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EFFECT OF HEAT TREATMENT ON MECHANICAL PROPERTIES AND MICROSTRUCTURE MORPHOLOGY OF LOW-ALLOY HIGH-STRENGTH STEEL

The paper analyzes the influence of different heat treatment processes on the mechanical properties of low-alloy high-strength steel denoted by Polish Standard (PN) as 10MnVNb6. One of the findings is that, after aging, the mechanical properties of rolled steel are high: the yield strength may reach > 600 MPa, and the ultimate tensile strength is > 700 MPa. These properties are largely dependent on the grain size and dispersion of the strengthening phase in the ferrite matrix. Aging applied after hot rolling contributes to a considerable rise in the yield strength and ultimate tensile strength. The process of normalization causes a decrease in the average grain size and coalescence (reduction of dispersion) of the strengthening phase. When 10MnVNb6 steel was aged after normalization, there was not a complete recovery in its strength properties.

Keywords: microalloyed steel, mechanical properties, microstructure

1. Introduction

10MnVNb6 microalloyed steel is classified as high strength low alloyed steel because of its chemical composition. The low carbon content is favourable for its good weldability [1]. The chemical composition of 10MnVNb6 steel was developed in the second half of the 20th century [2, 3]. As the steel exhibits high yield strength and ultimate tensile strength, it has been used, for instance, in the form of plates, in booms of truck-mounted cranes and STAR truck chassis frames [3]. In 1999, this steel was used to produce a trial batch of tubes (ϕ70 x 3.2 mm), which had good mechanical and technological properties [4]. The relatively high strength and plasticity are achieved as a result of grain refinement and precipitation strengthening of the ferrite matrix [5-8].

The yield strength and ultimate tensile strength of 10MnVNb6, which is a ferritic-pearlitic steel, is modelled by applying different strengthening mechanisms. The values of R_m and R_e are dependent, for example, on the friction impeding the dislocation motion, the degree of deformation of the ferrite crystal lattice by atoms of the alloy elements, the volume fraction of pearlite, the size of the strengthening phase particles, their dispersion, ferrite grain size, etc.

Assuming the additivity of various strengthening mechanisms, we can express their influence on the yield strength by [9]:

$$\sigma pl = \sigma i + \Delta\sigma f + \Delta\sigma p + \Delta\sigma od + \Delta\sigma cz + \Delta\sigma gz \qquad (1)$$

where: σ_i – stress due to friction impeding the dislocation motion, $\Delta\sigma_f$ – solution strengthening, $\Delta\sigma_p$ – strengthening due to the presence of pearlite in the microstructure, $\Delta\sigma_{od}$ – strengthening due to deformation (dislocation strengthening), $\Delta\sigma_{cz}$ – dispersion strengthening, $\Delta\sigma_{gz}$ – grain boundary strengthening ($k_y d^{-0.5}$).

The strengthening due to precipitation of the second phase (effect of the precipitation strengthening) is generally written as [10]:

$$\Delta\sigma_{cz} = 5.9 V_v^{0.5} \ln (r \cdot 10^{-4}/2.5) \qquad (2)$$

or [9]

$$\Delta\sigma_{cz} = (9.8 \cdot 10^3/l) \ln(2 \cdot l) \qquad (3)$$

where: V_v – volume fraction of precipitates, r – average size of precipitates, l – distance between particles [nm] $((l=D(\pi/bf)^{0.5}$, D – particle diameter, f – volume fraction of particles).

The results of the composition analysis conducted for 10MnVNb6 steel [3, 8] suggest that the increase in the yield strength and ultimate tensile strength results also from solution strengthening due to manganese (approx. 5–10%). Users of steel products cannot change their chemical composition. The only way to alter the mechanical properties of steel is by heat treatment, which may improve the microstructure morphology, and thus impart the desirable yield strength and ultimate tensile strength.

This paper discusses the effect of heat treatment on the strength properties and the microstructure of 10MnVNb6 steel.

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2. Material

The material studied − 10MnVNb6 microalloyed steel − was in the form of plates, 8 mm and 12 mm in thickness. Three heating variants of steel were used. As confirmed in the mill test certificates and follow-up analysis reports, their composition varied slightly (Table 1). The follow-up analysis showed that the steel in the form of plates with a thickness of 8 mm contained a small amount of titanium, i.e. approx. 0.002%. However, no titanium was present in the 12 mm thick plates.

