Ferrite Formation above the \( \text{Ae}_3 \) Temperature during the Torsion Simulation of Strip Rolling

Clodualdo ARANAS Jr.,* Samuel Filgueiras RODRIGUES, Rupanjit GREWAL and John Joseph JONAS

Department of Materials Engineering, McGill University, Wong Building, 3610 University St., Montreal, H3A 2B2 Canada.

(Received on June 1, 2015; accepted on July 16, 2015)

Torsion simulations of 7-pass strip rolling were carried out on a 0.06%C-0.3%Mn-0.01%Si and a 0.11%C-1.0%Mn-0.11%Si-0.03%Al-0.034%Nb steel using pass strains of 0.4 applied at 1 s\(^{-1}\). The deformations were imposed isothermally at 910°C and 930°C for the C–Mn and the Nb microalloyed steel, respectively. The flow curve levels decreased from pass to pass as a result of softening by both dynamic transformation (DT) and dynamic recrystallization (DRX). The application of double differentiation to the stress-strain curves led to average critical strains for the initiation of DT and DRX of about 0.06 and 0.11, respectively. Optical microscopy revealed that the volume fraction of DT ferrite increased continuously right up to the last pass. The fraction of DT ferrite formed and retained was significantly higher when short interpass times were used. Comparison of the behaviors of the C–Mn and Nb steels indicates that Nb addition retards both the forward as well as the reverse transformation.

KEY WORDS: thermomechanical processing; dynamic transformation; strip rolling; interpass time; torsion testing.

1. Introduction

The formation of ultrafine ferrite by dynamic strain induced transformation (DSIT) attracted considerable interest in the 1980’s and 1990’s.\(^1\) This involves the nucleation of ferrite by deforming austenite between the \( \text{Ae}_3 \) and \( \text{Ar}_3 \) temperatures. Grain sizes of about 1–3 \( \mu \)m were produced in both laboratory and pilot scale rolling.\(^1\)–\(^3\) However, transformation can also be initiated above the \( \text{Ae}_3 \) temperature, as shown by Yada and co-workers in the 1980’s.\(^3\) This is referred to here as dynamic transformation (DT). The microstructures from their hot compression tests and rolling simulations revealed that DT can occur as much as 166°C above the \( \text{Ae}_3 \). They also showed that reverse transformation takes place during isothermal holding after straining. This was followed by \textit{in-situ} x-ray diffraction experiments\(^4\) and dilatometric measurements\(^5\) to provide real-time evidence for DT. Thermodynamic explanations of this unusual phenomenon have been proposed in terms of the stored energy of dislocations,\(^6\) stress activation\(^7\) and transformation softening.\(^8\)

Early strip rolling simulations were mainly focused on the effects of static and dynamic recrystallization on the rolling load.\(^9\)–\(^13\) These studies indicated that short interpass times lead to significant reductions in the mean flow stress (MFS), which were then attributed to the occurrence of dynamic recrystallization. Moreover, rolling simulations performed by Bowden \textit{et al.}\(^14\) on microalloyed steels showed that Nb(C,N) precipitation and solute drag significantly retard both dynamic and static recrystallization. At this time, the dynamic transformation of austenite into the softer ferrite phase was not considered as playing a role.

In an earlier paper,\(^15\) it was shown that DT also contributes to the softening that takes place during rolling. In that work, torsion simulations of strip rolling were carried out on a C–Mn and a Nb microalloyed steel to determine the effect of interpass time on the rolling load. Once again, the flow stress levels and MFS’s decreased when short interpass times were employed. The nucleation and growth of ferrite from the austenite reduced the rolling load and modified the microstructure. Nevertheless, no quantitative information was provided regarding the volume fractions of ferrite being formed during rolling.

In this study, torsion simulations of strip rolling were carried out on a C–Mn and a Nb microalloyed steel and water quenching was performed after selected passes so that the volume fraction of ferrite formed in each pass could be quantified. The results obtained are described in detail below.

