Strengthening Mechanisms in High-Strength, Low-Alloy Structural Steel With Nb and High Ti

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ABSTRACT

Low-carbon structural steels, microalloyed with niobium and titanium, the latter in a hyperstoichiometric amount in relation to N in order to promote TiC precipitation after rolling, have been an answer to produce hot strips with extra-high levels of mechanical strength. However, their production is more complex than niobium microalloyed steels, since more rigid control of the soluble titanium content is required at the end of rolling, and because extra precipitation affects the toughness of the product. Due to this situation, this work had the aim to determine the contributions of the various strengthening mechanisms to the yield strength typical of such steels, originating from solid solution, grain size, dislocation density, fraction of MA (retained martensite-austenite) constituent and precipitation. Samples of this steel, were extracted from hot coils produced in a Steckel Mill. Their microstructures were analyzed by optical and scanning electron microscopy, as well by EBSD; hardness and tensile tests were also performed. It was verified that the greatest contribution to strength was provided by grain size, followed by solid solution and precipitation, dislocation density and MA constituent. However, the results obtained on the precipitation contribution showed greater dispersion, probably due to the fluctuations that occur in the soluble titanium content at the end of rolling.

Keywords: Microalloyed Steel, Alloy Design, Niobium, Titanium, Strengthening Mechanisms

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INTRODUCTION

The current search for reducing CO_2 emissions has encouraged the development of structural steels with higher levels of mechanical strength, thus allowing the reduction of the thickness of the elements of the structures and, consequently, their weight - or, in other words, promoting their partial dematerialization. This allows for reductions in carbon footprints in several ways: the amount of steel required for a given project is smaller; its transportation and handling require less energy; and vehicles made with it are lighter, generating fewer emissions as they move.

Specific structural steel applications, such as weightlifting, agricultural machinery and transportation, require materials with higher than usual levels of mechanical strength. However, this property must be achieved without sacrificing the ductility, toughness and weldability levels typical of commodity structural steels. This condition makes the easiest way to achieve this objective unfeasible, that is, simply increasing the carbon content of the steel. Therefore, several alloy designs have been proposed that, while maintaining or even slightly reducing the carbon content of the steel, allow for higher strength levels and a highly optimized balance between those seemingly incompatible properties.

Gerdau Ouro Branco, a Brazilian integrated steelmaker, recently began developing higher-strength hot-rolled coils. It is based on a microalloyed steel with niobium and a hyperstoichiometric content of titanium in relation to nitrogen, obtained in the form of hot-rolled coils [1]. The main feature of this product is the fact that it is obtained through the application of a Thermomechanical Controlled Processing (TMCP) in a Steckel rolling mill, a fact that is still unprecedented in the literature. The manufacturing of hot-rolled coils of the S600MC grade is currently consolidated, in accordance with the BS EN 10149-2 standard [2].

On the other hand, this alloy design based on high titanium content presents some problems. It is not yet possible to know precisely which titanium content is still soluble – and, therefore, available for later TiC precipitation in the coil - at the end of rolling, which impairs production stability and increases the dispersion of the final mechanical properties in the product. In addition, such precipitation may reduce the toughness of the final product. For this reason, it is advisable to know precisely the various contributions of the strengthening mechanisms and to prioritize the contributions of the solid solution, grain size and microstructural constituents, like MA (martensite-retained austenite), when defining the chemical composition and, particularly, the TMCP process conditions. The contribution of TiC precipitation strengthening should be minimized in order to achieve the mechanical strength values specified by technical standards [3]. This problem was addressed in a very recent paper. It was developed a mathematical model resident in the Level 3 automation of the Hot Strip Mill which, based on the actual nitrogen and titanium contents present in the steel slabs, automatically calculate the corresponding process windows through a multi-objective optimization algorithm, which are reported to Level 2 of the equipment and applied dynamically in the mill. According to this reference, the amplitude of dispersion in this product was decreased from 206 to 68 MPa (67%) in yield strength, from 191 to 68 MPa (64%) in tensile strength and from 10 to 4.5% (55%) in elongation [4].

So, aiming at an optimization of the chemical composition of the steel and the TMCP process parameters in the Steckel Mill, a detailed microstructural characterization of this new version of steel with hyperstoichiometric titanium content would be very opportune to quantify the contribution of the different strengthening mechanisms to the mechanical strength – more specifically, the yield strength. The recent advent of electron backscattering diffraction (EBSD) microstructural analysis now allows the calculation, with higher precision, of the contribution of various microstructural parameters to the mechanical strength of steels, as will be described below.

It was demonstrated that, in the case of a microstructure composed of acicular ferrite plus MA constituent, the contribution of the various strengthening mechanisms to the yield strength can be calculated by the following equation [5]:

$$\sigma_y = \sqrt{\left(\sigma_0 + \sigma_{ss} + \sigma_{ppt} + \sigma_{MA}\right)^2 + \sigma_\rho^2 + \sigma_{gs}^2} \tag{1}$$

where σ_0 is the iron lattice friction stress, σ_{ss} is the contribution of the solid solution strengthening, σ_{ppt} is the contribution of the precipitation, σ_{MA} is the contribution of the MA constituent, σ_ρ is the contribution of the dislocation density and σ_{gs} is the contribution of the grain size. The respective formulas needed to calculate such contributions can be found in [6] and are listed below.

