TOUGHNESS CONTROL THROUGH MICROSTRUCTURAL EVOLUTION DURING NORMALIZING ROLLING OF NIOBIUM MICROALLOYED STEELS*

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Abstract
Normalizing rolling (NR) requires a finishing rolling temperature range between 880°C and 920°C, of the same order as the austenitizing temperature used in the normalizing heat treatment furnace. It is very common to have partial austenite recrystallization between rolling passes in this temperature range during the normalizing rolling of low carbon structural steels microalloyed with niobium, a situation where the toughness of the product can be impaired. So, it is necessary to determine optimized NR process conditions and always comply with them. The use of a microstructural evolution tool as MicroSim®, which predicts the distribution of austenite grain size along hot rolling, can be extremely useful to reach such aim. This paper describes the use of such tool to correlate NR process conditions with plate toughness in some industrial cases. In one case MicroSim® predictions were checked with grain size distributions determined by EBSD analysis. Very satisfactory results were got, which indicated several rules to be followed to achieve good toughness results in NR plates of low carbon structural steels microalloyed with niobium.
Keywords: Normalizing rolling; Niobium; Plate Mill; Microstructural evolution.

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1 INTRODUCTION

In the past some grades of structural steel plates required an additional normalizing heat treatment to get the specified mechanical properties. Nowadays such plates have a microalloyed steel design and are processed according to a temperature-controlled hot rolling schedule, with finishing rolling temperature in the same range of the austenitizing temperature used in the former normalizing heat treatment. In this way, the plate shows a normalized condition directly from the rolling heat, allowing the suppression of the additional normalizing heat treatment. This specific process is the so-called Normalizing Rolling (NR) [1].

Usually, such NR plates are made of NbTi or NbTiV microalloyed steels. The finishing temperature range of normalizing rolling extends from 880°C to 920°C. As Nb content in such plates varies between 0.015 and 0.020%, this implies that partial austenite recrystallization will occur between the finishing stage passes, that is, a case of Type III Recrystallization. So, there is some decrease in the microstructural homogeneity which, in some critical cases, can impair plate toughness. Therefore, normalizing rolling must be carried out under strict and optimized process conditions to minimize toughness loss [1].

The objective of this work was to identify the reasons behind these toughness variations which, despite not disqualifying the product, indicated that process control must be improved. Therefore, microstructural evolutions during the NR of heavy plates with high and fair toughness were calculated and the comparison between them could provide hints about the causes of such differences. In some cases, the calculated results were checked by comparison with the grain size distribution of the final microstructure obtained through EBSD analysis.

2 DEVELOPMENT

Two cases were studied in this paper. The first one refers to four 10 mm heavy plates that were normalized rolled to meet the requirements of the EN 10025-2 S355 J2 standard. The nominal chemical composition of these plates was 0.16% C, 1.38% Mn, 0.19% Si and 0.090% Nb+Ti+V. As these plates were relatively light, there was no need to include a holding stage between the roughing and finishing stages of NR to reach a finish rolling temperature within the austenite range, according to the value of Ar_3 temperature calculated by the Ouchi equation [2].

The second case considered 15- and 30-mm gauge NR heavy plates, also attending the EN 10025-2 S355 J2 standard and with the same chemical composition of the former case. As plates in this case were thicker, a holding stage was included between the roughing and finishing stages to reach the specified value of finishing rolling temperature.

Charpy tests at -20°C were performed using 10 mm x 10 mm specimens (8,5 mm x 10 mm in the case of 10 mm plates) with a V-shaped notch machined in the longitudinal direction of the rolled plates. The microstructural evolutions occurred during the NR were calculated for both cases using the MicroSim® computer program, developed by Centro de Estudios e Investigaciones Técnicas de Gipuzkoa – CEIT, in Donostia-San Sebastian, Spain, under the sponsorship of the Companhia Brasileira de Metalurgia e Mineração - CBMM [3]. This software was previously used in a similar analysis carried out at Gerdau Ouro Branco [1], but the version used in this work was upgraded, as it was customized for use with the specific operation conditions of Gerdau plate mill. The grain size distribution of the final microstructure of the plates studied in the second case were additionally determined through EBSD at CEIT.

2.1 10 mm NR Plates

The four 10 mm gauge heavy plates studied in this case showed mixed toughness results expressed as mean absorbed energy values, as can be seen below:

- Maximum Toughness (Max): 96 J @ -20°C
- Intermediate Toughness #1 (Int #1): 58 J @ -20°C
- Intermediate Toughness #2 (Int #2): 52 J @ -20°C
- Minimum Toughness (Min): 30 J @ -20°C

Figure 1 shows the evolution of plate rolling pass schedules for these four cases, expressed in a generic graphic of percentual reduction versus temperature. The lines corresponding to the plates with maximum and minimum toughness create an envelope of rolling conditions. The rolling conditions corresponding to the so-called intermediate toughness cases fall inside this envelope.

