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The effect of cooling rate and coiling temperature on the niobium retention in Ultra-Thin Cast Strip steel

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Abstract
This laboratory study utilised a dilatometer to simulate the run-out table cooling rate and the coiling temperature to investigate the effect of the cooling rate and simulated coiling conditions on the age hardening response of a niobium microalloyed Ultra-thin Cast Strip (UCS®) steel, produced by the CASTRIP® Process. Three cooling rates of 1, 5 and 40 °C/s, covering very slow (1 °C/s) to typical run-out table cooling rates (40 °C/s), down to two coiling temperatures of 500 and 675 °C were used. Dilatation curves were used to determine the temperature range over which the \( \gamma \rightarrow \alpha \) phase transformations occurred and the final microstructures were characterized using an optical microscope equipped with an image analysis software. The subsequent age hardening response, which previous studies have shown, results from the retention of Nb in solid solution, was assessed by the hardness changes after a post heat treatment at 700 °C for 60 s. A range of age hardening responses were obtained, depending on cooling rates and cooling stop (coiling) temperatures, which indicate a different degree of Nb retention. At the same cooling rate, the lower coiling temperature of 500 °C resulted in higher Nb retention compared to the higher coiling temperature of 675 °C. As the coiling temperature of 675 °C was within the austenite to ferrite transformation range, the simulated slow cooling of the coil impacted the precipitation behaviour of Nb rendering the interpretation more complex and this will be discussed in this paper. For the 500 °C simulated coiling temperature, the higher cooling rate resulted in a higher age hardening increment thus more Nb retention.

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This laboratory study utilised a dilatometer to simulate the run-out table cooling rate and the coiling temperature to investigate the effect of the cooling rate and simulated coiling conditions on the age hardening response of a niobium microalloyed Ultra-thin Cast strip (UCS\textsuperscript{®}) steel, produced by the CASTRIP\textsuperscript{®} Process. Three cooling rates of 1, 5 and 40\textdegree C/s, covering very slow (1\degree C/s) to typical run-out table cooling rates (40\degree C/s), down to two coiling temperatures of 500 and 675\degree C were used. Dilatation curves were used to determine the temperature range over which the $\gamma$-$\alpha$ phase transformations occurred and the final microstructures were characterized using an optical microscope equipped with an image analysis software. The subsequent age hardening response, which previous studies have shown, results from the retention of Nb in solid solution, was assessed by the hardness changes after a post heat treatment at 700\degree C for 60s. A range of age hardening responses were obtained, depending on cooling rates and cooling stop (coiling) temperatures, which indicate a different degree of Nb retention. At the same cooling rate, the lower coiling temperature of 500\degree C resulted in higher Nb retention compared to the higher coiling temperature of 675\degree C. As the coiling temperature of 675\degree C was within the austenite to ferrite transformation range, the simulated slow cooling of the coil impacted the precipitation behaviour of Nb rendering the interpretation more complex and this will be discussed in this paper. For the 500\degree C simulated cooling temperature, the higher cooling rate resulted in a higher age hardening increment thus more Nb retention.

\textbf{Keywords:}

Dilatometry
CASTRIP steel
thermomechanical processing
age hardening
phase transformation
precipitation

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1. Introduction

CASTRIP® process is a revolutionary strip casting technique for producing Ultra-Thin Cast Strip steel, which has many advantages over conventional casting and rolling technologies, including a smaller carbon footprint, lower cost of production and simpler and more flexible operating conditions. The world’s first commercial installation of the CASTRIP process for the production of Ultra-Thin Cast Strip (UCS) is located in Nucor Steel’s Crawfordsville, Indiana plant. The facility has been producing low-carbon sheet steel since its start-up in 2002 [1]. The CASTRIP process is mainly composed of three parts: twin-roll casting, hot rolling reduction, controlled cooling process. The strip is produced from liquid steel with a rapid solidification rate by twin-roll casting, followed by hot rolling reduction to final thicknesses of 0.9-1.5 mm. Finally, on the run-out-table, the steel is controlled cooled by air-mist water cooling. The control of the cooling rate and the stop cooling temperature on the run-out-table plays an important role in achieving the desired microstructure and mechanical properties of the UCS products.