The solid solution contribution σ_{ss} can be calculated by the formula proposed by Pickering, assuming that σ_0 is equal to 53.9 MPa:

$$\sigma_{ss} = \sigma_0 + 32.3Mn + 83.2Si + 11Mo + 354\sqrt{N_{free}} \tag{2}$$

For its turn, the grain size contribution to yield strength can be estimated by a formula based in the famous Hall-Petch relationship, but incorporating some microstructural parameters determined by EBSD analysis:

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$$\sigma_{gs} = 1.05\ \alpha\ M\ \mu\ \sqrt{b} \left[\sum_{2° \leq \theta_i \leq 15°} (f_i \sqrt{\theta_i}) + \sqrt{\frac{\pi}{10}} \sum_{\theta_i \geq 15°} (f_i) \right] d_{2°}^{-0,5} \tag{3}$$

where α is a constant, equal to 0.3; M is the average Taylor factor, equal to 3; μ is the shear modulus, equal to 8 x 10^4 MPa; b is the magnitude of the Burgers' vector, equal to 2.5 x 10^{-7} mm; θ_i and f_i are, respectively, the mean disorientation angle (expressed in radians) and the corresponding relative frequency. Both parameters, as well $d_{2°}$, the mean equivalent diameter of the grain assuming grain boundaries with 2° disorientation, are determined by EBSD microstructural analysis.

The contribution of the dislocation density, σ_ρ, can be calculated by

$$\sigma_\rho = \alpha\ M\ \mu\ b\ \sqrt{\rho} \tag{4}$$

$$\rho = \frac{2\ \upsilon}{u\ b} \qquad (5) \tag{5}$$

where ρ is the dislocation density; υ is Kernel average misorientation for θ lower than 2°; u is the unit length, which is equal to 1.86 times the step size (that is, 0.186 μm), measured by EBSD, and b is the Burgers' vector.

The presence of MA islands in the microstructure contributes to yield strength according to this formula:

$$\sigma_{MA} = 900 f_{MA} \tag{6}$$

The following equation, proposed by Ashby-Orowan, quantifies the precipitation contribution to yield strength:

$$\sigma_{ppt} = 10.8\ \frac{\sqrt{f_v}}{x} ln\left(\frac{x}{6{,}125\ .\ 10^{-4}}\right) \tag{7}$$

where f_v is the volumetric fraction of precipitates and x is the mean planar intercept diameter in the particles, expressed in μm. However, given the extremely small size of the precipitates, these microstructural parameters, f_v and x, must be determined by transmission electron microscopy. This is a complex and laborious method, which analyzes very tiny portions of the material. Therefore, from a practical point of view, it is very common not to measure experimentally this contribution, but rather calculate it, by subtracting all other contributions of the strengthening mechanisms from the experimentally determined yield strength. This approach was adopted in this work.

The results of such microstructural analysis and calculation of the contributions of these strengthening mechanisms, performed in samples of hot rolled coils produced in the Steckel Mill of Gerdau Ouro Branco, are described below.

DEVELOPMENT

The alloy design adopted for the S700MC grade has the chemical composition shown in Table 1.

Table 1: Chemical composition ranges of the alloy studied in this paper.

C	Mn	Si	Nb	Ti	V	Cr	Mo	N
≤ 0.12	≤ 2.00	≤ 0.25	≤ 0.080	≤ 0.12	≤ 0.050	≤ 0.40	≤ 0.40	≤ 0.0080

The 250 mm slabs were reheated up to 1250°C in order to solubilize all niobium. They were rolled down to 12.5 mm strips in the Steckel Mill and cooled in a run-out table down to the coiling temperature. As has become routine, the applied TMCP included roughing and finishing stages, separated by a holding phase, to have total austenite recrystallization between passes in the first stage and minimal or even null recrystallization in the second. Final rolling was performed with the strip still austenitic, that is, slightly above Ar_3. A relatively wide range of coiling temperatures was tested to determine the region where an optimized amount of precipitation strengthening, especially caused by TiC, is got, assuring compliance with S700MC specifications. Figure 1 shows an outline of the Steckel Mill at Gerdau Ouro Branco.

After the still air cooling of the hot coils down to room temperature, samples were extracted from the tail of the strips/head of the coils to characterize their microstructure and determine mechanical properties. The hardness tests and microstructural

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analysis was carried out at the laboratories of CEIT, Spain. The microstructural quantitative parameters determined by EBSD were grain size considering misorientation of 4° ($d_{4°}$) and 15° ($d_{15°}$) between boundaries, and Kernel average misorientation for θ lower than 2° (υ). The region of the strips approximately at 1/4 of their thickness was analyzed using optical and scanning electronic microscopy, as well EBSD. Tensile tests were carried out at the Test Center of Gerdau Ouro Branco.

