



The Influence of the Thermodynamic Equilibrium Model of the Fe-M-C-N System on the Results of Calculations of Carbonitride Precipitation Kinetics

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Abstract

The microalloying elements such as: Ti, Nb, V are added into steels to control their microstructure and mechanical properties. High chemical affinity of these elements for interstitials (N, C) results in precipitation of binary compounds, nitrides and carbides and products of their mutual solubility product – carbonitrides. Influence of carbonitrides on the microstructure and mechanical properties depends on their basic stereological parameters: volume fraction, V_v , and their size, r . Proper analysis of the carbonitride precipitation kinetics and the influence of the steel chemical composition on the parameters of M(C,N) carbonitride precipitates formed under isothermal conditions and on the precipitation hardening effect is important in the process of modelling the microstructure and mechanical properties of microalloyed steels. The paper presents a comparison of two thermodynamic equilibrium models: Adrian and Maugis-Gouné, and their impact on the results of calculations of carbonitride precipitation kinetics.

Keywords: Thermodynamic model, Microalloyed steels, Carbonitrides, Precipitations, Mechanical properties

1. Introduction

Microalloyed steels constitute an important group of structural steels. The increasing demand for these steels is associated with their high mechanical properties, good fracture resistance, and weldability, achieved at economically production costs. The high mechanical properties of microalloyed steels result from the addition of small amounts of microalloying elements, such as Ti, Nb, V, or Al (<0.15 wt.%). These elements are introduced separately or comprehensively into steels containing only an increased Mn content (1.5–1.6 wt.%), provided that appropriate processing technology is applied [1, 2]. These steels may also

contain small amounts of elements such as Mo, Cr, Ni, and Cu. Their mechanical properties are additionally influenced by elements such as N, O, S, and P [3]. High chemical affinity of microalloying elements: Ti, Nb, V or Al for interstitials (N, C) results in precipitation of binary compound, nitrides and carbides and products of their mutual solubility – carbonitrides. Their composition depends on the chemical composition of the steel and the temperature. Microalloyed steels usually contain a small amount of aluminium, which is added as a deoxidizing element during steelmaking. Aluminium has a high affinity for nitrogen, forming aluminium nitride (AlN). Due to its hexagonal crystal structure, AlN does not dissolve in carbonitrides. Carbonitride precipitates of microalloying elements and aluminium nitride, due



to their wide range of dissolution temperatures, significantly influence the microstructure and, consequently, the mechanical properties of microalloyed steels.

The compounds formed between microalloying elements and carbon and/or nitrogen, as well as aluminium nitride, act as inhibitors of austenite grain growth at high temperatures. Their effectiveness depends on the chemical composition of the steel, which determines both the volume fraction of precipitates and their stereological parameters, such as particle size and volume fraction. Compounds formed at high temperatures in the liquid phase are the least effective in inhibiting austenite grain growth due to their large size. Among the compounds formed by Ti, V, and Nb with carbon and nitrogen, as well as AlN, only TiN precipitates form in the liquid phase. The remaining compounds precipitate at lower temperatures in the solid state.

Grain refinement is the most beneficial strengthening mechanism, as it increases strength and lowers the ductile-to-brittle transition temperature. A reduction in ferrite grain size positively affects the increase in the yield strength of steel. Solid solution strengthening provided by dissolved Mn, Si, and N is advantageous only in the case of Mn, as it does not cause the decrease in impact toughness observed for the other elements. The addition of Mn lowers the transformation temperature of undercooled austenite, which also contributes to ferrite grain refinement. Precipitation strengthening, manifested by an increase in yield strength ($R_{p0.2}$) while maintaining ductility, results from the presence of undissolved carbide and nitride precipitates of microalloying elements. Knowledge of the chemical composition of austenite, as well as the amount and parameters of undissolved precipitates inhibiting austenite grain growth, is essential for predicting the microstructure and properties of the material.

