

## AUSTENITE GRAIN GROWTH CALCULATION OF 0.028% Nb STEEL

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### Abstract

*Modeling of microstructural evolution has become a powerful tool for materials and process design by providing quantitative relationships for microstructure, composition and processing. Insufficient attention has been paid to predicting the austenite grain growth of microalloyed steel and the effect of undissolved microalloys. In this research, we attempted to calculate a mathematical model for austenite grain growth of 0.028% Nb steel, which can account for abnormal grain growth. The quantitative calculation of austenite grain growth generated from this model fit well with the experimental grain growth data obtained during reheating of niobium steels. The results of this study showed that increasing the temperature increases the austenite grain size, with a sharp gradient observed at higher temperatures.*

*Keywords: Austenite; Grain growth; Calculation; Niobium steel.*

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### 1. Introduction

It is well established that grain growth is the evolution of a microstructure due to the motion of grain boundaries driven by the reduction in grain boundary interfacial energy [1]. Given a sufficiently high temperature and no factors impeding grain

boundary migration, a polycrystalline material will evolve towards a single crystal.

In reality, this goal is rarely attained due to the unavoidable defects and impurities in materials, even for high purity zone refined metals.

The grain growth is characteristic of the growth of larger grain at the expense of

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smaller ones, leading to an increase in average grain size [1]. Grain growth control in microalloyed steels during hot working processing, critically depends on second phase particles [2, 3]. The appropriate employment of microalloying elements in high strength low alloy (HSLA) steels, coupled with hot working, can provide improvements in both strength and toughness [4, 5, 6]. This is achieved by suitable manipulation of the recrystallization and precipitation phenomena that take both place during deformation.

Austenite grain growth in microalloyed steels is influenced by numerous factors, including austenizing time and temperature, alloy composition, hot working history, initial grain size distribution, and rate of heating to the austenitizing temperature [6-8]. By suppressing austenite recrystallization, these elements act as ferrite grain refiners, thus increasing the yield strength and decreasing the impact transition temperature [6].

Manohar *et al.* [9], predicted the austenite grain growth for microalloyed steels, especially for Ti steel. They assumed that fine TiN precipitates control the austenite grain growth behavior at all temperatures. The influence of possible mixed precipitates such as Nb(C, N) was not accounted for in their mathematical model. Referring to their empirical predictions, the aim of this study is to observe and recalculate the austenite grain size and the influencing behavior of Nb(C, N) precipitates during reheating of Nb microalloyed steels.

Table 1. Steel Composition (Weight Percent)

C	Si	Mn	P	S	Al	Nb	Cu	N	Fe
0.085	0.222	1.45	<0.003	<0.003	0.049	0.028	0.045	<0.003	98.14

## 2. Experimental Procedures

Commercial Nb-microalloyed steel was used for this study; detailed composition of the steel is provided in Table 1. Rectangular samples in 30 mm width and 60 mm length (parallel to the rolling direction) were machined from hot-rolled coil (HRC) material. A hole of 2 mm in diameter was drilled in the center of a longitudinal-transverse (L-T) section of the sample for a thermocouple insertion.

Heat treatment of the sample fitted with the thermocouple was performed by heating at a constant rate of  $0.3^{\circ}\text{C}\cdot\text{s}^{-1}$  to temperatures of 900, 1000 and 1100 °C, followed by holding for 10 minutes to permit full austenitization of the material. Then the specimens were quenched in water to view the austenite boundary. Light microscopy of steel plates was conducted in an Olympus metallurgical, microscope on 4% picral-etched specimens.

## 3. Empirical Model

Empirical equations are widely used to describe grain growth behavior during isothermal heating. The power law relationship was first specified by Beck *et al.* (1948) for normal grain growth during isothermal conditions [10]:

$$d^n - d_o^n = k \cdot t \quad \dots(1)$$

where  $n$  is the grain growth exponent,  $k$  is the reaction rate constant, and  $d^n$  and  $d_o^n$  are grain sizes at time  $t$  and  $t=0$ , respectively. It

has been found by Sellars and Whiteman in 1978 that the  $k$  value can be expressed by an Arrhenius type equation [11]. They previously published grain growth data on low carbon-manganese steel. They arrived at the following general expression for evaluating the constant  $k$  in Eq. (1) as :

$$d^n - d_o^n = [A \cdot \exp(-Q_{gg}/RT)] \cdot t \quad \dots(2)$$

where  $n$  and  $A$  are constants which depend on material composition and processing conditions,  $Q_{gg}$  is the activation energy for grain growth,  $R$  is the universal gas constant and  $T$  is the temperature in Kelvin [10]. Empirical equations for grain growth of C-Mn steels are included in Table 2.