The preliminary results of the microscopic analysis indicated that there existed certain differences in the morphology of the microstructure of 10MnVNb6 steel plates. The most important difference − observed at e.g. 500x magnification − was in the grain size.

3. Heat treatment

To define the effect of some heat treatment processes on the mechanical properties and morphology of rolled 10MnVNb6 steel, we applied:

1. aging, with the plates being heated at 690°C for 0.5 h and then air cooled to room temperature (Fig. 1);
2. normalization, with the plates being heated at 950°C for 1 h and then air cooled to room temperature (Fig. 2);
3. aging after normalization, with the plates being heated at 690°C for 0.5 h and then air cooled to room temperature (Fig. 3).

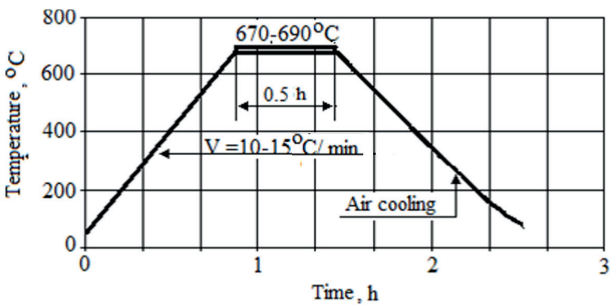

Fig. 1. Technological parameters of aging of 10MnVNb6 steel after rolling

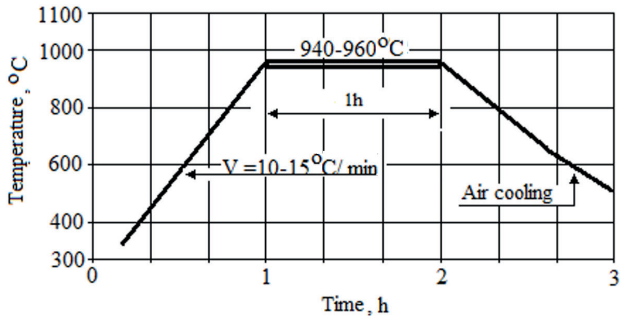

Fig. 2. Technological parameters of normalization of 10MnVNb6 steel

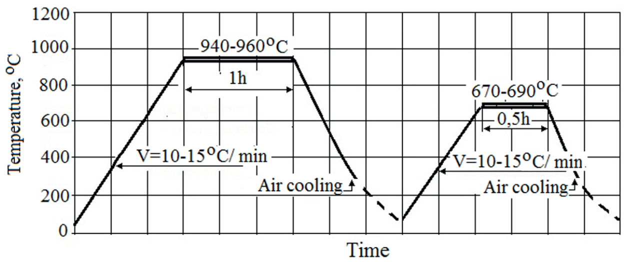

Fig. 3. Technological parameters of normalization and aging of 10MnVNb6 steel

4. Method

The mechanical properties of 10MnVNb6 were determined through static tensile strength tests conducted according to the requirements specified in PN-91/H-04310 (the current standard being PN- EN ISO6892-1:2010). The tensile strength tests were carried out using an AJ AMSLER 200 universal testing machine. The specimens were cut parallel to the rolling direction.

The microstructures of the microalloyed steel 10MnVNb6 before and after heat treatment were analyzed using the:

- Carl Zeiss Jena EPITYP-2 metallographic microscope. The observed sample surfaces were revealed with an etching agent containing 97 ml of ethylene alcohol (95%) and 3 ml of analytically pure nitric acid.
- JEOL JEM 2000FX transmission electron microscope.