Thermodynamic models have been developed to calculate the chemical composition of austenite, as well as the composition and volume fraction of carbonitrides [2–6]. These models are based on the regular solution model for stoichiometric phases proposed by Hillert and Staffansson [7]. In steels containing a single microalloying element (Ti, Nb, or V), a carbonitride described by the chemical formula MC_yN_{1-y} is formed, where y is the atomic fraction of carbon in the carbonitride. General information concerning the carbonitrides precipitation process in steels can be found in [7–10].

This paper presents a comparison of two thermodynamic equilibrium models: Adrian and Maugis-Gouné, and their impact on the results of calculations of carbonitride precipitation kinetics. These models were used in the comprehensive computational system for predicting the mechanical properties of microalloyed steels, which has been described and validated in [11,12].

2. Carbides, nitrides, carbonitrides

Precipitation in alloys occurs as a result of heating the solid solution to an appropriate temperature, solutionizing, and subsequent ageing at a specified temperature for a given period of time. The thermodynamic condition required for precipitation is the existence of a single-phase structure at high temperature in the solid state and a two-phase structure at lower temperature. Additionally, decreasing solubility with decreasing temperature and the

possibility of obtaining a supersaturated solution by cooling at an appropriate rate are necessary conditions. Supersaturated solution by cooling at an appropriate rate are necessary conditions. To obtain an alloy with high mechanical properties, precipitates should be distributed as uniformly as possible, with a relatively small particle radius and high number density. The matrix should exhibit relatively high ductility, whereas the precipitates should be hard. Continuous precipitation occurs when the second phase nucleates and grows within the supersaturated primary phase. The formation of the precipitated phase leads to a decrease in the solute concentration in the matrix, and the transformation proceeds uniformly throughout the entire volume of the alloy. The precipitated particles grow until the equilibrium concentration in the matrix is reached. The shape and crystallographic orientation of the matrix grains remain unchanged. Preferred nucleation sites include crystal lattice defects, dislocations, grain boundaries, and stacking faults, where the reduced energy barrier facilitates nucleation. Discontinuous (cellular) precipitation is characterized by the formation of transformation fronts that divide the microstructure. The region through which the transformation front has passed exhibits a modified chemical composition and crystallographic orientation relative to the parent grain from which the front originated. During this process, grain boundaries migrate, and precipitates form behind the moving boundary, only where the transformation front has passed. The transformed region consists of equilibrium phases $\alpha + \beta$, whereas the region ahead of the front remains untransformed and consists solely of phase α . Due to their identical crystal structure (NaCl-type), carbides and nitrides of microalloying elements exhibit complete mutual solubility, resulting in the formation of carbonitrides. Only TiN and ZrN show limited mutual solubility. The lattice parameter of the resulting carbonitride depends on its chemical composition.

The solubility of compounds formed by microalloying elements M reacting with interstitial elements X (C or N) in austenite is described by their solubility products according to the relation [13]:

$$\log_{10}([M][X]) = B - \frac{A}{T} \quad (1)$$

where: [M] and [X] - the concentrations (wt.%) of dissolved alloying element M (M = Ti, Nb, or V) and interstitial element X (X = C or N), respectively, T is the absolute temperature, A and B are constants related to the Gibbs free energy of formation of the compound (A is proportional to the enthalpy of formation and B to the entropy term).

Published literature data on the solubility products of carbides and nitrides formed by microalloying elements exhibit some scatter in the reported values of A and B. A comprehensive compilation of solubility product constants for carbides (TiC, NbC, VC) and nitrides (TiN, NbN, VN, AlN) is presented in [13]. The data selected for practical calculations of austenite composition and precipitate content significantly influence the results.