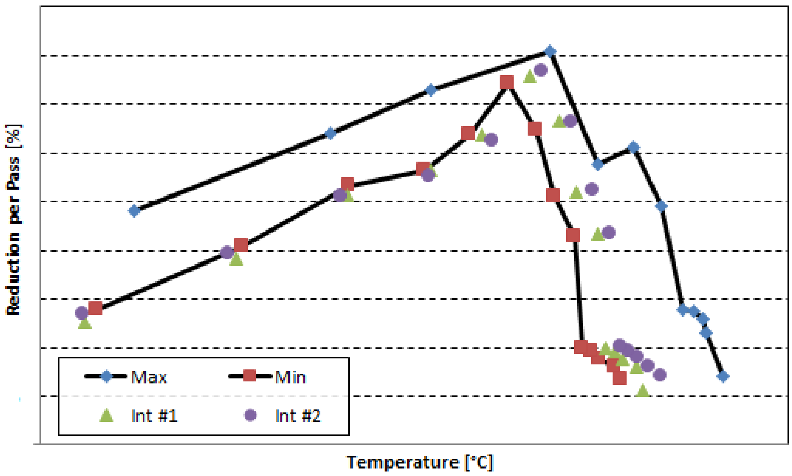
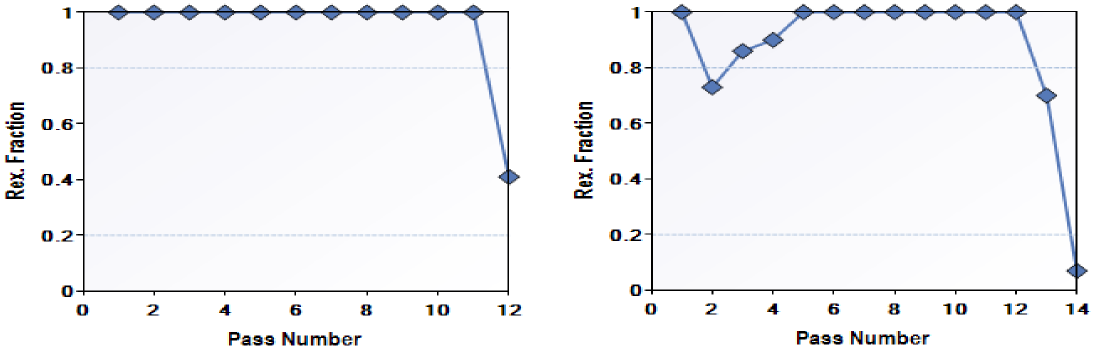


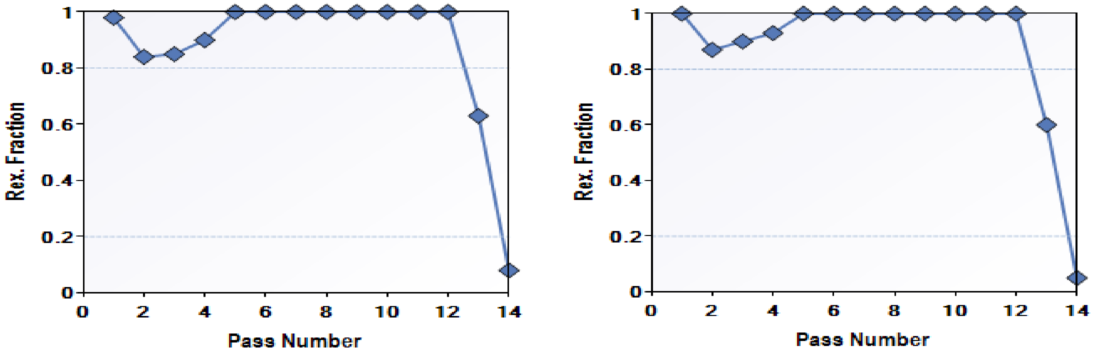
Figure 1. Generic evolution of percentual reduction per pass versus temperature in the plate mill pass schedules of the 10 mm NR plates studied in this work.

