A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
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MODEL OF PHASE TRANSFORMATION FOR NIOBIUM MICROALLOYED STEELS

MODEL PRZEMIANY FAZOWEJ DLA STALI Z MIKRODODATKIEM NIOBU

The paper presents the results of physical and numerical modeling of the kinetics of phase transformation, taking into account the precipitation of niobium carbonitride. Strain induced precipitation is a phenomenon, which controls the evolution of the microstructure in these steels during thermo-mechanical treatment. For the numerical simulation of precipitation Dutta-Sellars model was used, which describes the precipitation kinetics of Nb (C, N) at dislocations in the deformed and non-deformed austenite. The size of precipitates after continuous cooling of steel was calculated using the additivity rule. Numerical model combines a solution of the finite element method with model of phase transitions. Physical modeling included dilatometric study and rolling of rods made of niobium microalloyed steel. Microstructure studies were also carried out. Developed model allowed the assessment of the influence of precipitation on the progress of phase transition. Verification of model prediction by comparison with the experiments carried out in conditions close to semi-industrial is described in the paper, as well.

Keywords: niobium microalloyed steels, model of phase transformation, carbonitride precipitation process, thermomechanical treatment, multiscale modelling

W pracy przedstawiono wyniki modelowania fizycznego oraz model numeryczny opisujący kinetykę przemiany fazowej z uwzględnieniem procesu wydzieleniowego węglikoazotku niobu. Proces ten silnie wpływa na zmiany zachodzące w mikrostrukturze tych stali w trakcie obróbki cieplno – plastycznej. Do symulacji numerycznej procesu wydzieleniowego wykorzystano model Dutty-Sellarsa opisujący kinetykę procesu wydzieleniowego Nb(C,N) na dyslokacjach w austenicie odkształconym i nieodkształconym. Wielkość wydzieleń w warunkach ciągłego chłodzenia stali obliczona została z wykorzystaniem reguły addytywności. Model numeryczny łączy rozwiązanie metodą elementów skończonych z modelem przemian fazowych. Modelowanie fizyczne obejmowało badania dylatometryczne oraz walcowanie prętów ze stali z mikrododatkiem niobu uzupełnione badaniami mikrostruktury. Opracowany model pozwolił na ocenę wpływu procesu wydzieleniowego na postęp przemiany fazowej. Wyniki modelowania zweryfikowano doświadczalnie w warunkach zbliżonych do półprzemysłowych.

1. Introduction

The model of phase transformation was developed for carbon-manganese steel by a combination of conventional equations describing the kinetics of the transformation [1] with the solutions by finite element method (FEM) [2,3]. In the case of microalloyed steels the model should also consider the influence of precipitation processes.

The general objective of this study is experimental verification of the developed multi-scale model under conditions similar to industrial. In this order dilatometric tests were performed using the dilatometer DIL 805 at the Institute of Ferrous Metallurgy in Gliwice (Poland) and rod rolling in a continuous hot rolling mill at the TU Bergakademie Freiberg, Institut für Metallformung. Rods were rolled in four elongating passes of oval-round type. These experiments were used to identify the material models and to verify the global model of phase transformations.

The particular objectives of the work include quantitative description of the transformation fraction of ferrite in pearlite, the determination of ferrite transformation start temperature, and quantitative analysis of carbide niobium precipitation. Inverse analysis of experiments is used to determine the coefficients for the model of phase transitions.

Quantitative analysis of niobium carbonitride particles is the final objective of the paper. It is performed on

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the basis of comparison of microstructures obtained from experiments with the results of the model predictions.

2. Material for testing

Laboratory ingots weighing 20 kg and made of niobium microalloyed steel, which chemical composition shown in Table 1, were used in dilatometric and industrial tests. The study of phase transformation consisted of heating the material at the rate of 3°C/s to the austenitizing temperature – 1230°C, keeping at this temperature for 30 s, cooling to a temperature of deformation 850°C, deformation of a given reduction $\varepsilon = 0.2$ and $\varepsilon = 0.4$ and then cooled to ambient temperature at a constant speed. In studies cooling rates of 40, 20, 10, 5°C/s were applied. In the semi-industrial rolling process rod of dimensions 25×20 and a length of about 400 mm was heated to an austenitizing temperature of 1200°C, maintained for 20 min, cooled to a temperature of 1050°C in air and rolled. The interval between the passes was about 10 s. The temperature after the last pass was 930°C and the linear velocity of strip was 12 m/s. After leaving the last pass, the rod was cooled with compressed air to a temperature of 350°C. Illustration and parameters of the hot rolling mill in Freiberg can be found in [4]. Figure 1 shows the cooling curve of the rolled rod from the temperature 912 to 350°C. On this curve, the selected section was extrapolated from the temperature 912°C to a temperature of 930°C, in which the material was rolled in the last pass. The final diameter of the rolled bar was 12 mm.

TABLE 1

Chemical composition of steel used in the dilatometer tests and rolling, % mass

C	Mn	Si	P	S	Cr	Ni	Cu	Al	V	Nb	N
0.082	1.1	0.21	0.013	0.004	0.03	0.03	0.04	0.055	0.004	0.04	0.005

