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THE INFLUENCE OF THE KINETICS OF PHASE TRANSFORMATIONS DURING TEMPERING ON THE STRUCTURE DEVELOPMENT IN A HIGH CARBON STEEL

WPŁYW KINETYKI PRZEMIAN FAZOWYCH PRZY ODPUSZCZANIU NA KSZTAŁTOWANIE MIKROSTRUKTURY WYSOKOWĘGLOWEJ STALI STOPOWEJ

This work contains a detailed description of the kinetics of phase transformations occurring during tempering of a new, high carbon (1.22%C) Cr-Mo-V steel. The temperature ranges of the phase transformations occurring during heating from the quenched state (tempering) were determined from dilatometric tests. An interpretation of dilatograms of heating from the as-quenched state was also presented. The temperature ranges of the observed transformations during tempering of investigated steel were presented on CHT (Continuous Heating Transformations) diagrams. Moreover, the microstructure development in tested samples, reflecting the extend of the phase transformations during tempering, was discussed too.

Keywords: tool materials, steel, tempering, CHT – diagram, ε carbide, retained austenite

W pracy zamieszczono szczegółowy opis kinetyki przemian fazowych przy odpuszczaniu nowej, wysokowęglowej (1.22%C) stali chromowo-molibedenowo-wanadowej. Na podstawie badań dylatometrycznych wyznaczono zakresy występowania przemian fazowych podczas nagrzewania ze stanu zahartowanego (odpuszczania stali). W pracy zamieszczono również interpretację dylatogramów nagrzewania ze stanu zahartowanego. Zakresy poszczególnych przemian przy odpuszczania badanej stali przedstawiono na wykresie CTPc^{O}. Ponadto, zamieszczono mikrostruktury próbek w zależności od stopnia zaawansowania w nich przemian przy odpuszczaniu.

1. Introduction

During heating from the quenched state (tempering) of unalloyed, medium and high carbon steels, an occurrence of three principal transformations can be observed: precipitation of ε carbide, transformation of retained austenite into lower bainite 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 [1, 2].

During 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 [3]. Precipitation of highly dispersed ε carbide is believed to enhance strengthening in steel. However, the martensite with smaller carbon content is softer, what causes only a modest drop in strength and hardness of steel tempered in that temperature range [2].

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. It should be noticed, that this transformation occurs only in steels containing more than 0.3%C because the amount of the austenite remaining in the steel after quenching strongly depends on carbon content. The austenite does not occur in steels containing less than 0.3%C [1, 2].

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. The mechanism of the nucleation of cementite is, however, not fully understood yet. According to Ref. [4] precipitating cemen-

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tite 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 becomes occur as 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 [2].

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. The aim of this work is to determine the kinetics of phase transformations occurring during continuous heating after quenching of a high carbon alloy steel and its effect on the development of steel microstructure.

2. Material

The research was conducted on a Cr-Mo-V high carbon steel with the chemical composition given in Table 1.

TABLE

The chemical composition of investigated steel

C	Mn	Si	P	S	Cr	Mo	V	Al
1.22	1.93	0.19	0.018	0.02	1.52	0.36	0.17	0.04

A 50 kg ingot (120 mm diameter), produced in laboratory conditions, was forged into bars with a cross-section of 20×35 mm. The bars where then fully annealed and the samples for further investigations were cut off of these bars.

Austenitizing temperature (900°C) was determined from experimentally performed series of hardening treatments. The critical temperatures for investigated steel are: A_{C1s} = 730°C, A_{C1f} = 750°C.

3. Experimental procedure

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. Samples with a diameter of 2 mm and a height of 12 mm, after quenching from 900°C (austenitizing time of 1200 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 and help to build the CHT diagram for the investigated steel (in the Time-Temperature-Transformation system for continuous heating from quenched state). Dilatometric curves were graphically plotted by the DylArt software [6].

Hardened samples (Ø2×12 mm) were heated with a rate of 0.05°C/s to the temperatures of 210, 370 and 520°C. The microstructure of these samples were examined by a light microscope Axiovert 200 MAT and scanning electron microscope Stereoscan 120.

Similarly, heat treated samples, with a diameter of 3 mm and a height of 10 mm, were used for TEM investigations using JEM 200CX microscope.

4. Results and discussion

Fig. 1 shows a dilatogram of a sample (quenched from 900°C) heated with a rate of 0.05°C/s, along with a corresponding differential curve showing a method of an interpretation of dilatograms, basing on which a CHT diagram was created.

