

AUSTENITE SOFTENING IN THE FINISHING STAGE OF T.M.C.P. OF HIGH NIOBIUM-LOW MANGANESE MICROALLOYED STEELS

Antonio Augusto Gorni¹
José Herbert Dolabela da Silveira²
Kenji Camey¹
Emanuelle Garcia Reis²

Abstract

Recently several studies have been developed on the use of microalloyed steels with low Mn content (below 1%) in the controlled rolling of heavy plates for the manufacture of pipes. However, lower Mn contents should lead to the acceleration of NbCN precipitation kinetics during rolling, reducing the soluble Nb content in austenite. This fact, plus the heavy and fast passes applied under virtually isothermal conditions in a heavy plate rolling mill, increases the risk of partial recrystallization during the finishing stage of the controlled rolling, since strain hardening of austenite becomes significant and the interaction between precipitation and recrystallization is increasingly delayed. The objective of this study was to analyze the evolution of mean flow stress during the finishing stage of controlled rolling in these low Mn, high Nb microalloyed steels to better understand the metallurgical mechanisms acting during this process.

Keywords: Controlled Rolling; Low Mn high Nb microalloyed steels; Austenite recrystallization; NbCN precipitation.

AMACIAMENTO DA AUSTENITA DURANTE A FASE DE ACABAMENTO DO PROCESSAMENTO TERMOMECÂNICO DE AÇOS MICROLIGADOS COM ALTO NIÓBIO E BAIXO MANGANÊS

Resumo

Recentemente foram desenvolvidos estudos sobre o uso de aços microligados com baixo teor de Mn (abaixo de 1%) na laminação controlada de chapas grossas para a manufatura de tubos de grande diâmetro. Contudo, menores teores de Mn podem levar à aceleração da precipitação de NbCN durante a laminação, levando a uma redução no teor de Nb solúvel presente na austenita. Este fato, mais os rápidos e pesados passes aplicados sob condições virtualmente isotérmicas num laminador de chapas grossas, aumentam o risco de recristalização durante a fase de acabamento da laminação controlada, já que o encruamento da austenita se torna significativo e a interação entre precipitação e recristalização é cada vez mais atrasada. O objetivo desse estudo consistiu em analisar a evolução da resistência média à deformação durante a fase de acabamento da laminação controlada nestes aços com baixo Mn e alto Nb para se entender melhor os mecanismos metalúrgicos que atuam durante esse processo.

Palavras-chave: Laminação controlada; Aços microligados com baixo Mn e alto Nb; Recristalização da austenita; Precipitação de NbCN.

I INTRODUCTION

The use of Mn as an alloying element in steels has been virtually unquestionable for decades due to its relatively low cost and the benefits it provides, such as increased hardenability and hot ductility. Its usefulness has further increased with the advent of the so called Advanced High Strength Steels (AHSS), where its role is even more important. To crown this evolution, it is a key alloying

element in twinning-induced plasticity (TWIP) steels, where its amount is in the 20-22% range.

But, as one would expect, so much success ends up with disadvantages [1]. The increase in demand for manganese obviously can lead to a significant increase in the price of its ferroalloys, so its replacement is already being studied in technically viable cases. Recent studies

¹Gerência de Pesquisa e Desenvolvimento, Gerdau Ouro Branco, Ouro Branco MG, Brazil. E-mail: antonio.gorni@gmail.com

²Gerência de Chapas Grossas, Gerdau Ouro Branco, Ouro Branco MG, Brasil.



advocate its replacement by Nb microadditions, which have been done with good results both in technical and economic terms [2]. Other advantages resulting from the lowering of the Mn content, these already known for a long time, are the reduction of the load of ferroalloys in the ladle and the consequent loss of heat by the liquid steel and lower contamination by P, as well minimization of central segregation intensity in slabs and microstructural banding in the final products [3]. These advantages, plus the increase in the solubility of S in austenite, which minimizes or even suppresses the nucleation of MnS, constitute the basis for a relatively new type of steel used in heavy plates for the manufacture of sour service pipes, which combines extra-low Mn and significant Nb contents [4].

Although the new low Mn steel compositions are balanced in order to maintain or even improve the performance of the final products, their peculiar alloy design influences their metallurgical response during its thermomechanical controlled processing (TMCP), as discussed below.

