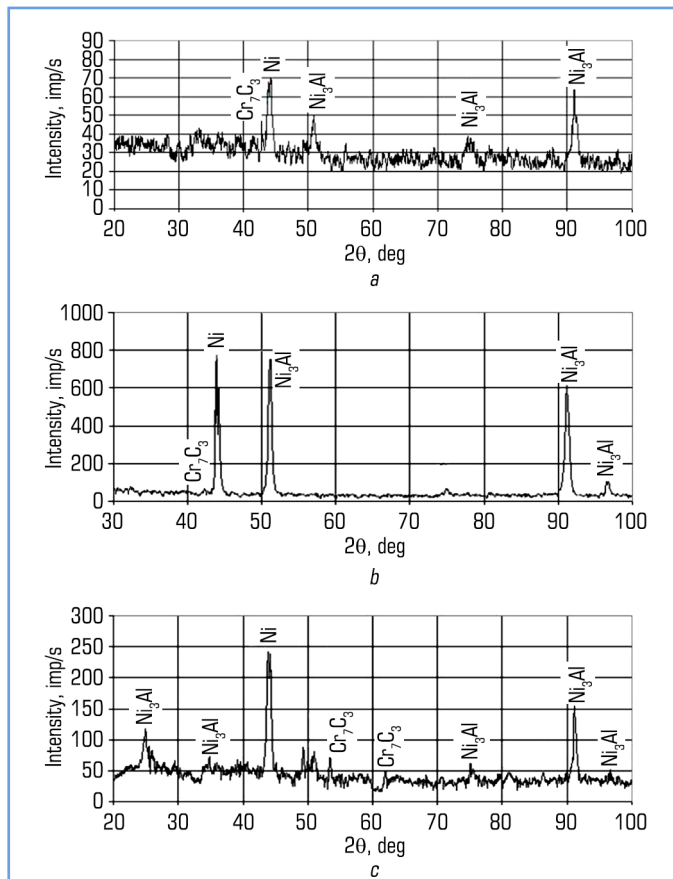


Fig. 3.26 Schemes of diffraction patterns of the nickel-chromium alloy "Nikorin" after quenching at different temperatures: *a* – hardening temperature 1390 °C; *b* – hardening temperature 1373 °C; *c* – hardening temperature 1286 °C
Source [53, 54]

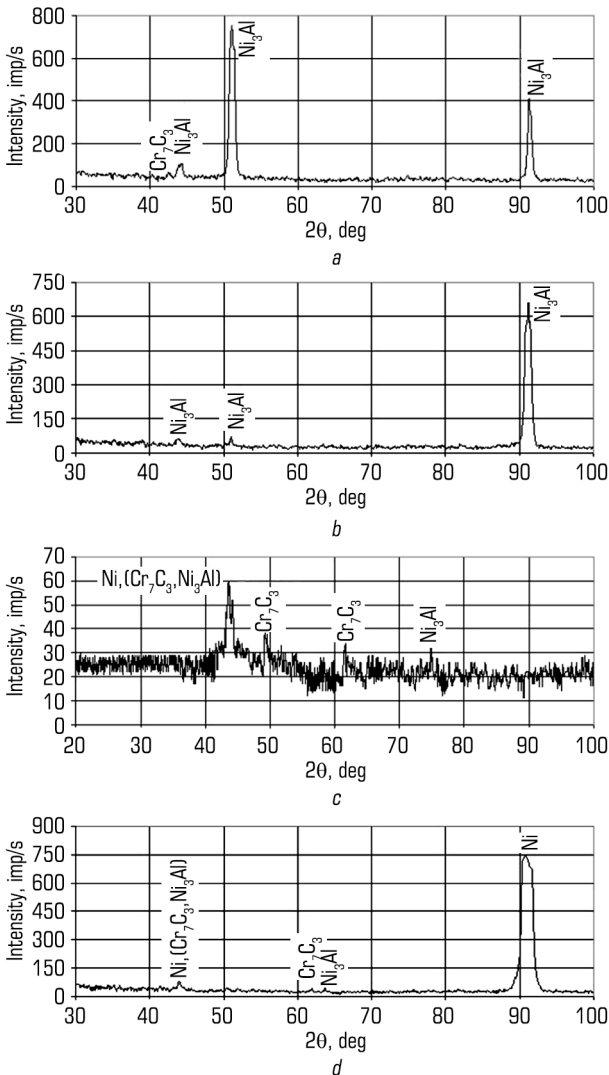


○ **Fig. 3.27** Schemes of diffraction patterns of the nickel-chromium alloy "Nikorin" after quenching from different temperatures: *a* – hardening temperature 1280 °C; *b* – hardening temperature 1190 °C; *c* – hardening temperature 1170 °C
 Source [53, 54]