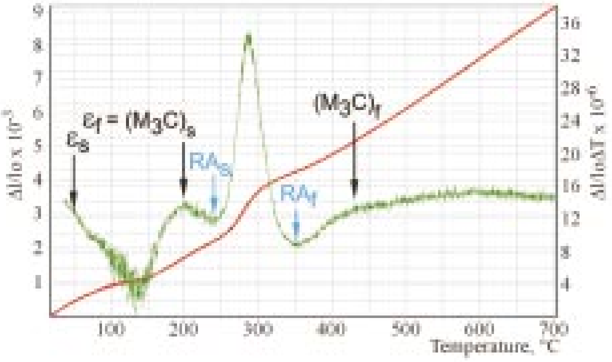

Fig. 1. Dilatogram of heating the samples with a heating rate of 0.05°C/s and a corresponding differential curve

It is apparent that during, the first stage of tempering, the investigated steel exhibits shrinkage, which may mainly be attributed to ε carbide precipitation. This shrinkage starts at ε_s temperature, and ends at ε_f one. When the ε_f temperature is reached, the second stage of shrinkage starts almost immediately and, at this time, it is associated with the precipitation of alloy cementite. That is why, it was assumed that the temperature of the termination of ε carbide precipitation (ε_f) is equal to the temperature at which the precipitation of alloy cementite starts $(M_3C)_s$. A strong positive dilatation effect connected with retained austenite transformation can also be noticed. This effect is noticeable in the temperature range of retained austenite starts (RA_s) ÷ retained austenite finish (RA_f). It can also be noticed, that retained austenite transformation takes place in the temperature range of cementite precipitation, that is between $(M_3C)_s$ i $(M_3C)_f$ temperatures. A more detailed description of the phase transformation kinetics in the investigated steel during tempering from the as-quenched state, based on dilatometric tests, is presented in Ref. [7, 8].

Fig. 2 shows a complete CHT diagram for the investigated steel. It shows the ranges of ε carbide and cementite precipitation as well as the temperature range for the transformation of retained austenite. As can be noticed, transformation start and transformation end temperatures increase with increasing heating rate from 0.05 to 35°C/s.

During tempering of the investigated steel, quenched from 900°C no effects associated with precipitation of independently nucleating MC carbides were recorded.

Fig. 3 and **4** show the microstructure of the investigated steel in the as-quenched state. In this condition (sample quenched from 900°C), the microstructure consists of martensite (partly twinned), retained austenite and secondary cementite undissolved during austenitizing process. Such cementite (large spheroidal precipitates) can be observed in TEM micrographs.

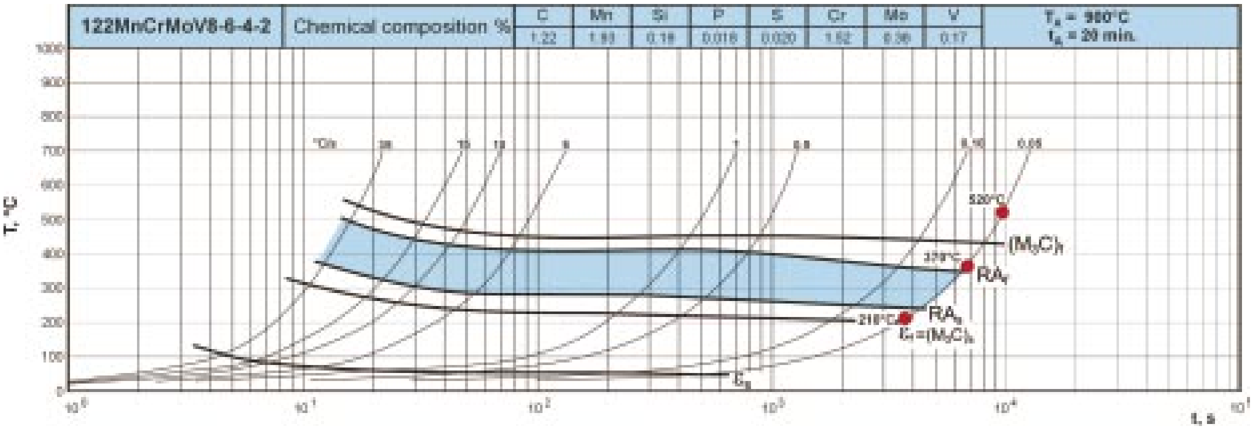

Fig. 2. CHT diagram for investigated steel. The temperatures of the end of heating the samples with a heating rate of 0.05°C/s are marked red

