STRUCTURE EVOLUTION OF 33NiCrMoV15 STEEL IN RELATION TO TEMPERING TEMPERATURE

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Abstract

The paper deals with the microstructure evolution and related properties changes of middle alloyed high strength 33NiCrMoV15 steel treated by various tempering temperatures in range from 200° to 650° C in order to optimize the treatment process. All tempering processes were simulated on real experimental samples with using of dilatometer DIL805A. Afterward, the samples were observed by light microscopy to describe microstructure after every tempering process. Finally, some structure constituents were investigated in more details by in-situ SPM scan and their local mechanical properties were evaluated by quasistatic nanoindentation. Finally, measured constituents were classified according to acquired data. Both SPM scan and nanoindentations were performed by nanoindenter Hysitron TI950.

Keywords: high strength steel; tempering; dilatometric analysis; nanohardness; reduced Young modulus

1. Introduction

Most steels that are hardened by quenching are subsequently subjected to the tempering process what involves controlled heating of the quenched work-piece to a temperature below the lower critical temperature (A_1). Tempering improves the toughness of martensitic microstructures but lowers strength and hardness. The most important structural changes during the tempering process involve the formation of various distribution of ferrite and carbides as the supersaturation of as-quenched martensite is relieved and equilibrium mixture of phases are approached with increased tempering intensity [1]. Certain alloying elements also have influence on the resulting microstructure after the tempering. The most effective elements in this regards are strong carbide formers such as chromium, molybdenum and vanadium which retard the rate of softening during tempering [2], [3], [4].

Investigated steel belongs to the group of ultra-high strength middle alloyed martensitic steels. Conventional heat treatment with commonly used parameters are often not suitable for high strength steels as some author shows [5], [6]. The 33NiCrMoV steel combine high strength with good ductility, fatigue strength and creep resistance. One of the main applications of the steel is howitzer gun barrel production where the described properties are advantageously employed.

The paper presents the evaluation of microstructure of the 33NiCrMoV steel after its processing by various tempering treatments from low-temperature to high-temperature range. In NiCrMoV type of steels, the presence of some types of

carbides based on Fe, Ni, Cr, Mo, or V has been reported such as M₂C, M₇C₃, M₂₃C₆ and M₃C [7], [8]. These particles contribute to grain retirement by retarding the recrystallization of austenite and cause the strengthening by this mechanism [9].

2. Experimental procedure

2.1. Experimental material

The base material used for this paper is a middle alloyed 33NiCrMoV15 steel used in weapon industry. Raw material was cast as bars subsequently forged, vacuum remelted and heat treated by quenching and tempering. At first, chemical composition of selected steel was verified. CCD-based Spectro Jr CCD optical emission spectrometer was used to measure chemical composition of the steel (Table 1). Mechanical properties of experimental materials were also verified. Vickers hardness measurement (HV5) of experimental samples was performed, using Instron-Wolpert testing device with loading 49.03 N and indentation time $t = 10$ s (EN ISO 6507-1). Standard tensile strength test (EN ISO 6892-1) was performed to acquire strength characteristics, while the standard Charpy Impact test (EN ISO 179-1) was used to measure toughness. All mechanical properties acquired by these measurements are summarized in Table 2. Experimental samples in the shape of a cylinder with dimensions $\varnothing 4 \times 10$ mm were made of the experimental steel which were subsequently subjected to dilatometric analysis (see chapter 2.2.).

C	Mn	Si	Cr	Ni	Mo	V	Fe
0,409	0,558	0,381	0,926	2,855	0,221	0,1363	bal

Table 1. Chemical composition of experiment steel (wt%)

Tensile strength R_m [MPa]	Proof stress 0.05 $R_{p0.05}$ [MPa]	Toughness KCU [J] at 20°C	Hardness [HV5]	Elongation A_5 [%]
1500	1079	23	500	20

Table 2. Basic mechanical properties of experimental steel

2.2. Heat treatment of experimental samples

Quenching dilatometer DIL805A was used to simulate the heat treatment of experimental samples in real conditions. Experimental sample required for the treatment by dilatometer is shown in Fig 1. The sample is inserted to the work chamber of the dilatometer and then subjected to the programmed thermal cycle. Heating phase of the cycle is performed by induction heating under vacuum and cooling phase with using of helium as protective and cooling gas. There are used equal quenching parameters for all experimental samples consist of slow heating (1°C per second) to the austenitizing temperature (850°C) with holding period on the temperature (30 min) to provide complete austenitizing of sample volume followed by stepped rapid cooling in order to create martensitic structure in whole sample volume. Next phase of the thermal processing was the tempering process where eight tempering temperatures were used in range from 200°C to 650°C ($200, 250, 300, 450, 500, 600$ and 650°C).

