The Onset Temperatures of $\gamma$ to $\alpha$-Phase Transformation in Hot Deformed and Non-deformed Nb Micro-alloyed Steels

X. Q. YUAN,1) Z. Y. LIU,1) S. H. JIAO,2) L. Q. MA1) and G. D. WANG1)

1) The State Key Laboratory of Rolling and Automation, Northeastern University, P. O. Box, No. 105, Shenyang, 110004, P. R. China. E-mail: zylu@mail.neu.edu.cn 2) Institute of Research and Development, Baosteel Co., P. R. China.

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In the present paper, continuous cooling transformation behaviors in Nb micro-alloyed steels were systematically investigated by the thermal dilatation method, during which the effects of Nb contents and hot deformation of austenite on phase transformation behaviors, especially the transformation start temperatures, were studied in detail. The tests were carried out with the samples having been reheated+hot deformed or reheated+non-deformed prior to the dilation measurements. It was found that the $A_{\gamma}$ temperatures measured from the dilatometric curves changed with increasing Nb content in parabolic ways under both hot deformed and non-deformed conditions. It is believed that dissolved Nb in austenite may have had the Nb solute drag effect that could delay austenite to ferrite transformation. On the other hand, Nb precipitates in austenite could retard the growth of austenite grains and act as potential nucleation sites, both of which could enhance the transformation kinetics. Also, the interaction of strain, precipitation and temperature was estimated by using the Sellars model, which predicted that the strain induced precipitation had occurred in hot deformed Nb steels before phase transformation started, and in non-deformed steels with Nb content greater than 0.023 mass%, precipitation was also likely to have occurred under slow cooling rate before phase transformation started, which could have played an important role in determining $A_{\gamma}$. These factors worked together to make $A_{\gamma}$ changing with Nb content in the parabolic way. Based on the experimental results, a mathematical model for the $A_{\gamma}$ calculation for Nb and C–Mn steels were developed, which exhibited a good accuracy in predicting the $A_{\gamma}$ of the steels with and without hot deformation.

KEY WORDS: Nb microalloyed steels; $A_{\gamma}$ temperature; CCT curve; hot deformation; solute drag effect.

1. Introduction

Properties of hot rolled steels are dependent upon their final state of microstructure, which results from the evolution processes such as recrystallization during hot rolling and phase transformation during accelerated cooling after hot rolling. Therefore, the relationship of microstructure, properties and processing plays a vital role in controlling the properties of hot rolled products.1–6) Modeling of the microstructure-processing-properties relationship has formed the core of SPPC technology (structure and properties prediction and control), which includes the modeling of phase transformation behavior as one of the most important parts.7–10)

When the phase transformation behavior of steels during continuous cooling is studied, the CCT curve for the formation of detailed phase products needs to be worked out by measuring the starting temperatures of phase transformations. Although numerous dilatometric studies have been conducted and a great number of CCT curves have been drawn,11–23) systematic work on the quantitative relationship of phase transformation starting temperatures, composition and processing are still lacking, especially for the starting temperature for austenite to ferrite transformation ($A_{\gamma}$) in high strength low alloy steels (HSLA), which is an important factor in determining the phase transformation kinetics during cooling after hot rolling.

As compared with plain C–Mn steels, Nb bearing steels possess both phenomena of solute drag and precipitation effects to greatly affect $A_{\gamma}$.23–27) Ouchi and co-workers28) studied the effect of hot rolling condition and chemical composition on $A_{\gamma}$, the $A_{\gamma}$ temperatures for the steels containing 0 to 0.1 mass% Nb were in the range of 750°C to 780°C, showing an obvious parabolic relationship between the $A_{\gamma}$ and Nb content when Nb content was less than 0.1 mass%, which had been neglected by the authors, and a conclusion of no influence of Nb on $A_{\gamma}$ after hot rolling had been reached. Other authors have measured the changing rates of $A_{\gamma}$ per 0.01 mass% Nb for different ranges of Nb contents, and great discrepancy was obtained among these values, with a decreasing rate of 10°C per 0.01 mass% Nb for the steels containing 0.031 and 0.04 mass% Nb by Manohor et al.,18) a decreasing rate of 7.4°C per 0.01 mass% Nb for the steels containing 0.0 and 0.052 mass% Nb at a cooling rate of 2°C/s by Hong et al.,19) and a decreasing rate of nearly 30°C per 0.01 mass% Nb for the steels containing 0.006, 0.011 and 0.015 mass% Nb at cooling rates of 1 and 10°C/s by Abe et al.22) The influences of dissolved microalloying elements (solute drag effects), precipitation, and hot deformation were not fully clarified.
Therefore, more systematic work still needs to be done to further explore the quantitative effects of Nb on $A_{\gamma3}$ in Nb bearing steels.

