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The kinetics of phase transformations during continuous heating from as-quenched state in high-speed steels

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ABSTRACT

Purpose: The reason for writing this paper was to describe the kinetics of phase transformations during continuous heating from quenched state. In this investigation two high-speed steels of the variable concentration of W and Mo were used. Moreover, the differences in hardness of samples of the investigated steels in relationship to the heating rate were evaluated.

Design/methodology/approach: The kinetics of phase transformations during continuous heating (tempering) from as-quenched state of investigated steels, was elaborated using a DT 1000 dilatometer of a French company Adamel. The microstructure of investigations steels in as-quenched state were examined by a light microscope Axiovert 200 MAT, scanning electron microscope Hitachi 3500N and transmission JEM200CX microscope. The measurements of hardness were performed with the Vickers HPO250 apparatus.

Findings: Change of heating rate during tempering has strong influence on the temperatures of beginnings and the ends of individual transitions as well as on the accompanying dilatation effects. It was shown that in quenched high-speed steels a part of retained austenite was already transforming during heating for tempering, but its significant part transformed only during cooling process after tempering.

Research limitations/implications: It has been found that the substitution of tungsten with molybdenum in HS6-5-2 steel has influenced mainly the stability of retained austenite and the temperature of precipitation beginning of MC (MCs) type carbides. Whereas the precipitation ranges of ε carbide and cementite in both steels are close.

Practical implications: Description of phase transformations kinetics during continuous heating from as-quenched state in high-speed steels.

Originality/value: This results should be of interest to engineers concerned with design new technologies of steel tempering.

Keywords: Tool materials; Tempering; High-speed steel; Retained austenite

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MATERIALS

1. Introduction

High speed steels are complex iron-base alloys of carbon, tungsten, molybdenum, vanadium, chromium, and substantial amounts of cobalt. Their typical as-quenched structure is a mixture of twinned plate martensite, retained austenite, and undissolved carbides. Martensite is a very strong phase but it is normally very brittle so it is necessary to modify the mechanical properties by heat treatment in the range 150-700°C, which is called tempering. This is one of the oldest heat treatments applied to steels [1].

During heating as-quenched martensite, the tempering (of unalloyed, medium and high carbon steels) takes place in three distinct but overlapping stages: precipitation of ε carbide, transformation of retained austenite into lower bainite and precipitation of cementite. In steels containing alloying elements contributing to an effect of secondary hardening (V, Mo, W), a fourth stage (transformation) occurs: precipitation of MC and M_2C-type alloy carbides, that nucleate independently [1-4].

The first transformation in the temperature range of 100-200°C. Metastable ε carbide ($Fe_{2.4}C$) with hexagonal crystal structure precipitates from the supersaturated martensite [5,6]. Precipitation of highly dispersed ε carbide is believed to enhance strengthening in steel [2,4,7,8].

Second transformation proceeding during tempering in the temperature range of 200-320°C is a transformation of retained austenite. As a result of this transformation, a non-homogeneous 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 recovery of the steel matrix [4,9]. The mechanism of the nucleation of cementite is, however, not fully understood yet. According to ref. [4] precipitating cementite nucleates independently or “in situ” on ε carbide particles. Whereas according to ref. [5] cementite nucleates independently, mainly on grain boundaries of former austenite or on subgrain boundaries of newly formed cell structure.

Above 400°C, diffusion of alloying elements such as V, Mo and W takes place. Then, the 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 tempered steel and is thus referred to as secondary hardening [1-4,10-16].

A majority of cited above investigations concerning transformations occurring during tempering were performed on samples tempered at the specific temperature during specific time. However, there is a lack of investigations pertaining to phase transformations occurring during continuous tempering on heating.

2. Test material

The research was conducted on HS18-0-1 and HS6-5-2 high speed steels with the chemical compositions is given in Table 1.

Table 1.
Chemical composition of the investigated steels

Grade	mass %						
	C	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 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 kinetics of phase transformations during continuous heating (tempering) from as-quenched state of investigated steels, was elaborated using a DT 1000 dilatometer of a French company Adamel. Samples with a diameter of 2 mm and a height of 12 mm, after quenching from 1260°C for HS18-0-1 and 1240°C for HS6-5-2 (austenitizing time of 150 s), were heated to 700°C with a heating rate in the range of 0.05 to 35°C/s. Digitally recorded dilatograms (engineering strain elongation $\Delta l/l_o$ in relation to the temperature T) for heated samples were differentiated, what facilitated determination of the start and end temperatures of consecutive transformations.

