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EFFECT OF MEDIUM-TEMPERATURE ADDITIONAL TEMPERING ON THE CARBIDE PHASE AND COLD RESISTANCE OF HEAT-HARDENABLE STEEL 09G2SA-A

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The interrelation between the fine structure and the cold resistance of forgings and rolled sheets from ferritic-pearlitic steel 09G2SA-A after heat hardening and additional tempering at 450°C is studied. The additional medium-temperature tempering is shown to raise effectively the cold resistance of the steel due to coagulation and spheroidization of the cementite-type carbides, which make the structure of the steel more equilibrium.

Key words: heat treatment, microstructure, medium-temperature additional tempering, carbide phase, cold resistance, heat-hardenable steel.

INTRODUCTION

It is acknowledged universally that the level of cold resistance of heat-hardenable steels can be elevated by stabilizing their microstructure by quenching with high-temperature tempering, when the carbide phase is conventionally "coagulated" and "globularized." At the same time, some data show that articles from heat-hardenable structural steels serving for a long term in the power industry and in other applications at a moderate temperature of 250 – 350°C may exhibit lowering of the cold resistance of the metal with respect to the initial level due to thermal aging [1 – 9]. Consequently, since the classical heat hardening that consists of quenching and high-temperature tempering does not provide the required structural stability, the problem may be solved by developing an advanced, i.e., three-stage heat treatment process.

A new approach to stabilization of the structure of heat-hardenable steels has been suggested in [10 – 14] and consists in using an additional medium-temperature tempering, which promotes precipitation of carbon from the supersaturated solid solution into α-Fe and growth of cementite-type carbides due to dissolution of the earlier formed precipitates of the less stable ε-carbide and their subsequent coagulation. Experiments have shown that additional tempering at 450°C is favorable for carbide formation and thus elevates the cold resistance of the steel. For example, the impact toughness of the steel of V-notched specimens (type 11 in the GOST 9454 Standard) at negative temperatures increases by a factor of 1.5 – 3 [10, 11]. At the same time, the kinetics of the precipitation and coagulation of the carbide phase in the additional tempering (450°C) with allowance for the heat hardening, the possibilities of which for elevation of the cold resistance of steels have been virtually exhausted, has not been studied sufficiently.

The aim of the present work was to study the changes in the carbide phase in the structure of low-carbon steel 09G2SA-A after a medium-temperature additional tempering (MAT) and its effect on the cold resistance of the metal.

METHODS OF STUDY

We studied low-carbon sparingly alloyed steel 09G2SA-A as delivered in two states, i.e., a ring forging (diameter 2100 mm, thickness 350 mm) and a rolled sheet 25 mm thick. The chemical composition of the steel is presented in Table 1. The rolled sheet and the forging were heat treated in three stages, i.e., quenching + high-temperature tempering + medium-temperature additional tempering. The

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TABLE 1. Chemical Composition of Steel 09G2SA-A

Semiproduct	Content of elements, wt.%										
	C	Mn	Si	P	S	Cr	Ni	Al	Ti	As	N
Sheet	0.07	1.44	0.54	0.006	0.004	0.08	0.08	0.042	0.005	0.008	0.008
Forging	0.09	1.46	0.60	0.008	0.003	0.13	0.25	0.031	0.005	0.008	0.008

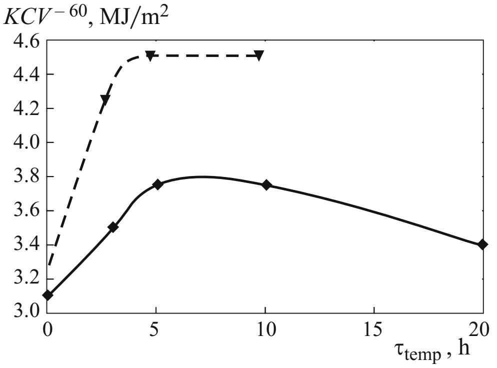

Fig. 1. Effect of the duration of coagulation and spheroidization of cementite-type carbides under medium-temperature additional tempering at 450°C on the impact toughness of a forging and of a rolled sheet from alloy 09G2SA-A at –60°C: ◆) forging; ▼) rolled sheet.

