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INFLUENCE OF HEAT TREATMENT ON STRUCTURE, PHASE COMPOSITION, HARDNESS AND ELECTRICAL CONDUCTIVITY OF VZhL14N-VI NICKEL SUPERALLOY

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Abstract. The phase composition of VZhL14N-VI nickel superalloy was analyzed in a wide temperature range – from room temperature to 1600 °C by means of CALPHAD (CALculation of PHase Diagrams) calculations. In light of the findings, the authors devised potential heat treatment modes for VZhL14N-VI superalloy. The impact of different heat treatment modes on the grain size, hardness, and electrical conductivity of VZhL14N-VI superalloy samples produced by ceramic mold casting was investigated, as well as the effect on the alloy of high-temperature annealing at 1070 – 1170 °C for 1 – 4 h. The alloy heat treatment resulted in a notable increase in grain size and a decrease in hardness. The influence of artificial aging temperature after high-temperature annealing and quenching on the hardness and electrical conductivity of the alloy in the range of 610 – 810 °C was studied. At 810 °C, the alloy exhibits the most pronounced aging effect, accompanied by a rapid increase in hardness, reaching approximately 370 HV. In contrast to the observed changes in hardness, the electrical conductivity of the alloy exhibited minimal variation during the aging process. The proposed heat treatment conditions diverge from those recommended by the OST 1 90126–85 Russian standard for this alloy. The developed heat treatment mode includes the alloy heat treatment at a temperature of 1170 ± 10 °C for 4 h, followed by air cooling and aging at a temperature of 810 ± 10 °C for 10 – 14 h, followed by air cooling. The proposed heat treatment mode is expected to result in an increase in hardness of VZhL14N-VI superalloy castings by 10 – 20 HV in comparison to the samples subjected to the standard heat treatment mode.

Keywords: VZhL14N-VI nickel superalloy, investment casting, heat treatment, castings

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ВЛИЯНИЕ ТЕРМИЧЕСКОЙ ОБРАБОТКИ НА ФАЗОВЫЙ СОСТАВ, СТРУКТУРУ, ТВЕРДОСТЬ И ЭЛЕКТРОПРОВОДНОСТЬ НИКЕЛЕВОГО ЖАРОПРОЧНОГО СПЛАВА ВЖЛ14Н-ВИ

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Аннотация. В работе проанализирован фазовый состав никелевого жаропрочного сплава ВЖЛ14Н-ВИ в широком температурном диапазоне – от комнатной температуры до 1600 °С с помощью расчетов по программе Thermo-Calc. На основании полученных данных авторы разработали возможные режимы термообработки жаропрочного сплава ВЖЛ14Н-ВИ. Исследовано влияние различных режимов термообработки на размер зерна, твердость и электропроводность образцов жаропрочного сплава ВЖЛ14Н-ВИ, полученных методом литья в керамические формы, а также влияние на сплав высокотемпературного отжига при температуре 1070 – 1170 °С в течение 1 – 4 ч. Термическая обработка сплава привела к заметному увеличению размера зерен и снижению твердости. Было изучено влияние температуры искусственного старения после высокотемпературного отжига и закалки на твердость и электропроводность сплава в диапазоне температур 610 – 810 °С. При температуре 810 °С сплав проявляет наиболее выраженный эффект старения, сопровождающийся быстрым повышением твердости, достигающим приблизительно 370 HV. В отличие от твердости, электропроводность сплава в процессе старения изменялась незначительно. Предлагаемый режим термической обработки отличается от рекомендованного стандартом ОСТ 1 90126–85 для этого сплава. Он включает отжиг при температуре 1170 ± 10 °С в течение 4 ч с последующим охлаждением на воздухе и старением при температуре 810 ± 10 °С в течение 10 – 14 ч с последующим охлаждением на воздухе. Предложенная термообработка позволяет повысить твердость отливок из сплава ВЖЛ14Н-ВИ на 10 – 20 HV по сравнению с образцами, подвергнутыми термообработке по стандартному режиму.

