Effect of Plate Rolling Strategy and Nb Microalloying in the HAZ Performance After Welding

The definition of hot rolling parameters to achieve the most suitable combination of final microstructure and precipitation is a key factor to control mechanical properties of welded plates. In this work, several Ti-Nb microalloyed steels were rolled following standard and optimized rolling strategies, resulting in different microstructures and precipitation states in the hot-rolled plates. Both simulated and real welding treatments were applied to the plates and the mechanical properties measured. The correlation between hot rolling parameters, precipitate population, microstructural features and mechanical performance was evaluated. High mechanical performance of welded plates was achieved for the optimized rolling conditions.

Low-carbon microalloyed steels are generally used for modern high-strength line pipe. The combination of Nb microalloying and the use of thermomechanical controlled processing (TMCP) are the essential measures for improving strength and toughness of the base microstructure by grain refinement and transformation strengthening. Obtaining a large reduction in the roughing pass during hot rolling is a key step to achieve a homogenous and fine recrystallized austenite, accompanied by controlled rolling below the non-recrystallization temperature to further refine the final transformed microstructure. Intensified recrystallization rolling has been applied for high-strength TMCP line pipe steels with excellent low-temperature toughness. Recently, a mathematical model to predict the austenite microstructure evolution of Nb microalloyed steels during hot rolling has been developed and used for optimizing through-thickness microstructure homogeneity, which depends strongly on composition and rolling conditions. By applying Nb microalloying and optimized rolling strategy, performance of line pipe steels can be maximized. However, line pipe steels also need to exhibit excellent toughness in the weldment as well as the base material. Since a large heat input is imposed by submerged-arc welding (SAW) in pipe production, the heat-affected zone (HAZ) toughness tends to deteriorate because of grain growth and formation of hard second phases, such as martensite-austenite (MA) constituents. Although formation of MA constituents in the simulated HAZ is enhanced with Nb addition, it was reported that Nb additions

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of up to 0.1% do not affect toughness of the coarse grain HAZ (CGHAZ) in the SAW joint\textsuperscript{10} or gave better simulated HAZ toughness.\textsuperscript{11} However, the HAZ in an actual SAW joint exhibits a variety of microstructures as shown in Fig. 1, and the intercritically reheated coarse grain HAZ (ICCGHAZ) typically shows lower toughness because of increased MA formation.\textsuperscript{12} In addition to MA formation, austenite grain growth is significant in CGHAZ since the heating temperature becomes far above the dissolution temperature of Ti and Nb carbonitrides which can pin austenite grain boundaries. There may be a possibility of grain growth even for the fine-grained line pipe steel produced by Nb addition and optimized rolling strategies. Therefore, the effect of plate rolling strategy and Nb addition on the HAZ performance should be of a great interest to the development of line pipe with excellent base metal and HAZ toughness.

In this work, Ti-Nb microalloyed TMCP steel plates with different Nb contents were produced using both standard and optimized rolling strategies, resulting in different microstructures in the hot-rolled plates. Both simulated HAZ heat treatment and submerged-arc welding were applied to the plates and the mechanical properties, especially Charpy toughness, were evaluated. The correlation between hot rolling parameters, microstructural features and properties was evaluated. Excellent mechanical performance of the welded plates was achieved with a combination of Nb microalloying and optimized rolling strategy.