The plate with maximum toughness (Max) also showed maximum width (3.088 mm), while all the other have widths between 2610 and 2650 mm. Despite presenting different broadening ratios, the number of passes in the broadsizing stage was the same for all cases. Therefore, thickness reduction per pass during the broadsizing stage was smaller for the narrower materials. To complicate the situation, the narrower (and with lower toughness) plates were rolled with a greater number of passes (14) than the wider one (11). Besides that, the rolling temperatures in the narrower plates were lower than the observed in the wider material, as their slab discharge temperatures were correspondingly lower.
Figure 2 shows the consequences of these different processing conditions of the plates over microstructure evolution. The maximum toughness plate (figure 2a) showed full recrystallization (Type I) between rolling passes, except in the last pass, when rolling temperature was below the recrystallization limit temperature (RLT). This is because the temperatures and reductions applied in almost all passes were high enough to promote recrystallization kinetics. On the other hand, the minimum toughness plate (figure 2b) showed partial recrystallization (Type III) after the first passes of the

schedule, that is, during broadsizing stage, as well as in the last two passes. Lower rolling temperatures and lighter passes can explain such behavior. The intermediate toughness plates showed similar tendency, but recrystallization fractions were greater after broadsizing passes than in the previous case.



(a) Maximum Toughness (b) Minimum Toughness

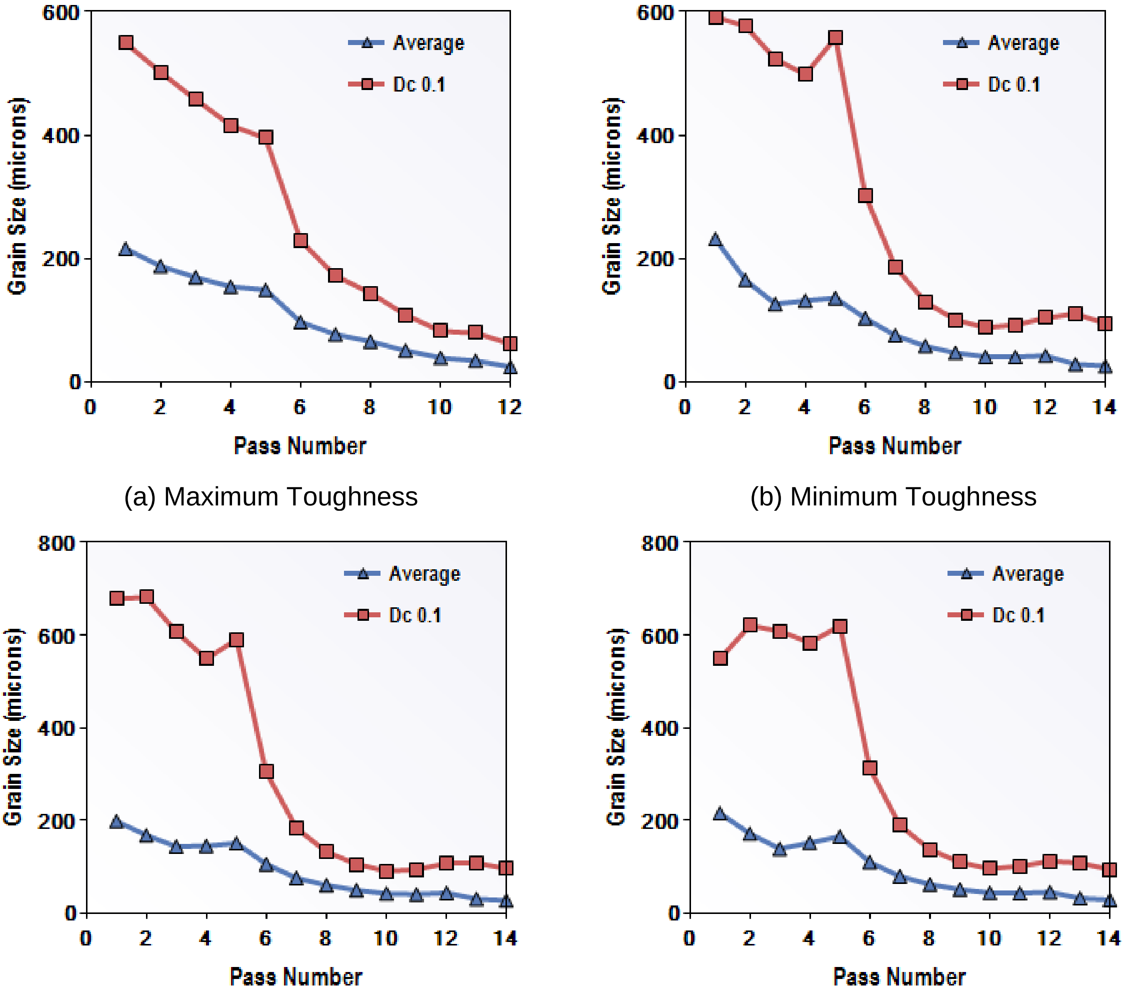


(c) Intermediate Toughness #1 (d) Intermediate Toughness #2

Figure 2. Recrystallized fraction of austenite along the pass schedules of the 10 mm NR plates studied in this paper, as calculated by MicroSim®.