The microstructure of investigated steels were examined by a light microscope Axiovert 200 MAT, scanning electron microscope Hitachi 3500N and transmission JEM200CX microscope.

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

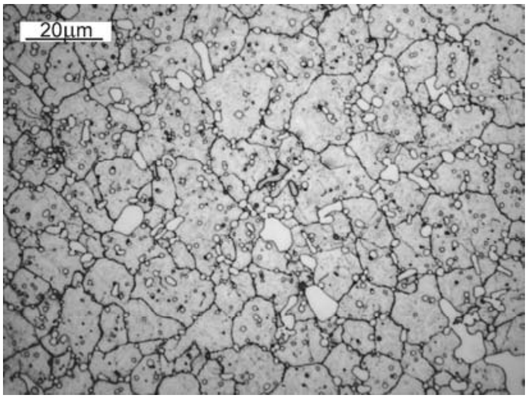
4. Research results and discussion

In as-quenched state the structure of HS18-0-1 steel (Fig. 1) consists of [eg 14] partially twinned martensite, retained austenite in amount of 24.4% and M_6C carbides undissolved during austenitizing. Similarly, the structure of HS6-5-2 steel (Fig. 2) 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 present MC. During heating from as-quenched state up to 700°C the morphology of primary carbides is not changed [12-14].

Fig. 3 presents HS18-0-1 steel dilatograms of heating from quenched state, together with corresponding differential curves, on which there are marked the temperatures of the beginning (letter s) and the end (letter f) of individual transition. Detailed description of phase transitions kinetics during tempering of HS18-0-1 steel in a form of CHT (Continuous Heating Transformations) diagram and change in the structure as a result of continuous heating from quenched state are present in studies [12,14].

Continuously tempered HS18-0-1 steel first exhibits the contraction related to precipitation of carbide ε. The contraction starts at temperature ε_s and ends at temperature ε_f. For the heating rate of 0.05°C/s, the beginning of ε carbides precipitation takes place at the 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 cementite precipitation $(M_3C)_s$, therefore it was assumed that they are equal to each other. Within the range of temperature $(M_3C)_s$ - $(M_3C)_f$ the cementite precipitates. Within this range, there is observed a positive dilatation effect connected with the transition of a part of retained austenite. The effect is visible within the temperature range RA_s - RA_f. For the heating rate of 0.05°C/s RA_s = 220°C while for 35°C/s RA_s = 340°C, whereas RA_f increases from 320°C (for 0.05°C/s) up to 440°C (for 35°C/s). The temperature at which precipitation of cementite caused increases from 480°C (0.05°C/s) to 550°C (35°C/s). At the MC_s temperature the independent nucleation of carbides of MC type begins. At temperature $(M_2C)_s$ = MC_f the independent nucleation of carbides of M_2C type begins. This temperature has been determined for the heating rate of 0.05°C/s only. Application of higher heating rates elevates this temperature to values higher than 700°C. It can be noticed, that increase of heating rate from 0.05°C/s to 35°C/s results in the increase of temperatures of beginnings and the ends of individual transitions and in reduction of dilatation effects which accompany these transitions [16].

a)



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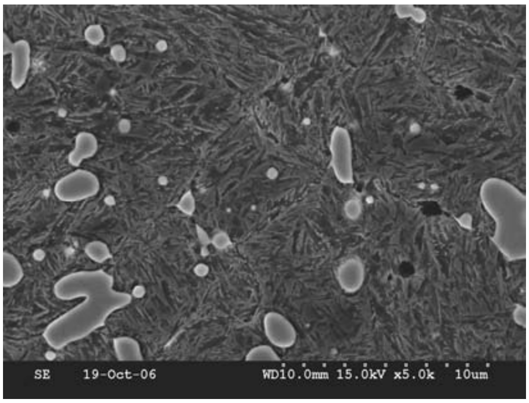
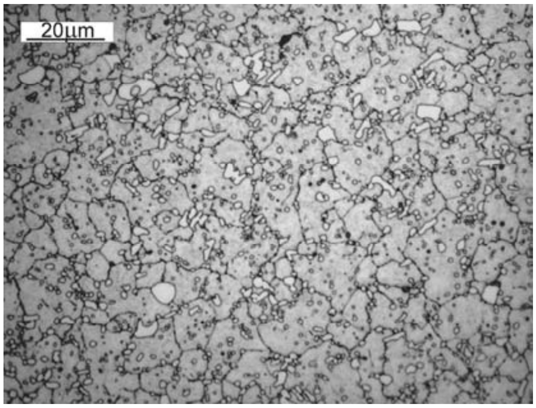