treatment parameters in these stages were 910°C, 1 h, water + 670°C, 2 h, air + 450°C for the sheet and 930°C, 10 h, water + 670°C, 30 h, air + 450°C for the forging. The duration of the MAT at 450°C was varied from 2 to 20 h for both cases; the cooling was conducted in air. To determine the effect of the MAT on the structural and mechanical condition of the steel, we measured its impact toughness at negative temperatures on specimens with a V-notch of type 11 of the GOST 9454 Standard (ISO-V-Probe). The impact toughness tests were conducted using MK-300 and RKP 450 Zwickau/Roell pendulum impact machines (Germany) with maximum impact energy 300 J (the forging) and 450 J (the rolled sheet), respectively. We tested three specimens for each variant of heat treatment with allowance for the variation of the MAT.

The structural features of the carbide phase differing in the fineness and morphology of the cementite depending on the temperature and time parameters of the MAT were studied by the method of transmission electron microscopy. After the tests for impact toughness, we fabricated round bars with diameter 3 mm and length 20 mm from halves of the specimens, cut them into strips with a thickness of 0.7 mm with the help of a Struers Minitom precision cutting mill, and ground them to a thickness of 0.1 mm. Thin foils were obtained by electrolytic etching in a Struers A-II chlorine-alcohol electrolyte at a voltage of 28 V and a temperature from 0 to +2°C using a Struers Tenupol 5 device.

The fine structure of the steel was studied under a Tecnai G2 30 S-TWIN FEI transmission electron microscope at an accelerating voltage of 200 kV. Images of the structure were obtained using a Gatan Ultrascan-1000 CCD camera attached to the transmission electron microscope. The dislocation density was determined by the method of secants. The phases were identified by the method of single reflections. The shapes and sizes of the carbide inclusions were studied using the Image J image-processing software, which allowed us to describe the shape of the projection of an individual carbide particle by the C (Circularity) method with the help of the relation

$$C = 4\pi \frac{A}{P^2},$$

where A and P are the area and the perimeter of the projection of the analyzed particle, respectively, and C is a parameter characterizing the degree of circularity of an individual inclusion [15]. We analyzed at least 200 particles for every specimen studied.

RESULTS AND DISCUSSION

The results of the complex studies of the effect of the temperature and time parameters, of the duration of the additional medium-temperature tempering at 450°C in the first turn, on the impact toughens at –60°C of the specimens fabricated from the forging and from the rolled sheet of steel 09G2SA-A are presented in Fig. 1. It can be seen that the best result is obtained after a 5 – 10-h exposure to MAT. At this duration of MAT, the impact toughness of the forging with a thickness of 350 mm exceeds 3.75 MJ/m^2. This a virtually ultimate value measurable by a pendulum impact machine with maximum impact energy 300 J used in the tests. It should be noted that the impact specimens treated under this mode of MAT have not fractured totally. Increase of the duration of the MAT at 450°C to 20 h lowers the impact toughness, which is an indirect evidence of some destabilization of the structure. It seems that this is a result of solid-phase dissolution of cementite carbides and subsequent formation of refractory carbides, for example, of niobium.

However, it should be noted that in this case too, the structure of the steel is more equilibrium than that provided by the traditional heat hardening. The situation is similar after a shorter (3 h) MAT, which also indicates higher completeness of the process of carbide formation in the structure of the steel as compared to the two-stage process.