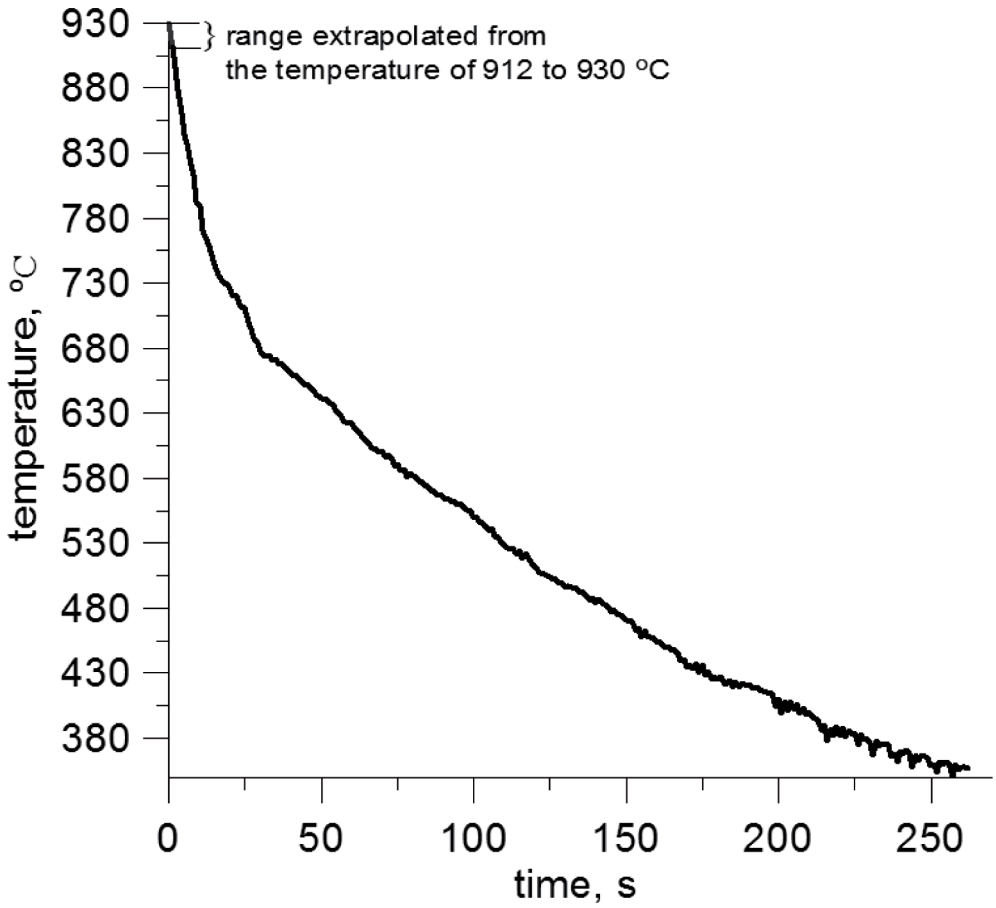


Fig. 1. Measured cooling curve of the steel rod niobium microalloyed temperature 930 to 350°C

3. Numerical modeling

Finite element method

Flow of metal in the plastometric tests was modeled using rigid-plastic finite element solution, where heat transport was described by the Fourier equation. A detailed description of the solution presented in publication [5] and it is not quoted in this paper.

Model of phase transitions

Models of phase transitions (ferritic, pearlitic and bainitic) consist of differential equations describing the incubation period and kinetics of the transformation. Design concept is described in [1], and its numerical implementation in [2,3,6]. In this model, the calculation of the progress of ferrite transformation begins at the equilibrium temperature $A_{c3.}$ Constitutive equations for ferrite transformation in general form are given below:

- incubation period for the ferritic transformation

$$\tau_f = \frac{x_1}{(A_{c3} - T)^{x_3}} \exp\left(\frac{x_2}{RT}\right) \tag{1}$$

- volume fraction of ferrite

$$\frac{dX_f}{dt} = x_5 S_v^{0.25} G^{0.75} I^{0.25} \left[\ln\left(\frac{1}{1 - X_f}\right)\right]^{0.75} \left(1 - X_f\right) \tag{2}$$

$$\frac{dX_f}{dt} = x_4 \frac{6}{D} G \left(1 - X_f\right) \tag{3}$$

where: t – time since the beginning of the transformation, D – austenite grain size, S_v – relative area of grain boundaries, G – the speed of the transformation front, T – temperature [K], R – gas constant, I – nucleation rate, $x_1 - x_5$ – coefficients, X_f – volume fraction of ferrite.

The full model of phase transitions, including the other changes taking place in steels, is described in the literature [2]. A method of identification of coefficients in the model is based on the algorithm of [7] and is described in [3], while work [6] presents results of this identification for a niobium microalloyed steel.

Dutta and Sellars Model

The model, which describes the incubation period and the progress of precipitation [8 ÷ 10], is used in the present work to calculate the number of precipitates per unit volume and average diameter of precipitates. The precipitation process of carbonitride Nb (C,N) strongly influences the final microstructure and properties of products. Precipitates inhibit the recrystallization of austenite, which leads to reduction of the size of the ferrite grains after transformation of non-recrystallized austenite. In this paper, the Dutta-Sellars model is used in the calculation of the precipitation kinetics of Nb (C,N) at dislocations, while it omitts others nucleation sites of carbonitride, such as grain/subgrain boundaries, sites with large plastic deformation or direct precipitation in the matrix. The basic equations of the model are quoted below.

– Solubility product of Nb, C and N in austenite [8]:

$$k_s = \frac{\log[Nb]\left[C + \frac{12}{14}N\right]}{10^q} \qquad q = 2.06 - \frac{6700}{T} \tag{4}$$

where [Nb] and [C + N] – the contents of niobium and suitably adjusted for the atomic weight aggregate content of interstitial elements in austenite, expressed in percentage by mass.

– Critical radius for nucleation, R_c is determined by propelling force and the equation is:

$$R_c = -\frac{2\gamma}{\Delta G_v} \tag{5}$$

where: γ energy of the interface equal to 0.5 [Jm $^{-2}$], V_m molar volume. For Nb (C, N), $V_m = 1.28 \times 10^{-5}$ [m ^{3} mol $^{-1}$].