Table 1: Chemical composition (wt.%) of the UCS steel.

<table>
<thead>
<tr>
<th>Element</th>
<th>C</th>
<th>P</th>
<th>Mn</th>
<th>Si</th>
<th>S</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Cu</th>
<th>Nb</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.031</td>
<td>0.014</td>
<td>0.738</td>
<td>0.159</td>
<td>0.003</td>
<td>0.047</td>
<td>0.057</td>
<td>0.017</td>
<td>0.095</td>
<td>0.059</td>
<td>0.005</td>
</tr>
</tbody>
</table>
The heat treated samples were divided into two parts by using an Accutom 5/50 sectioning machine. One part was used for the microstructure characterisation and hardness measurements, while the other part was utilised for the post-processing age hardening treatment, which was carried out in the dilatometer. These samples were aged at 700°C for 60s and cooled to room temperature by helium gas quenching, as shown schematically in Figure 3, then the hardness was measured.

Optical metallography was carried out on a LEICA DMR optical microscope. The samples were hot mounted on a Struers CitoPress-20 mounting machine, then ground and polished to a 1µm finish using a Struers grinding and polishing machine and etched in 2% Nital.

Vickers hardness and micro Vickers hardness tests were performed on an INDENTEC Vickers hardness testing machine with a load of 1 kg and LECO M-400-H1 micro Vickers hardness testing machine with a load of 50g, respectively. The Vickers hardness of the samples or micro Vickers hardness of each microstructure constituent was measured as an average of eight tests.

The dilatation curve shown in Figure 4 was obtained by combining the dilatometer data and the microstructure observations of the steel. Microsoft Excel was used to assist in drawing the dilatation curve.

Due to the difficulty in revealing the prior austenite grain boundaries of low carbon UCS steel by using picric acid, the austenite grain boundaries were delineated using ferrite, as shown in Figure 5. The austenite grain size was then measured using the linear intercept method.

The volume fraction of each constituent of the samples was estimated by using the imaging analysis software Axiovision and Microsoft Excel. There are a number of nomenclature systems for describing the range of ‘lower transformation ferritic products’ that form in steel [8-12]. In this paper, the ISIJ transformation products nomenclature [8] was adopted because it is well suited for low carbon steels [4, 8]. The typical order of ferritic transformation products are: Polygonal Ferrite (PF), Quasi-Polygonal Ferrite (QPF), Widmanstätten Ferrite (αw), Granular Bainite (GB), Bainitic Ferrite (BF) and Martensite.

3. Results

3.1 Microstructure

The prior austenite grain size was 179 µm, which is relatively coarse compared with the fine austenite grain size produced during conventional hot rolled strip rolling, and was similar to the austenite grain size produced by
Table 2: Microstructure summary of the samples cooled at the cooling rates of 1, 5 and 40°C/s from 900°C to the coiling temperatures of 500 and 675°C. (PF: polygonal ferrite; P: pearlite; GB: granular bainite and BF: bainitic ferrite)

<table>
<thead>
<tr>
<th>Coiling T(°C)</th>
<th>500</th>
<th>675</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cooling rate(°C/s)</td>
<td>1</td>
<td>5</td>
</tr>
<tr>
<td>Microstructure</td>
<td>PF + 5%P</td>
<td>PF+29%GB</td>
</tr>
</tbody>
</table>

Figure 6. Optical microstructures of the samples cooled at the cooling rate of 1°C/s with the coiling temperatures of (a) 675°C (b) 500°C. (PF: polygonal ferrite; P: pearlite)

Figure 7. Optical microstructures of the samples cooled at the cooling rate of 5°C/s with the coiling temperatures of (a) 675°C (b) 500°C. (GB: granular bainite; PF: polygonal ferrite)

The samples cooled at 1°C/s, regardless of the coiling temperature, had a similar microstructure, both consisting of polygonal ferrite and a small amount of pearlite, see Figure 6. The volume fractions of the polygonal ferrite were 95% and 98% for the coiling temperatures of 500 and 675°C, respectively, as shown in Table 2.