Yoshie and Nishizawa [9] presented and proposed another empirical model to predict grain growth of austenite in cast C-Mn steel. Their model represented the  $k$  constant as :

$$k = \sigma \cdot V \cdot D_{gb}/\lambda \cdot R \cdot T \quad \dots(3)$$

$$\text{and, } D_{gb} = D_{gb}^o \cdot \exp(-Q_D/RT)$$

where  $\sigma$  is grain boundary energy ( $= 800 \cdot 10^{-7} \text{ Jcm}^{-2}$ ) [9],  $V$  is the molar volume of austenite ( $= 6.97 \text{ cm}^3\text{mol}^{-1}$ ) [9],  $D_{gb}$  is the

diffusion constant at grain boundary for iron ( $\text{cm}^2\text{s}^{-1}$ ),  $D_{gb}^o$  is the diffusion coefficient ( $= 2.0 \text{ cm}^2\text{s}^{-1}$ ) [9],  $Q$  is activation energy for diffusion ( $= 171544 \text{ Jmol}^{-1}$ ) [9],  $R$  is the universal gas constant ( $= 8.31 \text{ Jmol}^{-1}\text{K}^{-1}$ ),  $T$  is the absolute temperature (K) and  $\lambda$  is thickness on grain boundary ( $=$  lattice parameter of austenite  $= 3.59 \cdot 10^{-10} \text{ mm}$ ) [9].

Empirical models were compared to observe relationships with each other. Assuming a hypothetical typical value for  $d_{RX}$  of  $80 \mu\text{m}$  and solving all above models by substituting  $T = 860, 960$  and  $1060 \text{ }^\circ\text{C}$ , for holding time  $t = 10 \text{ s}$  and  $1000 \text{ s}$ , the results obtained are shown in Figs. 1 and 2.

It can be observed from Figs.1 and 2, using empirical models for predicted grain growth of austenite in C-Mn steels giving different results. In general, if increase the holding time will take effect to  $d_o^n \ll d^n$  in C-Mn steels, then the Eq. (1) can be simplified to:

$$d^n = k \cdot t \quad \dots(4)$$

so the values of  $n$  and  $k$  can be obtained by regression of experimentally measured grain sizes and holding time [1].

Table 2. Summary of empirical models describing austenite grain growth [9,17].

Source	Steel	Austenite Conditions	$N$	$K$	$Q_{gg}$
Sellars <i>et al.</i> , (1979)	C – Mn	Statically recrystallized	10	$3.87 \cdot 10^{32}$ for $T > 1000^\circ\text{C}$ $10^{53}$ for $T < 1000^\circ\text{C}$	400000 for $T > 1000^\circ\text{C}$ 914000 for $T < 1000^\circ\text{C}$
Namba <i>et al.</i> , (1979)	Low C-Mn	Statically recrystallized	2	$4.27 \cdot 10^{12}$	66600
Hodgson and Gibbs (1992)	C-Mn and C-Mn-V	Statically or Metadynamically recrystallized	7	$1.45 \cdot 10^{27}$	400000
Hodgson and Gibbs (1992) Beynon and Sellars (1992)	C-Mn-Nb	Statically or Metadynamically recrystallized	4.5	$4.1 \cdot 10^{23}$	435000

The empirical model of C-Mn-Nb steel either underestimates or overestimates grain sizes due to its inability to account for undissolved microalloyed effects on austenite grain growth (Fig. 1 and 2). Actually, in microalloyed steels, e.g., Nb microalloyed steel, the austenite grain growth kinetics are expected to be slower than in plain C-Mn composition due to solute drag [12]. The final grain size after a certain heat treatment does not satisfy the requirement in Eq. (1), and therefore,

neglecting of initial grain size  $d_0^n$  would give rise to a large deviation on predicted values from the model. It is obvious that the grain growth exponent  $n$  is dependent on the temperature and materials. As a consequence, when Beck's formula is employed under continuous heating conditions, the  $n$  value should correlate with temperature [1]. So, developing an empirical model, to predict the undissolved microalloyed effect on austenite grain growth in Nb microalloyed steels is needed.

