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EFFECT OF HEAT TREATMENT ON STRUCTURE OF AUSTENITIC STEEL 07Cr25Ni13 OBTAINED BY WAAM

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Abstract. Currently, the use of additive technologies in industry is becoming more promising. The intensification of development of 3D technologies leads to the need for a more thorough study of the structure and properties of metals obtained by this method. In this paper, the effect of heat treatment on structure of the metal deposited by Wire Arc Additive Manufacturing (WAAM) is considered. The paper describes the effect of quenching at various temperatures and annealing on the structure of austenitic steel 07Cr25Ni13. As a result of the work, it was found that during metal deposition, crystallization occurs according to the FA type with the formation of a coarse dendritic structure with mainly skeletal and vermicular morphology, consisting of δ - and σ -phases. It is noted that quenching at 1070 °C practically does not change the metal structure. Despite the fact that quenching at elevated temperatures (1100 °C) leads to partial dissolution and spheroidization of the dendrites released during surfacing, there are no cardinal structural changes. The most complete dissolution of the dendritic component occurs during quenching at 1150 °C. The structure after this procedure is predominantly austenitic, remains of the dendritic component are represented by small spherical inclusions. The steel structure after annealing (1150 °C) practically does not differ from the structure obtained after quenching at the same temperature. A significant increase in grain size, typical for austenitic steels, is not observed in this case. Based on the structure obtained after heat treatment, the most promising treatment options for future physico-mechanical properties are quenching and annealing at 1150 °C.

Keywords: austenitic steel 07Cr25Ni13, additive technologies, WAAM, steel structure formation, δ -ferrite, quenching, annealing

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ВЛИЯНИЕ ТЕРМООБРАБОТКИ НА СТРУКТУРУ АУСТЕНИТНОЙ СТАЛИ 07Х25Н13, ПОЛУЧЕННОЙ МЕТОДОМ АДДИТИВНОГО ВЫРАЩИВАНИЯ WAAM

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Аннотация. В настоящее время все более перспективным является применение аддитивных технологий в промышленности. Интенсификация развития 3D-технологий приводит к необходимости более тщательного изучения структуры и свойств металлов, получаемых данным методом. В работе рассматривается влияние термообработки (ТО) на структуру наплавляемого методом электродуговой наплавки (WAAM) металла. Изучено влияние закалки при различных температурах и отжига на структуру аустенитной стали 07Х25Н13. Установлено, что при наплавке металла происходит кристаллизация по типу ФА с образованием грубой дендритной структуры со скелетной и вермикулярной морфологией и состоящей из δ - и σ -фаз. Закалка при температуре 1070 °C практически не изменяет структуру металла. При повышенных температурах (1100 °C) закалка приводит к частичному растворению и сфероидизации выделившихся при наплавке дендритов, однако кардинальных структурных изменений не происходит. Наиболее полное растворение дендритной составляющей происходит во время закалки при температуре 1150 °C. Структура после данной ТО преимущественно аустенитная, остатки дендритной составляющей представлены мелкими сферическими включениями. Структура стали после отжига (1150 °C) практически не отличается от получаемой после закалки при той же температуре. Значительного увеличения размера зерен, характерного для аустенитных сталей, в данном случае не наблюдается. Исходя из структуры, получаемой после ТО,

наиболее перспективными для будущих физико-механических свойств вариантами обработки являются закалка и отжиг при температуре 1150 °С.

Ключевые слова: аустенитная сталь 07X25H13, аддитивные технологии, WAAM, структурообразование стали, δ-феррит, закалка, отжиг

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INTRODUCTION

Currently, austenitic steels are widely used in the chemical, oil, and food industries, as well as in the production of medical and nuclear power plant equipment [1]. A number of industries in the modern world can hardly exist without austenitic corrosion-resistant steels. This type of material possesses unique properties, such as high corrosion resistance in acid and alkaline environments of varying corrosive strength, as well as paramagnetism. The combination of physical and mechanical properties is achieved not only through alloying with significant amounts of chromium, nickel, magnesium, and other elements but also through maintaining a homogeneous austenitic structure throughout the product's entire service life [2].