The microstructure of the samples cooled at 5°C/s varied with the coiling temperature, as shown in Figure 7. At the coiling temperature of 675°C, the microstructure consisted of about 95% polygonal ferrite and 5% pearlite/granular bainite, which is similar to the microstructure of the samples cooled at 1°C/s. At the coiling temperature of 500°C, the microstructure consisted of 71% polygonal ferrite and 29% granular bainite.

Figure 8 shows the microstructures of the samples cooled at 40°C/s, where both consisted of considerable amounts of granular bainite and bainitic ferrite. For the coiling temperature of 500°C, the sample consisted of 61% granular bainite and 31% bainitic ferrite and for the coiling temperature of 675°C the sample consists of 55% granular bainite and 45% of bainitic ferrite. The bainitic ferrite is the plate-like ferrite formed first at the austenite grain boundaries and the granular bainite formed in the remainder of the matrix. The size of the bainitic ferrite is much smaller than that of the polygonal ferrite produced in the sample cooled at 5°C/s.

3.2 Dilatation curves

Figure 9 displays the dilatation curves obtained from the samples in the various cooling conditions, in which
the slow cooling rate of 0.17°C/s, as shown in Figure 9e and 9f. Polygonal ferrite did not form during the simulated coil cooling stage, perhaps due to carbon segregation into the remaining austenite, which increased the hardenability, so bainitic ferrite formed. A marked difference between the sample cooled at 1°C/s and the sample cooled at 5°C/s is that the transformation of the austenite to granular bainite occurred in the sample cooled at 5°C/s (Figures 9c and 9d) but not in the sample cooled at 1 °C/s (Figures 9a and 9b). It should be noted that the inflection points in the cooling curves indicate the start and the finish of the phase transformation from γ to α in terms of volume as shown in Fig. 9. At the 675°C cooling temperature the γ to α transformation mainly occurred in the temperature ranges of 789-714°C, 757-668°C and 681-657°C for the cooling rates 1, 5 and 40°C/s, respectively. At the 500°C cooling temperature, the γ to α transformation mainly occurred in the temperature ranges of 799-724°C, 754-638°C and 662-577°C for the cooling rates 1, 5 and 40°C/s, respectively. The main γ to α transformation range occurred between peak and trough were labelled as shown in Figure 9.

3.3 Age hardening response

Table 3 tabulates the Vickers hardness and estimated yield strength of the samples for each of the various cooling conditions, before and after the age hardening heat treatment. The yield strength was converted from the Vickers hardness by conversion equation [13]. The results showed that a hardness increase from the age hardening heat treatments was recorded for all cooling conditions. For the 500°C cooling temperature, the age hardening increments were similar for the 1°C/s and 5°C/s cooling rates, while slightly higher for the highest cooling rate of 40°C/s.

The hardness before the age hardening heat treatment was similar for both the cooling temperatures for the cooling rates of 5 and 40°C/s, while for the 1°C/s cooling rate, the hardness before age hardening was higher for the 675°C cooling temperature, perhaps indicating that some precipitation hardening had occurred.

The micro Vickers hardness and associated estimated yield strength of the various microstructural constituents are shown in Table 4. The micro hardness results showed similar hardness increments from the age hardening heat treatment to that recorded from the macro Vickers hardness tests. Moreover, the micro Vickers hardness values for the polygonal ferrite phase were similar to the macro Vickers hardness before the age hardening for the 1°C/s cooling rates for the both cooling temperatures. However, for the other conditions, the micro hardness results were higher than the macro Vickers hardness results, particularly for the samples cooled at 40°C/s.

This was most likely due to the micro hardness tests sampling a higher percentage of harder phases, which would be more pronounced for granular bainite, due to the laminar like structure.