2. Experimental Procedure

The steels were supplied in the form of hot rolled plates with thicknesses of 12.5 mm. These were machined into torsion specimens with diameters of 6.3 mm and gauge lengths of 22.2 mm and their axes parallel to the rolling direction. The chemical compositions of the two steels are displayed in Table 1; their paraequilibrium and orthoequilibrium \( \text{Ae}_3 \) temperatures are also shown here. The latter were calculated using the FSstel database of the FactSage thermodynamic software.\(^16\)
2.1. Torsion Testing

Torsion simulations of strip rolling were carried out using a computer-controlled MTS torsion machine equipped with a horizontal radiation furnace and a temperature controller. A thermocouple was welded to the torsion samples to accurately measure the temperatures during deformation. The samples were protected by an argon gas atmosphere so as to minimize the amount of oxidation and decarburization at elevated temperatures. The torque/twist data were converted into equivalent stress/strain form using the Fields and Backofen formula.\(^1\)\(^7\) Corrections were also made for the increase in the sample diameter during free-end torsion testing.\(^1\)\(^8\)

The thermomechanical schedule employed during the rolling simulations is shown in Fig. 1. The samples were heated at 1°C/s from room temperature to 1 200°C. After austenitization for 20 minutes, the samples were cooled at 1°C/s down to the roughing pass temperature of 1 100°C. The temperature was held for 60 s before simulating the roughing pass by applying a strain of 0.4 at the rate of 1 s\(^{-1}\). After this, samples were held for another 90 s to allow recrystallization to take place before cooling at 1°C/s down to test temperatures of 930°C and 910°C for the C–Mn and Nb microalloyed steels, respectively. Seven deformations were applied isothermally with strains of 0.4 applied at 1 s\(^{-1}\) and employing interpass times of 0.5 s and 5 s. Samples were water quenched after selected passes, namely, after the i) 1st, ii) 3rd, iii) 5th, and iv) last passes. This procedure enabled the ferrite volume fraction formed in pairs of passes to be quantified and also led to assessment of the amount of reverse transformation taking place between passes.

2.2. Metallography

For microstructural analysis, the torsion samples were cut perpendicular to the longitudinal axis and rolling direction. These were hot mounted and then polished using silicon carbide papers with grits of 400, 600, 800, 1 000 and 1 200 that were lubricated with water. Diamond (3 μm and 1 μm) and colloidal silica (0.02 μm) suspensions were used for fine and final polishing. The polished samples were etched with a 2% nital solution followed by treatment with a 10% aqueous metabisulfite (Na\(_2\)S\(_2\)O\(_5\)) solution to improve the contrast between martensite and ferrite.

3. Results

3.1. Torsion Simulation of Strip Rolling

In torsion testing, the Fields and Backofen\(^1\)\(^7\) expression is generally employed to convert torque/twist data into equivalent stress/strain curves. For this purpose, average values of the twist hardening exponent (N) are used rather than the instantaneous ones. This method overestimates the critical strains for the initiation of dynamic transformation and dynamic recrystallization, as discussed in Ref. 19. Two stress-strain curves derived from one of the present tests on the Nb steel are presented in Fig. 2. Here both the average N (broken line) and local N (solid line) were used in the conversions. It can be seen that the level of the corrected curve is about 6 MPa below that of the average N curve and that it has a slightly different shape. For the latter reason, only the curves corrected in this way were used to determine the critical strains presented below.

The isothermal flow curves determined from the 7-pass rolling simulation carried out on the C–Mn steel are presented in Fig. 3. Here both interpass times of 0.5 s (Fig. 3(a)) and 5 s (Fig. 3(b)) were employed at 930°C. It should be noted that the simulations were performed isothermally at about 60°C above the paraequilibrium \(\text{Ae}_3\) temperature. No interpass cooling was employed so as to simplify the interpretation of the observed flow stresses. In the absence of a phase change, the flow stress level should not change significantly from pass to pass during deformation at a fixed temperature.

Table 1. Chemical compositions (mass%) and equilibrium transformation temperatures (°C).

