

The influence of kinetics of phase transformations during tempering on high-speed steels mechanical properties

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ABSTRACT

Purpose: The main purpose of the presented paper was the determination of the influence of the phase transformation kinetics, at tempering, on the properties of high-speed steels. In order to achieve this aim investigations of the influence of continuous heating and isothermal tempering from the as-quenched state on the investigated steels hardness were performed. The advancement degree change obtained by changing the heating rate and by pre-tempering was applied in strength properties studies.

Design/methodology/approach: Due to a high brittleness of tested steels it was decided to test their strength by means of the static bend test. The tests were carried out on 5 samples using the INSTRON testing machine. The microstructure of investigated steels in the as-quenched state was examined by the light microscope Axiovert 200 MAT and the scanning electron microscope Hitachi 3500 N. The measurements of hardness were performed with the Vickers HPO250 apparatus.

Findings: Changing heating rates for tempering one can influence steel properties. Better results are achieved when steels are heated up to the tempering temperature with the higher rate. The heating rate increased from 15°C/min to 300°C/min improved the investigated steels bending strength by 8%.

Research limitations/implications: Phase transformations at tempering, being diffusive transformations, are characterised by the determined kinetics depending on a temperature and time of heating. Complexity of processes occurring at tempering, as well as the diversity of influencing factors creates the possibility of searching for prolonging the life-span of tools made of high-speed steels. Regardless that only two, well known high-speed steels HS18-0-1 and HS6-5-2 were tested, the obtained results can be referred to the whole group of high-speed steels.

Practical implications: On the basis of the obtained results it is possible to select the tempering parameters in such a way as to achieve the optimal combination of strength and plastic properties needed for the anticipated application of the given high-speed steel.

Originality/value: This results should be of interest to engineers concerned with designing the new technologies of high-speed steels tempering

Keywords: Tool materials; Tempering; High-speed steel; Mechanical properties

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1. Introduction

Cutting tools are made of high-speed steels. They must be thermal tempering resistant, since during cutting a tool point can be heated up to a temperature of 650 °C. High-speed steels are very advanced materials on the iron-base matrix, in respect of their production as well as their chemical composition and the heat treatment.

High-speed steels are complex iron-base alloys of carbon, tungsten, molybdenum, vanadium, chromium, and substantial amounts of cobalt. Their typical as-quenched microstructure is a mixture of twinned plate martensite, retained austenite, and undissolved carbides. Martensite is a very strong phase but since it is normally very brittle, it is necessary to modify its mechanical properties by tempering in a temperature range of 150-700°C [1].

During heating as-quenched martensite, its tempering (of unalloyed, medium and high carbon steels) takes place in three distinct but overlapping stages: precipitation of ε carbide, transformation of the retained austenite into lower bainite and precipitation of cementite. In steels containing alloying elements contributing to the secondary hardening effect (V, Mo, W), a fourth stage (transformation) occurs: precipitation of MC and M₂C-type alloy carbides, that nucleate independently [1÷4].

The first transformation occurs in the temperature range of $100\div200^{\circ}$ C. Metastable ε carbide (Fe_{2.4}C) with a hexagonal crystal structure precipitates from the supersaturated martensite [5-8]. Precipitation of highly dispersed ε carbide is believed to enhance strengthening in steels [2,4].

The second transformation, proceeding during tempering in the temperature range of 200-320°C, is a transformation of the retained austenite. As a result of this transformation a nonhomogeneous mixture consisting of supersaturated ferrite and cementite, i.e. lower bainite, forms [1,2,4].

Cementite is formed during the third transformation occurring during tempering in the temperature range of 200-420°C. This results in further decarbonization of the matrix and dissolution of metastable ε carbides allowing for the steel matrix recovery [4,7]. However, the mechanism of the nucleation of cementite is not fully understood yet. According to Ref. [4] the precipitating cementite nucleates independently or "in situ" on ε carbide particles. Whereas according to Ref. [5] cementite nucleates independently, mainly on grain boundaries of the former austenite or on subgrain boundaries of the newly formed cell structure.