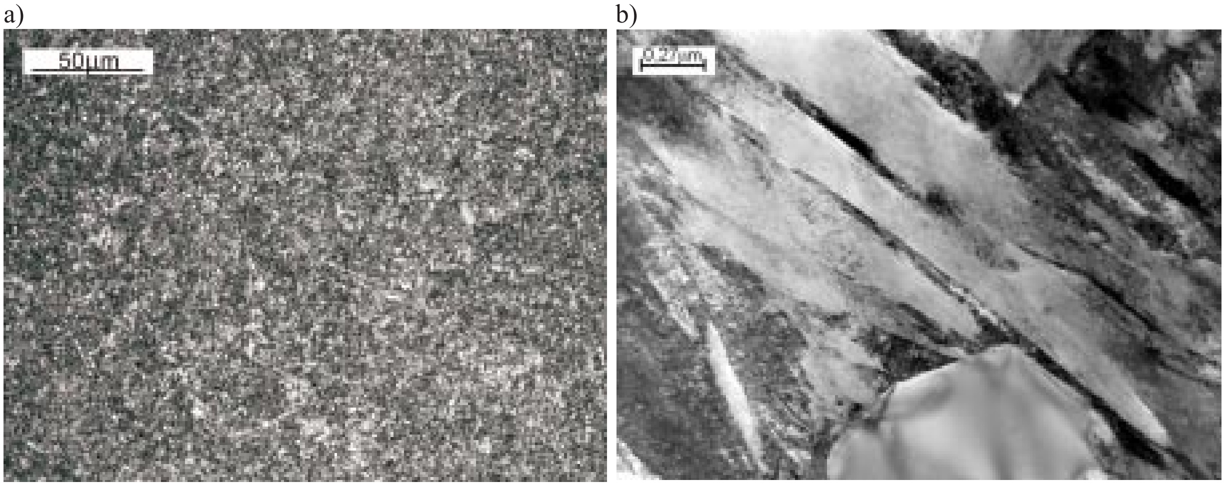

Fig. 3. Microstructures of the samples hardened from the temperature of 900°C: a) optical microscope, etched with nital, b) TEM