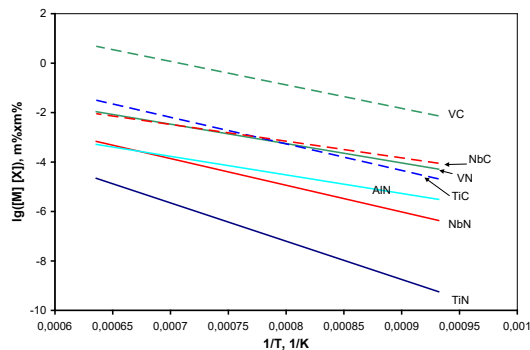


Fig. 1 The comparison of the temperature dependence of the solubility products of carbides and nitrides of V, Ti, Nb, Zr, and AlN [7]

Fig. 1 presents a comparison of the temperature dependence of the solubility products of carbides and nitrides of V, Ti, Nb, Zr, and AlN. The significant differences in chemical affinity of Ti, Nb, and V for carbon and/or nitrogen affect both the dissolution temperature and the chemical composition of the precipitates formed. In general, these elements exhibit a stronger affinity for nitrogen than for carbon. Carbides of Ti, Nb, and V are more soluble than their corresponding nitrides. Particularly large differences in solubility are observed between carbides and nitrides of Ti and V, whereas the difference between NbC and NbN is considerably smaller. Titanium exhibits the strongest affinity for nitrogen, and TiN has the lowest solubility among the compounds considered. Of all analysed compounds, VC is the most soluble, meaning that during cooling of steel it precipitates at the lowest temperature compared with the other compounds. Consequently, vanadium carbonitrides can inhibit grain growth at lower temperatures than titanium carbonitrides. The relatively high solubility of vanadium carbonitrides enables vanadium to perform a dual role: undissolved particles inhibit austenite grain growth, while vanadium dissolved in austenite increases hardenability and contributes to precipitation strengthening of the products of diffusional decomposition of undercooled austenite by V(C,N) particles forming below the A_{r3} temperature. For grain size control at elevated temperatures, TiN is the most effective precipitate. The solubility product of AlN is also shown in **Fig. 1**. Aluminium exhibits high chemical affinity for nitrogen and does not form compounds with carbon. Aluminium nitride has a hexagonal crystal structure and therefore does not participate in the formation of Ti-, Nb-, or V-based carbonitrides.

To improve the mechanical properties of metallic alloys, processes involving precipitation of a second phase are commonly applied. Examples illustrating the benefits of precipitation processes include:

- precipitation strengthening in solution-treated and aged aluminium alloys,
- control of austenite grain size in low-alloy steels by carbide and nitride precipitates.

The parameters characterizing precipitates include:

- crystallography,
- morphology,
- chemical composition,

- particle size distribution,
- volume fraction,
- number density (number of particles per unit volume).

3. Thermodynamic and kinetic models of carbonitride precipitation

Thermodynamic models of carbonitride precipitation under equilibrium conditions provide information on the amount of undissolved precipitates and the chemical composition of austenite. These models are based on thermodynamic calculations for systems containing iron, microalloying elements (V, Nb, Ti, Zr, and Al), and C and/or N. One of the most comprehensive thermodynamic models describing precipitation of carbides, nitrides, and carbonitrides is the Adrian model [13,14]. This model includes both simple compounds (carbides and nitrides) and complex carbonitrides. In the case of complex precipitates, one to three substitutional elements exhibiting chemical affinity for both carbon and nitrogen may be present in austenite. The model also accounts for the presence of aluminium in steel. Multicomponent systems containing one to three substitutional elements are described using the regular solution model for interstitial phases proposed by Hillert and Staffansson. The model is based on the following assumptions [13]:

- substitutional and interstitial elements (C, N) form dilute solutions in austenite and obey Henry's law;
- reactions between substitutional and interstitial elements lead to the formation of a carbonitride with ideal stoichiometry, where the total number of metal atoms equals the total number of carbon and nitrogen atoms;
- metal atoms and interstitial atoms occupy two independent sublattices in the carbonitride;
- vacancies in both sublattices and dissolution of iron in the carbonitride are neglected.

