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INFLUENCE OF HEAT TREATMENT MODES ON THE PROPERTIES OF 56DGNKH (Cu₂₀Ni₂₀Mn₂Cr) ALLOY

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Abstract. Alloys of the Cu–Ni–Mn system are used in many areas, and for some applications (watchmaking, dentistry, precision mechanics) they must have high hardness. A state of high hardness can be achieved by two-stage heat treatment – quenching and subsequent aging. To obtain a good set of performance characteristics, decomposition of the solid solution must proceed through a continuous mechanism, which can be regulated by additional alloying (for example, chromium) and aging parameters. In this work, we studied the influence of quenching and aging modes on microhardness of 56DGNKh (Cu₂₀Ni₂₀Mn₂Cr) alloy. It was shown that quenching from temperatures of 700 – 750 °C provides higher microhardness values than quenching from 800 °C. By varying the temperature and duration of aging, it was found that the maximum microhardness is observed at aging temperatures of 475 – 500 °C. Metallographic analysis shows that in this case, the supersaturated solid solution of Mn, Ni and Cr in copper decomposes into a less supersaturated solid solution and the precipitation of MnNi intermetallic particles occurs according to a continuous mechanism. The change in microhardness of 56DGNKh alloy depending on the aging time is multi-stage: its increase at short exposures is replaced by a subsequent decrease at increasing exposure with a clearly defined maximum or “plateau” between these two parts of the graph, and this type of dependence is observed at all aging temperatures. X-ray diffraction phase analysis shows that during the aging process, concentration of the solid solution decreases and MnNi particles are formed, the crystal lattice period of which differs from the period of the solid solution by 50 pm. The observed patterns of changes in hardness during the aging process are explained from the standpoint of the general theory of decomposition of supersaturated solid solutions. The maximum increase in microhardness (up to 450 kgf/mm² versus 130 – 160 kgf/mm² in the state after quenching) is achieved at a coherent or semi-coherent interface between MnNi particles and a Ni-based solid solution. This is observed after quenching from 750 °C and aging at 475 °C for 10 h.

Keywords: copper alloy, heat treatment, quenching, aging, microhardness, structure, X-ray phase analysis, decomposition of solid solution

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ИССЛЕДОВАНИЕ ВЛИЯНИЯ РЕЖИМОВ ТЕРМИЧЕСКОЙ ОБРАБОТКИ НА СВОЙСТВА СПЛАВА 56ДГНХ

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Аннотация. Сплавы системы Cu–Ni–Mn находят применение во многих областях и для некоторых из них (часовое производство, стоматология, точная механика) должны обладать высокой твердостью. Состояние с высокой твердостью достигается двухстадийной термической обработкой – закалкой и последующим старением. Для получения хорошего комплекса эксплуатационных характеристик распад твердого раствора должен идти по механизму непрерывного распада, что можно регулировать дополнительным легированием (например, хромом) и параметрами режима старения. В работе изучено влияние режимов закалки и старения на микротвердость сплава 56ДГНХ. Показано, что закалка от температур 700 – 750 °C обеспечивает большие значения микротвердости, чем закалка от 800 °C. Варьированием температуры и длительности старения найдено, что максимум микротвердости наблюдается при температурах старения

475 – 500 °С. Металлографический анализ показывает, что при этом происходит распад пересыщенного твердого раствора Mn, Ni и Cr в меди на менее пересыщенный твердый раствор и выделение частиц интерметаллида MnNi идет по механизму непрерывного распада. Изменение микротвердости сплава 56ДГНХ в зависимости от времени старения многостадийно. Ее рост при небольших выдержках сменяется последующим снижением при увеличении выдержки с отчетливо выраженным максимумом либо «плато» между этими двумя частями графика. Такой характер зависимости наблюдается при всех температурах старения. Рентгеноструктурный фазовый анализ показывает, что в процессе старения происходит уменьшение концентрации твердого раствора и образование частиц MnNi, период кристаллической решетки которых отличается от периода твердого раствора на 50 пм. Наблюдаемые закономерности изменения микротвердости в процессе старения объяснены с позиций общей теории распада пересыщенных твердых растворов. Максимум прироста микротвердости (до HV 0,5 = 45 кгс/мм² против HV 0,5 = 130 – 160 кгс/мм² в закаленном состоянии) достигается при когерентной или полукогерентной границе раздела частиц MnNi и твердого раствора на основе никеля. Это наблюдается после закалки от 750 °С и старения при 475 °С в течение 10 ч.

