Archives of Materials Science and Engineering

Volume 28
Issue 9
September 2007
Pages 517-524

International Scientific Journal
published monthly as the organ of
the Committee of Materials Science
of the Polish Academy of Sciences

Continuous heating from as-quenched state in a new hot-work steel

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Received 20.06.2007; published in revised form 01.09.2007

ABSTRACT

Purpose: This work contains a detailed description of the kinetics of phase transformations during tempering of a new hot-work steel. Moreover, the differences in hardness and microstructure of samples of the investigated steel in relationship to the heat treatment were evaluated.

Design/methodology/approach: CHT diagram, illustrating the kinetics of phase transformations during continuous heating (tempering) from as-quenched state of investigated steel, was elaborated using a DT 1000 dilatometer of a French company Adamel. In the case of investigations of the microstructural changes, quenched samples were heated with a heating rate of 0.05°C/s to the temperatures of 150, 280 and 650°C. The microstructure of investigated steel were examined using light microscope Axiovert 200 MAT, scanning electron microscope Stereoscan 120 and transmission JEM200CX microscope. The measurements of hardness were performed with the Vickers HPO250 apparatus.

Findings: Heating of the investigated steel from the as-quenched state resulted in the occurrence of 4 primary transformations: precipitation of ε carbide, M_3C precipitation, transformation of retained austenite and precipitation of alloy carbides of MC and M_2C type, nucleating independently. TEM investigations, focused on the determination of a degree of phase transformations during continuous tempering, showed compatibility of the microstructure with the CHT diagram for tested steel.

Research limitations/implications: Description of the kinetics of phase transformations during tempering of a new hot working steel

Practical implications: This results may be used to design new technologies of tempering of this steel and new designed hot-work steels.

Originality/value: Characterization of kinetics phase transformations during continuous heating from as-quenched state in new hot-work steel. Moreover it was shown, that the heating rate from as-quenched state has a strong influence on a hardness after tempering.

Keywords: Tool materials; Hot-work steel; Tempering; CHT - diagram

MATERIALS

1. Introduction

Knowledge of the kinetics of phase transformations during tempering alloys to carefully plan and conduct the operation of tempering, as a result of which optimum combination of resistance and plastic characteristics is obtained, including fracture toughness. After quenching in steel is obtained martensite structure, which mostly characterizes high hardness and strength, but low plasticity and fracture toughness. Using fact of metastable martensite during tempering is possible to change of mechanical properties of steel in a wide range [1].

Hot-work tool steels are used for manufacturing of tools applied within a wide range of temperatures. For example, the working temperature of some drop forging dies is about 200°C, while the extrusion press dies and pressure casting dies work at 600–700°C. Therefore, the most important properties of these steels are: strength, hardness, wear resistance at their working temperatures and thermal endurance, including thermal shock resistance. These properties are achieved by the appropriate composition and properly designed heat treatment [1-4]. Better properties are achieved in steels of complex composition than in steels containing equivalent amounts of one or two elements [1,5,6].

Present-day, a design of hot-work tool steels involves designing of complex chemical compositions, containing from 0,25 to 0,6%C, and characterizing of phase transformation during tempering and their kinetics. Only then a proper heat treatment, leading to the optimal combinations of mechanical properties and high fracture toughness, can be performed [1].

During tempering of unalloyed, medium and high carbon steels, an occurrence of three principal transformations can be observed: precipitation of ε carbide [7-9], transformation of retained austenite into lower bainite [10-12] and precipitation of cementite. In steels containing alloying elements causing an effect of secondary hardening (V, Mo, W), a fourth transformation occurs: precipitation of MC and M_2C-type alloy carbides, that nucleate independently [13-17].

A majority of cited above investigations concerning transformations occurring during tempering was 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 heating from quenched state.

The CHT diagrams [1,5,6,16,17] contribute to advances related to successive transformations during tempering (e.g. by means of the change of heating rate, temperature and time of soaking) and respectively, to achieving advantageous properties, and high fracture toughness in particular.

2. Test material

The research was conducted on a new hot-work steel with the chemical composition is given in Table 1.

Table 1.
Chemical composition of the investigated steel

mass %						
C	Mn	Si	Cr	Mo	Ni	V
0.30	0.30	0.30	2.50	2.60	1,59	0.18

Prior to testing the samples of investigated steels were soft annealed at 900°C/30 min. and successively cooled at the rate of 12°C/hour to 600°C, and after that to the room temperature together with the furnace.

