The microstructure changes in high-speed steels during continuous heating from the as-quenched state

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Abstract

The paper presents the research of the influence of continuous heating from the as-quenched state (continuous tempering) on the microstructure of high-speed steels. Two widely known high speed steels HS18-0-1 and HS6-5-2 of a different content of W and Mo were used for the investigation. The results confirmed the following order of phase transitions during continuous tempering: ε carbide precipitation, cementite precipitation with a simultaneous transformation of a part of the retained austenite, independent nucleation of MC carbides followed by M_2C carbides nucleation. The precipitated MC carbides are stable in the steel microstructure and exhibit low susceptibility to coarsen, whereas the precipitated M₂C carbides transform "in situ" into M_6C and $M_{23}C_6$ carbides. The ε carbides precipitate on dislocations within the martensite volume, while cementite initially nucleates independently at the preferential sites, i.e. on twin boundaries and between martensite plates, absorbing carbon mainly from the retained austenite, which leads to its destabilization. Such microstructure is formed within the temperature range of 250–300 °C and its morphology is similar to that of upper bainite. It suggests that the precipitating cementite mainly causes temper brittleness of type I. It is additionally intensified by the transformation of a part of the retained austenite and dissolution of ε carbides.

Key words: high-speed steels, tempering, carbides, retained austenite, TEM

1. Introduction

High-speed steels are complex iron-base alloys containing carbon, tungsten, molybdenum, vanadium, chromium, and substantial amounts of cobalt [1, 2]. High speed steels owe their name to the ability of fast machining and cutting of various materials, among others, alloys with the iron matrix. Their typical as-quenched microstructure is a mixture of twinned plate martensite, retained austenite and undissolved carbides. Martensite is a very strong phase but normally very brittle. Thus, it is necessary to modify the material mechanical properties by tempering within the temperature range: 150–700 °C.

One of more important attributes of these steels are their cutting properties dependent on the wear resistance, fracture toughness, and the resistance to heat tempering. The wear resistance depends on the type, content and form of primary carbides (VC and M_6C , M = Mo, W, Cr, V), and matrix hardness. Whereas the fracture toughness is determined by the tempered matrix structure, grain size of former austenite, spatial arrangement and size distribution of primary carbides. High-speed steels matrix should consist of the high tempered martensite and the carbides causing its secondary hardness [2].

During heating of as-quenched martensite, the tempering (of unalloyed, medium and high carbon steels) takes place in three distinct but overlapping stages: the precipitation of ε (Fe_{2.4}C) carbide [3–8], transition of the retained austenite into lower bainite [9, 10] and precipitation of cementite [1, 2, 7, 11]. In steels containing alloying elements contributing to the secondary hardening effect (V, Mo, W), the fourth stage (transformation) occurs: precipitation of MC and M₂C type alloy carbides, that independently nucleate [2, 12, 13].

Typical applications of high-speed tool steels are

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Table 1. Chemical composition of the investigated steels

Element	${ m wt.\%}$		
	HS18-0-1	HS6-5-2	
С	0.85	0.85	
Mn	0.27	0.30	
Si	0.31	0.31	
Cr	4.26	4.14	
W	17.0	6.55	
Mo	0.50	4.61	
V	1.26	1.94	
Fe	bal.	bal.	

all types of cutting tools, such as drills, mills, milling cutters, metal saws, and also cold work tools such as die inserts, tread-rolling dies, punching and blanking dies. All these applications have in common a high number of loading cycles during service. At those cycles tools are exposed to stresses and strains, and in consequence, these tools fail not only due to a wear of the cutting edges but also due to – a mechanical and/or thermal – cyclic loading [14, 15].

Properties of high-speed steels can be modified by changes in their chemical composition, additions of carbide forming elements (e.g. Nb or Ti) and matrix strengthening elements [16–18].

This research was performed in order to optimize the technology of tempering tools made of high-speed steels as well as for designing the tempering technology for new tool steels.

2. Materials and experimental procedure

The research was conducted on HS18-0-1 and HS6--5-2 high-speed steels of the chemical composition given in Table 1. Prior to testing, the samples were soft annealed at $840 \,^{\circ}C/4$ h, and successively cooled at the rate of $6 \,^{\circ}C h^{-1}$ to $600 \,^{\circ}C$, and then furnace cooled.

On the basis of the previously performed dilatometric tests the temperature range for adequate phase transformations during continuous heating from the as-quenched state (tempering) was determined [19– 21].