TABLE 1

Chemical composition of 10MnVNb6 microalloyed steel

Steel	Plate thickness mm / Heat variant	Element content, [wt %]						
10MnVNb6	8 / 033683	C	Mn	Si	P	S	V	Nb
		0.09	1.49	0.36	0.017	0.013	0.14	0.030
		Al	N	Cr	Mo	Ni	Ti	O
		0.031	0.010	0.10	0.02	0.10	0.002	0.0030
	12 / 800204	C	Mn	Si	P	S	V	Nb
		0.12	1.39	0.38	0.022	0.008	0.14	0.040
		Al	N	Cr	Mo	Ni	Cu	O
		0.038	0.013	0.08	0.03	0.08	0.11	0.0035
	12 / 800220	C	Mn	Si	P	S	V	Nb
		0.11	1.54	0.31	0.022	0.024	0.11	0.030
		Al	N	Cr	Mo	Ni	Cu	O
		0.030	0.014	0.10	0.03	0.09	0.13	0.0050

The thin foils used for transmission electron microscopy were prepared with a Struers Tenupol-3 electro-polisher. The preparation process was performed using perchloric acid 50 ml in glacial acetic acid 950 ml at a voltage of 80 V and a temperature of 13°C.

- TECNAI FEG (200kV) analytical transmission electron microscope. The thin foils used for transmission electron microscopy were prepared with a FIB technique.

5. Results

The results of the static tensile strength tests conducted for 10MnVNb6 steel (Table 2) indicate that, after rolling, heat No. 33683 had the highest yield strength and ultimate tensile strength (Fig. 4). For heats Nos 800204 (Fig. 5) and 800220 (Fig. 6), the values of $R_{0.2}$ and R_m were relatively lower. When aging was applied after rolling (690°C/0.5h), an increase in the yield strength and ultimate tensile strength for all the three heats was observed. Normalization of 10MnVNb6 steel at 950°C for 1 h caused a reduction in the strength properties; however, when normalization was followed by aging at 690°C for 0.5 h, the strength of 10MnVNb6 steel recovered slightly (Figs. 4-6).

<Tab 2>

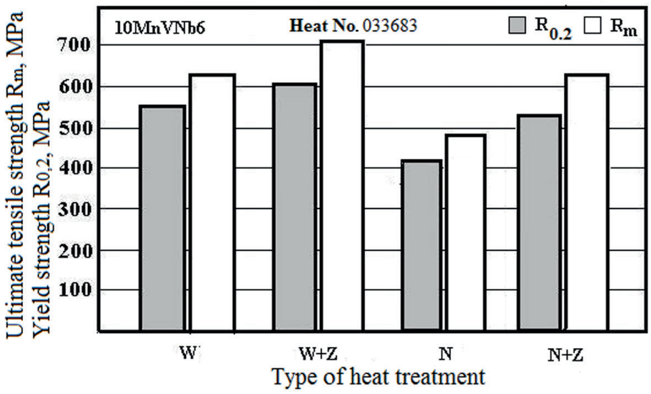

Fig. 4. Effect of the heat treatment processes on the yield strength and ultimate tensile strength of the rolled 10MnVNb6 steel. Plate thickness 8 mm, Heat No. 033683. W – before heat treatment, W+Z – aging (690°C/0.5 h), N – normalization (950°C/1 h), N+Z – normalization followed by aging (690°C/0.5 h)

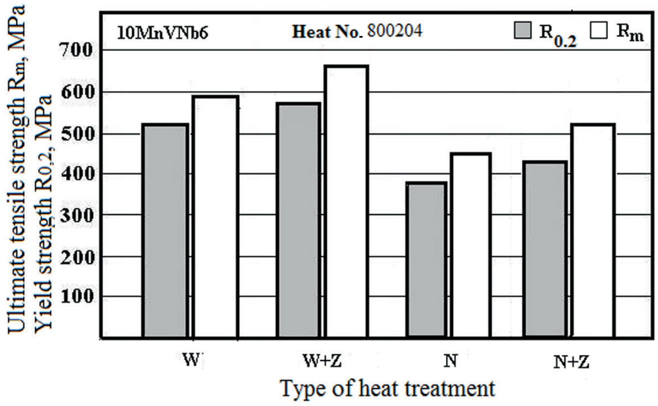

Fig. 5. Effect of the heat treatment processes on the yield strength and ultimate tensile strength of 10MnVNb6 steel after controlled rolling. Plate thickness 12 mm, Heat No. 800204. W – before heat treatment, W+Z – aging (690°C/0.5 h), N – normalization (950oC/1 h), N+Z – normalization followed by aging (690°C/0.5 h)