Ключевые слова: сплавы меди, термическая обработка, закалка, старение, микротвердость, структура, рентгеновский фазовый анализ, распад твердых растворов

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INTRODUCTION

Alloys of the Cu–Ni–Mn ternary system are used in watchmaking for the manufacture of high-precision, small-sized components. They are also employed as high-temperature brazing alloys for brazing components with a high coefficient of linear expansion (CLE), such as glass. Additionally, they are used as dental materials for crowns and bridges due to the similarity of their CLE to that of tooth tissues. For application in some of these fields, they must possess sufficiently high hardness [1].

Despite the existing research on the Cu–Ni–Mn ternary system, some of its alloys remain insufficiently studied. At present, they are considered promising for applications in precision mechanics, electronics, and medicine due to their excellent corrosion resistance, stable coefficient of linear expansion (CLE), adequate elasticity, and valuable aesthetic properties.

The aim of this study is to investigate the effect of various heat treatment modes on the mechanical properties of 56DGNKh alloy.

Alloys of the Cu–Ni–Mn system can exist in two states: metastable and stable [2; 3]. After rapid cooling from temperatures not exceeding 910 °С they exhibit the structure of a supersaturated solid solution of nickel and manganese in copper and remain in a metastable state. Heating the metastable state leads to the formation of a stable two-phase structure, consisting of a solid solution of nickel and manganese in the copper lattice and a θ -phase, which is a homogeneous ordered solid solution that can be represented by the general formula MnNi [4; 5].

Fig. 1 shows a segment of the isothermal section of the Cu–Ni–Mn ternary phase diagram at 450 °С. The line of equal mass fractions of nickel and manganese also represents the line of minimal copper solubility in the MnNi compound. In alloys with compositions lying on this line, the amount of θ -phase is at its maximum. Based on this, alloys with equal nickel and manganese

contents, specifically 60 % Cu – 20 % Ni – 20 % Mn, are considered technically promising [6; 7].

An important factor influencing the structural strength of the 56DGNKh alloy (i.e., the favorable combination of strength, ductility, and hardness) is the structure of the θ -phase. Depending on the temperature at which it forms, the θ -phase may develop either through a discontinuous decomposition mechanism or a continuous decomposition mechanism of the supersaturated solid solution [8]. Continuous decomposition results in a fine-dispersed structure uniformly distributed throughout the original copper grain, whereas discontinuous decomposition promotes the growth of θ -phase precipitates from the grain boundaries, which reduces the mechanical properties of 56DGNKh alloy [9 – 12].

All processes based on the phenomenon of diffusion-driven decomposition of a solid solution are governed by the rate of this phase transformation. Proper alloying enables the acceleration of these processes without

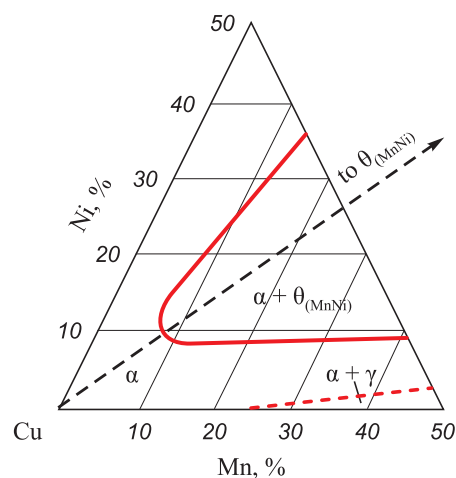


Fig. 1. Isothermal section of phase diagram of the Cu – Ni – Mn system at 450 °С [7]

Рис. 1. Изотермический разрез диаграммы состояния системы Cu – Ni – Mn при 450 °С [7]

degrading the structural characteristics of the precipitated particles. As calorimetric studies have shown [13], additional alloying of the Cu–Ni–Mn system with chromium in an amount of 1.8 – 2.2 wt. % allows for the highest rate of initial decomposition of the supersaturated solid solution and promotes the continuous decomposition mechanism. During continuous decomposition, a large number of MnNi particles exceeding 5 nm in size forms within the grain at the initial stage of the process, effectively preventing the growth of discontinuous decomposition regions from the grain boundaries [14; 15]. It should also be noted that regardless of the type of decomposition occurring, equilibrium is not fully reached even after prolonged aging treatments of more than 100 h [13; 16].

The aim of this study is to determine the optimal aging parameters for 56DGNKh alloy to maximize hardness under conditions that promote the continuous decomposition of its supersaturated solid solution.