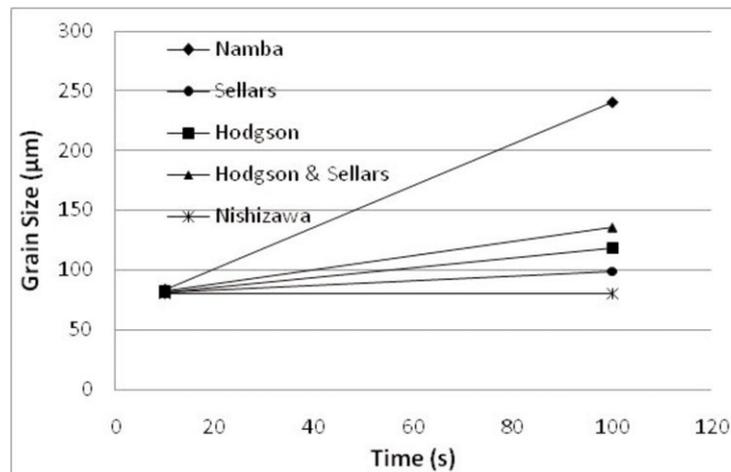


Fig. 1. Predicted austenite grain growth in C-Mn Steels,  $t = 10$  s

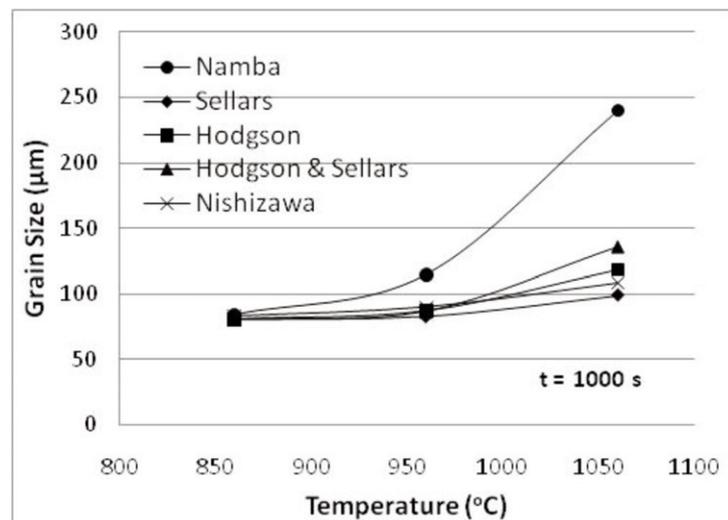


Fig. 2. Predicted austenite grain growth in C-Mn Steels,  $t = 1000$  s.

## 4. Calculation of a Model

### 4.1. Background

In the presence of second phase particles (such as in Nb microalloyed steels), a fundamental approach in identifying a theoretical solution for the grain growth behavior, centers on finding a hypothetical grain radius  $R_{cr}$ , such that any grain with radius equal to  $R_{cr}$  will neither grow nor shrink [9]. Any grain with a radius equal to  $R_{cr}$  is considered to be thermodynamically stable because the driving force for grain growth of such a grain exactly balances the pinning force exerted by particles on the moving grain boundary.

Zener proposed that the driving pressure for grain growth due to the curvature of the grain boundary would be counteracted by a pinning (drag) pressure exerted by the particles on the boundary. Zener [10] first assigned a quantitative value for a critical maximum grain radius  $R_{cr}$  as:

$$R_{cr} = k_z \cdot r/f_v \quad \dots(5)$$

where  $k_z$  is a constant,  $r$  is the mean particle radius,  $f_v$  is the volume fraction of particles. In the original Zener model,  $k_z$  was set to 4/3.