Currently, additive technologies are becoming increasingly promising for industry as they reduce the total cost of products, especially in single and small batch production. The rapid development of 3D printing technologies necessitates a thorough investigation of the mechanical properties, structure, and chemical composition of metals produced by these methods. To date, the main methods of metal 3D printing are layer-by-layer powder fusion (selective laser sintering, SLM), laser powder deposition (laser engineered net shaping, LENS/direct metal deposition, DMD), and electric arc deposition (wire arc additive manufacturing, WAAM) [3]. WAAM 3D printing, which we used for this study, is the most productive and technologically simple [4; 5].

The advantages of additive manufacturing methods are as follows:

- the process of obtaining products can be fully automatized;
- when products are manufactured of expensive materials, such as titanium and nickel alloys, the material consumption is considerably reduced;
- small batch production, unprofitable when using traditional production methods, becomes cost-efficient [6 – 8].

SLM technology enables the manufacture of complex products by laser melting of metal powder using CAD

models. At the powder melting point, the energy density is higher compared to other electric arc processes (e.g., welding), but lower than in the case of laser exposure [9]. The main problem of parts produced by the SLM method is relatively high surface roughness, which reduces fatigue resistance by increasing stress concentration on the sample surface [10].

Laser powder deposition is the process of overlay welding performed by fusing the powdered material layer onto the substrate. The laser beam creates a molten bath into which the powdered metal is introduced. The metal melts and solidifies in the bath to form metallic bonds with the substrate. During the surfacing process, the powdered metal is automatically fed from the feed system to the substrate, which is lowered to the height equal to the thickness of the layer to be deposited. However, it should be noted that the laser deposition method does not ensure reproducibility of the chemical composition and mechanical properties of the final products [11; 12], which is a significant disadvantage of this technology.

WAAM technology is a relatively new additive growth method that emerged in the 1990s. It includes depositing a common welding wire, widely available commercially, onto the substrate, resulting in a finished part. Compared to conventional manufacturing, WAAM can reduce manufacturing time by 40 – 60 % and post-processing time by 15 – 20 %, depending on the size of the part. For example, the use of this technology for manufacturing airplane landing gear stiffeners results in about 78 % raw material savings compared to conventional production [13]. Metals with good weldability can potentially be used for the WAAM process, and so far, researchers have successfully applied this method to fabricate parts from Ti [13], Al [14], steel [15], and Ni [16] based alloys.

In the additive growth process, the surfaced metal is in a liquid state and is subsequently subjected to multiple cycles of high-temperature heating, including to temperatures above critical. As a result, the microstructure of the surfaced metal differs from that of metal produced by conventional technologies, and consequently, the physicochemical and strength properties of the metal may also differ from those of rolled material.

Table 1. Chemical composition of the studied material

Таблица 1. Химический состав исследованного материала

Material	Content of chemical elements, %								
	C	Si	Mn	Cr	Ni	Mo	Ti	S	P
GOST 2246	<0.09	0.5 – 1.0	1.0 – 2.0	23.0 – 26.0	12.0 – 14.0	not regulated	not regulated	<0.018	<0.025
ER309LSI	0.016	0.7	1.9	23.3	13.7	0.1	0	0.004	0.019

An increasing number of foreign studies are investigating the application of additive technologies in industry. However, in Russia, these methods are developing locally and are not yet widespread. In addition to reducing the cost of small-batch products from austenitic steels, which are currently quite popular, additive technologies can also contribute to the advancement of Russian science.

The objective of this study is to explore the effect of heat treatment modes on the structure of austenitic steel 07Cr25Ni13 obtained by additive growth using the WAAM method.

