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The kinetics of phase transformations during tempering in the new hot working steel

P. Bała*, J. Pacyna, J. Krawczyk

Faculty of Metals Engineering and Industrial Computer Science, AGH University of Science and Technology, Al. Mickiewicza 30, 30-059 Kraków, Poland * Corresponding author: E-mail address: pbala@agh.edu.pl

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Materials

<u>ABSTRACT</u>

Purpose: This work contains a detailed description of the kinetics of phase transformations during tempering of new hot working steel. Moreover, the differences in 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 210, 320 and 420°C. The microstructure of investigated steel were examined using transmission JEM200CX microscope.

Findings: Heating of the investigated steel from the as-quenched state resulted in the occurrence of 4 primary transformations: precipitation of ε carbide, M₃C precipitation, transformation of retained austenite and precipitation of alloy carbides of MC and M₂C type, nucleating independently. TEM investigations focused on determination of a degree of phase transformations during continuous tempering, showed compatibility of the microstructure with CHT diagram for tested steel.

Research limitations/implications: The new CHT diagram of investigated steel was determined.

Practical implications: The obtained CHT diagram may be used to design new technologies of tempering of this steel. **Originality/value:** The new CHT diagram, characterization of tempering new hot working steel. **Keywords:** Tool materials; Hot working steel; Tempering; CHT - diagram

1. Introduction

The structure and properties of tools have significant influence on their performance efficiency and reliability what fosters the development of mechanization and automation of processing lines. Hot-work tool steels are used for manufacturing of tools working 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 very important properties of these steels are: strength, hardness, wear resistance at their working temperatures and thermal endurance, including thermal shock resistance. The above mentioned properties of these steels are achieved on the way of appropriate composition and properly designed heat treatment [1-3]. Better properties are achieved in steels of complex composition than in steels containing large amounts of one or two elements [1, 4, 5]. Nowadays, a design of hot working tool steels involves designing complex chemical compositions, containing from 0,25 to 0,6%C and characterizing special kinetics of phase transformations during tempering. Only then a proper heat treatment, leading to the optimal combinations of mechanical properties and high fracture toughness, can be performed [1].

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 [6-8], transformation of retained austenite into lower bainite [9-11] 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 M₂C-type alloy carbides, that nucleate independently [12-17].

2. Test material

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

Table 1.

Chemical composition of the investigated steel

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

Designing the tempering conditions for a new steel gives an opportunity to define the kinetics of phase transformations during tempering to obtain 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 DT1000 dilatometer manufactured by Adamel in France. The samples (\emptyset 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 using transmission JEM200CX microscope.

4. Research results and discussion

Fig. 1 presents a dilatogram of test steel sample heating at the rate of 0.05°C/s, previously quenched from 1050°C, along with corresponding differential curve, on which there is presented a method of dilatograms interpretation on the basis of which the CHT diagram has been made

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 . As one may notice the contraction related to precipitation of ε carbide is not significant, which is the caused by low (as for tool steels) carbon content (0,30%). Since almost immediately as soon as the temperature ε_f is reached, the transition of residual austenite begins, which is accompanied by the volume increase, therefore it has been assumed that the temperature at which the ε (ε_f) carbide precipitation process ends is equivalent to the temperature of the beginning of residual 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 (alloy). The end of cementite precipitation process takes place at temperature (M₃C)_f. The increase of volume within the temperature range $MC_s \div MC_f$ is related to precipitation of independently nucleating carbides of MC type. Comparing the test steel to rapid tool steels [15] the effect related to precipitation of MC carbides is much lower, what is connected with less carbon and vanadium content in matrix of guenched 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, and for HS18-0-1 and HS6-5-2 495°C respectively. The temperature (MC)_f is approximately equal to the temperature of precipitation start of $(M_2C)_s$ type carbides.



Fig. 1. The heating dilatogram of the samples investigated steel (heating rate of 0.05° C/s) and a corresponding differential curve

Fig. 2 presents full CHT diagram of investigated steel. There are marked on it 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 two the 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 CHT diagram (fig. 2) using red dots are marked 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 residual austenite transition and about 20°C below the end of precipitation process of independently nucleating carbides of MC type.



Fig. 2. Continuous heating transformations (CHT) diagram for investigated steel. After heating to the red point (marked on the 0.05°C/s curve) the TEM was made



Fig. 3. Microstructures of the investigated steel a) after hardening from 1050°C; after hardening from 1050°C and heating with the rate $0,05^{\circ}$ C/s to: b) 150°C, c) 280°C, d) 650°C, TE

Fig. 3 presents the microstructure of test steel samples in quenched state as well as quenched and successively heated at the rate of 0.05° C/s to 150, 280 and 650°C.

Immediately after quenching from 1050° C the microstructure consists of martensite and residual austenite (in amount of 5,9%) [16].

The heating to 150°C has resulted in ε carbides precipitation (mainly on dislocations), of which the particles may be observed in fig. 3b. Besides that the heating up to this temperature doesn't result in any other changes in the structure.

The changes of the structure as the result of heating up to 280° C are presented in fig. 3c. There are noticeable in this microphotograph (TEM), the precipitated during first stage of tempering, the ε carbides, which are still present in the structure of test steel. After heating to 280° C there were also observed the independent nucleation of cementite on the borders of martensite strips and still existing residual austenite [15].

The heating to 650° C (fig. 3d) has initiated cementite decomposition and independent nucleation of hardly identified alloy carbides of MC type. The fig. 3d presents the cementite surrounded by alloy carbides of MC type. After such tempering there was no residual austenite observed in the structure. There was also found no other type of alloy carbides.

5.Conclusions

During heating of samples of quenched steel 30CrMoNiV10-25-6-2 the presence of four basic transitions was found, i.e. precipitation of ϵ carbide, precipitation of M₃C, transformation of retained austenite and precipitation of alloy carbides of MC and M₂C type.

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

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.

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