Experimental data for the ratio of limiting grain radius to particle radius ( $R_{cr}/r$ ) as a function of volume fraction of particles ( $f_v$ ) shows that the value of solubility of (Ti,Nb)CN in austenite phase is lies directly on the line for  $k_z = 0.17$  [9] (Fig. 3).

Gladman [13] analysed the effect of grain sized distribution on the driving pressure (and therefore  $R_{cr}$ ) and found that :

$$R_{cr} = [\pi(0.25 - 0.33/Z)] \cdot r/f_v \quad \dots(6)$$

where  $Z$  is the ratio of growing grains to matrix grains ( $r_{max}/r_{mean}$ ), thus representing the heterogeneity of grain size distribution in the sample. Gladman found that  $Z$  values between 1.41 and 2 correlated closely with the experimental results. Thus, the value of the constant  $k_z$  could be between 0.05 and 0.26. So the value of  $k_z = 0.17$  (Fig.3) close to the Gladman value of  $k_z$ , then

$$R_{cr} = 0.17 \cdot r/f_v = d/2 \quad \dots(7)$$

where  $d$  is the critical grain diameter.

Solving Eq. (7) thus facilitates the calculations of stable austenite grain size under any combination of volume fraction and particle radius of precipitates. The dissolution and coarsening of Nb microalloy precipitates therefore play a pivotal role in deciding the stable austenite grain size. The methodology used to estimate these two parameters is described in the following sections.

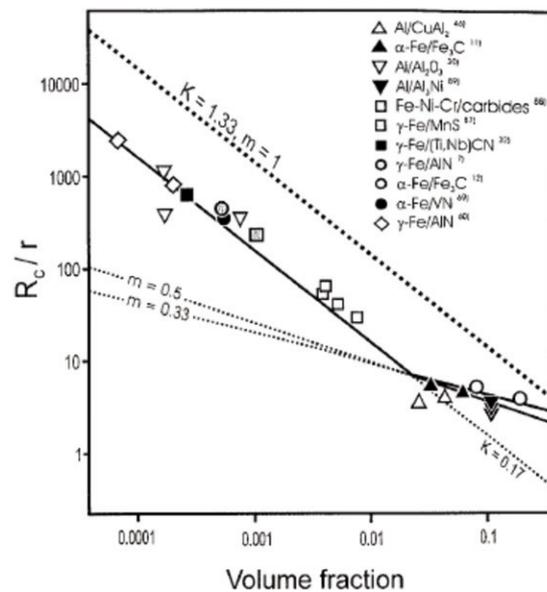


Fig.3. Experimental data of ratio of limiting grain radius to particle radius ( $R_{cr}/r$ ), as a function of volume fraction of particles ( $f_v$ ) [13].

## 4.2. Calculating Nb(C,N) Solubility

Choice of the equation describing solubility of Nb(C,N) in austenite is very important because partitioning of niobium in precipitates and in austenite matrix plays a crucial role in deciding grain growth of microalloyed austenite. In the current work, we have used the Nb(C,N) solubility product equation [10] :

$$\begin{aligned} \log[\text{Nb}][\text{C}]^{0.83}[\text{N}]^{0.14} &= 4.46 - 9800/T \\ [\text{Nb}][\text{C}]^{0.83}[\text{N}]^{0.14} &= \\ \log^{-1}(4.46 - 9800/T) &\dots(8) \end{aligned}$$

Multiplying both sides by  $[\text{Nb}]/[\text{C}]^{0.83}[\text{N}]^{0.14}$  results in:

$$[\text{Nb}]^2 = [\log^{-1}(4.46 - 9800/T)] \cdot \{[\text{Nb}]/[\text{C}]^{0.83}[\text{N}]^{0.14}\} \dots(9)$$

