DOI: 10.17580/cisisr.2019.02.02

STUDY OF THE EFFECT OF GRAPHITIZED ANNEALING ON MICROSTRUCTURE OF CHROMIUM-NICKEL CAST IRON

K. N. Vdovin[1], D. A. Gorlenko[1], N. A. Feoktistov[1]

[1] *Nosov Magnitogorsk State Technical University (Magnitogorsk, Russia)*

E-mail: kn.vdovin@gmail.com; gorldima@yandex.ru; fna87@mail.ru

AUTHOR'S INFO

K. N. Vdovin, Dr. Eng., Prof., Dept. of Foundry Production and Material Processing
D. A. Gorlenko, Cand. Eng., Senior Lecturer of Dept. of Foundry Production and Material Processing
N. A. Feoktistov, Cand. Eng., Associate Prof., Head of Dept. of Foundry Production and Material Processing

Key words:

mottled cast iron, graphitized annealing, graphite, carbide phase, residual austenite, microhardness.

ABSTRACT

The technology of manufacture of dual-layered centrifugally cast rolling rolls includes the process of casting and crystallization of work layer metal and, some time later, of core metal. It supports non-monotonous features of cooling of work layer metal that is subjected to the secondary heating and holding at high temperature, or to annealing. At the same time graphite is extracted in cast iron structure of work layer; its amount can be varied both by holding time and by alloying. Free carbon can be extracted by dissolution of carbide phase and by depletion of solid solution, what should effect on quantitative parameters of microstructure of mottled cast iron and its mechanical properties. Revealing of regularity of forming the structure of mottled cast iron after its graphitized annealing depending on holding time is the aim of this study.

Experimental cast samples were subjected to annealing in the protected medium at 900 °C during 30–330 min. Regularities of forming the microstructure were examined using optical metallography. Examination of experimental samples was resulted in obtaining of relationships between graphite amount, carbide phase and residual austenite (from one side) and time of graphitized annealing (from other side). Graphite amount increases up to certain holding time, and then additional extraction in the structure was not observed. On the contrary, carbide phase amount is essentially decreased at the same time up to certain holding time, and then its amount leaves practically the same.

Joint analysis of obtained relationships displays that graphite is partially extracted owing to dissolution of carbide phase, while quantitative relationship itself is characterized by power features based on the fact that graphite is also extracted due to carbon that is dissolved in metal base austenite. Alloying composition of austenite has effect on its resistance to decomposition during overcooling, thereby graphite amount in the structure of mottled chromium-nickel cast iron is considered as the main regulator of quantitative phase relationship in metallic base and of microhardness of samples in general.

Introduction

Mottled cast iron is considered as a material that is widely used in fabrication of a work layer of dual-layered centrifugally cast rolling rolls; it is characterized by carbon presented st the same time as carbide phase (in bound state) and as graphite (in free state) [1–6]. Continuous cooling of cast iron after its casting into mold finalizes in graphite forming in the structure in the small amount. However, non-monotonous features of cooling caused by the technology or rolling roll manufacture (core pouring by grey cast iron) leads to heating and additional high-temperature heat treatment (annealing) of crystallized material of work layer after certain cooling. Main graphite extraction in the structure of mottled cast iron occurs just at this stage [7]. It was displayed previously that quantitative parameters of graphite can be influenced by variation of chemical composition, i.e. by alloying [8–10]. Introduction of several carbide-forming elements in cast iron composition promotes decrease of carbide amount both in cast and annealed states.

Holding time during annealing is another possible way for varying of the amount of extracting graphite in the structure of mottled cast iron [11, 12]. Actuality of this method is confirmed by appearance of temperature and time gradients along radial cross section of the work layer during core pouring of a rolling roll); in other words, graphitization process takes different time for several different areas. From one side, time gradient during graphitized annealing of different layers should not be too essential, taking into account high material heat conductivity; from other side, it can be sufficient for extraction of different amount of graphite in microstructure of mottled cast iron. We also don't have any information about features of the relationship between amount of extracted graphite and annealing time, and if there is any maximal point on this graph or no.