The present work focuses on investigating the Nb effects on $A_{\gamma3}$ and developing modeling of $A_{\gamma3}$ for non-deformed and hot deformed Nb bearing C–Mn steels. Dilatometric testing was performed to build up the CCT curves for the steels containing different Nb contents during continuous cooling. $A_{\gamma3}$ temperatures were measured from the dilation curves and quantitative relationships of $A_{\gamma3}$, Nb content, austenite grain size, precipitation onset time, residual strain in austenite and cooling rate were established.

2. Experimental Procedures

The steels were melted by using a vacuum induction furnace and cast into ingots, the chemical compositions of the steels are given in Table 1. The ingots were forged into plates, from which specimens with a dimension of 8 mm in diameter and 12 mm in length were machined.

The dilatometric tests were carried out. A specimen was put in the chamber under high vacuum of 0.1 Torr, fixed between two anvils, and was reheated by induction. An R-type thermocouple was welded on the center of the specimen surface to measure the temperature. During cooling, helium gas was employed as the coolant when cooling rate exceeded 10°C/s, and nitrogen gas was employed when the cooling rate was slower than 10°C/s. Radial expansion of the specimen was measured by a scanning laser beam across the specimen center, where the thermocouple was welded.

Figure 1 shows the schematic drawing for the thermo-mechanical treatment. In order to get Nb completely dissolved in austenite, the Nb solution temperature was estimated by using Eq. (1).

$$\log_{10}[\text{Nb}] = 2.26 + \frac{838[Mn]^{0.246} - 1730[Si]^{0.594} - 6440}{T}$$

The Nb dissolution equilibrium temperatures for steels A2, A3 and A4 were calculated to be 922°C, 1037°C and 1060°C, respectively. Thus, the reheating temperature for these steels was chosen to be 1200°C and held for 5 min to be fully austenitized and get Nb sufficiently dissolved in austenite. After reheating, the steels were cooled down to 900°C at a cooling rate of 5°C/s, being held for 20 s and cooled down to 200°C at different cooling rates from 0.5 to 30°C/s. To study the effect of hot deformation on phase transformation behavior in the steels, a compression with a true strain of 0.693 was applied to the specimen at a strain rate of 5 s$^{-1}$ after being held for 10 s at 900°C, with the laser beam being able to follow the welding spot at the specimen center during the deformation.

Fig. 1. The schematic drawing for the thermo-mechanical treatment.

$A_{\gamma3}$ temperatures were determined on the dilatometric curves with the help of optical metallographic observations. In order to estimate the equilibrium temperature, $A'_{\gamma3}$, the dilatation tests during slow reheating and cooling at a rate of 0.2°C/s were carried out to determine the $A_{\gamma3}$ and $A_{\gamma3}$ of the steels, from which $A'_{\gamma3}$ temperatures were estimated as the average values, Eq. (2).

$$A'_{\gamma3} = \frac{1}{2} (A_{\gamma3} + A_{\gamma3})$$

Where, $A_{\gamma3}$ and $A_{\gamma3}$ are the start and end temperatures for $\gamma$ to $\alpha$ transformation measured at the heating and cooling rate of 0.2°C/s, respectively.

After the dilatation tests, quenching treatments were carried out to investigate the effect of the austenite grain size on transformation behavior. Without hot deformation, the samples were quenched at 900°C, just prior to the start of continuous cooling, and the grain size was applicable to all cooling rates. With hot deformation, the austenite grain size before transformation could be greatly different due to the recrystallization during the continuous cooling processes after hot deformation. The hot deformed samples, therefore, were quenched at the temperatures just above $A_{\gamma3}$ to determine the grain size prior to transformation, with the cooling rates of 0.5°C/s, 1°C/s and 5°C/s having been used before quenching.