EXPERIMENTAL METHODOLOGY

This study examined samples of 56DGNKh alloy, whose chemical composition is presented in the Table. The alloy was produced by induction melting in a protective atmosphere. The material was not subjected to homogenization annealing. Rods with a diameter of approximately 40 mm were obtained by hot forging of the ingot.

The samples used for testing measured approximately 5×5×7 mm. Their heat treatment was conducted in two stages: quenching and aging, both performed in vacuum inside an evacuated quartz ampoule. Quenching was carried out from temperatures ranging from 700 to 800 °C, with a 30-min holding time under a vacuum of approximately 10⁻² mmHg. Cooling involved removing the ampoule from the furnace and allowing it to cool without air admission. Under these conditions, the samples cooled from the heating temperature to approximately 150 °C in 2.5 min, followed by further cooling in air. The effect of quenching on the formation of a homogeneous solid solution was confirmed by the low hardness of the samples and the microstructure, which corresponded to a solid solution with approximately equiaxed grains ranging from 25 to 45 μm, with some annealing twins, this was also supported by X-ray phase analysis results. The decomposition of the metastable supersaturated solid solution was achieved through the thermal aging process. Aging was performed in a muffle furnace in air for 2, 7, 10, 12, and 25 h at temperatures ranging

Chemical composition of 56DGNKh alloy, wt. %

Химический состав сплава 56ДГНХ, мас. %

Cu	Ni	Mn	Cr
55 – 57	20 – 22	20 – 22	1.8 – 2.2

from 375 to 525 °C. After aging, the samples were cooled in air. Three samples were tested for each aging mode.

Microhardness measurements and microstructural analysis using metallographic methods were conducted on sections located at least 1 mm from the sample surface. The polished samples, prepared using standard surface preparation techniques, were etched with *aqua regia* [17] for 30 – 60 s. Microhardness (*HV*, in kgf/mm²) was measured using a PMT-3 microhardness tester following the Vickers method under a 500 g load. Variations in microhardness values between samples (up to 90 HV) due to liquation heterogeneity significantly exceeded the measurement error for an individual sample (maximum 11 HV). Therefore, the graphs present the average microhardness values obtained for each sample, without indicating the measurement spread.

The phase composition of the alloy was analyzed by X-ray phase analysis using a DRON-3M diffractometer with CoK_α radiation.

RESEARCH RESULTS

The microhardness of the 56DGNKh alloy samples after quenching is relatively low, measuring HV 0.5 = 100 – 130 kgf/mm². The microstructure at this stage does not contain any second-phase inclusions (Fig. 2, *a*).

During aging, the change in microhardness follows a multi-stage pattern: an initial increase at short aging times is followed by a subsequent decrease with increasing aging time, with a clearly defined maximum or a plateau between these two stages of the graph.

Fig. 3 presents data on the microhardness values obtained after different heat treatment modes.

Analysis of these dependencies reveals the following.

The maximum microhardness of 56DGNKh alloy achieved through aging is HV 0.5 = 456 kgf/mm², which is 3.5 – 4.5 times higher than the initial value.

When quenching temperatures are in the range of 700 – 750 °C, the microhardness attained after aging is at its highest. Increasing the quenching temperature to 800 °C leads to a reduction in maximum microhardness (Fig. 3, *a*). Therefore, the optimal quenching temperature should be considered 750 °C.

The maximum microhardness is observed at aging temperatures of 475 – 500 °C for aging durations of 7 to 12 h. This temperature and time range should be used for the aging process.

With varying aging times, the pattern of microhardness changes remains the same for all aging temperatures, but the degree of hardening achieved differs. The common characteristic of these dependencies is that microhardness increases with aging time up to a certain point, after