Ключевые слова: никелевый жаропрочный сплав ВЖЛ14Н-ВИ, литье по выплавляемым моделям, термическая обработка, отливки

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INTRODUCTION

Currently, a wide range of nickel-based heat-resistant cast superalloys have been developed and successfully applied for the production of heat-resistant structural components in aircraft engines [1; 2]. In Russian aircraft engineering, VZhL14N-VI alloy (OST 1 90126–85; base – Ni; up to 0.08 % C¹; up to 20.0 % Cr; up to 5.0 % Mo; up to 1.5 % Al; up to 2.9 % Ti; up to 2.8 % Nb; up to 10 % Fe) is widely used for large-scale structural castings, particularly in combustion chamber components. This alloy primarily contains chromium and iron as major alloying elements and has no direct foreign equivalents. The closest in composition are Inconel 718 (ASTM Ni B670) and Inconel 718Plus (UNS N07818) alloys. These belong to the second generation of superalloys, characterized by a high chromium content, often with cobalt, as well as refractory elements such as tungsten,

molybdenum, and niobium. A key feature of these alloys is their substantial γ' phase content (a face-centered cubic ordered Ni₃(Al, Ti) phase) [1; 3; 4]. The primary requirement for combustion chamber structural components, which operate at moderate temperatures of 500 – 900 °C and pressures up to 45 atm, is high strength combined with good ductility across the entire temperature range [5]. This is achieved through the complex alloying of the Fe–Ni alloy with solid-solution strengthening and carbide-forming elements. Chromium is a component of the Ni-based solid solution, contributing to its strengthening, while also being involved in carbide formation. This significantly enhances the oxidation resistance of the Ni–Fe alloy at operating temperatures [4 – 6]. Aluminum and titanium promote the formation of the γ' phase and strengthen the alloy during heat treatment. Molybdenum, tungsten, and niobium are added as carbide-forming elements responsible for the formation of fine-dispersed carbides, which enhance the alloy's high-temperature strength. They also strengthen the solid solution, while niobium, in addition, participates in the formation of strengthening

¹ Here and throughout the text, unless otherwise stated, element contents are given in wt. %.

precipitates in the nickel-based solid solution slightly reducing the processability of the alloy by impairing the weldability and machinability of castings [7; 8]. The addition of iron reduces the overall cost of the alloy due to the lower cost of iron compared to nickel, replacing part of the nickel content. At a moderate iron content, as in VZhL14N-VI alloy, its long-term strength at the operating temperatures of combustion chamber components remains at an acceptable level.

The operational properties of VZhL14N-VI alloy castings are strongly influenced by the quantity of alloying elements, the carbide and strengthening phases they form, and their distribution within the alloy structure. While the impact of individual alloying elements and structural components on the operational properties of VZhL14N-VI alloy has been well studied [1–5], the effect of structure and phase composition formation conditions on the mechanical properties of castings remains insufficiently explored. This often results in deviations of the alloy's properties from expected values [9; 10]. It is crucial to consider both the formation of the as-cast structure and the structure obtained after heat treatment [1; 10; 11]. The operational properties of cast components are determined by the combination of grain size and metal structure. The hardening of VZhL14N-VI-type alloys is primarily governed by the formation of γ' strengthening precipitates within the γ matrix phase. These precipitates exhibit greater thermal stability compared to γ'' (a body-centered tetragonal ordered Ni_3Nb phase), which forms in other heat-resistant nickel alloys. Additionally, the hardening of the alloy is influenced by the precipitation of δ (orthorhombic Ni_3Nb), η (hexagonal Ni_3Ti), and σ (hexagonal CrFeMoNi , CrMoNi , $(\text{Cr}, \text{Mo})_3\text{Ni}$) phases, along with carbide phases such as MeC , Me_{23}C_6 , and Me_6C (Me_7C_3 is rarely observed). These phases have a face-centered cubic lattice and limit grain growth in the alloy [11–14]. By adjusting the heat treatment modes to control the formation of these phases, the mechanical properties of cast components can be modified within a relatively broad range.

This study examines the effect of heat treatment modes on the macro- and microstructure, phase composition, and hardness of VZhL14N-VI alloy samples produced by ceramic mold casting to determine their influence on alloy hardening.