Designing the tempering conditions for a new steel gives an opportunity to define the kinetics of phase transformations during tempering. This let to obtain a die made out of that steel having good fracture toughness. For this purpose, a knowledge of CHT diagram, illustrating a kinetics of phase transformations during tempering (heating from quenched state), is needed.

3. Experimental procedure

The dilatometric tests were performed using a DT1000 dilatometer manufactured by Adamel in France. The samples (∅2x12 mm), after prior quenching from 1050°C (austenitizing time 20min), were heated with various rates up to 700°C. The digitally recorded heating dilatograms enabled drawing the CHT diagrams of tested steel in temperature – time system, according to the characteristic points read out from differential curves.

In the case of investigations of the microstructural changes, quenched samples were heated with a heating rate of 0.05°C/s to the temperatures of 150, 280 and 650°C. The microstructure of investigated steel were examined by a light microscope Axiovert 200 MAT, scanning electron microscope Stereoscan 120 and electron transmission JEM200CX microscope.

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

4. Results and discussion

Figure 1 presents tested steel dilatogram of heating from quenched state, together with corresponding differential curve, on which there are marked the temperatures of the beginning (letter s) and the end (letter f) of individual transition. This figure presents a method of dilatograms interpretation on the basis of which the CHT diagram has been made

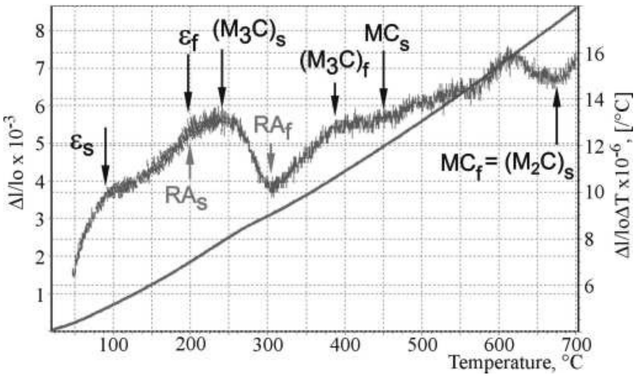

Fig. 1. The heating dilatogram for the heating rate of 0.05°C/s and the corresponding differential curve

During the first stage of tempering the investigated steel exhibits contraction related to the precipitation of ε carbide. The contraction begins at the temperature ε_s and ends at the temperature ε_f. The contraction related to precipitation of ε carbide is not significant, due to the low (as for tool steels) carbon content (0,30%). Since as soon as the temperature ε_f is reached, the transition of retained austenite begins almost immediately, what is accompanied by the volume increase, therefore it has been assumed that the temperature at which the ε carbide precipitation process ends (ε_f) is equivalent to the temperature of the beginning

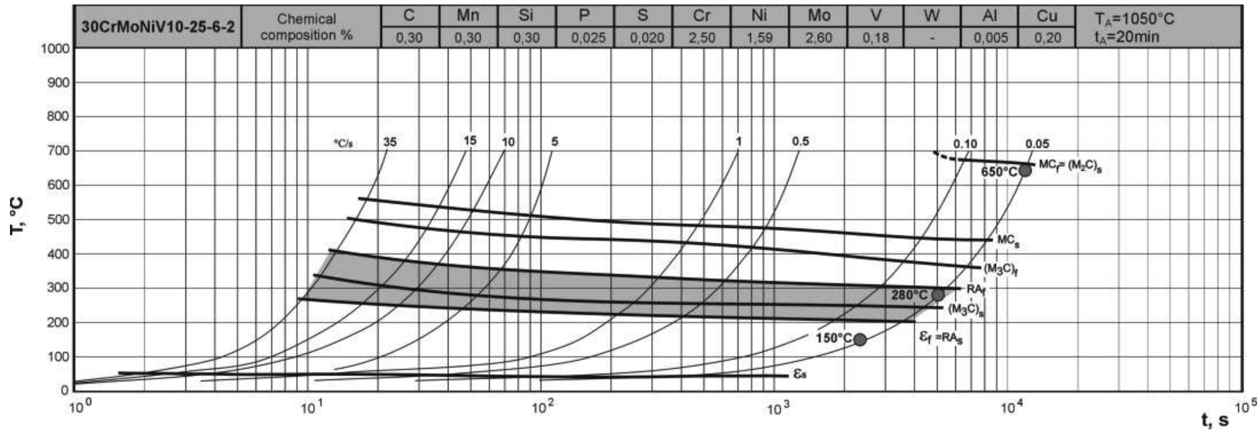

Fig. 2. Continuous heating transformations (CHT) diagram for investigated steel, indicate sampling for TEM investigation [17]