Maehara, in an early work [5], showed that the increase in Mn content slows down the kinetics of austenite static recrystallization at 900°C in CMn steels, occurring saturation of this effect when its content approaches values about 1%. The effect of Mn on the retardation of austenite recrystallization kinetics in Nb microalloyed steels was also studied, but considering exclusively their drag effect as dissolved atoms, a situation which occurs during the roughing stage of controlled rolling, where there is no NbCN precipitation due to the high rolling temperatures. Cho et al. [6] proposed several equations to quantify the effect of Mn on the kinetics of austenite recrystallization in a low C steel containing 0.050% Nb. In general, the equations proposed by him to calculate the time required for 50% static or metadynamic recrystallization of austenite indicate that the corresponding activation energy values were directly proportional to the Mn content of the steel in the range between 0.5 and 1.5%. For its turn, Lotter et al. [7] proposed an equation to calculate the time required by 50% of austenite recrystallization which simultaneously includes the effect of Mn and Nb, both contributing to increase the activation energy of this restoration process.

On the other hand, the effect of Mn on austenite recrystallization in microalloyed Nb steels is more complex at temperatures low enough for NbCN precipitation to occur. It is necessary to consider the mutual interaction between these two elements: Mn increases the stability of Nb in austenite, promoting its solubilization [8], and increasingly hinders its precipitation to a content of approximately 1.9%, when the effect stabilizes [9].

So, it seems logical to expect that, in Nb microalloyed steels with low levels of Mn, where precipitation would occur more quickly, there would be less time available for the occurrence of recrystallization, increasing its softening resistance. As a matter of fact, it was found in these steels that the faster Nb precipitation raises the temperature of no-recrystallization (T_{nr}) due to the occurrence of Nb

precipitation at higher temperatures. However, this early and intensified precipitation, plus the faster precipitate growth, already in the roughing stage of TMCP, may eventually deplete Nb dissolved in austenite in the finishing stage, reducing its drag effect and easing recrystallization [10].

It was also verified in steels with low Mn content that the immediate and extensive nucleation of NbCN precipitates over the dislocations present in the strained austenite promotes their pinning, which restricts or delays austenite recovery [11, 12]. For its turn, recovery suppression increases the thermodynamic potential for the subsequent recrystallization. In addition, lower Mn contents exert less drag effect on the recrystallization fronts [5]. All these factors can accelerate softening kinetics, so it could start before the beginning of NbCN precipitation.

Thus, it is plausible to conceive a pass schedule under slightly decreasing temperatures during the finishing stage of controlled rolling where the increase in the thermodynamic potential of austenite recrystallization resulting from the recovery blocked by intensified NbCN precipitation, associated with the impoverishment of soluble Nb that this causes in austenite, and the accumulation of strain hardening at each pass, ends up enabling austenite recrystallization, even if partial, at relatively low temperatures.

The analysis of mean flow stress values calculated from hot rolling loads in the finishing stage of industrial controlled rolling of a microalloyed steel with Mn content less than 1.0%, C less than 0.05% and Nb greater than 0.060% revealed a consistent softening occurrence at the third finishing pass (F3). This fact was unusual, since the finishing stage had started around 940-970°C, well below the of T_{nr} experimentally determined by a multiple hot torsion test for a similar steel, 1070°C [13], according to the procedure proposed by Boratto et al. [14], as shown in Figure 1. It should be noted that the difference between T_{nr} measured in the laboratory and the finishing start temperature adopted in the industrial controlled rolling was

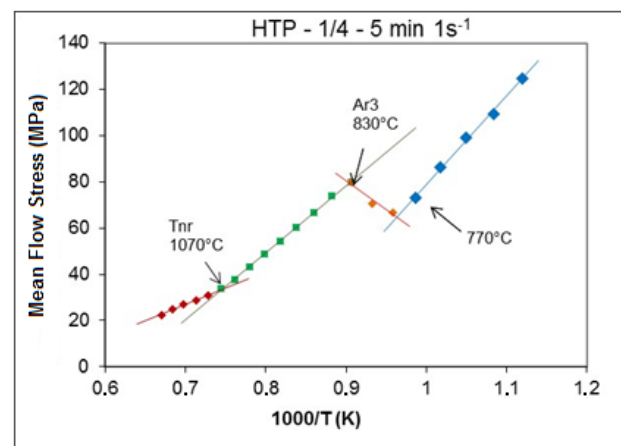


Figure 1. Austenite critical temperatures for a high Nb microalloyed steel with low Mn content [13].

above 75°C, as recommended by Bai et al. [15]. Another intriguing fact was the absence of softening of austenite in the laboratory test.