MATERIALS AND METHODS

The test samples were cut from cast plates measuring $100 \times 100 \times 10$ mm. The castings were produced using ceramic mold casting technology. Fused quartz of various fractions, manufactured by DINUR (Pervouralsk, Russia), was used as the filler for the slurry and stucco coating. To prepare the refractory slurry, Ultracast One+ and Ultracast Prime binders (Technopark LLC, Moscow,

Russia) were used. Pre-alloyed VZhL14N-VI, produced by VIAM (Moscow, Russia), was used as the charge material. The melting and pouring of the alloy were performed in a vacuum induction melting and casting unit (VAKETO, Moscow, Russia) using a mullite-corundum-zirconium crucible manufactured by ELEMET (Elektrogorsk, Russia). From the obtained castings, samples measuring $4 \times 9 \times 56$ mm were cut. The alloy samples were subjected to heat treatment in a muffle furnace in air at temperatures of 1070, 1120, and 1170 °C. The duration of high-temperature treatment ranged from 1 to 4 h, with 1 h increments. The samples were quenched by air blow cooling. For aging, samples quenched after 4 h of high-temperature soaking were used. The aging process was studied under three heat treatment modes, involving soaking at 610, 700, and 810 ± 10 °C for 2 to 14 h.

The microstructure of the alloy was examined using a TESCAN VEGA 3 SBH scanning electron microscope (SEM) equipped with an Oxford energy-dispersive microanalysis system and a Carl Zeiss Axio Observer D1m optical microscope. To reveal the grain structure, metallographic samples were etched using Marble's reagent (20 g Cu_2SO_4 , 100 mL hydrochloric acid, 100 mL ethanol) [15]. To examine the microstructure, an acid mixture (30 mL nitric acid, 0.5 mL hydrochloric acid, 70 mL acetic acid) was also used for etching [16]. The phases present in the microstructure were identified using energy-dispersive X-ray spectroscopy (EDS), based on literature data concerning possible compounds in heat-resistant nickel-based alloys [14; 17] and phase composition calculations.

The electrical conductivity of the alloys was measured using a non-contact eddy current conductometer VE-27NC3 (SIGMA, Russia), with a measurement range of 0.5–2.5 MS/m.

Vickers hardness (HV 10) was determined using a NEMESIS 9001 universal hardness tester (INNOVATEST) under a 10 kgf load, with a loading time of 12 s.

The equilibrium phase composition of VZhL14N-VI alloy in the temperature range of 20 to 1600 °C was calculated using the Thermo-Calc software with the TCS Ni-based Superalloys Database (TCNI8).

RESULTS AND DISCUSSION

To achieve a more precise phase identification, the phase composition of the VZhL14N-VI alloy was calculated in the temperature range of 20 to 1600 °C using Thermo-Calc software. The results of the equilibrium phase composition calculation, based on the average composition of VZhL14N-VI alloy in accordance with OST 1 90126–85, are presented in Fig. 1.

It can be observed that from the equilibrium solidus temperature (approximately 1280 °C) down to 1050 °C, the alloy is almost single-phase, consisting primarily

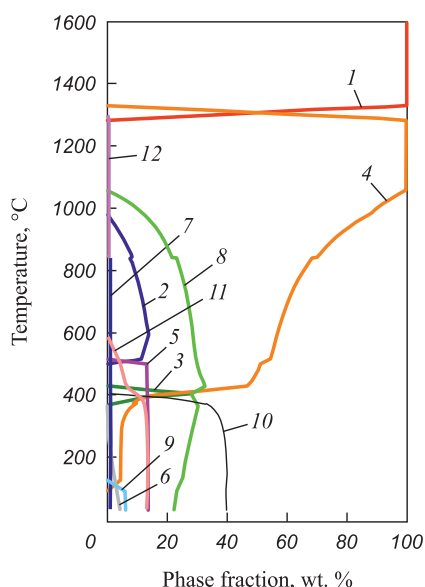


Fig. 1. Calculated phase composition of VZhL14N-VI superalloy (average composition) at temperatures from 20 to 1600 °C:

1 – L; 2 – $\sigma(\text{Cr,Mo})_3\text{Ni}$; 3 – Ni_3Fe ; 4 – γ ; 5 – $\text{P}(\text{NiCrMo})$; 6 – Ni_3Nb ; 7 – Cr_{23}C_6 ; 8 – γ' ; 9 – Ni_3Cr ; 10 – Ni_3Fe ; 11 – (Cr); 12 – (Nb,Ti)C