The impact toughness of the specimens from the rolled sheet KCV^{-60} has also attained almost ultimate values for

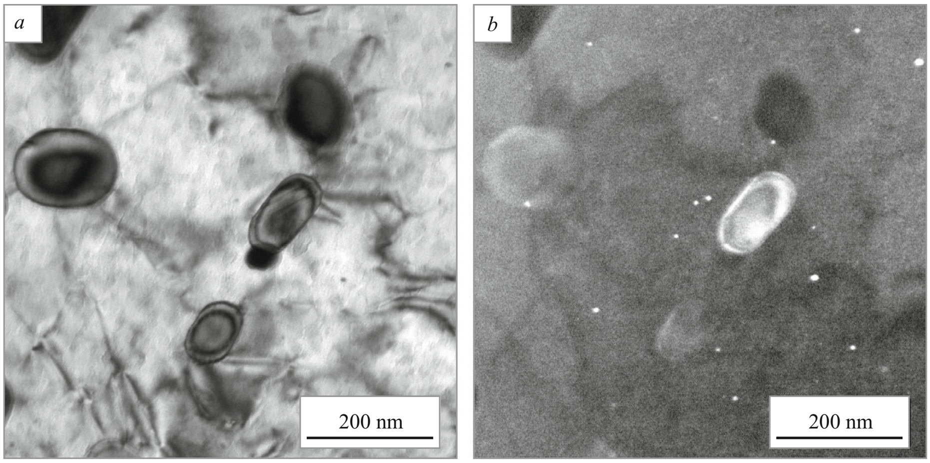

Fig. 2. Fine structure of a forging from steel 09G2SA-A after an additional 10-h tempering at 450°C (SEM): *a*) light-background image; *b*) dark-background image of cementite-type carbides and fine niobium carbides.

the impact machine with pendulum energy 450 J. It can be seen from Fig. 1 that after the MAT at 450°C the impact toughness of the rolled sheet from steel 09G2SA-A exceeds unprecedented 4.50 MJ/m^2. Like in the tests of the forging, the specimens of the rolled sheet have not fractured totally but were considerably deformed.

The results on the effect of medium-temperature additional tempering on the structure and mechanical properties of the rolled sheet and of the forging from steel 09G2SA-A have become a base for patenting the process in the Russian Federation [16, 17].

With allowance for the positive effect of MAT on the cold resistance of billets from steel 09G2SA-A, it is interesting to analyze the kinetics of the processes of coagulation and spheroidization occurring in the steel under such heat treatment.

To investigate the kinetics of formation of the carbide precipitates, we studied the fine structure of the steel after MAT at 450°C and determined the size and the shape of the cementite-type and niobium carbides and of the other excess phases in steel 09G2SA-A. We established that the dislocation density was independent of the duration of the MAT and amounted to $(3 - 5) \times 10^{13}$ m^{-2} for all the specimens subjected to MAT with duration of 3 – 10 h. The main two kinds of carbides in the specimens were cementite particles 100 – 350 nm in size and fine particles of niobium carbide 10 – 30 nm in size (Figs. 2 – 4). The cementite-type carbides were chiefly located in the bodies of grains of globular pearlite or over the boundaries of ferrite grains. The fine particles identified as niobium carbides (NbC, fcc-lattice, *a* = 1.115 nm) by analysis of diffraction patterns are chiefly located inside ferrite grains and among cementite particles in pearlite.

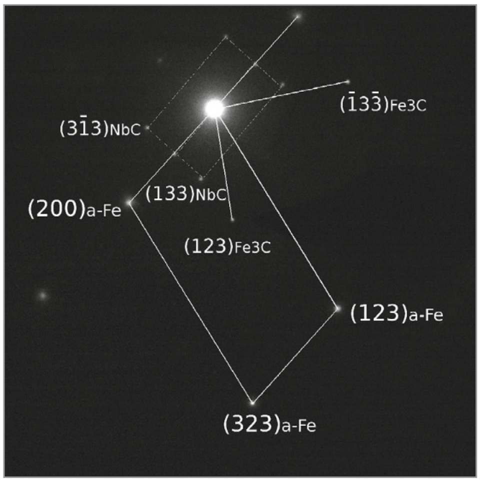

Fig. 3. Electron diffraction pattern of the structure and phase condition of cementite-type carbides and niobium carbides in the structure of steel 09G2SA-A (TEM).