Fig. 1. Microstructures of HS18–0–1 steel after quenching from 1260°C: a) light microscope, b) scanning microscope, nital etched

a)



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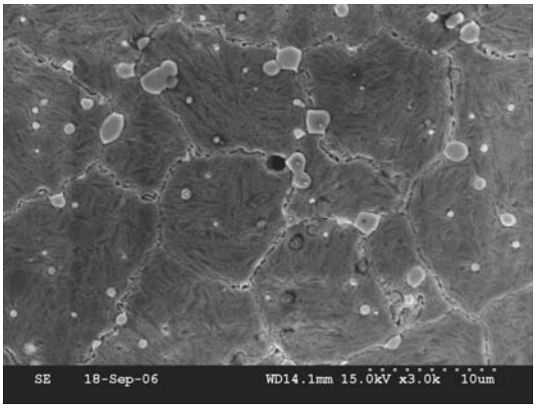

Fig. 2. Microstructures of HS6–5–2 steel samples after quenching from 1240°C: a) light microscope, b) scanning microscope, nital etched

Fig. 4 presents cooling dilatograms at the rate of 1°/s of HS18–0–1 steel samples previously heated to 700°C at the rate of: a) 0.05°/s, b) 35°/s. A positive dilatation effect from transition of retained austenite for previously heated sample (tempered) at the rate of 0.05°C/s is lower but more spread out over the temperature axis than for the sample previously heated (tempered) at the rate of 35°C/s. It is most probably related to more considerable diffusion of carbon and alloy elements from austenite (retained) to the boundary austenite – martensite (ferrite) at lower rate (0.05°C/s) of heating to 700°C and to transition of its larger amount during heating process (tempering). This fact is confirmed by lowering by about 75°C of temperature M_s with the increase of rate of previous heating to 700°C, from 400°C for $V_{heat.}$ = 0.05°C/s to 325°C for $V_{heat.}$ = 35°C/s.

Fig. 5 presents dilatograms of HS6–5–2 steel heated from quenched state, together with corresponding differential curves. Detailed description of phase transitions kinetics during tempering of HS6-5-2 steel in a form of CHT diagram and the changes in structure as a result of continuous heating from quenched state are presented in studies [13,14].

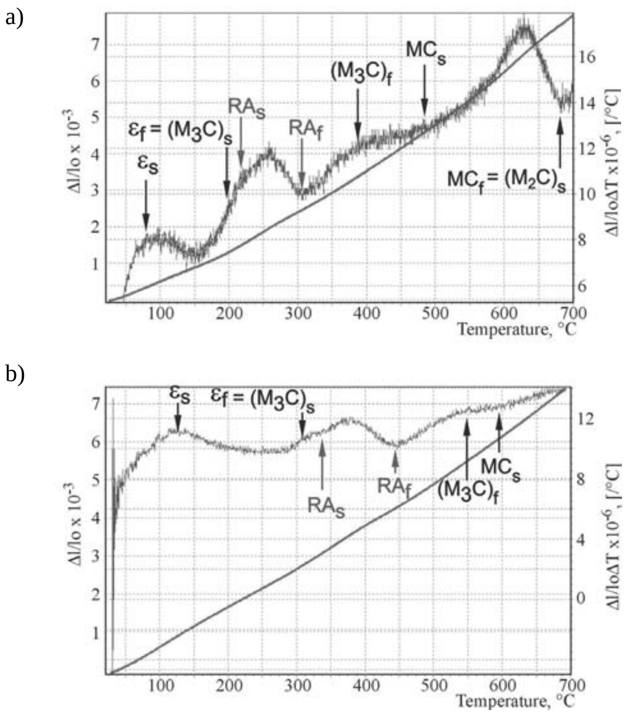

Fig. 3. Dilatograms of HS18-0-1 steel heated from quenched state at the rate of: a) 0.05°C/s, b) 35°C/s