Рис. 1. Расчетный фазовый состав сплава ВЖЛ14Н-ВИ (средний состав) при температурах от 20 до 1600 °C:

1 – L; 2 – $\sigma(\text{Cr,Mo})_3\text{Ni}$; 3 – Ni_3Fe ; 4 – γ ; 5 – $\text{P}(\text{NiCrMo})$; 6 – Ni_3Nb ; 7 – Cr_{23}C_6 ; 8 – γ' ; 9 – Ni_3Cr ; 10 – Ni_3Fe ; 11 – (Cr); 12 – (Nb,Ti)C

of the γ phase. Below 1050 °C, the main strengthening phase for this alloy, γ' , begins to precipitate [18]. At approximately 980 °C, the σ phase starts to form, which is commonly observed in heat-resistant nickel-based alloys containing iron and typically appears as irregularly shaped globules. With a further decrease in temperature, the volume fractions of γ' and σ phases gradually increase. At approximately 850 °C, carbide inclusions of Me_{23}C_6 start to appear in the structure, where Me is primarily chromium, along with iron and molybdenum.

At 600 °C, the equilibrium content of γ' and σ phases reaches its maximum. When the temperature drops below 600 °C, unwanted particles with a chromium-based solid solution lattice begin to appear in the equilibrium structure. Below 500 °C, the σ phase completely disappears, and other undesirable phases emerge in the structure. Additionally, the equilibrium volume fractions of γ and γ' phases decrease significantly below 400 °C. Thus, within the operating temperature range of combustion chamber cast components in aircraft gas turbine engines made from VZhL14N-VI alloy (750 – 950 °C) [19], the equilibrium phase composition consists of the γ phase, with dispersed γ' and σ phase particles, where the γ' phase predominates. Additionally, a small number of carbide particles are present in the structure, primarily Me_{23}C_6 carbides, with occasional occurrences of MeC carbides.

The as-cast structure of VZhL14N-VI alloy, produced by ceramic mold casting, is shown in Fig. 2, a. The microstructure consists of a nickel-based solid solution matrix (γ phase) with finely dispersed carbide precipitates containing niobium, titanium, and a significant amount of molybdenum, in addition to carbon (Fig. 2, b). According to the calculated phase composition of the alloy (Fig. 1), these carbides begin to precipitate directly from the liquid phase. During heat treatment at temperatures below 850 °C, they are expected to transform into Me_{23}C_6 -type carbides.

Fig. 3 presents the microstructure of VZhL14N-VI alloy after heat treatment following the mode specified in OST 1 90126–85 for this alloy. The treatment includes annealing at 1120 ± 10 °C for 3 h, followed by air cooling, and aging at 700 ± 10 °C for 16 h, with subsequent air cooling.

As seen in Fig. 3, a, heat treatment led to a reduction in dendritic segregation of elements, although complete chemical homogenization was not achieved. Despite ther-

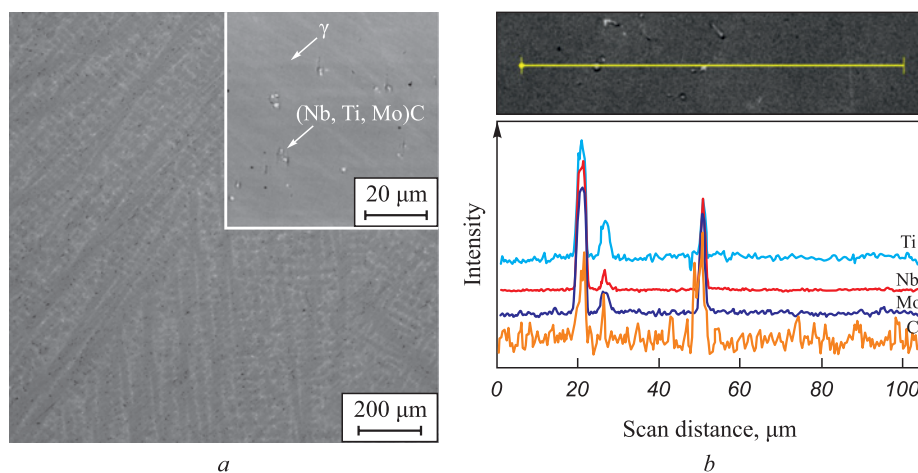


Fig. 2. Microstructure of VZhL14N-VI superalloy obtained by ceramic mold casting (a) and element distribution profile based on EDS results (b)