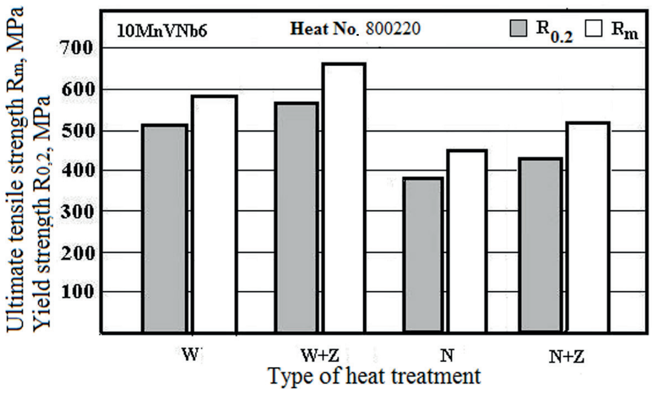

Fig. 6. Effect of heat treatment processes on the yield strength and ultimate tensile strength of 10MnVNb6 steel after controlled rolling. Plate 12 mm in thickness, Heat No. 800220. W – before heat treatment, W+Z – aging (690°C/0.5 h), N – normalization (950oC/1 h), N+Z – normalization followed by aging (690°C/0.5 h)

The metallographic analysis showed that 10MnVNb6 steel had a ferritic-pearlitic microstructure. The relative volume of the ferrite grains was approximately 95%; the rest was pearlite. After controlled rolling, the ferrite grains were 5-20 μm and 10-25 μm in diameter for plates 8 mm and 12 mm thick, respectively. Normalization at 950°C for 1 h caused a decrease in the grain diameter up to 3 μm to 10 μm in plates of 8 mm thick and 5 μm to 12 μm in plates of 12 mm thick (Fig. 7).

TABLE 2

Strength properties of rolled 10MnVNb6 steel before and after heat treatment

Heat No.	Yield strength $R_{0.2}$ and ultimate tensile strength R_m *), MPa							
	Before heat treatment (W)		After aging (W+Z)		After normalization (W+N)		After normalization and aging (W+N+Z)	
	$R_{0.2}$	R_m,	$R_{0.2}$	R_m,	$R_{0.2}$	R_m,	$R_{0.2}$	R_m,
033683	555	624	605	715	418	492	523	620
800204	515	596	591	660	392	452	417	519
800220	512	589	586	652	390	448	415	512

*) - The values given in the table have an error rate of less than 2%.

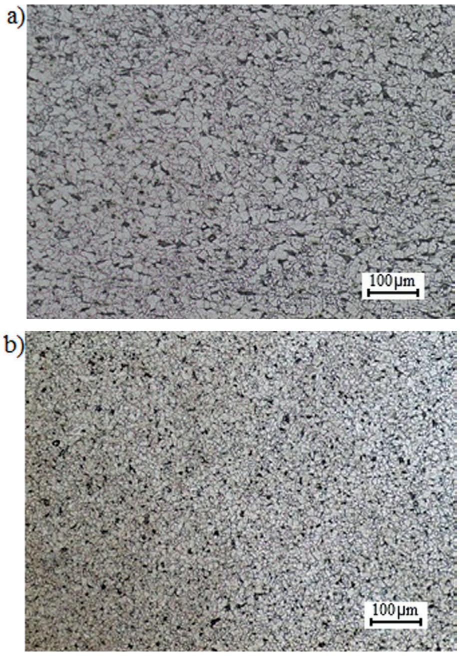

Fig. 7. Microstructure of 10MnVNb6 steel sample: aged after rolling at 690°C for 0.5 h (a); normalized after rolling at 950°C for 1 h + air-cooled (b). Plate thickness – 8 mm, Heat No. 033683

Transmission electron microscopy was used to determining the size and morphology of the particles of the strengthening phases as well as chemical composition of microareas of the microstructure.

After controlled rolling, strengthening phases formed in the ferrite matrix, with precipitates being greater than or equal to 10 nm in diameter in the grain interior (Fig. 8a) and up to 500 nm in size at the grain boundaries (Fig. 8b).

