The effects of hot rolling process and the nitrogen and sulphur content on the microstructural development of aluminium killed hot rolled low carbon strip steel

by C.W. Siyasiya* and W.E. Stumpf*

Synopsis

Process and steel chemistry differences in hot rolled Al-killed low carbon strip steel between the hot charge route (HCR) as practised in a compact strip production (CSP) plant and the conventional cold charge route (CCR) have been studied. This was done with reference to differences in reheating practice, hot rolling practice and steel chemistry through emphasis on the effects of solubility and reprecipitation of AlN in the two process/product combinations and its effect on ferrite grain size development. An earlier modelling study on the evolution of the austenite grain size in both of the above hot rolling processes has also now been extended to include an estimate of the possible effects of retained strain after the last finishing pass on the ferrite grain size. As part of this study the equilibrium solubility trends of AlN in a number of boron-free low carbon Al-killed strip steels with low and high sulphur content from the two respective processes was investigated by the thermoelectric power (TEP) technique and differences in the effects through the derived solubility equation of Log[Al][N]=2.6–9710/T at typical slab reheating temperatures was demonstrated. Ferrite grain size modelling using the significantly lower $A_{f}$ transformation temperatures generally found in hot rolled low carbon strip steel produced in the CSP plant using the HCR compared well to the larger grain size produced in the conventional CCR strip rolling plant.

Keywords: austenite grain size modelling; thermoelectric power (TEP); compact strip production (CSP); cold charge route (CCR); ferrite grain size

Introduction

Background

In South Africa ArcelorMittal South Africa Limited produces hot rolled low carbon strip steel at both its Vanderbijlpark as well as at its Saldanha Works. The former is a conventional strip producing plant using the cold charge route (CCR) in which the continuously cast slab is allowed to cool down to below the austenite ($gamma$) to ferrite ($alpha$) transformation temperature before entering the reheating furnace for hot rolling in the austenite phase. The Saldanha plant uses the hot charge route (HCR) in a compact strip production (CSP) plant where the cast slab still in the austenitic condition, enters the roller hearth furnace directly for reheating before hot rolling commences. Although both products are nominally of very similar composition, relatively small but nevertheless significant differences existed in their hot rolled microstructures, mechanical properties, $gamma$ to $alpha$ transformation behaviour and also in their static recrystallization behaviour after cold rolling.$^{1-5}$ The main differences between the two processes were reviewed in some detail by Stumpf$^2$ and consist mainly of the above difference in reheating practice, the number of hot rolling passes with the Saldanha HCR plant with its compact design having only two roughing and five finishing passes compared to the up to seven roughing and seven finishing passes of the Vanderbijlpark CCR plant but with the former again starting with a thinner slab thickness of 75 or 90 mm versus the 240 mm of the latter. These differences result in significant differences in the strain per pass, the strain rates per pass and pass temperatures. Furthermore, the Saldanha plant originally achieved a lower sulphur content in the steel than the equivalent Vanderbijlpark product in its steel making. Greater or lesser use of electric arc furnace (EAF) melting in any of the two types of plant may also result in differences in the nitrogen content of the steel.

Soon after start-up of the Saldanha plant, the two respective products were found to have slightly different characteristics, with the HCR product generally having a somewhat smaller ferrite grain size as well as a higher yield strength and a lower $A_{f}$ austenite-to-ferrite transformation temperature. Other differences in steel-making practice led to a generally lower sulphur content in the HCR product whereas differences in rolling practice also exist between the two plants. An in-depth
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modelling study of the differences in hot rolling practice\textsuperscript{1,2}, however, indicated that the product differences most likely cannot be ascribed directly to differences in rolling practice. In this further comparative study on the reasons for the product differences, the modelling of the rolling practices has firstly been extended to include the effects of retained strain in the austenite on the run-out table where the austenite to ferrite transformation occurs and, secondly, the solubility of AlN in these steels as well as a parallel study on the recrystallization behaviour after cold work\textsuperscript{3}, to finally understand the source of these differences in product behaviour in order to optimize each plant’s output.