**Effect of Plate Rolling Strategy on Base Metal Toughness**

**Material Preparation** — The chemical compositions of the steels used in this study are shown in Table 1. Low-carbon and Nb-Ti added chemistries were selected regarding modern practice for API Grade X60 to X65 line pipe for sour service. To investigate the effect of Nb content on base material and HAZ properties, the Nb addition varied from 0.030% to 0.055%. Other alloying elements were added to keep the Pcm values in the same level of 0.13–0.14%. P and S content were limited up to 60 ppm and 8 ppm, respectively, and 20–30 ppm of Ca was added for inclusion shape control. Two of the compositions (A and B) were produced as slabs by continuous casting, while the slab for Steel C was prepared by laboratory ingot casting. As-cast samples of the three compositions of 160-mm thickness were laboratory rolled. Heating temperature was varied depending on the NbC solubility temperature and set at 1,040, 1,080 and 1,110°C for Steels A, B and C, respectively. After heating for two hours, hot rolling was performed using two...
different rolling strategies as shown in Fig. 2. In rolling condition 1, relatively low reductions were applied in the roughing passes and gradually increased toward the later passes. On the other hand, higher reduction was applied in the roughing passes in condition 2, then reduction was reduced in the finishing rolling. Reduction in the finishing passes was set as the same level of between 15 and 20% to investigate the effect of the recrystallization rolling. This higher rolling reduction in the roughing rolling aimed to refine the recrystallized austenite and obtain homogeneous fine microstructure. These rolling strategies were analyzed by the recent developed model, MicroSim PM®, to predict microstructure evolution during the plate rolling. Fig. 3 shows recrystallization ratios during the roughing and finishing passes for the steel B. In condition 1, recrystallization was insufficient in the roughing passes because of lower reduction per pass. On the other hand, full recrystallization is expected to be achieved in the roughing passes in condition 2 by applying larger reduction per pass. Reduction below the non-recrystallization temperature was 72% for both conditions. The rolling finishing temperature was set at around 850°C and then accelerated cooling was applied from the temperature above the Ar3 temperature to prevent ferrite formation. Final plate thickness was 23 mm in all cases. The effects of the rolling reductions and Nb microalloying on the microstructure evolution during hot rolling can be indirectly evaluated by using mean flow stress (MFS) analysis. MFS analysis can assist in determining if the proper austenite softening behavior during hot rolling is occurring, as well as contrasting the robustness of the recrystallization suppression by strain-induced Nb precipitation. In the MFS curve, the total stress (MPa) increase from the last roughing pass to the first finishing pass will provide a reference on the strain accumulation due to Nb precipitation starting the finishing rolling process. From the first finishing pass to the last pass, the total stress increase will provide a reference on the softening behavior of the austenite and further strain accumulation due to Nb precipitation, resulting in a continued increase in MFS to the last pass, as shown in Fig. 4.

Rolled plate samples were then subjected to mechanical and microstructure characterization. Round bar uniaxial tension and V-notch Charpy specimens were taken from the mid-thickness region of the plates in the transverse direction. The microstructure of the plate was observed by scanning electron microscopy (SEM) in the longitudinal section.

**Microstructure of the Plates** — Fig. 5 shows microstructure of the steel plates in the mid-thickness region. All the microstructures were basically fine bainitic, consisting of mixtures of quasi-polygonal ferrite and acicular ferrite, with small amounts of martensite-austenite constituent (MA). It was seen that rolling condition 2 provided finer microstructures for all steels, as expected given the intensified recrystallization rolling and the estimated resulting recrystallization behavior shown in Figs. 2 and 3. Austenite grain size is reduced by about half by rolling condition 2, which applies intensified reduction per pass in the roughing rolling. It was also observed that the homogeneity of the grain size distribution was improved by applying rolling condition 2, which implies a reduced risk of isolated large grains in the final microstructure.

**Characterization of Precipitates** — An analysis of precipitates was carried out in the as-rolled plates by transmission electron microscopy (TEM) of carbon extraction replicas. In Fig. 6, TEM micrographs...
corresponding to steel B and rolling condition 1 are shown. Different precipitate populations are clearly observed. The coarsest particles (larger than 30–40 nm) are attributed to the lack of complete dissolution of Nb precipitates during the reheating prior to rolling. Additionally, finer strain-induced precipitates (between 10 and 20 nm) are also formed due to application of deformation below the non-recrystallization temperature. Microanalysis performed on these precipitates indicate that they are Nb-rich precipitates with some residual Ti presence.

Fig. 7a, 7b and 7c show the TEM images obtained after rolling using condition 1 for the different steel grades (A, B and C, respectively). In addition to non-dissolved particles and strain-induced precipitates, finer precipitates are detected (finer than 10 nm) that are expected to have been formed during final cooling (during or after phase transformation). As clearly shown in Fig. 7c, the presence of fine precipitates is more significant in Steel C, which has the highest Nb content in this study (0.055%).