The consequences on the uniformity of the grain size distribution resulting from the occurrence of partial recrystallizations between passes, especially during the broadsizing step, can be seen in figure 3, which shows the evolution of the average and $Dc_{0.1}$ (value of grain size for which at least 10% of the volume fraction of grains have a greater size than that) grain sizes. This last parameter is a measure of microstructure uniformity. The maximum toughness plate showed a continuous refining of the austenitic grain size along pass schedule. For its turn, the minimum toughness plate showed a discontinuous average grain size refining, and its $Dc_{0.1}$ parameter even increased in the final of the roughing stage, as well in the last rolling passes, although in a lighter way. It is interesting to note that both intermediate toughness plates showed similar evolutions as the minimum toughness plate case, but the increases in $Dc_{0.1}$ were not so high in these cases.

A summary of the results obtained from the MicroSim® simulations can be seen in Table 1. As expected, the plate with maximum toughness had lowest values austenite and ferrite mean grain sizes, as well as austenite maximum grain size. Besides that, it showed lowest values of quantitative parameters that express austenite microstructural uniformity, that is, $Dc_{0.1}$ and Zd, which values are inversely proportional to grain size dispersion. For their turn, all other plates, with minimum or intermediate toughness, showed higher values of the mentioned microstructural parameters. Specially the values of austenite maximum grain size, $Dc_{0.1}$ and Zd were far higher than those observed for the plate with maximum toughness. The values of accumulated strain were relatively low and similar for all plates studied here.



(a) Maximum Toughness

(b) Minimum Toughness

(c) Intermediate Toughness #1

(d) Intermediate Toughness #2

Figure 3. Average and $Dc_{0.1}$ grain size of austenite along the pass schedules of the 10 mm NR plates studied in this paper, as calculated by MicroSim ®.

Table 1. Final austenite grain size parameters for the 10 mm NR plates studied in this paper, as calculated by MicroSim®

Plate	Mean GS [μm]	Max GS [μm]	$Dc_{0.1}$ [μm]	Zd	ε_{ACC}	Ferrite GS [μm]
Maximum Toughness	24.5	151.8	61.9	2.53	0.20	12.8
Minimum Toughness	26.1	237.5	94.9	3.64	0.26	13.4
Intermediate Toughness #1	27.9	293.9	96.4	3.46	0.26	13.6
Intermediate Toughness #2	29.1	240.9	94.3	3.24	0.32	13.6

GS: Grain Size; ZD = $Dc_{0.1}$/Mean GS; ε_{ACC}: Accumulated Strain; Ferrite Grain Size calculated according to [4]

The correlation between the results obtained in terms of performance and the parameters calculated by MicroSim® seems to indicate that the full and successive recrystallizations between the roughing passes are vital to obtain high and consistent toughness levels in NR plates. As a matter of fact, the roughing stage of the plate with maximum toughness was characterized by the application of passes with greater reductions and temperatures, which accelerated recrystallization kinetics.

So, to obtain high and consistent toughness in such NR plates, it is recommended to use a higher slab discharging temperature and broadsizing stage with maximum reduction per pass. Slab thickness and width must be the standardized according to the corresponding plate dimensions to avoid different broadsizing ratios and different pass schedules for similar product grades with similar dimensions.

2.2 15 and 30 mm NR plates

Two pairs of plates, 15 and 30 mm, were studied here, each pair with one plate with good and the other with fair toughness:

- 15 mm: 186 J @ -20°C and 30 J @ -20°C
- 30 mm: 141 J @ -20°C and 40 J @ -20°C

Figure 4 shows the evolution of plate rolling pass schedules for the two 15 mm plates, expressed in a generic graphic of percentual reduction versus temperature. It is interesting to note in this figure that the broadsizing stage of the 15 mm NR plate with good toughness was carried out with lighter passes than the lower toughness plate, despite its start rolling temperature being much higher than that of the plate with poor toughness. It must be noted that the width of both plates was the same (2,615 mm). Finishing stage was almost similar for both plates.