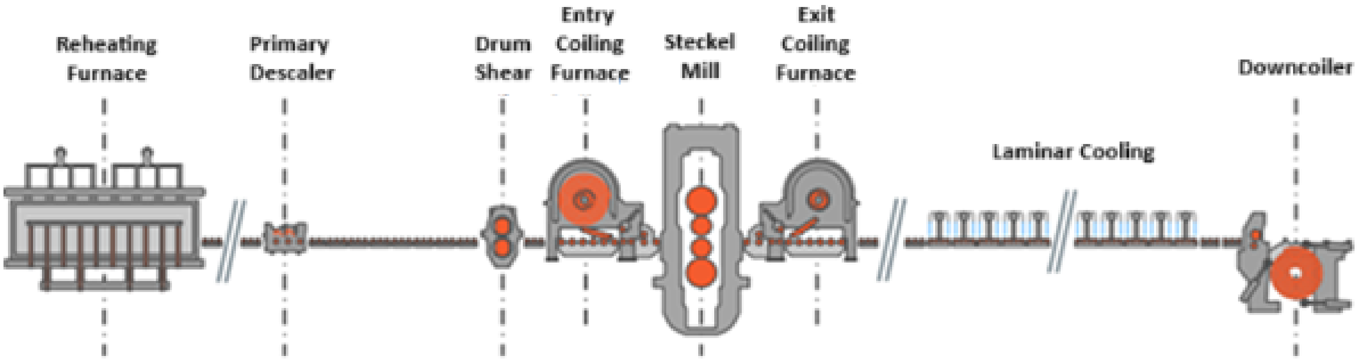

Figure 1: Outline of the Steckel Mill at Gerdau Ouro Branco at the time of the experiments.

DISCUSSION

Figure 2 shows the correlation between yield and tensile strength with the hardness of the coils. One can note that one case is clearly an outlier, as both yield and tensile strength values are clearly lower than the others. This is since its microstructure was predominantly composed of ferrite and pearlite, while the other samples presented microstructures of acicular ferrite, degenerated pearlite and MA constituent. The coiling temperature of the sample with lower mechanical strength was much higher than in the other cases, which caused this strong change in its microstructure matrix. The adoption of this very high coiling temperature was intentional, having occurred during the product development process, when this parameter was varied over a wide range to verify its effect on the microstructure and mechanical properties of the material. Figures 3(a) and 4(a) shows the microstructure of this sample with lower mechanical strength values, while figure 3(b) and 4(b) shows a microstructure where the strength values were higher.

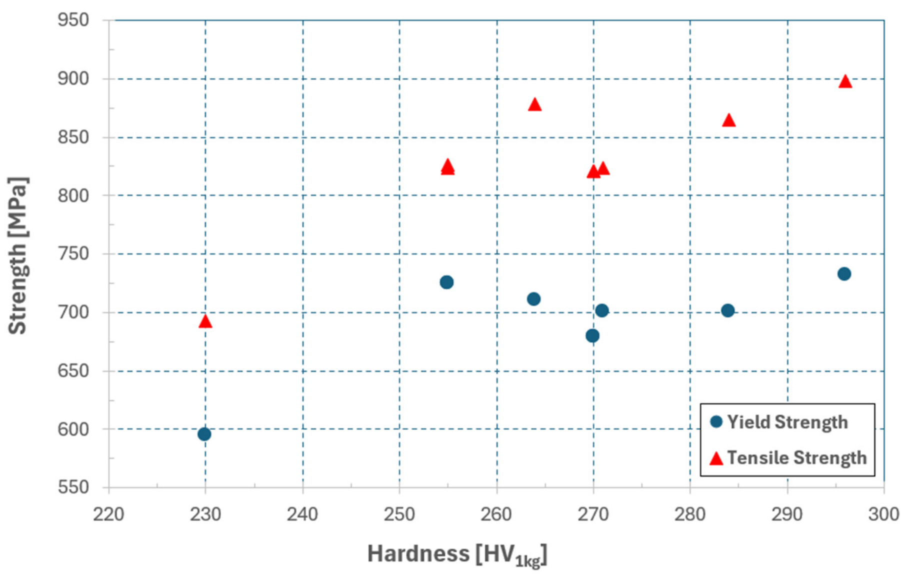

Figure 2: Correlation between yield and tensile strength with hardness for the coils.

The following equation, that correlates yield strength with hardness, was proposed for acicular ferrite/bainite microstructures of this kind of steel [7]:

$$YS\ [MPa] = 2.86\ HV - 45.5 \tag{8}$$

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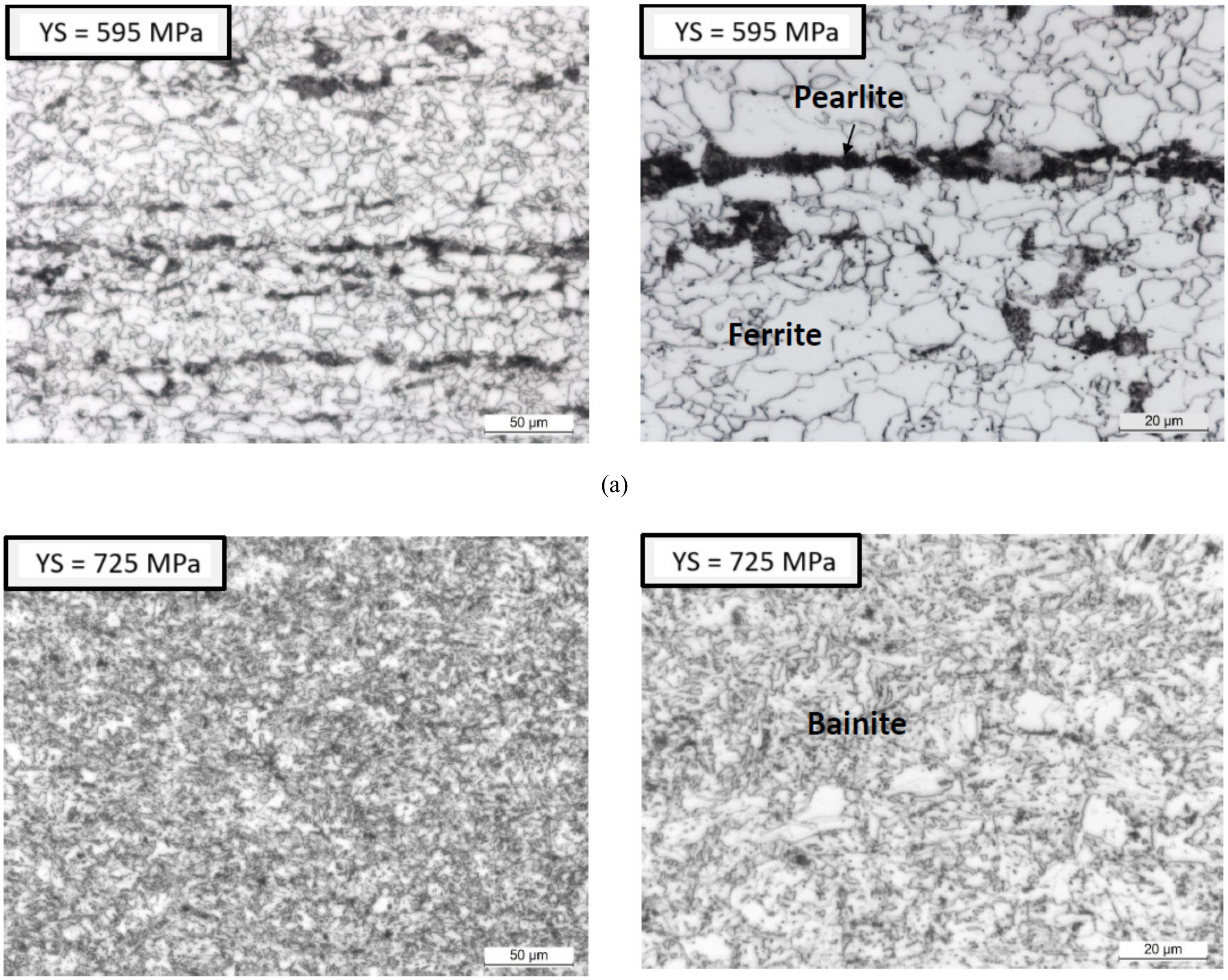
However, one can see in figure 2 that, visually, there is no recognizable relationship between yield strength and hardness in this case for steels with acicular ferrite/bainite. As a matter of fact, a linear regression equation developed using only data from these steels resulted in a zero intercept and a constant value of 707 MPa.



(a)

(b)

Figure 3: Optical microstructures of samples: a) with lowest strength: polygonal ferrite and pearlite; b) with maximum strength: acicular ferrite/bainite, degenerated pearlite and MA constituent. Nital etching.

Figure 5 shows several dispersion graphics between yield/tensile strength and microstructural parameters measured through EBSD analysis. One can see that the only sample with ferrite-pearlite microstructure, with minimum values of yield and tensile strength, showed significantly higher values of $d_{4°}$, $d_{15°}$ and lower values υ when compared with the samples with acicular ferrite/bainitic microstructure and higher mechanical strength. The samples with acicular ferrite/bainite microstructure did not present a clear correlation with these microstructural parameters. It is more likely that these results show that other strengthening mechanisms are more important than grain size and dislocation density for these specific samples, probably due to the application of different TMCP parameters.