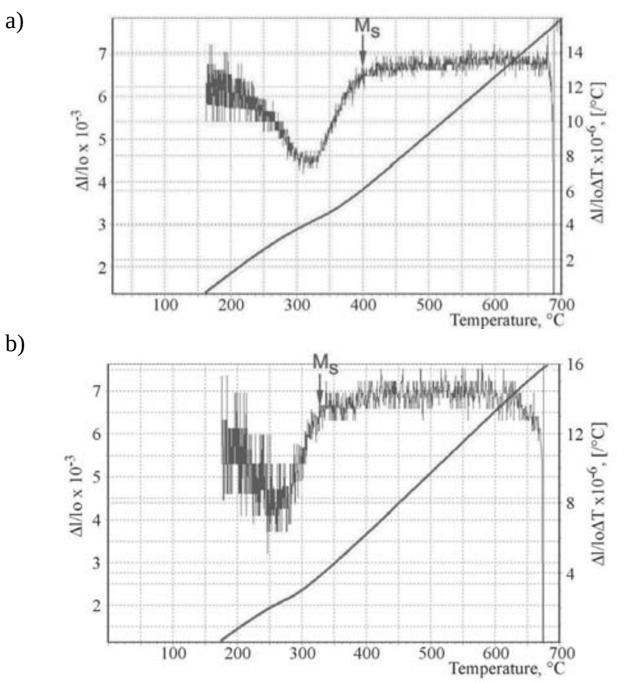

Fig. 4. Dilatograms of cooling at the rate of 1°C/s of HS18-0-1 steel, together with corresponding differential curves. First samples was heated from quenched state to 700°C at the rate of: a) 0.05°C/s, b) 35°C/s

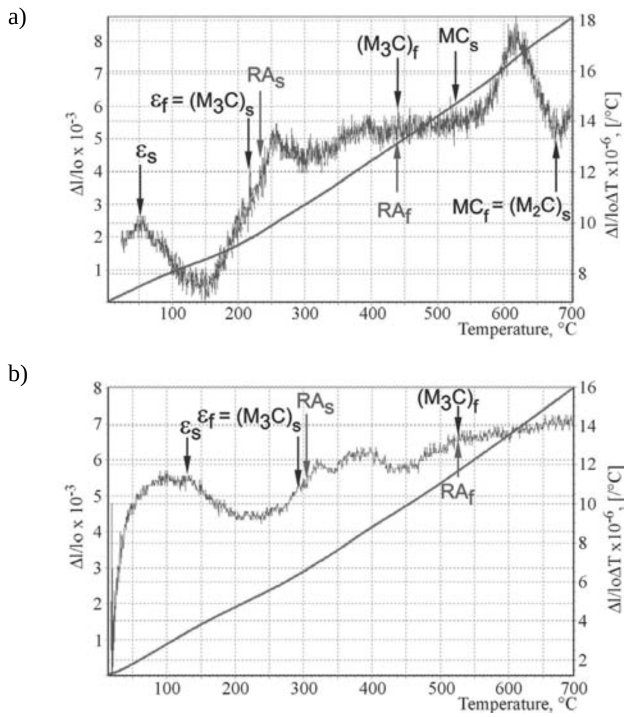

Fig. 5. Dilatograms of HS6-5-2 steel heated from quenched state at the rate of: a) 0.05°C/s, b) 35°C/s

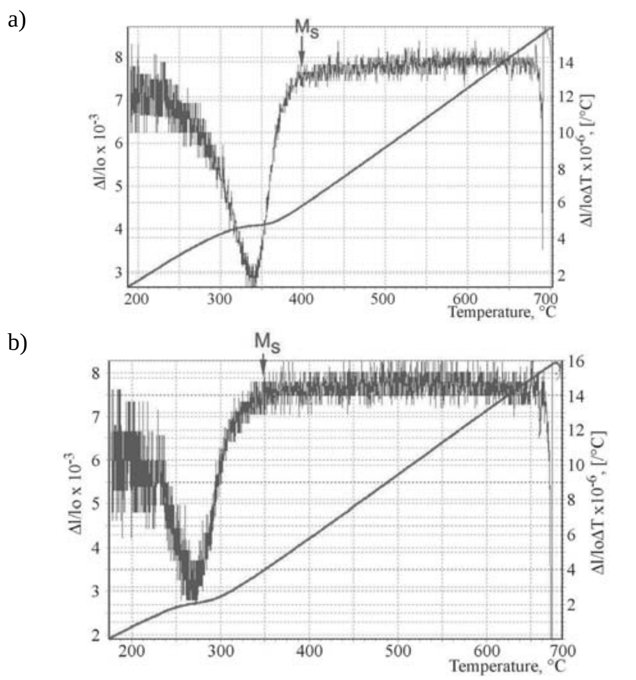

Fig. 6. Dilatograms of cooling at the rate of 1°C/s of HS6-5-2 steel, together with corresponding differential curves. First samples was heated from quenched state to 700°C at the rate of: a) 0.05°C/s, b) 35°C/s