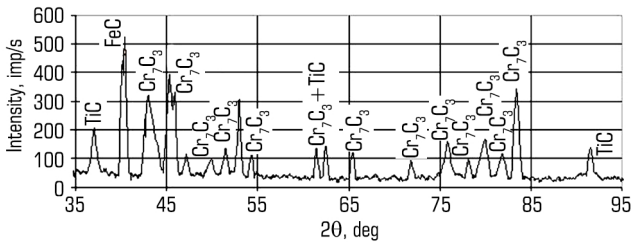
In order to clarify the phase composition of the alloy, carry out carbide analysis. Additionally, carbide analysis was carried out on samples quenched from 1150–1100 °C (**Fig. 3.29, 3.30**), which, in addition to Cr₇C₃ carbide, made it possible to reveal titanium carbide TiC and the σ-phase (FeCr) in the structure of the heat-resistant chromium-nickel alloy.

Table 3.8 shows the measurement data for the microhardness of the matrix and Cr₇C₃ carbide.

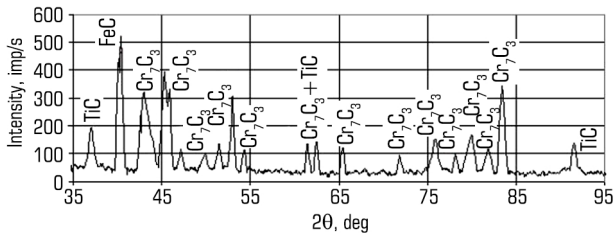
In accordance with the structural changes and the phase composition of the nickel-chromium alloy "Nikorin", the microhardness also changes (**Table 3.8**).



○ **Fig. 3.28** Schemes of the diffraction patterns of the nickel-chromium alloy "Nikorin" after quenching at different temperatures: *a* – hardening temperature 1150 °C; *b* – hardening temperature 1110 °C; *c* – hardening temperature 1085 °C; *d* – hardening temperature 526 °C
Source [53, 54]



● **Fig. 3.29** Schematic of the diffraction pattern of a sample of nickel-chromium alloy "Nikorin" quenched from a temperature of 1150 °C
Source [53, 54]



● **Fig. 3.30** Schematic diffraction pattern of a sample of nickel-chromium alloy "Nikorin" quenched from a temperature of 1100 °C
Source [53, 54]

● **Table 3.8** Microhardness of the matrix and carbides in the samples of the nickel-chromium alloy "Nikorin" after quenching from various temperatures

Sample No.	Quenching temperature of "Nikorin" alloys, °C	Microhardness of research alloys, MPa	
		Matrix	Carbide
1	1390	4120.89	–
2	1373	3405.69	21285.58
3	1286	2294.99	16483.55
4	1280	2043.68	23361.05
5	1190	2595.67	25755.55
6	1170	2227.98	25755.55
7	1150	1933.23	16483.55
8	1110	2365.06	16483.55
9	1085	2438.39	15239.97
10	526	3532.99	16483.55

Source [53, 54]

Data analysis **Table 3.8** shows that in the temperature range 1286–1280 °C, where peritectic-eutectic transformations are realized, the microhardness of the carbide changes extremely, which is probably associated with a change in the quantitative ratio of carbides. In the temperature range 1150 °C–1110 °C, the microhardness of the carbide is stable and amounts to 16483.55 MPa. However, at a temperature of 1150 °C, a significant decrease in the microhardness of the matrix (up to 1933 MPa) is observed, which is probably associated with the precipitation of the σ -phase. Quenching from a temperature of 526 °C leads to an increase in the microhardness of the matrix to 3533 MPa.

Based on the data in the table, it is possible to conclude that, as a result of grating from different temperatures, a redistribution of alloying elements occurs, which leads to jumps in the values of microhardness.

