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STUDY OF THE EFFECT OF ISOTHERMAL HOLDING ON PARAMETERS OF GRAPHITE PHASE IN INDEFINITE CHROMIUM-NICKEL CAST IRON ALLOYED BY NITROGEN AND VANADIUM

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ABSTRACT

The paper presents the effect of isothermal annealing at the temperatures higher than the temperature of carbide phase graphitization in indefinite cast iron on quantitative and qualitative parameters of graphite inclusions, depending on vanadium content and nitrogen presence in the alloy. This material is used for manufacture of the working layer of a dual-layered rolling roll. Absolute and relative variations of amount and average size of graphite inclusions are determined depending on the degree of alloy alloying in as-cast and heat treated states. Regularities of variation of graphite phase morphology depending on the degree of alloy alloying and presence of high-temperature heat treatment are examined.

Introduction

The technology of manufacture of dual-layered rolling rolls, where the working layer is produced from alloyed molted iron and the core is produced from grey iron, is widely distributed worldwide. It allows to combine in this product wear-resistant and hard working surface with more cheap and “soft” base that is more suitable for final mechanical working (such as forming of wobbler and necks). Manufacturing technology of such rolls includes centrifugal casting of the working layer and static pouring of upper and lower necks as well as roll core. In this case grey iron is poured in crystallized working layer, what leads to heating and partial melting of this working layer. Therefore, the features of temperature variation after pouring become non-monotonous. Pouring of large amount of core metal, its crystallization and cooling lead to isothermal holding of iron working layer at higher temperature compared with initial temperature of carbide phase graphitization. Thereby cast iron structure of the working layer consists of martensite-austenite metallic base, eutectic and secondary carbides as well as graphite (its presence is mandatory for indefinite iron) [1–6]. In addition to iron rolls, steel rolls also can be applied for hot rolling [7–9], and they displays good results, especially in heavy plate mills.

Graphite has a positive effect on wear resistance of rolls due to following advantages: locations of spheroidal graphite are reservoirs for lubrication; solid abrasive particles are absorbed by large graphite inclusions; iron heat conductivity increases; kinetics of oxide film forming on roll surface improves; thermal wear resistance enlarges. It should be mentioned in this connection that excessive graphite amount loosens metallic base and decreases wear resistance, while absorption of abrasive particles by graphite inclusions can be observed only in the case of correspondence of sizes. Appearance of thermal fatigue cracks and graphite extraction promote oxidation of iron structural components, and in the conditions of friction forces influence in the deformation area lead also to detachment of cementite particles. However, this process does not distribute inside metal owing to graphite. It should be noted that both quantitative and qualitative parameters of graphite inclusions have the effect on working layer resistance. Cast iron with uniformly distributed graphite is characterized by thermal fatigue limit by 40% higher than cast iron with graphite conglomeration in the form of nests and sockets, while fine-comminuted inclusions provide minimal susceptibility to origination of erosion cracks [10–15].

Correlation of the amounts of structural components determines the level of operating properties and reliability of a rolling roll. Heat treatment — tempering for release of stresses, more complete decomposition of overcooled austenite and additional extraction of secondary phases — is a mandatory technological stage in manufacture of rolling rolls. However, heat treatment in the form of low- and medium-temperature tempering has no essential effect on amount and morphology of graphite inclusions [16–21].

The main method for variation of the amount and form of graphite inclusions is alloying. Vanadium (i.e. nitrided one) can be considered as one of the prospective alloying compositions for the examined iron. Vanadium itself is a carbide-forming element, thereby its introduction in the chemical composition of molted iron will have the effect on quantitative and qualitative parameters of both carbide phase and graphite [6].

Examination of the effect of alloying by nitrided vanadium and ferrovanadium without nitrogen on the amount and average size of graphite inclusions in chromium-nickel molted iron of the working layer of dual-layered rolling rolls is the aim of this work.

Table 1. **Chemical composition of the "basic" sample**

Element	C	Si	Mn	S	P	Cr	Ni	Mo	B	Nb	V	Al
Mass part, %	3,05	0.93	0.87	0.021	0.049	1,80	4,46	0,34	0.032	0.19	0.03	0.03

Technique of experimental investigation

Four series of samples in as-cast and annealed states have been obtained. The first batch was alloyed by vanadium, its content in the samples was varied from 0.13 to 0.50%. The second series of experimental samples was alloyed by nitrided ferrovanadium of FV35N9 (ФВ35Н9) grade.

