Journal of Physics: Conference Series

PAPER • OPEN ACCESS

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To cite this article: A Smith et al 2019 J. Phys.: Conf. Ser. 1270 012030

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IOP Conf. Series: Journal of Physics: Conf. Series **1270** (2019) 012030 doi:10.1088/1742-6596/1270/1/012030

The effect of Niobium on Austenite Evolution during Hot **Rolling of Advanced High Strength Steel**

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Abstract. Two AHSS steels (0.2C-2.4Mn-1.5Si with/without Nb+Cr addition) were subjected to pilot reversing mill hot rolling in 5 passes varying the finish rolling and coiling temperature. After coiling simulation, the final microstructures were characterised by the EBSD technique measurements. One sample was analysed to determine the presence and the level of Nb precipitated by TEM and chemical extraction. The austenite evolution was inferred from the measured transformation textures and reconstructed final austenite texture and microstructure. Further insights were obtained by comparing the results to a semi-empirical austenite evolution model, which predicted recrystallization behaviour and level of Nb precipitation. From the combined experimental and modelling work it was shown that Nb addition led to recrystallization inhibition via partial strain induced precipitation for this high Mn and high Si steel. Applying the austenite evolution model to a more realistic industrial hot strip rolling schedule revealed earlier Nb precipitation during hot rolling and higher strain accumulation with a finer grain size. The final Nb level in solution was lower for the industrial schedule, although a significant amount was not precipitated (two thirds of the total).

1. Introduction

Austenite evolution during hot rolling has been intensively studied and modelled in the last decades for plain carbon, (C-Mn) and low carbon microalloyed (Nb,V,Ti) steels [1,2]. Recently there has been explosive growth in the development of advanced high strength steels (AHSS) for the automotive sector [3]. Since these steels are mainly produced as thin sheets following cold rolling and heat treatment, the microstructural changes occurring during hot rolling have received much less attention. Nevertheless, understanding and control of austenite evolution in AHSS during hot rolling is of importance, to ensure e.g. cold rollability and to optimise the microstructure for processing after cold rolling [3]. The latest AHSS chemistries employ higher carbon contents and significant levels of Mn (e.g. up to 3 wt%) and Si (e.g. 1.5 wt%), with or without microalloying [4]. These rich alloying chemistries may significantly affect austenite evolution in terms of recrystallization behavior and Nb strain induced precipitation.

The present work explores the role of niobium addition on austenite evolution in laboratory rolling of AHSS steels. The Nb state, (i.e. as solute or as strain induced precipitate) and austenite microstructure were inferred from a combination of texture measurements, and prior austenite reconstruction, precipitation characterization, and microstructure evolution modelling. Finally, based on the results obtained, the validated austenite evolution model was applied to an example industrial strip rolling schedule.

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2. Experimental methods and models used

Two advanced high strength steel chemistries were cast into 50 kg ingots using vacuum induction melting. The measured compositions are shown in table 1.

Table 1. AHSS steel chemistries. Element amounts in wt% with balancing Fe.

	С	Mn	Si	Cr	Nb	Al	Ν
Base Nb	0.20 0.20	2.4 2.4	1.5 1.25	0 0.3	0 0.025	0.02 0.02	0.002 0.002

The ingots were first rough rolled into slabs of 20 mm thickness. After reheating at 1250° C for 1 hour, hot rolling was performed down to a strip thickness of 5 mm in 5 passes (percent reductions of 30-25-25-20-20). The temperature of the final pass was varied i.e. finishing temperatures (FT) of 920° C and 860° C were studied. Temperature was measured during rolling via a thermocouple inserted at the mid thickness of the slab. Since a reversing mill was used, the interpass times were relatively long i.e. in the range 10-20s. After finish rolling, the strips were subjected to air or water cooling to the target coiling temperature (CT) i.e. 620° C or 540° C. The coiling was simulated by furnace cooling the strips at a rate of 20° C/h.

Samples were taken parallel to the rolling direction and prepared for EBSD examination via grinding and electro polishing. Orientation distribution functions were determined to obtain the bulk texture. In addition, samples were analysed after mechanical polishing via optical microscopy (Nital/Le Pera etch). For the Nb steel, analysis of precipitates was carried out for one condition only (FT = 920°C and CT = 540°C), using both TEM replicas and chemical extraction followed by inductively coupled plasma optical emission spectroscopy (ICP-MS).