**Hot rolling practices**

From a large number of mill logs from the two plants, it was established that the two local plants of ArcelorMittal South Africa Limited produce hot rolled low carbon Al-killed strip steel, firstly using the HCR in the CSP plant with the typical lower soaking temperature of 1120 °C whereas secondly, in the conventional strip producing plant that uses the CCR, the reheat temperatures are higher at about 1180 °C. The steels from these two plants at the time of this study also differed significantly in their sulphur content, i.e. low sulphur content (~20 ppm) steels were generally produced in the CSP plant whereas the medium to high sulphur content (50< S<150 ppm) steels were produced in the conventional CCR strip plant. Furthermore, the hot rolled strip steel from the CSP-HCR plant generally had a smaller ferrite grain size of about 9 to 11 µm (compared to the 12 to 15 µm of the conventional CCR plant) and had a yield strength about 50 MPa higher, was more ‘sluggish’ in recrystallizing after cold work and had $A_r$ temperatures at typical run-out-table cooling rates that were up to 40 °C lower than in the equivalent strip steel from the conventional CCR plant\textsuperscript{4,5}. As it was suspected that differences in reheating practice may partly contribute to a finer ferrite grain size in the CSP plant, a study of the AlN solubility behaviour by TEP was also introduced.

Apart from the differences in thermal history between the CSP-HCR process and the conventional CCR process, the former has less roughing and finishing passes, a significantly larger starting austenite grain size as the slabs lack the refinement introduced by the first austenite to ferrite transformation in the slab, higher finishing strains and slower strain rates than the equivalent CCR process. The total strains in the last pass are typically about 0.28 to 0.32 in the HCR compared to 0.18 to 0.2 in the CCR process. The starting austenite grain size after reheating was introduced into the model as 150 µm for the CCR steel and 270 µm for the HCR steel. Despite these differences, the final austenite grain size at the finishing mill head, which was modelled using data from both plants from a large number of mill logs and also some published constants for low carbon strip steels, was found to be approximately the same for both processes\textsuperscript{1,2}, Figure 1. Although this study did not clearly confirm what the actual reasons for the product differences were, it did eliminate the above process differences as an actual cause and pointed towards a more in-depth study concerning the subtle chemical differences in the two steels, particularly the nitrogen and sulphur differences and the AlN precipitation and dissolution behaviour and how these would affect the product behaviour in the two respective processes.

It is worthwhile mentioning that the CCR mill at Vanderbijlpark runs relatively faster and, therefore its run-out-table’s cooling rate is less than the CSP mill at Saldanha i.e. roughly at most 80 versus 125°C/s respectively. Despite this substantial difference in run-out-table cooling rates between the two processes, ferrite transformation grain size models predict no significant grain size refinements\textsuperscript{6,7}, i.e. at most 5%, and this does not explain the industrially observed differences of up to 35% in ferrite grain size.

It was recognized that the rolling practice also affects the thermal history of the AlN, i.e. in the HCR steel the AlN freshly precipitates from the high temperature as-cast austenite, whereas in the CCR steel it reprecipitates after dissolution during reheating, which starts from below the $A_r$ temperature and ends with the solution treatment itself at 1180°C. This difference in process can cause differences in the amount of nitrogen in solid solution particularly if, in the latter case of the CCR process, the reheat temperatures are not high enough to redissolve all of the AlN.