of retained austenite transition RA_S. This effect may be noticed within the temperature range RA_S-RA_f. The second contraction beginning at temperature $(M_3C)_S$ is related to precipitation of cementite (alloyed). The end of cementite precipitation process takes place at temperature $(M_3C)_f$. The increase of volume within the temperature range MC_S-MC_f is related to precipitation of independently nucleating carbides of the MC type [17].

Comparing the examined steel to high-speed steels [16] the effect related to precipitation of MC carbides is weaker, what is connected with less carbon and vanadium content in matrix of quenched steel. However, it is important to notice that, the temperature of the beginning of MC carbides precipitation is relatively low and for heating rate of 0.05°C/s is 450°C, while for HS18-0-1 and HS6-5-2 is 495°C. The temperature $(MC)_f$ is approximately equal to the temperature of precipitation start of $(M_2C)_s$ type carbides.

Figure 2 demonstrates the full CHT diagram of investigated steel. Horizontal lines mark the ranges of ε carbide precipitation, cementite precipitation, the range of residual austenite transition and precipitation of independently nucleating carbides of MC and M_2C type. The temperature $(M_2C)_s$ has only been determined for the two lowest rates of heating 0,05°C/s and 0,01°C/s, for the other applied rates of heating this temperature is higher than 700°C. It can be noticed, that with heating rate increase from 0.05 to 35°C/s the temperatures of the beginnings and ends of particular transitions also increase. On the CHT diagram (Fig. 2) red dots mark the temperatures at which the heating of samples was interrupted (at the rate of 0.05°C/s) in order to perform the microscopic examination. These are the temperatures of highest precipitation rate of ε carbide, the end of retained austenite transition and about 20°C below the end of precipitation process of independently nucleating carbides of MC type.

Figure 3 presents the microstructure of test steel samples in quenched state. Immediately after quenching from 1050°C the microstructure consists of martensite and retained austenite (in amount of 5,9%) [16].

Figures 4-6 shows the microstructures of the samples quenched from 1050°C, and then heated with a heating rate of 0.05°C/s (see Fig. 2) to 150 (Fig. 4), 280 (Fig. 5) and 650°C (Fig. 6) respectively.

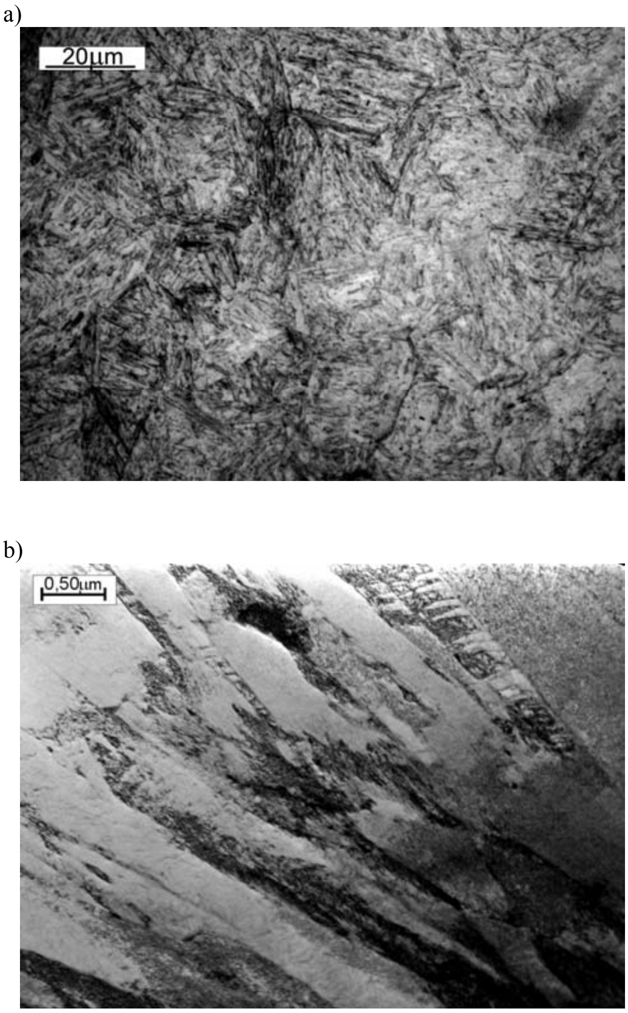

Fig. 3. Microstructures of investigated steel after quenching from 1050°C: a) light microscope, nital etched, b) TEM