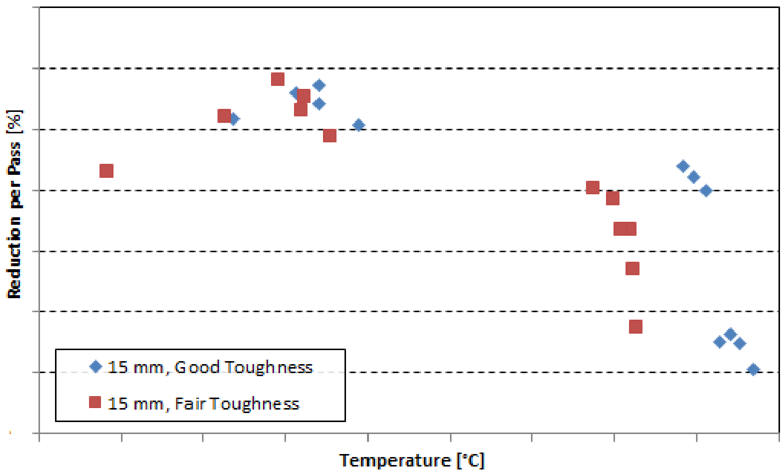


Figure 4. Generic evolution of percentual reduction per pass versus temperature along the plate mill pass schedules of the 15 mm NR plates studied in this work.

Figure 5 shows the evolution of several austenite microstructural parameters along the pass schedules of the 15 mm NR plates as calculated by MicroSim®. It can be seen in this figure that, despite the unfavorable start, at the end of the roughing stage the austenite mean grain size of the good toughness plate was only slightly greater that of the fair toughness plate; the $Dc_{0.1}$ values of both plates were approximately the same. However, this situation changed along the finishing stage, as the fair toughness plate showed high recrystallization fractions, above 80%, after the F2, F3 and F4 passes, while the good toughness plate showed a nearly high recrystallization fraction only after the F2 pass. Probably this can be due to the lower slab discharging temperature of the fair toughness plate, at a temperature not high enough to promote full Nb solubilization, which can explain its faster austenite recrystallization kinetics in these three finishing passes. From this point on, the fair toughness plate showed slightly higher values of austenite mean grain size and $Dc_{0.1}$. Besides that, accumulated strain (that is, austenite strain hardening) was lower for this plate due to the already

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mentioned high recrystallization fractions between rolling passes during the finishing stage.

