Understanding the static recrystallization of Ni-Mo alloyed steels for rolling optimization in high strength thick plates

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Abstract. The development of high strength/high toughness steel grades for thick plates requires an optimum combination of alloy design and processing conditions. In this contribution, the effect that Molybdenum and Nickel have on the static recrystallization kinetics during roughing passes is analyzed. High alloying additions, especially in the case of Mo, imply intense solute drag delay for static recrystallization. This mechanism, added to the low reductions typically applied during roughing of thick plates, reduces the possibility for a complete recrystallization between passes and limit austenite conditioning and grain size refinement. Under these circumstances, the understanding and modelling of static recrystallization becomes relevant. Hot torsion tests were carried out and the effect of alloying elements on the recrystallization kinetics and recrystallized grain size was quantified in four different steel grades with several Molybdenum (0.25-0.5%) and Nickel combinations (0-0.5%). Finally, the softening behavior and microstructural evolution during multipass roughing rolling simulations was compared with MicroSim® model predictions, showing a reasonable agreement with experimental results.

1. Introduction
The demanding market requirements for applications such as super heavy lift cranes, offshore cranes, ships and constructions, requires the development of innovative thermomechanical processing routes in addition to an optimum alloy design. These high strength/high toughness combination requirements are especially challenging as the thickness of the plates (80 mm and above) increases. The definition of a suitable hot rolling sequence becomes essential [1,2] to ensure homogeneous microstructures through thickness and avoid heterogeneities in the center of the plate. Thermomechanical processing must be carefully designed to obtain an optimum grain size that ensures a good balance between tensile and toughness properties. For the final application, thick plates are usually produced by conventional quenching (CQ) followed by tempering. Even when CQ is applied, the effect of the pre-austenite conditioning during rolling on the final mechanical properties and through-thickness homogeneity is a very important factor, and therefore, the microstructural evolution must be controlled and well understood.

The addition of high Mo contents becomes crucial in order to avoid microstructural gradients through thickness and the presence of softer phases (ferritic regions) in the centerline of the plate [3]. However, for very thick plates, to increase hardenability and ensure good toughness properties, Mo addition is not sufficient and additional alloying elements such as Ni are required [4]. It is widely known that the addition of Mo retards static recrystallization due to solute drag effect [5]. When Ni is alloyed to austenitic steel it increases the stacking fault energy (SFE) of the iron matrix [6]. The level of SFE has an impact on the deformation characteristics of the material such as the work-hardening and softening behavior. The increase of the SFE by nickel alloying and the correspondingly improved dislocation...
mobility should promote recovery as softening mechanism during hot deformation. However, the overall recrystallization kinetics of the steel is not noticeably changed by nickel. The combined effect of Mo and Ni alloying on recrystallization kinetics though, remains unclear for the composition ranges in these applications. Moreover, in the production of thick plates, the low reductions applied during roughing step impedes full recrystallization between passes, limiting the austenite conditioning and microstructural refinement. In addition to the static recrystallization kinetics, the austenite microstructure evolution during hot deformation was evaluated in four different steel grades with different Mo (0.25-0.5%) and Ni combinations (0-0.5%). Furthermore, the applicability of available microstructural evolution models was analyzed for these alloy concepts and the applied low strain conditions using MicroSim® model predictions.

2. Experimental procedure

Table 1 summarizes the chemical compositions of the steels studied in the current paper. Four different laboratory melts with two different levels of Mo (0.25-0.5%) and Ni (0-0.5%) were casted.