After the thermo-mechanical treatment, all specimens were cut in the middle along the longitudinal direction across the thermocouple welding points and prepared for optical metallographic examination in the conventional way to observe the transformed microstructure. Image analysis system was used to measure the average size of grains by using the line-intercept method.

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3. Results

3.1. The $A'_{13}$ Temperature

Figure 2 shows a typical dilation curve, Fig. 2(a), and the relevant ferrite and pearlite microstructure of A1 steel, Fig. 2(b), after the slow heating and cooling at 0.2°C/s. By using Eq. (2), the $A'_{13}$ values of the steels were calculated, as shown in Table 2. It can be seen that when Nb content is less than 0.023 mass%, the $A'_{13}$ temperature decreases with the increase of Nb content, when Nb content increases from 0.023 to 0.038 mass%, the $A'_{13}$ temperature increases with the increase of Nb content.

3.2. The CCT Curves

Figure 3 shows the CCT curves of the steels with and without hot deformation. It can be seen that hot deformation can greatly increase the $A_{13}$ temperatures. For steel A1, the compression led to an increase of $A_{13}$ temperature by about 60°C, Fig. 3(a). For Nb bearing steels A2 and A3, the increase of $A_{13}$ temperatures caused by the compression was dependant on cooling rates, with the increase of about 110°C at slower cooling rates, and about 40°C at faster cooling rates, as shown in Figs. 3(b) and 3(c), respectively. For steel A4, as shown in Fig. 3(d), the degree of $A_{13}$ increase by hot deformation was almost the same as that of steel A1, which also decreased with increasing cooling rates.

Table 2. $A'_{13}$ temperatures of the steels after the heating and cooling treatments at a rate of 0.2°C/s.

<table>
<thead>
<tr>
<th></th>
<th>A1</th>
<th>A2</th>
<th>A3</th>
<th>A4</th>
</tr>
</thead>
<tbody>
<tr>
<td>$A_{13}$ °C</td>
<td>856.0</td>
<td>842.0</td>
<td>823.0</td>
<td>832.0</td>
</tr>
<tr>
<td>$A'_{13}$ °C</td>
<td>773.0</td>
<td>769.0</td>
<td>736.0</td>
<td>755.0</td>
</tr>
<tr>
<td>$A'_{13b}$ °C</td>
<td>814.5</td>
<td>805.5</td>
<td>779.5</td>
<td>793.5</td>
</tr>
</tbody>
</table>

Fig. 2. (a) Dilatometric curve of steel A1 during the slow heating and cooling rate; (b) The microstructure of A1 steel after the slow heating and cooling treatments.

Fig. 3. CCT curves of (a) Steel A1; (b) Steel A2; (c) Steel A3; (d) Steel A4 with and without hot deformation on austenite phase prior to transformation, the deformation condition: $T=900^\circ$C, $\varepsilon=0.693$, $\dot{\varepsilon}=5$ s$^{-1}$. 

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From the CCT curves, it can also be seen that hot deformation also increased the starting temperature of austenite to pearlite transformation, and its effect on the starting temperature of austenite to bainite transformation was not clear. The bainite start temperatures of steels A1 and A2 were increased by hot deformation, but those of steel A3 and A4 were decreased. More work needs to be carried out to understand this mechanism.

3.3. The Relationships between \( A_{s3} \) and Nb Content

Figures 4(a) and 4(b) show the relationships between \( A_{s3} \) and Nb contents for non-deformed and hot deformed steels, respectively. With increasing dissolved Nb in austenite, the \( A_{s3} \) temperature changes with Nb content in a parabolic way, decreasing from 0 to 0.023 mass% Nb and starting to increase thereafter, with the same tendency as that of \( A_{e3} \) temperature. In the range of 0 to 0.023 mass% Nb, the decreasing rates were measured to be 22°C and 35°C per 0.01 mass% Nb for the hot deformed steels at cooling rates of 1°C/s and 10°C/s, respectively, which were close to the results in Ref. 22, and showing a clear increasing trend with increasing the cooling rate. For the non-deformed steels, the decreasing rates were measured to be 39°C and 26°C at cooling rates of 1°C/s and 10°C/s, respectively, showing a clear decreasing trend with increasing the cooling rate. \( A_{s3} \) temperatures start to increase when the Nb content is higher than 0.023 mass%, with the increasing rates were measured to be 47°C and 34°C per 0.01 mass% Nb for non-deformed steels at cooling rates of 1°C/s and 10°C/s, respectively, and 12°C and 9.3°C per 0.01 mass% Nb for the hot deformed steels at cooling rates of 1°C/s and 10°C/s, respectively, showing a similar increasing trend with the results given in Refs. 18 and 19). With the continuous cooling rate increasing, the \( A_{s3} \) curves move down.