– The difference in free energy per unit volume ΔG_v is calculated from the formula:

$$\Delta G_v = \frac{-RT \ln ks}{V_m} \tag{6}$$

A special feature of metallic systems at elevated temperatures is coagulation of the particles of precipitates. Kinetics of the coagulation equation is described by the Wagner equation [9]:

$$r^3 - r_0^3 = \frac{8}{9} \frac{\gamma D_{Nb} C_0 V_m^2}{RT} t \tag{7}$$

where $r_{0,}r$ – initial radius of particles and the radius after time t at temperature T, D_{Nb} – diffusion coefficient of Nb as an element controlling the speed of coagulation, C_0 – the concentration of N ($C_0 = k_s C_r$ where$C_r = 8.4 \times 10^2 [Nb]$)

– Time to start the process of precipitation, $t_{0.05p}$ is calculated from equation of classical nucleation theory, modified on the basis of experimental studies [9]:

$$t_{0.05p} = 3 \times 10^{-6} [Nb]^{-1} \varepsilon^{-1} Z^{-0.5} \exp\left(\frac{270000}{RT}\right) \exp\left[\frac{2.5 \times 10^{10}}{T^3 (\ln k_s)^2}\right] \tag{8}$$

where [Nb] – percentage by mass of niobium in solid solution in equilibrium with carbon and nitrogen calculated from equation (5) for the applied temperature of soaking stock, T – temperature at which the precipitation process proceed.

– Critical energy for the process of nucleus of carbonitride formation [9]:

$$\Delta G^* = \frac{16\pi f^3 \gamma^3}{3(\Delta G_v)^2} \tag{9}$$

where: $f = 0.41$ (empirical constant)

Model described above is used to calculate the number of precipitates per unit volume and average diameter of precipitates. The above components of the precipitation model were combined with the FEM program and the model of the kinetics of phase transitions, as it is described in detail in [10].

4. Results

Diagram of the developed multiscale model is shown in Figure 2. The general idea of the multiscale modeling [11,12,13] is applied. Description of rheological model for the investigated steel and the model of evolution of dislocation density are in the works [14,15]. Below are the results of testing and validation of the two components of the complex model: parts which calculate the phase transformation and the kinetics of precipitates. Start temperature of ferrite transformation for the deformed austenite ($\varepsilon = 0.2$ and $\varepsilon = 0.4$) was calculated using the developed model of phase transformation with the coefficients set by inverse analysis, which are presented in Table 2. Figure 3 shows the results of measurements and calculations of start temperature for ferrite transformation (F_s). As can be seen, a good agreement of measurement and calculation of that temperature is obtained. Pictures of microstructures of rolled bar, which were obtained using an optical microscope NEOPHOT, are presented in Fig. 4. Table 3 summarizes the results of measurements and calculations of start temperature of ferrite transformation for the two strains: 0.2 and 0.4 and the hardness of the samples measured by Vickers method.

Quantitative analysis of chemical composition is based on 300 measurements of grains and process simulation for the experimental conditions, and the result is volume fraction of ferrite of 94.7, while the predicted volume fraction is 90.7. It confirms good compatibility of the results of the volume fractions of phases obtained from modeling and measurement.

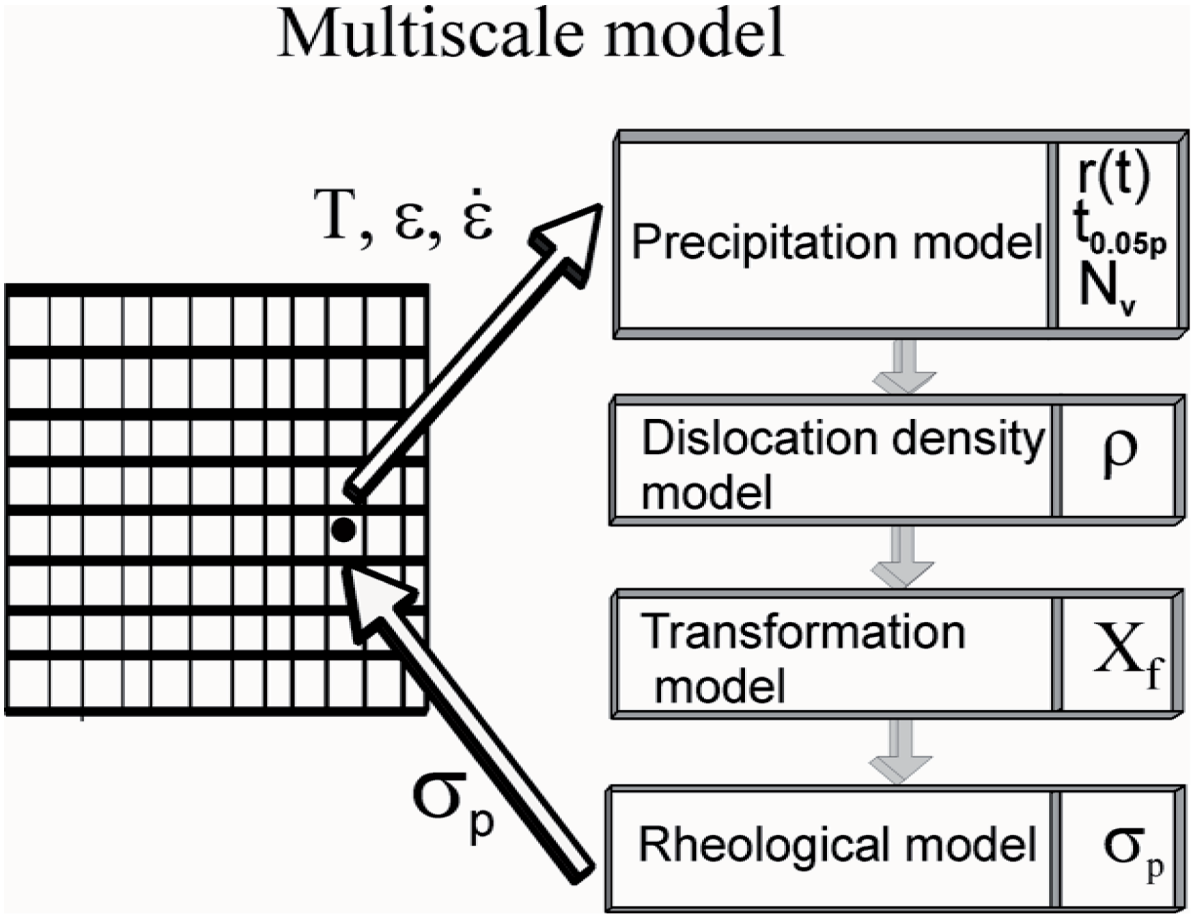


Fig. 2. Block diagram of the multiscale model simulating the deformation process of two-phase steel