**The solubility of AlN**

The precipitation and growth of AlN in these steels during and after hot rolling and during subsequent cooling has long been known to be one of the fundamental metallurgical processes that affect the final properties (strength, grain size, hardness, etc.) of these steels\textsuperscript{8} and, hence, one of the first aspects to be understood in this comparative study was the solubility of AlN in these particular steels and how this may be affected directly or indirectly in steels that also had varying sulphur contents. As part of this wide-ranging study on the role and behaviour of AlN in low carbon strip steels\textsuperscript{9}, the AlN dissolution kinetics at typical industrial reheating temperatures of about 1150 °C and the AlN equilibrium solubility in austenite during reheating was determined by the TEP technique in a number of commercially produced low carbon Al-killed strip steels with varying sulphur and nitrogen contents. Although a number of solubility equations for AlN in austenite have been published, these were either determined by the chemical extraction technique based on the Beegly process\textsuperscript{9} or from fundamental thermodynamic
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calculations\(^{10,11}\). Both of these procedures, however, have inherent limitations. The first method is unable to extract and recover the very fine AlN particles and also cannot distinguish AlN from any other co-extracted nitrides. The second method relies on fundamental thermodynamic data and neglects any indirect effects from other impurities and alloying elements present in commercially produced hot strip steel. The TEP technique, whose theory is well covered in literature\(^ {11,12}\), avoids these particular limitations but, of course, has also some limitations of its own that need to be addressed, such as possible stray TEP contributions from dislocations, strain aging, any coherency strains around particles and solid solution carbon atoms\(^ {13,14}\). For AlN solubility determination, these effects need to be eliminated or kept constant by appropriate experimental design. Brahmi et al\(^ {15}\) used the TEP technique to study the equilibrium solubility of AlN in an experimentally prepared pure Fe-Al-N alloy and the individual determinations were in reasonable agreement with the Leslie solubility equation\(^ {16}\), i.e.,

\[
\log_{10}[\text{AlN}] = 1.03 - \frac{6770}{T},
\]

although no TEP-derived solubility equation was determined for commercial steels. Therefore, this part of the wide-ranging study partially focused on the determination of the TEP-derived AlN solubility equation. It would appear that the TEP technique has not been used in the study of the solubility of AlN in austenite of commercial steels due to the fact that there might be other nitrides that may also be precipitating or dissolving within the AlN precipitation and dissolution temperature range (800° to 1300 °C), in particular BN, if the steel contains some boron. Therefore, in this study, steels containing boron (> 4 ppm) were excluded.

Experimental procedures

**Material preparation and solution treatment**

Slab sections of seven low Al-killed carbon steels whose chemical compositions are given in Table I were received from ArcelorMittal South Africa in the as-cast condition. These steels were hot and cold rolled in laboratory rolling mills down to 1 mm sheets. The nitrogen content was measured by LECO analysis and the acid soluble Al-content by chemical dissolution, while the content of the remaining elements were determined by spectrographic analysis. Strips of 90 mm long, 4 mm wide and 1 mm thick were cut from these plates for the TEP measurements. All the specimens were first annealed at 800°C for 6 hours in an argon atmosphere and water quenched. This was done to ensure complete precipitation and coarsening of the incoherent AlN before subsequent solution treatment that was carried out at various test temperatures followed by water quenching. Furthermore, to avoid strain aging effects, TEP measurements were taken immediately after the solution treatment and quenching. The AlN dissolution as measured by TEP was confirmed metallographically by EDS-TEM on both carbon extraction replicas as well as on thin foils.

**TEP measurements**

The schematic diagram and apparatus for the TEP measurement are given in Figures 2 and 3 respectively. Two copper blocks were kept at a temperature difference of 15ºC in order to maintain a temperature gradient \(\Delta T\) between the cold and hot junctions of the specimen. One block was filled with ice and the temperature at the cold junction was maintained at 5° ± 1°C while the other one at the hot junction was filled with water at room temperature at 20° ± 1°C. K-type thermocouples were spot-welded onto the hot and cold junctions of the specimen and a data logger was used to record both the temperature and the voltage drop \(\Delta V\) across the hot and cold junctions into different analog channels.

![Figure 2—Schematic diagram for the TEP measurements]

![Figure 3—Photograph of the TEP measurement arrangement]

| Table 1

<table>
<thead>
<tr>
<th>Chemical compositions of the investigated steels</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Element</strong> (ppm)</td>
</tr>
<tr>
<td>-------------------</td>
</tr>
<tr>
<td>C</td>
</tr>
<tr>
<td>Mn</td>
</tr>
<tr>
<td>S</td>
</tr>
<tr>
<td>Ti</td>
</tr>
<tr>
<td>Al (total)</td>
</tr>
<tr>
<td>Al (acid sol)</td>
</tr>
<tr>
<td>N</td>
</tr>
<tr>
<td>HS = high sulphur and LS = medium to low sulphur, the first numeral is for the sulphur content and the second for the nitrogen content, both in ppm.</td>
</tr>
</tbody>
</table>
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Voltage drop was measured directly from the Chromel wires from the cold and hot junctions, ensuring that \( \Delta V \) was measured instantaneously with the corresponding \( \Delta T \) between the cold and the hot junctions out of which the instantaneous absolute TEP measurement was obtained.