Above 400°C, diffusion of alloying elements such as V, Mo and W takes place. Then, cementite gradually dissolves to make the nucleation of MC and M_2C carbides coherent with the alloy matrix possible. This leads to an increase of hardness of the tempered steel and is thus referred to as the secondary hardening [1-4,10-17].

Microstructure of the correctly heat treated high-speed steel should consist of a hard and homogeneous matrix with a high volume fraction of fine and uniformly distributed carbides both the undissolved during austenitizing and being formed during tempering. The main objective of the research presented in this paper was the evaluation of the influence of the phase transformation kinetics during tempering on the properties of high-speed steels.

2. Test material

The research was conducted on HS18-0-1 and HS6-5-2 highspeed steels of the chemical compositions given in Table 1.

Table 1.

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Grade	mass %							
	С	Mn	Si	Cr	Mo	W	V	
HS18-0-1	0.85	0.27	0.31	4.26	0.50	17.0	1.26	
HS6-5-2	0.85	0.30	0.31	4.14	4.61	6.55	1.94	

Prior to testing, the samples of the investigated steels were soft annealed at 840° C/4hours, and successively cooled at the rate of 6°C/hour to 600°C, and after that to the room temperature together with the furnace.

3. Experimental procedure

The microstructure of the investigated steels were examined by the light microscope Axiovert 200 MAT, and the scanning electron microscope Hitachi 3500N.

The measurements of hardness were performed with using the Vickers HPO250 apparatus.

Due to high brittleness of the tested steels it was decided to test their strength by means of the static bend test. The tests were carried out on $\phi 5$ samples using the INSTRON testing machine.

4. Research results and discussion

In the as-quenched state the microstructure of HS18-0-1 steel (Fig. 1) consists of partially twinned martensite, retained austenite (in amount of 24.4%) and M_6C carbides undissolved during austenitizing [14]. Similarly, the microstructure of HS6-5-2 steel according to [14] consists of partially twinned martensite, retained austenite (in amount of about 27.9% vol.), and carbides undissolved during austenitizing, mainly M_6C and occasionally MC [12-14].

Fig.2 presents the HS18-0-1 steel dilatograms of heating from the as-quenched state, together with corresponding differential curves, on which temperatures of the beginning (letter s) and the end (letter f) of individual transformations are marked. Detailed description of phase transformations kinetics during tempering of HS18-0-1 steel in a form of the CHT (Continuous Heating Transformation) diagram and change in the structure as a result of continuous heating from the as-quenched state are presented in several studies [12,14,16,17].

Continuously tempered HS18-0-1 steel first exhibits the contraction related to the precipitation of ε carbide. The contraction starts at the temperature ε_s and ends at the temperature ε_f . For the heating rate of 0.05°C/s, the beginning of ε carbides precipitation takes place at a temperature of about 80°C, which increases along with the heating rate increase up to about 150°C (35°C/s). The temperature ε_f approximately equals to the temperature of the beginning of the cementite precipitation



Fig. 1. Microstructures of the investigated steels after quenching: a,b) Light microscope; c,d) Scanning microscope, nital etched

 $(M_3C)_{s}$, therefore it was assumed that they are equal to each other. Within the temperature range: $(M_3C)_s \div (M_3C)_f$ the cementite precipitates. Within this range, a positive dilatation effect connected with the transformation of a part of the retained austenite is observed. The effect is visible in the temperature range: $RA_s \div RA_f$. For the heating rate of $0.05^\circ C/s$ $RA_s = 220^\circ C$ while for 35°C/s $RA_s = 340^\circ C$, whereas RA_f increases from 320°C (for $0.05^\circ C/s$) up to 440°C (for 35°C/s). The temperature at which the cementite precipitation occurs increases from 480°C ($0.05^\circ C/s$) to 550°C (35°C/s). At the MC_s temperature the independent nucleation of carbides of MC type begins. At the temperature ($M_2C)_s = MC_f$ the independent nucleation of carbides of M_2C type begins. This temperature was determined for the heating rate of $0.05^\circ C/s$ only. An application of higher heating rates elevates this temperature to values exceeding 700°C.

It can be noticed, that the heating rate increase from 0.05°C/s to 35°C/s results in the increase of temperatures of the beginnings and ends of individual transformations and in the reduction of dilatation effects which accompany these transformations [16].