MAT also affects the size and the shape of the carbide particles (Fig. 4*c* and *d*). Statistical analysis of the values of parameter *C*, which is the ratio of the area of the projection of the carbide precipitate analyzed to the squared perimeter of this projection (i.e., characterizes the closeness of the projection of an individually taken precipitate to a circle), shows that the shape of the precipitate approaches a sphere when the duration of the MAT is increased. Parameter *C* varies from 0 to 1, where 0 corresponds to precipitation of maximally elongated particles and 1 corresponds to precipitation of perfectly round particles.

The data of Table 2 obtained for MAT of different durations (3 – 10 h) show that parameter *C* increases with growth

TABLE 2. Sizes and Morphology of Carbide Particles in the Structure of Steel 09G2SA-A

Duration of MAT at 450°C, h	Mean size of carbides, nm	Degree of circularity *C*	Faction of particles with *C* > 0.85, %
Initial condition (after heat hardening)	230	0.81	42
3	200	0.85	68
5	220	0.87	70
10	210	0.88	78

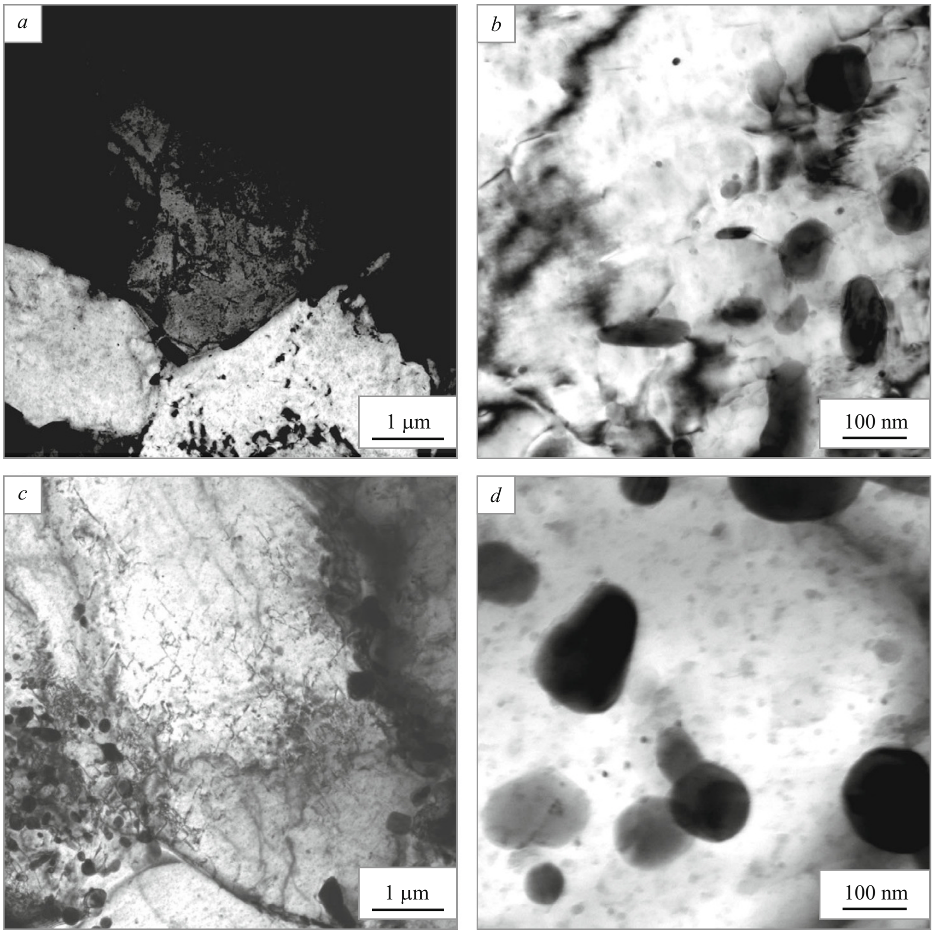

Fig. 4. Fine structure of a forging from steel 09G2SA-A with different shapes of globular-elliptical cementite-type carbides after heat hardening (*a*, *b*) and after 5-h MAT at 450°C with round cementite-type carbides (*c*, *d*) (TEM).

of the time of the MAT at 450°C. The proportion of the particles with parameter *C* exceeding the value averaged for all the measurements increases too.

