
STRUCTURE, PHASE TRANSFORMATIONS,
AND DIFFUSION

Structural and Phase Changes in Carbides of the High-Speed Steel upon Heat Treatment

A. S. Chaus

Slovak University of Technology, Faculty of Materials Science and Technology, Paulinska 16, Trnava, 91724 Slovakia
e-mail: alexander.chaus@stuba.sk

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Abstract—The effect of austenitizing temperature on structural and phase changes in carbides of the tungsten–molybdenum high-speed steel has been studied. The results of metallographic analysis and energy dispersive microanalysis have been discussed. It has been shown that an increase in austenitizing temperature from 1180 to 1260°C causes structural transformations in carbide particles of eutectic origin crushed upon hot plastic deformation, which are related to their dissolution and coalescence, and changes in the phase composition of the carbides themselves.

Keywords: high-speed steel, carbides, heat treatment, austenitizing

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INTRODUCTION

High-speed steels are alloys of the ledeburitic class due to the presence of a eutectic constituent in their structure, which is formed at the final stage of the solidification of the crystallization of melt and forms a developed network of carbides at the grain boundaries of the matrix. The nature of the carbides precipitated upon the eutectic reaction depends on the chemical composition of the high-speed steel and on the conditions of melt solidification, especially on its cooling rate [1–5]. During the crystallization of tungsten–molybdenum high-speed steels, including the R6M5 steel, which is the most widespread, the eutectic is formed based on M_2C , M_6C , and MC carbides. The volume ratio of these carbides can be varied in a cast state. The introduction of small additions of pure elements or their compounds in the form of carbides, nitrides, and borides [2, 6, 7] into the melt or variations in the solidification and crystallization rate [2, 5, 8] makes it possible to purposefully control the amount of a given eutectic in the alloy.

After solidification, ingots of high-speed steel are generally subjected to annealing and subsequent hot plastic deformation to remove the carbide network at the grain boundaries of the matrix via the deformation and crushing of the carbides of eutectic origin. During the annealing and hot plastic deformation, the high-speed steel is exposed to a temperature of up to 1200°C (or, in the case of R6M5 steel, 1130°C) [9]. Furthermore, from the viewpoint of obtaining an optimum complex of mechanical and exploitation properties of the cutting tools, the thermodynamic stability of individual eutectic carbides plays an important role. In particular, it has been shown that M_6C and MC car-

bides are very stable phases, whereas the carbides of M_2C type have low thermodynamic stability. As a rule, during heating to the above-mentioned temperatures, M_2C carbides decompose completely and form a mixture of M_6C and MC carbides. As a consequence, the mechanism and kinetics of decomposition of the M_2C carbides were studied in much detail upon the high-temperature treatment of metallurgical ingots [10–13] or samples produced using different casting processes [14–18], including the electroslag remelting [19] and centrifugal casting [20]. Moreover, it was argued in review paper [2] that no M_2C carbides are present in the microstructure of final products made from high-speed steels of M2 type (analog of the R6M5 steel) and M7 type (steel containing 1 C, 3.75 Cr, 1.75 W, 8.7 Mo, and 2 V wt %), which are subjected to the temperature in the ranges investigated during heat treatment or thermomechanical processing (hot plastic deformation). However, investigations that would confirm or deny the presence of the M_2C carbides in the microstructure of wrought high-speed steel after complete cycle of heat treatment, are absent. With regard to changes in carbides of eutectic origin during final heat treatment, cast high-speed steels that are not subjected to hot plastic deformation are better studied compared with deformed steels [20–22]. In this context, in the present paper, we studied the structural and phase transformations in carbides of the wrought high-speed R6M5 steel after a complete cycle of the heat treatment at which the austenitizing temperature was changed in the range of 1180–1260°C.

Table 1. Chemical composition of the investigated high-speed R6M5 steel

Wrought steel	Content of elements, wt %										
	Fe	C	Si	Mn	Ni	P	S	Cr	Mo	W	V
	Base	0.85	0.44	0.39	0.34	0.021	0.023	4.01	5.49	6.14	2.02
Cast steel	Base	0.88	0.26	0.29	0.26	0.023	0.028	4.03	5.34	5.95	1.89

EXPERIMENTAL

R6M5 steel, the chemical composition of which is shown in Table 1, was supplied in the as-annealed state in the form of a rolled rod with a diameter of 30 mm. Samples with a height of 10 mm were cut from the rod for the metallographic analysis and electron microprobe analysis. The microstructure of cast R6M5 steel of close chemical composition (Table 1) was studied using samples $10 \times 10 \times 55$ mm in size, which were cut from 1.3-kg ingots cast into a ceramic mold.

The heat treatment of samples included austenitizing, quenching, and tempering. The heating of samples was carried out in two stages, i.e., after heating to 850°C in a melted salt (70% $\text{BaCl}_2 + 30\%$ NaCl), the temperature of samples was increased to a preset temperature in a 95% $\text{BaCl}_2 + 5\%$ MgF_2 solution. During austenitizing, the samples were kept at temperatures of 1180, 1200, 1220, 1240, and 1260°C with the same exposure of 10 s per 1 mm of the cross section for all samples. During quenching, the cooling was carried out at 550°C in a mixture of 50% $\text{CaCl}_2 + 50\%$ NaCl and further in air. The heat treatment of the investigated samples was completed by triple tempering at 560°C for 1 h in a salt bath of 90% $\text{KNO}_3 + 10\%$ NaOH .