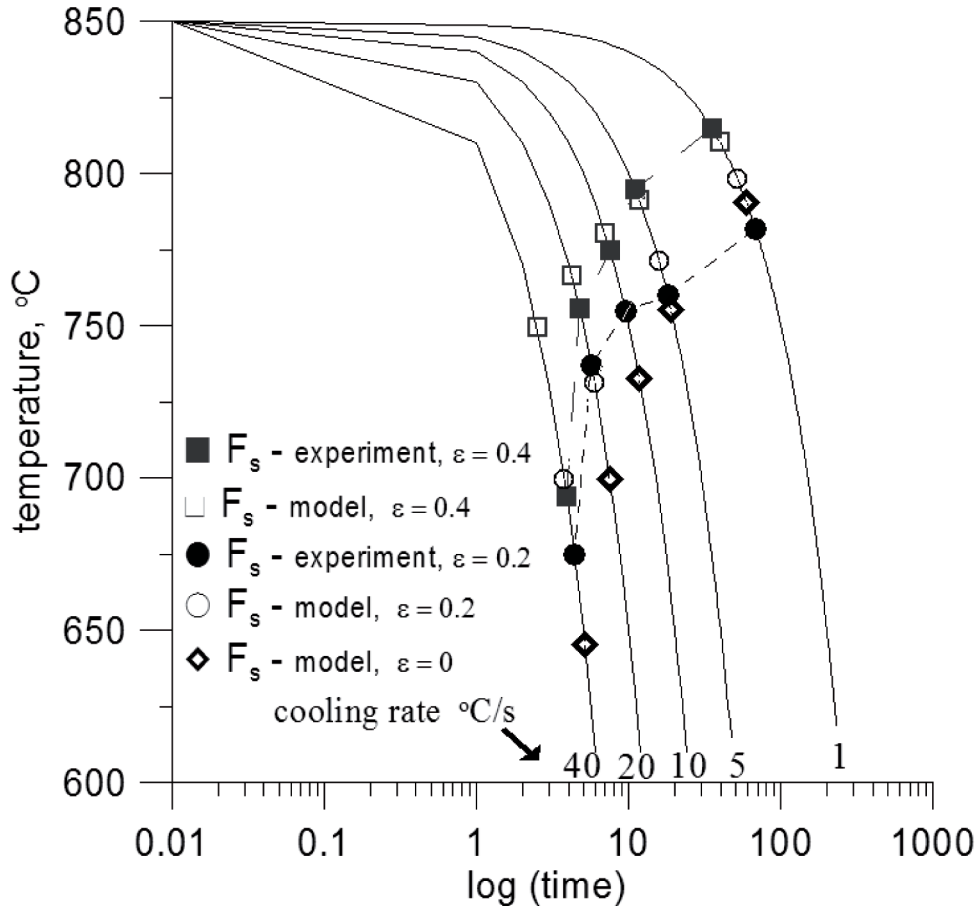


Fig. 3. The results of measurements and calculations ferrite transformation start temperature for the rolled bar

TABLE 2

Coefficients of phase transformation model determined by inverse analysis

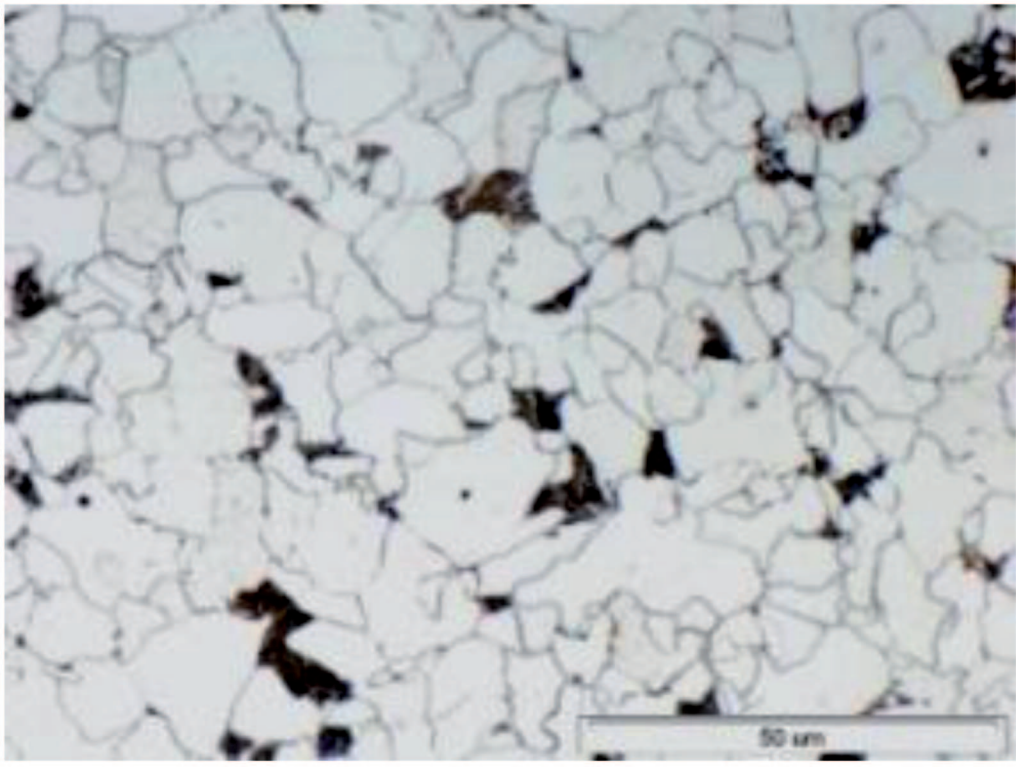
ε	x_1	x_2	x_3	x_4	x_5	x_6
0	4.2	109.5	2.8	0.8	46000	0.05
0.2	3.7	108.5	2.9	0.8	46000	0.05
0.4	3.4	102.5	3.0	0.8	46000	0.05