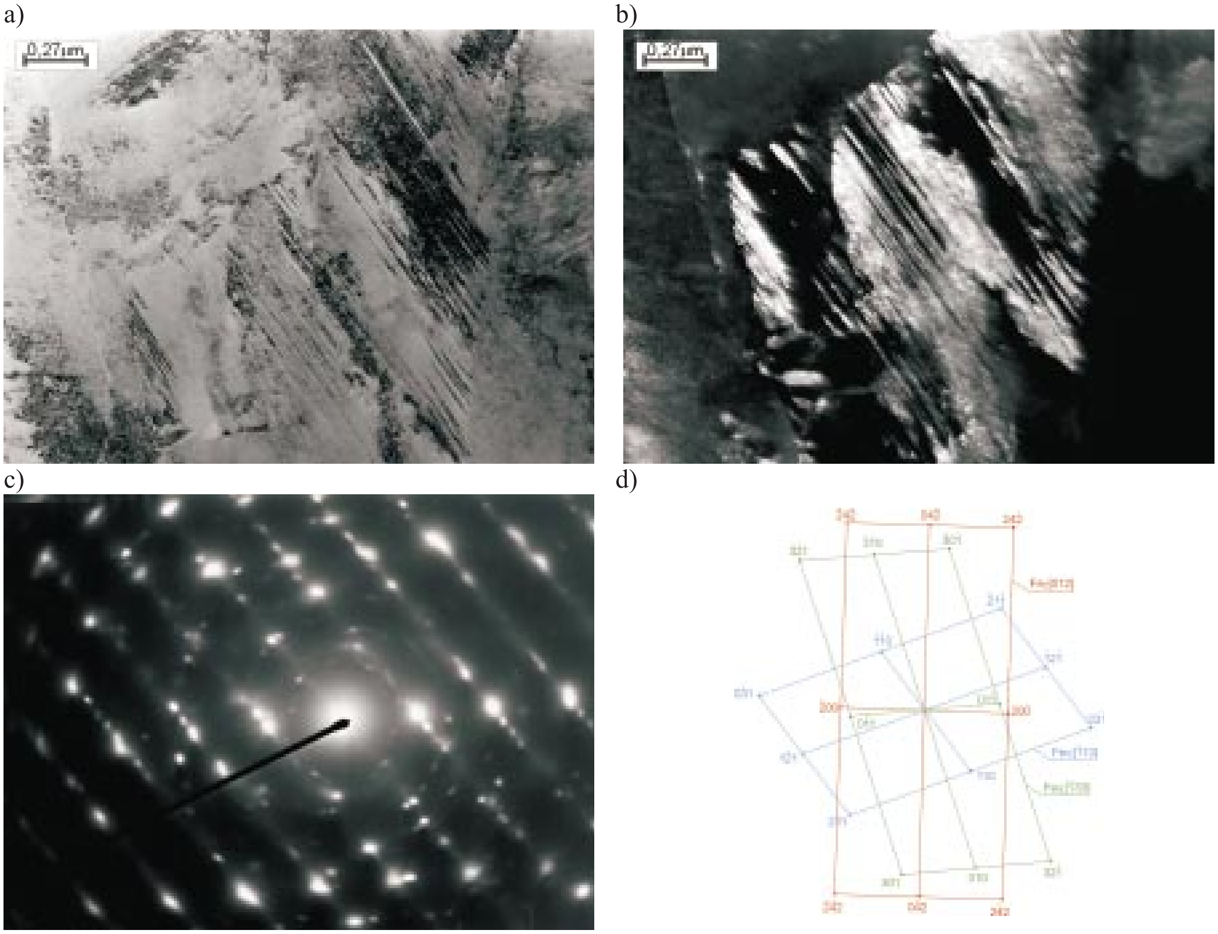

Fig. 4. Microstructure (TEM) of steel quench from 900°C: a) bright field, b) dark field from the reflex (200)Feγ, c) diffraction pattern from the area visible on figure a, d) solution of the diffraction pattern from figure c

Figures **5÷10** show the microstructures of the samples quenched from 900°C, and then heated with a heating rate of 0.05°C/s (see Fig. 2) to 210, 370 i 520°C respectively. These are specific temperatures, at which, for a given heating rate (0.05°C/s) the following phenomena were noticed: at 210°C – the end of ε carbide precipitation (before the start of retained austenite transformation) and at 370°C – the end of the transformation of retained austenite into lower bainite. Next part of the research was performed at 520°C, when precipitation of cementite was completed. The pictures show diversified advancement of transformation during tempering, depending on the temperature to which the hardened samples were heated. During heating, the morphology of secondary carbides does not change, what can be seen on the optical micrographs and from SEM (Fig. 5). Heating to 210°C caused almost complete precipitation of ε carbide (compare with Fig. 1), whose small and dispersed particles can be noticed on TEM micrograph (Fig. 6a). Heating to 210°C additionally ease precipitation of cementite. The twinned boundaries of martensite were observed in the microstructure (Fig. 7). No other changes comparing to the as-hardened state were observed in the structure.

Well-defined changes in microstructure are only mainly observed after heating to of 370°C. SEM micrographs shows a surface relief coming likely from the products of a retained austenite transformation (Fig. 8 b). Moreover, TEM micrographs (Fig. 8c, d) show the martensite strip on the boundaries, on which precipitated cementite was found. Additionally, any retained austenite was detected in the microstructure (Fig. 9). Heating to 520°C brings about coarsening of precipitated particles of cementite (Fig. 10), what is clearly demonstrated in the microstructure shown in TEM micrographs.