Рис. 2. Микроструктура сплава ВЖЛ14Н-ВИ, полученного литьем в оболочковую керамическую форму (a), и профиль распределения элементов по результатам микрорентгеноспектрального анализа (b)

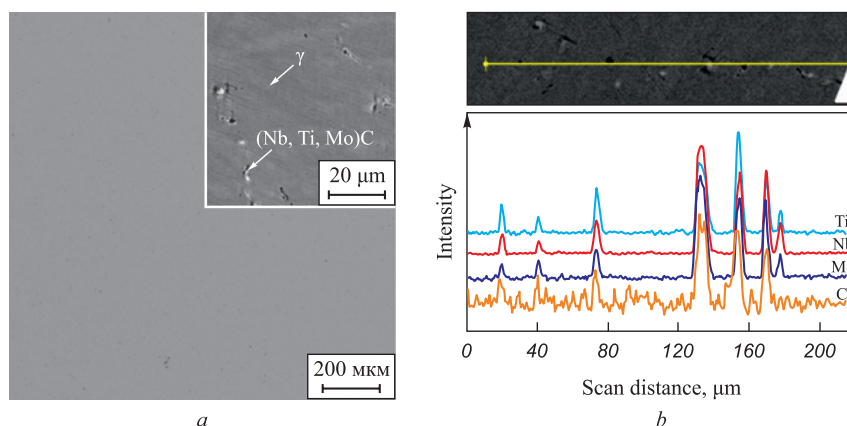


Fig. 3. Microstructure of VZhL14N-VI superalloy after heat treatment including annealing at $1120 \pm 10^\circ\text{C}$ for 3 h followed by air cooling and aging at $700 \pm 10^\circ\text{C}$ for 16 h followed by air cooling (a) and element distribution profile based on EDS results (b)

Рис. 3. Микроструктура сплава ВЖЛ14Н-ВИ после термической обработки по режиму, включающему отжиг при температуре $1120 \pm 10^\circ\text{C}$ в течение 3 ч с последующим охлаждением на воздухе и старением при температуре $700 \pm 10^\circ\text{C}$ в течение 16 ч с последующим охлаждением на воздухе (a), и профиль распределения элементов по результатам микрорентгеноспектрального анализа (b)

modynamic calculations predicting that niobium and titanium carbides should dissolve during high-temperature annealing and be replaced by ultrafine chromium-based Me_{23}C_6 carbide precipitates during aging, niobium and titanium carbides were still observed in the alloy structure after heat treatment. Additionally, some carbide particles exhibited increased nitrogen content, which most likely inhibits their dissolution.

It is well known that, according to the Hall–Petch effect, the grain size of polycrystalline nickel-based alloy castings significantly affects their mechanical properties [20]. The influence of various high-temperature heat treatment modes on grain size was studied. Fig. 4, a shows the grain structure of samples solidified under identical cooling conditions in the as-cast state and after heat treat-

ment. Grain coarsening occurs during annealing. Fig. 4, b illustrates the effect of high-temperature heat treatment duration on the grain density on the polished sample surface. During 4 h of annealing, the number of grains per 1 cm^2 in the metallographic section decreased by approximately 2.6 times, regardless of the annealing temperature. Thus, high-temperature annealing significantly increases the grain size of the alloy, which is a negative factor that reduces the mechanical properties of cast components. Therefore, excessive annealing time is undesirable. Changing the annealing temperature within the studied range ($1070 - 1170^\circ\text{C}$) had virtually no effect on the grain size of the alloy (Fig. 4, b).