MATERIALS AND METHODS

The research was conducted using metastable austenitic steel 07Cr25Ni13 obtained by the WAAM method. The samples for investigating the deposited metal were produced on a specialized bench for additive arc surfacing [17]. Surfacing was performed with the following parameters: $I = 120$ A; $U = 24$ V; $v = 350$ mm/min. A shielding gas mixture of 98 % Ar and 2 % CO₂ was used. The samples for metallographic studies were cut from the obtained blanks using waterjet cutting followed by milling. ER309LSI welding wire was used as the initial material for surfacing. The chemical composition of the wire is presented Table 1.

The metal surfacing process can result in the loss of alloying elements. The chemical composition of the surfaced metal was determined using a Foundry-Master optical emission analyzer.

The structural affiliation of the deposited material to the austenitic class was derived from the Scheff-

ler diagram. According to the literature data [15 – 20], the phase composition obtained after surfacing depends on the Cr_{eq} and Ni_{eq} ratio and can be specified from the Scheffler diagram.

We know from the literature sources [18 – 23] that phase transformations during crystallization and the final phase composition depend on the Cr_{eq}/Ni_{eq} ratio and are divided into the following types:

- A (<1.25): $L - (L + \gamma) - \gamma$;
- AF (1.25 ≤ 1.48): $L - (L + \gamma) - (L + \gamma + \delta) - (\gamma + \delta)$;
- FA (1.48 ≤ 1.95): $L - (L + \delta) - (L + \delta + \gamma) - (\delta + \gamma)$;
- F (>1.95): $L - (L + \delta) - (\delta + \gamma)$.

These equivalents were determined using the following formulas

$$\text{Cr}_{\text{eq}} = \text{Cr} + \text{Mo} + 1.5\text{Si} + 0.5\text{Nb} + 2\text{Ti}; \quad (1)$$

$$\text{Ni}_{\text{eq}} = \text{Ni} + 30\text{C} + 0.5\text{Mn}. \quad (2)$$

Metallographic studies were performed in cross section relative to the surfacing direction at magnifications of 100 and 200, and the milled surface of the samples was also examined. The metallographic sections were prepared following the standard procedure – the samples were sanded mechanically using the sandpaper of different grits and polished with pastes. The solution consisting of 5 cm³ HNO₃, 50 cm³ HCl and 50 cm³ H₂O was used as a chemical etching reagent.

To investigate the impact of heat treatment on the structure of the samples, we performed quenching in three modes followed by metal annealing. The heat treatment parameters are presented in Table 2.

Table 2. Modes of the samples heat treatment

Таблица 2. Режимы термообработки образцов

Mode number	Heating temperature, °C	Holding time, min	Cooling medium
1	1070	30	Air
2	1100	60	Air
3	1150	60	Air
4	1150	60	Furnace

RESULTS AND DISCUSSION

The study of the chemical composition of the surfaced metal revealed the decrease in the content of all alloying elements (AE), except silicon. The diminishing AE concentration is typical for metal welding and smelting processes due to their losses. The increased wire content in the surfaced metal compared to the original wire can be attributed to the heterogeneity of its chemical composition along its length. It should be noted that as the steel

Table 3. Chemical composition of the surfaced metal

Таблица 3. Химический состав наплавленного металла

Material	Content of chemical elements, %								
	C	Si	Mn	Cr	Ni	Mo	Ti	S	P
ER309LSI	0.016	0.700	1.90	23.3	13.7	0.100	0	0.004	0.0190
Surfaced metal	0.018	0.820	1.75	23.2	13.4	0.036	0	0.005	0.0114

chemical composition changed after surfacing, the AE content variations did not exceed the permissible limit (in accordance with GOST 2246 – 70). The final composition of the surfaced metal is presented in Table 3.

Chromium and nickel equivalents are calculated by the formulas (1), (2): $Cr_{eq} = 23.2578$; $Ni_{eq} = 14.815$. The Cr_{eq}/Ni_{eq} ratio is 1.57, hence, in this case the transformations during crystallization can be described by the FA mode (the Cr_{eq}/Ni_{eq} ratio exceeds 1.48). The approximate ferrite content in the surfaced metal can be determined from the Scheffler diagram (Fig. 1).