Besides the characterization of precipitates, quantification of the precipitate size was also performed. Fig. 7d confirms that for rolling condition 1, the precipitate diameter distribution varies depending on the base composition. The overall finest precipitate diameter distribution was observed in Steel A, with Steel B coarser and Steel C showing the overall coarsest on average. Mean precipitate diameters of 11.6, 15.8 and 18.0 nm are estimated for A, B and C steels, respectively. For the steel containing the highest Nb level (Steel C), the precipitate size distribution shows a distinctly bimodal shape, confirming the formation of differently sized Nb precipitates (finer and coarser than 10 nm) at different stages of the plate thermomechanical history. The size and volume fraction of these precipitates, and their resulting coarsening and dissolution behavior during the heating of the welding process, will affect the grain growth behavior in the HAZ.

**Figure 6**
Transmission electron microscopy (TEM) micrographs corresponding to Steel B and condition 1 (different magnifications).

**Figure 7**
TEM micrographs corresponding to A, B and C steels, respectively (condition 1) (a,b,c); and comparison between precipitate diameter distributions measured for each steel grade (d).

**Mechanical Properties of the Plate** — The results of the tensile and Charpy tests are summarized in Table 2. All the steels have strength level comparable to API Grade X60 to X65, with excellent ductility. This excellent ductility is reflected in extremely high Charpy absorbed energy, which was over 300 J because of the
homogeneous fine bainitic microstructure all three steels. Fig. 8 shows the estimated Charpy transition curves for Steels A, B and C with rolling conditions 1 and 2. The Charpy test data was analyzed by the exponential equations as:\textsuperscript{15}

$$vE(T) = \frac{vE_{shelf}}{\exp\left\{-a \left(T - vT_E\right) + 1\right\}}$$

(Eq. 1)

$$BA(T) = \frac{100}{\exp\left\{-b \left(T - vT_S\right) + 1\right\}}$$

(Eq. 2)

where

\begin{itemize}
  \item $vE(T)$ = the absorbed energy at temperature $T$,
  \item $BA(T)$ = the brittle fraction at temperature $T$,
  \item $vT_E$ = the energy transition temperature,
  \item $vT_S$ = the ductile-to-brittle transition temperature (DBTT)
  \item $vE_{shelf}$ = the upper shelf energy and
  \item $a$ and $b$ = constants.
\end{itemize}

The transition curves in Fig. 8 and DBTT in Table 2 were obtained by the least-squares approximation with Eqs. 1 and 2.

Even with condition 1, which gradually increases the reduction per pass from the roughing pass, excellent toughness was achieved with the DBTT of about $-130^\circ$C and lower. However, toughness was further improved by the condition 2, in which DBTT was lowered by about $-15^\circ$C for all the steels for all the chemistries.

### Table 2

<table>
<thead>
<tr>
<th>Steel</th>
<th>Rolling pass condition</th>
<th>Tensile properties</th>
<th>Charpy test</th>
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<td></td>
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<td>YS (MPa)</td>
<td>TS (MPa)</td>
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<tr>
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<td>468</td>
<td>567</td>
</tr>
<tr>
<td></td>
<td>Condition 2</td>
<td>501</td>
<td>565</td>
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<tr>
<td>B</td>
<td>Condition 1</td>
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<td>528</td>
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<td></td>
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<td>475</td>
<td>551</td>
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<tr>
<td>C</td>
<td>Condition 1</td>
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<td>585</td>
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<tr>
<td></td>
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Charpy transition curves of the plates: Steel A (a), Steel B (b) and Steel C (c).
Effect of Plate Rolling Strategy and Nb Addition on Heat-Affected Zone Toughness

Experimental Procedures — Steels A, B and C with rolling condition 1, and Steel B with rolling condition 2, were used for HAZ simulation tests. First, block samples of dimensions 15 mm (t) x 75 mm (W) x 80 mm (L) were taken from the plate in the mid-thickness region. Then, the heat treatment simulating for the CGHAZ and the ICCGHAZ, which are schematically shown in Fig. 1, were applied using a Gleeble thermal-mechanical simulator. The maximum heating temperature was 1,400°C in all cases. Heating and cooling rates were precisely controlled to simulate the welding thermal cycle in DSAW line pipe. Fig. 8 shows the heating and cooling cycles for CGHAZ and ICCGHAZ, which represents the heat input equivalent to about 50 kJ/cm. Charpy specimens were taken from the block sample after the thermal simulation and subjected to Charpy impact testing. Light optical microscope and SEM-based metallographic examination was also conducted on the same samples. For the SEM observation, a two-stage etching procedure consistent of Nital etching followed by electrochemical etching was used to dissolve cementite and then the remaining MA phase.