3.4. The Effect of Austenite Grain Size

Table 3 shows the average sizes of austenite grains for all quenched samples and their quenching temperatures. Figures 5(a) and 5(b) show the relationships between the austenite grain size and Nb contents for non-deformed steels and cooling rates for hot deformed steels, respectively. For non-deformed steels, with increasing Nb content, the effect of Nb content on grain refinement is obvious, Fig. 5(a). Figure 5(b) shows the linear relationships of grain size and cooling rates for the specimens after hot deformation, with the slope of steel A1 being greater than those of the Nb bearing steels. The slopes for steels A2 and A3 are

<table>
<thead>
<tr>
<th>Cooling rate (°C/s)</th>
<th>A1</th>
<th>A2</th>
<th>A3</th>
<th>A4</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5—30.0</td>
<td>248.0</td>
<td>344.0</td>
<td>213.0</td>
<td>69.0</td>
</tr>
<tr>
<td>0.5</td>
<td>25.3</td>
<td>23.4</td>
<td>17.8</td>
<td>24.3</td>
</tr>
<tr>
<td>1.0</td>
<td>27.6</td>
<td>24.5</td>
<td>18.8</td>
<td>27.2</td>
</tr>
<tr>
<td>5.0</td>
<td>46.5</td>
<td>36.1</td>
<td>28.4</td>
<td>34.1</td>
</tr>
<tr>
<td>QT (°C)</td>
<td>900</td>
<td>900</td>
<td>900</td>
<td>900</td>
</tr>
<tr>
<td>QT (°C)</td>
<td>810</td>
<td>777</td>
<td>756</td>
<td>787</td>
</tr>
<tr>
<td>QT (°C)</td>
<td>800</td>
<td>770</td>
<td>742</td>
<td>760</td>
</tr>
<tr>
<td>QT (°C)</td>
<td>775</td>
<td>700</td>
<td>642</td>
<td>685</td>
</tr>
</tbody>
</table>

Table 3. The austenite grain size (D) of the quenched samples and their corresponding quenching temperatures (QT).
greater than that for steel A4.

The relationship of A_r3 temperatures and grain sizes of a 0.14C–1.5Mn–0.7Si steel at a cooling rate of 2°C/s was observed to fit an exponential function (Eq. (3))^{19}:

\[ A_{r3} = A + B \exp \left(-\frac{D}{C}\right) \] .................(3)

where, \(D (\mu m)\) is austenite grain size, \(A, B\) and \(C\) are constants, and dependant on composition of the steels. By using the experimental results obtained for non-deformed and hot deformed steel A1 at a cooling rate of 1°C/s in this paper, Eq. (3) was modified for steel A1, with the constants of \(A, B,\) and \(C\) being 634, 370 and 6.7, respectively.

3.5. Nb Precipitates

Coarse Nb carbides formed at a prior austenite grain boundary accelerated ferrite nucleation,^{25} which was believed to increase A_r3 temperature. Dutta and Sellars developed a model of Nb precipitation for non-deformed and deformed steels under isothermal conditions.^{30} By using the method proposed by Jonas et al.,^{31} the precipitation start time during continuous cooling can be calculated. Figures 6(a) and 6(b) show the calculated results. For the non-deformed samples, Fig. 6(a), with the constant of B in the Dutta–Sellars model to be \(3.6 \times 10^8 - 3.6 \times 10^{10} K^{-3}\), Nb precipitation was likely to have occurred in austenite of steels A3 and A4 at the cooling rate of 1°C/s, but almost exerted no effect on A_r3 when the cooling rate was 10°C/s. In the hot deformed steels, however, the Nb precipitation had already taken place in austenite before ferrite transformation, Fig. 6(b), which could have strongly affected A_r3.