The results of the point analysis indicated that the precipitates in the ferrite grains, ranging from 5 nm to 20 nm in size, contained vanadium and niobium. Their quantitative ratio was variable, depending on size of the precipitates. The amount of vanadium in large precipitates was generally lower than that in small precipitates (Figs. 9a, b). The precipitates found at the grain boundaries contained little contents of vanadium or its amount was within the margin of measurement error (Figs. 10 a and 11).

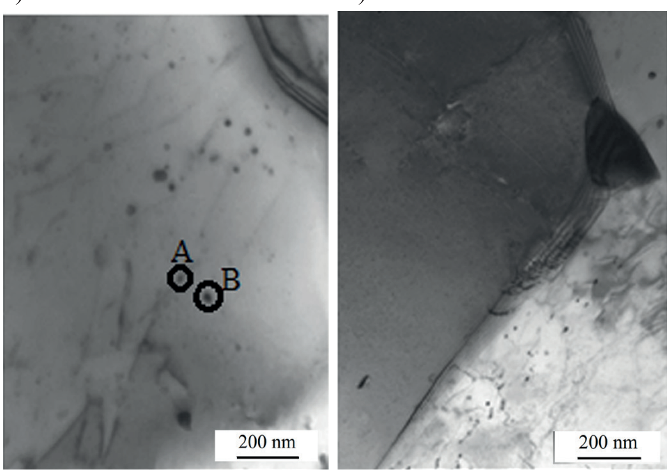

Fig. 8. TEM JEOL JEM 2000FX. Microstructure of 10MnVNb6 steel after rolling. Precipitation of the strengthening phase: ferrite grain interior precipitates (a) and ferrite grain boundary precipitates (b)

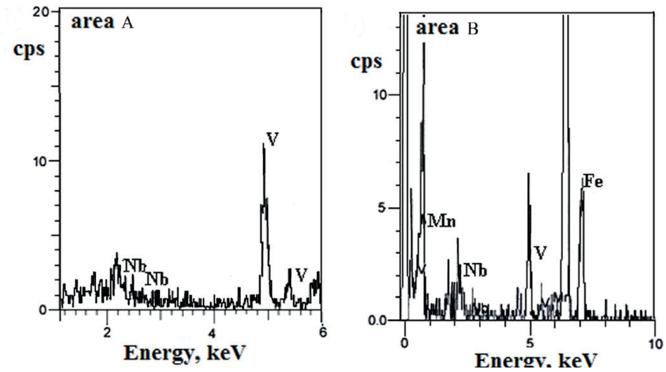

Fig. 9. Energy-dispersive X-ray spectrum of carbonitrides (Fig.8a). (a) – area A, (b) – area B

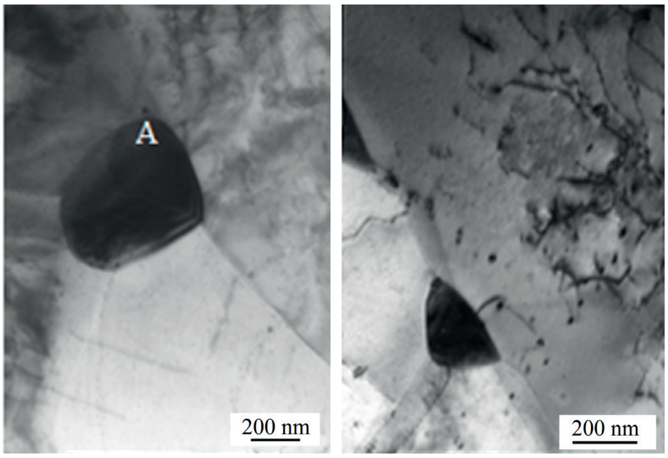

Fig. 10. TEM JEOL JEM 2000FX. Microstructure of 10MnVNb6 steel aged at 690oC for 0.5h after rolling (a, b); (). Plate thickness – 8 mm