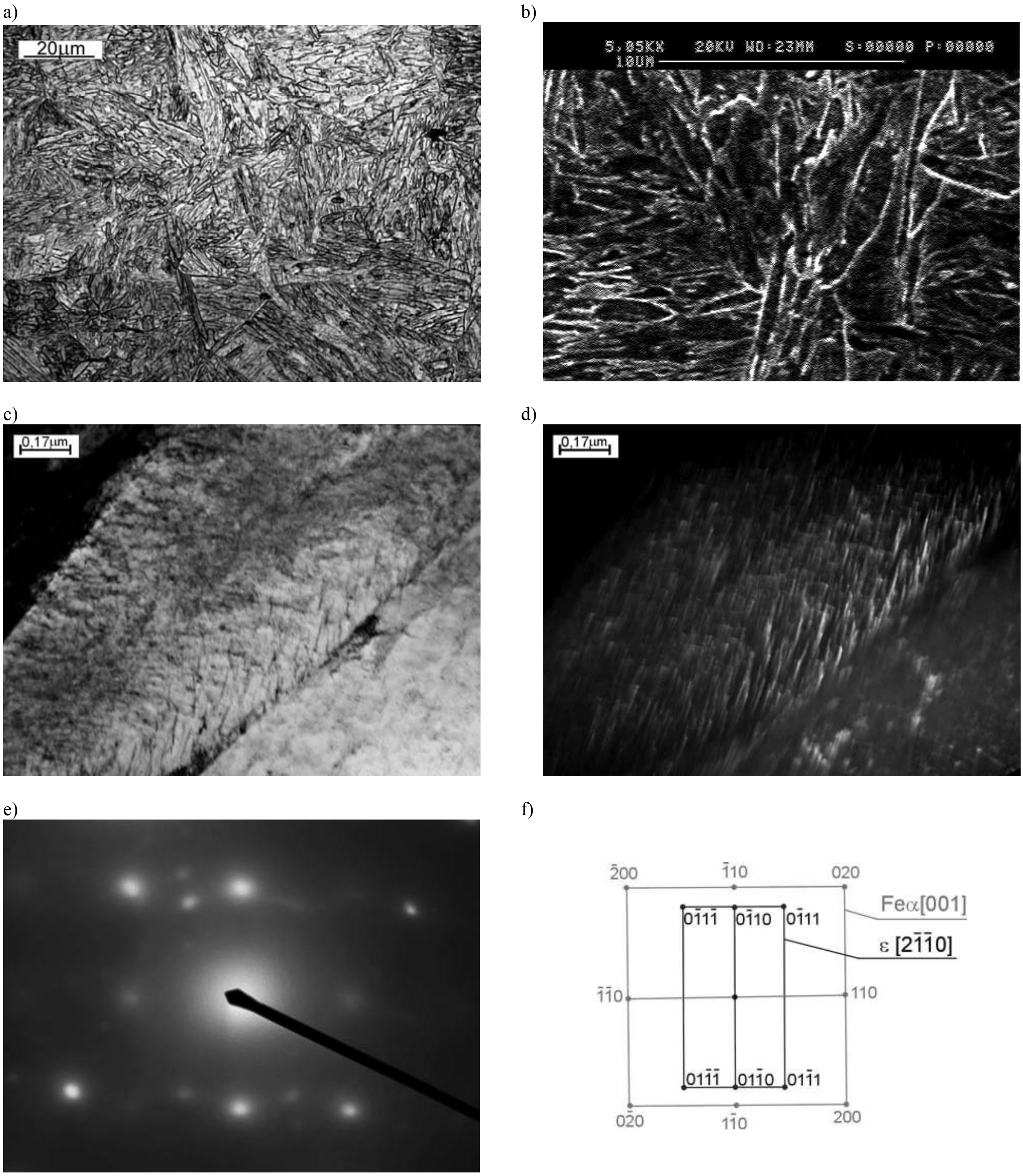

Fig. 4. Microstructure after quenching from 1050°C and subsequent heating with the rate 0.05°C/s to 150°C: a) light microscope, b) scanning microscope, nital etched, c) TEM d) dark field from the $(0\bar{1}1\bar{1})$ reflex ε carbides, e) diffraction pattern from the area as in figure d, f) solution of the diffraction pattern from figure e