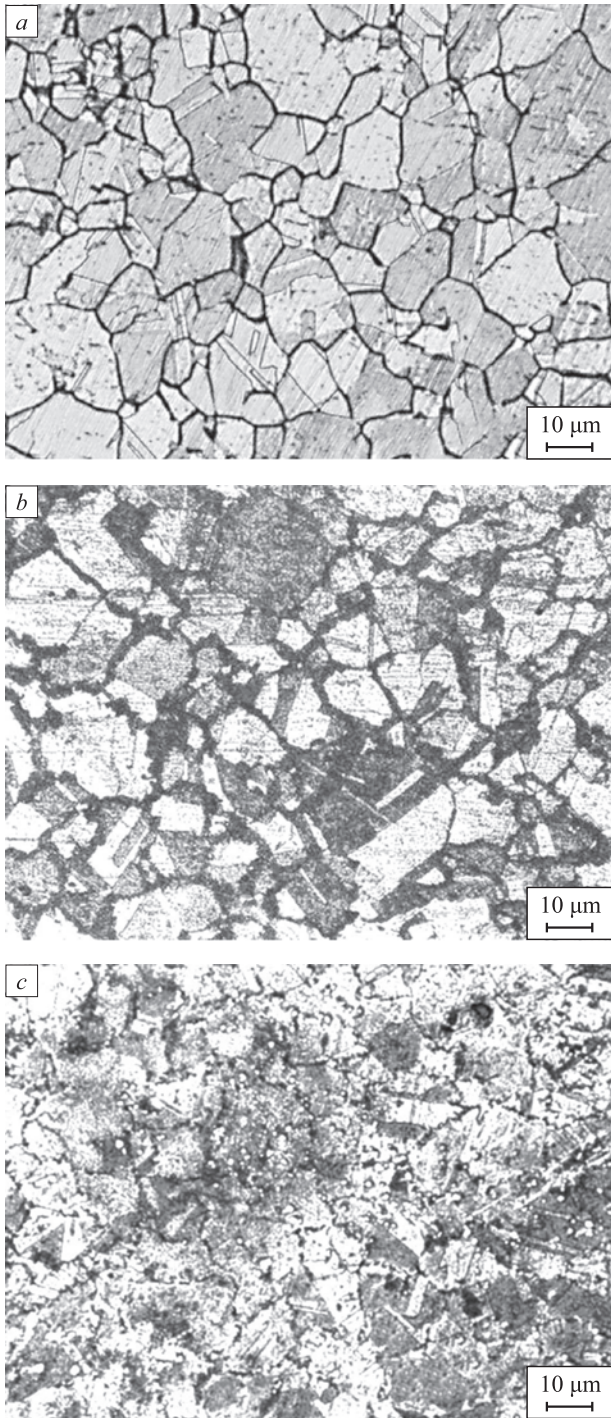


Fig. 2. Microstructure of 56DGNKh alloy after quenching (a), aging by intermittent decomposition at 525 °C for 2 h (b) and aging by continuous decomposition at 475 °C for 10 h (c)

Рис. 2. Микроструктура сплава 56ДГНХ в состоянии после закалки (a), старения по механизму прерывистого распада при 525 °C в течение 2 ч (b) и старения по механизму непрерывного распада при 475 °C в течение 10 ч (c)

which it begins to decrease. The rate of decrease is higher at higher aging temperatures. The aging time after which microhardness starts to decline shortens as the aging temperature increases (Fig. 3, b). The optimal aging mode for achieving the maximum increase in microhardness in

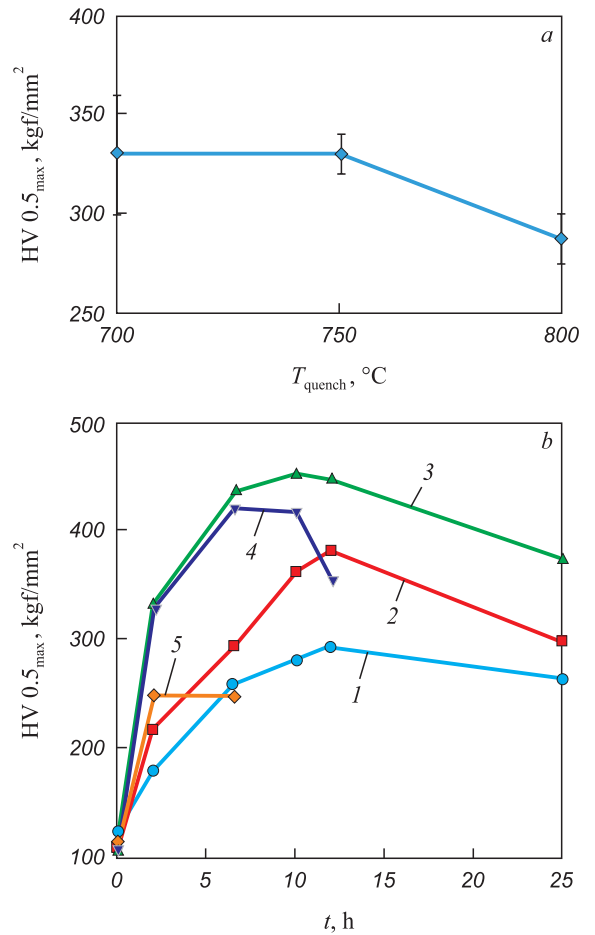


Fig. 3. Dependence of microhardness of 56DGNKh alloy on the quenching temperature (a) and aging duration at temperatures °C:

1 – 475; 2 – 425; 3 – 475; 4 – 500; 5 – 525, quenching from 750 °C (b)

Рис. 3. Зависимость микротвердости сплава 56ДГНХ от температуры закалки (a) и продолжительности старения при температуре старения, °C:

1 – 475; 2 – 425; 3 – 475; 4 – 500; 5 – 525, закалка от 750 °C (b)

56DGNKh alloy is heating to 475 °C with a 10 h holding time. Under these conditions, the microhardness increases to HV 0.5 = 450 kgf/mm².

Phase and structural analysis of 56DGNKh alloy in different states revealed that after quenching, X-ray diffraction (XRD) patterns show only lines corresponding to a solid solution based on copper with an FCC lattice. No effects of isomorphous regions in the matrix with a similar lattice parameter are observed in diffraction lines, even for reflections with large indices (e.g., (220)) (Fig. 4, a), confirming the homogeneity of the quenched solid solution. For this solid solution state, a slight shift in diffraction lines from the tabulated values characteristic of pure copper is observed, which is caused by lattice distortions due to the high concentration of nickel and manganese in the solid solution.

The essence of the aging process is the formation of MnNi compound particles, which leads to a decrease

in the concentration of alloying elements (Mn, Ni, Cr) in the solid solution. The early stages of this process are not detected by X-ray diffraction analysis because the amount of the precipitated hardening phase is still low, and the lattice parameters of the copper-based solid solution and the MnNi compound are very similar. However, this process can be observed metallographically: if the phase transformation follows a discontinuous decomposition mechanism, dark precipitate bands of the MnNi second phase begin to form along the grain boundaries of the α -solid solution present in the alloy after quenching (Fig. 2, *b*). When a sufficient amount of the MnNi phase has precipitated, its formation is indicated by changes in the shape of diffraction lines [18] on X-ray diffraction patterns (Fig. 4, *b, c*).

The absence of this effect in the first diffraction lines with low indices is explained by the close lattice parameters of the tetragonal θ -phase and copper. The observed shift of the primary (220) line from $2\theta = 87.5^\circ$ (Fig. 4, *a*) toward higher angles (almost $2\theta = 90^\circ$, Fig. 4, *b*) indicates that the lattice parameter of the solid solution is approaching the equilibrium lattice parameter values characteristic of pure copper (tabulated $2\theta = 88^\circ 54'$).

According to the general theory of supersaturated solid solution decomposition, the two-stage nature of hardness changes during aging can be explained by the structural and crystallographic characteristics of the alloy [10; 19; 20]:

- during the initial hardness increase, the amount of precipitated MnNi phase is small, but the particles remain coherent with the matrix, leading to a gradual hardness increase as their volume fraction grows;
- as the number of precipitated particles increases, they begin to lose coherence with the matrix, but their increasing volume fraction results in maximum hardening;
- at later aging stages, as the particle size continues to grow and coherence with the matrix is lost, their hardening effect diminishes due to an increased interparticle distance, despite their continuing increase in volume fraction.

With increasing aging temperature, the described sequence of transformations exhibits a wave-like pattern. At higher aging temperatures, MnNi intermetallic particles precipitate rapidly and quickly lose coherence with the matrix. As a result, the maximum microhardness is reached sooner, but the overall hardening effect is lower. At lower aging temperatures, the precipitation process occurs more slowly, and the number of MnNi particles increases at a slower rate. The loss of coherence also progresses gradually, shifting the hardness peak to longer aging times while allowing for a higher overall strengthening effect. Based on this analysis, the optimal aging conditions are those that provide the longest possible coexistence of coherent copper-based α -solid solu-

