

THE EFFECT OF CARBIDES ON THE KINETICS OF BAINITIC TRANSFORMATION AND THE RESULTING NANOBAINITIC MICROSTRUCTURE IN 90SiCrMn6-4 STEEL

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Abstract

The aim of the work was to determine the influence of the quantity and distribution of carbides on the bainitic transformation process that takes place in 90SiCrMn6-4 steel. The kinds of possible carbides that can be formed in steel and the kinetics of their precipitation were examined by the use of computer simulations. Various thermal treatments, leading to obtaining of different size and distribution of carbides in steel, were carried out. The obtained microstructures were studied by the use of Light Microscopy (LM) and by Transmission Electron Microscopy (TEM). The pre-heat-treated steel samples with different carbide size and distribution were subjected to austempering processes. The kinetics of bainitic transformation in samples with different carbide size and distribution were analysed by means of dilatometric tests and the resulting microstructures were examined by use of TEM. The obtained microstructure was composed of carbide free bainitic matrix in which carbides were observed. Thickness of the bainitic ferrite plates and austenite layers, in samples with different carbide distributions, was measured. Finally, hardness of the heat treated samples was determined.

Keywords: Steel, bainitic matrix, carbide precipitations, nanostructuring process

1. INTRODUCTION

An effective method used to ensure high hardness and plasticity in steels is the reduction of the size of phase grains. Other methods that improve hardness lead to diminishing of plasticity of steel. This effect significantly reduces suitability of such steels in many applications [1-4]. Unprecedented combination of high strength, fracture toughness and ductility was obtained in a new generation of nanobainitic steels [5]. A nanobainitic microstructure can be achieved in steels containing an increased content of carbon (0.6 ÷ 1.1 wt.%) and silicon (c.a 1.5 wt.%) by the use of specially designed heat treatment. This treatment consists of the following steps: austenitization, followed by rapid cooling to the temperature lying in the lower range of the bainitic transformation zone, annealing at this temperature for the time necessary to complete the bainitic transformation, and, finally cooling to ambient temperature. It allows the formation of the nanobainite composed of bainitic ferrite plates of nanometric thickness separated by thin layers of the carbon-enriched retained austenite [1,4].

For some applications (machine - building industry, tool industry) the hardness of nanobainitic steels is too low, which excludes the use of these attractive steel grades. In this work, we have assumed that greater hardness and abrasion resistance can be achieved through the formation in steel a nanobainitic matrix containing highly dispersed carbides. Grained carbides will significantly increase hardness and wear resistance. The aim of this work was to create carbide free bainitic matrix containing hard carbides through specially designed heat treatment. The two types of heat treatment is designed and the influence of the quantity and distribution of carbides precipitation on the bainitic transformation process is determined.

2. EXPERIMENTAL

2.1. Material

The chemical composition of the steel investigated in this study is given in **Table 1**.

Table 1 Chemical composition of 90SiCrMn6-4 steel

Element	C	Mn	Si	P	S	Cr	Ni	Mo	W	V	Cu
Weight (wt%)	0.90	0.40	1.32	0.011	0.02	1.05	0.016	0.03	0.01	0.01	0.10

It can be noticed that the investigated steel contained about 1.35 wt.% of silicon which prevents the precipitation of carbides during bainitic transformation. The addition of Si together with increased content of carbon (0.9 wt.%) are essential for obtaining bainitic nanostructure in the steel. Furthermore, a low content of alloying elements makes 90SiCrMn6-4 steel relatively cheap.

2.2. Heat treatment

In order to properly design the heat treatment parameters, the kinetics of phase transformations in steel was determined through dilatometric tests performed in Bähr DIL 508 L dilatometer. Cylindrical samples with a diameter of $\phi = 3$ mm and drilled 4 mm x 3 mm and height $h = 10$ mm were used for the tests. The recording of extension changes, as a function of time in different temperatures, was carried out using the DIL805L PRO program, which is connected with the dilatometer. As a result, a TTT (Time-Temperature-Transformation) diagram was constructed. To determine characteristic temperatures of phase transformations, steel samples were heated to 1050 °C at the heating rate of 2 °C/min. and then cooled to room temperature. The critical temperatures were determined from the diagrams recorded during the cooling. The heat treatments were also conducted in a dilatometric furnace.