TABLE 3

The results of measurement and modeling of start temperature of ferrite transformation and hardness of the samples for different cooling rates

Cooling speed°C/s	ε = 0.2			ε = 0.4		
	F_s. model °C	F_s. measurement. °C	Hardness HV_{10}	F_s. model. °C	F_s. measurement. °C	Hardness HV_{10}
1	796.4	782	166	810.4	815	154
5	766.5	760	188	790.5	795	184
10	746.6	755	193	777.5	775	193
20	720.6	737	199	762.6	756	198
40	683.6	675	215	741.6	694	210

a)



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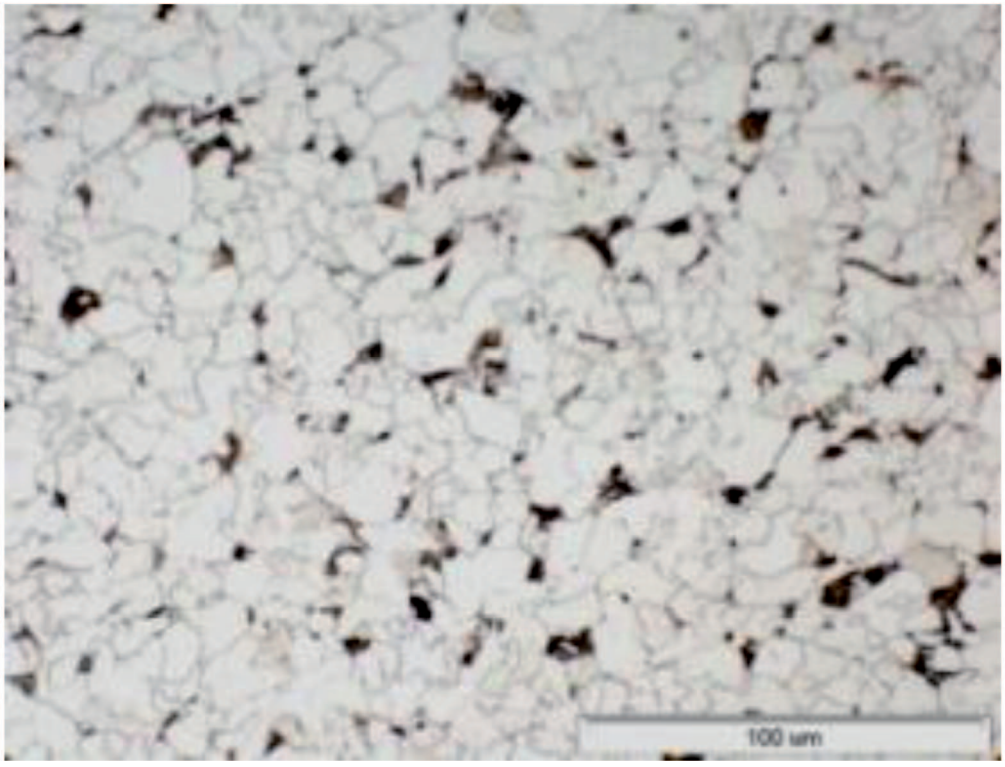


Fig. 4. Microstructure of the rolled steel rod microalloyed niobium after cooling to a temperature of 350° C

To calculate the fraction of static recrystallization (t_{rx}), the JMAK equation was used for the two models: Sellars [8,9] (t_{rx}_Dutta_Sellars) and Hodgson [16] (t_{rx}_Hodgson), and time of beginning of precipitation ($t_{0.05p}$) was obtained by the Dutta-Sellars model (Figure 5). Times, after which there was a beginning of a process of precipitation ($t_{0.05p}$) for non-isothermal conditions is calculated on the basis of the additivity rule:

$$x_{i+1} = x_i + \frac{\Delta t_i}{t_{0.05p}(T_i)} \tag{10}$$

where: Δt_i – time step, $t_{0.05p}(T_i)$ – the time of the transformation under isothermal conditions at the temperature T_i.

When the total incubation period reaches the value of one, the precipitation begins:

$$\sum_{i=1}^{n} \frac{\Delta t_i}{t_{0.05p}\,(T_i)} = 1 \tag{11}$$

Figure 5 shows the incubation period of formation of precipitates and the static recrystallization calculated for the experimental process. It is clear that precipitates after the last pass calculated by Dutta-Sellars model occur after the time a bit shorter than the time of static recrystallization, and the results obtained using the model of Hodgson gives a much longer times recrystallization. This is justified because the study by Hodgson covered steel with a higher content of niobium. The computer simulations provide information on incubation period of the formation of precipitates and fraction of recrystallization during the rolling of the material at different temperatures, and different linear speeds of deformed strip (Table 4). Austenite grain size after each pass through the rolling stand were calculated based on the Sellars [17] and Hodgson [16] models, see Figure 6.