Fig.3 presents dilatograms of HS6-5-2 steel heated from the as-quenched state, together with corresponding differential

curves. Detailed description of the phase transformation kinetics during tempering of HS6-5-2 steel in a form of the CHT diagram and the changes in structure as a result of the continuous heating from the as-quenched state are presented in papers [13,14].

As it can be noticed, during heating from the as-quenched state in HS6-5-2 steel, at first there is a contraction related to the precipitation of ε carbide. The temperature ε_s for the heating rate of 0.05°C/s is about 50°C and for 35°C/s is about 140°C, whereas ε_f increases from 210°C to 290°C. The temperature ε_f approximately equals to the temperature (M₃C)_s, at which cementite begins to precipitate. Within the temperature range of RA_s-RA_f a part of the retained austenite is transformed. It should be noticed that a positive dilatation effect from this transformation is less intensive in comparison with the previously discussed HS18-0-1 steel, yet it spreads out over almost the whole range of contraction resulting from the cementite precipitation (it significantly reduces the effect of contraction) [14].

Comparing the heating dilatograms from the as-quenched state for the two tested high-speed steels one may notice, that the substitution of tungsten with molybdenum in HS6-5-2 steel has influenced mainly the stability of the retained austenite and the

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temperature of the precipitation beginning of MC (MC_s) type carbides. Whereas the precipitation ranges of ϵ carbide and cementite in both steels are close. The heating rate influence on the MC_s temperature, which increases from 530°C for the heating rate of 0.05°C/s to above 700°C for 35°C/s, is stronger in HS6-5-2 steel. Thus the difference is about 170°C. In HS18-0-1 steel at the heating rate of 0.05°C/s the alloy carbides of MC type begin to precipitate already at 490°C while at the heating rate of 35°C/s at 600°C[16].

7 MC 16 (M₃C)_f 6 Δl/lo x 10⁻³ 5 RA $\varepsilon_{f} = (M_{3}C)$ Δl/loΔT x10⁻⁶ 4 Es. $MC_f = (M_2C)$ 3 10 2 100 200 300 400 600 700 Temperature, °C 500 a) $\varepsilon_{f} = (M_{3}C)_{s}$ Es 12 6 MC. <u>М/IoAT x10⁻⁶, [/°C]</u> RA $(M_3C)_f$ Δl/lo x 10⁻³ RAs 3 300 400 500 600 700 Temperature, °C b)

Fig. 2. Dilatograms of HS18-0-1 steel heated from the asquenched state at the rate of: a) 0.05° C/s, b) 35° C/s [16]

Figs. 4 and 5 show the CHT diagrams for the investigated steels. The diagrams contain the ranges of the precipitation of ε carbide, transformations of retained austenite, precipitation of cementite and alloy carbides of MC and M₂C type [12,13,16].

Influences of the heating rate from the as-quenched state to 700°C on hardness of the investigated steels are shown in Fig. 9 and 10. As can be seen, for low heating rates $(0.05^{\circ}C/s \text{ and } 0.1^{\circ}C/s)$ hardness is clearly smaller, which confirms a greater contribution of phase transformations during tempering.



Fig. 3. Dilatograms of HS6-5-2 steel heated from the as-quenched state at the rate of: a) 0.05°C/s, b) 35°C/s [16]



Fig. 4. CHT diagram of HS18-0-1 steel [12]



Fig. 5. CHT diagram of HS6-5-2 steel [13]



Fig. 6. Influence of the heating rate from the as-quenched state on hardness of HS18-0-1 steel

Application of the heating rate higher than 0.5° C/s does not cause much changes in hardness of the investigated steel, though an increase in hardness with increased heating rate can be noticed, what is believed to be the result of a smaller degree of phase transformations.