For a system containing a single microalloying element ($Fe-M-C-N$), the thermodynamic equilibrium conditions according to Adrian are expressed by the system of equations [13,14]:

$$\begin{cases} \ln \frac{y k_{MC}}{[M_a][C_a]} + (1-y)^2 \frac{L_{CN}}{RT} = 0 \\ \ln \frac{(1-y) k_{MN}}{[M_a][N_a]} + y^2 \frac{L_{CN}}{RT} = 0 \end{cases} \quad (2)$$

where: k_{MX} - the solubility product of carbide or nitride MX, and L_{CN} is the interaction parameter describing the mutual interaction between the metallic element M and the interstitial elements C and N. Its value is commonly taken as -4260 J/mol [13].

The above equations are supplemented by mass balance equations for the elements forming the carbonitride MC_yN_{1-y} .

An alternative approach is the Maudis-Gouné (MG) model [15], which can be used for quantitative description of precipitation kinetics of single- and multicomponent phases in ferrite and austenite during heat treatment, when nucleation, growth, and coarsening occur simultaneously. Based on kinetic functions, time dependencies of nucleation rate (J), particle radius (r), number

density (N_V), volume fraction (V_V), particle size distribution $N_V(r)$, and matrix concentration, it is possible to determine the time ranges corresponding to individual precipitation stages. In the case of V(C,N), the C/N ratio depends on the steel composition and heat treatment parameters. Therefore, V(C,N) is assumed to be an ideal solid solution of VC and VN, and particle growth is controlled by vanadium diffusion in austenite.

The MG model considers:

- chemical composition of nuclei,
- nucleation rate,
- growth rate,
- time-dependent change in precipitate composition,
- coarsening (Ostwald ripening).

The principal assumptions of the MG model are:

- precipitates are spherical,
- V(C,N) is an ideal solid solution of VC and VN,
- nucleation is homogeneous and follows classical nucleation theory,
- growth is diffusion-controlled by vanadium diffusion in the matrix,
- coarsening is governed by the Gibbs–Thomson effect.

The thermodynamic equilibrium conditions for the Fe–V–C–N system are expressed as [15]:

$$\begin{cases} yK_{VC} = X_V^i X_C^i \\ (1-y)K_{VN} = X_V^i X_N^i \end{cases} \quad (3)$$

where: K_{VC} and K_{VN} are the solubility products of VC and VN, respectively.

The system of equations (3), together with mass balance equations, was used to calculate the equilibrium concentrations of V, C, and N in the solid solution as well as the composition of the V(C,N) carbonitride, y , VC_yN_{1-y} .

The MG model considers three stages of precipitation: nucleation, growth, and coarsening. However, the MG model is somewhat simplified as it neglects the interaction parameter between the metallic element and interstitials, $L_{CN} = 0$. In contrast, the Adrian model (2) includes L_{CN} , making it closer to the real thermodynamic process.

In this work, the influence of the chosen thermodynamic equilibrium model (Adrian, (2), MG, (3)) on the predicted kinetics of M(C,N) precipitation was analyzed. Based on this analysis, an optimal computational approach was selected for implementation in a comprehensive numerical system developed to predict mechanical properties of microalloyed steels.

The precipitation kinetics model developed here, based on [16], employs a Lagrange approximation. The chemical composition of the outer layer of the growing MC_yN_{1-y} carbonitride is obtained by solving the following system of nonlinear equations:

$$\begin{cases} \ln \frac{y_i K_{MC}}{X_M^i X_C^i} + (1-y_i)^2 \frac{L_{CN}}{RT} = 0 \\ \ln \frac{(1-y_i) K_{MN}}{X_M^i X_N^i} + y_i^2 \frac{L_{CN}}{RT} = 0 \\ D_C(X_C^i - X_C) = y_i D_M(X_M^i - X_M) \\ D_N(X_N^i - X_N) = (1-y_i) D_M(X_M^i - X_M) \end{cases} \quad (4)$$

where: X_M, X_C, X_N are atomic fractions of M, C, and N in the matrix; X_M^i, X_C^i, X_N^i denote concentrations at the matrix–precipitate interface; D_M, D_C, D_N are diffusion coefficients; and $K_{MC}(r), K_{MN}(r)$ are solubility products corrected for the Gibbs–Thomson effect.