Theoretically extraction occurs owing to dissolution of carbide phase, what leads to decrease of its amount in the structure as well as to variation of certain mechanical properties. Depletion of austenite by carbon is considered as another possible way for conducting the process of graphite forming. Variation of chemical composition of a solid solution will have effect on its resistance to decomposition during consequent overcooling, what will be finalized in variations in quantitative relationship "martensite — residual austenite" of metallic base after complete cooling. As a result, the values of metallic base microhardness will be varied and it will also have effect on operation properties of cast iron used for work layer of dual-layered centrifugally cast rolling rolls [13–17].

Table 1. **Chemical composition of examined cast iron**												
Element	C	Si	Mn	P	S	Cr	Ni	Cu	B	Ti	V	Mo
Mass part, %	3.05	0.97	0.81	0.047	0.015	1.85	4.46	0.087	0.025	0.037	0.28	0.36

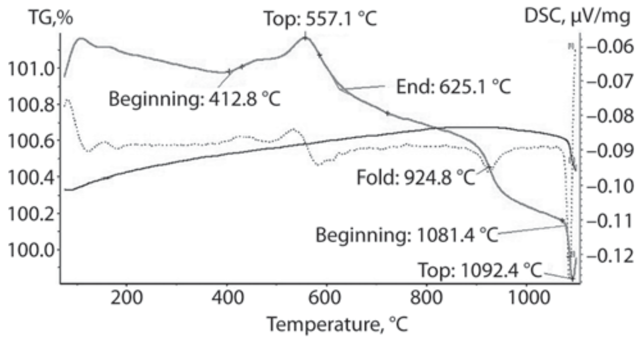

Fig. 1. Differential Scanning Calorimetry (DSC) and Thermogravimetry (TG) heating curves of examined cast iron with rate 10 °C/min

The aim of this study is reveal of regularities in forming the structure of mottled cast iron as a result of graphitized annealing. It is necessary for this purpose to examine dynamics of the process of graphite extraction during annealing. It is also required to determine the degree of influence of graphite amount on amount of carbide phase and residual austenite in metallic base of cast iron, as well as its microhardness.

Technique of experimental investigation

Experimental samples with dimensions 35×35×10 mm (their chemical composition is presented in the **Table 1**) were molten in the induction furnace with charge mass 2 kg and basic lining.

Chemical composition of the samples was determined by SPECTRO spectrometer, MAXx model.

Annealing time was varied from 30 to 300 min with 0.5 hour pace. Annealing temperature was chosen based on the results of thermal analysis of the examined cast alloy during continuous heating with rate 10 °C/min (**Fig. 1**).

Thermal analysis was conducted using STA 449 F3 Jupiter thermal analyzer made by Netsch company. Thermal gravimetrical analysis data testify that gradual mass increase takes place up to 900 °C temperature, what can be connected with sample oxidation. When heating above 900 °C temperature, gradual mass decrease occurs, and this process is characterized by larger intensity for the temperature above 1080 °C. This mass decrease can be explained by alloy decarbonization, this process is more dynamic after starting of dissolution of chromium-alloyed cementite. Carbide phase graphitization during continuous heating starts at 924.8 °C, therefore the temperature of isothermal holding during annealing is accepted to equal to 900 °C. The samples were placed in the protective medium (flux with melting temperature appr. 700 °C) during heat treatment to prevent oxidation and decarbonization.

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 Thixomet 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.

X-ray structural analysis was conducted at X-ray diffractometer Shimadzu XRD-7000 with 40 kV voltage and 30 mA electric current; X-ray tube anode material was chromium.

Microhardness was determined by Buchler Mikromet hardness measuring instrument in accordance with GOST 9450-60 via diamond pyramid pressing, with 136° angle between opposite planes, with 1 kg load and 10 s loading time.

Obtained results

Crystallization is accomplished with high speed owing to melt pouring in metallic mould, and the formed structure consists of martensite-austenite metallic base, carbide phase and graphite. High-temperature heat treatment is presented by graphitized annealing and finalizes in quantitative redistribution of phases in the structure of mottled cast iron (**Fig. 2**).