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Nb</th>
<th>(\text{Ae}_{3(p)})</th>
<th>(\text{Ae}_{1(p)})</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plain C–Mn</td>
<td>0.06</td>
<td>0.30</td>
<td>0.01</td>
<td>–</td>
<td>870°C</td>
<td>877°C</td>
</tr>
<tr>
<td>Nb microalloyed</td>
<td>0.11</td>
<td>1.0</td>
<td>0.11</td>
<td>0.034</td>
<td>827°C</td>
<td>843°C</td>
</tr>
</tbody>
</table>

Fig. 1. Torsion testing schedules for the simulation of strip rolling. The deformation temperatures were 930°C and 910°C for the C–Mn and Nb steel, respectively, and interpass times of 0.5 s and 5 s were employed.

Fig. 2. Two stress-strain curves derived from the torque/twist data obtained during a torsion test of the present Nb steel. The curve shown as a broken line was derived using the average N while the solid curve was obtained using the local values of N. There appears to be slightly more dynamic softening when the local N is employed and lower values of the critical strains are obtained.
However, here the peak flow stress was reduced by about 25% from the first pass to the final pass when the 0.5 s interpass time was employed and by roughly 15% when the longer pass intervals were employed. These reductions can be directly associated with the volume fractions of ferrite formed and retained from pass to pass, as will be discussed in more detail in the section that follows. This interpretation will be examined again below when the microstructures pertaining to the two simulations are presented.

A progressive decrease in peak stress is also evident in the Nb steel simulations displayed in Fig. 4. These tests were carried out at about 80 °C above the paraequilibrium $A_{e3}$ temperature. Note that the rate of decrease in stress level is markedly higher in the 0.5 s interpass time tests (Fig. 4(a)) than in those involving 5 s interpass times (Fig. 4(b)). The shapes of the flow curves indicate that there is considerably less static recrystallization between passes in the first case, leading to the appreciable retention of work hardening. As a result, the average flow stresses are higher when full recrystallization occurs. High austenite flow stresses in turn promote ferrite formation by transformation softening, so more ferrite formation is expected to occur under the interpass conditions of Fig. 4(a). The resulting higher average flow stresses in the austenite phase are then responsible for the greater rate of ferrite formation in the first case. The gradual decrease in the flow stress levels of the successive pass flow curves can thus be attributed to progressive ferrite formation.

4. Discussion

4.1. Mean Flow Stress

In hot rolling, the evolution of the mean flow stress (MFS) provides evidence for the progress of metallurgical phenomena such as work hardening, recrystallization, precipitation as well as transformation. These events are dependent on the applied strain, strain rate, temperature of deformation, interpass time and alloy composition. In the current study, the MFS behavior provides considerable insight into the volume fraction of DT ferrite formed and retained from pass to pass. For this purpose, the areas under the flow curves of the rolling simulations of Figs. 3 and 4 were calculated; these are presented in Figs. 5(a) and 5(b), respectively. Here the dotted lines refer to the 0.5 s interpass time tests while the solid lines are associated with the 5 s intervals.

It can be seen that the MFS increases somewhat from the first to the second pass for the C–Mn steel (Fig. 5(a)) due to the appreciable retention of work hardening. As a result, the average flow stresses are higher than when full recrystallization occurs. High austenite flow stresses in turn promote ferrite formation by transformation softening, so more ferrite formation is expected to occur under the interpass conditions of Fig. 4(a). The resulting higher average flow stresses in the austenite phase are then responsible for the greater rate of ferrite formation in the first case. The gradual decrease in the flow stress levels of the successive pass flow curves can thus be attributed to progressive ferrite formation.
The presence of Nb in the simulation of Fig. 5(b) delays the occurrence of static recovery and recrystallization. As a result, the amounts of work hardening retained after the first pass are comparable in both the short and long interpass time simulations. This appears to be largely a solute drag effect, as there is insufficient time for any significant amount of niobium precipitation. However, in the succeeding passes, it is evident from the change in the flow curve shape that the sample with the longer times displays significantly more retained hardening than the one with the shorter times. This can be attributed directly to the carbonitride precipitation taking place during this more time-consuming simulation.

4.2. Critical Strains
The stress levels of the flow curves in Figs. 2 and 3 decrease steadily even though the strains were applied at a fixed temperature. To investigate the reason for this unusual behavior, the stress-strain curves were submitted to double differentiation. This was done by fitting 9th order (or higher in some cases) polynomials using the MatLab software. The initiation of softening was then identified with the inflection points in the strain hardening rate versus stress plots. The details of this approach are described in Ref. 20). These calculations confirmed that two distinct softening mechanisms are initiated well before the peak stress is attained. The first of these has been identified as dynamic transformation (DT) and the second as dynamic recrystallization (DRX). As shown in earlier investigations, the critical strain for DT is always lower than that for DRX.