It is widely known that any occurrence of austenite recrystallization during the finishing stage of controlled rolling is undesirable, as it affects the homogeneity of the grain size distribution in the microstructure of the final plate. This, for its turn, potentially impairs its toughness features. So, it was decided to develop this work to better understand the origin of this phenomenon and identify its countermeasures.

2 MATERIAL AND METHODS

Slabs with 250 mm thickness, made of microalloyed steel with Mn content lower than 1.0%, C lower than 0.05% and Nb higher than 0.060%, were controlled rolled to 20 mm plates. After roughing and a holding stage, the finishing stage started at temperatures between 940 and 970°C, except where otherwise noted. Mean Flow Stress (MFS) values corresponding to each rolling pass were calculated using process data routinely available at the plate mill supervisory system. The following Formula 1 was used:

$$MFS = \frac{P}{w\sqrt{R\Delta h} Q} \quad (1)$$

where P is the rolling load; w is the width of the rolling stock; R is the radius of the work rolls; Δh is the difference between the initial and final thicknesses of the rolling stock; and Q is a dimensionless factor defined by the model to calculate hot rolling loads proposed by Sims [16].

A correlation of the values of MFS thus obtained with the specific controlled rolling conditions was performed to verify which process circumstances could promote austenite softening during the finishing stage.

3 RESULTS AND DISCUSSION

Figure 2 shows the evolution of the MFS values along the controlled rolling pass schedules. As can be observed in this figure, normal variations during the execution of the same nominal process conditions led to different trends for the MFS value observed in the F3 pass. Normally it is expected a continuous and monotonous increase in the values of MFS along the finishing stage of controlled rolling, but this was not always the case here: there was a slight decrease of 6 MPa between the values of MFS for passes F2 and F3 in the case of Figure 2a. In the case of Figure 2b such decrease in MFS was not observed, although its increase was negligible: only 7 MPa. In general terms, it can be seen a steep increase of MFS at the beginning of the finishing stage until the F2 pass, occurring then a “plateau” in F3 pass and, from there, constant increase, but at a much slower pace than previously observed.

This discrepancy prompted a survey of the effective plate rolling conditions and their correlation with the different trends of MFS value in the F3 pass. Pass temperature versus accumulated strain plots, shown in Figure 3, were made in order to identify which process conditions could lead to softening. Figure 3a, F2 pass temperature versus cumulative strain in F1 and F2 passes, was proposed to map the favorable conditions for a possible static recrystallization between F2 and F3 passes, while Figure 3b, F3 pass temperature versus cumulative strain in F1, F2 and F3 passes, had the same objective, but focusing on a possible dynamic recrystallization or austenite-to-ferrite transformation in F3 pass.

It can be observed that, in both cases, the softening in F3 pass has occurred when temperature in this pass was above approximately 950°C, regardless of the previously accumulated strain. This softening could also occur at lower temperatures, down to 930°C, provided that previously accumulated strain was large enough: above 0.45 for F1 and F2 passes or 0.65 for F1, F2 and F3 passes.

So, graphs at Figure 3 allow establishing an important recommendation: the finishing stage of controlled rolling of the steel studied here should be started at temperatures below 930°C to avoid any potential occurrence of any softening