The aim of the research was to investigate the influence of the quantity and distribution of carbide precipitations on the bainitic transformation process that occurs in 90SiCrMn6-4 steel. In order to obtain samples with different carbides volume fraction, distribution and morphology, the annealing at the temperature between Ac_{1k} and Ac_{cm} was performed (Ac_{cm} is the boundary temperature for austenite - secondary cementite equilibrium, below which cementite starts to dissolve; Ac_{1k} is the temperature of the end pearlitic transformation). Two types of heat treatments leading to a variable amount and morphology of carbide precipitates were carried out. The heat treatment routes designed in this work followed the sequence displayed in **Figure 1**.

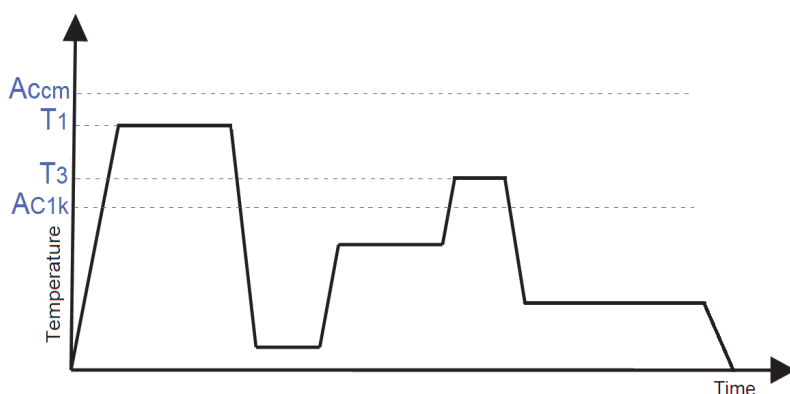


Figure 1 a) The scheme of the first heat treatment

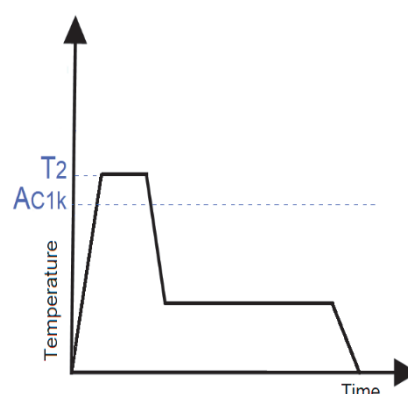


Figure 1 b) the second heat treatment

The first stage of first treatment consisted of heating the steel to austenitizing temperature T_1 (960 °C) lying about 20 - 30 °C below Ac_{cm} temperature, thereby causing a significant dissolution of the carbides (mainly cementite). Then, steel samples were quenched to room temperature, and then tempered (at 500 °C, 550 °C and 600 °C). Afterwards, the samples were heated to a temperature T_3 (880 °C) lying 20 - 30 °C above the Ac_{1k} temperature. The next stage involved the nanostructuring process that consisted of isothermal quenching of the samples at bainitic transformation temperature. For this purpose, steel was quenched from the temperature T_3 to a temperature lying about 20 - 30 °C above the M_s i.e.: martensite start temperature. In this variant of heat treatment the additional cementite particle precipitated during tempering. The computer simulations using the commercial software JMatPro showed, that only cementite can be formed in steel at used temperature range. The first stage of the second treatment consisted in heating the samples to austenitizing temperature T_2 (20 - 30 °C above Ac_{1k} temperature), which resulted in obtaining of austenite and carbides, which did not dissolve during austenitization. The second stage, similarly as in the first treatment, involved bainitic nanostructuring process that was performed at 20 - 30 °C above the M_s temperature. The duration of the individual processing steps has been determined experimentally on the basis of registered dilatometric curves.