Figure 4 shows a schematic illustration of the heat treatment cycles and TEP measurement sequences for the study of the dissolution of AlN in austenite during reheating. The absolute TEP values designated \( S_0, S_{800} \) and \( S_i \) correspond to the as-received condition, after annealing at 800°C/6 h, and after soaking for 12 minutes at the test reheating temperature respectively. Assuming that the diffusion of electrons was more predominant than the phonon drag at room temperature\(^{17,18}\) and as the concentrations of both the aluminium and nitrogen were low (< 0.1 atomic-per cent), the Gorter-Nordheim rule can be used with confidence. For a stoichiometric composition of AlN, the relative TEP value \( \Delta S_N \) can be obtained through the linear relationship:

\[
\Delta S_N = S_{800} - S_i = K_{AlN}[N]
\]

where \( K_{AlN} \) is the aluminium nitride's TEP coefficient for the nitrogen content \([N]\) in weight per cent in solid solution in the austenite at the test temperature.

Rolling process modelling

The rolling process modelling of these steels has been dealt with in detail elsewhere\(^1\)\(^,\)\(^2\). In HCR steel, the model predicted that DRX (dynamic recrystallization) takes place within the rolls during the two roughing and the first two finishing passes whereas SRX (static recrystallization) occurs after the rolls in the last three passes. In the CCR steel, DRX occurs in the first two roughing and SRX takes place in the rest of the passes, Figure 1. The retained strain and the fraction softening by SRX during the last three finishing passes were also predicted using the same model. Data from the mill logs on strains, strain rates, rolling temperatures and roll forces were used in the modelling of the SRX volume fraction and the retained strain during the last three finishing passes. The models for the prediction of the ferrite grain size nucleated from the recrystallized and unrecrystallized austenite are also given in Table II.

Results

Dissolution kinetics of AlN as measured by TEP

Figure 5 shows the evolution of the \( \Delta S_N \) after the isothermal soaking of two steels with high and low sulphur content, i.e. HS140-104 and LS2-65 respectively at 1150°C for various times and then quenching them into water. (Note that the first numeral in each steel’s identification is for the sulphur content and the second for the nitrogen content, both in ppm.) The relative TEP values were taken with reference to the ‘no dissolution’ TEP values at 800°C. These values levelled off after soaking for 5 minutes, which was an indication that equilibrium conditions in the dissolving AlN were attained.

Equilibrium solubility trends of AlN in low and high sulphur strip steels

The logarithm of the solubility product \( \log[Al][N] \) derived from \( \Delta S \) through Equation [1], is plotted against the inverse of the absolute solution temperature in Figure 6. From regression analysis the TEP-derived solubility equation for AlN in austenite was found to be:

Table II

<table>
<thead>
<tr>
<th>Ferrite nucleation</th>
<th>Model</th>
<th>Remarks</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nucleation of ferrite grains from recrystallized austenite (no retained strain)</td>
<td>( D_a_{SRX} = (6.77 - 10[C] - [Mn][C]/[C]_0^{0.177})/O^{2/3} )</td>
<td>( D_a_{SRX} = ) a grain size from SRX aust. (µm), ( D_a = ) austenite grain size from prior grain growth (µm), ( C = ) cooling rate in °C/s, ([C]) and ([Mn]) =</td>
<td>6</td>
</tr>
<tr>
<td>Nucleation of ferrite grains from unrecrystallized austenite grains with retained strain</td>
<td>( D_a_{UnSRX} = (6.77 - 10[C] - [Mn][C]/[C]_0^{0.177})/O^{2/3} )</td>
<td>( D_a_{UnSRX} = ) ferrite grain size from unrecrystallized austenite grains (µm), ( \varepsilon = ) retained strain in the unrecrystallized austenite</td>
<td>6</td>
</tr>
<tr>
<td>Weighted average of ferrite grain size after rolling</td>
<td>( D_a = D_a_{SRX}X_{SRX} + D_a_{UnSRX}(1 - X_{SRX}) )</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
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\[
\text{Log}[\text{Al}][\text{N}] = 2.6 - \frac{9710}{T} \tag{2}
\]