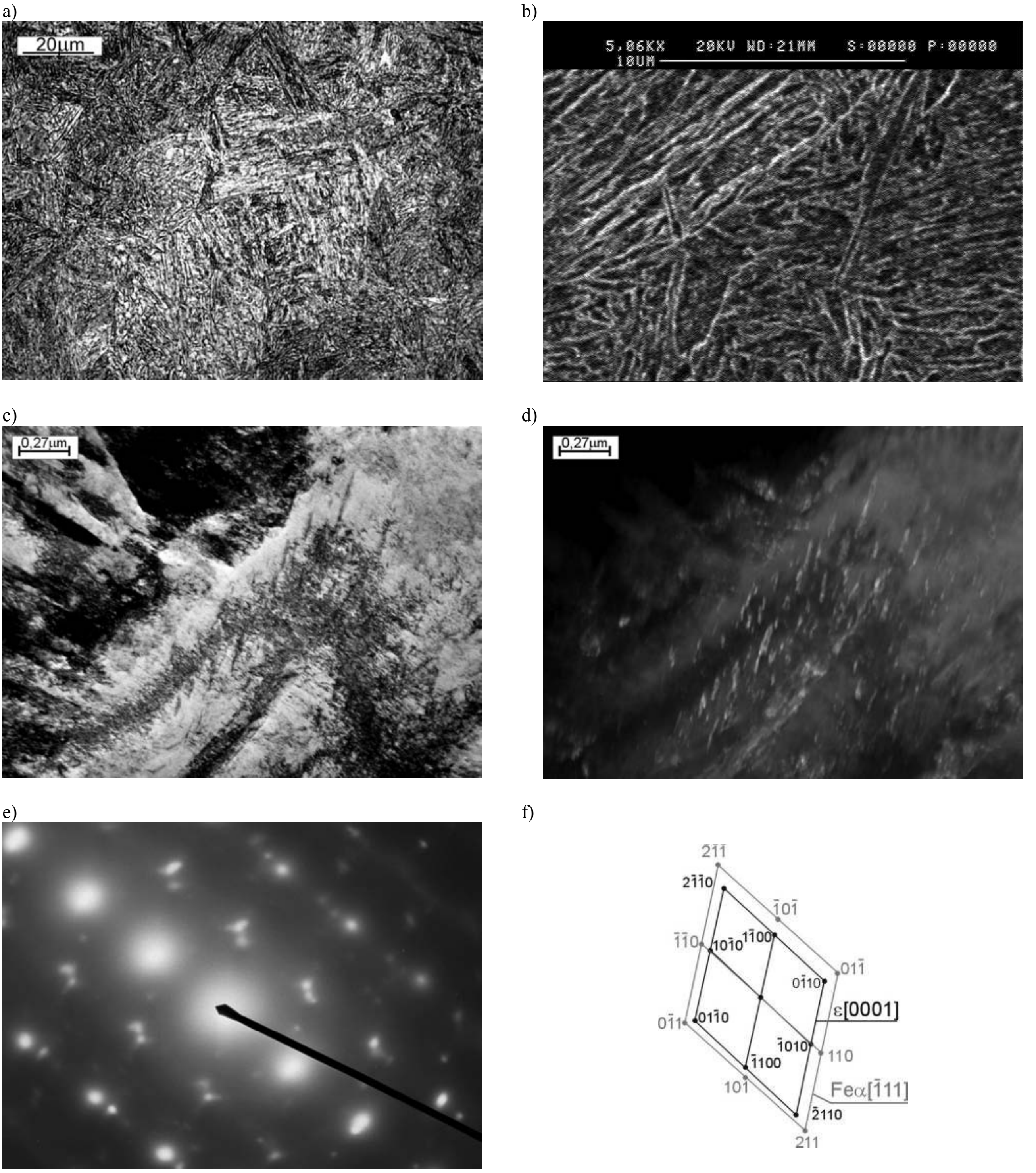

Fig. 5. Microstructure after quenching from 1050°C and subsequent heating with the rate 0.05°C/s to 280°C: a) light microscope, b) scanning microscope, nital etched, c) TEM d) dark field from the $(1\bar{1}00)$ reflex ε carbides, e) diffraction pattern from the area as in figure d, f) solution of the diffraction pattern from figure e