Fig. 5 presents the effect of heat treatment on the hardness and electrical conductivity of VZhL14N-VI

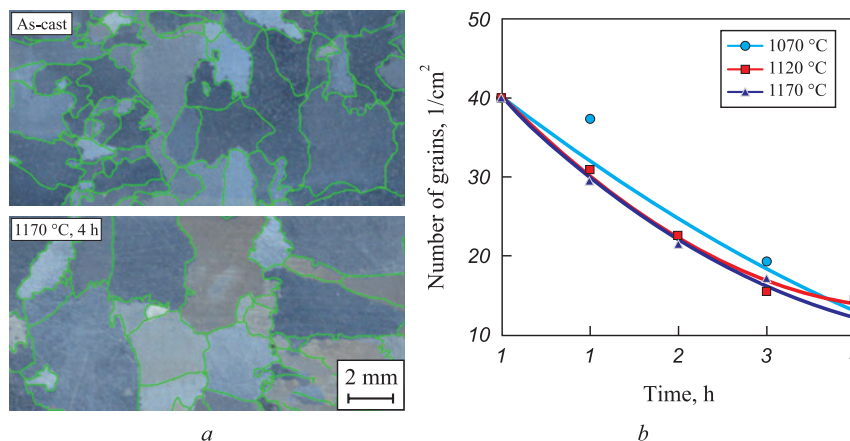


Fig. 4. Grain structure of VZhL14N-VI superalloy in as-cast condition and after high-temperature heat-treatment at 1170°C for 4 h with following air cooling (a) and dependence of the number of grains per 1 cm^2 in the metallographic section on temperature and time of annealing (b)

Рис. 4. Макроструктура сплава ВЖЛ14Н-ВИ в литом состоянии и после высокотемпературного отжига при 1170°C в течение 4 ч с последующим охлаждением на воздухе (a) и зависимость количества зерен на 1 cm^2 поверхности шлифа от температуры и времени отжига (b)

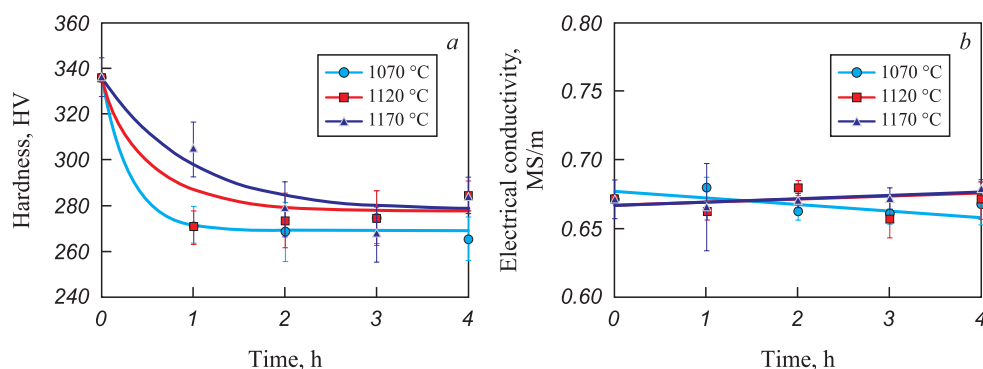


Fig. 5. Influence of temperature and time of holding during high-temperature annealing on hardness (a) and electrical conductivity (b) of VZhL14N-VI superalloy

Рис. 5. Влияние температуры и времени выдержки в процессе высокотемпературного отжига на твердость (a) и электропроводность (b) сплава ВЖЛ14Н-ВИ

alloy samples. The temperature of high-temperature annealing within the examined range had a negligible effect on hardness (Fig. 5, a) after quenching. During high-temperature annealing, the hardness of the alloy rapidly decreased from 336 to ~280 HV for all temperatures used. The influence of high-temperature annealing on the electrical conductivity of the alloy was even smaller (Fig. 5, b). It can be assumed that the electrical conductivity of the alloy remains unchanged during annealing. However, it should be noted that the electrical conductivity measurement method used in this study is relatively crude. Given the small variations in measured electrical conductivity values observed for VZhL14N-VI alloy, the measurement error is relatively high. It is possible that more precise measurement techniques could detect changes in electrical conductivity.