Figure 8. Optical microstructures of the samples cooled at the cooling rate of 40°C/s with the coiling temperatures of (a) 675°C (b) 500°C (BF: Bainitic ferrite).
Figure 9. Dilatation curves obtained at the various combinations of cooling rates and coiling temperatures: (a) 1°C/s, 500°C (b) 1°C/s, 675°C (c) 5°C/s, 500°C (d) 5°C/s, 675°C (e) 40°C/s, 500°C (f) 40°C/s, 675°C (PF: polygonal ferrite; BF: bainitic ferrite; GB: granular bainite).

Table 3: Vickers hardness (HV1) and yield strength (MPa) of the samples before and after age hardening.

<table>
<thead>
<tr>
<th>Coiling temp (°C)</th>
<th>Cooling rate (°C/s)</th>
<th>Before age hardening</th>
<th>After age hardening</th>
<th>Age hardening</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>HV</td>
<td>YS</td>
<td>HV</td>
<td>YS</td>
</tr>
<tr>
<td>500</td>
<td>1</td>
<td>178</td>
<td>205</td>
<td>499</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>198</td>
<td>224</td>
<td>554</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>201</td>
<td>233</td>
<td>579</td>
</tr>
<tr>
<td>675</td>
<td>1</td>
<td>191</td>
<td>206</td>
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<tr>
<td></td>
<td>40</td>
<td>204</td>
<td>212</td>
<td>519</td>
</tr>
</tbody>
</table>
Table 4: Micro Vickers hardness (HV 0.05) and estimated yield strength (MPa) of the polygonal ferrite and granular bainite before and after age hardening.

<table>
<thead>
<tr>
<th>Coiling temp (°C)</th>
<th>Cooling rate (°C/s)</th>
<th>Before age hardening</th>
<th>After age hardening</th>
<th>Age hardening</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>PF</td>
<td>GB</td>
<td>PF</td>
<td>GB</td>
</tr>
<tr>
<td></td>
<td>HV</td>
<td>YS</td>
<td>HV</td>
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</tr>
<tr>
<td>500</td>
<td>1</td>
<td>177</td>
<td>418</td>
<td>-</td>
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<tr>
<td></td>
<td>5</td>
<td>185</td>
<td>441</td>
<td>207</td>
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<td></td>
<td>40</td>
<td>-</td>
<td>230</td>
<td>571</td>
</tr>
<tr>
<td>675</td>
<td>1</td>
<td>192</td>
<td>461</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>203</td>
<td>493</td>
<td>221</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>-</td>
<td>227</td>
<td>562</td>
</tr>
</tbody>
</table>

Table 5: The γ→α phase transformation temperature range and dwell time in the γ→α phase transformation temperature range of the samples in various cooling conditions.

<table>
<thead>
<tr>
<th>Coiling Temp. (°C)</th>
<th>Cooling rate (°C/s)</th>
<th>λ→α temperature range (°C)</th>
<th>Dwell time in λ→α range (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Entire</td>
<td>Major</td>
<td>Entire</td>
</tr>
<tr>
<td>500</td>
<td>1</td>
<td>810-630</td>
<td>799-724</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>786-615</td>
<td>754-638</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>735-540</td>
<td>662-577</td>
</tr>
<tr>
<td>675</td>
<td>1</td>
<td>810-667</td>
<td>789-714</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>786-660</td>
<td>757-668</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>726-640</td>
<td>681-657</td>
</tr>
</tbody>
</table>

Table 5 summarises the γ→α phase transformation temperature ranges and the dwell time in the γ→α phase transformation temperature range of the samples in the various cooling conditions. It is clear that for the cooling temperature of 500°C, the dwell time of the sample at the cooling rate of 40°C/s in the major range is just 2 seconds which is as expected much shorter than that of the samples cooled at 1 and 5°C/s. For the cooling temperature of 675°C the dwell time of the sample in the major region at 1°C/s is 75 seconds which is a little shorter than 89 seconds for the sample cooled at 5°C/s but shorter than 106 seconds for the sample cooled at 40°C/s as shown in Table 5.