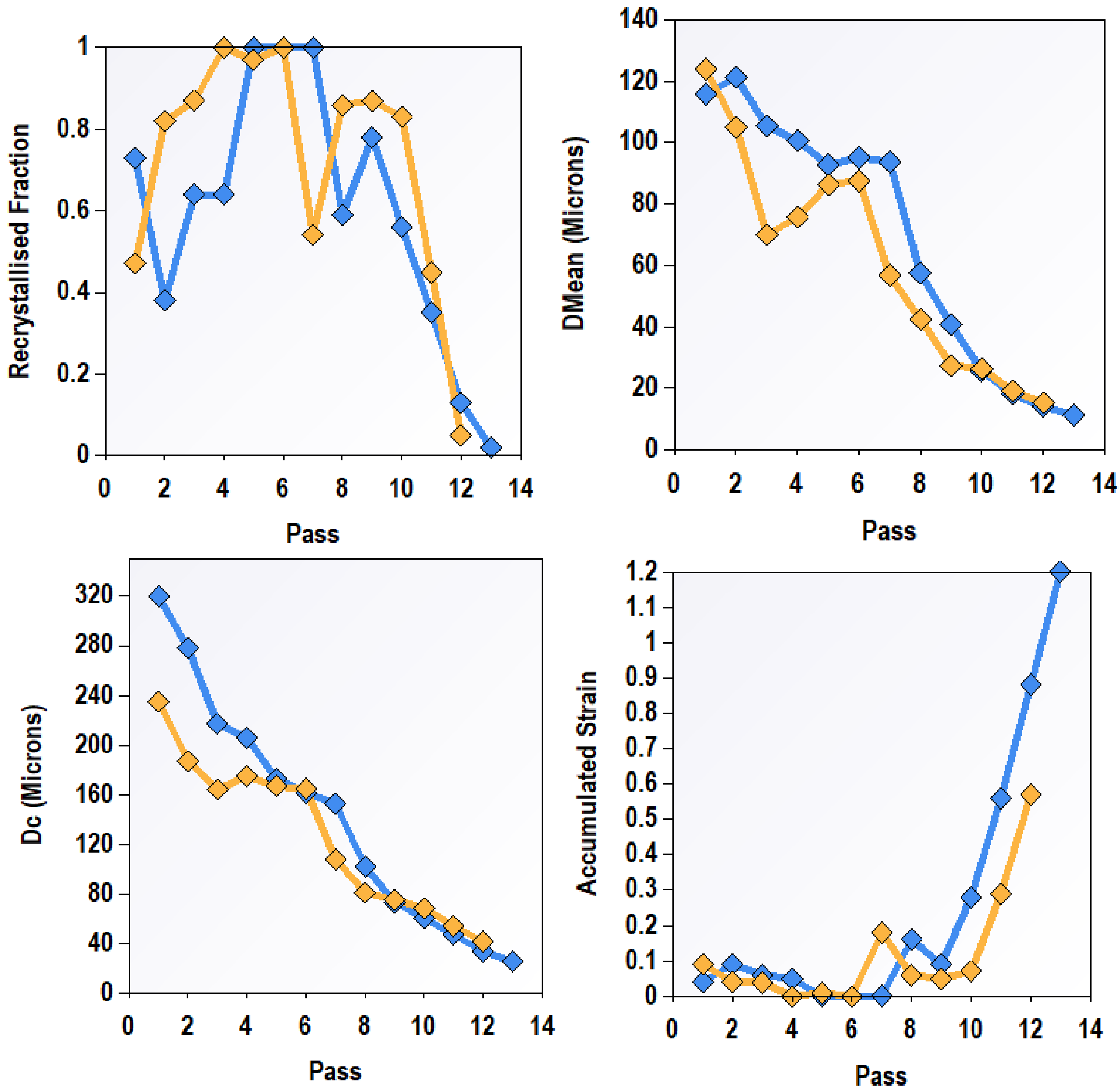

Figure 5. Several austenite microstructural parameters got along the pass schedules of the 15 mm NR plates studied in this paper, as calculated by MicroSim ®. The blue line indicates the good toughness plate, while the yellow one indicates the fair toughness plate.

Table 2 shows a summary of the results calculated by MicroSim® for the 15 mm NR plates. As expected, the good toughness plate showed the minimum values of austenite mean, maximum and $Dc_{0.1}$ grain sizes, as well Zd ratio. Accumulated strain was maximum, which contribute to refine even more the mean ferrite grain size.

Table 2. Final austenite grain size parameters for the 15 mm NR plates studied in this paper, as calculated by MicroSim ®

Plate	Mean GS [µm]	Max GS [µm]	$Dc_{0.1}$ [µm]	Zd	ε_{ACC}	Ferrite GS [µm]
Good Toughness	8.9	102.4	24.4	2.74	1.17	4.2
Fair Toughness	10.5	115.6	39.0	3.71	0.67	5.8

GS: Grain Size; ZD = $Dc_{0.1}$/Mean GS; ε_{ACC}: Accumulated Strain; Ferrite Grain Size calculated according to [4]

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Now, considering the case of 30 mm thick plates, figure 6 shows that the good toughness plate showed a normal roughing stage, with increasing pass reductions as it thickness fell. For its turn, the fair toughness plate showed a weird roughing stage, starting well but, after the 4th pass, pass reductions have fallen significantly, requiring a greater number of rolling passes to reach holding thickness – 8, instead of the 6 applied in the good toughness plate. Finishing stage was nearly similar for both cases.

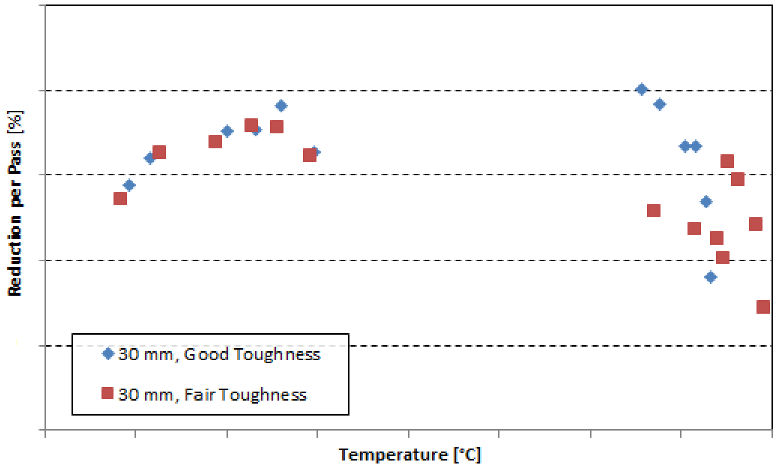

Figure 6. Generic evolution of percentual reduction per pass versus temperature along the plate mill pass schedules of the 30 mm NR plates studied in this work.