The microstructure of the steels was studied using a NEOPHOT-22 optical microscope and a JEOL JSM-7600F scanning electron microscope (SEM),

which was equipped with an energy dispersive attachment (Oxford Instruments) for the electron microprobe analysis. The samples for metallographic studies were prepared according to the conventional procedure. After grinding and polishing, the samples for studies in an optical microscope were etched in Vilella-Bain etchant (5 g of picric acid + 5 mL of hydrochloric acid + 95 mL of ethanol). Unetched samples were used for the SEM study. The statistical processing of the microstructure images of the investigated steel to determine the sizes and volume fractions of the carbide particles of eutectic origin was performed using an ImageJ software. A total of 400 measurements were carried out upon the determination of each characteristic.

RESULTS AND DISCUSSION

The typical morphology of the $M_2\text{C}$ and $M_6\text{C}$ eutectic carbides, which are usually present in the microstructure of the R6M5-type high-speed steel in the as-cast state is shown in Figs. 1a and 1b, respectively. The carbides differ not only in a specific morphology, but also in the contents of the alloying elements. It can be seen from the presented data given in Table 2 that the concentrations of chromium and especially vanadium in the $M_6\text{C}$ carbides is lower than in the $M_2\text{C}$ carbides. At the same time, the MC carbides have the highest vanadium contents compared to

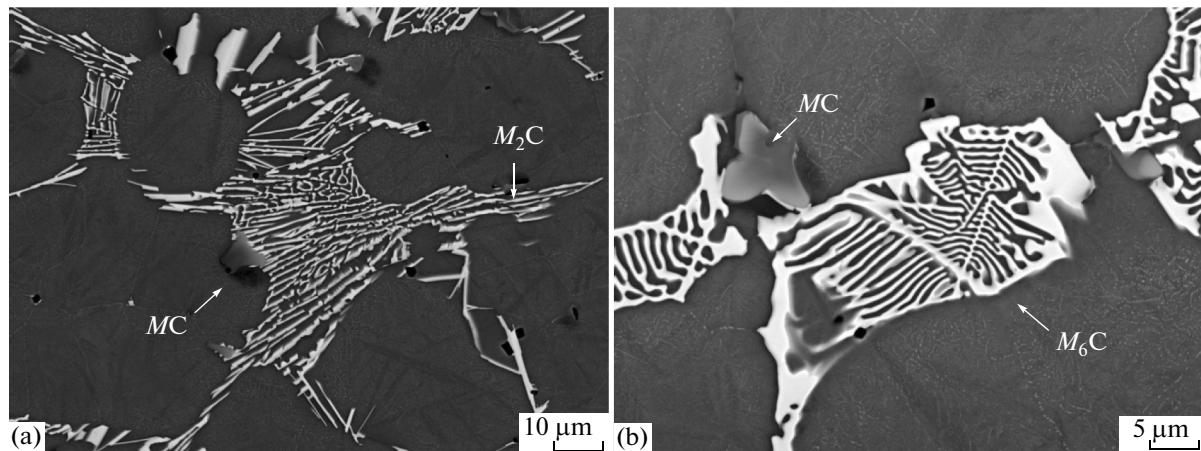


Fig. 1. Morphology of (a) $M_2\text{C}$ and (b) $M_6\text{C}$ carbides in the microstructure of cast high-speed R6M5 steel; SEM.

Table 2. Chemical composition of carbide phases in the microstructure of high-speed steel in the wrought and cast states

Phase, Figure	Content of elements*, wt %					
	C	V	Cr	Fe	Mo	W
R6M5-type cast steel						
M_2C , Fig. 1a	9.62	10.85	6.19	12.70	22.90	37.74
MC , Fig. 1a	15.28	36.92	3.52	17.09	9.26	17.93
M_6C , Fig. 1b	6.73	3.33	4.19	27.77	21.80	36.18
MC , Fig. 1b	13.61	32.25	4.34	13.30	13.13	23.37
Deformed R6M5 steel after annealing						
M_6C , Fig. 3b	5.13	2.74	4.78	35.55	21.68	30.13
MC , Fig. 3b	12.63	29.86	7.10	16.64	13.04	20.73
Deformed R6M5 steel after quenching and tempering						
M_2C , Fig. 4c	10.22	11.09	3.88	54.66	8.87	11.28
M_6C , Fig. 4c	6.97	3.11	3.18	28.16	24.69	33.88
M_6C , Fig. 5a	6.69	3.53	2.75	27.93	26.99	32.12
M_2C (A), Fig. 5a	10.97	11.92	6.01	5.64	32.74	32.73
M_6C (B), Fig. 5a	6.71	2.69	3.18	28.40	25.97	33.05
MC , Fig. 5a top part	15.90	39.58	3.05	3.60	16.29	21.56
MC , Fig. 6a	14.62	40.63	4.21	6.78	17.47	16.30
M_6C , Fig. 6a	6.83	3.28	2.92	27.53	25.29	34.15

* Data on carbon are semiquantitative.

two other types of carbides. Looking ahead, it should be noted that the established features of the chemical composition of carbides in the as-cast state will be important later for interpreting changes that occur in carbides upon the heat treatment of the R6M5 steel.