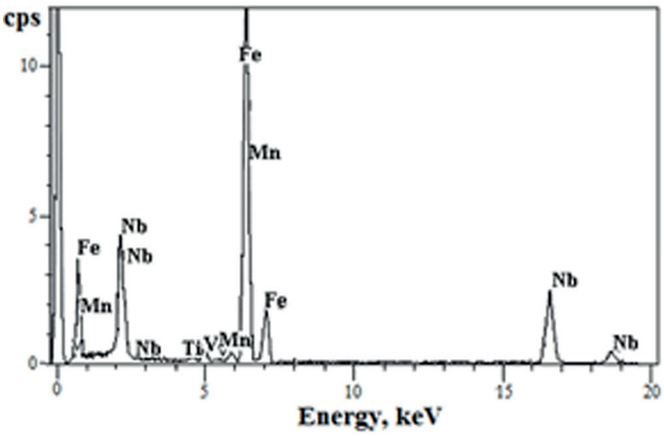

Fig. 11. Energy-dispersive X-ray spectrum of a carbonitride – area A in Fig. 10a

Aging at 690°C for 0.5 h performed after rolling resulted in the occurrence of numerous precipitates less than 10 nm in size in the ferrite grains.

The treatment led to the occurrence of many precipitates ≤ 5 nm in size in the ferrite matrix microstructure (Figs. 12a, b, and 13a).

The point analysis by TEM show that the particles precipitated in the ferrite matrix of 10MnVNb6 steel contained vanadium; there was no niobium, or its amount was within the margin of measurement error (Fig. 13b).

a) b)

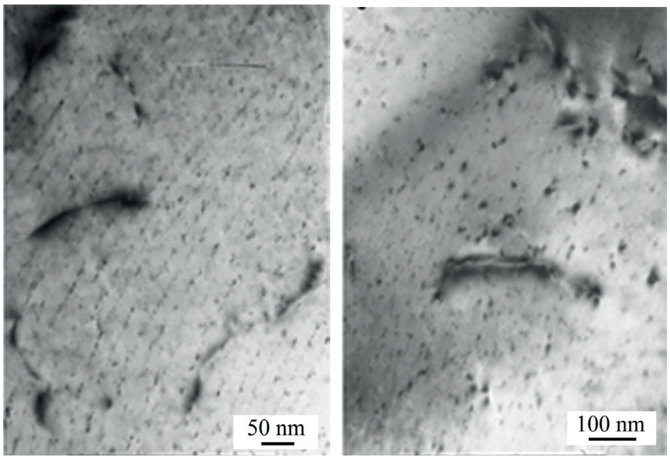

Fig. 12. TEM-JOEL JEM 2000FX. Microstructure of the ferrite matrix in 10MnVNb6 steel after rolling and then aging at 690°C for 0.5 h;. Plate thickness – 8 mm

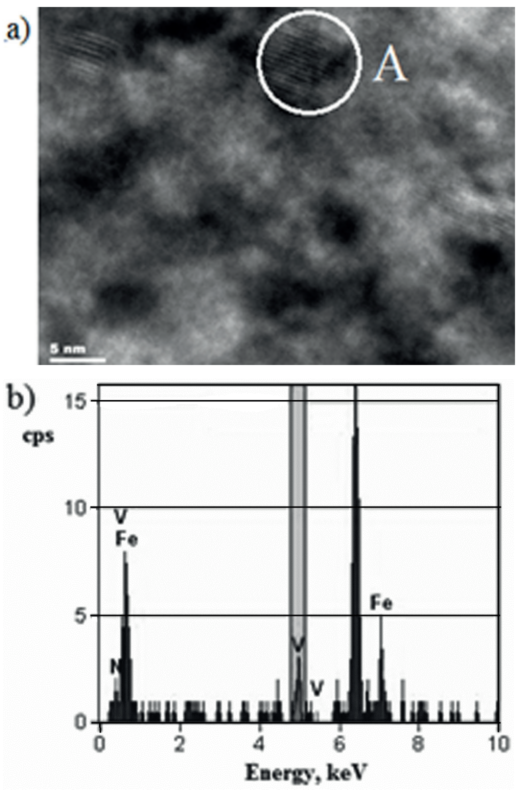

Fig. 13. TEM-TECNAI FEG (200kV). Microstructure of a small fragment of the ferrite matrix of 10MnVNb6 steel after aging at 690 °C for 0.5 h (a); an energy-dispersive X-ray spectrum of area A. Plate thickness – 8 mm