As it can be noticed, during heating from quenched state in HS6-5-2 steel first there is a contraction related to the precipitation of ε carbide. The temperature ε_s for 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 $(M_3C)_s$ at which the 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 transition is lower (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 cementite precipitation (it significantly reduces the effect of contraction resulting from the precipitation of this carbide). It can be noticed, in cooling dilatograms after heating (tempering) to 700°C (Fig. 6), that by far greater part of retained austenite is transformed only by cooling after tempering (heating to 700°C). The decrease of M_s temperature that was found during cooling with the increase of rate of first heating (tempering) to 700°C, from 400°C for $V_{heat.}$ = 0.05°C/s to about 350°C for $V_{heat..}$ = 35°C/s (i.e. about 50°C) is smaller than for HS18-0-1 steel, what also indicates that during heating to 700°C the chemical destabilization of retained austenite in HS6-5-2 steel takes place to a greater degree than in HS18-0-1 steel. It may be connected with higher diffusion coefficient of Mo than W and with higher diffusion coefficients of other elements (including carbon) in the presence of Mo than in the presence of W. This is indicated by so called temperability of matrix standard alloys of quenched high-speed steels with diversified of Mo and W content, described among others in work [14].

Comparing the heating dilatograms from quenched state for the two test high-speed steels one may notice that the substitution of tungsten with molybdenum in HS6-5-2 steel has influenced mainly the stability of retained austenite and the temperature of precipitation beginning of MC (MC_s) type carbides. Whereas the precipitation ranges of ε carbide and cementite in both steels are close. The influence of heating rate on 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 indicated 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 from 600°C.

Figs. 7 and 8 show a CHT diagrams for investigated steels. The diagrams contains the ranges of precipitation of ε carbide, transformations of retained austenite, precipitation of cementite and alloy carbides of MC and M_2C type.

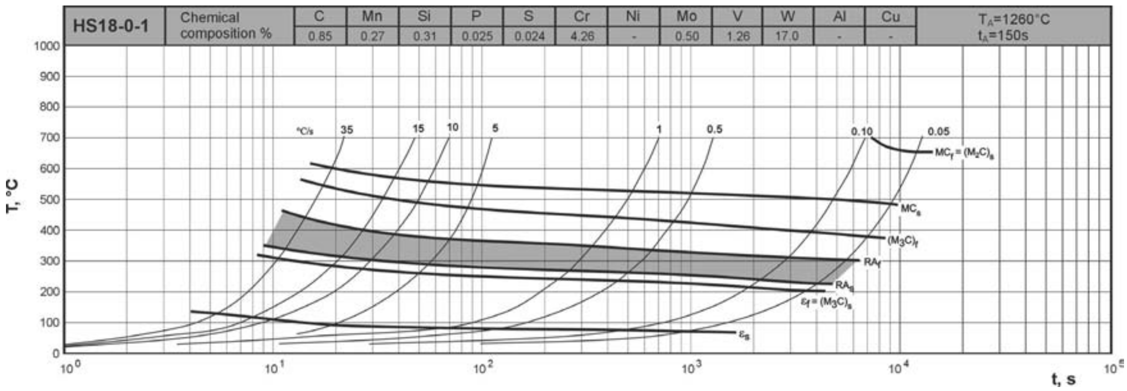

Fig. 7. CHT diagram of HS18-0-1 steel [12]

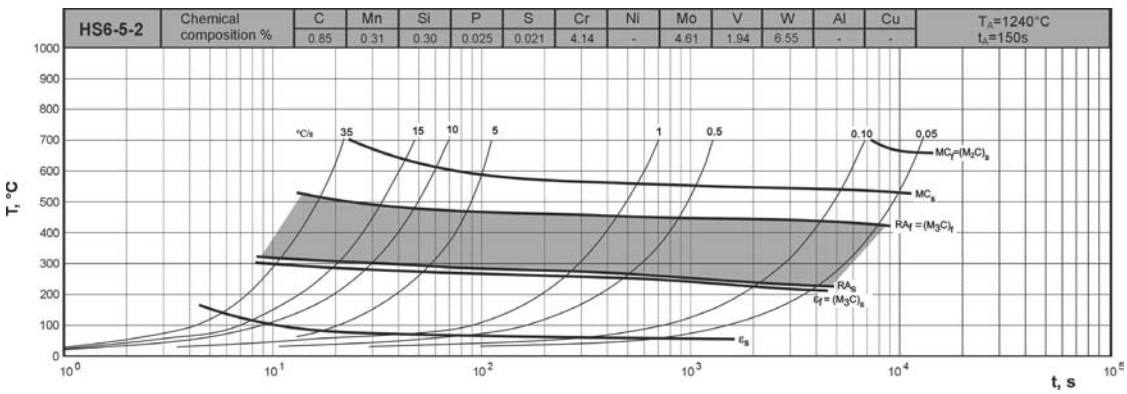

Fig. 8. CHT diagram of HS6-5-2 steel [13]