For modelling studies, the EBSD measurements were used to reconstruct the prior austenite grain structure and its texture, using the APRGE software [5]. In addition, the austenite microstructure evolution during hot rolling was simulated using an in-house model [6]. This model is based on a combination of classical semi-empirical recrystallization models [1,2]. Strain induced precipitation was treated using the original physical model of Dutta and Sellars [7], extended it to predict the fraction of niobium in solution according the approach of Pereda et al. [8]. The interaction of recrystallization with precipitation was treated in the most simple fashion i.e. assuming that once precipitation had started that no further softening (recrystallization) could take place during the remaining passes [1]. Finally, the austenite evolution model should be adapted consider the effect of steel chemistry, since the AHSS compositions are richer than those used to develop the original equations. This was taken into account in the following way. Firstly, static recrystallization was modelled using the activation energy (composition dependent) i.e. in a linear fashion for Mn and Si solute drag according to Medina [2]. To extrapolate this model to high Si levels, a modification was made considering the non-linear effect of Si seen in TRIP steels by Suikkanen et al. [9]. For Mn no modification to the Medina equation was made i.e. linear behavior was assumed. This is considered justified, considering the much weaker effect of Mn on recrystallization kinetics in microalloyed steels and the smaller extrapolation i.e. 2.4 wt% Mn studied here, versus 1.2-1.4 wt% Mn steels mainly used to generate the Medina model equation. Another influence of Mn and Si is on the Nb strain induced precipitation kinetics [1]. Thus Si can accelerate precipitation and Mn retards it. Although the Siciliano model takes this into account by correction factors, the Si and Mn levels used were significantly different to those of AHSS chemistries. Thus for the present study, the original Dutta Sellars model was used, adjusting the pre factor for precipitation start time if necessary.

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3. Results and Discussion

3.1. Optical microscopy and TEM

Microstructures obtained for air/water cooling with coiling at 620°C, were ferrite-pearlite structures for both steels and finishing temperatures. For coiling at 540°C, a bainitic structure was obtained for the Nb steel with air/water cooling. The base steel with reduced hardenability (no Cr or Nb) led to a partially bainitic structure with water cooling. Figure 1 shows example microstructures for the steels.





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Figure 1 Microstructures for base (left) and Nb (right) steels for finish rolling temperature of 860°C followed by water cooling and coiling at 540°C.

For the Nb steel an example TEM replica micrograph is shown by figure 2 below.



Figure 2 Nb steel TEM replica after hot rolling with finish rolling temperature of 920°C followed by air cooling and coiling at 540°C.

As can be seen in figure 2, TEM analysis revealed the presence of Nb (C,N) precipitates with a size range of 10-32 nm. For the conditions given in figure 2 the chemical extraction results revealed that 0.020 wt% Nb remained in solution i.e. 0.005 wt% or 20% of the Nb was present as Nb(C,N) precipitates.

3.2. EBSD results and simulations

EBSD scans are shown by the orientation distribution functions (ODFs) in figure 3. The results were compared to the theoretical textures expected for different austenite states [10,11].

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Figure 3 (A) ODF's of hot rolled samples of the base alloy with CT 620°C and FT 920°C (a) and FT 860°C (b) as well as similarly for the Nb alloy FT 920°C (c) and FT 860°C (d). (B) Theoretical formation of transformation texture in steels [11].

As can be seen from figure 3, for both steels and finishing temperatures, a clear transformation fiber texture is observed. This texture would suggest that before transformation deformed austenite was present i.e. having a texture with copper and brass orientations. In addition, the rotated cube texture component is present in all cases (strong and weak). This component may indicate the presence of recrystallized austenite i.e. having a texture with cube orientation. Unfortunately, the rotated cube component can also be formed from deformed austenite with a brass orientation. However, since the measured rotated cube component for the base steel at 920°C finishing temperature was significantly higher than that for the Nb steel, at the same finishing temperature (maximum intensity of 2.3 versus 1.4), one can infer a greater final recrystallized fraction for the base steel. For the lower finishing temperature, the situation is more complex since the rotated cube component for both steels is weak. Rather, it can be seen that the intensity of the general deformation fiber is more intense for the Nb steel (for example transformed Goss component). Thus it can be tentatively concluded that the final austenite recrystallized fraction in the base steel was also higher for finishing at 860°C.

To gain further insights, the austenite texture was reconstructed using the APRGE software, applied to the measured textures at the lower finish rolling temperature. Since this software requires the presence of diffusionless transformation products, the measured EBSD textures from samples water cooled and coiled at 540°C were used as the input. The results are shown in figure 4.



Figure 4 ODF's of reconstructed prior austenite structure just before transformation of the Base (c) alloy and Nb alloy (d), both in FT860°C-water cooling-CT540°C condition.