4. Discussions

4.1 Effect of cooling rate and coiling temperature on transformation products

As the cooling rate increased, the change in ferritic transformation products followed the typical sequence as outlined by ISIJ Bainitic committee [8]. That is, with increasing cooling rate; Polygonal ferrite becomes more irregular in shape, i.e. Quasi-Polygonal ferrite, then bainitic transformations take place, Granular Bainite (G.B.) followed by Bainitic Ferrite (B.F.). Normally, GB forms before BF [8], but this was not the case for the samples cooled at 40°C/s (See Fig 8). Once BF had exhausted the preferred nucleation sites of the austenite grain boundaries, the intragranular nucleation of GB became more favourable. Nucleation of intragranular GB was possible in this study, due to the large austenite grain size and continuous cooling conditions. The austenite grain size was large enough to prevent BF from consuming the entire matrix before the temperature dropped low enough for intragranular nucleation to become favourable [4,9]. Granular Bainite has a lath-less, coarse structure, which does not have the classical needle-like structure that is associated with acicular ferrite. In conventional processing, intragranular nucleation is not observed as the austenite grain size is quite small prior to transformation.

4.2 The effect of the coiling temperature on the Nb retention in the UCS steel

Before age hardening, the greatest difference in hardness between the 675°C and 500°C cooling temperature results was at 1°C/s, where the hardness of the sample at 675°C was 191Hv, while at 500°C it was 178Hv, as shown in Table 3. For the cooling rates of 5 or 40°C/s, the hardness of the samples was slightly higher at the cooling temperature 675°C. However, according to the microstructural analysis (Table 2), more granular bainite was produced at the cooling rate of 5°C/s for the lower cooling temperature. Based on this, the hardness of the sample at 500°C should be higher than that of the sample at 675°C, which is obviously contradictory to the results. The reason for the contradiction is that the strength of the steel is not only determined by grain refinement, but also determined by other strengthening mechanisms, as shown in equation 1 [14].

\[ \sigma_p = \sigma_y + \sigma_s + \sigma_g + \sigma_p + \sigma_d \]  

(1)

where \( \sigma_y \) is the yield strength, \( \sigma_s \) is the ferrite lattice strength, \( \sigma_p \), \( \sigma_g \) and \( \sigma_d \) are the strengthening contributions caused by solid solution, grain refinement, precipitation hardening and dislocation hardening respectively. Of the above mechanisms, grain refinement hardening and precipitation hardening are the two most
likely candidates to cause an increase in hardness for the samples in the present study.

From Table 3, at the same cooling rate, the age hardening increment with the low cooling temperature of 500°C is much higher than that with 675°C, which means more Nb atoms were retained in the solid solution before the age hardening heat treatment during the simulated strip cooling process. Previous studies have shown that microalloy carbides are mainly produced during γ-α phase transformation in cooling because there is a large solubility difference of alloy carbides between the austenite and ferrite [5, 15]. The solubility of Nb carbides in austenite is much higher than that in ferrite and thus the Nb carbides will typically precipitate out during γ-α phase transformation in a cooling process [16-21]. As can be seen from Figure 9, that 500°C is outside of the γ-α phase transformation temperature range but 675°C is within the range in all the dilatation curves. The cooling rate was just 0.17°C/s below the cooling temperature, so there was sufficient time for the sample with a 675°C cooling temperature to dwell in the γ-α phase transformation temperature range. This was not the case when cooled at 500°C, so the time spent during the γ-α phase transformation was much shorter Therefore, at the same cooling rate, fewer precipitates, if any, would be expected to be produced in the sample at the lower cooling temperature of 500°C compared to the sample at 675°C. Furthermore, the drive for Nb precipitation at 500°C is very low, so even with the slow coil cooling, Nb would remain in solid solution.

Consequently, for samples cooled at 500°C, only microstructural hardening and solid solution hardening occurred, but for samples cooled at 675°C, precipitation hardening also occurred. Therefore, the pre-aged hardness was higher for samples cooled at 675°C for all cooling rates, especially at 1°C/s, where both microstructures were polygonal ferrite. The diminished age hardening response for the 675°C samples for all cooling rates agrees that a significant amount of Nb was lost during the cooling process.