Content of nitrided vanadium in the samples was varied from 0.13 to 0.48%. To provide correct building of relationships, the "basic" alloy with vanadium content less than 0.05% has been melted (**Table 1**).

Experimental samples have been melted in the induction furnace with basic lining and operating volume 2 kg. Standard samples with dimensions 35×35×10 mm have been used for researches.

Heat treatment of the samples imitated isothermal holding in production conditions: both batches of experimental samples were subjected to annealing at the temperature 1000 °C during 20 hours and consequent slow cooling together with a furnace after holding termination.

Optical metallography has been conducted through optical microscope Axio Observer with magnification from 50 to 1000 times. Quantitative analysis has been done using the program Ticsomet Standard Pro in accordance with GOST 5639-82. Polished sections for microanalysis have been prepared according to the standard technique via pressing of samples in "Transoptic" resin at the automatic press Simplimet 1000 in the sample preparation line of Buechler company.

To examine microstructure, surface of polished sections has been subjected to pickling in the 4% solution of nitric acid in ethylic alcohol via dipping of polished surface in the bath with reagents.

Scanning electron-microscopic analysis has been conducted using scanning electronic microscope JEOL JSM-6490 LV with accelerating voltage 20 kV. Investigations have been executed on transversal polished sections used for light microscopy, in the conditions of secondary electrons with magnification from 30 to 50,000 times. X-ray microanalysis has been conducted using special additional block to the scanning microscope of INCA Energy. These investigations have been done by the Center of Collective Usage of the Scientific and Research Institute of Nanosteels at the Nosov Magnitogorsk State Technical University.

Obtained results

Structure of the sample of a basic alloy contains appr. 1.0% of graphite after crystallization and monotonous cooling. Graphite amount increases slightly up to 1.2% after additional alloying of the examined alloy by vanadium (0.05%). Consequent rise of vanadium content in the alloy leads to gradual lowering of graphite amount to 0.2% (for the alloy with vanadium content about 0.4%). Introduction of 0.5% vanadium in the alloy does not finalize in lowering of graphite amount in alloy structure, it remains at the level 0.2% (**Fig. 1**).

In the case of alloying the basic alloy by nitrided ferrovanadium, the features of variation of graphite amount looks similar to alloying only by vanadium. They differ only in vanadium concentration (from 0.1 to 0.2%), when maximal graphite amount in the structure is observed up to 1.3–1.6%. Consequent rise of the amount of introducing alloying composition leads to monotonous lowering of graphite amount. So, if vanadium content in the examined alloy is equal to 0.3%, then graphite amount corresponds to its amount in the basic alloy (1.0%). Introduction of nitrogen and about 0.5% of vanadium in the alloy leads to graphite forming in this structure in the amount 0.2% and its corresponds to the amount obtained in alloying only by nitrogen.

Variation of qualitative parameters of graphite inclusions was observed together with variation of their quantitative parameters. Relationship between average size of graphite inclusions and vanadium amount in the alloy has extremal feature (**Fig. 2**). If vanadium is introduced in the alloy (both separately and together with nitrogen) in the amount up to 0.3%, the average size of graphite inclusions increases from 8/3 to 10.0 μm. Consequent enlargement of vanadium concentration in the alloy leads to harsh decrease of average size of graphite inclusions to 5 μm (for vanadium content in the alloy ~ 0.5%).

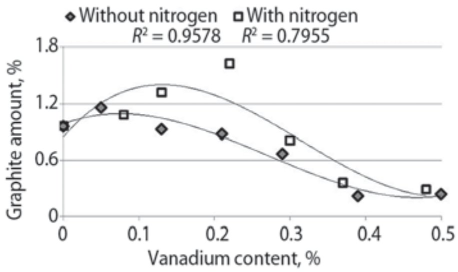


Fig. 1. Relationship between graphite amount in as-cast alloy and vanadium content, nitrogen presence