Figs. 9 and 10 show the microstructures of the samples of investigated steels quenched and then heated with a heating rate of 0.05°C/s. HS18-0-1samples were heated up to 200, 370, 560 and 790°C and HS6-5-2 samples were heated up to 210, 410, 560 and 790°C, respectively. These are specific temperatures, at which, for a given heating rate (0.05°C/s), the following phenomena were noticed: the end of precipitation of ε carbide (before the beginning of transformation of retained austenite), the end of cementite precipitation, the beginning of precipitation of MC alloy carbides and the end of precipitation of carbides of M_2C type. The presented microphotographs indicate a diversified rate of advancement of transformations during tempering, depending on the temperature, that the quenched samples of the tested steel were heated up to. During heating up to 790°C the morphology of primary carbides is not changed. Heating up to ε_f temperature caused the precipitation of ε carbide, which could be observed on the pictures taken with the transmission electron microscope. Besides, heating up to this temperature did not cause changes in the structure of high-speed steels. On the contrary, heating up to $(M_3C)_f$. caused some changes in the microstructure of investigated steels. TEM microphotographs show clear martensite strips with cementite precipitations. Heating up to 560°C initiated the dissolution of cementite and nucleation of carbides of MC type. Dispersive precipitations seen after such tempering in the TEM microstructures are most probably these carbides. In HS6-5-2 steel, after heating it up to 560°C, numerous precipitations of cementite were observed. The presence of cementite after that heat treatment involving heating the samples up to this temperature seems to be a cause of initiation of the nucleation of MC carbides. Heating up to 790°C caused the transformation of martensite into ferrite and precipitation of carbides which can be observed on TEM (Figs. 9d and 10d). The carbides revealed on the microphotographs of samples of the tested steel after such tempering, observed in TEM, are of MC, M_2C, M_6C and $M_{23}C_6$ type [12-14].