It is assumed that the formation and dissolution of Nb(C,N) precipitates proceed such that stoichiometric relationships of Nb:C [=7.74] and Nb:N [=6.63] [10] are maintained whenever the overall Nb(C,N) ratio in the given composition of steel is close to the stoichiometric ratio of  $0.14 \cdot [\text{Nb}]/[\text{Nb}]^{0.97}$ . Therefore, substituting  $[\text{Nb}]/[\text{C}]^{0.83}[\text{N}]^{0.14} = 0.14 \cdot [\text{Nb}]/[\text{Nb}]^{0.97}$  in Eq. (9) gives:

$$[\text{Nb}]^{1.97} = [\log^{-1}(4.46 - 9800/T)] \cdot 0.14 \dots(10)$$

Using this equation, the amount of niobium out of solution, i.e., in precipitate

form ( $X^p$ ), can now be calculated as =Total Nb- $X^s$ , where  $X^s$  is the percent of niobium in the solution of austenite for differing values of T. Total niobium in this case is 0.028%. Results of these calculations are presented in Table 3.

## 4.3. Calculating Volume Fraction of Precipitates

Volume ' $V$ ' of a given mass ' $m$ ' of a material can be given as [9]:

$$V = m \cdot Na \cdot Vu / (Nu \cdot Ar) \dots(11)$$

where  $Na$  is Avogadro's number,  $Vu$  is the volume of one unit cell of the material,  $Nu$  is the number of atoms per unit cell of the material, and  $Ar$  is the atomic weight of the material. Nb(C,N) phase is regarded as a complete ideal solid solution of NbC and NbN because the interaction between C and N in Nb(C,N) can be ignored [14]. Therefore, the volume and the mass of Nb(C,N) can be written as:

$$\begin{aligned} V_{\text{Nb(C,N)}} &= V_{\text{NbC}} + V_{\text{NbN}} \\ &= 0.128 \cdot m_{\text{NbC}} + 0.119 \cdot m_{\text{NbN}} [15] \dots(12) \end{aligned}$$

where

$$\begin{aligned} m_{\text{NbC}} &= m_{\text{Nb}} + m_{\text{C}} = m_{\text{Nb}} + (m_{\text{Nb}}/7.74) = \\ &1.129 \cdot m_{\text{Nb}} \\ m_{\text{NbN}} &= m_{\text{Nb}} + m_{\text{N}} = m_{\text{Nb}} + (m_{\text{Nb}}/6.63) = \\ &1.151 \cdot m_{\text{Nb}} \end{aligned}$$

Table 3. Predicted partitioning of Nb in dissolved and precipitated forms at temperatures of interest

Temperature (°C)	[Nb]= $X^s$ =Predicted % Nb in solution in austenite	$X^p$ = Predicted % Nb in ppt form = 0.028- $X^s$
860	0.00102	0.02698
960	0.00435	0.02365
1060	0.01489	0.01311

so  $V_{Nb(C,N)} = 0.282 \cdot m_{Nb}$  and  
 $V_{Fe} = 0.125 \cdot m_{Fe}$  [9] ... (13)

where  $m_{Fe} = 1 - m_{Nb(C,N)}$  and  
 $m_{Nb(C,N)} = X^p/100$ .

Volume fraction of precipitates can now be calculated from:

$f_v = V_{Nb(C,N)} / (V_{Nb(C,N)} + V_{Fe})$  ... (14)

For different values of T we can thus calculate the corresponding volume fraction of precipitates (Fig. 4).

**4.4. Calculating Particle Coarsening**

The Nb(C,N) particle in Nb-HSLA steels plays an important role in retarding the recrystallization process. After the formation of the new phase, the high density of small precipitation tends to coarsen into a lower density of larger particles with a smaller total interfacial area. The diffusional growth and diminution of precipitated particles in a solid matrix is generally known as Ostwald ripening [16].

