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Designing and controlling the microstructure of 37MnNiMo6-4-3 hypoeutectoid steel after continuous cooling

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<u>ABSTRACT</u>

Purpose: Present work corresponds to the research on the kinetic of phase transformation of undercooled austenite of 37MnNiMo6-4-3 hypoeutctoid steel. The kinetic of phase transformation of under cooled austenite of investigated alloy was presented on CCT diagram (continuous cooling transformation). Also the methodology of a dilatometric samples preparation and the method of the critical points determination were described.

Design/methodology/approach: The austenitising temperature was defined in a standard way i. e. $30-50^{\circ}$ C higher than Ac₃ temperature for hypoeutectoid steels. The technology of full annealing was proposed for the iron based alloy. The CCT diagram was made on the grounds of dilatograms recorded for samples cooled with various rates. The microstructure of each dilatometric sample was photographed after its cooling to the room temperature and the sample hardness was measured. Also EDS analysis was performed using scanning microscope.

Findings: The test material has been hypoeutectoid steel. These steels represent a groups of alloy steels for quenching and tempering. The microstructure of test 37MnNiMo6-4-3 hypoeutectoid steel on CCT diagram changes depending on the cooling rate.

Research limitations/implications: The new hypoeutectoid steel and new CCT diagram.

Practical implications: The paper contains a description of one from a group of iron based model alloys with 0.35-0.40% carbon content. According to PN-EN 10027 standard this steel should have a symbol 37MnNiMo6-4-3.

Originality/value: The new hypoeutectoid steel (Mn-Ni-Mo iron based model alloy). **Keywords:** Metallic alloys; Hypoeutectoid steel; CCT diagram; EDS analysis

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1. Introduction

The aim of this study was to determine the appropriate austenitizing temperature T_A of 37MnNiMo6-4-3 hypoeutectoid steel and the kinetic of phase transformation of undercooled austenite of tested steel [1-12]. These steels represent a groups of alloy steels for quenching and tempering [13, 14]. The hypoeutectoid steels belong to elementary materials used for production of construction for metal forming. The basic requirements for these steels are: sufficient hardenability, wear resistance as high as possible strength and fracture toughness.

The analysis of microgradients of chemical compositions seems to be important for these steels. It should be noted that the interaction of two or more alloying elements is significantly different from the sum of effects of these elements added separately. The most important may be the common effect of molybdenum and chromium, molybdenum and nickel, chromium and nickel, manganese and chromium, manganese and nickel, manganese and molybdenum, manganese and cobalt. Until now, the impact of each element was considered separately, only sometimes, pointing to a group of alloys, in which this interaction was evaluated.

Therefore, this work concerns the kinetics of transformations of undercooled austenite in hypoeutectoid steels. The tests are aimed at a preliminary analysis of the impact of three elements such as manganese, nickel and molybdenum on the microstructure of the tested alloy.

2. Experiments procedure

The chemical composition of the hypoeutectoid steel was designed in the Laboratory of Phase Transformations, Department of Physical and Powder Metallurgy, AGH University of Science and Technology.

The microstructure of the investigated material was examined by the light microscope Axiovert 200 MAT

The hardness measurements were performed with the Vickers HPO250 apparatus.

The research of kinetics of the phase transformations of the undercooled austenite has been performed with a dilatometric method. The Continuous- Cooling-Transforation (CCT) diagram was made by means of a L78R.I.T.A. optical dilatometer.

EDS analysis was performed using Hitachi SU-70 scanning microscope at the Department of Non-Ferrous Metals AGH in Krakow.

3. Material for investigations

This hypoeutectoid steel was melted and cast in the Faculty of Foundry Engineering of AGH University of Science and Technology in Cracow then reforged in INTECH-MET in Gliwice. According to PN-EN 10027 standard this steel should have a symbol 38MnNiMo6-4-3. Chemical compositions of this steel is given in Table 1.

The microstructure of tested steel after forging is shown in Figure 1. It is clear that the microstructure of the tested alloy after forging consists of ferrite and pearlite. Ferrite is precipitated along

grain boundaries of the former austenite in Widmannstätten structure. Hardness of the studied steel after forging is 197 HV30.

Table 1.