As can be seen, the base steel austenite texture exhibits copper, Goss and cube components, indicating a partially recrystallized state, whilst the Nb steel reveals a strong copper component i.e. indicating deformed austenite. Combining these results with those from figure 3 clearly illustrate the role of Nb in the rich AHSS chemistry, namely that for both finishing temperatures, Nb addition has led to more significant inhibition of recrystallization.

3.3. Recrystallization modelling of lab scale hot rolled strips

From the previous sections, several important questions arise i.e. is the Nb effect observed due to solute drag or to strain induced precipitation? Are the observed precipitates formed before or during/after transformation? Can recrystallization modelling of lab rolling reproduce the same trends as those inferred from the texture results?

Figure 5 below shows the results of the modelling activity for the cases of air cooling after finishing to the coiling temperature of 620° C. Only a summary is shown i.e. final total retained strain (indicating the degree of austenite deformation retained after 5 passes), and the final recrystallized fraction (X). Note that similar trends were obtained if water cooling after finishing to coiling at 540°C was used.



Figure 5 Recrystallization modelling results summary for base (left) and Nb steel (right).

As shown in figure 5, for both finishing temperatures, the accumulated strain values between base and Nb steels are very similar when the Nb effect on recrystallization inhibition is considered only as solute drag. (Note that the slightly lower strain for Nb solute drag at the higher temperature is due to the actual temperature of the last passes being slightly higher than for the base steel). On the other hand, when Nb strain induced precipitation was allowed to occur, (started between the last two passes F4-F5), the accumulated strain was significantly higher, i.e. recrystallization was completely supressed for both finishing temperatures starting between F4 and F5. In addition, figure 5 also shows the effect of finishing temperature on recrystallization kinetics i.e. for all three cases the accumulated strain in austenite was higher for the lower temperature. Comparing these simulations to the trends from the texture analysis reveals good qualitative agreement, i.e. partial recrystallization of the base steel for both finishing temperatures, and a much higher accumulated strain in the Nb steel, (considering strain induced precipitation rather than a solute drag effect).

Concerning the predicted final amount of Nb in solution for when strain induced precipitation was active, values of 0.018-0.019 wt% were obtained for all schedules, including the schedule where TEM and chemical extraction analysis was made afterwards. Thus, good agreement was obtained with chemical extraction results with no modification to the original Dutta Sellars model coefficients. This suggests that the opposite influence of Si and Mn on precipitation kinetics for the current steel chemistry would tend cancel each other out.

3.4. Recrystallization modelling of 0.025wt% Nb steel for an industrial hot strip schedule The model was extended to an industrial strip rolling schedule of 7 finishing passes (with final pass at 900°C) taken from literature [12]. Table 2 shows the comparison of results with the lab schedule.

Fable 2.	Recrystallization	modelling results	for 0.2C-2.4Mn-	1.25Si-0.025Nb-0.3Cr steel
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Schedule	Total reduction	Interpass	Final Nb level in	Final residual	Final fraction	Final austenite grain
	ratio	times (sec)	solution (wt%)	strain	recrystallized	size (microns)
Lab	4	10-20	0.019	0.44-0.51	0	15
Industrial	9	1-4	0.016	1.18	0	7

For the industrial schedule with much higher total reduction, Nb precipitation occurred earlier (F2-F3 compared to F4-F5 for lab rolling), this led to greater strain accumulation, a finer final austenite grain size, and a lower amount of Nb remaining in solution. Interestingly the model predictions suggest that a significant amount of Nb remains in solution (two thirds of 0.025 wt%) for the industrial schedule. This information is of great interest when considering optimization of AHSS mechanical properties. Thus Nb remaining in solution is available for downstream processes e.g. assisting steel hardenability during transformation or for precipitation hardening after cold rolling and annealing.

4. Conclusions

From a combination of precipitate analysis, texture analysis, and recrystallization modelling adapted to AHSS chemistries, a picture was built up of the austenite evolution during laboratory hot rolling of two AHSS steel compositions (with and without 0.025 wt% Nb). The base steel final austenite microstructure was partially recrystallized, whilst for the Nb steel a mainly deformed austenite structure was obtained. For both steels, a lower finish rolling temperature increased the amount of retained strain. For the Nb steel, results were best explained by strain induced precipitation rather than by solute drag. Kinetics of strain induced precipitation were in agreement with classical model predictions, suggesting that Mn and Si effects cancelled each other out. Finally, extending the validated model to an industrial strip rolling schedule revealed earlier Nb strain induced precipitation i.e. giving much more strain accumulation and a finer grain size. Nevertheless, a significant amount of Nb remained in solution, potentially available for downstream processes.

Acknowledgments

This research has received funding from the European Union's Research Fund for Coal and Steel under grant agreement N°709755, OptiQPAP project.

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