Heat treatment procedures were performed using Adamel DT1000 dilatometer. Samples of diameter of 3 mm and height of 10 mm, after quenching from 1260 °C for HS18-0-1 and 1240 °C for HS6-5-2 (austenitizing time of 150 s, cooling rate $10 \,^{\circ}\text{C} \,^{\text{s}-1}$), were heated, with the heating rate of $0.05 \,^{\circ}\text{C} \,^{\text{s}-1}$, up to certain characteristic temperatures. Those were temperatures of the end of ε carbide precipitation (prior to the start of the retained austenite decomposition), end of the cementite precipitation, start of the alloy



Fig. 1. Microstructures of HS18-0-1 steel after quenching from 1260 $^{\circ}\mathrm{C}.$



Fig. 2. Microstructures of HS6-5-2 steel after quenching from 1240 $^{\circ}\mathrm{C}.$

carbides MC type precipitation and the end of M_2C type carbides precipitation. The microstructure of the samples was analysed by means of the JEM 200CX transmission electron microscope.

3. Results and discussion

The microstructure of HS18-0-1 steel (Fig. 1) in the as-quenched state consists of the partially twinned martensite, retained austenite (24 %) and M_6C carbides undissolved during austenitizing. Similarly, the structure of HS6-5-2 steel (Fig. 2) consists of the partially twinned martensite, retained austenite (app. 28 vol.%), and carbides undissolved during austenitizing, mainly M_6C and occasionally also MC.

Microstructures of the investigated steels quenched and then heated with a heating rate of $0.05 \,^{\circ}\text{C} \,^{\text{s}-1}$, are presented in Figs. 3 and 4. HS18-0-1 samples were continuously heated up to 200, 370, 560 and 790 $^{\circ}\text{C}$ while HS6-5-2 samples were continuously heated up to 210,



Fig. 3. Microstructures of HS18-0-1 steel after hardening from 1260 °C and heating with the rate 0.05 °C s⁻¹ up to: (a) 200 °C; (b) 370 °C; (c) 560 °C and (d, e) 790 °C; (f) dark field from the (022) reflex M₂₃C₆ carbide, dark field from the (0110) reflex M₂C carbides, diffraction pattern from the area as in Fig. 3e and solution of the diffraction pattern.

410, 560 and 790 °C. These are specific temperatures, at which according to [21], for the applied heating rate $(0.05 \,^{\circ}\mathrm{C}\,\mathrm{s}^{-1})$, the following phenomena were noticed: the end of ε carbide precipitation (before the start of the retained austenite transformation), the end of the cementite precipitation $(M_3C)_f$, the start of the MC alloy carbides precipitation. The presented microphotographs indicate a diversified rate of advancement of transformations during tempering, depending on the temperature that the quenched samples were heated

up to. During the continuous heating up to 790 °C the morphology of the primary carbides is not changed. Heating up to 200–210 °C caused the precipitation of ε carbide, which could be observed in Figs. 3a and 4a. Beside that, heating up to this temperature did not cause other noticeable changes in the structure of the investigated high-speed steels. On the contrary, heating up to $(M_3C)_f$ temperature caused some changes in the microstructure of investigated steels. TEM microphotographs displayed in Figs. 3b and 4b show martensite strips with cementite precipitations.





MGC

of martensite into ferrite accompanied by the precipitation of carbides. The carbides were revealed on TEM microphotographs for samples of the tested steel after tempering, and assumed to be of MC, M₂C, M₆C, and $M_{23}C_6$ type.

The sequence of carbides precipitation and temperature ranges of their occurrence during a continuous heating from the as-quenched state (with the rate $0.05 \,^{\circ}\mathrm{C}\,\mathrm{s}^{-1}$) are schematically presented in Table 2. This list was prepared on the bases of the given above results as well as the dilatometric tests given in the paper [21]. Unfortunately, the temperatures of the start of the carbides Mo and W, type M_2C precipitation, were not precisely determined in the steel.

Fig. 4. Microstructures of HS6-5-2 steel after hardening from 1240 °C and heating with the rate 0.05 °C s⁻¹ up to: (a) 210 °C; (b) 410 °C; (c) 560 °C; (d) 790 °C and (e) 790 °C bright field, dark field from the (022) reflex M₆C carbide; suitable diffraction pattern and solution of the diffraction

pattern.

Heating up to 560 °C initiated the dissolution of cementite and nucleation of MC type carbides, which is illustrated in Figs. 3c,d and 4c,d. In HS6-5-2 steel, after the sample heating up to 560 °C, numerous cementite precipitations were observed. The presence of cementite after that heat treatment (continuous heating of samples up to this temperature) seems to result in the initiation of the MC carbides nucleation. Moreover, in the microstructure of both investigated steels, the retained austenite was not observed. Probably a part of the austenite was transformed during tempering and another part during the samples preparation.