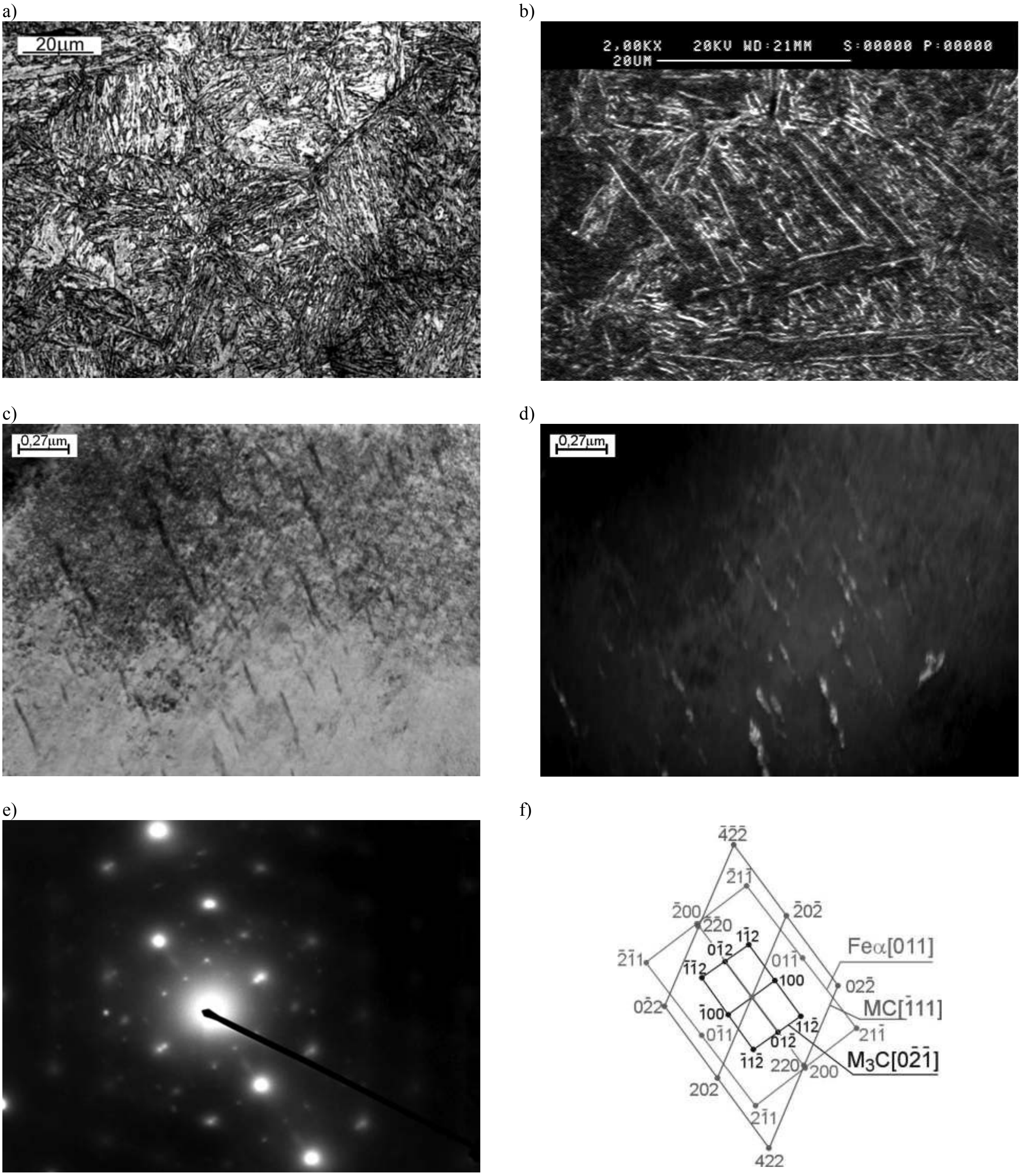

Fig. 6. Microstructure after quenching from 1050°C and subsequent heating with the rate 0.05°C/s to 650°C: a) light microscope, b) scanning microscope, nital etched, c) TEM d) dark field from the $(1\bar{1}2)$ reflex M_3C carbides, e) diffraction pattern from the area as in figure d, f) solution of the diffraction pattern from figure e