where the Al and N contents are in weight percentage, \(T\) is the absolute solution temperature in Kelvin. The standard error on the y-intercept was 2.6 ±0.8 and on the slope (temperature coefficient) was 9710 ±1100 K.

Metallographic analysis

Steels HS140-104 and LS2-65 were chosen for metallographic analysis to confirm the dissolution treatments. The micrograph in Figure 7a shows coarse and fine AlN particles in these two steels after isothermal annealing at 800°C for 6 hours. It was also observed that in the as-coiled condition (simulated hot rolling and coiling on a Gleeble 1500™) and in the steels with medium to high sulphur content, i.e. > 50 ppm, the AlN precipitated heterogeneously on MnS particles, Figure 7b, as opposed to low S steel, with virtually no MnS particles, in which the AlN was homogeneously nucleated in the matrix or heterogeneously on dislocations, sub or grain boundaries, Figure 7c. Partial dissolution of the AlN occurred in the higher nitrogen steel HS140-104, whereas almost complete dissolution in the medium nitrogen steel LS2-65 took place after soaking for 12 minutes at 1150°C. Full dissolution of the AlN in steel HS140-104 occurred at 1250ºC and this was in reasonable agreement with TEP results.

Effects of retained strain on the ferrite grain size

The modelled retained strain in the austenite at the transformation from austenite to ferrite was found to be higher in the CCR steel than in the HCR steel, Table III. As opposed to the conventional CCR, the higher finishing strains in the HCR process induced SRX during the last two passes, as shown by the modelled fraction softening by SRX in Figure 8, and this resulted in lower retained strains in this steel.

As may be seen from Figure 1, the modelled final austenite grain size \(D_f\) was found to be approximately the same for both processes, i.e. about 25 \(\mu\)m. The modelled fraction softened by SRX after the last passes were found to be 0.75 for the HCR steel and 0.1 for the CCR steel, Figure 8.
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The ferrite grain size arising from the austenite to ferrite transformation was predicted using the models in Table II and, at typical run-out table cooling rates of about 80°C s⁻¹ (for a 2 mm thick strip), they were found to be about 9.4 µm for the HCR steel and 9 µm for the CCR steel, providing a ratio of Dₘ₋CCR/Dₘ₋HCR of 0.96, Table V. This is contrary to what is observed at the two plants whereby the Dₘ₋CCR/Dₘ₋HCR ratio is on average 1.35. This suggests that the higher retained strain in the CCR may not have a significant influence on the ferrite grain size and that the main cause of the differences in grain size lies elsewhere, as discussed later.

Discussion

Effect of sulphur on the dissolution kinetics and equilibrium solubility of AlN

As may be seen from Figure 5, the TEP values levelled off after soaking for more than 5 minutes at 1150°C, indicating equilibrium conditions in the solubility of the dissolving AlN in both steels. Therefore, the presence of sulphur does not appear to have any effect on the dissolution kinetics of the AlN under equilibrium conditions. i.e. at least beyond 5 minutes soaking time. Furthermore, from Figure 6, the sulphur content of the steel also does not appear to have any measurable effect on the equilibrium solubility of AlN in these steels, at least within the experimental scatter found. This is in agreement with earlier observations made by others in similar steels using other techniques than TEP¹⁶,¹⁹ with the exception, however, that these TEP results predict slightly higher solution temperatures of the AlN when compared with the results obtained through the Beeghly technique. On the one hand, the Beeghly technique mainly lacks sensitivity to particles of less than 10 nm in diameter and fails to isolate the AlN from other nitrides. Consequently, lower solubility temperatures are reported by the Beeghly technique compared to other techniques. On the other hand, the accuracy of the TEP-derived solubility Equation [2] is dependent on the precision of the chemical analysis as the TEP values are correlated to the LECO-analysed nitrogen content. Effects of strain aging may also lead to lower solubility values in the TEP technique although this effect was minimized by immediate TEP measurements after quenching the specimens in water. Despite the differences in the predicted AlN solubility limits, all models including the one from this work indicate that the sulphur content (that includes effects from the nucleation of AlN on MnS) in these low carbon Al-killed strip steels does not influence the equilibrium solubility of AlN.