Figure 2 shows the different thermodynamic stability of M_2C and M_6C carbides present in the microstructure of the cast high-speed steel of R6M5 type upon subsequent heat treatment. It can be seen from Fig. 2a that, at relatively low austenitizing temperature (1200°C), the precipitation of particles of the carbide occurs during the decomposition of the M_2C eutectic carbide. Under the action of the higher austenitizing temperature, the decomposition is completed by the formation of a mixture that consists of pure M_6C and MC carbides (Fig. 2b). The M_6C carbide keeps its initial morphology unchanged after austenitizing at both the temperature of 1200°C (Fig. 2c) and at 1240°C (Fig. 2d).

It is known that, under the action of the hot plastic deformation, a refinement of the carbide particles of eutectic origin occurs [9], which as a result, lose their initial morphology and exhibit carbide banding clearly visible in the longitudinal section of the rolled rod (Fig. 3a). The typical morphology of the eutectic carbides of M_6C and MC types prevailing in the microstructure of the annealed deformed high-speed steel of R6M5 type in the as-supplied state is shown in Fig. 3b; their chemical composition is given in Table 2.

The microstructure of the steel of R6M5 type in the tempered state after quenching from different austenitizing temperatures is shown in Fig. 4 [23]. Against a background of the matrix represented by the tempered martensite, individual carbide particles of eutectic origin are observed that are more or less uniformly distributed therein. With an increase in the austenitizing temperature, the coarsening of the carbide particles occurs. The difference in the size of carbide particles depending on the austenitizing temperature is clearly seen in Fig. 5.

Undoubtedly, the above-mentioned changes relating to the carbide constituent are a result of two simultaneous processes that occur during austenitizing holding, i.e., the dissolution of the less stable carbide particles in the matrix and the coalescence and coarsening of the more stable particles. It should be noted that this is primarily related to smaller particles with a rounded morphology that prevail in the microstructure of steel, rather than to less numerous coarse, elongated particles, which seem to be more stable against an increase in the austenitizing temperature. The more completed dissolution of the elongated coarse particles accompanied by a change in their shape to become rounded only occurs after austenitizing at 1260°C, as can be seen in Fig. 4d.

According to the results of the static processing of data on the parameters of the microstructure of samples presented in Table 3, upon the austenitization, the