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Mo</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>LowMo</td>
<td>0.14</td>
<td>0.28</td>
<td>1.3</td>
<td>0.24</td>
<td>0.07</td>
</tr>
<tr>
<td>HighMo</td>
<td>0.15</td>
<td>0.3</td>
<td>1.3</td>
<td>0.50</td>
<td>0.03</td>
</tr>
<tr>
<td>LowMoNi</td>
<td>0.15</td>
<td>0.3</td>
<td>1.3</td>
<td>0.24</td>
<td>0.52</td>
</tr>
<tr>
<td>HighMoNi</td>
<td>0.14</td>
<td>0.34</td>
<td>1.3</td>
<td>0.5</td>
<td>0.52</td>
</tr>
</tbody>
</table>

Torsion tests were designed to analyze the hot deformation and austenite evolution behavior. The torsion samples had a central gauge section of 17 mm in length and 7.5 mm in diameter. To define the static recrystallization kinetics, double pass torsion tests were performed, whereas for simulating different roughing sequences, multipass torsion tests were carried out. A schematic representation of the designed thermomechanical cycles is shown in Figure 1 and figure 2. Concerning the double pass torsion tests, in most of the cases, a reheating treatment at 1150 ºC for 10 minutes was applied, followed by a refining deformation step to ensure homogeneous and fine austenitic structure prior to double pass deformation. Different refining step was applied depending on the alloy-concept. To avoid the presence of coarse austenite grains prior to double pass deformation, as illustrated in figure 1a, for the steels containing Mo (Low Mo, High Mo and HighMoNi), two deformation passes of 0.3 are required at 1150 ºC. However, for LowMoNi steel (see figure 1b), two additional refining deformation passes are needed to avoid austenite heterogeneity. After refining deformation step, the torsion specimens were cooled down to the deformation temperature in the interval between 1100 and 900 ºC and a strain of 0.2 was applied. The fractional softening was defined assuming the 2% offset method, which is reported as the procedure that most accurately eliminates the effect of recovery in the absence of strain induced precipitation [7]. For the HighMoNi steel, deformation temperature range was extended to higher deformation temperatures (1250, 1150 and 1100 ºC). Furthermore, for all the chemical compositions, the specimens were quenched before the double pass deformation torsion tests and the initial austenite grain sizes (D₀) were quantified in all cases. To reveal the previous austenite grain boundaries in the quenched samples, picric acid etching was employed and the austenite is characterized by optical microscopy. The austenite grain size measurements were carried out in terms of the mean equivalent diameter.
In order to reproduce roughing sequences and to analyze the recrystallization kinetics during roughing step, two different multipass torsion tests were conducted, as presented in figure 2. The Constant Strain cycle (see figure 2a) contains 8 deformation passes of 0.15. In the Variable Strain cycle, after reheating treatment, 2 deformation passes of 0.1 and 0.15 were applied, followed by only one deformation pass of 0.2, 0.25 and 0.3 (in total, 7 deformation passes). An interpass time of 8 s was used in both sequences and three different deformation temperatures of 1250, 1150 and 1100 °C were applied. In all cases, the austenitic structure after roughing step was characterized and to that end, the samples have been quenched after roughing cycle.

![Figure 1](image1.png)

**Figure 1.** Schematics of the thermomechanical cycles applied for the definition of recrystallization kinetics for the different steels: (a) LowMo, HighMo, HighMoNi and (b) LowMoNi.

In order to fit experimental data and define the fractional softening curve, Avrami-type approach is considered:

\[
X_{REX} = 1 - \exp \left[ -0.693 \left( \frac{t}{t_{0.5}} \right)^n \right]
\]

where \(X_{REX}\) is the recrystallized fraction corresponding to an interpass time \(t\), \(t_{0.5}\) is the time to reach a 50% recrystallized volume and \(n\) is the Avrami exponent. As clearly observed in figure 3a-d, the reduction of deformation temperature (from 1100 °C to 900 °C) shifts the softening curves to longer times. For the HighMoNi steel and the highest deformation temperatures (between 1250 °C and

![Figure 2](image2.png)

**Figure 2.** Schematics of the thermomechanical cycles applied for the simulation of different roughing strategies: (a) Constant Strain cycle and (b) Variable Strain cycle.