The chemical composition (wt. %) of the investigated steel

| С | Mn | Мо | Ni | Si | Р | S | Cu | Al |
|------|------|------|------|-------|---------|---------|-------|-------|
| 0.37 | 1.50 | 0.31 | 1.01 | 0.050 | < 0.001 | < 0.010 | 0.030 | 0.017 |

In order to determine the correct critical temperatures (break points) for the investigated 37MnNiMo6-4-3 steel after forging, the heating at the rate of 0.05° C/s to a temperature of 1100° C and then cooling at a rate of 1° C/s to a room temperature with dilatometric method was performed. The determined critical temperature A_{C3} of the steel (after forging) was as following: 790°C.



Fig. 1. Microstructure of the 37MnNiMo6-4-3 steel after reforging. Etched by 3% nital

The next step of research was to make a full annealing, which was performed in a laboratory oven Carbolite RHF16/19. A sample of the tested alloy was heated to a temperature of 850°C, hold 2 hours, and then cooled at a rate of 3° C/min to 500°C and further cooled at a rate of 30° C/min to a room temperature.

a)



Fig. 2. Microstructure of 37MnNiMo6-4-3 after full annealing. Etched with 3% natal

Microstructure of 37MnNiMo6-4-3 after such annealing is shown in Figure 2. It is clear that the microstructure is ferropearlitic. Ferrite is precipitated in Widmannstätten structure, similarly as in case of steel after forging. Hardness of the studied steel is 186HV30.

Again, a set of break points for the investigated 37MnNiMo6-4-3 hypoeutectoid steel after such annealing was determined, which is respectively: $Ac_{1s} = 690^{\circ}C$, $Ac_{1f} = 750^{\circ}C$, $Ac_{3} = 800^{\circ}C$.

The next step of research was to determine the kinetics of phase transformations of undercooled austenite in tested steel with dilatometric method using a L78 R.I.T.A. dilatometer. Individual cooling curves were differentiated, allowing for precise temperature determination of the beginning and end of each transformation. All obtained cooling curves allowed for plotting the CCT diagram. Austenitizing temperature for 37MnNiMo6-4-3 steel was assumed as $T_A = 850^{\circ}$ C, i.e. 50°C above the Ac₃ temperature.

To draw the CCT diagram one used samples with dimensions of Φ 3 x 10 mm form annealed condition (detailed technology of annealing the test steels is described in Chapter 3 of this work). The samples were heated at the rate of 5° C/s up to the austenitizing temperature in this case T_A = 850°C, annealed for 20 min, followed by cooling at different rates (within the range of 47°C/s-1°C/s for 37MnNiMo6-4-3 steel). Then, each of dilatometric samples was subjected to hardness testing three times using Vickers hardness tester type HPO 250, with indenter load of F=30 kG. Hardness measurement results are found in the CCT diagram of test hypoeutectoid steel.

On the other hand, the analysis of chemical composition of the phases present in the studied hypo-eutectoid steels for different cooling rates was made by electron microprobe, called X-ray microanalyser. It allows to determine the chemical composition of selected micro-area of micro-section of a fixed diameter or fixed length. After placing the test samples in the chamber and reaching a desired level of vacuum, using light microscope one searched the spots for the analysis which was then made with EDS technique.



Fig. 3. The CCT diagram of 37MnNiMo6-4-3 steel

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a) Cooling rate = 47° C/s 548 HV30



c) Cooling rate = 10° C/s 212 HV30





d) Cooling rate = 5° C/s 198 HV30



d) Cooling rate = $1^{\circ}C/s$ 156 HV30





Fig. 4. Microstructures of the 37MnNiMo6-4-3 hypoeutectoid steel after dilatometric test. Etched with 3% nital

4. Research results and discussion

Figure 3 contains a CCT diagram of 37MnNiMo6-4-3 steel after austenitizing at temperatures $T_A = 850^{\circ}$ C. It was supplemented by metallographic examinations, which are given in Figure 4.

As can be seen, the transitions start curves are C-shaped, while the ends of the curves indicate the extended time of their end. According to the classification of Wever and Rose [15] this chart is of type III. For the austenitizing temperature $T_A = 850^{\circ}C$ the beginning of ferrite precipitation as well as of the pearlite was observed already on the cooling curve of $25^{\circ}C/s$ while the intermediate transition is observed on the cooling curves $47^{\circ}C/s - 10^{\circ}C/s$. Martensitic transformation start temperature Ms = $365^{\circ}C$. It should be noted that diffusion transformations, despite the presence in steels' composition of about 1% Ni, are shifted to shorter times, probably due to the influence of manganese and nickel (austenite stabilizing elements) on significant lowering of diffusion transitions temperatures (precipitation of ferrite and pearlite formation), which could significantly speed up the transformation of austenite.