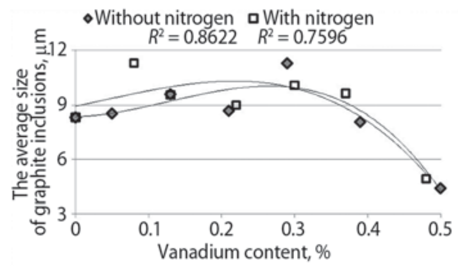
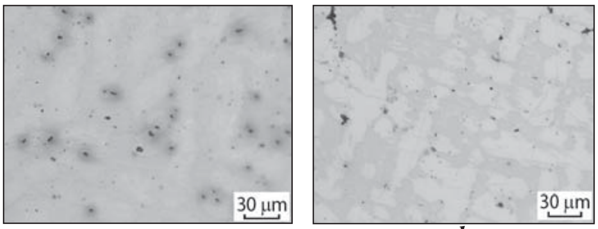


Fig. 2. Relationship between average size of graphite inclusions in as-cast alloy and vanadium content, nitrogen presence



a *b*

Fig. 3. Microstructure of examined iron in as-cast state, alloyed by vanadium without nitrogen in the amount 0.13% (*a*) and 0.5% (*b*), without pickling

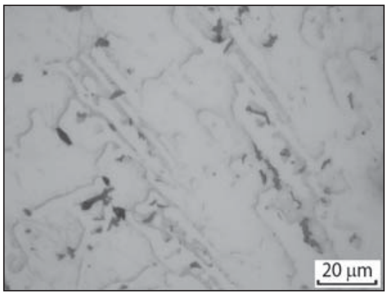


Fig. 4. Microstructure of the "basic" alloy after annealing, ×1000, without pickling

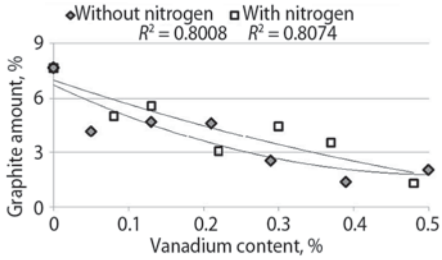


Fig. 5. Relationship between graphite amount in annealed alloy and vanadium content, nitrogen presence

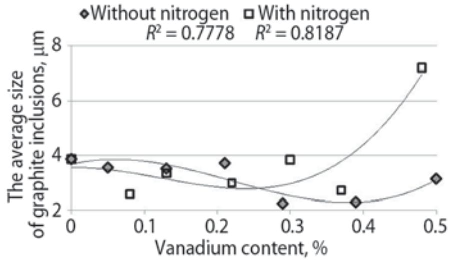


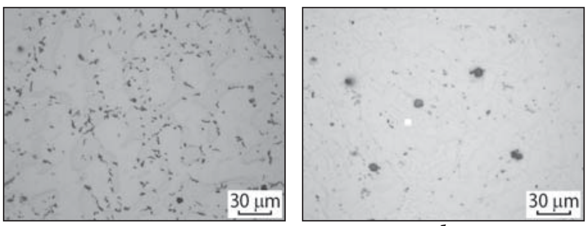
Fig. 6. Relationship between average size of graphite inclusions in annealed alloy and vanadium content, nitrogen presence

Introduction of nitrided vanadium and vanadium without nitrogen in the examined alloy changes morphology of graphite inclusions. Form of graphite inclusions is mainly round – as in the basic alloy, as well as in the alloy with vanadium addition up to 0.3%. If vanadium content is larger, graphite inclusions become more stretched (**Fig. 3**).

Isothermal annealing at high temperature leads to partial graphitization of carbide phase, mainly along its boundaries. The areas where formed carbide inclusion was located perpendicular to carbide boundary, was observed rarely (**Fig. 4**).

Graphite amount in the "basic" alloy increases from 1.0 to 7,7%. Introduction of ferrovanadium in the melt (i.e. nitrided ferrovanadium) leads to substantial lowering of the amount of graphite inclusions. Thus, if vanadium content in the alloy makes 0.05%, graphite amount in the structure decreases to 4.2–5.0%. Consequent elevation of the alloying element concentration leads to monotonous lowering, and if we introduce 0.5% vanadium in the alloy, graphite amount will decrease to 1.2–2.0% (**Fig. 5**).