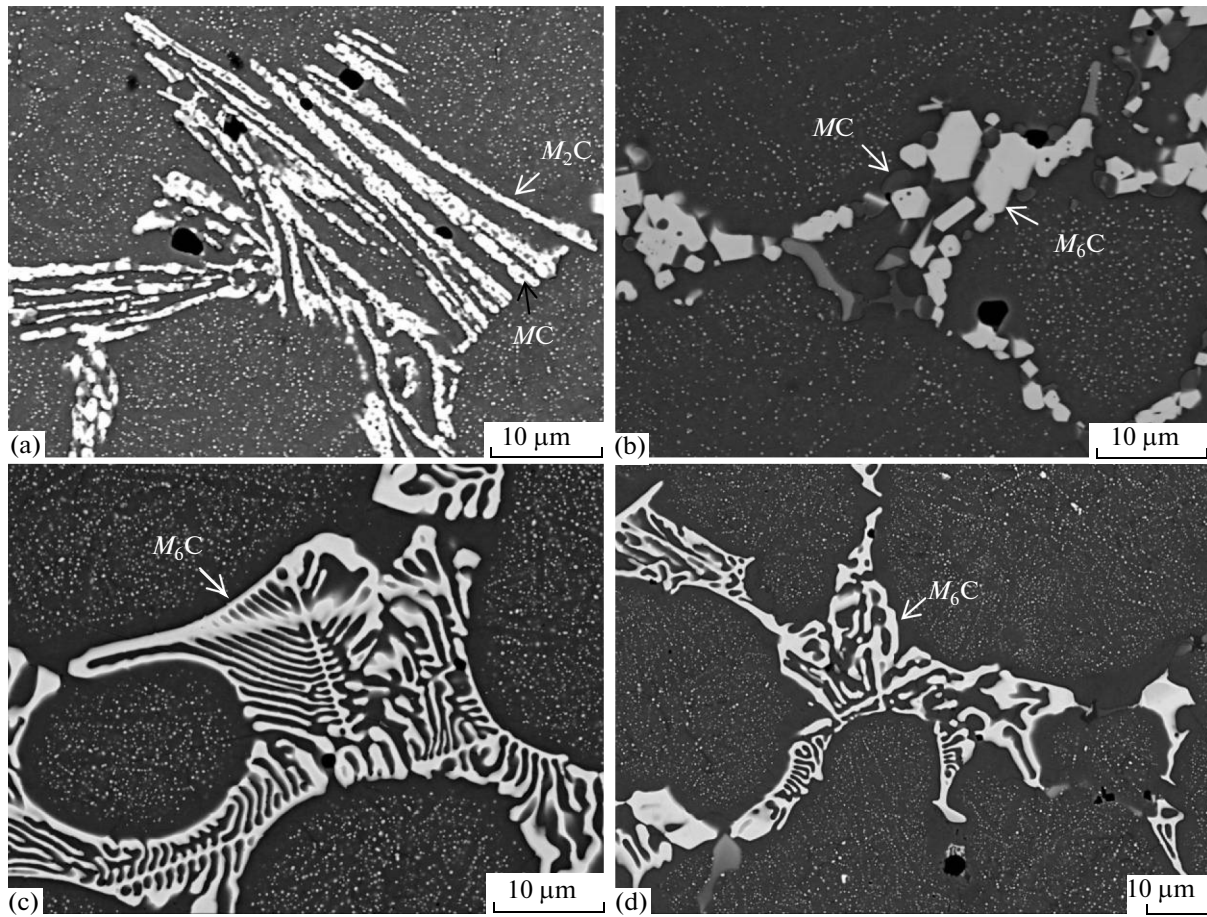


Fig. 2. (a, b) Products of the decomposition of the M_2C carbide and (c, d) morphology of the M_6C carbide in the microstructure of the cast high-speed steel of R6M5 type after quenching and tempering: (a, c) austenitizing temperature 1200°C; (b, d) austenitizing temperature 1240°C; SEM.

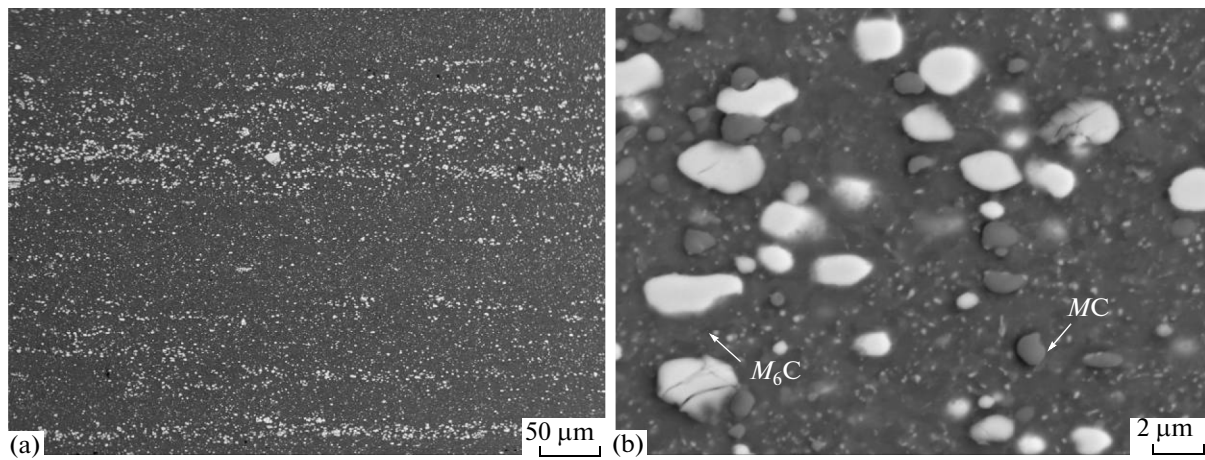


Fig. 3. Microstructure of the wrought high-speed steel of R6M5 type in the as-supplied state (after annealing) at (a) low and (b) high magnifications; SEM.