Normalization of 10MnVNb6 steel at 950°C for 1 h led to essential decrease in the grain size. From the microstructure analysed at a large magnification it was clear that changes in the morphology were larger after normalization than after aging process. The number of vanadium-niobium carbonitride particles decreased, but their average diameter increased. Particles with a diameter of ≤ 5 nm were not present in the ferrite matrix microstructure, or their presence was rare (Fig. 14a). Aging after normalization resulted in the occurrence of a small amount of vanadium carbonitride precipitates in the microstructure, their diameter being ≤ 5 nm (Figs. 14b, 15a and b). After normalization, the number, size and morphology of niobium carbonitrides did not change significantly; niobium carbonitrides were present at the grain boundary. However, considerable changes were observed in the pearlite morphology. Rolling or rolling followed by aging caused that the plates of pearlite cementite were degenerate. After normalization, however, pearlite had a lamellar (plate-like) structure (Figs. 16a and b)

a) b)

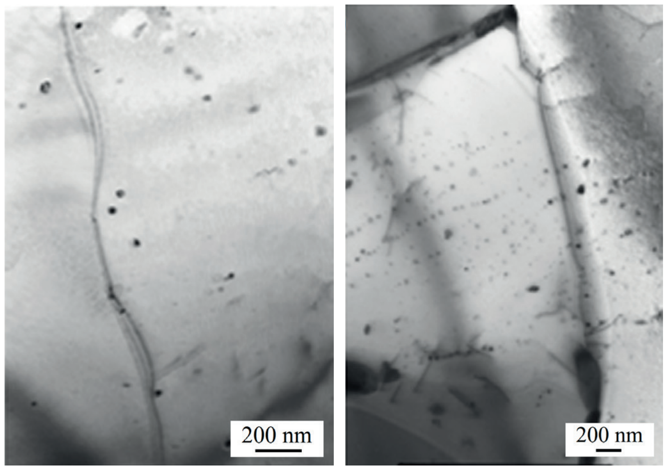

Fig. 14. TEM-JEOL JEM 2000FX. Microstructure of the ferrite matrix of 10MnVNb6 steel normalized at 950oC for 1 h (a), normalized and then aged at 690°C for 0.5 h (b). Plate thickness – 12 mm (heat No. 800220)

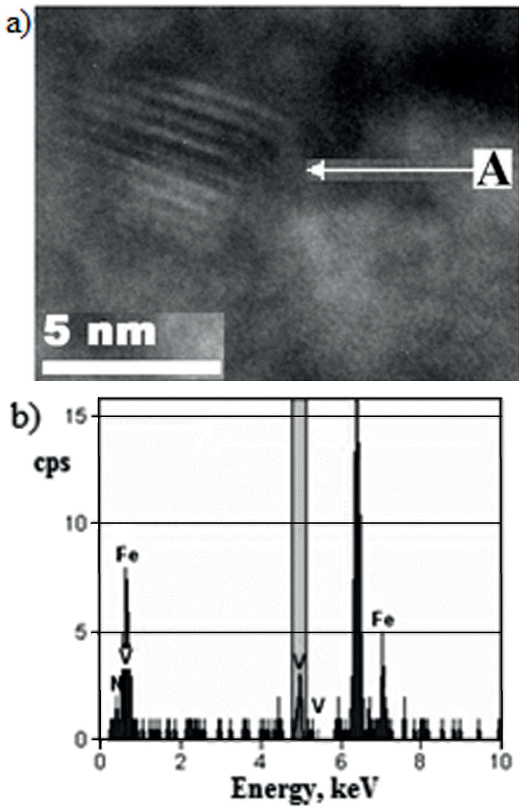

Fig. 15. TEM TECNAI FEG. Microstructure of the ferrite matrix of 10MnVNb6 steel normalized at 950oC for 1 h and aged at 690oC for 0.5 h (a) and an energy-dispersive X-ray spectrum of area A (b). Plate thickness – 8 mm (heat No. 033683)

a) b)

