Original Article

Niobium addition effect of SAE 4320 steel properties after annealing and normalizing stages of the automotive carburized component manufacturing

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Abstract

Steel is the material mostly used in automotive industry to produce lighter and economy cars without abstaining from safety and minimizing pollutant emissions. However, this material faces challenges in meeting mechanical, competitive, and good formability properties. Microalloyed steels emerges as a response to this demand as it comines high mechanical strength with good toughness and ductility due to an appropriate alloy design that is linked to low carbon contents and use of microalloys such as Nb, V and Ti which provide precipitation hardening, solid solution, and grain refinement. This study investigated the effect of Nb addition at different processing stages of case-hardened automotive component (after annealing and normalizing) by quantifying how this element influences grain size, hardness, yield strength, ultimate strength, elongation, area reduction and impact test. Results indicate that niobium addition between 200 to 500 ppm improves yield strength by 7%, ultimate strength by 3%, and hardness by 10% for annealed condition, whereas for normalized condition the absorbed energy increases by 18%. Although it has a widely known effect on grain refinement, Nb showed no consistent effects on this feature. For properties in which there was no substantial improvement, Nb presented no deleterious effect.

Keywords: Microalloyed steel; Cold forging; Annealing; Normalizing.

1 Introduction

Steel has been the mainstay of the automotive industry since its inception, and despite competing materials like composites or the use of lighter metal alloys such as aluminum and magnesium, to this day steel remains the most used in vehicle production [1]. Generally, the goal is to achieve high strength levels without sacrificing other properties like ductility and toughness [2]. Among several alternatives, many components undergo a carburizing process to obtain these in-use properties (high hardness at the surface combined with a more ductile, strong, and durable core). Case hardening also induces compressive residual stress at the surface, thereby improving fatigue resistance [3].

According to Wise and Matlock [4], different parameters influence the bending fatigue endurance limit. Based on the statistical analysis of a large set of experimental fatigue data, their results indicated that the most effective metallurgical way to improve fatigue endurance was refining prior austenite grain size in both the core and the hardened case. For adequate mechanical properties and durability of carburized steels, the following key requirements must be complied with: adequate chemical composition for grain size stability and hardenability, macroscopic and microscopic cleanliness, and homogeneity [5].

Microalloyed steels, also known as high strength low alloy (HSLA) steels, emerge as an answer to this issue. They are a group of steels that contain small quantities of microalloying elements such as niobium, vanadium, titanium, either as a single addition or in combination. Generally, the total amount of microalloying elements in these steels is less than 0.2% [6]. Combined with the forging process, the addition of niobium, titanium and vanadium at levels of up to 0.15% by weight promotes solid solution hardening and grain refinement. By forming finely dispersed carbonitrides in the matrix during the austenite-ferrite transformation, they are effective in increasing mechanical strength and tenacity [7]. Niobium is one of the most effective elements for improving steel strength and toughness [8]. One of the important uses of Nb in steels relates to grain pinning effect

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by the niobium carbonitride precipitates formation. Also, niobium in solid solution acts as a solid solution hardener and retards the recovery of dislocations in steel [9].

Conventional high strength forging steels are necessary for producing automotive power train and chassis parts. Most of these steels are usually subjected to quenching (heat-treatment) and tempering after cold or hot forging to increase yield stress, impact toughness, fatigue strength, etc [10]. The preferential use of cold forging as a viable alternative to hot forging is due to increasing interest in near-net-shape processing and product quality enhancement. With the potential for reducing production costs along with recently improved cold forging processes, many carburized part manufacturers favor near-net-shape cold-forging processes rather than the more expensive conventional hot-forging and extensive machining processes [11].

Forming processes are highly dependent on steel ductility which is completely conditioned by the initial steel microstructure [12]. Industrially, steels in the raw rolling condition present a microstructure resulting from continuous cooling (composed basically of ferrite, pearlite and/or bainite). These constituents do not present sufficient ductility characteristics for cold forming processes, and are dependent on subsequent annealing or spheroidization treatments [13]. During these treatments, the microstructural matrix (normally composed of ferrite and cementite) softens via the spheroidization or globularization process of the cementite. This spheroidized microstructure is known to improve the cold formability of the material, ensuring the imposition of high plastic deformation with a reduction in the forming load during the manufacturing process.

Cold forged components require control of austenite grain size during long-time high-temperature carburizing treatments. Cold work can increase the susceptibility to abnormal austenite grain growth. To control this factor, microalloying has been used to form stable precipitates that limit grain boundary movement [14].

The as-forged steel microstructure is generally composed of coarse ferrite and banded pearlite which consequently lead to limited impact toughness. Given the characteristics of the forging process, conventional technology such as normalizing heat treatment is necessary for grain refinement to improve toughness and machinability [15].