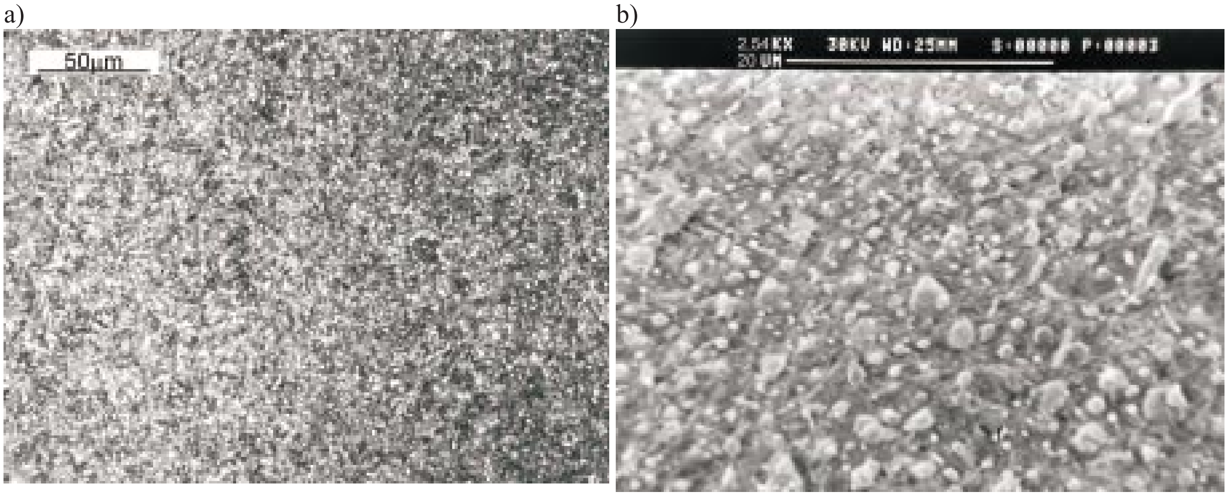

Fig. 5. Microstructure of investigated steel after hardening from 900°C and subsequent heating to the temperature of 210°C with a heating rate of 0.05°C/s: a) picture from an optical microscope, b) picture from SEM. Etched with nital