Based on the above diagram, the approximate content of δ -ferrite in the metal after surfacing is about 7.5 %, which is consistent with the theoretical data [24].

We studied the cross direction relative to the metal surfacing axis based on the microstructural analysis of the samples before and after the heat treatment. Fig. 2 shows the structure of the surfaced metal prior to heat treatment.

The dendrites are oriented normal to the surface of the laser track due to the direction of heat removal. The dendrites located deep within the surfaced metal have a more developed boundary structure. The dendritic structure refinement on the laser track surface can be put down to the supply of additional thermal energy as the next metal layer is deposited. In general, the struc-

ture of the surfaced metal is similar to the microstructure resulting from the crystallization of austenitic steel.

It was noted in [25] that the dendrites formed during surfacing may include δ -ferrite and σ -phase. Fig. 3, *b* shows that δ -ferrite has mainly skeletal and vermicular morphology. Surfacing of AISI 316L [25] and AISI 316 [26] steel resulted in a similar structure. The interdendritic space is filled with γ -phase (austenite).

Austenitization according to mode I (1070 °C, 30 min) did not result in visible changes in the grain structure, which is indicative of insufficient holding time

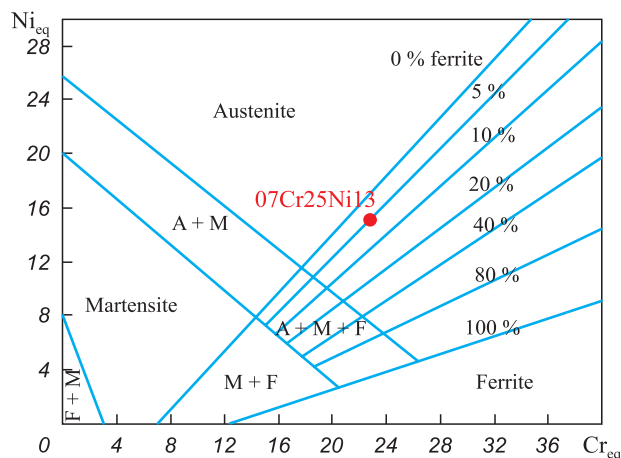


Fig. 1. Location of steel 07Cr25Ni13 on the Scheffler diagram

Рис. 1. Расположение стали 07X25H13 на диаграмме Шеффлера

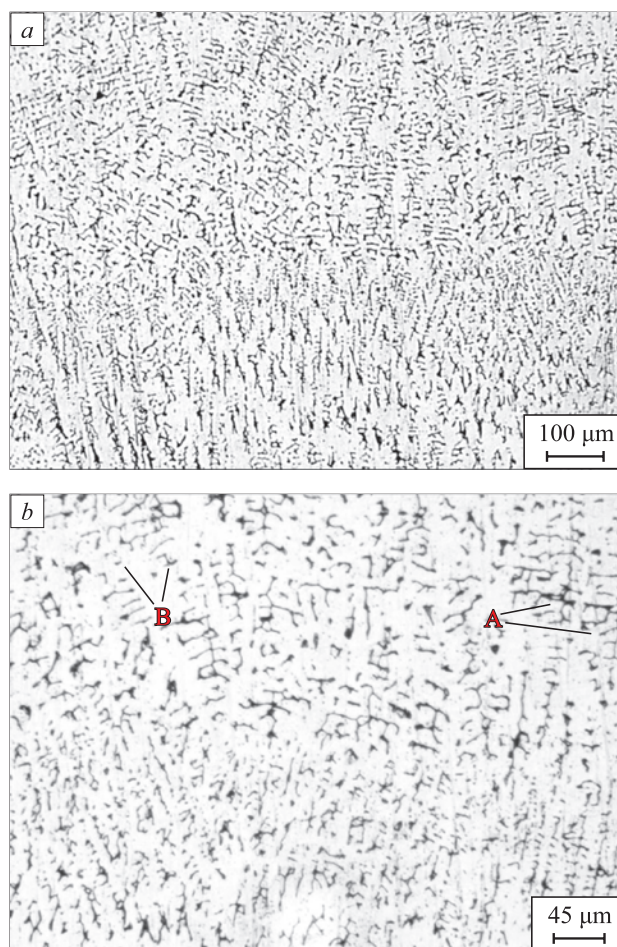


Fig. 2. Structure of the sample after surfacing: $\times 100$ (a); $\times 200$ (b)