The temperature and duration of artificial aging had a much stronger effect on alloy hardness than the annealing temperature prior to quenching (Fig. 6). The most significant increase in hardness was observed at the maximum aging temperature of 810 °C. At this temperature, the hardness of the alloy reached its near-maximum value of approximately 370 HV within just 2 h of aging. Lower aging temperatures did not result in such significant hardening. The lowest hardness values were observed at 610 °C, where the hardness of the alloy continued to increase throughout the aging process, indicating incomplete decomposition of the supersaturated solid solution. It is evident that this temperature is insufficient for achieving maximum strength through artificial aging. An intermediate hardness level was obtained after aging at 700 °C. The maximum hardening effect, achieved at 810 °C, was practically independent of the annealing temperature before quenching (Fig. 6, d), showing consistent results.

The degree of supersaturated γ -phase decomposition can be indirectly assessed based on changes in electrical conductivity [19]. For the VZhL14N-VI alloy, a weak dependence of electrical conductivity on aging tempera-

ture and duration was observed (Fig. 7). However, while the electrical conductivity of samples aged at 610 and 700 °C was nearly identical, at 810 °C, a significant difference exceeding the measurement uncertainty interval was detected. The electrical conductivity of samples aged at 810 °C was higher than that of samples aged at 610 and 700 °C. This suggests that at 810 °C, γ phase decomposition occurs at a higher rate. At the same time, the electrical conductivity of alloys subjected to high-temperature annealing for solid solution treatment at different temperatures varies insignificantly during aging (Fig. 7, d).

Thus, the highest hardening effect was achieved in VZhL14N-VI alloy samples that were annealed at 1170 °C for 4 h and then artificially aged at 810 °C for 10 – 14 h. The proposed heat treatment mode differs slightly from the current standard specified in OST 1 90126–85 and results in a small hardness increase of 10 – 20 HV in VZhL14N-VI alloy. This mode may be of interest for heat treatment of cast components operating at temperatures above 800 °C.

CONCLUSIONS

The as-cast grain structure of VZhL14N-VI alloy primarily consists of a γ phase solid solution with (Nb, Ti, Mo)C carbide inclusions.

During heat treatment, which includes high-temperature solution annealing followed by artificial aging, a noticeable reduction in coring of alloying elements within the γ phase solid solution is observed. However, the morphology of niobium and molybdenum carbides, identified through X-ray microanalysis, remains unchanged.

Thermodynamic calculations have shown that aging at temperatures below 600 °C may lead to the formation of undesirable phases in the alloy structure, while aging above 850 °C results in a reduction in the volume fraction of hardening γ' and σ particles and inhibits the precipitation of $Me_{23}C_6$ -type carbides.