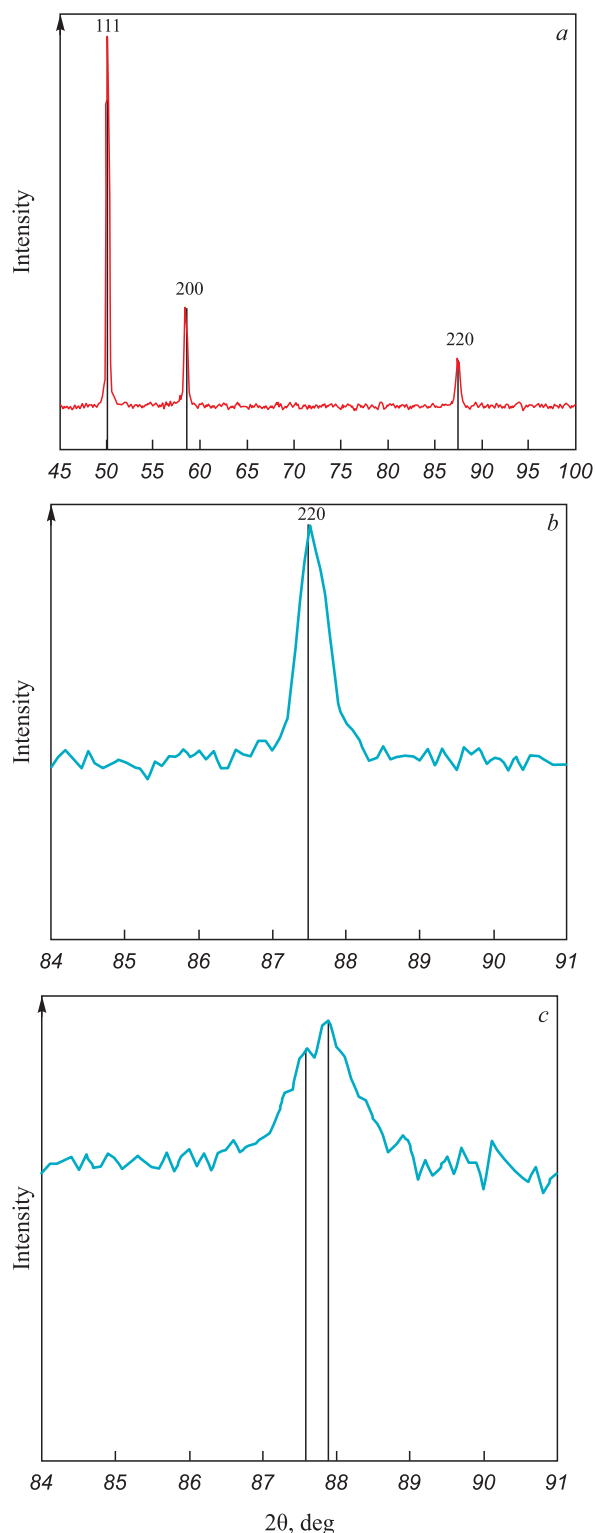


Fig. 4. X-ray diffraction pattern of 56DGNKh alloy after quenching from 800 °C for 30 min (*a*); the third diffraction maximum of this pattern, corresponding to the $\langle 220 \rangle$ line of the copper lattice (*b*) and the same line for the alloy after quenching and aging at 475 °C for 25 h (*c*)

Рис. 4. Рентгенограмма сплава 56ДГНХ после закалки с 800 °C в течение 30 мин (*a*); третий дифракционный максимум рентгенограммы сплава 56ДГНХ после закалки от 800 °C с выдержкой в течение 30 мин, соответствующий линии (220) решетки меди (*b*); та же линия для сплава после закалки и старения при 475 °C в течение 25 ч (*c*)

tion regions and MnNi hardening phase particles. This is achieved at aging temperatures of 475 – 500 °C.

CONCLUSIONS

In the quenched state, the microhardness of 56DGNKh alloy ranges from 100 to 130 kgf/mm², which is characteristic of a homogeneous copper-based solid solution.

As the aging temperature increases from 375 to 475 °C, the microhardness exhibits a monotonic increase from HV 0.5 = 156 – 190 kgf/mm² to HV 0.5 = 440 – 456 kgf/mm², with higher temperatures leading to a faster attainment of peak hardness; however, the ultimate hardness value depends on the specific aging temperature.

At 500 – 525 °C, a sharp drop in maximum attainable microhardness is observed, reducing it to HV 0.5 = 250 – 290 kgf/mm², indicating overaging of the alloy. The microhardness dependence on aging time follows a curve with a distinct peak across all quenching and aging temperatures.

The optimal two-stage heat treatment for this alloy consists of quenching, which involves heating to 750 °C, holding for 30 min, and cooling at a rate of no less than 300 °C/min, followed by aging by reheating to 475 °C, holding for 10 h, and subsequent cooling in air. After this treatment, the microhardness reaches HV 0.5 = 450 kgf/mm².

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D. A. Kozlov – providing the radiographic part of the work (recording, interpretation, analysis of spectra).

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