The different pass schedules for the 30 mm plates promoted different microstructural evolutions, as can be seen in figure 7. The fair toughness plate showed grain size more refined than the good toughness plate in the end of the roughing stage due to the more cases of partial recrystallization between rolling passes in the first case; however, its $Dc_{0.1}$ value was higher already. Curiously, the fair toughness plate showed higher recrystallization fractions between passes in the finishing stage. According to MicroSim®, the effect of such pass schedule variations on the final ferrite mean grain size was virtually null, as can be seen in table 3. Maximum austenite grain size, $Dc_{0.1}$ and Zd values were higher for the fair toughness plate, although the last parameter was not so different. This indicates a more heterogeneous microstructure, which can explain its lower toughness.
Both pairs of 15 mm and 30 mm NR plates were analyzed through EBSD at CEIT in order to validate the microstructural parameters determined by MicroSim®. The results of grain size distribution, considering only high angle boundaries (15°), can be seen in figure 8 and table 4. A direct comparison between the microstructural parameters determined by MicroSim® and EBSD is not recommendable, as they analyzed different phases (austenite and ferrite, respectively). Besides that, the values of mean ferrite grain size shown in this paper were not calculated by MicroSim®.
One can see that only the 15 mm plate with good toughness showed a sharper grain size distribution, with minimum values of mean grain size, $Dc_{0.2}$ and Zd ($Dc_{0.2}$/Dmean). All the other three plates showed similar values of such parameters. The comparison between the two 30 mm plates revealed somewhat contradictory results: the good toughness plate showed a nominal smaller medium grain size than the fair toughness plate (9.1 microns versus 10.0 microns, respectively), but a more heterogeneous microstructure (Zd equal to 2.7 and 2.3, respectively). Possible causes for such discrepancies can be the analysis of a non-representative sample and/or segregation and pearlite fraction issues. But it is interesting to note that, in the specific case of the

30 mm NR plates, the results of microstructural parameters got by MicroSim® showed a better agreement with the toughness values than the EBSD analysis.