Fig. 1. Shape and dimensions of the experimental sample for DIL805A

Fig. 2. Thermal treatment used for heat treating of the samples

Holding period during tempering was the same for all used temperatures (120 min). Used quenching and tempering treatment are schematically depicted in Fig 2. As a result of this processing, eight different groups of the experimental samples were prepared.

2.3. Microstructure analysis and hardness measurements

The microstructure of each experimental sample previously subjected to the specific thermal regime in dilatometer was subsequently observed by optical microscopy. The samples were treated by a standard metallographic procedure consisting of grinding, polishing and etching by Nital (3% HNO₃ in ethanol) to emphasize the microstructure. After that, the hardness of all samples was evaluated by standard Vickers hardness test with $F = 49,03$ load force and $t=10$ s indentation time. The samples were indented in the center line to measure the core hardness and final hardness of the sample was evaluated as the average value from three measurements.

2.4. Quasistatic nanoindentation

Quasistatic nanoindentation and SPM in-situ scanning was performed on Hysitron TI-950 measuring device equipped with Triboscan software. All nanoindentation tests were performed at room temperature inside the CEDITEK Laboratory of Mechanical testing in Trencin (Faculty of special technology). Nanoindentation tests were performed in purpose to determine nanomechanical properties of some specific phases and structure constituents after different heat treatments of experimental specimens. Samples for nanoindentation were chosen with the help of metallographic analysis and specific places or structure constituents on the sample surface have been identified by in-situ SPM scanning included in the nanoindenter system.

Quasistatic nanoindentation tests involve pushing a diamond tipped indenter head into a material under either load or displacement control. Displacement (h) is monitored as a function of the load (F) throughout the load-unload cycle where resulting relation $F-h$ is known as the nanoindentation curve. Plastic part of deformation is typically used to determine Young's Modulus, while the elastic-plastic part along with indent surface is used to evaluate hardness. The area bounded by both loading and unloading curves is equivalent to dissipation energy [10]. During all performed nanoindentation tests, load together with displacement were recorded, while Cube Corner indenter was pressed onto the measuring specimen surface with the standard loading and unloading profiles. The young modulus E_r (Pa) was obtained with the initial unloading stiffness S ($N \cdot m^{-1}$). Stiffness contact is then related to the reduced Young modulus E_r according to the Eq. (1):

$$E_r = \frac{\sqrt{\pi}}{2\beta} \frac{S}{A_c} \quad (1)$$

whereas β [m^{-1}] is a constant which depends on the geometry of used Cube Corner indenter and A_c [m^2] is the contact area of indentation. For all performed indentation standard trapezoid load curve with the maximum test load $F = 2000 \mu N$ and $t = 2$ s holding period is used. These parameters have been proven in previous research focused on high strength 33NiCrMoV15 steel [11].

3. Results and discussion

3.1. Evolution of the microstructure and corresponding hardness course in relation to the tempering temperature

Before nanoindentation tests, all treated samples were observed by standard metallographic analysis. There are shown all microstructures corresponding to all used tempering temperatures (T) in Fig. 3. The microstructures for tempering temperature from 200° to 300 °C (Fig. 3a÷d) are full martensitic where the amount of tetragonal martensite decreased with increasing of the temperature as an effect of transformation of supersaturated tetragonal martensite to tempered cubic martensite. This temperature range corresponds with low temperature tempering. The martensite is almost fully transformed to ferritic – carbidic mixture from $T=400$ °C where grain coarseness increases with temperature increase (Fig. 3e÷h). Also some alloys element carbides are observed in the structure for $T=500^\circ$ and $600^\circ C$. This indicates the effect of secondary hardening.

The surface hardnesses of samples treated by each of the used tempering processes were measured. The values of the hardness HV5 versus tempering temperature (Fig. 4) also supports the conclusions from the metallographic analysis. Hardness decreases with the increase of temper temperature with exception in high temperature area from $T = 500$ °C where some increase in hardness occurs. The experimental steel consists of considerable content of carbide-forming alloying elements that diffuse at elevated temperature ($500^\circ - 600^\circ C$) and form new carbides. However, hardness values increased by secondary hardening do not reach the hardness of initial martensitic structure in this case.

Measured hardness course does not reflect the possibility of high temperature reversible or low temperature irreversible temper embrittlement. Toughness versus temperature course has to be measured to detect these negative effects. However, fracture properties cannot be evaluated with using of the small cylinder samples ($\varnothing 4 \times 10$ mm) required by the dilatometer. This goal will be met as a separate part of the research project focused on properties and behaviour of 33NiCrMoV15 under high load.