2.3. The characterization of microstructure

The obtained microstructures were examined with the use of Light Microscopy (LM). Thin foils, cut from samples after bainitic nanostructuring processes, were subjected to the microstructural observations using JEOL 1200EX Transmission Electron Microscope (TEM), operated at 120 kV, to reveal the quantity and distribution of carbide precipitates in steel. Additionally, the thickness of the ferrite plates and the austenite layers was determined with the following formula:

$$d = \frac{2}{\pi} L \quad (1)$$

where: d - the real thickness of the ferrite plates or austenite layers and L - the width of the ferrite plates of the austenite layers measured on the TEM image [6]. The measurements of the volume fraction of the austenite were made by the magnetic saturation method.

2.4. The characterization of hardness

Hardness in Vickers scale was measured using the ZWICK/ROELL testing machine. The applied load was 19.6 N.

3. RESULTS

3.1. Selection of austenitization temperature and time

The Ac_{cm} and Ac_{1k} temperatures were 985 °C and 860 °C respectively. Consequently, to maintain a certain fraction of undissolved carbides, the austenitization was carried out at $T_1 = 960$ °C and $T_2 = 880$ °C. The use of different austenitizing temperatures allowed differentiation of the content and special distribution of carbides. During the austenitizing, a significant increase of the sample length was initially observed in the time-elongation curve. After reaching a maximum, the elongation begins to decline, which may indicate the dissolution of carbides. The value of time corresponding to the maximum elongation of the sample before the subsequent decrease in elongation was taken as the duration of the austenitizing process. For the sample austenitized at $T_1 = 960$ °C and $T_2 = 880$ °C, the austenitizing time was determined for 8 min and 10 min respectively. The values of M_s temperatures after the short (8 min and 10 min respectively) and long (1.5 h) austenitization time were compared. The shortening of the austenitizing time caused an increase in M_s temperature from 250 °C to 320 °C for $T_1 = 960$ °C and an increase from 220 °C to 270 °C for $T_2 = 880$ °C. Nanostructuring process

on the steel was conducted. For this purpose, steel was annealed at 20 - 30 °C above the M_s temperature. The austempering parameters were determined experimentally based on dilatometric curves. The austempering temperature and time values are summarized in the **Table 2** below.

Table 2 Heat treatment parameters

	Austenitization		Quenching	Tempering		Austenitization		Austempering	
	T (°C)	t (min)		T (°C)	t (min)	T (°C)	t (min)	T (°C)	t (h,min)
960_500	960	8	+	500	90	880	10	310	9h
960_550	960	8	+	550	90	880	10	335	7h50'
960_600	960	8	+	600	90	880	10	290	14h5'
880	880	10	-	-	-	-	-	290	12h45'

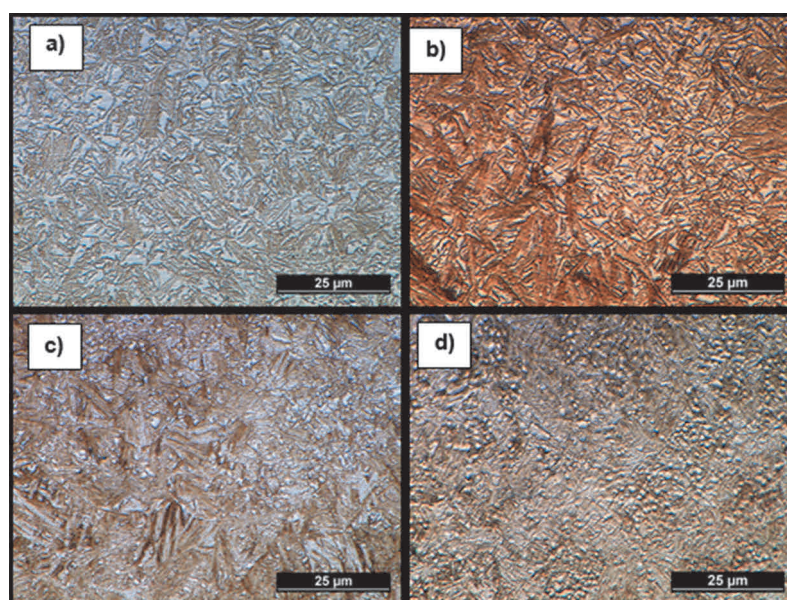


Figure 2 LM figures of microstructure obtained by heat treatment of 90SiCrMn6-4 steel after a) the 960_500 procedure, b) the 960_550 procedure, c) the 960_600 procedure, d) the 880 procedure