Equation (4) represents the approximate thermodynamic equilibrium at the precipitate–matrix interface and differs from the MG model, which assumes $L_{CN} = 0$. Following Grieveson [13], the interaction parameters for V and Nb with interstitials are assumed equal to that of Ti: $L_{CN}^V \approx L_{CN}^{Ti} \approx L_{CN}^{Nb} = L_{CN} = -4260 \text{ J/mol}$.

Two variants were implemented in this study: the “simplified version” ($L_{CN} = 0$) and the “complete version” ($L_{CN} = -4260 \text{ J/mol}$). Computation times differed significantly due to the complexity of solving the nonlinear system. For steel containing 0.2% C, 0.015% N and 0.2% V, aged at 820°C after solution treatment at 1200°C for 100 minutes, computation times on a laptop (16 GB RAM, Intel i7, 8 cores, 3.4 GHz) were 50 s for the simplified version and 142 s for the complete version. Despite the difference, the predicted particle radius distribution differed by less than 1%. Therefore, the simplified version was more frequently used in subsequent analyses due to its efficiency, while the complete version was used for verification.

4. The influence of the thermodynamic equilibrium model of the Fe–M–C–N system

Using the developed computational system, a comparative analysis of the precipitation kinetics of V(C,N) was performed using the Adrian (2) and MG (3) models. For steel containing 0.2% C, 0.015% N and 0.2% V, heated to 1200°C and held at 820°C for 10000 min, the results are shown in Fig. 2-8.

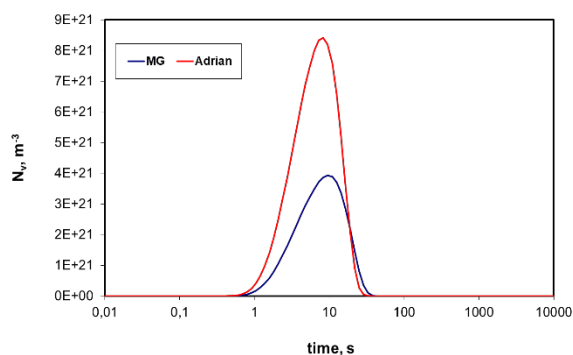


Fig. 2. The comparison of the parameters of the analysis of the kinetics of carbonitride V(C,N) (Adrian and MG models), $J=f(\text{time})$

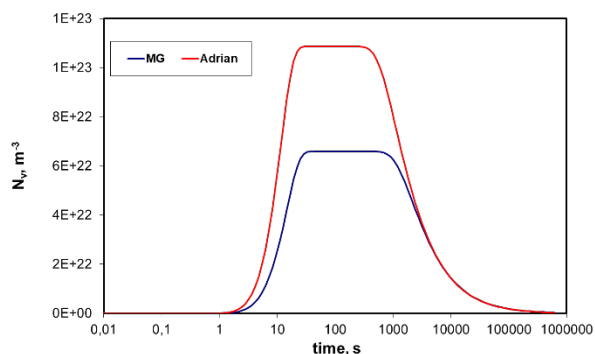


Fig. 3. The comparison of the parameters of the analysis of the kinetics of carbonitride V(C,N) (Adrian and MG models), $N_v=f(\text{time})$

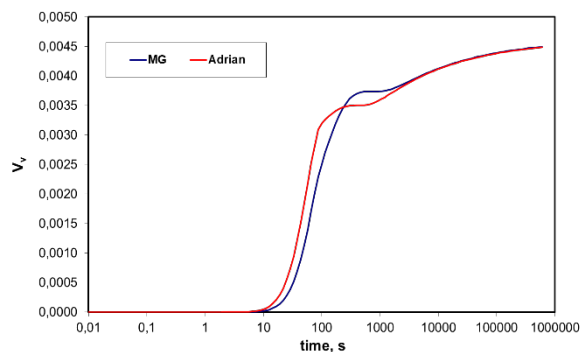


Fig. 4. The comparison of the parameters of the analysis of the kinetics of carbonitride V(C,N) (Adrian and MG models), $V_v=f(\text{time})$