3.6 INFLUENCE OF THE STRUCTURAL-PHASE STATE ON THE MECHANICAL AND TRIBOLOGICAL PROPERTIES OF CHROMIUM-NICKEL ALLOYS IN THE CAST STATE

The work carried out the measurement of hardness at elevated temperatures of samples of chromium-nickel alloy "Nikorin" in the initial state.

This type of study allows one to make an express assessment of the heat resistance characteristics of a given material without expensive tensile tests. The hardness, which is measured by the indentation of the indenter, characterizes the value of the resistance of the material [43].

Table 3.9 shows the data of hardness measurements at high temperatures on samples of the nickel-chromium alloy "Nikorin" in the cast state.

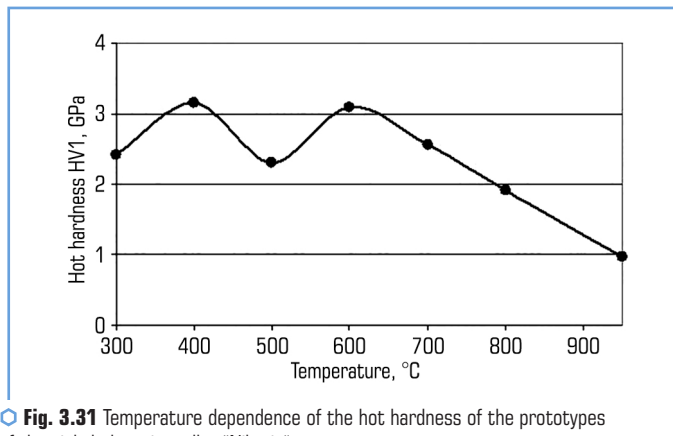
● **Table 3.9** Measurement data of the high-temperature hardness of the nickel-chromium alloy "Nikorin"

Test temperature, °C	300	400	500	600	700	800	950
Hardness HV1, GPa	2.41	3.15	2.3	3.09	2.56	1.92	0.98

Source [59, 60]

The analysis of the table shows that at a test temperature of 300 °C the hardness of the chromium-nickel alloy is small and amounts to 2.41 GPa, with an increase in temperature to 400 °C the hardness of the alloy slightly increases (3.15 GPa). A temperature of 500 °C leads to the fact that the hardness index drops sharply and amounts to 2.3 GPa. Thereafter, the hardness of the research chromium-nickel alloy increases.

On the curve of the temperature dependence of the hardness of the heat-resistant chromium-nickel alloy "Nikorin" (**Fig. 3.31**) in the temperature range of 500–600 °C, there is a sharp change in hardness. Probably, in this temperature range, dissolution of secondary excess carbides in nickel austenite occurs, which explains the sharp drop in hardness at 500 °C.



● Fig. 3.31 Temperature dependence of the hot hardness of the prototypes of the nickel-chromium alloy "Nikorin"
Source [59, 60]

Also, tests were carried out for wear resistance under friction conditions at elevated temperatures and various loads of samples of the nickel-chromium alloy "Nikorin" in the cast state. The research results are shown in **Tables 3.10, 3.11**.

Table 3.10 shows the results of tribotechnical tests of samples of chromium-nickel alloy "Nikorin" in the cast state at room temperature.

- **Table 3.10** Results of studies on the wear resistance of the nickel-chromium alloy "Nikorin" under friction conditions at various loads

State of research samples	Specific load, kg/mm ²	Speed m/s	Sliding distance, km	Wear rate <i>i</i>	Wear resistance index <i>n</i>
Chromium-nickel alloy "Nikorin"	0.02	1	7.2	2.6×10^{-8}	7.58
	0.03	1	7.2	4.92×10^{-8}	7.3
	0.04	1	7.2	7.3×10^{-8}	7.13

Source [61–63]

- **Table 3.11** The results of studies on the wear resistance of the nickel-chromium alloy "Nikorin" under friction conditions at elevated temperatures and a load of 500 N

State of research samples	Load, [N]	Speed m/s	Sliding distance, km	Wear rate <i>i</i>	Friction coefficient μ	Wear resistance coefficient, <i>n</i>
Chromium-nickel alloy "Nikorin"	500	2	0.6	9.3×10^{-7}	0.4	6.03

Source [61–63]

Analysis of the results of tribological studies carried out at specific loads of 0.02–0.04 kg/mm² (**Table 3.10**) indicates that with an increase in the specific load, an increase in the intensity of wear (i) of the nickel-chromium alloy "Nikorin" in the cast state is observed with 2.6×10^{-8} to 7.3×10^{-8} , and the wear resistance coefficient (n) decreases (from 7.58 to 7.13).