3.6. The Residual Strain

After hot deformation, residual strain may be stored in austenite if recrystallization is not complete, which can increase the deformation stored energy to enhance the ferrite transformation.^{32} Table 4 shows the calculated accumulative strain in the specimens before ferrite transformation by using the recrystallization models developed by Dyja and co-workers.^{33} Figure 7 shows the relationships of residual strain and Nb content, indicating that the residual strain in austenite before transformation increases with increasing Nb content and cooling rate.

4. Discussion

In the present work, the A_r3 temperature changed with Nb content in steel in a parabolic way, as shown in Figs. 4(a) and 4(b). For hot deformed steels, according to the results in Fig. 6(b), at the cooling rate of 1°C/s, time could be long enough for the formation of coarse Nb precipitates before the transformation, while at a faster cooling rate of 10°C/s, only fine Nb precipitates could be formed due to short time before the transformation, which were believed to hinder austenite to ferrite transformation,^{25,32} making the decreasing rate of A_r3 at slower cooling rate smaller than that at faster cooling rate. For the non-deformed steels, however, when the cooling rate is slow, Nb precipitation might have been formed according to the results in Fig. 6(a), which could have hindered the ferrite transformation.

<table>
<thead>
<tr>
<th>Cooling rate (°C/s)</th>
<th>Steel A1</th>
<th>Steel A2</th>
<th>Steel A3</th>
<th>Steel A4</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5</td>
<td>0</td>
<td>0</td>
<td>0.55</td>
<td>0.66</td>
</tr>
<tr>
<td>1</td>
<td>0</td>
<td>0</td>
<td>0.58</td>
<td>0.66</td>
</tr>
<tr>
<td>5</td>
<td>0</td>
<td>0.17</td>
<td>0.66</td>
<td>0.67</td>
</tr>
</tbody>
</table>

Table 4. The calculated residual strain in austenite after the hot deformation with a true strain of 0.693.
to result in a decreasing trend of $A_r$ decreasing rate with increasing the cooling rate.

By using Eq. (3), $A_r$ of the steels was calculated at the cooling rate of 1°C/s, which only took the grain size into consideration. The measured $A_r$ curve with the same cooling rate of 1°C/s is lower than the calculated, as shown in Fig. 8(a). The differences were estimated to be about 4°C, 7°C, 44°C, 38°C for steels A1, A2, A3 and A4, respectively. Since Nb precipitation could hardly occur before transformation in non-deformed steels, the difference between the measured and calculated $A_r$ is believed to be caused by mainly the Nb solute drag effect by the dissolved Nb in austenite.\(^{23}\) Suehiro\(^{24}\) calculated the dissipation of the driving force for transformation by the solute drag effect, which showed that when Nb content increased from 0.01 to 0.02 mass%, the driving force drastically dissipated, indicating the start of strong solute drag effect to result in the abrupt decrease of $A_r$. It can be noticed that the most abrupt difference between the calculated and measured $A_r$ happened when Nb content was in the range of between 0.01 mass% and 0.023 mass%, indicating that the solute drag effect by the dissolved Nb had started to work, in a good agreement with the calculation results by Suehiro et al.\(^{24}\)

The $A_r$ of the hot deformed steels was also calculated by using Eq. (3). Again, the measured $A_r$ curve is lower than the calculated one, and the differences were 3°C, 41°C, 86°C, 44°C for steels A1, A2, A3 and A4, respectively, generally greater than those for non-deformed steels. However, more dissolved Nb is believed to have been consumed by the strain induced Nb precipitation to result in less Nb solute drag effect than the non-deformed steels.

Therefore, there must be other effects to retard the phase transformation. It has been proved that fine Nb precipitates formed at the early stages were incapable of acting as the preferential ferrite nucleation sites because of their lower energy than that of grain boundaries. On the contrary, they might prevent the nucleation of ferrite.\(^{23,24}\) Nb precipitation was formed in all of the hot deformed steels before the phase transformation, Fig. 6(b), which may have been the main cause for the retardation of transformation. For the steels containing higher Nb contents such as steel A4, the strain induced precipitation occurred at higher temperatures, as shown in Fig. 6(b), to have longer time for the coarsening of Nb precipitates prior to the phase transformation to meet the requirement for the ferrite nucleation, resulting in higher transformation start temperature than lower Nb containing steels such as steel A3. With increasing Nb content, recrystallization was more retarded by the precipitation to have higher accumulative strain in austenite.\(^{25}\) Nb precipitation was formed in all of the hot deformed steels before the phase transformation, Fig. 6(b), which may have been the main cause for the retardation of transformation. For the steels containing higher Nb contents such as steel A4, the strain induced precipitation occurred at higher temperatures, as shown in Fig. 6(b), to have longer time for the coarsening of Nb precipitates prior to the phase transformation to meet the requirement for the ferrite nucleation, resulting in higher transformation start temperature than lower Nb containing steels such as steel A3. With increasing Nb content, recrystallization was more retarded by the precipitation to have higher accumulative strain in austenite, Fig. 7, which increased the deformation energy to accelerate the transformation, leading to a higher $A_r$.