3.2. Microstructure

Observations made with the use of light microscope revealed that the microstructure after both variants of heat treatments led to obtaining different distributions of carbides in 90SiCrMn6-4 steel (**Figure 2**). The first variant of heat treatment (regardless of tempering temperature, **Figures 2 a, b, c**) resulted in a microstructure composed of retained austenite and bainitic ferrite plates which formed groups or packages. The ferritic bainite plates were either parallel to each other or arranged at different angles to each other. An increase in the tempering temperature resulted in increase of the size of carbides and decrease of areas of the retained austenite. The microstructure of sample after second heat treatment variant (**Figure 2d**) contains clearly visible, undissolved, relatively large carbides.

The microstructure, formed in 90SiCrMn6-4 steel after two selected heat treatments (marked with symbols: 960_500 and 880) was analysed by TEM. Typical images of the microstructure are shown in **Figure 3**. The matrix of steel after the 960_500 procedure was composed of bainitic ferrite plates surrounded by retained austenite. The thickness of bainitic ferrite plates varied between 50 nm and 248 nm (the mean value was 119 nm) and the thickness of austenite layers varied between 10 nm and 90 nm (the mean thickness was 37 nm). The volume fraction of retained austenite was 37.2 % \pm 2.7 %. Detailed TEM observations of the steel

microstructure after the first variant of heat treatment confirmed the presence of some secondary carbides (**Figure 3 a**), which did not dissolve during austenitizing. The fine spherical carbides which formed clusters were also present (**Figures 3 a, b**). In a microstructure obtained after second heat treatment marked with symbol 880, the bainitic ferrite plates and austenite layers had more differentiate size as compared to those which were present in the microstructure after first heat treatment (960_500). The thickness of ferrite plates varied between 16 nm and 306 nm (with the average value of 111 nm). In this microstructure austenite was present in form of layers between the ferrite laths and in form of blocks. The austenite volume fraction was $14.9 \% \pm 2 \%$, which was significantly less than in the case of the first variant of heat treatment. The thickness of the austenite layers varied between 9 nm and 159 nm (with the mean value of 61 nm). This microstructure contained also higher density of secondary carbides, which remained after the austenitizing at 880 °C below the boundary temperature for austenite (the temperature at which carbides begin to dissolve - 985 °C) in this steel (**Figure 3d**).