Effect of Nb and Rolling Strategy on Simulated HAZ Toughness and HAZ Microstructure — Charpy test results of the simulated HAZ for the three steels are shown in Fig. 11 and consist mainly of bainite (acicular ferrite or lath bainite) and quasi-polygonal ferrite. While precise microstructure investigations would be needed to clarify the effect of Nb on HAZ microstructure, Steels B and C clearly exhibited smaller grain size and improved toughness when compared to Steel A for both CGHAZ and ICCGHAZ after HAZ simulation. To explain the significant deterioration of toughness in ICCGHAZ, it is postulated that massive MA formation occurs because heating to the ferrite-austenite two-phase region results in carbon enrichment to the newly formed austenite phase, resulting in forming of MA phases during cooling after the second welding pass. Fig. 12 shows SEM images of the simulated CGHAZ and ICCGHAZ. Small volume fractions of fine, blocky MA particles, which are shown in Fig. 12 with the yellow arrows, are frequently observed in the ICCGHAZ in all samples. However, the volume fraction of MA is reduced in Steels B and C when compared to Steel A, which could be attributed to the finer and more homogeneous pre-austenite grain size during the heating cycles, as otherwise the carbon content and overall hardenability of the three chemical compositions is very similar. In addition to the MA formation in the ICCGHAZ, precipitation of Nb carbides can also contribute to the deterioration of ICCGHAZ toughness. Since NbC or other carbonitrides are assumed to be dissolved by the first heating cycle up to 1,400°C, it is expected that this solute Nb can precipitate new carbides during the second heating cycle. Table 3 shows the measured Vickers hardness of the CGHAZ and ICCGHAZ samples rolled using condition 1 after HAZ simulation. It can be seen that the ICCGHAZ samples exhibit higher hardness than the CGHAZ sample, likely because of precipitation and/or dislocation hardening induced by precipitation and bainitic phase transformation during the second heating and cooling cycle. Therefore, it is concluded that the toughness reduction observed
in the ICCGHAZ is caused by a combination of the formation of blocky MA and precipitation hardening.

Regarding the effect of rolling strategy, Steel B with the rolling condition 2 exhibited a smaller average grain size after HAZ thermal simulation than when rolled with condition 1, both in the CGHAZ and the ICCGHAZ. This is exemplified by the microstructure images in Fig. 11. Quantitative analysis of solute Nb and precipitates by the extraction residual method performed on the sample after the full CGHAZ heat cycle as described in Fig. 9a yielded a mass balance indicating that almost all Nb exists as solute in the CGHAZ. Therefore, it is concluded that the majority of the Nb carbonitrides dissolve during rapid heating to 1,400ºC, and their austenite boundary pinning capacity is lost. On the other hand, it is known that the solute Nb exhibits a strong solute drag effect which retards grain growth. The combination of the grain size homogeneity of the parent plate microstructure, the initial size distribution, volume fraction, and thermal stability of precipitates and their dissolution kinetics will be important in controlling the subsequent grain growth behavior of the steel microstructure during the very high-temperature phase of heating to the peak temperature of the welding cycle. After the majority of the Nb carbonitride precipitates are dissolved, first the initial austenite microstructural homogeneity and subsequently the presence of whatever residual thermally stable precipitates such as TiN exist, and the presence of solute Nb will become the main factors retarding very high-temperature grain growth in the CGHAZ. Delineating the relative contribution of starting grain size, residual precipitates and solute drag at very high temperatures on the net kinetics of austenite grain growth and the final HAZ microstructure in these steels is complex and will require further investigation. However, the overall net effect is an appreciable refinement in the final CGHAZ and ICCGHAZ microstructures through a combination of these mechanisms.