For evaluation purposes, we assessed particle coarsening in terms of the mean cube

radius and the Ostwald ripening theory. According to the Wagner's diffusion-controlled model [16], particle size under Ostwald ripening conditions can be described by:

$r^3 - r_o^3 = k \cdot t$  ... (15)

Wagner, Lifshitz and Slyozov analyzed the particle coarsening more rigorously, maintaining that the growth process begins when the grains (of second phase) have reached appreciable size and the degree of supersaturation of the matrix has become very slight [9]. In this situation, coalescence starts (i.e., growth of larger grains by incorporation of smaller ones begins). This theory predicts particle grain growth through the equation given below:

$r^3 - r_o^3 = [8 \cdot \sigma \cdot V \cdot D \cdot t \cdot X^s] / [9 \cdot R \cdot T]$  ... (16)

where  $r$  is the average final particle radius (cm) at time  $t$ ,  $r_o$  is the average initial particle radius (cm) at time  $t_o$ ,  $\sigma$  is interfacial energy (= grain boundary energy =  $3 \cdot 10^{-7} \text{ Jcm}^{-3}$ ) [14],  $V$  is the molar volume of Nb(C,N) ( $= 6.72 \text{ cm}^3 \text{ mol}^{-1}$ ) [14],  $D$  is the diffusivity of solute (Nb) in matrix (austenite) ( $D = \text{cm}^2 \text{ s}^{-1}$ )

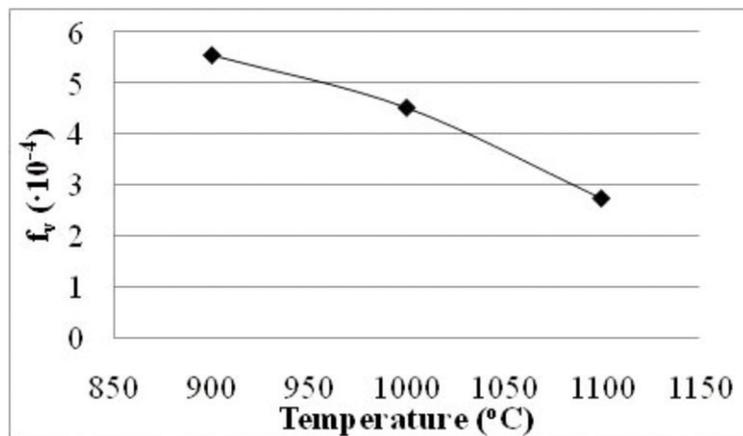


Fig. 4. Predicted volume fraction of precipitate at temperatures of interest.

<sup>1</sup>),  $t$  is the time for particle coarsening (s),  $X^s$  is the concentration of the saturated solution (Table. 3),  $R$  is the universal gas constant (=  $8.31 \text{ Jmol}^{-1}\text{K}^{-1}$ ) and  $T$  is the absolute temperature (K).

Diffusivity of Nb in austenite [14] is given by:

$$D_{\text{Nb}} = 5.6 \cdot \exp(-286000/RT) \text{ cm}^2\text{s}^{-1} \quad (17)$$

Niobium diffusivities are given in Fig. 5, for selected temperatures.

By considering initial mean particle radius,  $r_0 = 1.3 \cdot 10^{-7} \text{ cm}$  [14],  $t = 600 \text{ s}$ , and substituting appropriate numerical values in

Eq. (16), we can estimate particle coarsening at different temperatures (Fig. 6).

#### 4.5. Calculation of Austenite Grain Size

The micrographs of the samples after continuous heating at different temperatures held for  $t = 600 \text{ s}$  are shown in Fig. 7. The measured mean linear intercept and the calculated real grain size, combined with the substitution of predicted  $f_v$  values (Fig. 4) and predicted  $r$  values (Fig. 6) in Eq. (7), are predictive of stable austenite grain size (Figs. 8 and 9).