Рис. 2. Структура образца после наплавки: $\times 100$ (a); $\times 200$ (b)

or temperature or both during austenitization (Fig. 3). It should be noted that the overall etchability of the samples increased.

The grain boundaries only begin to emerge in the structure after heat treatment according to mode 1, which is also indicative of insufficient holding at the austenitization temperature (Fig. 3). The dendritic structure does not decrease, which indirectly shows that the content of δ - and σ -phases did not reduce. The δ -ferrite morphology does not significantly change.

We performed quenching using mode 2 (1100 °C, 60 min) to study the effect of increased austenitization temperature and holding time. Fig. 4 shows the sample microstructure in the direction transverse to the surfacing axis after heat treatment according to mode 2.

In the micrographs of the sample after austenitization at 1100 °C, the grains formed can be seen more clearly. The dendrite size generally decreases compared to the microstructure after surfacing and treatment according to mode 1. This effect indicates more complete diffusion processes during austenitization. Spheroidization of dendritic components is observed, their general orientation remaining the same. The percentage of δ -ferrite and σ -phase should significantly decrease after this treatment.

After quenching according to mode 3 (1150 °C, 60 min), grain boundaries and austenite twins are clearly visible in the metal structure (Fig. 5). The dendritic structure dissolved almost completely; the dendrites that failed to do so are represented by small spheroidal inclusions.

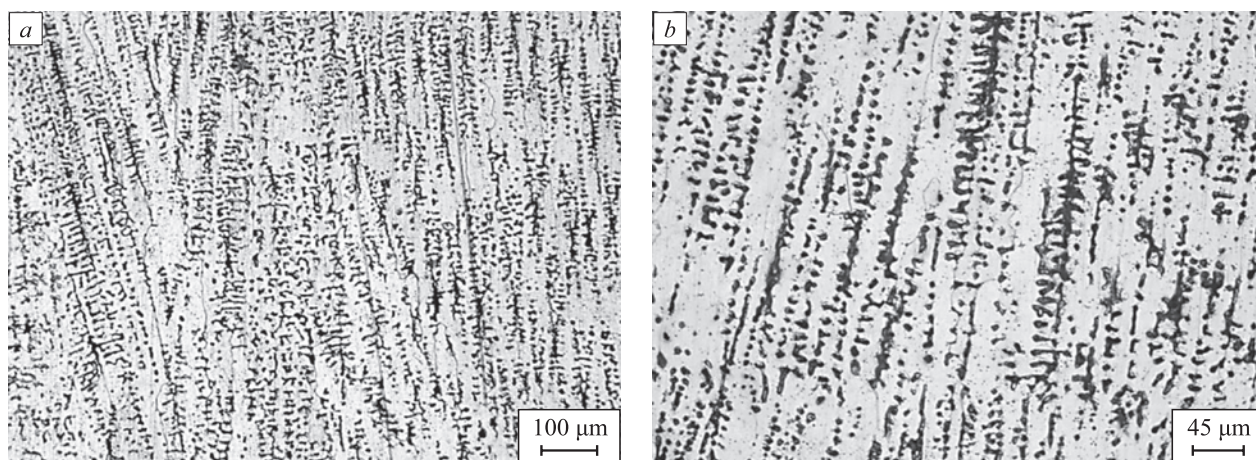


Fig. 3. Structure of the sample after quenching according to mode 1:
×100 (a); ×200 (b)

Рис. 3. Структура образца после закалки по режиму 1:
×100 (a); ×200 (b)