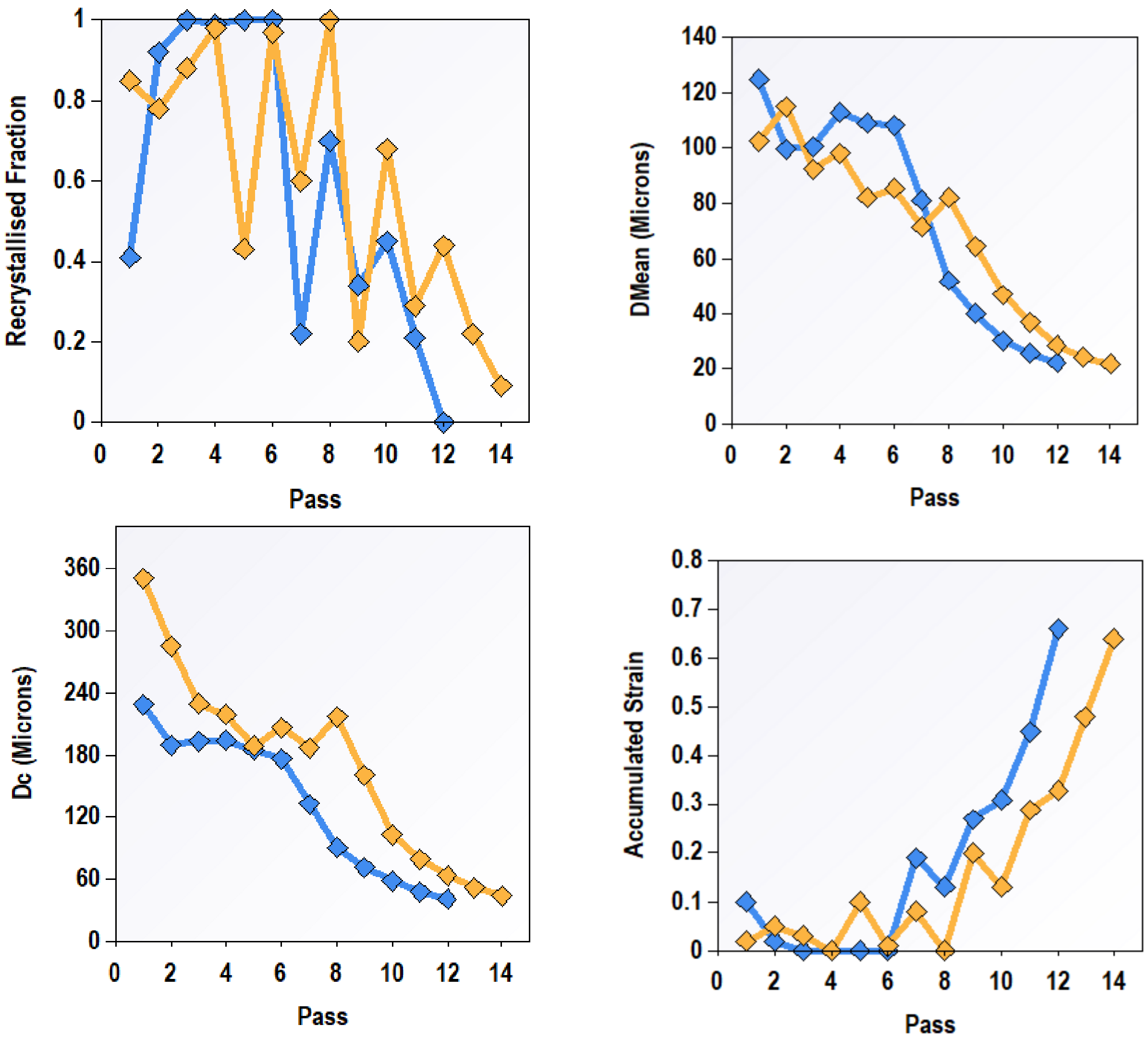

Figure 7. Several austenite microstructural parameters got along the pass schedules of the 30 mm NR plates studied in this paper, as calculated by MicroSim ®. The blue line indicates the good toughness plate, while the yellow one indicates the fair toughness plate. The number of roughing passes was 6 for the good toughness plate and 8 for the fair toughness plate.

Table 3. Final austenite grain size parameters for the 30 mm NR plates studied in this paper, as calculated by MicroSim ®

Plate	Mean GS [µm]	Max GS [µm]	$Dc_{0.1}$ [µm]	Zd	ε_{ACC}	Ferrite GS [µm]
Good Toughness	16.3	165.7	43.4	2.66	0.69	6.5
Fair Toughness	17.7	265.6	52.5	2.97	0.71	6.6

GS: Grain Size; ZD = $Dc_{0.1}$/Mean GS; ε_{ACC}: Accumulated Strain; Ferrite Grain Size calculated according to [4]

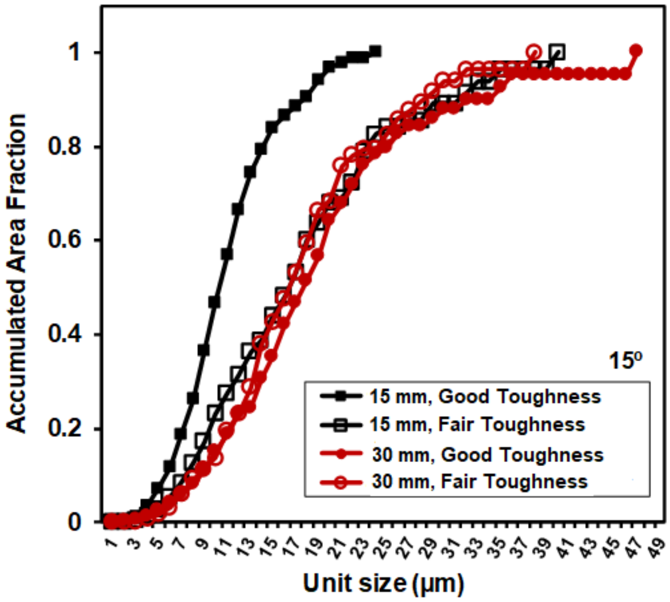

Figure 8. Grain size distribution considering high angle boundaries for the 15 and 30 mm plates determined by EBSD.

Table 4. Grain size distribution parameters, for the 15 and 30 mm plates, considering high angle boundaries, determined by EBSD

Plate	$D_{15°}$ [μm]	$Dc_{0.2}$ [μm]	$Dc_{0.2}/D_{15°}$
15 mm, Good Toughness	6.8	14.2	2.1
15 mm, Fair Toughness	8.7	22.5	2.6
30 mm, Good Toughness	9.1	24.3	2.7
30 mm, Fair Toughness	10.0	23.3	2.3

3 CONCLUSION

The analysis using the results of microstructural evolution in normalized rolled plates of niobium microalloyed steels calculated by the MicroSim® model presented interesting results that helped to correlate the applied rolling process parameters with the different toughness obtained in the plates considered for this study. The results for this work showed that it is very important to fully solubilize niobium in the reheating furnace and promote full austenite recrystallization between passes using minimum number of rolling passes with high thickness reductions during the roughing stage. Fully solubilized niobium will contribute to restrict recrystallization between passes of the finishing stage as much as possible. So, in this way, it is possible to achieve a refined and homogeneous grain structure in the normalized rolled plate and, consequently, high levels of toughness.

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