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TEM studies of tempered structural steels with Ni

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Methodology of research

<u>ABSTRACT</u>

Purpose: The reason for writing this paper was to describe the influence of Ni addition on the microstructure of structural steels after tempering. In this investigation, four model alloys of the variable concentration of Ni and constant concentration of carbon and other elements were used.

Design/methodology/approach: The alloys were melted in air and hot forged into bars 20×35 mm. The samples for investigations were machined after normalizing. All samples for TEM investigations were used in quenched and tempered conditions. Quenching was performed in oil at room temperature. After quenching samples were tempered at 200°C for 2 h. The microstructure of the investigated steels were investigated using JEM200CX transmission electron microscope.

Findings: An increase of Ni content in investigated structural steels causes a decrease of ε carbide concentration in their microstructure after tempering at 200°C for 2 hours. Cementite precipitates in these steels independently on the boundaries of martensite needles boundaries and on the twin boundaries in the areas in which the precipitates of ε carbide dissolved.

Research limitations/implications: Presented results may be used to design new technologies of tempering of structural steels with nickel addition.

Originality/value: Morphology of ε carbides precipitates.

Keywords: Electron microscopy; Structural steel; Nickel; ε carbide

1. Introduction

Nickel is known to increase the resistance to cleavage fracture of iron and reduce a ductile-to-brittle transition temperature. The medium-carbon low-alloy martensitic steels attain the best combination of properties in a low tempered condition, with tempered martensite, retained austenite and transition carbides in the structure [1]. Nickel decreases the ductile to brittle transition temperature by promotion of a cross-slip of dislocations in ferritic [2,3] as well as martensitic steels [4]. This effect promotes deformation rather than cleavage fracture and therefore increases toughness. Also, nickel is known as the alloying element, which slightly increases the hardness of martensite and has a weak effect on hindering the decrease of hardness with the tempering temperature [5-7].

Nickel enlarges the γ phase region in Fe-C phase diagram and therefore enables lower austenitizing temperature of steel, which can promote refinement of structure. Decreasing in the martensite packet diameter similar to the decreasing of the ferrite grain size improves the strength as well as toughness of steel [8]. Nickel can also influence increasing a stability of retained austenite [9,10] and the morphology of cementite precipitation at tempering [11].

The work presented here is concerned with the quantitative nickel effect (in the range from 0.35 to 4.00%) on microstructure (TEM) of model alloys in the quenched and tempered condition.

2. Experimental procedure

In this investigation the authors used four model alloys of constant concentration of C (about 0.30%) and the variable concentration of Ni ($0.35 \div 4.00\%$). The concentration of other elements was kept constant. Table 1 presents the chemical compositions of tested alloys.

All samples for TEM investigations were used in quenched and tempered condition. The austenitizing temperatures were determined in [12] as $Ac_3 + 50^{\circ}C$. Quenching was performed in oil at room temperature.

After quenching samples were tempered at 200°C for 2 h.

The microstructure of the investigated steels were investigated using JEM200CX transmission electron microscope.

3. Investigation results and discussion

According to CCT diagrams from work [12], all investigated alloys had enough hardenability to get full martensitic microstructure in cross-section of tested samples after oil quenching. An increase of Ni content gave also an increase of the amount of retained austenite [13,14].

The microstructure (TEM) investigations (Fig. 1-4) for tested alloys nos. 1 and 2 shows the ε carbide according to CHT diagrams presented in Zając and Pacyna work [13,14]. The precipitates of ε carbide are uniformly distributed inside the tempered martensite needles (Figs 1, 2, 3a and 4). Figs 2b and 4b show ε carbide precipitates observed in a dark field. They have a shape of a needle, with approximate length of up to 0.2 µm. However, the precipitates of ε carbide were not observed in each tempered martensite needle (Fig. 3b).