TABLE 4

Characteristics of parameters of experimental rolling of bars

Parameter	Stand 1	Stand 2	Stand 3	Stand 4
deformation temperature, °C	1050	1030	990	930
linear velocity spectrum, m/s	3.92	5.68	8.23	12
strain rate, s^{-1}	59.14	111.74	177.55	281.54

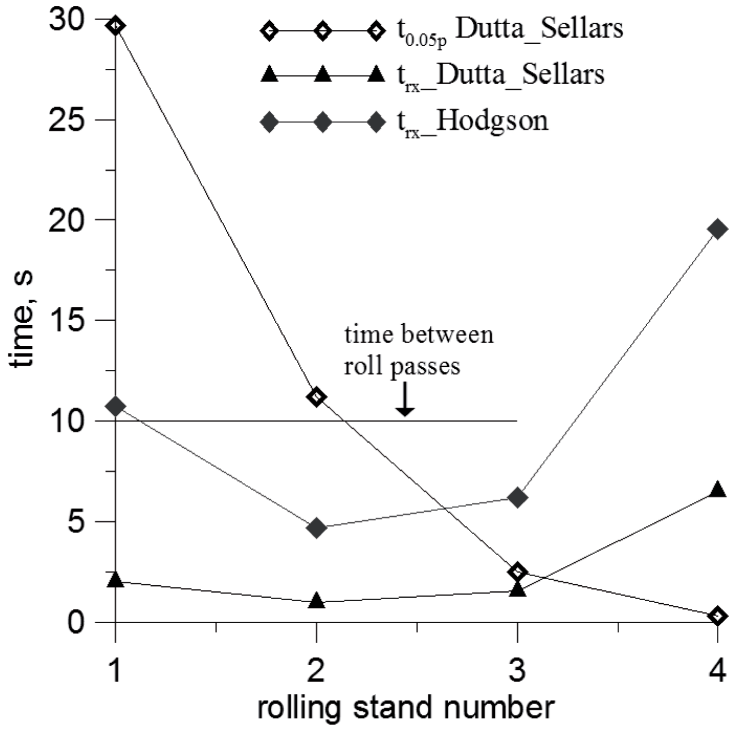


Fig. 5. Calculation of time of the beginning of the precipitation ($t_{0.05p}$) and the fraction of static recrystallization, calculated on the basis of the Dutta-Sellars (t_{rx}_ Dutta_Sellars) and Hodgson (t_{rx}_Hodgson) models

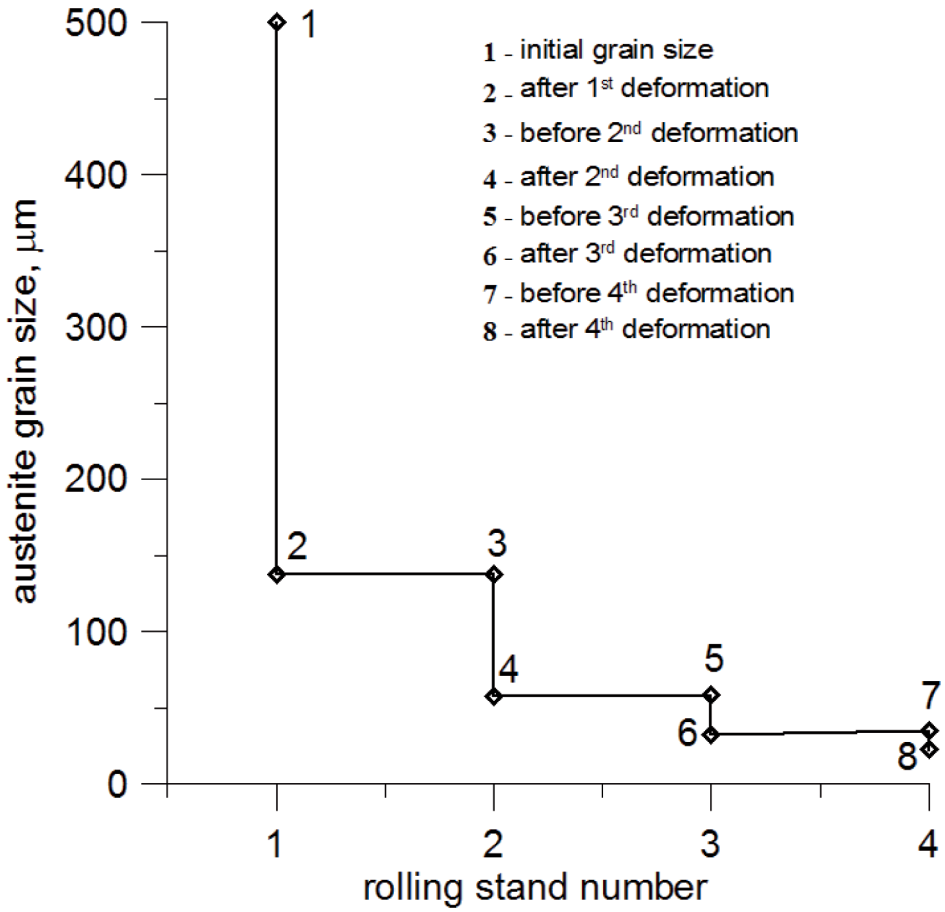


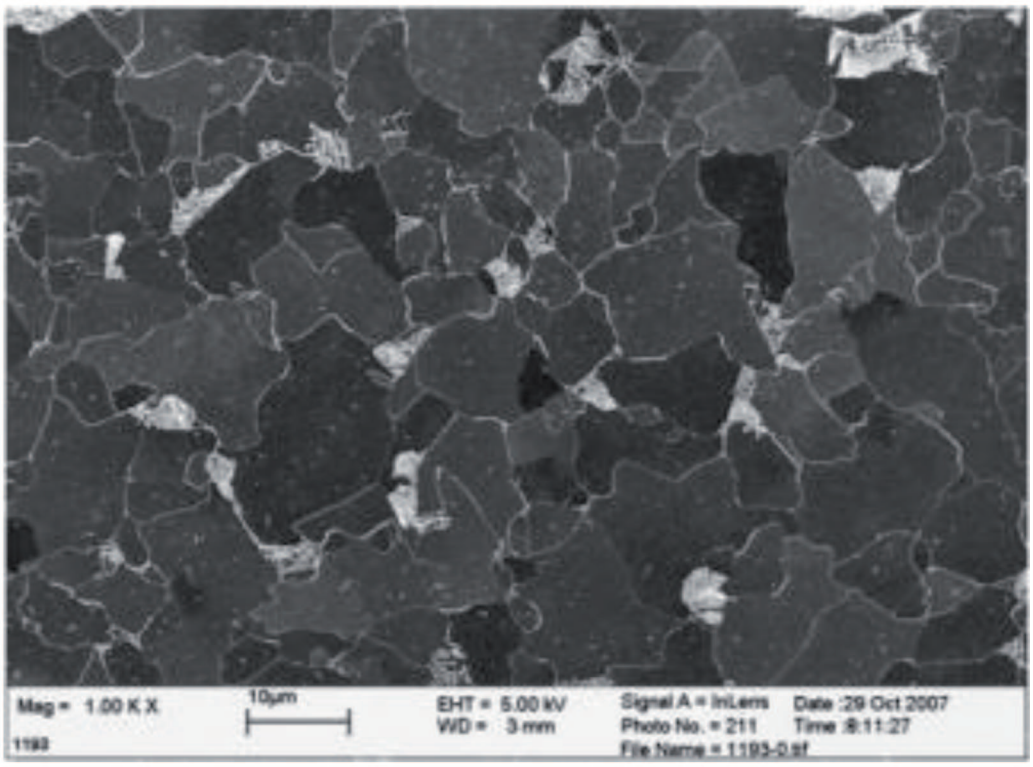
Fig. 6. Austenite grain sizes before and after the exit from the rolling stand