The relative contributions of the strengthening mechanisms to the yield strength are shown in Figure 6a) to e). No clear relationship could be detected between yield strength and all strengthening mechanisms considered here, except for the case of solid solution. Probably different strengthening mechanisms are acting in each sample of hot strip, according to the specific conditions of TMCP applied to it. In the case of solid solution, figure 6a), which is independent from the rolling process, its strengthening contribution decreases as yield strength increases, ranging from 17% to 22%. This tendency was already expected, as all samples came from the same heat and so they have the same chemical composition and the same value of solid solution strengthening. The contribution from grain size, figure 6b), is highest, ranging from 42% to 50% but, as already verified with the influence of $d_{4°}$ and $d_{15°}$, there is no clear relationship between this contribution and yield strength. As expected, the

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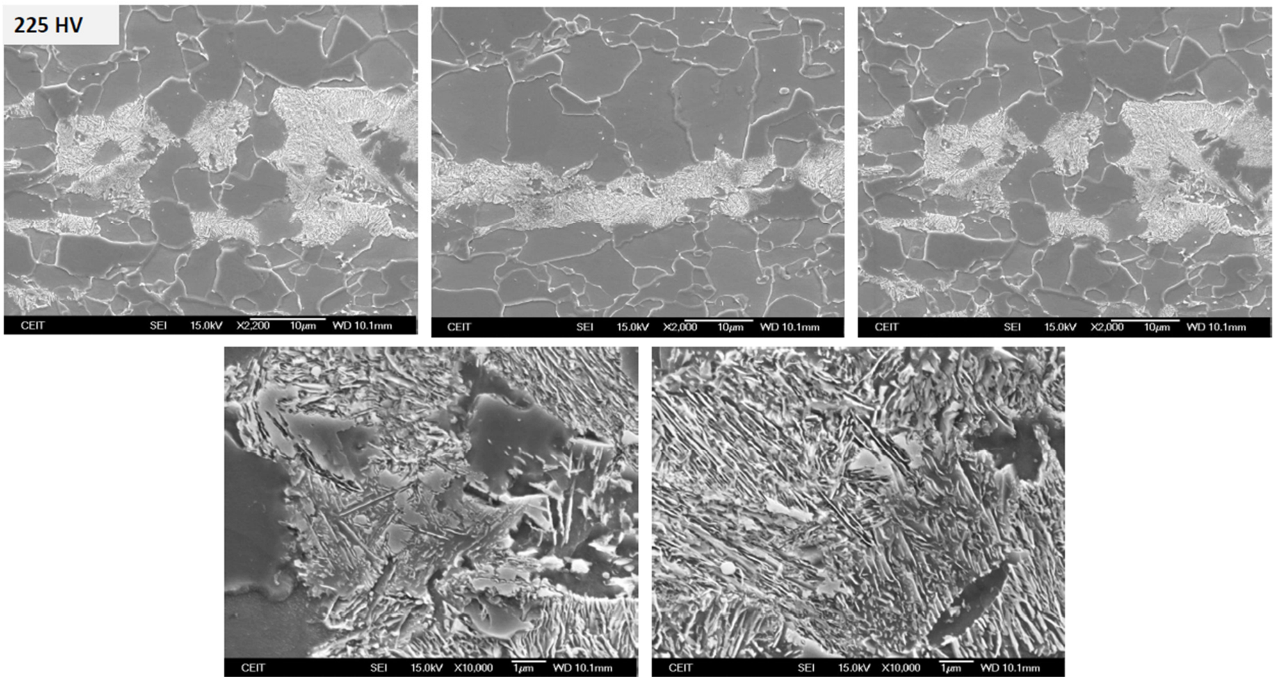
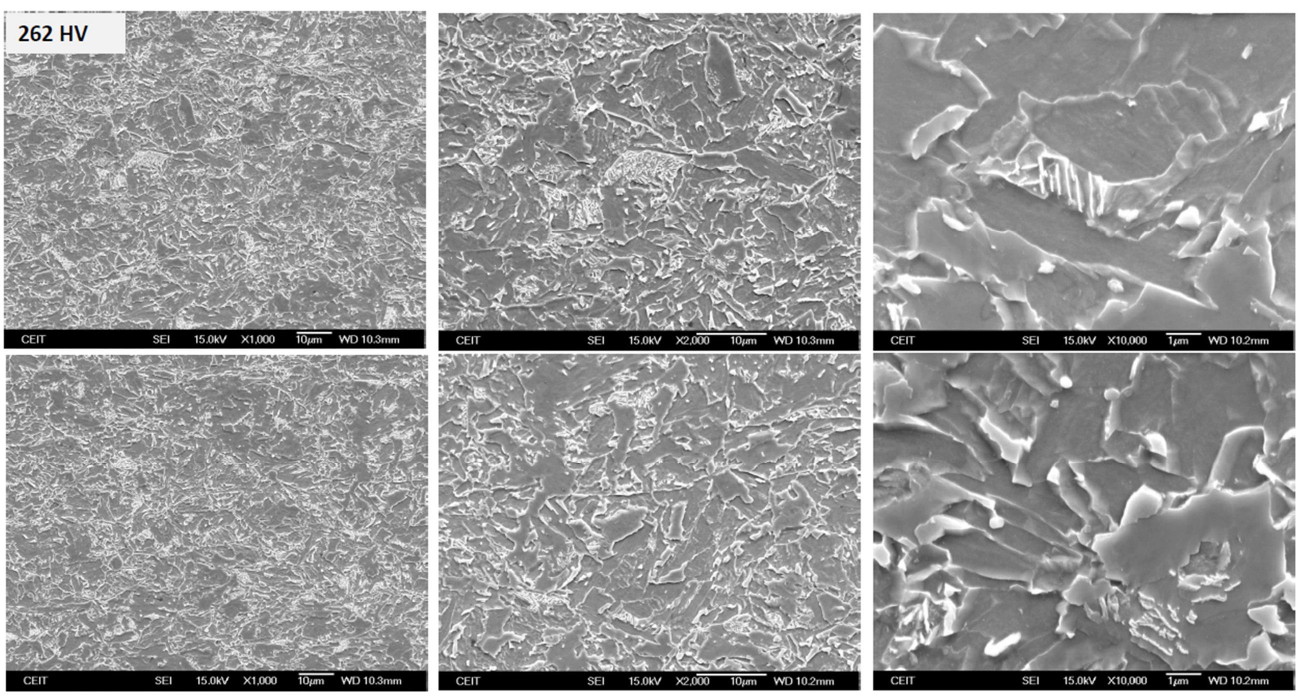
strengthening contribution from grain size was very low for the sample with ferrite-pearlite structure and minimum yield strength. Dislocation density contributions, figure 6c), were lower ranging from 8 to 13%. As expected, the ferritic-pearlitic sample with minimum yield strength, showed minimum contribution from dislocation density, which is lower for this kind of microstructure than for the bainitic matrix of the other samples. The contribution of the MA constituent present in microstructure was like the precedent case, ranging from 2 to 13% and, of course, was minimum in the case of the ferritic-pearlitic sample. Finally, the contribution precipitation strengthening was significant, but showed a broader range, from 7 to 41%.