In the case of alloys 3 and 4 the number of areas, in which the precipitations of similar morphology (as in the case of alloys 1 and 2) were observed, was much smaller (Fig. 5 and 6). It may suggest, that in these alloys ϵ carbides dissolve during tempering at 200 °C. It is in agreement with mentioned above dilatometric investigations of these alloys [13,14]. Nevertheless, it has to be pointed out, that an influence of Ni on hardness after tempering, analysed in work [15,16] showed, that the alloys 3 and 4, hardened and tempered at 200 °C, have higher hardness than alloys 1 and 2. Also, an ultimate tensile strength (UTS) of alloy 3 after hardening and tempering at 200 °C was significant higher than that of similar heat treated alloy 1, and reduction of area (R_A) for the samples made of alloys 1 and 2 was higher than for alloys 3 and 4 [15,16].

Table 1.	
Chemical compositions of the tested alloys (mas	s %



Fig. 1. Microstructure of alloy 1 (0.35% Ni) after hardening and tempering at 200°C. Bright field



Fig. 2. Microstructure of alloy 1 (0.35% Ni) after hardening and tempering at 200°C: a) bright field; b) dark field; c) diffraction pattern; d) diffraction pattern description

The area of nucleation of the precipitates of cementite was revealed in the microstructure of hardened and tempered alloy 2 (Fig. 3b). This cementite nucleates on the grain boundaries of prior austenite and also on twin boundaries inside martensite needles. It is in agreement with the investigations of other researchers, pointing out an independent nucleation of cementite after dissolution of ε carbide.

Alloy No	С	Mn	Si	Р	S	Cr	Ni	Мо	Al	Cu
1	0.27	0.78	0.24	0.022	0.019	1.84	0.35	0.40	0.003	0.142
2	0.28	0.75	0.24	0.015	0.018	1.69	1.02	0.40	0.021	0.057
3	0.37	0.72	0.12	0.010	0.014	1.60	2.48	0.36	0.028	0.019
4	0.29	0.79	0.14	0.024	0.024	1.40	4.00	0.36	0.023	0.158



Fig. 3. Microstructure of alloy 2 (1.02% Ni) after hardening and tempering at 200°C. Bright field



Fig. 4. Microstructure of alloy 2 (1.02% Ni) after hardening and tempering at 200°C: a) bright field; b) dark field; c) diffraction pattern; d) diffraction pattern description



Fig. 5. Microstructure of alloy 3 (2.48% Ni) after hardening and tempering at 200°C. Bright field



Fig. 6. Microstructure of alloy 4 (4.00% Ni) after hardening and tempering at 200°C. Bright field

However, as it can be noticed, cementite nucleates already when significant amount of carbon is bounded in precipitates of ε carbide.

Moreover, an increase of Ni concentration in investigated alloys caused decrease of martensite needles dimensions and increase of twinned martensite content. An example of twinned martensite, observed in bright and dark field along with diffraction pattern description, is shown in Fig. 7.

TEM investigations did not reveal the presence of retained austenite in the alloys tempered at 200 °C for 2 hours. It may be due to very small amount of such austenite after hardening. According to the results obtained from quantitative X-ray analysis by Zając and Pacyna [15,16], the volume fractions of retained austenite in the microstructure of quenched alloys were 1.37% in the case of the lowest Ni content alloy (no. 1) and 3.54% in the case of the highest Ni content alloy (no. 4). Moreover, retained austenite may undergo destabilization during thin foil preparation [17].



Fig. 7. Microstructure of alloy 4 (4.00% Ni) after hardening and tempering at 200°C: a) bright field; b) dark field; c) diffraction pattern; d) diffraction pattern description

4.Conclusions

The investigations of the effect of nickel on the microstructure of structural steel alloys in quenched and tempered condition lead to the following conclusions:

- an increase of Ni content causes:
 - acceleration of dissolution of ε carbide,
 - decreasing the dimension of martensite needles,
 - increase of twinned martensite content,
- ε carbide precipitates in a form of a needle, with approximate length of up to 0.2 μm and is uniformly distributed in martensite needles,
- cementite nucleates independently on the grain boundaries of prior austenite and also on twin boundaries inside martensite needles. It nucleates already when the precipitates of ε carbide exist in the adjacent needles of martensite.

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