The DT and DRX critical strains determined in the C–Mn simulation steel are illustrated in Figs. 6(a) and 6(b) for interpass times of 0.5 s and 5 s. Critical strains in the Nb steel subjected to interpass times of (c) 0.5 s and (d) 5 s. Here the first pass always displays the highest critical strain in all the simulations.

Fig. 5. MFS’s calculated from the stress-strain curves of Figs. 2 and 3. (a) For the C–Mn steel, the MFS decreases are immediately evident as a result of ferrite formation during each pass. (b) In the Nb steel, however, there is more work hardening retained from pass to pass. Because of carbonitride precipitation during the longer interpass times, there is little decrease in the MFS.

Fig. 6. The critical strains for dynamic transformation (DT) and dynamic recrystallization (DRX) in the C–Mn steel determined by means of double differentiation: (a) 0.5 s and (b) 5 s. Critical strains in the Nb steel subjected to interpass times of (c) 0.5 s and (d) 5 s. Here the first pass always displays the highest critical strain in all the simulations.
first pass DT critical strain is about 0.08 while the critical strains for the succeeding passes (from the 2nd to the 7th pass) have approximately constant values of about $\varepsilon=0.05$. This is because the first pass is applied to undeformed austenite whereas retained work hardening is present in the succeeding passes, reducing the DT critical strain in this way.

A similar plot of critical strain versus pass number associated with the longer interpass times is shown in Fig. 6(b). Here the first pass critical strain agrees with the value presented in Fig. 6(a). However, the DT critical strains calculated for the succeeding passes increase slightly with the pass number. This can be attributed to the greater amounts of recrystallization taking place during the longer interpass times so that the retained austenite gradually approaches the undeformed state of the first pass material more and more closely. As a result, higher strains are required to initiate transformation in the succeeding passes.

A similar double-differentiation procedure was applied to the Nb steel flow curves of Fig. 4. The results obtained are presented in Figs. 6(c) and 6(d) for interpass times of 0.5 s and 5 s, respectively. Here the first pass DT critical strains (about 0.09) are slightly higher than the values for the C–Mn steel. In the succeeding passes, they remain approximately constant at about 0.05. In this particular case, there is very little to no retransformation. For this reason, the amount of ferrite accumulated from pass to pass is expected to be comparable for both the 0.5 s and 5 s interpass times. The microstructures pertaining to the two simulations will be presented in the section that follows.

The initiation of DT and DRX at the double minima was verified by means of both optical and electron microscopy. An example of the microstructure produced in a torsion test carried out to a strain of 0.4 at 930°C and 1 s$^{-1}$ is presented in Fig. 7(a). Here it can be seen that ferrite formation was initiated at strains in the range 0.08 to 0.1, while recrystallization began at higher strains, about 0.15 to 0.2. These values are consistent with the calculated critical strains displayed in Fig. 6. It is important to note that the “initiation” of DT corresponds to the presence of about 5% volume fraction of ferrite. The fresh ferrite formed in this way consists of plates of Widmanstätten ferrite, as illustrated in Fig. 7(b). As these plates are of near identical orientation, they coalesce during continued straining into polygonal grains.

4.3. Volume Fraction of Ferrite Formed Per Pass

The transformation from austenite to ferrite at temperatures above the $\gamma$+$\eta$ involves the displacive conversion of the fcc crystal structure followed by carbon diffusion, as discussed elsewhere. Widmanstätten ferrite plates are nucleated and colonies of these plates coalesce into polygonal grains upon further straining. The volume fraction of DT ferrite depends on the strain, interpass time, chemical composition and temperature. The dilatation associated with ferrite formation increases the volume flow rate (but not the mass flow rate) as a bar passes through a strip mill. These changes can lead to shape control problems if not properly taken into account.