Fig. 3.32 shows the dependence of the wear rate on the specific load of the nickel-chromium alloy "Nikorin". Analysis of the graphical dependencies indicates that the wear rate of the nickel-chromium alloy "Nikorin" increases significantly with increasing specific load. At a specific load of 0.04, this indicator is the highest.

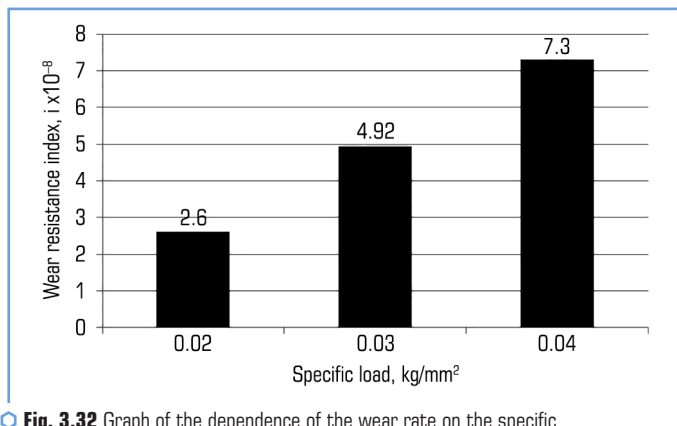


Fig. 3.32 Graph of the dependence of the wear rate on the specific load of the nickel-chromium alloy "Nikorin"

Fig. 3.33 shows the dependences of the wear resistance index of the nickel-chromium alloy "Nikorin" on the specific load at room temperature of tests. The analysis of the graphical dependencies indicates that the wear resistance indicator decreases with an increase in the specific load.

The work also carried out tribotechnical studies of samples of the nickel-chromium alloy "Nikorin" in the cast state at a load of 500 N. The test temperature in contact was 950 °C (**Table 3.11**).

Analysis of the test data for wear resistance under frictional conditions (**Table 3.11**) indicates that at a load of 500 N the nickel-chromium alloy "Nikorin" has a high wear rate (9.3×10^{-7}) and, accordingly, a low wear resistance coefficient (6.03), and the coefficient friction (μ) is 0.4. With an increase in loads to 600–750 N, the nickel-chromium alloy "Nikorin" began to collapse during testing.

Thus, the nickel-chromium alloy "Nikorin" in the cast state demonstrates the best wear resistance at loads of 0.02–0.04 kg/mm² (minimum wear rates and maximum wear resistance rates).

Frictional wear leads to a change in the structure, phase composition and properties of the nickel-chromium alloy "Nikorin".

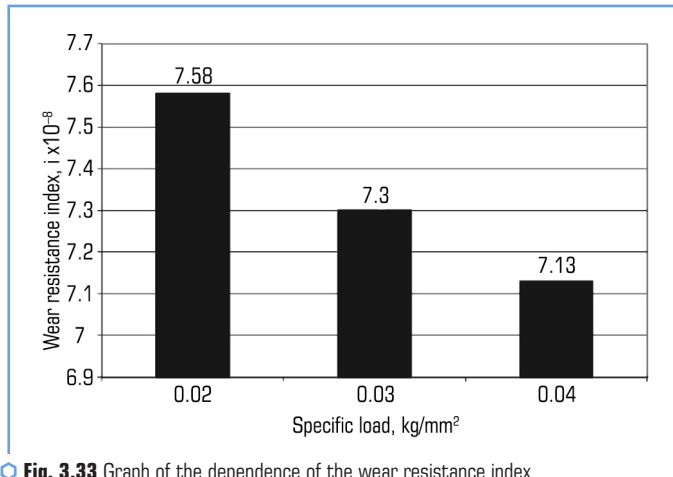


Fig. 3.33 Graph of the dependence of the wear resistance index of the nickel-chromium alloy "Nikorin" on the specific load

In Fig. 3.34 shows the structure of the nickel-chromium alloy "Nikorin" in the cast state after tribological tests at room temperature. The structure contains TiC carbides, which are located in the bulk of Cr_7C_3 carbides, which indicates their initial role in crystallization. Cr_7C_3 carbides are also destroyed during drift. However, since the chromium carbide in this alloy is not alloyed, its destruction occurs to a lesser extent and mainly at the TiC/ Cr_7C_3 interface. Significant fracture zones were also found at the TiC/matrix interface.