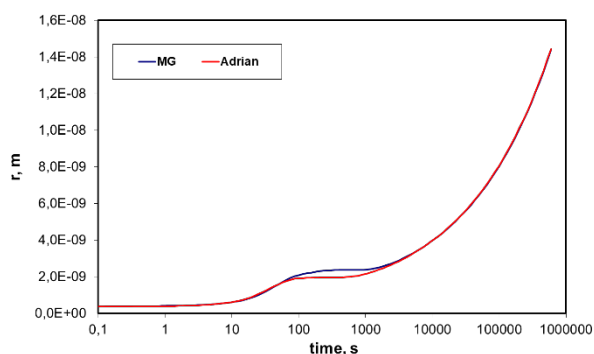


Fig. 5. The comparison of the parameters of the analysis of the kinetics of carbonitride V(C,N) (Adrian and MG models), $r=f(\text{time})$

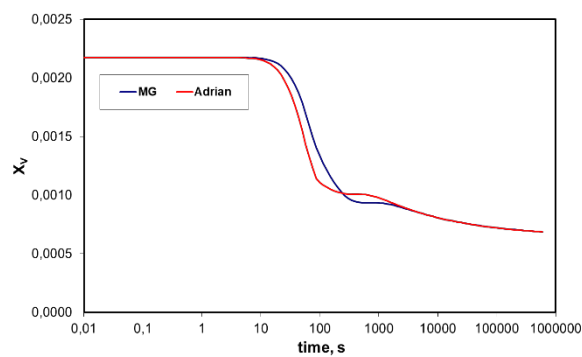


Fig. 6. The comparison of the parameters of the analysis of the kinetics of carbonitride V(C,N) (Adrian and MG models), $X_v=f(\text{time})$

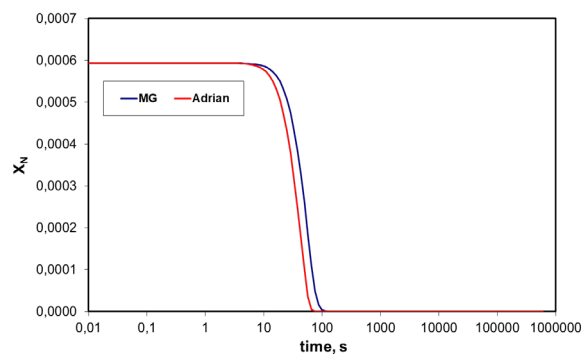


Fig. 7. The comparison of the parameters of the analysis of the kinetics of carbonitride V(C,N) (Adrian and MG models), $X_N=f(\text{time})$

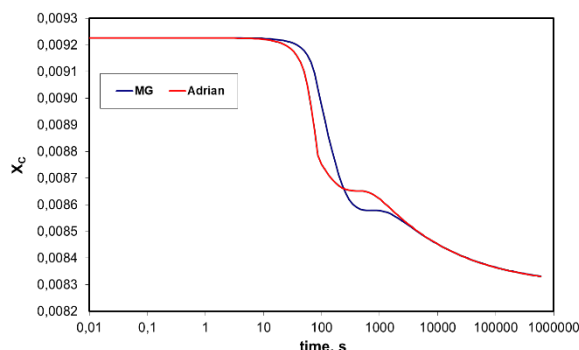


Fig. 8. The comparison of the parameters of the analysis of the kinetics of carbonitride V(C,N) (Adrian and MG models), $X_C=f(\text{time})$

As can be seen, the compared results exhibit significant differences, particularly during the second stage of precipitation: the growth of particles. The maximum nucleation rate, J , and the number of precipitates, N_V , calculated using the Adrian model are substantially higher than those obtained with the Maugis model. As previously mentioned, the computation time for the precipitation cycle using the Adrian model, both for calculating the equilibrium composition of the matrix and the composition of the growing precipitate layer is considerably longer compared to the MG model.