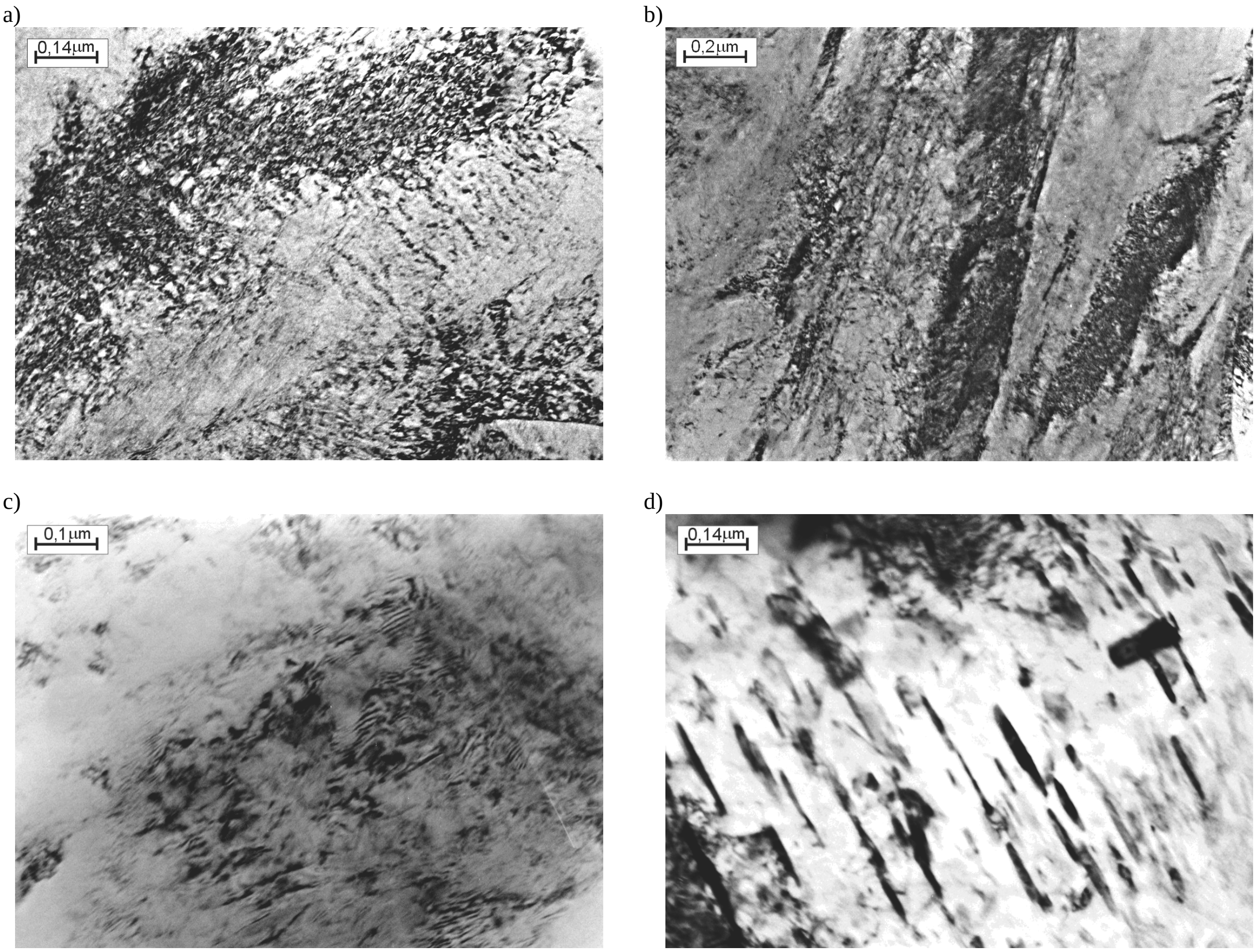

Fig. 9. Microstructures of the HS18-0-1 steel after hardening from 1260°C and heating with the rate 0,05°C/s up to: a) 200°C, b) 370°C, c) 560°C and d) 790°C, TEM