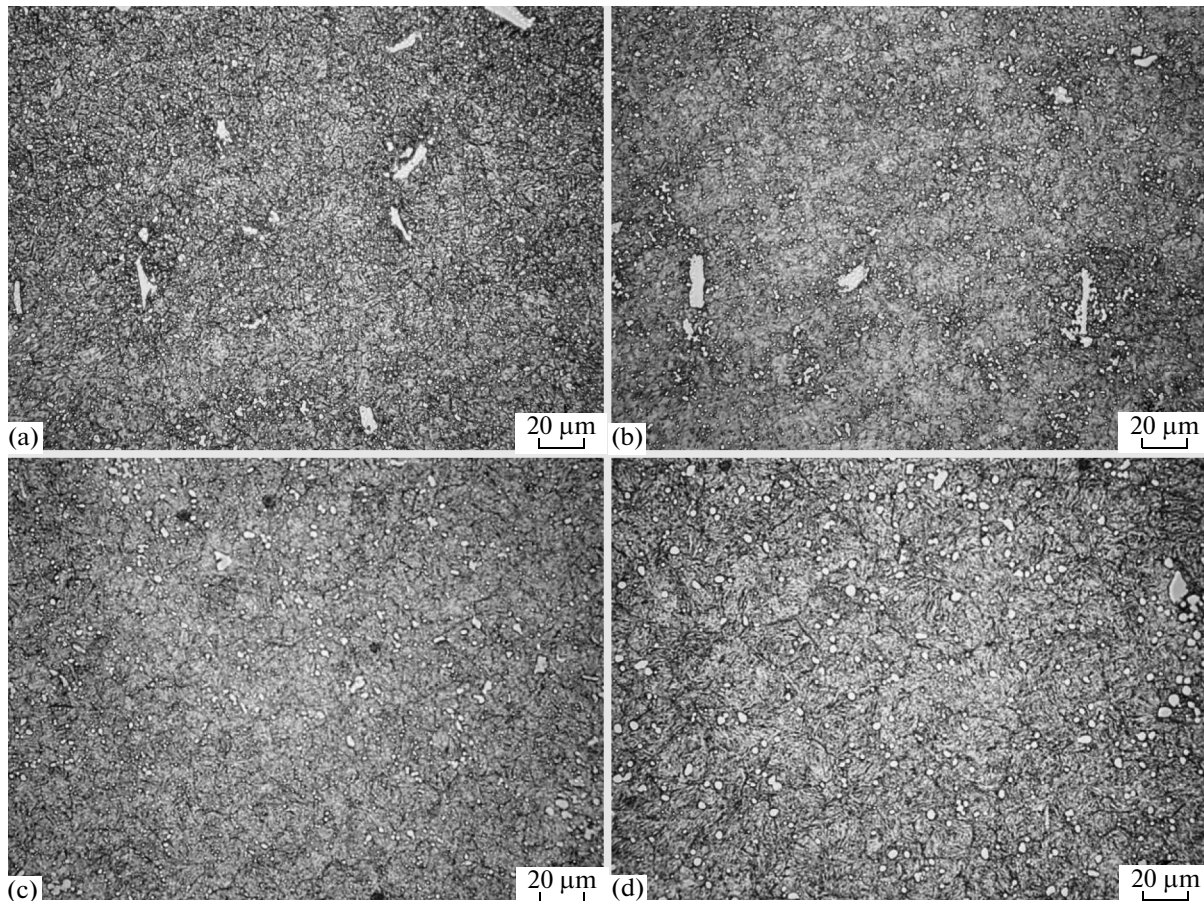


Fig. 4. Microstructure of tempered wrought high-speed R6M5 steel after austenitizing at (a) 1180, (b) 1200, (c) 1220, and (d) 1260°C [23]. Optical micrographs.

process of the dissolution of the carbide particles apparently occurs faster than the process of coarsening and coalescence. As a consequence, despite the increase in the sizes of the carbide particles in steel, their total volume decreases with increasing the austenitizing temperature (Table 3).

The change in the phase composition of carbides in the microstructure of the wrought R6M5 steel under heat treatment is of greater interest. For example, in the microstructure of the tempered wrought steel subjected to austenitizing at a temperature of 1200°C,

Table 3. Results of statistical processing of the microstructure of the carbide constituent of the investigated wrought high-speed steel after complete cycle of heat treatment [23]

Parameter	Austenitizing temperature, °C		
	1180	1220	1260
Average size of carbide particles, μm	4.0	4.7	8.8
Volume fraction of carbides, %	16	14	13

coarse particles of carbides of eutectic origin have been revealed, which, as can be seen in Figs. 6a and 6b, have a two-phase structure. It has been found using the electron microprobe analysis that the chemical composition of the central part of these particles corresponds to the M_2C carbide. The chemical composition of its peripheral region, which contains precipitated particles of MC carbide, corresponds to the M_6C carbide (Table 2). It should be noted that these two-phase particles were also found in the structure of the cast high-speed steel of R6M5 type after heat treatment [21] (Figs. 6c, 6d). Taking into account the precipitated particles of the MC carbide in the peripheral regions of coarse particles of the M_6C carbide, it should be emphasized that these coarse particles in fact have a three-phase rather than two-phase microstructure.

Using an electron microprobe analysis, the small rounded particles were also found in the microstructure of the wrought steel of R6M5 type after heat treatment at which the austenitizing temperature was 1200°C (Fig. 5c). Apparently, these are particles that are incompletely dissolved upon austenitizing, the vanadium content in which corresponds to the M_2C