It is a unique phenomenon, not yet "raised" by undercooled austenite phase transformations experts, observable in steels having a model (special) chemical composition. This means, that in case of elements which open the austenite field, i.e. Mn and Ni, their effect on hardenability is reduced by lowering the temperature of supercooled austenite transformation. It is possible that in the same way one would explain the influence of Co, which as everyone knows, reduces the hardenability. This may be due to a strong reduction, by this element, of range of diffusion transformations of austenite, which as one can notice based on the results presented in this study, results in shortening the time of transformation start.

Below, Figure 4, presents the microstructures of dilatometric samples of 37MnNiMo6-4-6 test steel used for drawing of the CCT diagram (compare Figure 3).

As can be seen, the microstructure of samples of 37MnNiMo6-4-3 steel, cooled at the highest rate (of about $47^{\circ}C/s$) contains mainly martensite. After applying the cooling of about $25^{\circ}C/s$ next to the martensite there appears bainite as well as ferrite and pearlite in the microstructure. However, after cooling at the rates in the range of $10^{\circ}C/s$ to $1^{\circ}C/s$ the microstructure of test steel is consisting mainly of pearlite and ferrite, which sometimes forms Widmannstätten structure again. It can be seen that the steel containing up to 1% of Ni is particularly prone to precipitation of ferrite in Widmannstätten structure.

The next stage of the study was the EDS analysis for selected cooling rates of the investigated hypo-eutectoid steels. For this purpose, the samples used for the dilatometric tests were etched again to expose the particular structural components of the test steel.

For 37MnNiMo6-4-3 steel two cooling rate were selected: 50°C/s and 1°C/s (compare Figure 3), where, for the above mentioned rates, the analysis of distribution of the alloying elements present in the investigated steel in areas of former austenite grains and their boundaries has been made (see Figure 5-6). The analysis of EDS did not register any significant changes (micro-gradients) of alloying elements in the area of the former austenite grains for selected cooling rates.

It should be noted that the above-presented studies are the beginning only of an accurate (detailed) analysis of the effects of

alloying elements and of evaluation of their microgradients within the group of temperable steels.

a) microscope scanning, cooling rate = $50^{\circ}C/s$



b) distribution of alloying elements: Ni and Mo made EDS analysis



c) distribution of alloying elements: Mn and Mo made EDS analysis



Fig. 5. Distribution of alloying elements: Mn, Ni and Mo made EDS analysis for 37MnNiMo6-4-3 hypoeutectoid steel. $T_A=850^{\circ}C$; cooling rate = 50°C/s

a) microscope scanning, cooling rate = $1^{\circ}C/s$



b) distribution of alloying elements: Ni and Mo made EDS analysis



Fig. 6. Distribution of alloying elements: Ni and Mo made EDS analysis for 37MnNiMo6-4-3 hypoeutectoid steel. $T_A = 850^{\circ}C$; cooling rate = 1°C/s

5. Summary and conclusions

In this paper, an analysis of the kinetics of phase transformations of super-cooled austenite in 37MnNiMo6-4-3 steel with model chemical composition was made. A CCT diagram, supported by the metallographic examinations was prepared. Then an analysis of the distribution of alloying elements: Mn, Ni and Mo for different, selected cooling rates was made. The EDS analysis was performed to reveal the changes in distribution of above mentioned alloying elements in the areas of the former austenite grains.

It can be concluded that the investigated steel has low content of alloying elements. Nevertheless, this affects the phase transformations (CCT diagrams) and temperability. The CCT diagram of test steel (according to the classification of Rose and Wever [15]) is of type III, which means that the curves of beginning of the diffusion transitions form a letter "C" and are not separated from bainitic transformation by the stability range of the supercooled austenite.

One should remember that both the manganese and molybdenum are the elements that have significant influence on the hardenability of steel (even with a weak background of alloying elements). It may be noted that the times to start bainitic transformation are prolonged. This results in a shift of the diffusion transitions (pearlite and ferrite) towards longer times (to the right in the CCT diagram). The addition of nickel to hypoeutectoid test steel in an amount of about 1%, however, did not cause a significant increase in the hardenability of the steel. This is most likely the result of the shift of diffusion transitions to lower temperatures.

The EDS analysis performed for the selected cooling rates did not reveal any changes in the distribution of alloying elements of hypo-eutectoid 37MnNiMo6-4-3 test steel. However, these studies, from the point of view of the effect of alloying elements (acting jointly) are only the preliminary assessment analysis (beginning) of the impact of the chemical composition gradient in micro-scale in iron-based alloys.

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