The MFS’s associated with the strip rolling simulations described above indicated that significant amounts of ferrite formed and were retained during the schedule and that the volume fraction of ferrite increased continuously right up to the last pass. Some of the optical microstructures related to the 0.5 s interpass time C–Mn simulation are presented in Fig. 8. Here the martensite (prior austenite) appears dark while the ferrite is light. It can be seen that the volume fraction of ferrite present after the 1st pass (Fig. 8(a)) is about 15%, and that it is largely of plate form. These plates coalesced into polygonal grains in the succeeding passes, as shown in Fig. 8(b) (after the 3rd pass), Fig. 8(c) (after the 5th pass) and Fig. 8(d) (after the 7th pass). Here the ferrite fractions increased to about 56%, 65% and 72%, respectively.

These microstructures reveal that the volume fraction of ferrite increased progressively with pass number (i.e. with strain) when the 0.5 s interpass times were employed. The microstructures associated with the 5 s interpass time simulation are illustrated in Fig. 9. In this case, the volume fractions of ferrite after the 3rd (Fig. 9(a)), 5th (Fig. 9(b)) and 7th (Fig. 9(c)) passes are about 48%, 54% and 52%, respectively. These values are significantly lower than those associated with the shorter times. This can be attributed to the retransformation that takes place between passes, resulting
in the retention of reduced amounts of DT. In this example, about 20% of the ferrite retransformed back into austenite when the interpass times were increased from 0.5 s to 5 s.

The microstructures related to the Nb steel simulation with interpass times of 0.5 s are illustrated in Fig. 10. The presence of significant amounts of ferrite, about 25%, is evident in the 1st pass microstructure (Fig. 10(a)), in which both polygonal grains and ferrite plates can be seen. During the succeeding passes, the volume fraction of ferrite increases to 30%, 39%, and finally to 51% after the 3rd (Fig. 10(b)), 5th (Fig. 10(c)) and 7th (Fig. 10(d)) passes, respectively. The ferrite is mostly present as polygonal ferrite grains, which have nucleated both along the grain boundaries and within the prior austenite grains.

The microstructures pertaining to the Nb steel simulation with interpass times of 5 s are illustrated in Fig. 11. Here the volume fractions of ferrite are about 32%, 40% and 49% after the 3rd (Fig. 11(a)),

Fig. 8. Optical microstructures of the C–Mn steel subjected to the 7-pass simulation at 930°C with interpass times of 0.5 s. The samples were quenched immediately after the: (a) first pass, (b) 3rd pass, (c) 5th pass, and (d) final pass. Dark regions are martensite (prior austenite) while the light regions are ferrite. The amount of ferrite increases progressively right up to the last pass.

Fig. 9. Optical microstructures of the C–Mn steel subjected to the 7-pass simulation at 930°C with interpass times of 5 s. The samples were quenched immediately after the: (a) 3rd pass, (b) 5th pass, and (c) final pass. Dark regions are martensite (prior austenite) while the light regions are ferrite. There is less ferrite than in the short time simulations because of reverse transformation.
formation in the Nb steel even when the interpass time is increased from 0.5 s to 5 s. In this case, it is probably Nb solute drag that prevents the reverse transformation.\textsuperscript{15) As the nature of the latter is diffusional rather than displacive,\textsuperscript{24) the addition of Nb can have a larger effect on the kinetics than during the forward transformation. The current results thus support those of an earlier study\textsuperscript{26) regarding another Nb steel, in which retransformation was delayed by up to 200 s.}

The ferrite volume fractions produced in the C–Mn and Nb steels are summarized in Figs. 12(a) and 12(b), respectively. Here the Vickers hardness measurements pertaining to each pass of the 0.5 s simulation have been added and are

5th (Fig. 11(b)) and 7th (Fig. 11(c)) passes, respectively. These values are similar to those associated with the shorter interpass times. It thus appears that there is little retransformation in the Nb steel even when the interpass time is increased from 0.5 s to 5 s. In this case, it is probably Nb solute drag that prevents the reverse transformation.\textsuperscript{15) As the nature of the latter is diffusional rather than displacive,\textsuperscript{24) the addition of Nb can have a larger effect on the kinetics than during the forward transformation. The current results thus support those of an earlier study\textsuperscript{26) regarding another Nb steel, in which retransformation was delayed by up to 200 s.}