(a)



(b)

Figure 4: Scanning electronic microstructures of samples: a) with lowest strength: polygonal ferrite and pearlite; b) with maximum strength: acicular ferrite/bainite, degenerated pearlite and MA constituent. Nital etching.

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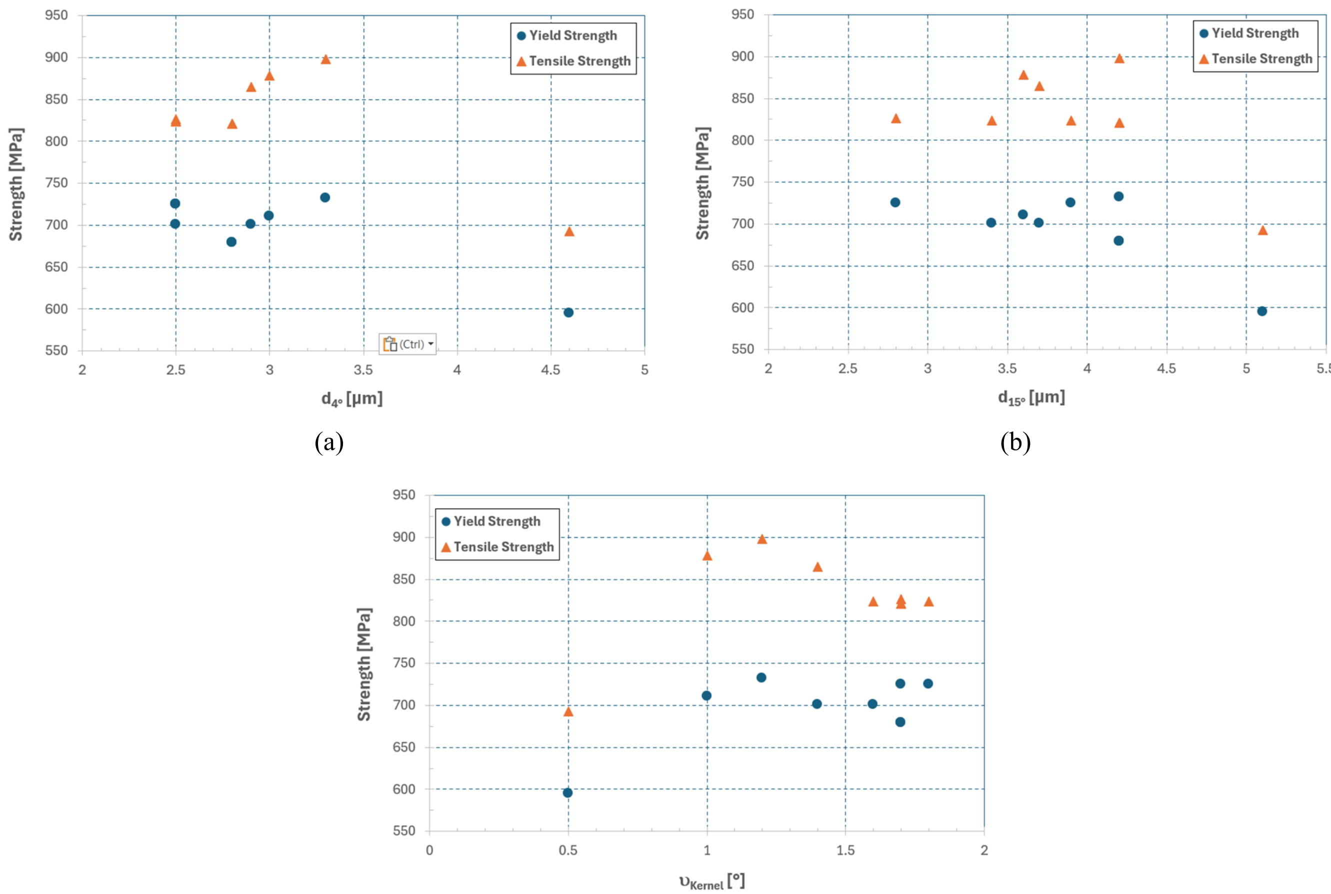
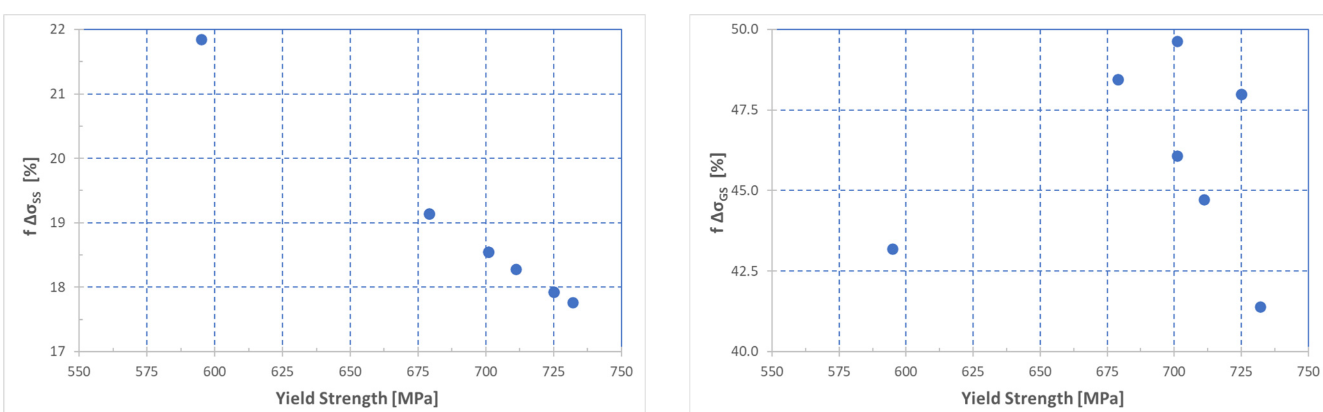


