Austenite grain size control in upstream processing of Nb microalloyed steels by nano-scale precipitate engineering

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Outline

- What is the market driver? Why should we control austenite grain size before pancaking?
  There is a growing demand for thicker gage pipes particularly for deep off-shore projects.
  Austenite grain size control before pancaking is essential to obtain excellent DBTT and DWTT properties in thicker gage product.
- What causes austenite grains to coarsen in upstream processing and how can we control it?
- Concept of nano-scale precipitate engineering of TiN-NbC composites to prevent grain coarsening of austenite by Zener drag in upstream processing
- Basic science aspects of nano-scale precipitate engineering
- Technological aspects: (i) control of TiN dispersion and (ii) control of roughing temperature window for NbC growth on pre-existing TiN to pin austenite grains of the required size
- Accelerated cooling upstream to retain adequate solute niobium on entry to finish rolling and to enter below temperature of no recrystallization
- Application of nano-scale precipitate engineering for plate rolling (X-100), conventional hot strip rolling (X-90) – validation of the concept.
- Potential application for near net shape processing of Nb microalloyed grades
- Conclusions - Technological take-away - Acknowledgements
DBTT and DWTT properties are controlled by crystallographic domain size, i.e. the density of high angle misorientation boundaries.

--- J.Y. Koo

Effect of pancaked austenite grain size (thickness) on high angle boundaries (>45º) as shown by yellow lines. Grain refinement of austenite before pancaking is key to suppress competition from brittle fracture as measured by %age shear in DWTT. High angle boundaries arrest microcracks before they initiate brittle fracture.
Austenite grains in the centre region of X80 plate steel with large thickness

<table>
<thead>
<tr>
<th>Heat</th>
<th>$A_k$ -20 °C</th>
<th>DWTT (-15 °C)</th>
<th>Average SA %</th>
</tr>
</thead>
<tbody>
<tr>
<td>a</td>
<td>328</td>
<td>55</td>
<td></td>
</tr>
<tr>
<td>b(OHTP)</td>
<td>372</td>
<td>98</td>
<td></td>
</tr>
</tbody>
</table>

Austenite grain refined before pan-caking

Courtesy Wenjin Nie, Shagang
DBTT as a function of the Slab to Strip Thickness Ratio for different steel grades

In the absence of intentional austenite grain size control upstream, heavy pancaking is required to obtain consistent DBTT and DWTT properties downstream, which limits the gage thickness of final product.

-M. Guagnelli and P. Bobig
High nitrogen: 0.0075 N, 0.015 Ti, 0.06 C, 0.09 Nb (in wt percent)

<table>
<thead>
<tr>
<th>I.D. Steel-A</th>
<th>TiN size (nm)</th>
<th>Particle spacing (nm)</th>
<th>TiN-NbC size (nm)</th>
<th>TiN-NbC volume fraction</th>
<th>Limiting austenite grain size (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>High N</td>
<td>60</td>
<td>553</td>
<td>83</td>
<td>0.00186</td>
<td>62</td>
</tr>
</tbody>
</table>

Present  Technology with no intentional control of austenite grain size upstream before pancaking

Heavy rolling reduction: 66–80%

Thinner gage product: 10–17 mm
Novel use of Kozazu’s Diagram on Sv factor for producing thicker gage product by austenite grain refinement upstream
Effect of rolling reduction on $S_v$ factor as influenced by prior austenite grain size

*Austenite grain size is not intentionally controlled upstream in current technology and is generally about 60 microns.

**Austenite grain size is controlled below 30 micron by nano-scale precipitate engineering according to the new technology.