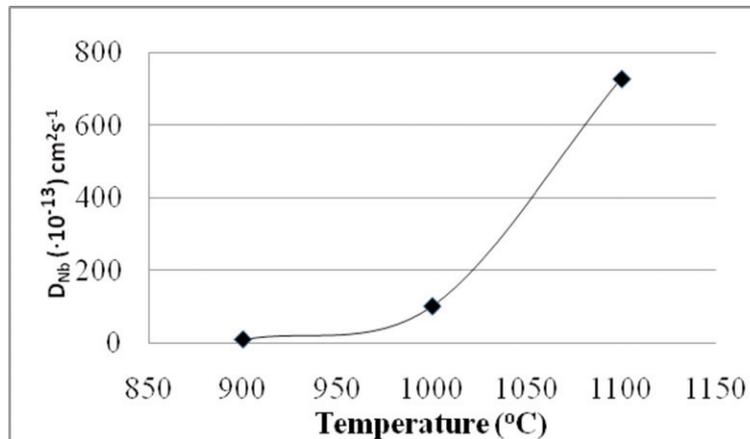


Fig. 5. Diffusivity of Nb in austenite at temperatures of interest

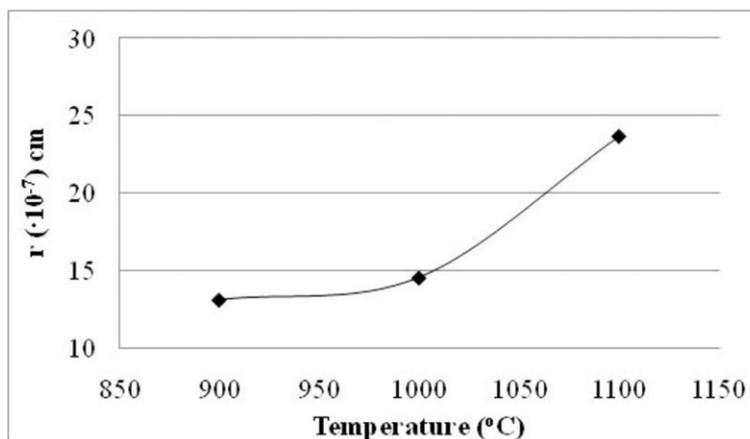


Fig. 6. Predicted particle coarsening with respect to temperature at  $t = 600 \text{ s}$ .

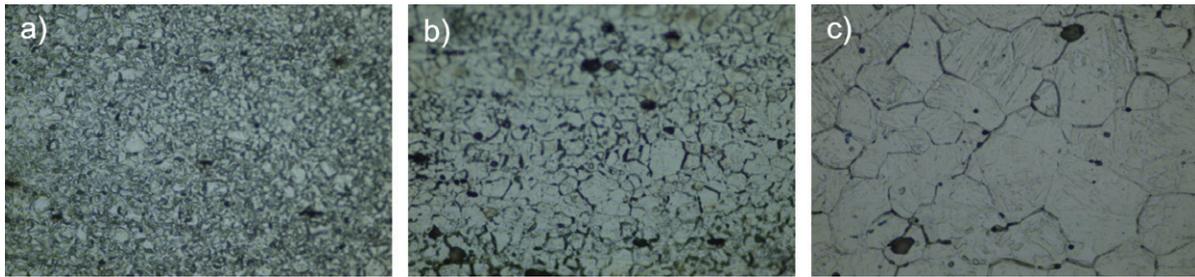


Fig. 7. Micrographs grain size (1000x) at different temperatures in a continuous reheating at various temperatures held for 600 s, a). 860°C, b). 960°C, c). 1060°C.

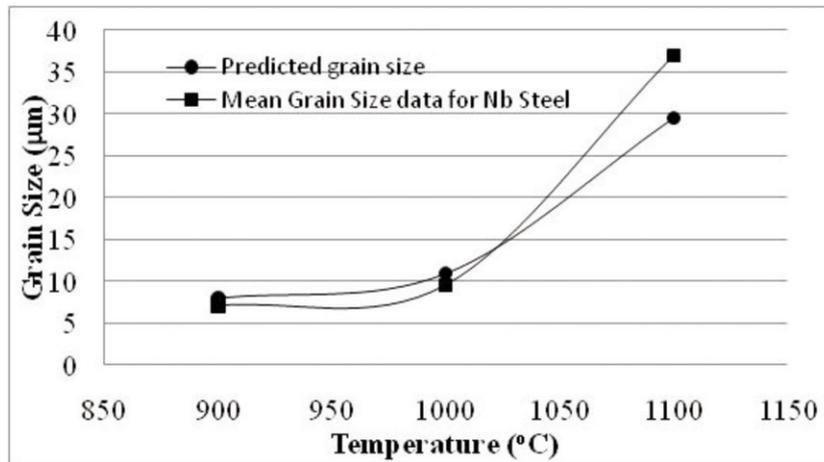


Fig. 8. Comparison of predicted and experimental grain growth behavior in 0.028% Nb microalloyed steel.