Graphite amount in the structure of researching cast iron increases from 0.2% to 1.5% after annealing during 150 min. Additional increase of heat treatment time

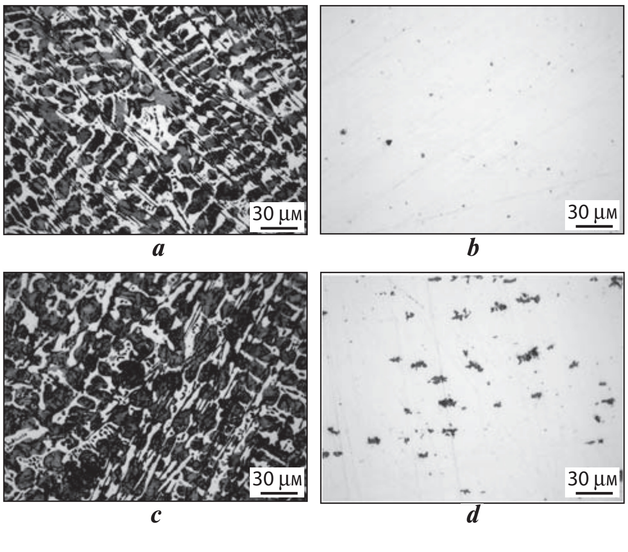

Fig. 2. Cast iron microstructure in cast state (*a*, *b*) and after annealing at 900 °C during 150 min (*c*, *d*), ×500; *a*, *c* — pickled; *b*, *d* — not pickled

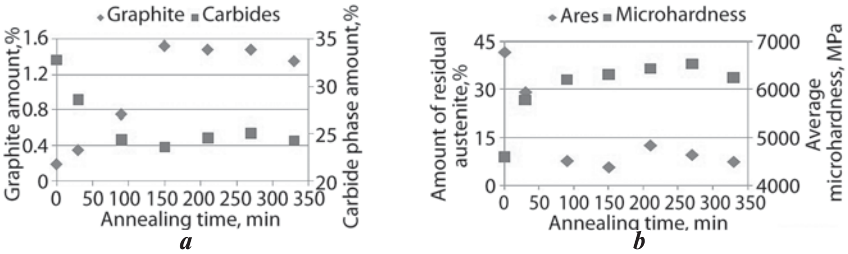

Fig. 3. Relationship between graphite and carbide phase amounts (*a*), amount of residual austenite in the structure and average microhardness (*b*) of researching samples relating to annealing time at the temperature 900 °C

carbides, while quantitative relationship itself is characterized by power features (**Fig. 4**).

Variation of the amount of residual austenite in metallic base testifies about variation of its stability to decomposition during cooling owing to varying alloying degree. First of all, carbon amount in solid solution varies due to graphite extraction and carbide phase dissolution.

Joint analysis of quantitative relationships between residual austenite, graphite and carbides (from one side) and annealing time (from another side) shows that graphite has the main effect on the amount of residual austenite. Increase of its amount in the structure of researching cast iron leads to decrease of stability of overcooled austenite and more complete transformation into martensite during cooling (**Fig. 5, *a***). Dissolution of carbide phase during annealing does not lead to any increase of decomposition resistance for residual austenite (**Fig. 5, *b***). Based on this conclusion it was established that graphite is extracted simultaneously due to dissolution of carbide phase and austenite depletion by carbon, while the process of carbides dissolution does not lead itself to increase of decomposition stability of solid solution during overcooling.

Amount of residual austenite in metallic base of mottled cast iron has the main effect on its average microhardness and is characterized by inverse proportion: the less is amount of residual austenite in the structure, the more is cast iron microhardness (**Fig. 6**). It should be noted that decrease of microhardness of researching cast iron starts at residual austenite content more than 15%. Increase of the part of "soft" component in the structure presented by graphite does not lead to decrease of average microhardness of annealed samples, as well as increase of the part of "hard" (carbide) phase does not lead to its growth.

leads to slight gradual decrease of graphite amount in the structure to 1.35% (annealing during 330 min), what can be connected with decarburization process starting after holding during 300 min.