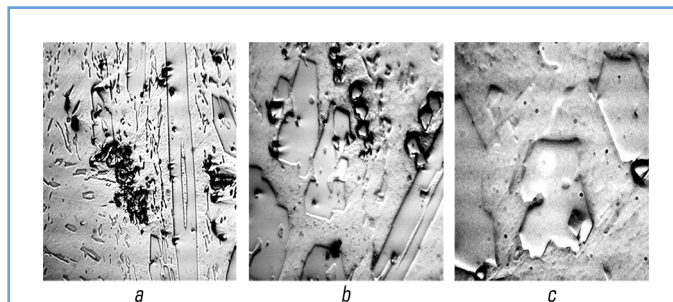


Fig. 3.34 Microstructure of the nickel-chromium alloy "Nikorin" in the cast state after testing at a temperature of 20 °C and a specific load of 0.04 kg/mm²: a – $\times 200$; b – $\times 500$; c – $\times 1000$

Friction wear of the nickel-chromium alloy "Nikorin" at a test temperature of 20 °C leads to a change in its phase composition.

In **Fig. 3.35** shows the diffraction patterns of the nickel-chromium alloy "Nikorin" in the cast state after tests at room temperature and a specific load of 0.04 kg/mm². Phase X-ray diffraction analysis made it possible to reveal in the "Nikorin" alloy Cr₇C₃ carbide, Fe₃C carbide and a matrix consisting of 14 % austenite and 86 % ferrite.

Table 3.12 shows the quantitative data of X-ray structural analysis of the research chromium-nickel alloy "Nikorin" before and after friction wear: the value $\beta_{0.5}$ of the (011) line_a, characterizing the degree of imperfection of the phase; amount of retained austenite (% γ); the residual austenite parameter (a_γ) and the α -phase parameter (a_α).

Data analysis **Table 3.12** indicates that the structure of the chromium-nickel alloy after frictional drift contains the α -phase, the degree of imperfection ($\beta_{0.5}$) of the lattice of which is 0.43, and the lattice parameter (a_α) is 2.91.

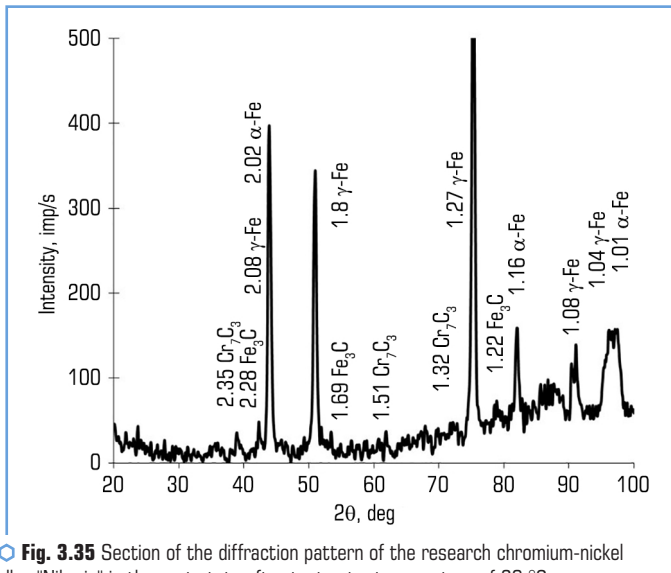


Fig. 3.35 Section of the diffraction pattern of the research chromium-nickel alloy "Nikorin" in the cast state after tests at a temperature of 20 °C and a specific load of 0.04 kg/mm²

Table 3.12 Quantitative data of X-ray structural analysis of the research chromium-nickel alloy "Nikorin" before and after friction wear at room temperature