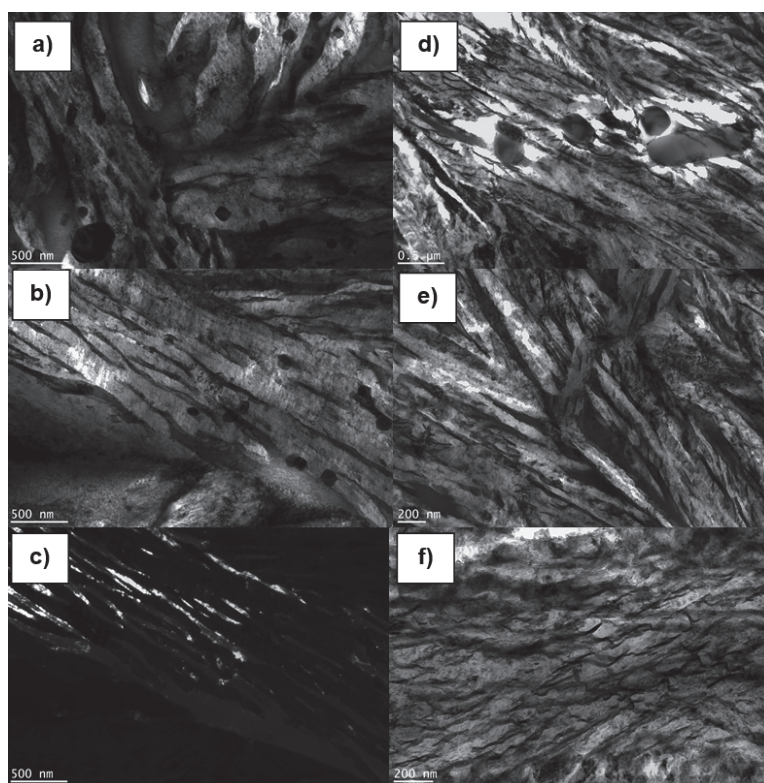


Figure 3 TEM image of microstructure of 90SiCrMn6-4 steel after nanostructuring processes marked with symbols: a), b), c) - 960_500, d), e), f) - 880

3.3. Hardness

Hardness and volume fraction of retained austenite data for the tested steel samples obtained in various heat treatment parameters are shown in **Figure 4**. Hardness of the tested steel after first type of heat treatment (marked with symbols: 960_500, 960_550 and 960_600) increased with an increase of tempering temperature. The increase in hardness is correlated with the decrease in austenite content. Austenite has a lower hardness compared to bainitic ferrite, so increasing the austenite content in the microstructure leads to a decrease in hardness of steel. The hardness of steel treated according to the 880 variant is slightly higher than the hardness obtained for the 960_600 variant. It could be caused by more refined microstructure obtained in the 880 variant as a result of applying lower austempering temperature. As may be observed in **Figure 4**, the austenite volume content has a tendency to decrease with the increase of the tempering temperature. The maximum volume fraction of retained austenite - $37.2 \% \pm 2.7 \%$ was reached after first type of heat treatment

using the lowest tempering temperature (500 °C). The application of higher tempering temperature led to higher volume fraction of carbides in the microstructure and thus to lower carbon in the austenite which is necessary to stabilize it during austempering. As a result, smaller volume fraction of retained austenite was observed in variants where more carbides occurred in the microstructure.

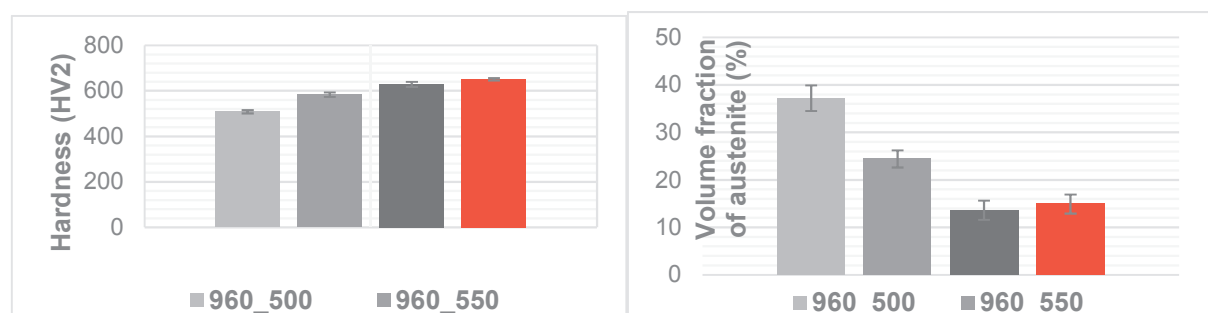


Figure 4 Hardness and volume fraction of retained austenite data for samples of 90SiCrMn6-4 steel after analysed heat treatments

4. CONCLUSION

As a result of specially designed heat treatments of 90SiCrMn6-4 the microstructure with carbide precipitates submerged in a carbide free bainitic matrix was obtained. The thickness of bainitic ferrite plates was submicron or nanometric depending on the heat treatment used. The austenite was in the form of thin, nanometric layers and in form of blocks. Depending on a heat treatment the retained austenite content in the microstructure varied in a wide range, which affected steel hardness. The obtained results show that by changing the content and morphology of carbides it is possible to control the phase composition and hardness of the steel.

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