Relationship between carbide phase amount and holding time is characterized by inverse character relating to graphite: its amount decreases from 32.8% in cast state to 23.6% after annealing during 150 min. Further increase of graphitized annealing time does not lead to any distinct variation and remains on the level 23.5–25.0% (**Fig. 3, *a***).

Quantitative relationship between martensite and austenite in metallic base also varies: the amount of residual austenite decreases from 41.7% (in cast state) to 7.3% (after annealing during 330 min). At the same time minimal content of residual austenite makes 5.6% after annealing during 90 min (**Fig. 3, *b***). Variation of quantitative relationship between metallic base phases leads to variation of average microhardness of researching samples: in makes 4,600 MPa in cast state. Annealing at 900 °C promotes increase of average microhardness to 6,200–6,500 MPa.

Essential increase of microhardness value is observed during 90 min of annealing (to 6,200 MPa), then microhardness value is varying in the range 6,200–6,500 MPa.

Discussion of results

Analysis of the relationships between amounts of graphite and carbide phase and annealing time displays that graphite is partially extracted due to dissolution of

Conclusions

Annealing of mottled nickel-chromium cast iron, used for manufacture of work layer of centrifugally cast

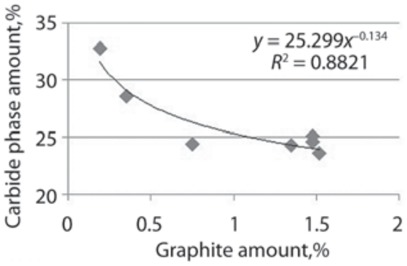

Fig. 4. Relationship between carbide phase amount and graphite amount in the structure of mottled cast iron

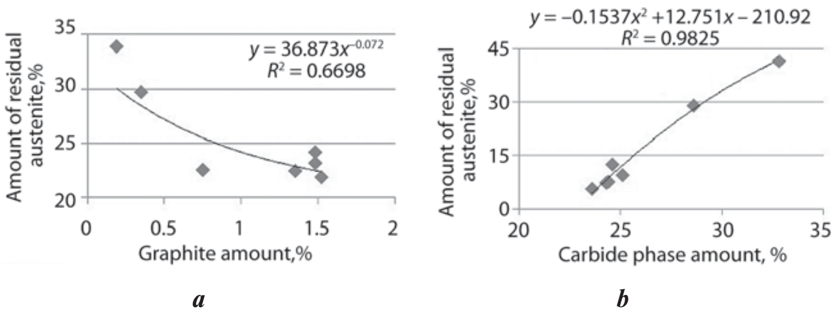

Fig. 5. Relationship between amount of residual austenite and graphite amount (*a*) and carbide phase (*b*) in the structure of mottled cast iron

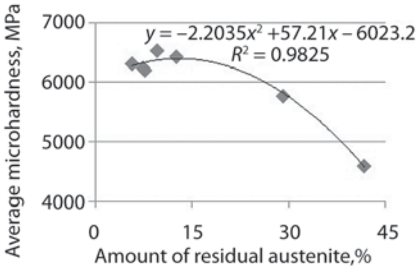

Fig. 6. Relationship between average microhardness of mottled cast iron and amount of residual austenite in its structure

rolling rolls, at 900 °C temperature leads to partial dissolution of carbide phase with simultaneous graphite extraction. Maximal graphite amount (1.5%) is extracted after annealing during 150 min, further high-temperature isothermal holding does not support additional graphite extraction and leads on the contrary to slight decrease of its amount in the structure.

Graphite is extracted simultaneously owing to dissolution of carbide phase as well as decrease of carbon amount in austenite.

Graphite also provides regulation of residual austenite in the structure of researching cast irons; increase of its amount accompanies by increase of martensite amount in metallic base. Amount of carbide phase has no effect in this case on quantitative relationship of phases in metallic base.

Average microhardness of annealed samples of mottled cast iron depends on amount of residual austenite: the less is this amount, the higher is average microhardness. At the same time amount of graphite and carbide phase in the structure has no effect on this parameter of examined cast iron.

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