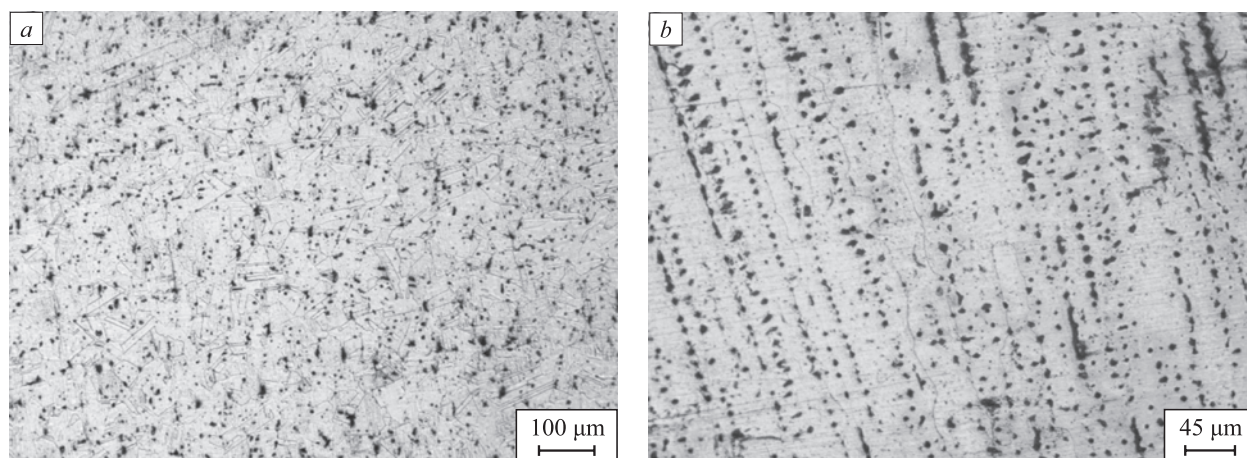


Fig. 4. Structure of the sample after quenching according to mode 2:
×100 (a); ×200 (b)

Рис. 4. Структура образца после закалки по режиму 2:
×100 (a); ×200 (b)

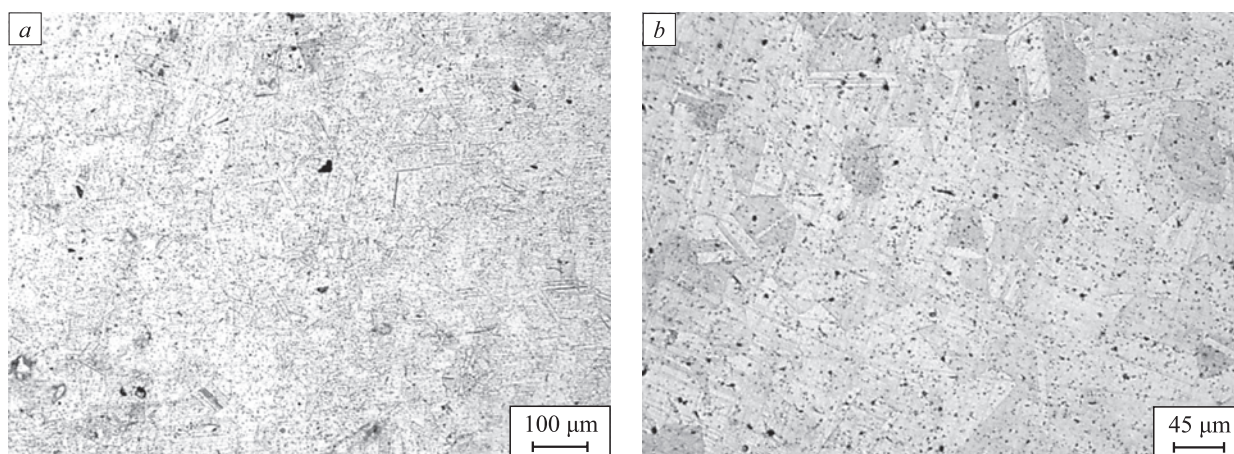


Fig. 5. Structure of the sample after quenching according to mode 3:
×100 (a); ×200 (b)