By using the experimental results, mathematical models of $A_r$ for Nb bearing steels with or without hot deformation were developed. With no deformation, $A_r$ was considered to be a function of continuous cooling rate in °C/s, $C_R$, Nb content in mass%, [Nb], and austenite grain size in μm, $D_g$. The equation can be expressed as:

$$A_r = 370 \exp \left( - \frac{\sqrt{D_g}}{6.7} \right) - 325C_R^{0.1} - 5.649[Nb]$$

$$+ 78194[Nb]^2 + 1019 \quad \text{..........................}(4)$$

With the specimens subjected to hot deformation, precipitation start time, $t_{0.05}$ and residual strain in austenite, $\Delta \varepsilon$, also need to be taken into account. Therefore, for the hot deformed steels, the equation for $A_r$ is expressed as

$$A_r = 370 \exp \left( - \frac{\sqrt{D_g}}{6.7} \right) - 198C_R^{0.1} - 6.646[Nb]$$

$$- 2.327[Nb]^2 + 66 \times \left( \frac{1}{t_{0.05}} + \Delta \varepsilon \right) + 830 \quad \text{.....}(5)$$

Figure 9 shows the comparison of the measured and calculated values, which are in good agreements, indicating that the models have good accuracies to predict the $A_r$ va-
5. Conclusions

(1) The $A_{13}$ temperature of Nb bearing steels changes with the Nb content of 0–0.038 mass% in a parabolic way. When the Nb content was in the range of 0–0.023 mass%, the $A_{13}$ temperature decreased at the rates of about 22°C and 35°C per 0.01 mass% Nb for hot deformed steels at cooling rates of 1°C/s and 10°C/s, respectively, and 39 and 26°C for non-deformed steels at cooling rates of 1°C/s and 10°C/s, respectively. $A_{13}$ started to increase when the Nb content was higher than 0.023 mass%, with the increasing rates of 47°C and 34°C per 0.01 mass% Nb for the non-deformed steels at cooling rates of 1°C/s and 10°C/s, respectively, and 12°C and 9.3°C per 0.01 mass% Nb for hot deformed steels at cooling rates of 1°C/s and 10°C/s, respectively.

(2) For non-deformed steels, with increasing Nb content, fine Nb precipitates are likely to form in austenite before ferrite transformation only at slow cooling rate, which makes the $A_{13}$ decreasing rate faster than that at fast cooling rate per 0.01 mass% Nb. For hot deformed steels, with increasing Nb content, coarse Nb precipitates form in austenite before ferrite transformation at slow cooling rate, which makes the $A_{13}$ decreasing rate smaller than that at fast cooling rate per 0.01 mass% Nb.

(3) Based on the experimental results, models for calculating $A_{13}$ temperature of Nb bearing steels have been developed, for non-deformed Nb bearing steels,

$$A_{13} = 370 \exp \left( - \frac{-\sqrt{D_g}}{6.7} - 325C_g^{0.1} - 5.649[Nb] ight)$$

$$+ 78194[Nb]^2 + 1019$$

where $D_g$ is the austenite grain size, $C_g$ the continuous cooling rate, [Nb] Nb content in steels, and for hot deformed Nb bearing steels,

$$A_{13} = 370 \exp \left( - \frac{-\sqrt{D_g}}{6.7} - 198C_g^{0.1} - 6.646[Nb] ight)$$

$$- 2327[Nb]^2 + 66 \times \left( 1 \over t_{0.05} + \Delta \varepsilon \right) + 830$$

where $t_{0.05}$ the precipitation start time and $\Delta \varepsilon$ residual strain in austenite. The models showed good accuracies in predicting $A_{13}$ temperatures of Nb-bearing steels.

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