The heating up to $790 \,^{\circ}\mathrm{C}$ caused the transformation

Table 2. Types of carbides precipitated during continuous heating from as-quenched state of the investigated steels and the temperature ranges of their precipitation

	HS18-0-1	HS6-5-2	
Precipitation of ε (Fe _{2.4} C)	$70-200 ^{\circ}\mathrm{C}$	$60-210 ^{\circ}\mathrm{C}$	
Dissolving of ε , precipitation of M ₃ C	200-370 $^{\circ}\mathrm{C}$	210-410 $^{\circ}\mathrm{C}$	
Dissolving of M ₃ C, independent nucleation of MC	490-650 $^{\circ}\mathrm{C}$	540-660 $^{\circ}\mathrm{C}$	
Independent nucleation of M ₂ C	560 $^{\circ}\mathrm{C}$ <	560 $^{\circ}\mathrm{C}$ <	
Transformation "in situ" of M ₂ C into M ₆ C, M ₂₃ C ₆	650 $^{\circ}\mathrm{C}$ <	660 $^{\circ}\mathrm{C}$ <	



Fig. 5. Dependence of hardness of samples made of the tested steels on the heating temperature after quenching: (a) HS18-0-1, (b) HS6-5-2.

The hardness changes of the investigated steels samples, in dependence on the heating temperature after quenching, are presented in Fig. 5. As can be seen, the investigated steels exhibit the highest hardness in the quenched state. Heating the HS18-0-1 steel from the as-quenched state (Fig. 5a) to the temperature of $200 \,^{\circ}\text{C}$ – corresponding to the temperature of the end of the carbide ε precipitation – causes an insignificant hardness decrease from 853HV30 to 810HV30. Heating to 370 °C, i.e. to the temperature of the finish of the cementite precipitation, causes a further hardness decrease to 742HV30. The hardness similar to the one in the as-quenched state exhibited the sample heated to 560° C (840HV30), i.e. to the temperature from the range of the MC type carbides precipitation. The lowest hardness had the sample heated to 790 °C (412HV30), where the most probably the precipitation coherence of MC and M₂C carbides was ruptured and the transformation of M₂C carbides into M_6C and $M_{23}C_6$ type of carbides occurred. Similar characteristic exhibits HS6-5-2 steel (Fig. 5b), which in the as-quenched state has a slightly higher hardness (876HV30) than HS18-0-1 steel. After heating to 210 °C, corresponding to the temperature of the end of the carbide ε precipitation, the hardness decrease is larger than in HS18-0-1 steel (by 70 HV30 units). Heating to the temperature of the finish of the cementite precipitation in this steel $(410^{\circ}C)$ caused a further hardness decrease to 733HV30. Heating to a temperature being slightly above the temperature of the start of the MC carbides precipitation $(560 \,^{\circ}\text{C})$ caused the hardness increase to 773HV30, but since the precipitation process of the independently nucleating MC type carbides was at the very initial stage the hardness increase was quite small. Heating to a temperature of 790 °C caused the hardness decrease (400HV30), the most probably due to a rupture of the precipitation coherence of MC and M₂C carbides as well as their transformation into M₆C and M₂₃C₆ carbide types.

4. Conclusions

The results confirmed the following order of phase transformations during continuous heating from the as-quenched state:

1. Precipitation of ε carbide;

2. Precipitation of cementite with simultaneous transformation of the part of retained austenite;

3. Independent nucleation of MC carbides followed by the nucleation of M_2C carbides;

4. Transformation 'in situ' of the precipitated M_2C carbides into M_6C and $M_{23}C_6$ carbides. (The precipitated MC carbides are structurally stable and exhibit low susceptibility to coarsen.)

The ε carbides precipitate on the dislocations within the martensite volume, while the cementite initially nucleates independently at the preferential sites. It nucleates e.g. on twin boundaries and between martensite plates, absorbing the carbon mainly from the retained austenite what – in turn – leads to its destabilization. Such a structure is formed within the temperature range of 250-300 °C and its morphology is similar to that of upper bainite. It suggests that precipitating cementite causes mainly the temper brittleness of type I. An effect of lowering the fracture toughness within this temperature range (about 300° C) was observed by numerous authors. This effect is additionally intensified by the transformations of a part of the retained austenite and by dissolving of ε carbides. Basing on these observations one may conclude, that the temperature of the temper brittleness of type I occurrence is determined by the influence of the chemical composition on the range of ε carbide precipitation and on the stability of the retained austenite. Only heating up to the temperature of ε carbides dissolution allows cementite to nucleate independently within the whole volume of the material.

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