A calculation variant was therefore tested in which the Adrian model was used for determining the equilibrium composition of the matrix (complete version), while a simplified model was employed to calculate the chemical composition of the growing precipitate layer, assuming $L_{CN} = 0$ (simplified version). The differences between the compared parameters and those obtained using the full Adrian model (complete version) were negligible. An example of the compared results is shown in Fig. 9-11. Consequently, for the analysis of carbonitride precipitation kinetics and the calculations, a modified approximate method was adopted due to the shorter computation time. In this approach, the equilibrium chemical composition of the matrix is calculated using the Adrian model (system of equations (2)), whereas the chemical composition of the growing precipitate layer is determined using the MG model (system of equations (3)).

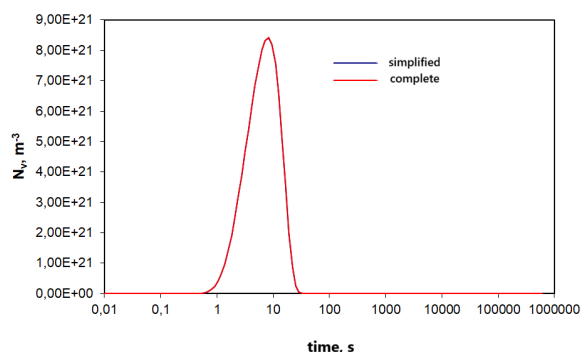


Fig. 9. The comparison of selected precipitation parameters of vanadium carbonitride V(C,N) using the complete and the simplified method: $J = f(\text{time})$

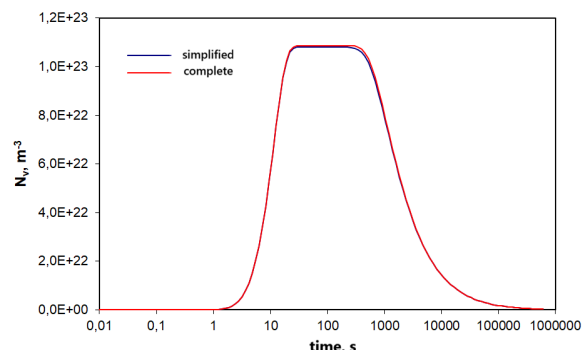


Fig. 10. The comparison of selected precipitation parameters of vanadium carbonitride V(C,N) using the complete and the simplified method: $N_V = f(\text{time})$

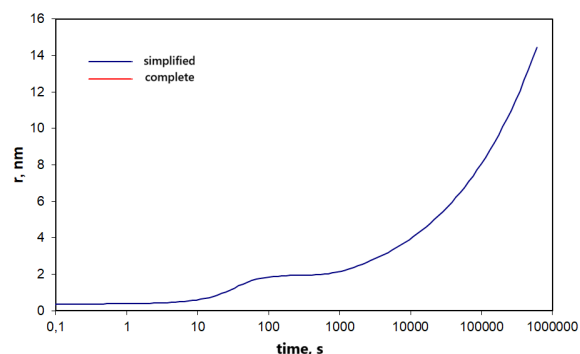


Fig. 11. The comparison of selected precipitation parameters of vanadium carbonitride V(C,N) using the complete and the simplified method: $r = f(\text{time})$

The calculated parameters can be used to analyze the hardenability of steel (austenite chemical composition) as well as to predict changes in grain size and the mechanical properties of steels containing a microalloying element (stereological parameters: r , V_V).