Implications of the observed AlN solubility results on hot rolling practices

The AlN solubility isotherms at 1180°C for the often quoted Leslie equation as well as for the TEP equation from this work are shown in Figure 9. The five steels studied here are also plotted on this figure. Firstly, these five steels all appear to contain an excess of soluble Al and secondly, whereas reheating practice at 1180°C according to the Leslie equation predicts complete dissolution of the AlN in four of the five steels, the TEP solubility equation predicts incomplete dissolution in four of the steels with the fifth one possibly a borderline case. It is worthwhile to consider the implications of such differences in the predicted solubility on industrial hot strip mills that employ somewhat different reheating temperatures of 1120 and 1180°C as is the case in the respective HCR and CCR processes being compared here.

Table III

The modelled retained and total strain for the HCR and CCR processes

<table>
<thead>
<tr>
<th>Process</th>
<th>Retained strain</th>
<th>Last pass total strain</th>
</tr>
</thead>
<tbody>
<tr>
<td>HCR</td>
<td>0.09 to 0.11</td>
<td>0.26 to 0.32</td>
</tr>
<tr>
<td>CCR</td>
<td>0.18 to 0.22</td>
<td>0.18 to 0.2</td>
</tr>
</tbody>
</table>

Figure 8—Modeled fraction softening by SRX in the last three passes of the CCR and HCR processes

Figure 9—The chemical compositions for the steels that were studied are superimposed on the isotherm solubility curves at 1180°C obtained from this work and from Leslie et al.¹⁴
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In the CSP-HCR plant, the full nitrogen content (excluding minor amounts locked up in TiN) from continuous casting will likely be in solid solution during reheating at 1120°C as no dissolution of any AlN is relevant. Secondly, it has been shown that during hot rolling at lower temperatures, any AlN needs to nucleate homogeneously in the matrix in the low sulphur steels produced by the HCR process at the time of this study, due to the absence of any significant quantities of MnS/[Mn,Cu]S particles in these steels with generally less than 50 ppm of sulphur. The homogeneous nucleation of the AlN presents a thermodynamic barrier to its nucleation in austenite\textsuperscript{8,20} in the CSP-HCR steel, resulting in slow kinetics and relatively large concentrations of nitrogen are likely still to be in solid solution at the point of transformation to ferrite on the runout table. Nitrogen in solid solution is one of the strongest austenite formers\textsuperscript{21} and the relatively high concentration of nitrogen in solid solution, therefore, lowers the $A_{3}$ temperature and leads to the observed smaller ferrite grain size in these steels.

On the other hand, in the CCR of the conventional plant, the TEP-derived isotherm of 1180°C in Figure 9 predicts that the reheate temperatures employed in this plant may not be high enough to completely dissolve the AlN in these steels and, therefore, less nitrogen will be in solid solution if compared to the low sulphur content steels produced in the CSP-HCR plant. For instance, estimates from Figure 9 show that four of the steels examined here, may still have contained up to about 60 per cent of their total nitrogen still locked up in AlN after reheating. Secondly, in these generally medium to high sulphur content steels, the presence of MnS/[Mn,Cu]S allows the rapid heterogeneous nucleation of AlN on these particles at higher temperatures, which will deplete the already nitrogen-poor matrix even further from nitrogen at the point of transformation to ferrite\textsuperscript{22,23}. This will lead to the measured higher $A_{3}$ temperatures of about 40°C at a cooling rate of 80°C/s (Table IV) than those exhibited in the CPS-HCR plant and consequently, a larger ferrite grain size will be found in the CCR product, which is in agreement with observed behaviour.