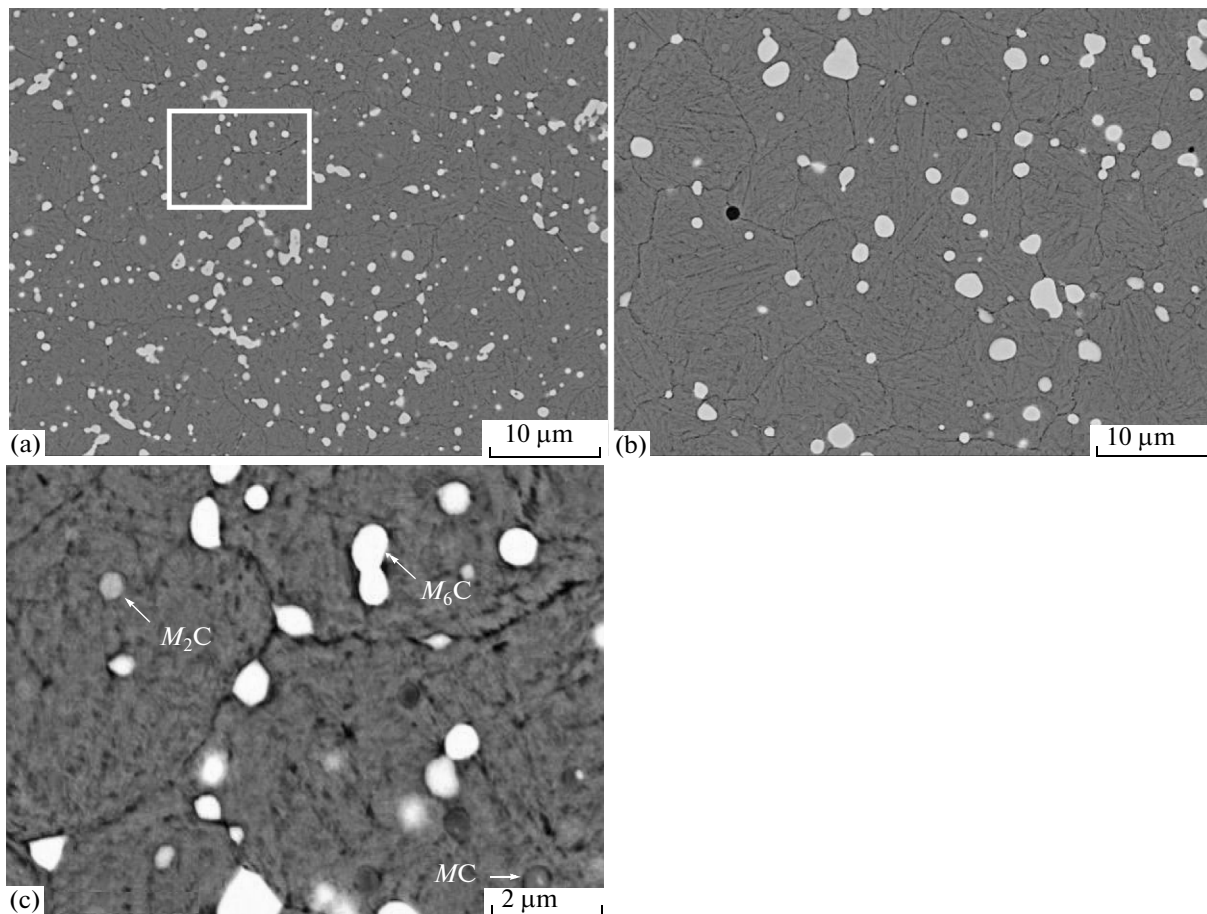


Fig. 5. Microstructure of the tempered wrought high-speed steel of R6M5 type after austenitizing at (a, c) 1200°C and (b) 1260°C; (c) fragment of the microstructure outlined in Fig. 5a at a higher magnification; SEM.

carbide (Table 2). However, to confirm or refute the presence of wrought particles of M_2C carbide in the microstructure of high-speed R6M5 steel after a complete cycle of heat treatment using X-ray diffraction analysis, it was unfortunately not methodically or technically possible.

As for coarse particles of M_6C carbide, the chemical composition of which after heat treatment is presented in Table 2, inclusions of MC carbide can be seen in them. This is shown based on the example of steel samples subjected to austenitizing at 1200°C (Fig. 6a, left part) and 1260°C (Fig. 7a). The character of the distribution of elements over the cross section of the coarse particle of the M_6C carbide (Figs. 7b–7d) shown in Fig. 7a confirms the presence of particles of the MC carbide with a high vanadium content (see Table 2) [23]. The presence of particles of the MC carbide in the coarser particles of the M_6C carbide suggests that their precipitation is related to the incompleteness of their decomposition process of the initial M_2C carbide at the previous stages of the high-temperature treatment (first of the ingot and later of the rolled rod following annealing), or is a regular consequence

of diffusion processes that spontaneously occur in the M_6C carbide.

In our previous works [24, 25], the following mechanism of the decomposition of the M_2C carbide into a mixture of M_6C and MC carbides in the cast high-speed steel of R6M5 type upon its heat treatment was proposed. During the austenitization, vanadium diffuses from the M_2C carbide and, interacting with the austenitic matrix, forms its own carbide of MC type. The continuing precipitation of particles of the M_2C carbide and the simultaneous escape of vanadium and, to a lesser degree, of chromium from the initial M_2C carbide is accompanied by a change of its crystal lattice and the transformation of the M_2C carbide into the M_6C carbide. As a result, when using higher austenitizing temperatures (1240°C and higher), in the microstructure of the cast high-speed steel, after heat treatment, a mixture consisting of isolated carbides of M_6C and MC types instead of the initial carbide of M_2C type is observed [6, 21, 22, 24, 25]; this exerts a significant influence on the final mechanical properties, primarily on the impact toughness and wear resistance of steel [6]. Since, in the wrought high-speed R6M5 steel, even after austenitizing at high tempera-