4.3 The effect of cooling rate on the Nb retention in the UCS steel

The cooling temperature of 500°C is outside of the γ-α phase transformation temperature range in all the samples, so the dwell time of the samples in the phase transformation range depends on the cooling rate. The higher the cooling rate the shorter the dwelling time, thus NbC precipitation is less likely. It is expected that more Nb will be retained in the samples cooled at the high cooling rate of 40°C/s than the samples cooled at the lower cooling rates of 1 and 5°C/s.

As expected for the same cooling temperature of 500°C, the age hardening of the samples cooled at 40°C/s is the highest at 32Hv, as shown in Table 3. At the cooling rate of 40°C/s, it is clear from Table 5 that the dwelling time of the samples in the major γ-α phase transformation temperature range is the shortest, giving the least time for NbC to precipitate.

The 675°C cooling temperature is within the γ-α phase transformation temperature range, which renders the interpretation of the age hardening results more complex. For example, the age hardening increment was higher at the lower cooling rate compared with the faster cooling rates, indicating that more Nb was retained in solution at the slower cooling rate through the transformation, rather than at the higher cooling rates, which was not expected.

At the cooling rate of 1°C/s, most of the austenite had transformed into polygonal ferrite above the cooling temperature of 675°C, as shown in Figure 9a. While the very slow cooling below the cooling temperature of 675°C would add to the transformation time, there was very little phase transformation left to occur. Therefore, the γ-α phase transformation and NbC precipitation mainly occurred above the cooling temperature of 675°C for the sample cooled at 1°C/s. In contrast, the samples cooled at the higher cooling rate, such as 40°C/s, only some of the austenite had transformed into bainitic ferrite above the cooling temperature of 675°C (Figure 9e). A larger volume fraction of the austenite transformed into the granular bainite at the temperature range below 675°C at the cooling rate of only 0.17°C/s. Such a slow cooling rate will enable more Nb to precipitate out as NbC and less Nb was retained in solid solution for the age hardening. As can be seen from Table 5, the dwell time of the sample in the major γ-α phase transformation range at 40°C/s is much longer than that of the sample at 1°C/s. The nucleation sites for the NbC precipitation during the γ-α phase transformations for the samples at 40°C/s are mainly the interfaces between the γ and α. The number of the nucleation sites is related to the volume transformation of the γ→α and therefore it is likely that more NbC precipitated in the sample cooled at 40°C/s than in the sample cooled at 1°C/s to the cooling temperature of 675°C before the aging hardening treatment. As for the sample cooled at the cooling rate of 5°C/s, the dwell time of the sample in the major γ-α phase transformation was also longer than that of the sample at 1°C/s, as shown in Table 5. This implies that less Nb was retained in the solid solution of the sample cooled at 5°C/s than the sample cooled at 1°C/s. This is consistent with the results that larger age hardening was obtained in the simulated slow coil cooling, NbC precipitated in 1°C/s than the sample cooled at 5°C/s as shown in Table 3.

5. Conclusions

1. The hardness of the UCS steel is not only determined by the microstructures, but also determined by precipitates produced during CAST RIPS process. The findings suggest that even if more granular bainite were produced in the sample with the lower cooling temperature of 500°C, the hardness of the samples cooled to 675°C was higher due to precipitation hardening.

2. The cooling rate effect on the Nb retention was very clear for the samples which have the cooling temperature of 500°C. The faster cooling rates results in lower γ-α transformation temperatures and the specimens spent less time in the γ-α transformation temperature range, so more Nb was retained in solid solution and a large age hardening increment was produced.

3. The cooling rate effect on the Nb retention was complicated for samples cooled at 675°C, because the cooling temperature was within the γ-α transformation range. Due to the simulated slow coil cooling, there was
ample time for Nb to precipitate, thus diminishing the age hardening response for all cooling rates.

4. High coiling temperatures should be avoided for the CASTRIP Process for Nb-bearing steels to prevent unwanted Nb precipitation during coiling and therefore, diminishing the age hardening response of the product.

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References