3. **Results and discussion**

3.1. **Analysis of recrystallization kinetics**

In figure 3a,b,c and d, the impact of deformation temperature on fractional softening curves can be evaluated for each steel grade, respectively. In order to fit experimental data and define the fractional softening curve, Avrami-type approach is considered:

\[
X_{REX} = 1 - \exp \left[ -0.693 \left( \frac{t}{t_{0.5}} \right)^n \right]
\]
1150 °C), very short times are needed to complete recrystallization. Concerning the effect of chemical composition, in figure 3(e), the fractional softening curves corresponding to the deformation temperature of 1100 °C and different alloy-concepts are plotted together. The fastest softening kinetic corresponds to Low Mo steel and the longest times are measured for LowMoNi alloyed grade. However, HighMo and HighMoNi steels show very similar behavior. Reducing the deformation temperature to 1000 and 900 °C, the effect of chemical composition is less evident and similar curves are obtained in the different steels. From the softening curves shown in figure 3, the time for 50% of recrystallized fraction \( t_{0.5} \) and the Avrami exponent \( n \) were estimated for the entire range of deformation temperature and alloys. In addition, the initial mean austenite grain sizes \( D_0 \) were also measured. Regarding the initial mean austenite grain sizes \( D_0 \), no considerable differences are noticed depending on the steel and reheating treatment (mean austenite sizes between 29.9 and 47.2 μm are quantified). Values for the Avrami exponent \( n \) are in the 0.4-1.1 range.

![Figure 3](image)

**Figure 3.** (a,b,c,d) Effect of deformation temperature on fractional softening curves for the different steels (LowMo, HighMo, LowMoNi and HighMoNi, respectively). (e) Impact of chemistry on recrystallization kinetics at 1100 °C.

The time for 50% of recrystallization, \( t_{0.5} \), depends on the initial austenite size \( D_0 \), deformation conditions (temperature, strain, and strain rate) and the microalloying content in solid solution. Several empirical equations for predicting \( t_{0.5} \) can be found in the literature. In the current study, the one proposed by Fernandez et al. [9] and assuming the solute drag effect established by Jonas [10] was considered:

\[
t_{0.5} = 9.92 \times 10^{-11} \cdot D_0 \cdot \varepsilon^5 \cdot 6.6^{0.15} \cdot 0.53 \cdot \exp \left( \frac{1800000}{RT} \right) \cdot \exp \left( \frac{275000}{T} \right) \cdot 185 \cdot [Nb + 0.0936Mo]
\]  \( (2) \)

In figure 4 the experimental \( t_{0.5} \) times are plotted against the predicted values using equation (2). The results suggest that reasonable prediction could be achieved in most of the cases taking into account this approach. In a recent work, and considering this approach, good correlation is also obtained with high Mo and boron microalloyed steels [5]. However, for the LowMoNi steel, the predictions underestimate the experimental measurements to a higher degree. As reported in [4], nickel might have an effect increasing restoration kinetics in austenite due to its higher SFE, and this would retard further the
recrystallization kinetics. Additional strain relaxation tests will be performed in the near future in order to confirm this interaction.

![Graph showing comparison between experimental and predicted $t_{0.5}$ values]

**Figure 4.** Comparison between experimental and predicted $t_{0.5}$ values by equation (2).

3.2. Simulation of hot rolling by multipass torsion tests

In figure 5 the influence of deformation temperature on recrystallized evolution can be evaluated for both sequences for the HighMoNi steel. For the determination of the fractional softening, anisothermal interpass conditions were considered, assuming the approach proposed by Liu and Akben [11]. For 1150 and 1250 °C, partial recrystallization is achieved and alternate softening is detected every two passes. For the lowest deformation temperature of 1100 °C, the recrystallized fraction increases gradually from pass to pass. Concerning the effect of deformation temperature, as expected, for the highest deformation temperature, higher recrystallization fractions are achieved after the last deformation pass, for both thermomechanical cycles. In terms of the effect of roughing strategy, slightly higher recrystallization fraction is obtained at the last deformation pass for the Variable Strain Cycle.