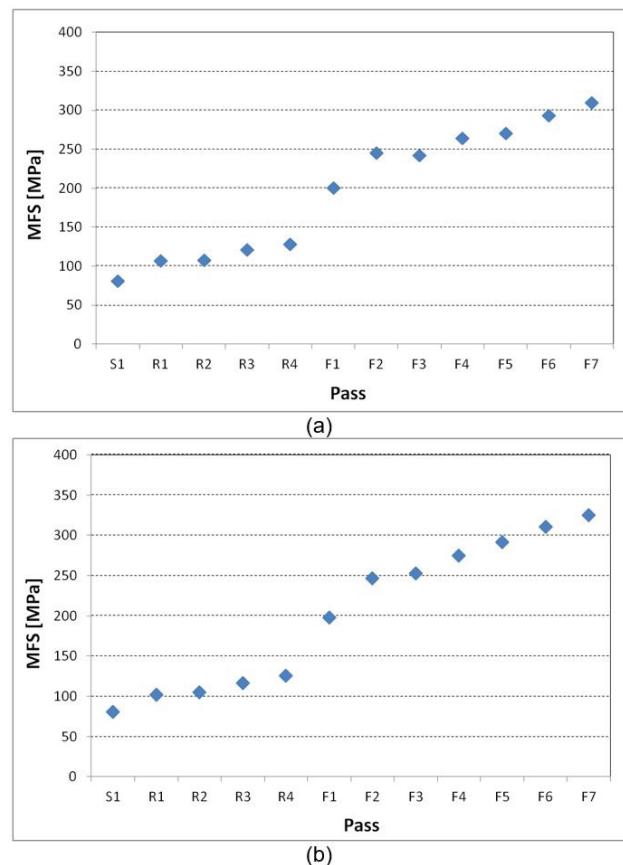


Figure 2. Evolution of MFS values observed along two pass schedules for the same steel and TMCP nominal conditions. Nevertheless, there were different trends for the MFS value in the F3 pass: (a) softening; (b) strain hardening (although slight).

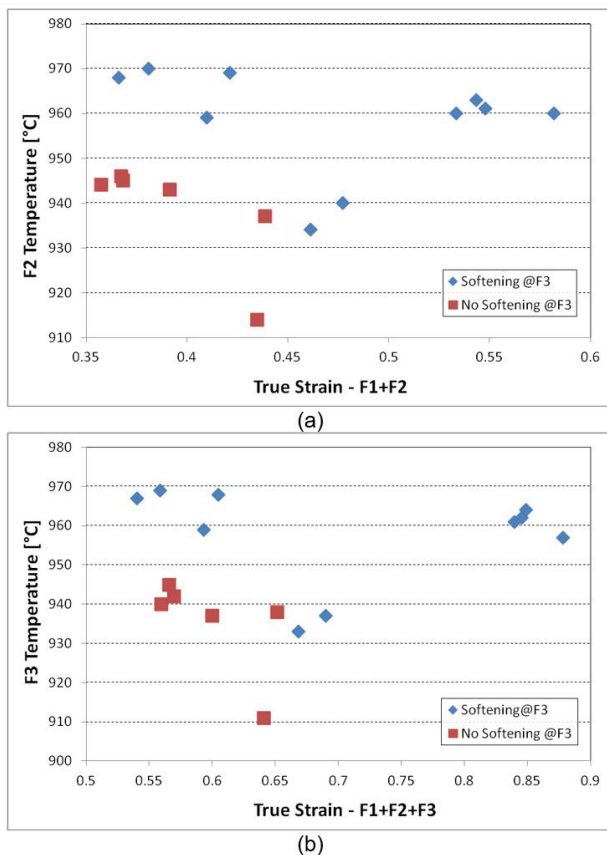


Figure 3. Softening condition maps in the F3 pass: (a) temperature on F2 pass versus accumulated strain on F2 and F3 passes; (b) temperature on the F3 pass versus accumulated strain on F1, F2 and F3 passes.

between passes, in order to preserve homogeneity of the final microstructure and ensure a high toughness performance.

It would be interesting to know what kind of softening is occurring in the F3 pass - if static recrystallization in the interval between F2 and F3 pass, or dynamic recrystallization or ferrite transformation in F3 pass, but this is difficult to verify under industrial conditions. Calculations of microstructural evolution using models available in the literature [17] cannot even predict the occurrence of austenite softening in the F3 pass, most probably due to the lack of adequate equations to describe recrystallization and precipitation kinetics for Nb microalloyed steels with low Mn. However, a parallel additional experience, where time elapsed between passes F2 and F3 was purposely increased from 10-12 seconds to 75 seconds, showed that there was no softening in the F3 pass as it would be expected, since this case fitted in the regions favorable to softening mapped in the graphs of Figure 3. On the contrary, in this case the value of MFS in the F3 pass was 12 MPa higher than that in the F2 pass, value higher than the 7 MPa of variation that was usually observed in cases where softening did not occur. This fact seems to indicate that the softening observed in the F3 pass under the conditions mentioned here is probably due to some dynamic softening process, like dynamic recrystallization or even dynamic transformation [18], since a longer time

available for restoration between F2 and F3 passes seems to have contributed to reduce the accumulated strain in austenite and avoided (or at least minimized) the dynamic softening in the F3 pass.