Charpy transition curves of simulated CGHAZ (a) and ICCGHAZ (b).
Table 3

<table>
<thead>
<tr>
<th>Steel</th>
<th>Rolling strategy</th>
<th>DBTT Base metal (°C)</th>
<th>CGHAZ</th>
<th>ICCGHAZ</th>
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<tr>
<td>A</td>
<td>Condition 1</td>
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<td>77</td>
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<td>Condition 1</td>
<td>-123</td>
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<td>26</td>
</tr>
<tr>
<td></td>
<td>Condition 2</td>
<td>-140</td>
<td>-29</td>
<td>9</td>
</tr>
<tr>
<td>C</td>
<td>Condition 1</td>
<td>-133</td>
<td>-43</td>
<td>5</td>
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Table 4

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<thead>
<tr>
<th>Steel</th>
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<tr>
<td></td>
<td>CGHAZ</td>
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<tr>
<td>A</td>
<td>191</td>
</tr>
<tr>
<td>B</td>
<td>200</td>
</tr>
<tr>
<td>C</td>
<td>185</td>
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</table>

Figure 11

Microstructure of simulated CGHAZ and ICCGHAZ.

Figure 12

SEM micrographs of simulated CGHAZ and ICCGHAZ.
Effect of Nb on Heat-Affected Zone Toughness of DSAW Weld Joint

Experimental Procedures — DSAW joints were prepared using Steels A and B. Plate rolling conditions were almost the same as those used to prepare the material for HAZ simulation, but final plate thickness was set as 25 mm to simulate thicker line pipe. To obtain a finer microstructure and excellent base metal toughness, intensified rolling at the recrystallization temperature (condition 2 in Fig. 2) was adopted as the rolling strategy. SAW was conducted using laboratory equipment with the heat input of about 50 kJ/cm for both inside and outside welding passes. Fig. 13 shows an indicative macrostructure of the weld joints produced. Charpy-V specimens were taken from the welds with the notch positions oriented to match the CGHAZ and ICCGHAZ positions in the point, as shown in Fig. 14.

HAZ Toughness of DSAW Joint and Microstructure — Fig. 15 shows the Charpy transition curves for CGHAZ and ICCGHAZ. Both steels showed a fairly excellent HAZ toughness for the low-temperature use. The DBTT was the same for Steels A and B in CGHAZ, while Steel B exhibited far better toughness in ICCGHAZ. Figs. 16 and 17 show microstructure of CGHAZ and ICCGHAZ of the DSAW joints, respectively. It is observed that Steel B shows a smaller prior austenite grain size than Steel A in the region connected to the fusion line in both the CGHAZ and ICCGHAZ. Although precise and statistical analysis over a large number of welds is required for final confirmation, these results show that detrimental grain growth could be prevented in Steel B with a higher Nb content in the welded joint in the same fashion as was shown by the simulated HAZ testing under comparable conditions.
Conclusions

The effect of plate rolling strategy and Nb addition on the HAZ performance was investigated by using laboratory-rolled plates, HAZ simulation and DSAW welding tests. By applying the optimized rolling condition with the intensified reduction in the roughing passes, a fine bainitic microstructure with homogeneous grain size distribution was achieved with steels containing 0.030% to 0.055% Nb, resulting in excellent base metal toughness. The HAZ-simulated DSAW welds proved that HAZ toughness is improved by the Nb addition for both the CGHAZ and ICCGHAZ because of the refined microstructure produced. This refinement is likely due to a combination of optimal grain boundary pinning and dissolution characteristics of Nb precipitates in the initial plate microstructure during heating, and the subsequent solute drag effect of Nb on grain growth at very high temperatures. The refined microstructures produced by the optimized rolling strategy were effective at improving the final HAZ toughness by maintaining a finer grain size in the HAZ. Finally, actual DSAW welded joints with a heat input of about 50 kJ/cm using the plate with Nb addition of 0.045% and applying the optimized rolling strategy exhibit fairly excellent toughness both in CGHAZ and ICCGHAZ. A refined microstructure close to the fusion line was shown to be the reason for the excellent HAZ toughness obtained.

References