The size distribution and the distribution of the degree of circularity of the cementite-type carbides is presented in Fig. 5. It can be seen that the structure of the steel in the initial condition (heat hardening) and after the 3-h MAT is characterized by lower homogeneity of the carbide phase, and the peak near the mean value is wider than after the MAT with duration 5 h and 10 h. The size distribution of the carbides and the distribution of the degree of their circularity in the structure the steel after the MAT with duration 5 h and 10 h behave virtually similarly.

More detailed studies have shown that prolongation of MAT causes decrease in the content of disperse carbides 50 – 100 nm in size. The content of coarser carbides with a size of 200 – 300 nm increases, which is a feature of a carbide-forming process of coagulation of the carbide phase, which occurs virtually without preservation of coherent bonding with the matrix. Thus, we may expect that MAT at 450°C for 5 – 10 h should make the structure of steel 09G2SA-A more stable due to the changes in the sizes and in the morphology of the carbide phases. Since this structure is more equilibrium, it provides maximum cold resistance in the steel (Fig. 1). The results obtained allow us to infer that the substantial effect of coagulation and spheroidization of the carbide phase, which raises the cold resistance of the steel, is provided by cementite-type carbides when their structural state is controlled by the method of MAT.

It should be noted that the mean size of the cementite-type particles in steel 09G2SA-A varies less than their shape. When the duration of the MAT is increased from 3 to 10 h, the shape of such particles becomes progressively more spherical. Globular shape of the cementite-type carbides obviously causes lowering of the effect of stress concentration and deformations on the carbide-matrix interface, which also promotes growth of the cold resistance of the steel [8, 18, 19].

After analyzing the transformation of the cementite-type carbides, we studied the kinetics of the precipitation of fine niobium carbides. It is shown in Fig. 6 that the 3-h MAT increases the size of the refractory carbide particles from 10 to 15 nm. The proportion of the particles 20 – 25 nm in size is somewhat lower. After the 5-h MAT, the number of particles 15 – 25 nm in size increases noticeably and this is accompanied by simultaneous formation of new carbide particles with a size of 10 nm. After the 10-h MAT, the size distribution of the particles becomes more uniform, which should also influence positively the cold resistance of the steel.

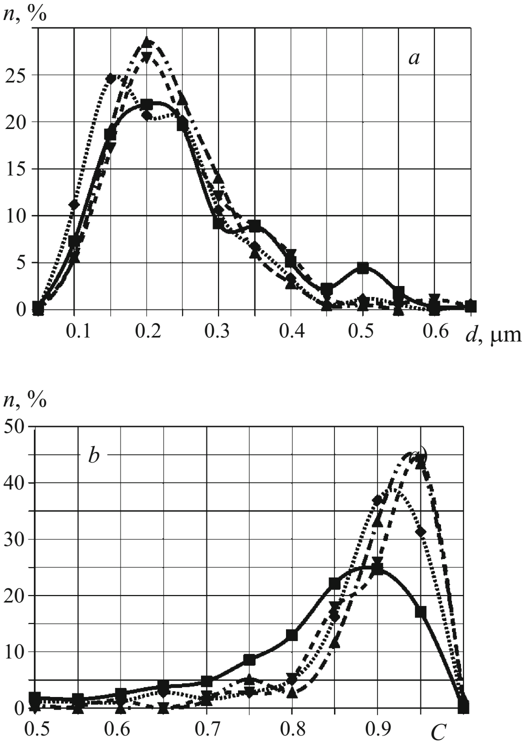

Fig. 5. Size (*d*) distribution of cementite-type carbides (*a*) and distribution of parameter *C* (*b*) in the structure of steel 09G2SA-A prior to (■) and after MAT at 450°C for 3 h (◆), 5 h (●) and 10 h (▲) (*n* is the fraction of particles).