TABLE 5

Quantitative analysis of carbides of niobium Nb (C, N) obtained on the basis of measurement and model

Parameters	Measurement	Model
Number of precipitates per unit volume	$5.014 \times 10^{22}/m^3$	$1.7 \times 10^{22}/m^3$
The average diameter of precipitates	6.3 nm	4.0 nm

a)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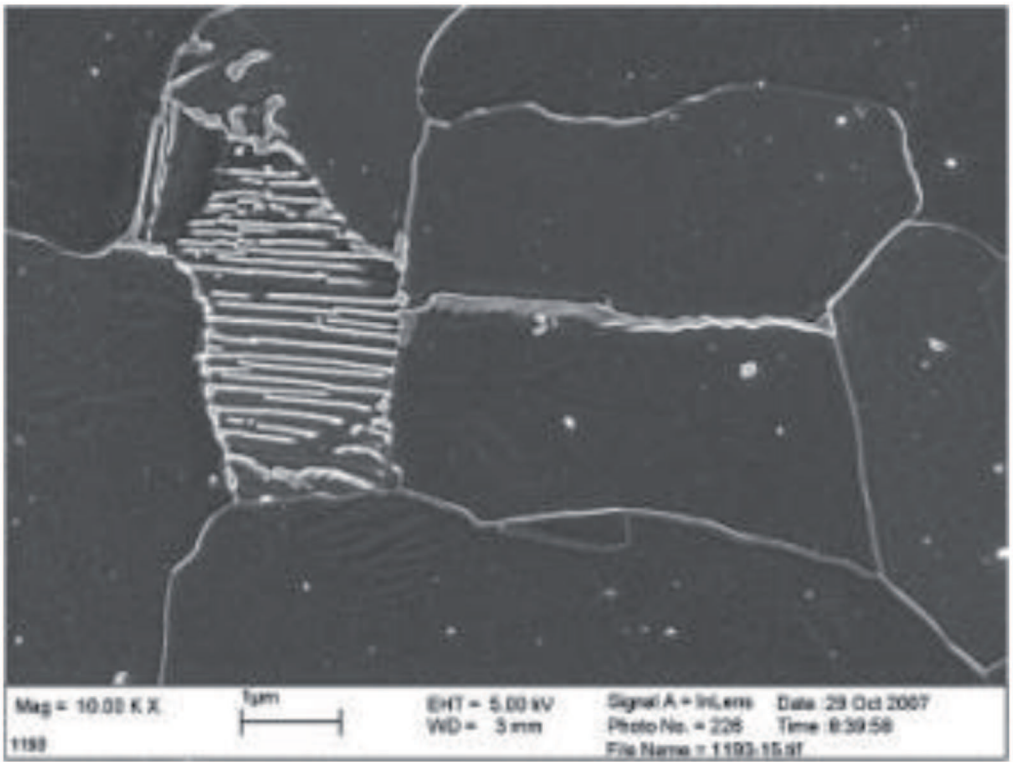
b)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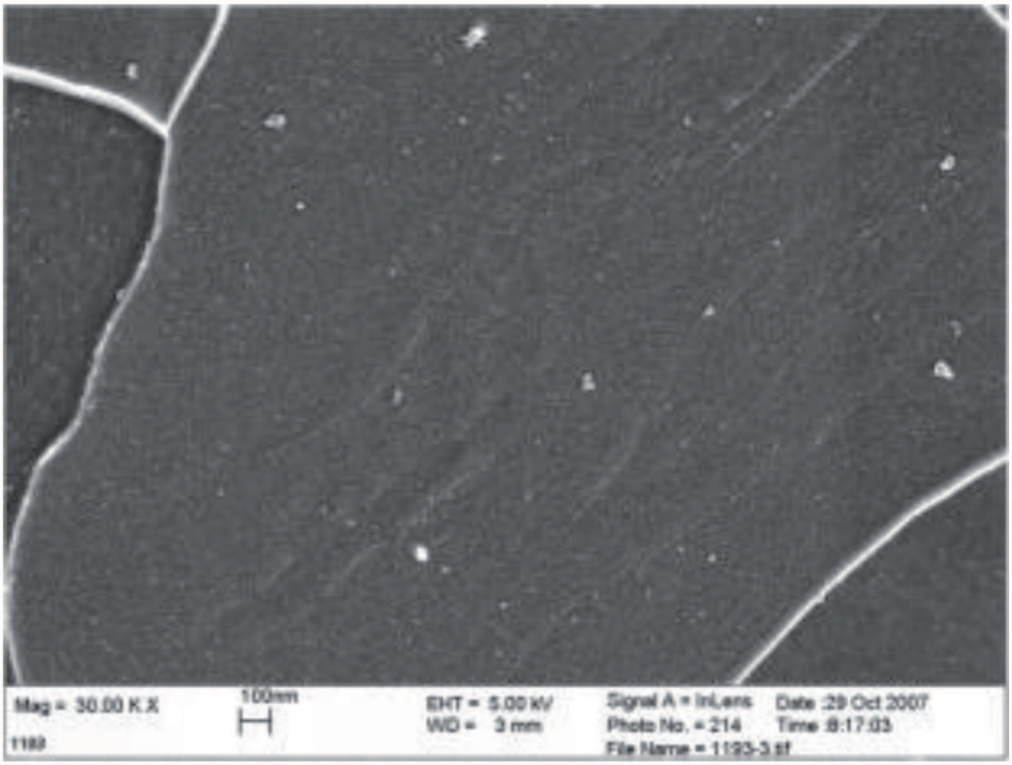
c)