5. Example of application

The developed and implemented comprehensive computational system was validated by comparing its results with the experimental data on Nb(C,N) carbonitride precipitates presented in [17]. Maugis et al. [17] investigated the precipitation kinetics of Nb(C,N) in steel with the chemical composition of 0.0059%C, 0.0064%N, 0.0843%Nb, and 0.0013%Mn. Laboratory-cast ingots were hot-rolled and austenitized at a high temperature to fully dissolve the precipitates, followed by isothermal aging at 650°C and 750°C for various times, after which the samples were quenched in water. The carbonitride precipitation occurred in the ferrite phase. As can be seen in Figure 4, there are some differences in the dependence of precipitate radius, r , on holding time at 650°C and 750°C over the entire investigated time range. The experimental values of the average precipitate radius at 750°C lie

above the calculated curve, whereas at 650°C they are below. These differences may result from other necessary simplifications applied in the computational system. Considering that the deviations are on the order of a few nanometers, they are considered acceptable. Results of validation is shown on Fig. 12.

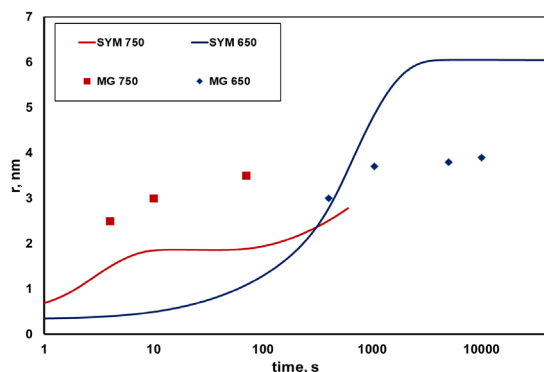


Fig. 12. The comparison of experimental [17] and calculated dependencies of the average radius of Nb(C,N) precipitates on holding time at the specified temperature for steel C at 650°C and 750°C

6. Conclusions

The mechanical properties of microalloyed steels are determined by their chemical composition and the parameters of the processing route, which influence the size of the carbonitride precipitates. In these steels, carbonitride precipitates play a dual role: in the austenite phase, they inhibit grain growth, while in the ferrite phase, they contribute to precipitation strengthening. Grain refinement resulting from the decomposition of austenite together with precipitation strengthening, allows for high mechanical properties of the steel after cooling and plastic deformation, without the need for additional heat treatment.

The final effect of microalloying elements such as V, Nb, and Ti, added either individually or in combination, depends on two stereological characteristics of the resulting carbonitride precipitates: their volume fraction, V_p , and the size distribution of the precipitates. These parameters can be calculated based on the steel's chemical composition and processing conditions using an appropriate model. The selection of the proper model, as well as potential modifications, has a significant impact on the obtained results.

In this work, a comprehensive computational system developed by the author is presented. It applies the classical nucleation theory based on the general Kampmann-Wagner model [16] and compares the results using the MG [15] and Adrian [13] approaches. Each model individually produced results that differed both in the individual stages of the process and in computational time. The obtained results allowed for an appropriate modification involving partial application of both the MG thermodynamic model and Adrian's approach. A positive outcome of this modification was a significant acceleration of the simulation time, a critical factor, while the simulation results remained very similar to each other.

Despite the simplified version of the model, it allows for the prediction of trends in the precipitation kinetics caused by changes in the steel's chemical composition. The developed model enables the analysis of carbonitride precipitation kinetics under isothermal conditions in steels containing one of the commonly used microalloying elements: V, Nb, or Ti.

A key advantage of the developed model is its ability to track the individual stages of precipitation kinetics (nucleation, growth, and coarsening) that occur simultaneously. The calculation results strongly depend on the physical parameters of the model, particularly the interfacial energy, which can be influenced by both the chemical composition of the precipitates and the matrix, as well as by temperature.

The model needs further development, which should include:

- Extending the range of chemical composition of steel for case of simultaneous addition of several microalloying elements in steel, on the carbonitride precipitation process.
- Including of calculation the temperature field during heat treatment processes and its effect on size distribution of carbonitride particles.
- More experimental data and samples to compare with numerical model.
- Calculation of final mechanical properties of steel after heat treatment.

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