Average size of graphite inclusions decreased after annealing from 8.3–10.0 µm to 2.0–4.0 µm for the basic alloy and the alloys alloyed additionally by vanadium in the amount up to 0.4%. It was not finalized in observation of essential difference in the average size of graphite inclusions both for alloys alloyed by usual alloying composition or by nitrided alloying composition. Alloying of the basic alloy only by vanadium in the amount 0.4–0.5% does not lead to variation of the average size of graphite inclusions, which remains on the level 2.0–4.0 µm. However, if we introduce nitrided ferrovanadium in the melt to reach vanadium concentration in the alloy 0.5%, harsh enlargement of graphite particles occurs, while their average size increased to 7.0 µm (**Fig. 6**).



a *b*

Fig. 7. Microstructure of investigated cast iron in annealed state, alloyed by vanadium and nitrogen in the amount 0.13 (*a*) and 0.5 (*b*), without pickling, ×500

Shape of graphite inclusions in the structure of investigated alloy becomes stretched in comparison with the samples cooled monotonously after crystallization. In other words, growth of graphite inclusions extracted during annealing is realized in the advantageous direction. This regularity is true for all experimental alloys except one of them. The sample alloyed by nitrided vanadium in the amount 0.48% is characterized by large round graphite inclusions in its structure, together with small stretched inclusions, i.e. distribution has distinctly expressed bimodality (**Fig. 7**).

Discussion of results

Isothermal annealing at the temperature higher than graphitization temperature of a carbide phase leads in all examined samples to enlargement of graphite amount in the structure of molted iron that is used for manufacture of the working layer of rolling rolls. Maximal graphite increase was observed in the basic alloy. Additional alloying of the investigated alloy by vanadium (i.e. nitrided vanadium) leads to decrease of graphite amount after annealing. Introduction of carbide-forming vanadium in the melt facilitates forming of alloyed eutectic carbides that probably have larger resistance to graphitization.

It should be mentioned that the alloys alloyed additionally by nitrogen are characterized by increase of graphite amount in average by 0.5% less than in the alloys alloyed only by vanadium. It can be connected with forming of carbonitrides and decrease of total amount of carbide phase and, respectively, with decrease of number of graphitization areas.

Relative variation of graphite amount in the structure of investigated cast iron has extremal features, different to its absolute variation. Graphite amount in the basic alloy increases by 6 times in average. Additional alloying of the investigated alloy by vanadium leads to lowering of relative enlargement of graphite amount. Minimal rise was observed in the alloys with vanadium content as much as 0.1–0.3% (by 2.5–3.0 times). Increase of concentration of the introducing alloying element more than by 0.3% finalizes in gradual elevation of relative rise of carbide phase to 6-7 times with vanadium content 0.5%. Introduction of nitrogen in the investigated alloy increases relative variation of the graphite amount appr. by 50%.

Elevation of graphite amount in structure of the basic alloy occurs mainly due to extraction of new more fine particles, what facilitates diminishing of their average size by 5 μm. Additional alloying of cast iron by vanadium in the amount up to 0.3% leads to decrease of average size of inclusions down to 7–8 μm. Alloying by vanadium in the amount from 0.3 to 0.5% leads to forming of more coarse particles in the structure; they are smaller than those in not annealed state by 1–6 μm depending on alloying degree. And if we introduce 0.5% of vanadium together with nitrogen in the alloy, the average size of particles becomes larger than before heat treatment. At the same time relative decrease of average size of graphite particles for the basic alloy and alloys alloyed by vanadium in the amount up to 0.4% is equal and makes 50–70%. If the alloy is alloyed by 0.5% of vanadium without nitrogen, relative decrease of average size of graphite particles makes 25%, while the same parameter in the alloy alloyed by corresponding amount of vanadium together with nitrogen relatively increases of by 85%.

Conclusions

Annealing of indefinite cast iron, used for manufacture of the working layer of dual-layered rolling rolls, at the temperature higher than the temperature of the beginning of carbide phase graphitization leads to increase of graphite amount in the structure from 1.0 to 7.7%.

Additional alloying of cast iron by vanadium, i.e. together with nitrogen, leads to monotonous decrease of graphite amount to 1.2%. This graphite is forming after annealing accompanied by introduction of 0.5% of vanadium in the alloy due to alloying of eutectic carbides and rise of their resistance to graphitization.

Increase of graphite amount in the structure of molted iron occurs both due to extraction of the new more fine particles and growth of already formed particles. The form of these particles themselves varies from round to stretched, while the average sixe decreases approximately by 2.0–2.2 times.

The obtained data on alloying of cast iron for rolling rolls allow not only to adjust the amount of extracted graphite, but also to predict its morphology, what shall be considered in the next article.

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