Fig. 7. Influence of the heating rate from the as-quenched state on hardness of HS6-5-2 steel

The hardness change of the investigated steels samples, in dependence on the heating temperature after quenching, are presented in Fig. 8. As can be seen, the investigated steels exhibit the highest hardness in the as-quenched state. Heating the HS18-0-1 steel from the as-quenched state (Fig. 8a) to a temperature of 200°C - corresponding to the temperature of the finish 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 exhibited 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 M_2C carbides into M_6C and $M_{23}C_6$ type of carbides occurred. Similar characteristic exhibits HS6-5-2 steel (Fig. 8b), 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 finish 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°C), caused a further hardness decrease (to 733HV30). Heating to a temperature being slightly above the temperature of the beginning of the MC carbides precipitation (560 °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 this hardness increase was quite small. Heating to a temperature of 790°C caused the hardness decrease (400HV30), the most probably due to the rupture of the precipitation coherence of MC and M2C carbides as well as their transformation into M_6C and $M_{23}C_6$ carbide types.

Changes of the investigated steels hardness caused by isothermal tempering (holding) for 8 hours are shown in Fig. 9. As can be seen the hardness changes in both high-speed steels are nearly identical (Fig. 9a and b). After tempering at 100° C hardness slightly decreases as compared to the as-quenched state. In a similar fashion, as at a continuous heating from the as-quenched state, tempering of HS6-5-2 steel in the range of the ϵ carbide precipitation causes a more significant hardness decrease than tempering of HS18-0-1 steel.



Fig. 8. Dependence of hardness of samples made of the tested steels on the heating temperature after quenching: a) HS18-0-1; b) HS6-5-2

Tempering at 200 and 300°C, it means within the ε carbide precipitation range and its transformation into cementite, causes a further hardness decrease and hardness of both steels are similar (app. 700HV30). After tempering at 400°C hardness of HS18-0-1 steel slightly increases as compared to a temperature of 300°C, which is related to the beginning of the retained austenite transformation. On the other hand, tempering at 400°C causes in HS6-5-2 steel a further insignificant hardness decrease, which confirms the previous observations of the higher stability of the retained austenite in this steel. Tempering at 500°C causes a hardness increase related to the retained austenite transformation in both steels. Tempering at 600°C causes a hardness decrease due to the rupture of the MC carbides precipitation coherence, advanced M₂C precipitation, their increase and eventual transformation into the M_6C and $M_{23}C_6$ types carbides. After tempering at 700°C hardness of both steels rapidly decreases to app. 350HV30.

An influence of the heating rate for tempering (at a temperature of 580°C for 1 hour) on bending strength of the investigated high-speed steels is illustrated in Fig. 10. As it is seen, much better results are obtained when the higher heating rate is applied. The bending strength difference is app. 200MPa and 400MPa for HS18-0-1 and HS6-5-2 steels, respectively. These results constitute a good prognosis in searching for new

technologies of steel tempering, based on knowledge of their transformations (kinetics) occurring during heating from the asquenched state.



Fig. 9. Influence of 8 hours of isothermal tempering on the investigated steels hardness: a) HS18-0-1; b) HS6-5-2



Fig. 10. Influence of the heating rate - for tempering - on bending strength of the investigated high-speed steels (tempering time 1 hour)

The influence of pre-tempering (advancement of the selected transformations) on the final bending strength and hardness of the investigated steels is shown in Fig. 11 and 12. Temperatures and time (1.5h) of pre-tempering were selected on the bases of the previously performed investigations of high-speed steels [14]. Thus, pre-tempering was performed at the following

temperatures: 250°C - it means slightly above the temperature of the finish of the ε carbide precipitation and the start of the cementite precipitation, 450°C - it means after finishing the cementite precipitation and transformation of the retained austenite part, 520°C - for HS18-0-1 and 550°C for HS6-5-2 - it means slightly above the temperature of the precipitation start of independently nucleating MC-type carbides.

All applied variants of pre-tempering caused decreases of bending strength and hardness of HS18-0-1 steel (Fig. 11) as compared to the standard tempering: $3x580^{\circ}$ C/1h. In the case of pre-tempering at 250°C, the temperature was sufficiently high for starting the cementite precipitation and for lowering the retained austenite stability before the main tempering. As a result the standard tempering process ($3x580^{\circ}$ C/1h) was facilitated. Thus, it is obvious that pre-tempering at 250°C causing the pre-advanced cementite precipitation decreases the final (after the main tempering) hardness and strength. Maybe pre-tempering of this steel should be carried out at a temperature enabling the precipitation of ε carbide alone, which means not exceeding 200°C.