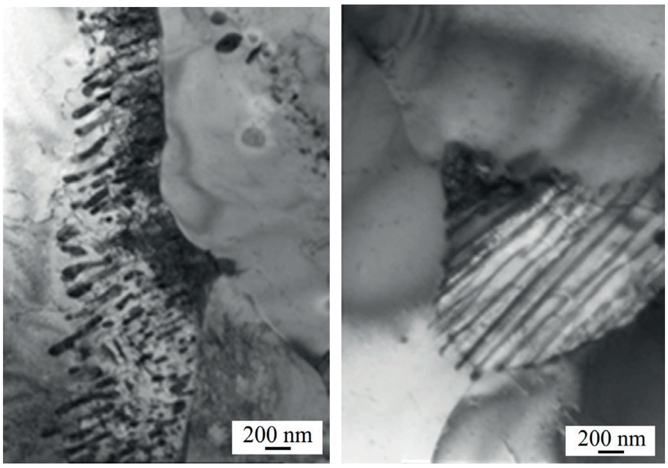

Fig. 16. TEM-JOEL JEM 2000FX. Microstructure of 10MnVNb6 steel rolled and then aged at 690oC for 0.5h (a), normalized at 950oC for 1 h and then aged at 690oC for 0.5h (b). Plate thickness – 8 mm (heat No. 033683)

6. Discussion

The results of the strength tests and microscopic analysis indicated that 10MnVNb6 steel after rolling process had relatively high yield strength and high ultimate tensile strength, irrespective of heat or plate thickness. The highest strength properties were observed for the plate of 8mm in thickness (heat No. 033683). The strength properties of 12 mm thick plates were lower than those of 8mm thick plates; yield strength R0.2 was approx. 10% lower and the ultimate tensile strength Rm was about 5% lower. The size of grains ranged from 5 μm to 20 μm in 8 mm thick plates, and from 10 μm to 25 μm in 12 mm thick plates. Aging at 690°C for 0.5 h of the rolled 10MnVNb6 steel caused an increase in the yield strength and ultimate tensile strength. There was significant increase in R0.2 and Rm for all heating variants; the highest values were reported for 033683 one. When 10MnVNb6 steel was subjected to aging, the grain size remained unchanged, whereas the microstructure morphology of the ferrite matrix changed substantially. There was an increase in the amount of small size particles (≤5 nm) of the carbonitride phase, which are frequently arranged in parallel lines (Fig. 12a). The results of microanalysis confirmed that the precipitate particles contained vanadium. No niobium was present.

Normalization at 950°C for 1 h was responsible for significant changes both in the strength properties as well as microstructure morphology. The strength properties decreased essentially (Table 2). The changes in microstructure morphology referred to the grain size and dispersion of precipitates. The grain diameter decreased, ranging approximately from 3 μm to 10 μm in 8 mm thick plates and from 5 μm to 12 μm in 12 mm thick plates. The dispersion of carbonitride phase in the ferrite matrix was lower; small diameter precipitates (≤ 5 μm) diminished (Fig. 13a). Aging after normalization led to a slight rising in the strength properties, an increase in the dispersion of precipitates in the ferrite matrix and the occurrence of a small amount of precipitates with a diameter of ≤5 nm (Figs. 14b and 15). The pearlite morphology also changed (Fig. 16)

7. Conclusions

Strong correlations between the strength test results and those of the microscopic analysis indicate that the following conclusions can be formulated.

1. The strength properties of micro-alloyed 10MnVNb6 steel retained relatively high.
2. An increase in dispersion of vanadium and/or niobium carbonitride precipitates in the ferrite matrix due to aging after rolling or aging after normalization led to a rise in the yield strength $R_{0.2}$ and ultimate tensile strength R_m.
3. A decrease in dispersion of vanadium and/or niobium carbonitrides in the ferrite matrix due to aging at a high temperature (normalization at 950°C for 1 h) caused a decrease in the strength properties of the steel.
4. The increase in the component of strengthening resulting from a decrease in the average size of ferrite grains (the Hall-Petch equation) did not counterbalance the decrease in the component of precipitation strengthening of the steel subjected to normalization.
5. The high strength properties of 10MnVNb6 steel were mainly due to precipitation strengthening at the grain boundary and in the ferrite matrix.
6. The changes observed in the microstructure indicate that it is possible to significantly improve the properties of the structural steel using a method based on the modification of its chemical composition (steel with microadditives) and a respective heat treatment process.

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