![Graph showing recrystallized fraction from pass to pass]

**Figure 5.** Recrystallized fraction from pass to pass (calculated from multipass torsion tests) for the different deformation temperatures and both thermomechanical cycles: (a) Constant Strain sequence and (b) Variable Strain sequence.

Additionally, as shown in figure 6, the austenitic structures after roughing were characterized and the austenite grain size distributions were quantified for the different sequences and deformation temperatures. In figure 7a the measured austenite grain size distributions (in terms of accumulated area...
fraction) have been plotted together. At 1100 and 1150 ºC, very similar austenite size distributions are obtained following both cycles (~20 and ~32 µm, for 1100 and 1150 ºC, respectively). At 1250 ºC, on the contrary, slight differences are noticed depending on the sequence. Slightly finer distribution is obtained for the cycle containing 8 deformation passes of 0.15. The impact of deformation temperature and roughing strategy on austenite grain sizes can be analyzed in figure 7b. At 1250 ºC, mean austenite sizes of 56.4 and 44.2 µm are measured, for Variable Strain and Constant Strain schedules, respectively.

![Figure 6](image-url)

**Figure 6.** Austenitic structure after the different roughing schedules (Variable Strain cycle and Constant Strain cycle) and the different deformation temperatures: (a,d) 1100 ºC, (b,e) 1150 ºC and (c,f) 1250 ºC.

![Figure 7](image-url)

**Figure 7.** (a) Comparison between austenite grain size distributions obtained in the entire range of deformation temperatures and both roughing strategies. (b) Effect of deformation temperature and cycle on mean austenite sizes.

Finally, MicroSim-PM model was employed for predicting Fractional Softening (FS) during plate hot rolling simulation and the predictions have been compared with experimental data. This plate mill
model is able to predict the evolution of austenite conditioning and can be a very useful tool for optimizing the hot rolling schedule and alloy design. In figure 8a, the comparison between predicted recrystallized fractions and experimentally measured values are shown for 1150 ºC deformation temperature and both schedules. Comparing the experimental data to the MicroSim-PM simulation results, the model correctly predicts the effect of thermomechanical cycle on the evolution of the austenite fractional softening along the pass schedule. In figure 8b and c, measured austenite thickness distributions after roughing (at the exit of last deformation) have been plotted for both rolling strategies and 1150 ºC, with the predicted ones by the model. Satisfactory correlation between experimental and predicted austenite thickness distributions is noticed for both roughing schedules.

![Figure 8](image.png)

**Figure 8.** Comparison between (a) predicted recrystallized fractions and (b,c) austenite thickness distributions after roughing sequence by MicroSim-PM model at 1150ºC and experimentally measured values.

4. Conclusions
Concerning recrystallization kinetics (double hit torsion tests), the addition of Mo and Ni, as well as decreasing deformation temperature promote the delaying of softening kinetics. Reasonable correlation between experimental and predicted t0.5 are observed for the LowMo, HighMo and HighMoNi steels. For the LowMoNi steel grade, slightly longer experimental t0.5 values are measured compared to the predicted ones.

Regarding the simulations of roughing schedules, for both multipass sequences and entire range of deformation temperatures, partial recrystallization was detected. Regarding the austenitic structure after roughing sequence, for 1100 and 1150 ºC deformation temperatures, similar mean austenite sizes were
measured for both cycles (~20 and ~32 µm, for 1100 and 1150 °C, respectively). However, for the deformation temperature of 1250 °C, slightly finer mean austenite size was quantified for Constant Strain sequence compared to Variable Strain cycle. MicroSim-PM software was used to reproduce the microstructural evolution during these sequences. It is observed that for 1150 °C, MicroSim predicts reasonably well the fractional softening curves and the austenite grain size distributions.

Acknowledgments
The authors would like to acknowledge the International Molybdenum Association (IMOA) and the Nickel Institute for funding this project.

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