The data on the interrelation between the structure and the properties of steel 09G2SA-A speak in favor of steady influence of the coagulation and spheroidization of carbide particles in the structure on growth of the cold resistance of the metal after medium-temperature additional tempering of different durations. The level of the cold resistance attained after MAT exceeds considerably the one provided by the standard heat hardening, which is assumed to be preferable for the "most equilibrium" state of the metal.

According to the classical physical metallurgy, quenching and tempering provide the demanded combination of properties in a conventionally "equilibrium" condition when the formed structure is fine-grained. This occurs due to lowering of the internal stresses caused by quenching. It is also known that high-temperature tempering is accompanied by a number of carbide processes that to do not develop completely, i.e.,

– decomposition of martensite, which yields carbides (about 350°C);

– transformation of ε-carbide into cementite (350 – 400°C);

– coagulation and spheroidization of carbides (450°C and higher temperatures).

However, it would not be correct to think that the individual processes of coagulation and spheroidization of carbides under high-temperature tempering, which are accompanied by other processes (for example, decomposition of the retained austenite, cell formation and recrystallization of the ferrite matrix) are sufficient conditions for formation of an "equilibrium" state in the metal. In this case the relatively high level of the impact toughness of the steel at reduced temperatures is explainable by the fact that the determining factors are the temperature and time parameters of the high-temperature tempering with allowance for the quenching cooling intensity [20].

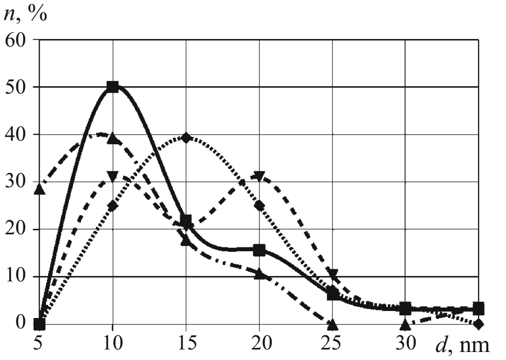

Fig. 6. Size distribution of niobium carbides as a function of the duration of MAT at 450°C for steel 09G2SA-A (*n* is the fraction of particles; *d* is the particle size): ■) initial condition; ◆) duration of MAT 3 h; ●) 5 h; ▲) 10 h.

The results of the study of the efficiency of the new operation (medium-temperature additional tempering) show that it is expedient to replace the classical two-stage heat treatment process by a new three-stage one. Even the less effective modes of MAT, for example the too short 3-h tempering (precipitation of cementite-types carbides) or the too long 20-h tempering (dissolution of cementite-type carbides) produce a higher level of cold resistance than the transitional heat hardening. This proves that the coagulation and spheroidization process occurring in the structure of the steel under heat hardening have a partial or episodic nature, whereas in the case of MAT they develop fully and cause substantial and virtually direct increase in the cold resistance of the steel.

CONCLUSIONS

Methods of optical and electron microscopy have been used to detect effective occurrence of processes of coagulation and spheroidization of carbide phase in the structure of steel 09G2SA-A in the course a novel heat treatment operation, i.e., medium-temperature additional tempering at 450°C. After the medium-temperature additional tempering integrated into the classical heat treatment, i.e., heat hardening represented by quenching and high-temperature tempering, the cold resistance of forgings and sheets from steel 09G2SA-A increases considerably.

Medium-temperature additional tempering of steel 09G2SA-A at 450°C for 5 – 10 h conducted after the heat hardening makes it possible to

– increase the size of a maximum amount of cementite-type carbides from 130 – 230 nm to 160 – 260 nm, which indicates that their medium-temperature coagulation is a carbide-forming process;

– change the shape of the cementite-type carbides from an elliptical one to a spherical one, which doubles the circularity of the carbide particles;

– obtain a more equilibrium structure in the steel;

– raise the resistance of the metal of forgings and sheets from heat-hardenable steel 09G2SA-A to brittle fracture at low temperatures, i.e., the impact toughness KCV^{-60} is increased by more than a factor of 1.5.

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