Fig. 3. Evolution of microstructure in relation to the tempering temperature:
a) 200 °C, b) 250 °C, c) 300 °C, d) 400 °C, e) 450 °C, f) 500 °C, g) 600 °C, h) 650 °C

Fig. 4. Course of hardness HV5 versus tempering temperature

3.2. Nanoindentation study of selected structure components

Two characteristic types of the treated samples were chosen for quasistatic nanoindentation. First one was tempered at 200 °C with tempered martensite structure what corresponds with low temperature tempering (Fig. 5a, b). The treatment of second one corresponds to high temperature tempering at 600 °C with ferritic – carbide mixture microstructure as a result (Fig. 5a, b). The sample surfaces were first scanned by SPM (Scanning probe microscopy). To form images, indenting tip oscillates with specific frequency over the surface while scanning the selected area without causing any physical changes.

The SPM scans acquired for these two samples are depicted in Fig. 5. In both cases, first $50 \times 50 \mu\text{m}$ area was scanned as an overall view. Then a detailed scan of selected $10 \times 10 \mu\text{m}$ area was performed. All further described structure constituents were classified according to their measured local mechanical properties.

Scan area in Fig. 5a consists of tempered martensite mainly. Nanoindenting locations were chosen in some martensitic needles (M1÷M3). Detailed scan (Fig. 5b) shows some polygonal particles where three of them (P1÷P3) were chosen as indentation location. Last two indents in this area are also located in martensitic needles (M4, M5). Measured nanohardness and reduced Young modulus of all described locations are shown in Table 3. Based on measured local mechanical properties, the particles are assumed as carbides of alloying elements.

Figure 5c shows $50 \times 50 \mu\text{m}$ scan of the structure after tempering at 600°C . Ferritic grains with some carbides on their boundaries are observable in the scan. Locations in the grains (FG1, FG2) and a particle (P4) were selected as nanoindentation locations. Detailed scan in Fig. 5d shows particles diffused on grain boundaries within secondary hardening (P5÷P7). The places that appeared on the scan as dark filled places (FG3, FG4) are etched ferritic structure. Measured nanohardness and reduced Young modulus of all these locations are shown in Table 4.

Fig. 5. In Situ SPM scans of measured surfaces with indentation locations: a), b) tempering 200°C ; c), d) tempering 600°C

Location	a0	a1	a2	b0	b1	b2	b3	b4
H [Gpa]	15	17	15	27	24	23	14	13
Er [GPa]	376	377	382	408	449	336	380	352
Constituent (assumed)	M1	M2	M3	P1	P2	P3	M4	M5

Table 3. Local mechanical properties of microstructure constituents for tempering temperature 200°C

Location	c0	c1	c2	d0	d1	d2	d3	d4
H [GPa]	8	9	17	15	14	12	9	8
Er [GPa]	283	327	430	456	414	496	230	314
constituent (assumed)	FG1	FG2	P4	P5	P6	P7	FG3	FG4

Table 4. Local mechanical properties of microstructure constituents for tempering temperature 600°C

4. Conclusion

Microstructures of 33NiCrMoV15 steel obtained by its quenching with same parameters and then tempering at eight different tempering temperatures were evaluated in the paper. Evolution of the microstructure in relation to the tempering temperature as well as hardness course corresponds with general knowledge about phase transformation during tempering of steel. Tempering at low-temperature range ($200^\circ \div 300^\circ\text{C}$) led to tempered martensite microstructure while high-tempering temperature ($500^\circ \div 650^\circ\text{C}$) produced ferritic – carbidic mixture. However, investigated steel consists of considerable content of alloying elements (Cr, V, Mo, Ni) which forms carbides with a noticeable effect on whole heat treatment process as a structure softening factor at high temperatures. The measured course of hardness HV5 shows secondary hardening in temperature range $500^\circ \div 600^\circ\text{C}$ also caused by carbide presence. However, hardness elevated by the secondary hardening effect do not reach the hardness values of martensitic structure measured after low-

temperature tempering. Values of nanohardness and reduced Young modulus measured on some structure constituent for both low and high temperature tempering confirm all these conclusions.

Next phase of this research will be focused on an evaluation of toughness and related fracture properties of investigated steel what cannot be realised within the experiment presented in the paper. Measured hardness course does not reflect the possibility of reversible or irreversible temper embrittlement development.

Complete information about behaviour and properties of investigated steel after heat treatment can only be obtained by combining the presented research with planned investigation of its plastic and fracture properties.

The main purpose of complete research consisting of quenching, tempering and thermomechanical treatment of this steel and related behaviour and changes in properties is to optimize heat treatment parameters to achieve mechanical properties as a combination of high strength without decreasing of plastic properties (toughness). These properties are required for extreme high shock strain-rate loadings

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6. References

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