Figure 5: Relationship between yield and tensile strength with some microstructural parameters: grain size assuming a) misorientation of a) 4° ($d_{4°}$) or b) 15° ($d_{15°}$) between boundaries, and c) Kernel average misorientation for θ lower than 2° (υ).

Finally, Figure 7 presents the average contribution of each strengthening mechanism, considering only the samples with acicular ferrite/bainite microstructures, while Table 2 shows the standard deviation values associated with these averages. It can be observed that, in global terms, grain size was the main strengthening mechanism, contributing with 46% of the yield point value, followed by solid solution and precipitation, both tied at 18%, dislocation density, with 11%, and MA with 7%. In terms of the dispersion of these values, solid solution presented a minimum value, with 0.5%, and precipitation the maximum value, with 12.5%.

The high dispersion observed in the contributions of precipitation strengthening is inherent to this metallurgical mechanism, as previously mentioned, due to the fluctuations that occur in the soluble titanium content at the end of rolling, and which depend on the refining, solidification and TMCP conditions applied to the steel, as well as the methodology adopted for its calculation.



(a) (b)

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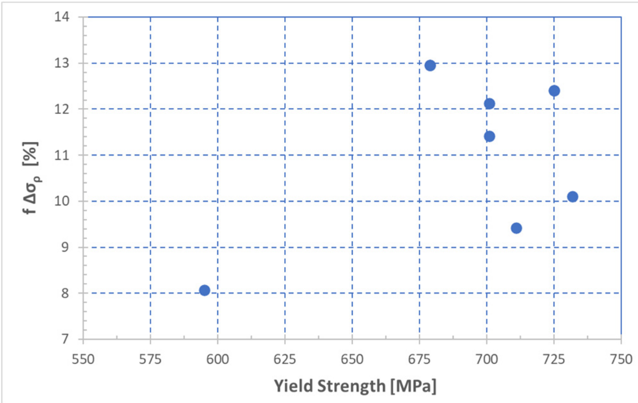
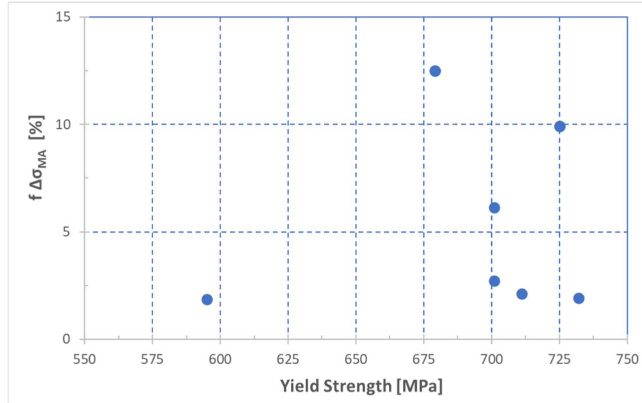
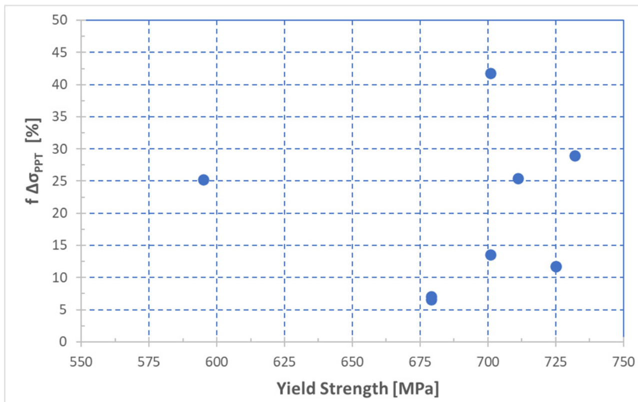