Modelling the ferrite grain size differences in HCR and CCR produced strip steels

Although the above qualitative reasoning behind the experienced ferrite grain size differences between the CSP-HCR and the conventionally produced CCR low carbon strip steels may appear plausible, it needs to be supported by a semi-quantitative model. Firstly, the extension of the original modelling study to include the effects of any retained strain in the austenite as it transforms to ferrite, has been shown not to be a significant factor in any eventual differences in grain size between the two types of products. In these steels, most ferrite grain size models do not take into account the nitrogen in solid solution as it is assumed that it is already tied up in AlN during the austenite-to-ferrite phase transformation\textsuperscript{6,7}. Hence, instead of relying on the empirical models alone, a more fundamental approach, which takes into account the influence of the nitrogen in solid solution on the $A_{3}$ temperatures as well, would be more appropriate. The ferrite grain size $D_{\alpha}$ that forms by heterogeneous nucleation on austenite grain boundaries can be obtained as a function of the nucleation rate $I$ by\textsuperscript{24}:

$$D_{\alpha} = 0.784K_{2}D_{c}^{1/3}I^{1/9}$$

where $D_{c}$ is the austenite grain size and from modelling was found to be approximately the same at the finishing mill heads of both processes, Figure 1. $K_{2}$ is the parabolic rate constant for the thickness of the nucleating ferrite grain and it is also assumed to be the same for both steels from the two processes.

From classical nucleation theory, the nucleation rate $I$ of ferrite grains from austenite, in this case on the run-out table of the hot strip mill, is given by\textsuperscript{25}:

$$I = K_{2}D_{c}^{1/2} \exp \left( -\frac{K_{c}}{A_{c}^{2}kT} \right)$$

where $K_{c}$ is the nucleation site density for ferrite on austenite grain boundaries, which is assumed to be the same for both sets of steels with their similar austenite grain sizes at the point of transformation\textsuperscript{3}. $K_{s}$ is the surface energy term, which is also assumed to be the same for both sets of steels, $k$ is the Boltzmann constant and $D_{c}$ is the diffusivity of carbon in austenite at the transformation temperature $T_{A_{3}}$ and is given by\textsuperscript{26}:

$$D_{c} = 0.5 \exp (-30 C_{e})$$

$$\exp \left( \frac{38300 - 1990000C_{e} + 550000C_{e}^{2}}{19877} \right) \text{ cm s}^{-1} \text{ [5]}$$

Therefore, the ratio of the ferrite grain size $D_{HCR}/D_{CCR}$ for these two sets of steels is given by:

$$\frac{D_{CCR}}{D_{HCR}} = \frac{D_{T_{c}-1}(kT_{c})^{1/2} \exp \left( -\frac{K_{c}}{A_{c}^{2}kT_{c}} \right)}{D_{T_{2}-1}(kT_{c})^{1/2} \exp \left( -\frac{K_{c}}{A_{c}^{2}kT_{c}} \right)}$$

where $T_{2}$ and $T_{c}$ are the $A_{3}$ temperatures in the CCR and CSP-HCR production processes respectively.

The laboratory determined $A_{3}$ temperatures from Verdoorn at $a^{6}$ for these same steels and the ferrite grain size modelling constants from previous workers are given in Table IV. As may be seen from Equation [6], the final ferrite grain size is a function of the $A_{3}$ temperatures, which is directly influenced by the cooling rate and chemical composition of the steel, i.e. the amount of nitrogen in solid solution.