One aspect to be considered here is the greater reduction of the Nb content dissolved in austenite due to the precipitation intensified by the low Mn content. Calculations performed through a microstructural evolution model [17], assuming the specific conditions of the TMCP performed here, but for a relatively high Mn steel, indicate that the loss of Nb in solid solution in the F3 pass can reach around 32%, which would contribute to reduce the minimum deformation necessary for the occurrence of dynamic recrystallization. However, it should be noted that no specific equations are known yet to calculate the kinetics of recrystallization and precipitation of these new low Mn steels, which would probably indicate an even greater loss of solute Nb. Another factor that may facilitate recrystallization is the lower content of Mn itself, since there will be less solute atoms in austenite to hinder the movement of the recrystallization interfaces, as previously reported [5].

A question then arises: why then did the test for determination of T_{nr} performed in [13] not presented any softening of austenite below this temperature, which was approximately 1070°C? A probable explanation for this fact is the continuous application of strain passes during this test. So, several of these passes were applied in the region of austenite partial recrystallization, that is, the range between the so called Recrystallization Limit Temperature (RLT) and Recrystallization Stop Temperature (RST). This fact, plus the relative long time intervals between passes (30 s), reduces the accumulation of residual strain that could promote an eventual recrystallization. As a matter of fact, double deformation tests showed the occurrence of partial recrystallization starting 10 seconds after the application of a 0.3 strain at 975°C [13]. On the other hand, the industrial controlled rolling includes a holding period which prevents rolling while the plate shows temperatures within the range of partial recrystallization of austenite. The finishing stage started at relatively low temperatures, 940 to 970°C, and time intervals between passes were relatively short, about 10 to 12 s. Such conditions apparently allowed an accumulation of residual strain high enough to deflagrate dynamic recrystallization or ferrite transformation in the F3 pass.

4 CONCLUSION

When performing TMCP of Nb microalloyed steels it is necessary to ensure that its finishing stage occurs in such a way that no recrystallization of austenite occurs between passes. This contributes to an adequate level of microstructural homogeneity that ensures the achievement of good toughness characteristics in the final product. This is why the finishing stage of controlled rolling starts at

temperatures below the RST, where recrystallization of austenite no longer occurs between the rolling passes. From a practical point of view, it can be considered that RST is 75 to 100°C below RLT (or T_{nr}). However, under specific process conditions, austenite softening was detected during the finishing stage of controlled rolling of a Nb microalloyed steel with low Mn content, even when this stage started at temperatures 100°C below the measured T_{nr} value. This fact was attributed to the relatively low Mn content of this steel,

which promotes an immediate and copious nucleation of NbCN precipitation in the dislocations after hot deformation, preventing the recovery of austenite and increasing the thermodynamic potential for its recrystallization. The most evident solution to this problem, as shown by the results of this work, is an even greater lowering of the start finishing temperature of controlled rolling in order to restrict the eventual occurrence of austenite recrystallization, despite its higher thermodynamic potential.