Ouchi, Sampei and Kozasu, ISIJ, 22, 1982

New Technology with intentional control of austenite grain size upstream by nano-scale precipitate engineering of TiN-NbC composite

Low nitrogen: 0.004 N, 0.015 Ti, 0.05 C, 0.09 Nb (in wt percent)

<table>
<thead>
<tr>
<th>I.D.</th>
<th>TiN size (nm)</th>
<th>Particle spacing (nm)</th>
<th>TiN/NbC size (nm)</th>
<th>TiN/NbC volume fraction</th>
<th>Limiting austenite grain size (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low N</td>
<td>22</td>
<td>221</td>
<td>32</td>
<td>0.00159</td>
<td>27</td>
</tr>
</tbody>
</table>

Less rolling reduction: 50~66%

Thicker gage product: 16~27 mm
Microstructural Control

Morphology of prior-austenite grains after 25% deforming and holding: (a) 3 s, (b) 60 s and (c) 240 s at 1000 °C.

\[ G_t = \frac{1}{2} \rho \mu b^2 \]

Dislocation density is the driving force for recrystallization.

\[ \langle r_t \rangle = \langle r_0 \rangle + \int_0^t MG_t \, dt \]

\[ M = M_0 e^{\frac{-Q}{RT}} \]

\[ M(t) = \left( \frac{1}{M_{\text{pure}}} + \alpha C_{\text{Nb}} \right)^{-1} \]

\[ G_t = 2 \frac{\gamma}{\langle R_t \rangle} \]

Capillary force is the driving force for grain coarsening.

Coarsening kinetics of recrystallized austenite grains driven by capillary force.
Growth of NbC on well dispersed TiN causes deviation from Sellar’s model prediction of RLT, which is based on strain induced precipitate interaction with recrystallization.
Figure 9  Driving and pinning pressures for boundary mobility after Ashby (19). The arrow shows the required pinning pressure to counteract the driving pressure from deformation.
Maximum restraining force exerted by each precipitate is $\pi r \gamma$

Total restraining force on the boundary is $N_s$ times the restraining force of each precipitate, i.e., $N_s \pi r \gamma$, where $N_s$ is the number of precipitates per unit area on the boundary, which determines the interparticle spacing $\lambda$.

Zener pinning force is increased by (i) increasing $r$ and (ii) decreasing interparticle spacing $\lambda$.
Increasing base Nb concentration

<table>
<thead>
<tr>
<th>Inter Particle Distance, nm</th>
<th>10</th>
<th>15</th>
<th>20</th>
<th>25</th>
<th>30</th>
<th>35</th>
<th>40</th>
<th>50</th>
<th>60</th>
<th>70</th>
<th>80</th>
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</thead>
<tbody>
<tr>
<td>150</td>
<td>85</td>
<td>38</td>
<td>21</td>
<td>14</td>
<td>10</td>
<td>7</td>
<td>5</td>
<td>3</td>
<td>2</td>
<td>1.8</td>
<td>1.3</td>
</tr>
<tr>
<td>200</td>
<td>203</td>
<td>90</td>
<td>51</td>
<td>32</td>
<td>22</td>
<td>17</td>
<td>13</td>
<td>8</td>
<td>6</td>
<td>4</td>
<td>3</td>
</tr>
<tr>
<td>250</td>
<td>397</td>
<td>176</td>
<td>99</td>
<td>63</td>
<td>44</td>
<td>32</td>
<td>25</td>
<td>16</td>
<td>11</td>
<td>8</td>
<td>6</td>
</tr>
<tr>
<td>300</td>
<td>687</td>
<td>305</td>
<td>171</td>
<td>110</td>
<td>76</td>
<td>56</td>
<td>43</td>
<td>28</td>
<td>19</td>
<td>14</td>
<td>11</td>
</tr>
<tr>
<td>350</td>
<td>1091</td>
<td>485</td>
<td>273</td>
<td>174</td>
<td>121</td>
<td>89</td>
<td>68</td>
<td>44</td>
<td>30</td>
<td>22</td>
<td>17</td>
</tr>
<tr>
<td>400</td>
<td>1629</td>
<td>724</td>
<td>407</td>
<td>260</td>
<td>181</td>
<td>133</td>
<td>102</td>
<td>65</td>
<td>45</td>
<td>33</td>
<td>25</td>
</tr>
<tr>
<td>450</td>
<td>2320</td>
<td>1030</td>
<td>580</td>
<td>371</td>
<td>257</td>
<td>190</td>
<td>145</td>
<td>93</td>
<td>65</td>
<td>47</td>
<td>36</td>
</tr>
<tr>
<td>500</td>
<td>3183</td>
<td>1414</td>
<td>795</td>
<td>509</td>
<td>353</td>
<td>259</td>
<td>199</td>
<td>127</td>
<td>88</td>
<td>65</td>
<td>50</td>
</tr>
<tr>
<td>550</td>
<td>4236</td>
<td>1882</td>
<td>1059</td>
<td>678</td>
<td>471</td>
<td>346</td>
<td>265</td>
<td>170</td>
<td>118</td>
<td>86</td>
<td>66</td>
</tr>
</tbody>
</table>