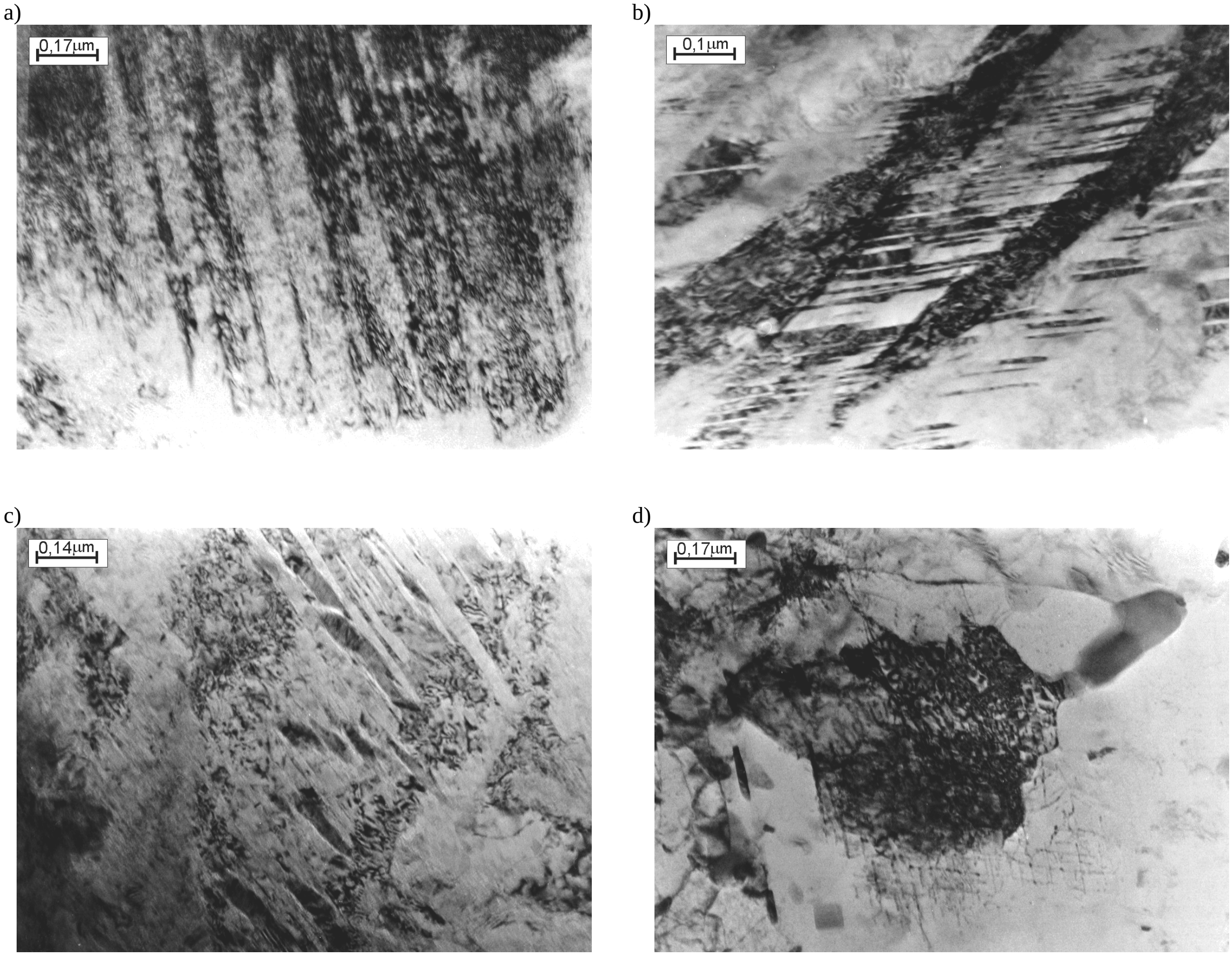

Fig. 10. Microstructures of the HS6-5-2 steel after hardening from 1240°C and heating with the rate 0,05°C/s up to: a) 210°C, b) 410°C, c) 560°C and d) 790°C, TEM

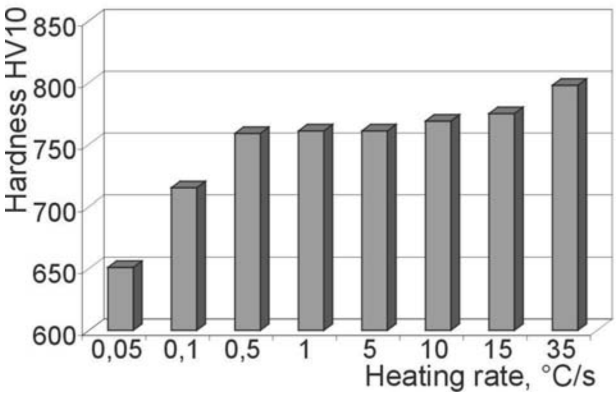

Fig. 11. Influence of heating rate from as-quenched state on hardness of HS18-0-1 steel

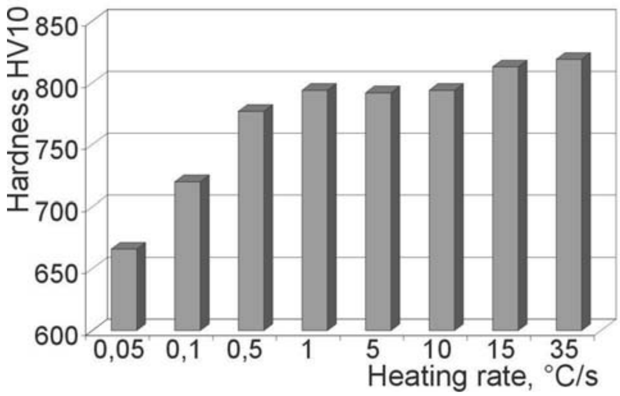

Fig. 12. Influence of heating rate from as-quenched state on hardness of HS6-5-2 steel

Influence of heating rate from quenched state to 700°C on hardness of investigated steels are shown on Figs. 11 and 12. As can be seen, for low heating rates (0.05°C/s and 0.1°C/s) hardness is clearly smaller, what confirms a greater contribution of phase transformations during tempering.

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

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 M_3C, retained austenite transition and precipitation of alloy carbides of MC and M_2C type.

It has been found that in quenched high-speed steels a part of retained austenite has already transformed during heating for tempering, but its significant part transformed only during cooling process after tempering. Change of heating rate during tempering has strong influence on the temperatures of beginnings and the ends of individual transitions as well as on the accompanying dilatation effects.

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

Examination of the microstructures of investigated steels, mainly focused on microstructural development relating to the advancement of transformations during continuous tempering, showed an adequacy of the microstructural changes to CHT diagrams.

Investigations of influence of heating rate from quenched state to 700°C on hardness of steel showed, that application of heating rate higher than 0.5°C/s doesn't cause much changes in hardness of investigated steel.

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