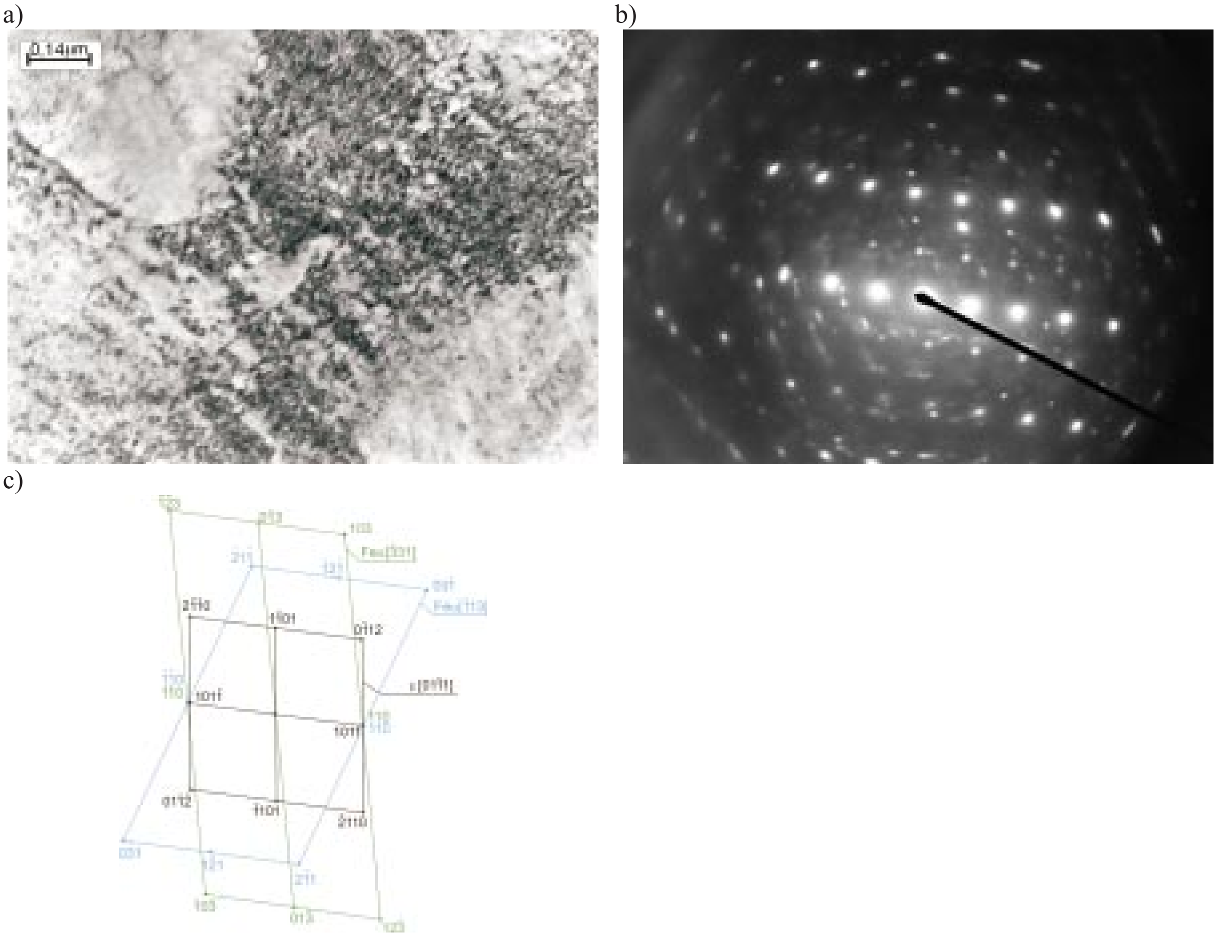

Fig. 6. Microstructure of investigated steel (TEM) quench from 900°C and subsequently heated to 210°C with a heating rate of 0.05°C/s: a) picture taken in a bright field, b) diffraction pattern for the area visible on figure a, and c) solution of diffraction pattern for figure b

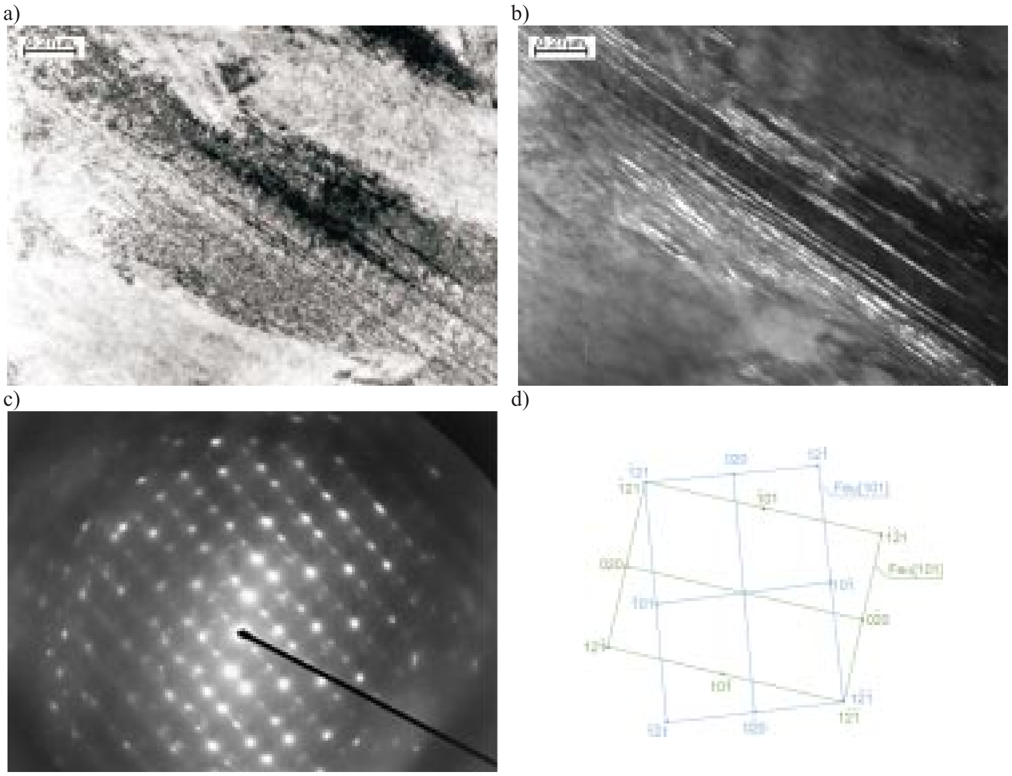

Fig. 7. Microstructure of investigated steel (TEM) quenched from 900°C and subsequently heated to 210°C with a heating rate of 0.05°C/s: a) bright field, b) dark field, c) diffraction pattern for the area visible on figure a, and d) solution of diffraction pattern for figure c