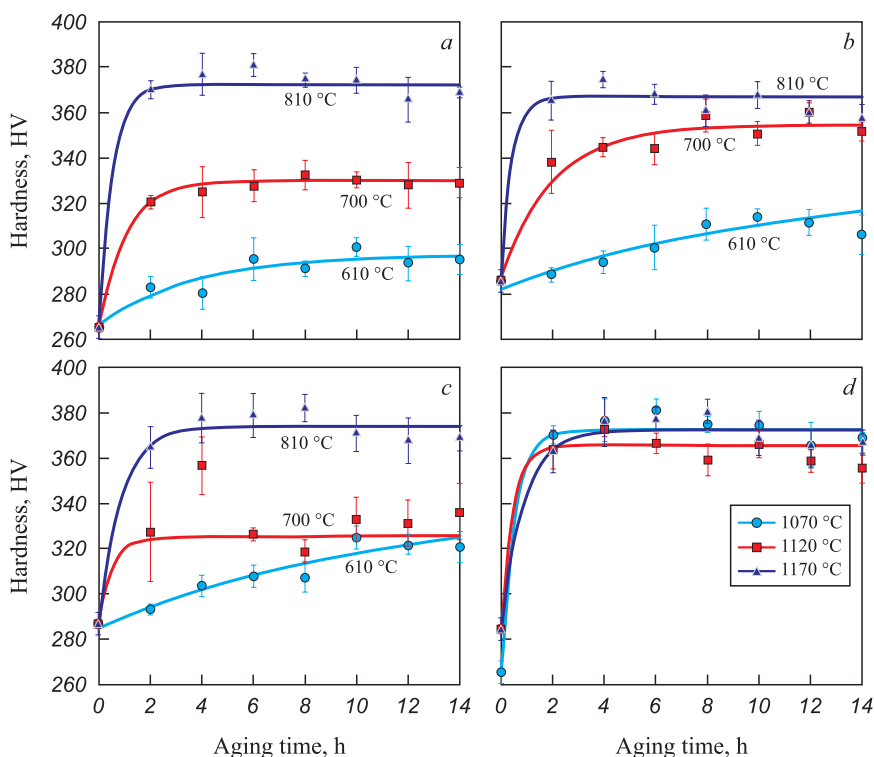


Fig. 6. Influence of high-temperature annealing at 1070 (a), 1120 (b), 1170 °C (c) and aging temperature on hardness of VZhL14N-VI superalloy, and effect of changing the annealing temperature on hardening of the alloy during aging at 810 °C (d)

Рис. 6. Влияние температуры высокотемпературного отжига 1070 (a), 1120 (b), 1170 °C (c) и температуры старения на твердость сплава ВЖЛ14Н-ВИ и изменения температуры отжига на твердый раствор на упрочнение сплава при старении при 810 °C (d)

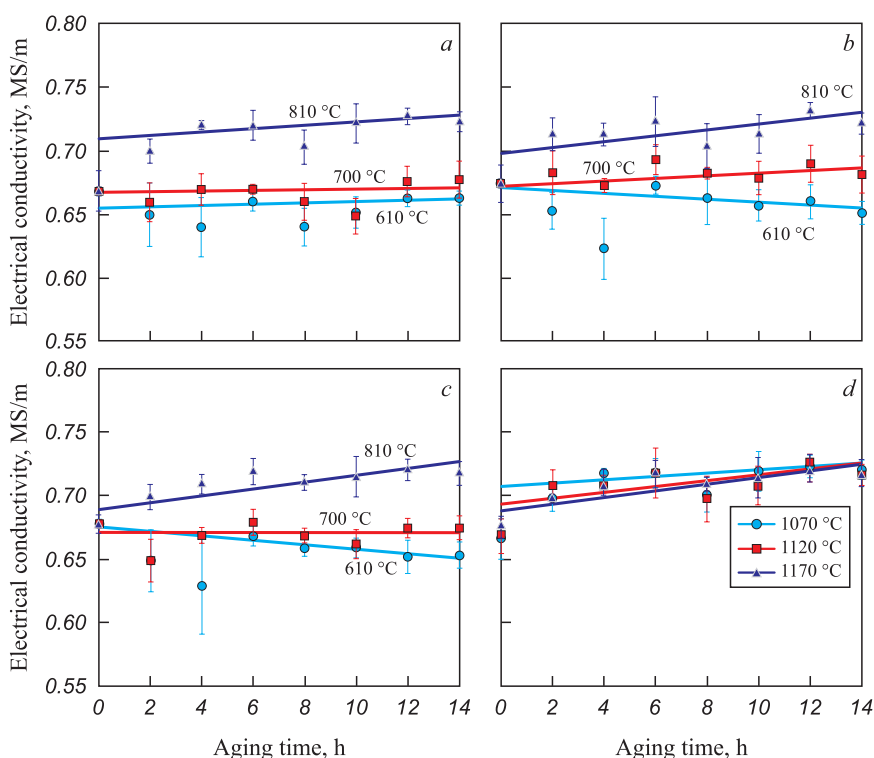


Fig. 7. Influence of high-temperature annealing at 1070 (a), 1120 (b), 1170 °C (c) and aging temperature on electrical conductivity of VZhL14N-VI superalloy, and effect of changing the annealing temperature on electrical conductivity of the alloy during aging at 810 °C (d)

Рис. 7. Влияние температуры высокотемпературного отжига 1070 (a), 1120 (b), 1170 °C (c) и температуры старения на электропроводность сплава ВЖЛ14Н-ВИ и изменения температуры отжига на электропроводность сплава при старении при 810 °C (d)

It has been established that the highest hardening effect is observed in alloys aged at 810 °C, ensuring the most complete decomposition of the supersaturated γ phase solid solution. At the same time, within the studied range of 1070 – 1170 °C, the solution annealing temperature has a significantly smaller effect on the hardening of the alloy compared to the aging temperature. The maximum hardness during aging is reached within 4 – 6 h and remains practically unchanged up to 10 – 14 h.

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