(c)



(d)



(e)

Figure 6: Relative contribution of each strengthening mechanism to the yield strength: a) solid solution, b) grain size, c) dislocation density, d) MA fraction and e) precipitation.

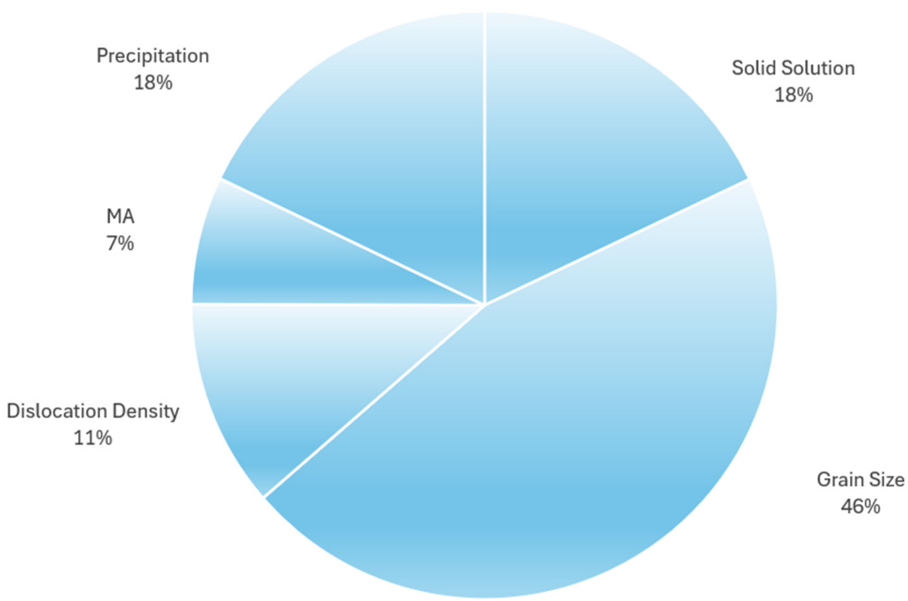


Figure 7: Mean relative contributions of each strengthening mechanism to the yield strength in the samples with acicular ferrite/MA microstructure.

Table 2: Standard deviation percentual values relative to the average contributions of each strengthening mechanism for the hot coils with acicular ferrite/MA microstructure.

Percentual Standard Deviation				
$\Delta\sigma_{ss}$ [%]	$\Delta\sigma_{gs}$ [%]	$\Delta\sigma_{\rho}$ [%]	$\Delta\sigma_{MA}$ [%]	$\Delta\sigma_{ppt}$ [%]
0.5	2.7	1.3	4.6	12.5

CONCLUSIONS

The study demonstrated that the yield strength of higher-strength hot-rolled coils produced in a Steckel mill using a Nb and hyperstoichiometric Ti alloy design is primarily governed by grain size refinement, followed by contributions from solid solution strengthening and precipitation. Dislocation density and the presence of MA (martensite-austenite) constituents provided comparatively smaller but still relevant contributions. This balance of strengthening contributions highlights the beneficial effect of niobium during hot working, achieving final fine and homogeneous microstructures, which combined with the TiC strengthening, give a strong support to reach the high mechanical properties.

Among the samples analyzed, a clear outlier with ferrite-pearlite microstructure exhibited significantly lower strength, attributed to excessively high coiling temperature that suppressed the formation of acicular ferrite and MA phases. EBSD-based microstructural characterization enabled accurate quantification of grain size and dislocation density effects, although some uncertainty remained in precipitation strengthening due to the difficulty of directly measuring TiC particles and the variability in soluble Ti content at the end of rolling.

The lack of consistent correlation between yield strength and EBSD parameters across all samples suggests that distinct TMCP paths influenced the dominant strengthening mechanisms in each case. These findings highlight the importance of optimizing rolling and coiling conditions to minimize precipitation-induced toughness losses while ensuring strength compliance via grain refinement and solid solution effects. The developed methodology offers a valuable tool for guiding alloy and process adjustments in advanced HSLA steel production.

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