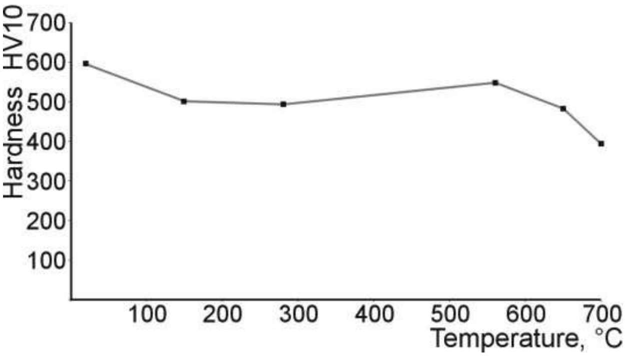

Fig. 7. Dependence of hardness of samples made of tested steel on the heating temperature after quenching (heating rate 0.05°C/s)

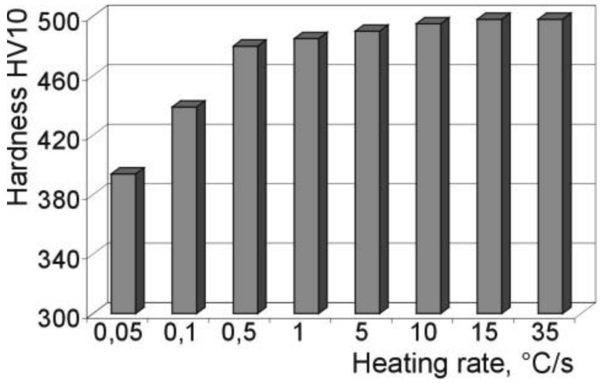

Fig. 8. Influence of heating rate from as-quenched state on hardness of the investigated steel

The heating to 150°C has resulted in ε carbides precipitation (mainly on dislocations, Fig. 4c). No other changes in microstructure were observed upon heating to this temperature.

The changes of structure as a result of heating up to 280°C are presented in fig. 5. It is noticeable that the precipitated during the first stage of tempering ε carbides, are still present in the microstructure of examined steel. After heating to 280°C there were also observed the independent nucleation of cementite on the boundaries of martensite strips and still existing retained austenite [16].

The heating to 650°C (Fig. 6) has initiated cementite decomposition and independent nucleation of hardly identified alloy carbides of MC type. The Fig. 6c presents the cementite surrounded by alloy carbides of MC type. After such tempering there was no retained austenite observed in the microstructure. No other type of alloy carbides was found in this sample.

Figure 7 shows the change of hardness of tested samples, depending on the heating temperature after quenching. Hardness after quenching is 596HV10. The heating after quenching to temperature 560°C doesn't caused vehemently increase of hardness, what may resulted from low content of carbon. The highest hardness exhibited the quenched sample, while the lowest one the sample heated up to 700°C, when the coherence of MC carbides precipitates was broken and the progress of precipitation of M_2C occurred .

Influence of heating rate from quenched state to 700°C on hardness of investigated steels are shown on Figure 8. 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 significant 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

During heating of quenched steel 30CrMoNiV10–25–6–2 the occurrence of four basic transitions was found, i.e. precipitation of ε carbide, precipitation of M_3C, transformation of retained austenite and precipitation of alloy carbides of MC and M_2C type.

The ε carbides precipitate on dislocations within the whole volume of martensite, while the cementite initially nucleates independently in preferred sites, i.e. on martensite strips boundaries, drawing out the carbon first from retained austenite (leading to its destabilization), and after that from dissolving ε carbides what makes possible for cementite to independently nucleate within the whole volume.

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 changes in hardness with tempering temperature showed, that tested steel characterize small inclination to softening with tempering temperature.

The increase of heating rate (from 0.05 to 35°C/s) results in the increase of temperatures of the beginnings and the ends of particular transitions, and in decrease of accompanying dilatation effects.

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

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