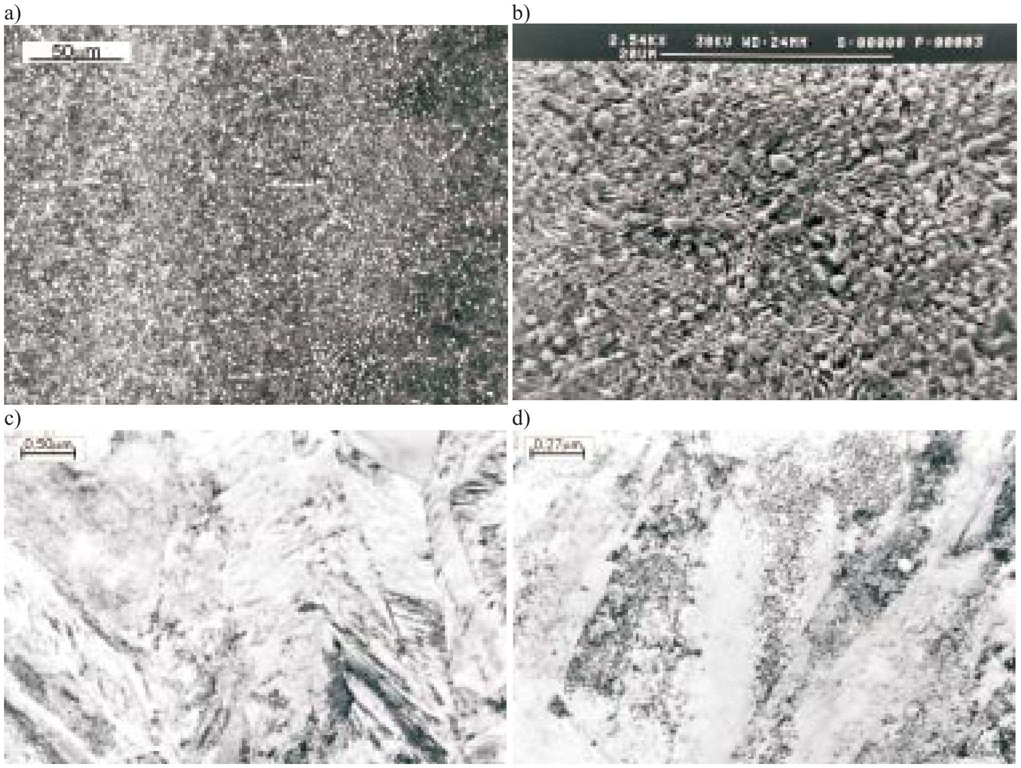

Fig. 8. Microstructure of investigated steel (TEM) quenched from 900°C and subsequently heated to 370°C with a heating rate of 0.05°C/s: a) picture from an optical microscope (sample etched with nital), b) SEM picture (sample etched with nital), and c, d) TEM pictures