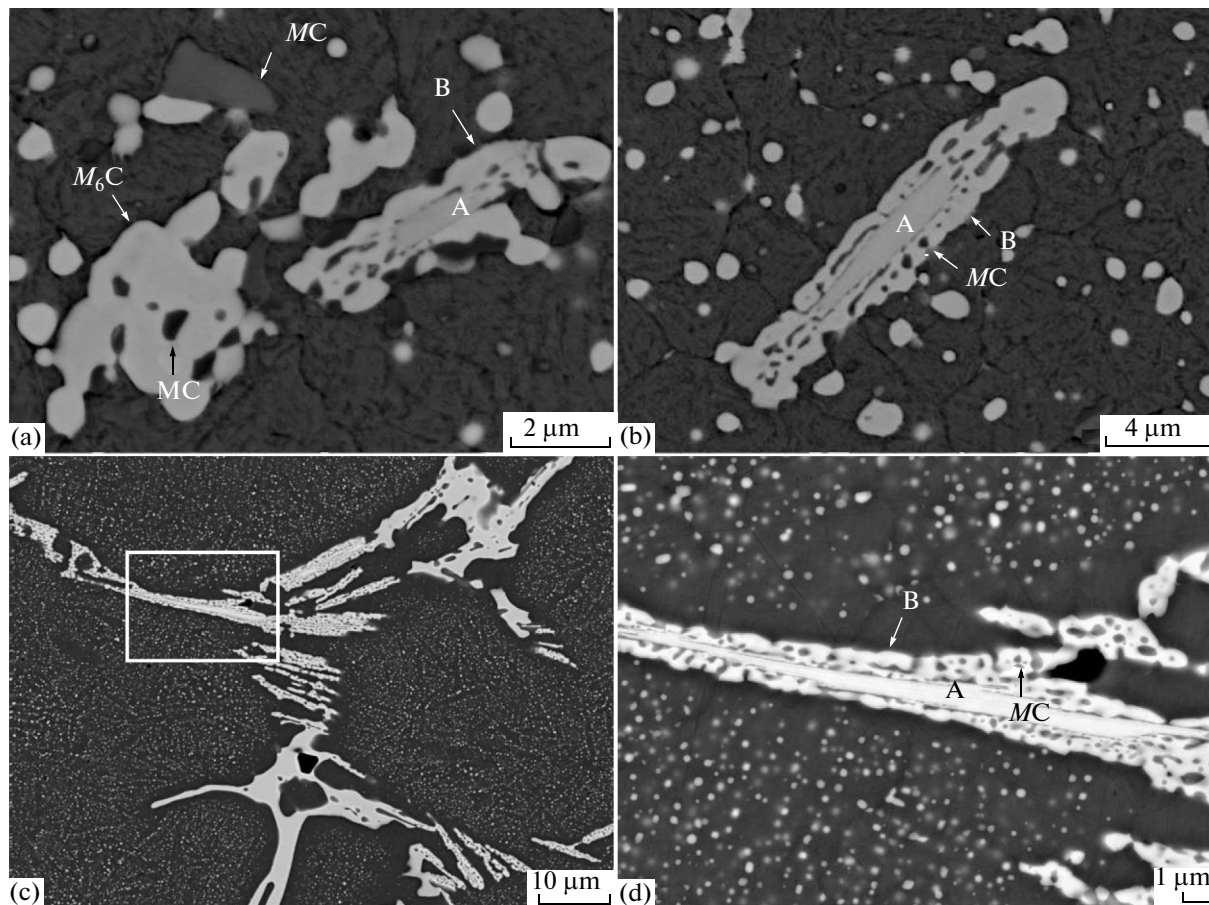


Fig. 6. Microstructure of (a, b) tempered wrought and (c, d) cast high-speed steel after austenitizing at (a, b) 1200 and (c, d) 1180°C; (d) fragment of the microstructure outlined in Fig. 6c, at a higher magnification. SEM. In Figs. 6a, 6b, and 6d, A indicates the central part of the particle, the chemical composition of which corresponds to the M_2C carbide and B indicates the peripheral part of the particle, the chemical composition of which corresponds to the M_6C carbide.

ture, the formation of such a distinctly pronounced mixture of M_6C and MC carbides does not occur upon the decomposition of the M_2C carbide as in the case of the cast steel with close chemical composition (see Fig. 2b), it can be assumed with high probability that the MC particles at high austenitizing temperatures already precipitate from the M_6C carbide, rather than from M_2C .

CONCLUSIONS

In this work, we investigated the structural and phase transformations in carbides of the deformed high-speed steel of R6M5 type after a complete cycle of heat treatment (quenching + triple tempering at 560°C), in which the austenitizing temperature was varied in the range of 1180–1260°C. The following results were obtained:

(1) An increase in the austenitizing temperature causes the coarsening of carbide particles of M_6C and MC types and a decrease in their total amount, which is confirmed by the results of statistical processing of

the microstructure parameters. The above-mentioned changes in the carbide constituent are the result of two simultaneous processes that occur upon the austenitization, i.e., the dissolution of the less stable carbide particles in the matrix and the coalescence and coarsening of the more stable particles.