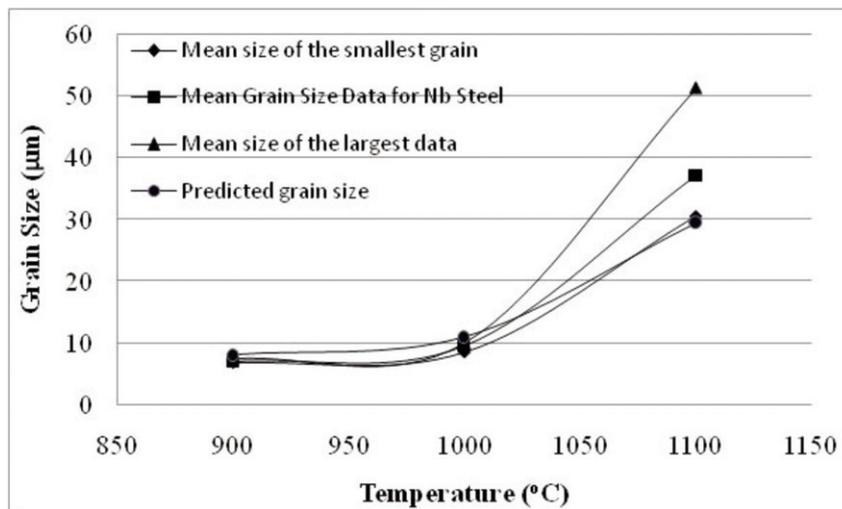


Fig. 9. Smallest and largest mean grain sizes and predicted grain growth for 0.028% Nb microalloyed steel at  $t = 600$  s.

## 5. Discussion

The evolution of the microstructure during the thermal history was observed by quenching the specimens at specified temperatures (Fig. 7). After quenching a specimen at 860°C, the microstructure showed only martensite, with fully undissolved precipitates at the austenite grain boundary. Fine and uniform distributed austenite grain size was observed due to the presence of precipitates as the second phase particles. Increasing the reheating temperature reduced the undissolved precipitates. When the specimen was quenched at 1060°C, the microstructure was entirely martensite, denoting a fully austenitic structure prior to quenching, because the reheating temperature exceeded the solubility temperature equation of Nb(C,N) [Eq. (8)].

Two distinct stages in grain growth behavior of 0.028% Nb steels were observed (Figs. 8 and 9). At lower temperatures (<1030°C), small and uniform grain size was observed which very closely approached the predicted grain sizes. Low temperatures indicated a marginal increase in mean grain size and an insignificant differentiation between the smallest and the largest grain sizes. According to Hillert's size distribution theory [9], a grain radius greater than 1.8 times the mean grain radius is unstable and predictive of abnormally growth. This means that abnormal grain growth due to the size different effect is either insignificant in niobium steels at lower temperature. Grain growth in this temperature region, therefore, can be

considered normal in the presence of second phase particles.

At temperatures above 1030°C, the largest grain size increased sharply to exceed the predicted grain size. At this stage, we observed mixed grain sizes, with most grain remaining small, and a small number of grains developing into unusually large sizes compared with their neighbors. A significant ratio difference between the largest and smallest observed grain size is indicated; the ratio of the mean size of the largest grain is 1.74 times the mean size of the smallest grain (Fig. 8). This behavior is typical of abnormal grain growth as described by Zener and Gladman [9] and is due to progressive particle dissolution and particle coarsening.

## 6. Conclusions

1. Grain growth of 0.028% Nb steels is influenced by heating temperature, where grain sizes increase with increasing reheated temperature.
2. A plot of the size of the largest and smallest grain versus temperature provides an effective and sensitive method for detecting the onset and termination of abnormal grain growth in 0.028% Nb steels.
3. Abnormal grain growth increases rapidly at the temperature above 1030°C.
4. Grain growth behavior and the effect of Nb microalloy precipitates can be successfully explained by developing the calculated model in this work.
5. Grain growth predictions for Nb steels fit well with the experimental grain growth data for these steels.

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