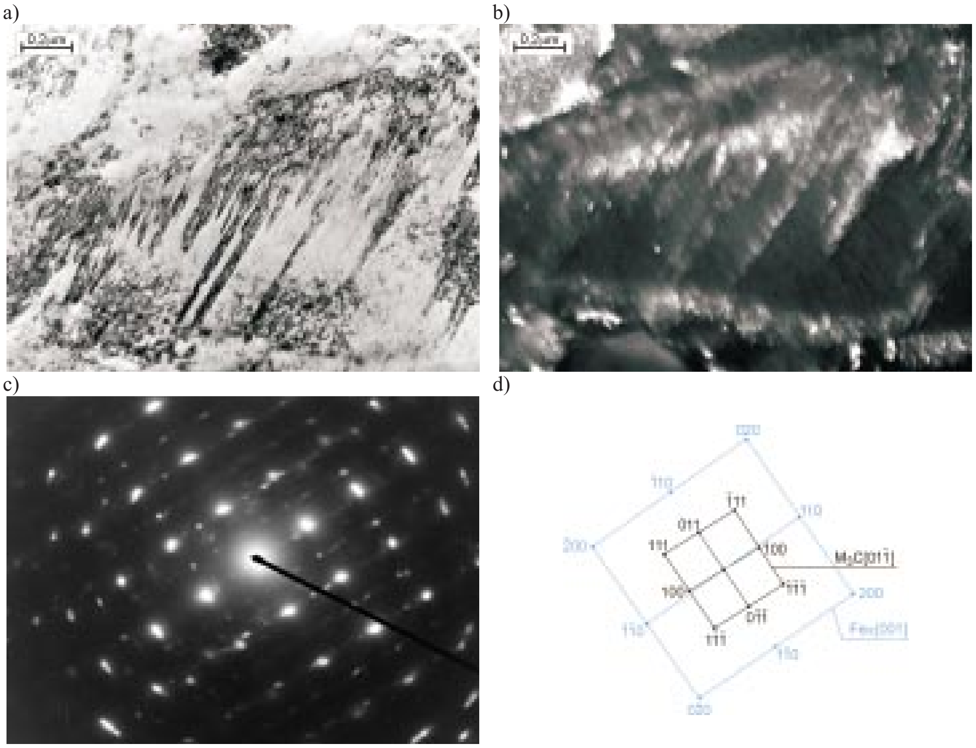

Fig. 9. Microstructure of investigated steel (TEM) quenched from 900°C and subsequently heated to 370°C with a heating rate of 0.05°C/s: a) bright field, b) dark field - from (111)M_3C reflection, c) diffraction pattern for the area visible on figure a, and d) solution of a diffraction pattern for figure c

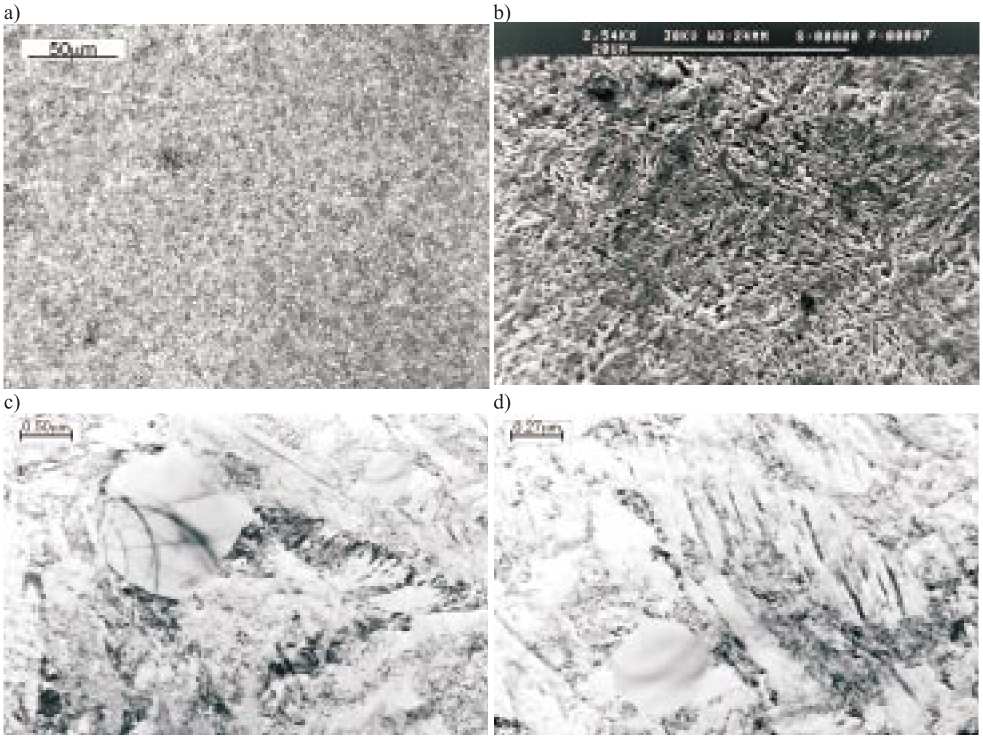

Fig. 10. Microstructure of investigated steel quenched from 900°C and subsequent heated to the temperature of 520°C with a heating rate of 0.05°C/s: a) optical microscope, etched with nital b) SEM, etched with nital and c), d) TEM

5. Summary

Heating of the investigated steel from the as-quenched state resulted in the occurrence of three primary transformations: precipitation of ε carbide – connected with the first range of the sample shrinkage, precipitation of M_3C cementite – connected with the second shrinkage of the samples, and transformation of a retained austenite resulting in a strong, positive dilatation effect. It was also noticed, that transformation of a retained austenite occurs in the same temperature range as formation of cementite.

Examination of the microstructure of investigated steel, mainly focused on microstructural development relating to the advancement of transformations during continuous tempering, showed an adequacy of the microstructural changes to CHT diagrams. The most preferential sites for precipitation of cementite, are martensite strips, indicating that its nucleation is independent. It was also noticed, that precipitation of cementite takes place only when precipitations of metastable ε carbide are present in the microstructure. The morphology of the obtained structures is similar to that of lower bainite, suggesting that it is a likely reason for a temper brittleness.

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