(2) The presence of coarse particles of carbides of eutectic origin, which have a two-phase structure, is fixed in the microstructure of the tempered wrought steel. The chemical composition of the central part of such particles corresponds to the M_2C carbide; the chemical composition of their peripheral regions, where precipitates of particles of the MC carbide are seen, corresponds to the M_6C carbide. Moreover, small rounded particles that were apparently not completely dissolved during austenitizing were found, the vanadium content in which corresponds to the M_2C carbide. Both of these facts indicate the incomplete decomposition of the metastable carbide of M_2C type upon heating during hot plastic deformation and subsequent heat treatment of the high-speed steel. This has not been yet mentioned in the literature devoted to

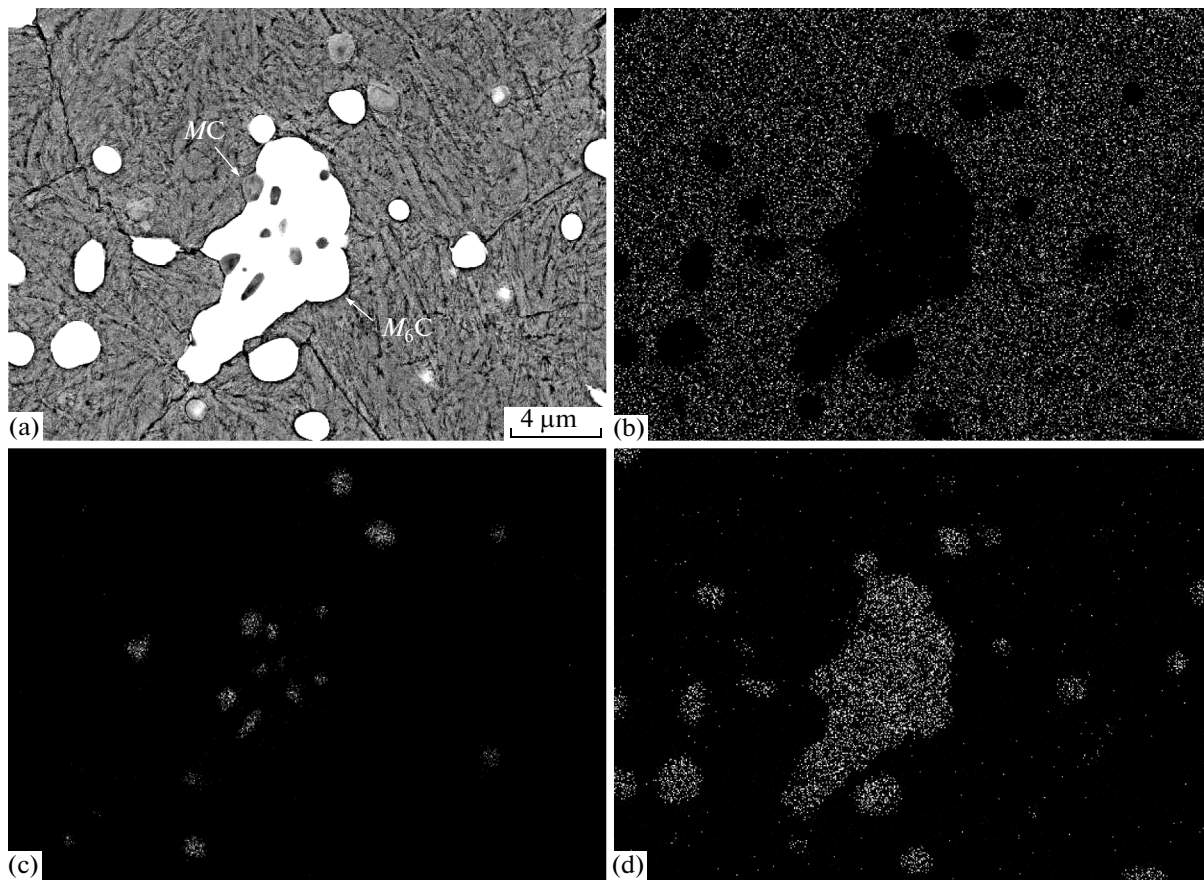


Fig. 7. (a) SEM image of the microstructure of the tempered wrought high-speed steel of R6M5 type after austenitizing at 1260°C; (b–d) distribution of alloying elements: (b) iron, (c) vanadium, and (d) molybdenum [23].

the final heat treatment of the wrought high-speed steel.

(3) Despite the similarity of changes in the initial microstructure and in the phase composition in the deformed and cast high-speed R6M5 steel under the action of the heat treatment, these changes are less pronounced in the case of deformed steel. In particular, in contrast to the cast steel, the formation of such a pronounced mixture of M_6C and MC carbides as the decomposition of M_2C carbide occurs in it. At a high austenitizing temperature, the MC particles presumably precipitate from the M_6C carbide rather than from M_2C .

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