Рис. 5. Структура образца после закалки по режиму 3:
×100 (a); ×200 (b)

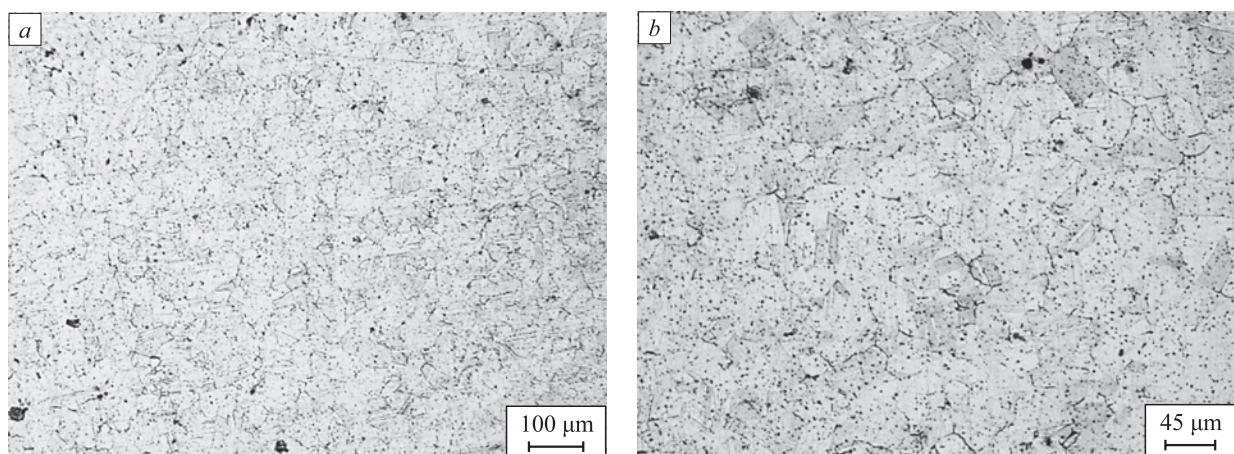


Fig. 6. Structure of the sample after annealing according to mode 4:
×100 (a); ×200 (b)

Рис. 6. Структура образца после отжига по режиму 4:
×100 (a); ×200 (b)

Mode 4 (1150 °C, 60 min) enables more complete dissolution of the remaining dendrites because at such metal temperatures, the diffusion is active for a longer time. The austenitic structure of the metal with characteristic twins is clearly visible (Fig. 6).

It should be noted that after annealing according to mode 4, there is no significant grain increase compared to quenching at the same temperature and holding time.

CONCLUSIONS

It is found that after WAAM surfacing, steel 07Cr25Ni13 forms a rough dendritic structure, which may consist of δ -ferrite and σ -phase. The post-deposition structure features δ -ferrite of skeletal and vermicular morphology, with the interdendritic space filled with γ -phase.

The phase composition of the deposited material is consistent with that determined from the Scheffler diagram.

Austenitization at 1070 °C with a 30 min holding time practically does not change the structure of the deposited metal. After aging the samples at 1100 °C for 60 min, we can clearly see the formation of austenitic grains and a reduction in dendrite size. Thus, structural-phase transformations in steel 07Cr25Ni13 require heating to temperatures above 1100 °C during heat treatment.

Quenching according to mode 3 (1150 °C, 60 min) results in almost complete dissolution of dendrites. The remaining undissolved dendrites appear as small spheroidized particles.

The metal structure after heat treatment according to mode 4 (1150 °C, 60 min, furnace cooling) is prac-

tically the same as that of the metal quenched from the same temperature. A significant increase in grain size, typical for austenitic steels, is not observed in this case.

In terms of potential physical and mechanical properties, heat treatment modes 3 and 4 proved to be the most favorable.

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