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**Table IV**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>HS130-50 $A_{3}$ (K)</th>
<th>LS2-65 $A_{3}$ (K)</th>
<th>Ref. $A_{3}$ (K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20°C/s</td>
<td>1148</td>
<td>1098</td>
<td>5</td>
</tr>
<tr>
<td>50°C/s</td>
<td>1125</td>
<td>1063</td>
<td></td>
</tr>
<tr>
<td>80°C/s</td>
<td>1048</td>
<td>987</td>
<td></td>
</tr>
<tr>
<td>$\Delta A_{3}$ (J/mol)</td>
<td>-90</td>
<td>-90</td>
<td>27</td>
</tr>
<tr>
<td>$K_{c}$ (J/mol)</td>
<td>$6.33 \times 10^{-15}$</td>
<td>$6.33 \times 10^{-15}$</td>
<td>28</td>
</tr>
</tbody>
</table>
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solution. At a typical run-out-table cooling rate of 80°C s⁻¹, the ratio of $D_{CCR}/D_{HCR}$ from the above equation was found to be 1.35, somewhat closer to the observed 1.35 for the industrially produced steels. The lower ratio in practice could be attributed to the influence of the higher retained strain in the CCR steel, which provides an extra driving force for the nucleation of ferrite and this offsets the ferrite grain growth effect, which arises from the higher austenite to ferrite $A_\gamma$ transformation temperatures, Table V. This confirms that the lower $A_\gamma$ temperatures in the low sulphur low carbon Al-killed strip steels produced in the CSP-HCR plant significantly contribute to the finer ferrite grain size and these grains remain finer during coiling due to the presence of the fine homogeneously nucleated AlN particles. In the higher sulphur content steels produced by the CCR process, the AlN has been found to be very coarse as it forms MnS-AlN compound particles through its heterogeneous nucleation on the MnS particles and, therefore, will not be effective in grain boundary pinning of the ferrite grain boundaries immediately after transformation and during the subsequent coiling process. Hence, some further grain growth may even contribute additionally to a somewhat coarser ferrite grain size in the CCR processed steels.

Conclusions

The following conclusions were drawn from this study:

- AlN dissolves relatively quickly in both low and high sulphur content low carbon Al-killed strip steels during a reheating treatment. In both steels, it was observed that equilibrium was attained after soaking for only 5 minutes.
- The sulphur content does not appear to have any significant effect on the equilibrium solubility of the AlN in austenite of both the low and high sulphur content low carbon Al-killed strip steels. The heterogeneous nucleation of the AlN on MnS in the medium to high sulphur content (≥ 50 ppm) steels opposed to the homogeneous nucleation of the AlN in low sulphur content steels, therefore, does not have any noticeable influence on the equilibrium dissolution of the AlN in these steels upon reheating.
- The TEP-derived AlN solubility equation was found to be

$$\log[\text{Al}][\text{N}] = 2.6 - \frac{9710}{T}$$

which predicts lower solubilities than most other published equations determined either by the Beeghly technique or by purely thermodynamic means.

- Reheating practices in conventional strip producing plants may, therefore, underestimate the solution temperatures of the AlN, likely leading to higher $A_\gamma$ temperatures as a result of less nitrogen in solid solution during the austenite to ferrite phase transformation.
- On the contrary, in CSP-HCR plants that also produce low sulphur content Al-killed strip steels, it appears likely that the nitrogen remains largely in solid solution due to the difficulty in the homogeneous nucleation of the AlN in these steels. This will lead to a significantly lower $A_\gamma$ temperature (i.e. higher undercooling) and a finer ferrite grain size, as was broadly confirmed by modelling the grain size differences between the two sets of steels.
- The presence of a greater level of retained strain in the austenite of the CCR process than in the HCR case, has been shown not to be a significant factor on its own in quantifying the differences in ferrite grain size and, in fact, predicts that the CCR product should have had the finer ferrite grain size and not the HCR product as found in practice. This proves that this factor is not the only determinant in the final ferrite grain size although it appears from the modelling carried out here, that it may play a contributory role to the overriding effect of the nitrogen in solution.

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References


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<td>Comparison of the ratio of $D_{\alpha}-CCR/D_{\alpha}-HCR$ as determined by different models and the actual ferrite grain size ratio</td>
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<td>Empirical (with retained strain)</td>
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5. VIERDORNS, F.A. and STUMPF, W.E. Witwatersrand Metallurgy Conf. 11 and 12 October 2004