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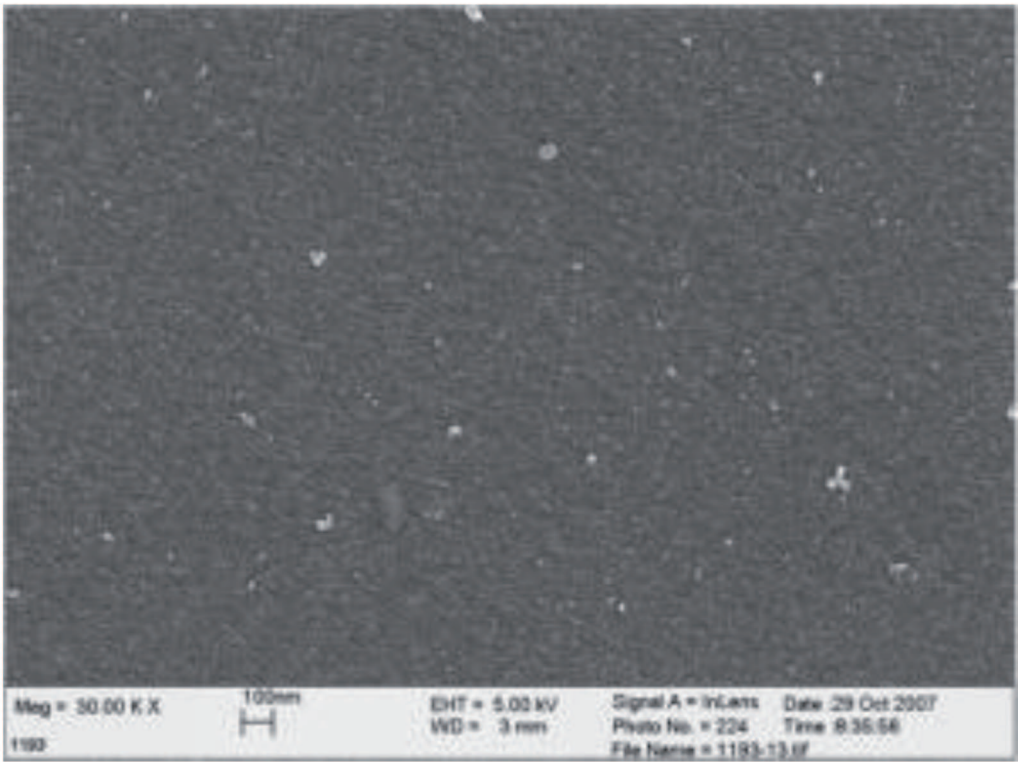

Fig. 7. Pictures of the scanning electron microscope showing a carbonitride particle Nb (C,N)

After the thermal-mechanical treatment microstructures were tested in different areas of rolled bar, using a scanning electron microscope FEGSEM. Selected results are presented in Fig. 7. A lot of small niobium carbonitride precipitates are seen inside the ferrite grains. Quantitative analysis of these particles was performed on the basis of microstructure in Figure 7d and on the basis of simulations. The results are presented in Table 5. The number of precipitates per unit area ($450/\mu m^2$), which was obtained from measurements, was converted to per unit volume by the inverse of the diameters of Saltykov [18].

5. Discussion

The use of multiscale model, which combines the macro-scale (where temperature, stresses and deformation are calculate) with micro and nano scale (where the kinetics of phase transformation and the precipitation process of carbonitride niobium are calculated), allows the modeling of quantitative changes in fractions of phases during the thermo-mechanical processes. In order to verify the multiscale model, the rolling of rods and dilatometric tests were performed. It is seen from calculations and the measurements that the start temperature of ferrite transformation increases with the decreasing speed of cooling and with increasing strain in the material. Hardness of niobium microalloyed steel increases with increasing cooling rate and decrease of strain. For the rolling process precipitates are formed after a shorter time comparing to static recrystallization time in the last pass. These calculations provide information on the incubation period, a fraction of the formation of precipitates and recrystallization during rolling of the material at different temperatures and speeds. A good agreement between measurement and prediction of temperature of start of ferritic transition (F_s) for different conditions of thermo-mechanical treatment for the executed rod rolling process was obtained. The calculations of ferrite and pearlite grain size, radius and number of precipitates and the volume fraction of ferrite and pearlite, are compared with the results of the measurements and a good agreement is observed. Therefore, it can be stated that consideration of the kinetics of precipitation and the dislocation density in the γ–α transition model and combination of these models with the finite element method allows the realistic prediction of microstructure evolution for different conditions of thermo-mechanical treatment.

The simulations provide information about the influence of the precipitation processes on the phase transformation, the period of incubation for the formation of precipitation, austenite grain size and fraction of recrystallization during rolling of the material at different temperatures, strain rates, and different linear band speed. Model of the phase transformation γ–α combined with model of the kinetics of precipitation gives good agreement with the measurement of start temperature of ferrite transformation *Fs*, and good quantitative agreement for volume fractions of phases in the deformed austenite.

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