REFERENCES

- 1 Gorni AA, Silveira JHD, Camey K, Reis EG. Austenite partial recrystallization in the finishing stage of controlled rolling of niobium microalloyed steels with low manganese. In: Proceedings of the 11th International Rolling Conference: ABM Week; 2019; São Paulo. São Paulo: ABM; 2019.
- 2 Jansto S. The integration of process and product metallurgy in niobium bearing steels. *Metals*. 2018;8(671):1-20.
- 3 Gorni AA, Reis JSS, Silva CNP, Cavalcanti CG. Otimização da composição química e do processo de laminação controlada de chapas grossas navais. In: Anais do 32º Seminário de Laminação: Processos e Produtos Laminados e Revestidos; 1995; Curitiba. São Paulo: Associação Brasileira de Metalurgia e Materiais; 1995. p. 373-392.
- 4 Gray JM. Ultra low manganese high toughness HTP sour service linepipe steel. In: Proceedings of the Microalloyed Pipe Steels for the Oil & Gas Industry Congress; 2013; Moscow, Russia. Moscow: CBMM; 2013. 14 p.
- 5 Maehara Y, Kunitake T, Fujino N. The effect of alloying elements on the static recrystallization of mild steels in the austenite range. *Tetsu To Hagane*. 1981;67:362-371.
- 6 Cho SH, Bang KB, Jonas JJ. Effect of manganese on recrystallisation kinetics of niobium microalloyed steel. *Materials Science and Technology*. 2002;18:389-395.
- 7 Lotter U, Schmitz HP, Zhang L. Application of the metallurgically oriented simulation system “TKS-StripCam” to predict the properties of hot strip steels from the rolling conditions. *Advanced Engineering Materials*. 2002;4:207-213.
- 8 Koyama S, Ishii T, Narita K. Effects of Mn, Si, Cr and Ni on the solution and precipitation of niobium carbide in iron austenite. *Journal of the Japanese Institute of Metals*. 1971;35:1089-1094.
- 9 Akben MG, Weiss I, Jonas JJ. Dynamic precipitation and solute hardening in a V microalloyed steel and two Nb steels containing high levels of Mn. *Acta Metallurgica*. 1981;29:111-121.
- 10 Karmakar A, Biswas S, Mukherjee S, Chakrabarti D, Kumar V. Effect of composition and thermomechanical processing schedule on the microstructure, precipitation and strengthening of Nb-microalloyed steel. *Materials Science and Engineering A*. 2017;690:158-169.
- 11 Miao CL, Shang CJ, Zhang GD, Zhu GH, Zurob H, Subramanian S. Studies on softening kinetics of niobium microalloyed steel using stress relaxation technique. *Frontiers of Materials Science in China*. 2010;4:197-201.
- 12 Subramanian S, Zurob H, Miao C, Shang C. Studies on softening kinetics of low manganese steel microalloyed with niobium for high-strength sour service ERW pipe. In: Proceedings of the International Symposium on the Recent Developments in Plate Steels; 2011; Winter Park, Colo. USA. Warrendale : Association for Iron & Steel Technology; 2011. p. 365-374.
- 13 Oliveira NJL, Andrade MS, Castro CSB, Escobar DP, Borba EC, Silva JCP, et al. Precipitação de Nb(C,N) durante a laminação a quente de chapas grossas de um aço com baixo Mn e Alto Nb. Belo Horizonte: Centro de Inovação e Tecnologia SENAI-FIEMG; 2017. 102 p. Relatório Final de Projeto.
- 14 Boratto F, Barbosa R, Yue S, Jonas JJ. Effect of chemical composition on the critical temperatures of microalloyed steels. In: *Thermec-88: Proceedings of the International Conference on Physical Metallurgy of Thermomechanical Processing of Steels and Other Metals*; 1988; Tokyo, Japan. Tokyo: Iron and Steel Institute of Japan; 1988. p. 383-390.
- 15 Bai D, Bodnar R, Ward J, Dorricott J, Sanders S. Development of discrete X80 line pipe plate at SSAB Americas. In: Proceedings of the International Symposium on the Recent Developments in Plate Steels; 2011; Winter Park, Colo. USA. Warrendale: Association for Iron and Steel Technology; 2011. p. 13-22.

- 16 Sims RB. The calculation of roll force and torque in hot rolling mills. Proceedings of the Institution of Mechanical Engineers. 1954;168:191-214.
- 17 Pereda B, Rodriguez-Ibabe JM, López B. Improved model of kinetics of strain induced precipitation and microstructure evolution of Nb microalloyed steels during multipass rolling. ISIJ International. 2008;48:1457-1466.
- 18 Rodrigues SF, Aranas C Jr, Wang T, Jonas JJ. Dynamic transformation of an X70 steel under plate rolling conditions. ISIJ International. 2017;57:162-169.

Received: 15 Nov. 2019

Accepted: 8 Dec. 2019