Pre-tempering at 450°C (Fig. 11) caused even larger drop of bending strength at slightly smaller hardness decrease than the standard tempering (3x580°C/1h). At 450°C a transformation of the retained austenite is already possible due to which the matrix tempering process is facilitated.



Fig. 11. Influence of pre-tempering on bending strength and hardness of HS18-0-1 steel

Whereas, an advancement of the cementite precipitation suppressed the hardness decrease during the main tempering at 580°C since cementite had - at first - to dissolve to enable the precipitation of alloy carbides, which are causing the secondary hardness effect

After pre-tempering at 520°C the part of the retained austenite underwent the transformation and the precipitation of the alloy carbides of MC-type was advanced. Consequently, after the main tempering hardness decreased significantly, while the bending strength decrease was stopped (Fig. 11).

Fig. 12 presents the influence of pre-tempering on bending strength and hardness of HS6-5-2 steel after the main tempering. The temperatures and duration (1.5h) of the pre-tempering were selected on the basis of previous research [13,14,17].

During pre-tempering at 250°C mainly the ε carbide has been advanced. Negligible cementite precipitates on a boundary between martensite and austenite did not cause a decrease of the carbon content in austenite and, in result of it, during the main tempering the austenite was acting like there had been no pretempering, that is why the bending strength had not changed. However, the precipitation of ε carbides (mainly) has resulted in a more uniform distribution of carbon after their solubilisation inhibiting the precipitation of cementite and, by it, the creation of more favourable conditions for independent precipitation within the whole volume of steel. Hence the sample hardness after pretempering at 250°C is higher by about 25HV30. It confirms the above stated suggestion that on the way of advancing of ε carbide precipitation (exclusively) one may increase the hardness of steel after the main tempering.

Similarly, the obtained bending strength and hardness after pre-tempering at 450°C, can be explained. They are almost identical as the ones after pre-tempering at 250°C. Most probably it is a result of a higher stabilization of the retained austenite and a distinct decrease of advancement of the cementite precipitation in HS6-5-2 steel as compared to HS18-0-1 steel [12,14,16].

Pre-tempering at 550°C started the transformation of the retained austenite and caused the advancement of the cementite precipitation, also initialising the precipitation of MC carbides. Most probably it has postponed the precipitation of MC carbides during the main tempering resulting in distinctly higher hardness of the steel if compared to standard tempering. Whereas a decrease of strength is most probably connected with the transformation of a part of the retained austenite during pre-tempering.



Fig. 12. Influence of pre-tempering on bending strength and hardness of HS6-5-2 steel

5. Conclusions

Heating of the investigated steels from the as-quenched state resulted in the occurrence of four primary transformations: precipitation of ϵ carbide, precipitation of M₃C, retained austenite transformation and precipitation of alloy carbides of MC and M₂C type.

The substitution of tungsten with molybdenum in HS6-5-2 steel influenced mainly the stability of the retained austenite and the temperature of the precipitation beginning of MC (MC_s) type carbides. Whereas the precipitation ranges of ε carbide and cementite in both steels are close to each other.

Investigations of the influence of the heating rate from the asquenched state to 700°C on the steel hardness indicate, that an application of the higher than 0.5°C/s rate does not cause much changes in the investigated steel hardness. The isothermal tempering in the temperature range of ε carbide precipitation does not decrease significantly the investigated steels hardness. More serious hardness decrease is caused by isothermal holding at the temperature range of the cementite precipitation beginning. The isothermal tempering temperature increase within the range of the cementite precipitation and the retained austenite transformation does not significantly decrease hardness due to the retained austenite transformation. Tempering in the range of the secondary carbide precipitation increases hardness. A further increase of the isothermal tempering temperature causes, in a similar fashion as at continuous heating, the hardness decrease.

It is possible to influence the steel properties by changing the heating rate for tempering. Better results are obtained when heating to the tempering temperature is carried out with a higher rate. The increase of the heating rate from 15°C/min to 300°C/min increased bending strength of the investigated high-speed steels by 8%.

An advancement (by pre-tempering) of the selected phase transformations at tempering, can influence steel properties after the main tempering. A favourable influence of pre-tempering on ε carbides precipitation was found.

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