Test temperature	a_α to (011) α		a_γ to (022) γ		Imperfection degree α – phase ($\beta_{0.5}$)		% γ , in the matrix	
	before wear	after wear	before wear	after wear	before wear	after wear	before wear	after wear
+20 °C	–	2.91	3.59	3.57	–	0.43	100	14

In the process of testing, measurements were made of the microhardness of the decomposition products of austenite, the eutectic component and the hardness of prototypes of the nickel-chromium alloy "Nikorin". **Table 3.13** shows the data of measurements of the microhardness of the matrix, eutectic carbide and the hardness of samples of the chromium-nickel alloy "Nikorin" before and after wear by friction at room temperature and a specific load of 0.04 kg/mm².

● **Table 3.13** Microhardness of matrix, eutectic carbide and hardness of samples of chromium-nickel alloy "Nikorin" before and after friction wear

Test temperature	Microhardness of structural components, MPa				Hardness, HRC	
	Decomposition products of austenite		Eutectic carbides		before wear	after wear
	before wear	after wear	before wear	after wear		
+20 °C	2769	2468	18921	17886	47	33

The frictional wear of the nickel-chromium alloy "Nikorin" leads to a decrease in the microhardness of the matrix (from 2769 MPa to 2468 MPa), carbide (from 18921 MPa to 17886 MPa) and the general rigidity of the test specimens. The hardness of the Nikorin chromium-nickel alloy after tribological tests is 33 HRC.

This nature of the change in the microhardness of the matrix, carbide and the total rigidity of the prototypes of the nickel-chromium alloy "Nikorin" is due to structural changes, as well as the redistribution of alloying elements between phases and structural components in the process of friction wear.

CONCLUSIONS

The structure, phase composition and properties of the nickel-chromium alloy "Nikorin" in the cast state were investigated:

- it has been found that the structure of the heat-resistant chromium-nickel alloy in the initial cast state consists of high-nickel austenite, high-chromium carbide Cr₇C₃, titanium carbide TiC and intermetallic compound Ni₃Al;
- by the method of X-ray structural analysis, the phase composition has been studied and the carbide analysis of research alloys has been carried out. It has been shown, that:
 - the presence of high-chromium carbides ensures high hardness of research alloys;
 - the σ-phase has been found in the nickel-chromium alloy "Nikorin", which negatively affects the operational properties of the alloy;
 - Ni₃Al intermetallic compounds have been found in the structure of the "Nikorin" alloy;

- the distribution of alloying elements between the phases and structural components in the nickel-chromium alloy "Nikorin" in the cast state has been studied;
- it has been established that heat-resistant chromium-nickel alloys have a homogeneous structure with a uniform distribution of alloying elements over the section of the casting;
- calorimetric analysis of research alloys during heating/cooling has been carried out. The crystallization/melting temperatures of the nickel-chromium alloy "Nikorin" were determined;
- specific structure, phase composition, microhardness of the structural components of the heat-resistant chromium-nickel alloy "Nikorin" after quenching from different temperatures;
- a carbide analysis has been, which confirmed the presence in the structure of a heat-resistant chromium-nickel alloy quenched from a temperature of 1150, 1100 °C of the σ -phase (FeCr);
- a comprehensive study of structure formation and phase transformations during crystallization and in the solid state of the nickel-chromium alloy "Nikorin" confirms the realization of peritectic-eutectic transformation in these alloys;
- a high-temperature hardness of the nickel-chromium alloy "Nikorin" in the cast state has been investigated;
- tests for wear resistance have been conducted under friction conditions at room and elevated temperatures and various loads of samples of nickel-chromium alloy "Nikorin" in the cast state;
- it has been found that the best indicators of wear resistance (minimum indicators of wear intensity and maximum indicators of wear resistance coefficient) are demonstrated by samples of nickel-chromium alloy "Nikorin" in the cast state at loads of 0.02–0.04 kg/mm². Frictional wear leads to a change in the structure, phase composition and properties of the nickel-chromium alloy "Nikorin";
- a decrease in the microhardness of the decomposition products of austenite, eutectic carbide and the total hardness of the tested samples has been observed. The hardness of the "Nikorin" chromium-nickel alloy after tribological tests is 33 HRC.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

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