Zener limiting Austenite grain size in micrometer

(decreasing N concentration)

(increasing r)

(decreasing interparticle spacing λ)
Temperature window of roughing has to be optimized for growth of NbC on TiN.
Effects of N, C, on precipitate spacing, austenite grain size:

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<td>0.00186</td>
<td>62</td>
</tr>
<tr>
<td>(low N)</td>
<td>20</td>
<td>218</td>
<td>32</td>
<td>0.00164</td>
<td>26</td>
</tr>
<tr>
<td>(low C,N)</td>
<td>20</td>
<td>218</td>
<td>29</td>
<td>0.00128</td>
<td>31</td>
</tr>
</tbody>
</table>

(high N): 0.0075 N, 0.015 Ti, 0.06 C, 0.09 Nb  
(low N): **0.0035 N**, 0.014 Ti, 0.07 C, 0.08 Nb  
(low C, N): **0.0035 N**, 0.015 Ti, **0.045 C**, 0.08 Nb
The smaller the austenite grain size, the greater is the driving force for grain coarsening. High density of fine precipitation of TiN-NbC is required for Zener drag to prevent grain coarsening. This requires nitrogen control in steel to low level (30-40 ppm) with stoichiometric addition of Ti to form high density and uniform dispersion of TiN. Growth of NbC on pre-existing TiN develops adequate Zener drag to counteract increased driving force associated with fine grains.
(high N): 0.0075 N, 0.015 Ti, 0.06 C, 0.09 Nb

High N – Large Inter-particle Spacing: 553nm

(low N): 0.0035 N, 0.014 Ti, 0.07 C, 0.08 Nb

Low N Inter-particle spacing: 220nm

Nano-scale precipitate engineering requires control of both chemistry and processing.
Size and dispersion of Ti-Nb rich precipitates in X90 strip

High number density of nano-scale precipitation of TiN fixes the inter-particle spacing 200-300nm upstream. Growth of NbC on pre-existing particles increases the Zener pinning force to counteract capillary force for grain coarsening.
TEM-EELS characterization of TiN-NbC precipitates
Band contrast maps from EBSD characterization on surface and center of X90 strip with thickness of 16.4mm showing pancaked austenite grain size under 10µm in the surface and center of the strip.
Technological take-away:
HTP is the workhorse for line pipe steel. There are four functional roles of niobium in conventional thermo-mechanical rolling of classical HTP: (i) Retardation of recrystallization by strain induced precipitation and solute drag, (ii) strain accumulation in pancaked austenite, (iii) transformation hardening and (iv) precipitation strengthening by interphase precipitation and precipitation in ferrite.

In order to produce thicker gage, the functional roles of niobium are expanded in the new technology (HTP-PLUS) to include austenite grain size control upstream before pancaking by nano-scale precipitation engineering of TiN-NbC composites. This innovation offers a general platform for austenite grain size control over a wide range of product applications including line pipe as well as non-line pipe applications such as infra-structure steel and super-martensitic stainless steel.
CONCLUSIONS:

By taking advantage of low N chemistry and accelerated cooling, the new technology enables limiting of austenite grain size on entry to finish rolling, and so permits the application of classical HTP to thicker grades of higher strength linepipe steels: less pancaking is required on finish rolling.

The science underlying the formation of NbC/TiN pinning precipitates has been verified, and the new technology (HTPPLUS) has been validated in mill trials on X-80, X-90 and X100. Further applications of the underlying principle are suggested, including the processing of super-martensitic stainless grades and near net-shape processing.
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