The ferrite volume fractions produced in the C–Mn and Nb steels are summarized in Figs. 12(a) and 12(b), respectively. Here the Vickers hardness measurements pertaining to each pass of the 0.5 s simulation have been added and are
displayed on the right-hand axes. In Fig. 12(a), the increases in the amount of ferrite are accompanied by corresponding decreases in the overall hardness, from 259 HV before the first pass down to about 175 HV immediately after the final pass. Note that since the C–Mn steel only contains about 0.06 wt% C, the martensite hardness falls in the expected 200 to 300 HV range.27 However, the hardness of the martensite in the Nb steel (with 0.11%wt C) is somewhat higher, as shown in Fig. 12(b). Here the measured hardness before the initial pass is 341 HV and the presence of ferrite introduced by the deformation lowers it to about 273 HV.

As a check, hardnesses of local ferrite and martensite regions were also measured, as illustrated in Fig. 13. The Vickers microhardness of the ferrite in the C–Mn steel is approximately 158 HV (Fig. 13(a)) while that of the martensite is 264 HV (Fig. 13(a)). These values are in agreement with the macrohardnesses shown above.

4.4. Effect of Nb Addition on the Behavior

The amounts of ferrite formed in the C–Mn and Nb steels during the rolling simulations are presented in Figs. 14(a)
and 14(b). During the 0.5 s simulations (Fig. 14(a)), more ferrite was produced in the C–Mn steel than in the microalloyed steel. This amounted to about 20% more ferrite in the C–Mn than in the Nb steel. These results confirmed that the addition of Nb hinders the forward displacive transformation and not just the reverse diffusional transformation. These observations complement the higher measured DT critical strains in the Nb steel than in the C–Mn steel. Given that Si addition decreases the driving force for transformation softening, it is possible that Nb has a similar effect. This would explain why less ferrite is formed in the Nb steel on the application of a given strain than in the C–Mn steel.

The situation is somewhat more complicated in the case of the 5 s simulations (Fig. 14(b)). As shown above, the retransformation of ferrite back into austenite in the C–Mn steel lowers the ferrite volume fraction by about 20% because of the longer times available for this to occur (see Fig. 12(a)). However, in the Nb steel, no retransformation was observed during the 5 s interpass times. Although the presence of Nb retards the production of DT ferrite, it also retards the reverse transformation. In this way, comparable amounts of ferrite were present in the final pass in the two steels.

5. Conclusions

1. The strip rolling simulations carried out on the C–Mn steel revealed that ferrite volume fractions of about 10% form during each $\varepsilon=0.4$ pass, with the volume fraction increasing continuously until the last pass. Concurrent decreases in flow stress level and MFS were also observed during these simulations. The view that the ferrite forms dynamically is supported by the extensive optical microscopy results, which were accompanied by hardness measurements.

2. The strip rolling simulations carried out on the Nb steel revealed that ferrite volume fractions of about 7% form during each $\varepsilon=0.4$ pass, with the volume fraction increasing continuously until the last pass. Concurrent decreases in flow stress level and MFS were also observed during these simulations. Extensive optical microscopy results again supported the conclusion that the ferrite formed dynamically. Comparison of the behaviors of the C–Mn and Nb steels during the short interpass time simulations indicates that more ferrite forms in the C–Mn steel than in the Nb steel because Nb addition impedes the forward transformation.

3. Critical strain measurements provided further insight into the forward transformation. The DT critical strains are always highest in the first pass and then adopt lower values in the succeeding passes because of the retention of work hardening.

4. The fraction of DT ferrite formed and retained in the C–Mn simulations is significantly higher when short interpass times are employed. Here the metastable DT ferrite retransforms back into the more stable austenite during the longer intervals. This led to about a 20% volume fraction decrease when the interval was increased from 0.5 s to 5 s.

5. The addition of Nb impedes the displacive forward transformation and retards the diffusional reverse transformation rates. The former occurs because Nb addition reduces the effectiveness of transformation softening; the latter by means of a solute drag effect.

Acknowledgements

The authors acknowledge with gratitude funding received from the McGill Engineering Doctoral Award (MEDA) program and the Natural Sciences and Engineering Research Council of Canada.

REFERENCES