Hence, this work investigated and compared the metallurgical and mechanical behavior of SAE 4320 as standard and with Nb addition at different heat treatments of carburized automotive component production (after annealing and normalizing). By analyzing the grain size, steel microstructure and the values of tensile, hardness, and impact tests, it evaluated the influence of Nb addition on these properties.

2 Materials and method

This study analyzed two steels, one SAE 4320 standard and another with Nb added as a microalloying element. Both were produced in an electric arc furnace (EAF) followed by vacuum degassing (VD) and continuous casting. Table 1 presents the steel chemical composition. C and S values were obtained using the combustion and inert gas fusion technique with LECO CS 230, according to ASTM E1019 [16]. For the other elements, an ARL Model 4460 optical emission spectrometer was used in accordance with ASTM E415 [17]. The samples were obtained during the continuous casting process. Importantly, the elements presented in the table refer to the addition of intentional elements in the steel manufacture. However, some other elements may have been incorporated unintentionally due to the steel manufacturing process.

The billets produced in the casting process have nominal size of 240 x 240 mm. After having the billets rolled for a size of 155 x 155 mm, they were grinded and then rolled for round bars with final dimension of 57.15 mm. Both rolling processes are characterized as conventional. For final rolling, the billets were reheated at a temperature around 1055 °C (average value), standard deviation of 8 °C, for approximately 2 hours. These values correspond to the measurements of the first pyrometer located immediately outside the heating furnace.

Steel microstructure was evaluated in specimens cut cross-sectionally. Microstructure revelation was achieved through etching with a 2% Nital reagent to facilitate phase identification. Images were captured using an Olympus microscope, model BX51M, attached to a Leica camera DMC2900 using Leica Software LAS V4.13. Austenitic grain size was evaluated using samples from the middle radius of a randomly selected section of the bar.

The testing methodology followed the ASTM E112-13 [18] standard for direct hardening steels (885 °C for 1 hour). Austenitic grain size was determined using an Olympus BX60 microscope, along with the Leica QGrain application from Leica Microsystems Imaging Solutions Ltd.

Six samples, each around 300 mm in length, were randomly taken from the bars for mechanical testing.

Table 1. Chemical composition of steels used in this study and SAE specification, wt%. For Al, Nb and N, the values are in ppm

Steel	C	Mn	P	Si	Cr	Ni	Mo	S	Al	Nb	N
SAE 4320	0.17-0.22	0.45-0.65	≤0.030	0.15-0.35	0.40-0.60	1.65-2.0	0.20-0.30	≤400	-	-	-
4320 STD	0.18	0.57	0.017	0.23	0.53	1.74	0.24	<300	200 to 300	<100	100 to 200
4320 Nb(1)	0.20	0.59	0.017	0.23	0.53	1.70	0.21	<300	200 to 300	200 to 500	100 to 200

Five of them underwent tensile tests on the sample cores following ASTM A370 standard, using a Kratos ECC universal testing machine with a 50,000 kgf capacity and strain gauge.

Hydraulic grippers prevented slippage, and the TRClv614301 system handled data control and analysis [19]. The final sample was used for hardness and impact tests. Impact test was performed in accordance with ASTM E23-18 using a Heckert impact machine with a capacity of 294 J [20]. HB hardness measurement was conducted with a Durometer from Shimadzu Seisakusho LTD, using a ϕ10 mm ball indenter with a maximum capacity of 3,000 kgf, according to ASTM E10-18 [21]. Hardness analysis was performed in triplicate on the middle radius of the bar sample's cross-section and the impact test was performed in quintuplicate on test specimens from the same sample region.

Intending to replicate the production of carburized automotive components, the bars followed the typical production flow of a carburized automotive component (Figure 1). Although the flow presented exemplifies all the processes that a carburized automotive component is subjected to, we emphasize that our objective here is to characterize the steels after the annealing and normalization heat treatments. Figure 2 details the thermal processing. Figure 3 shows the dimensions of blanks (after cutting) and forged parts. After forging, there was no further change in part geometry. Thus, steel characterization after annealing was done on steel bars and after normalization on parts in the hatched region (Figure 3).

3 Results and discussion

Using the Jmat Pro software, it calculated the AC_1 and AC_3 temperatures for the steels studied to characterize the annealing process. Thus, 703 °C for AC_1 and 787 °C for AC_3 were obtained for 4320 STD and 704 °C and 785 °C for 4320 Nb(1). Comparing these temperatures with that during annealing, it can consider that in both cases the real temperature of 850 °C was higher than AC_3, characterizing full annealing. Similarly, normalization must be performed above the AC_3 temperature, as evinced by the data obtained.

Figure 4 shows the cross-sectional microstructure characterization for steel 4320 STD and 4320 Nb (1) after the annealing process and Figure 5 after the normalization heat treatment, both using Nital etching.

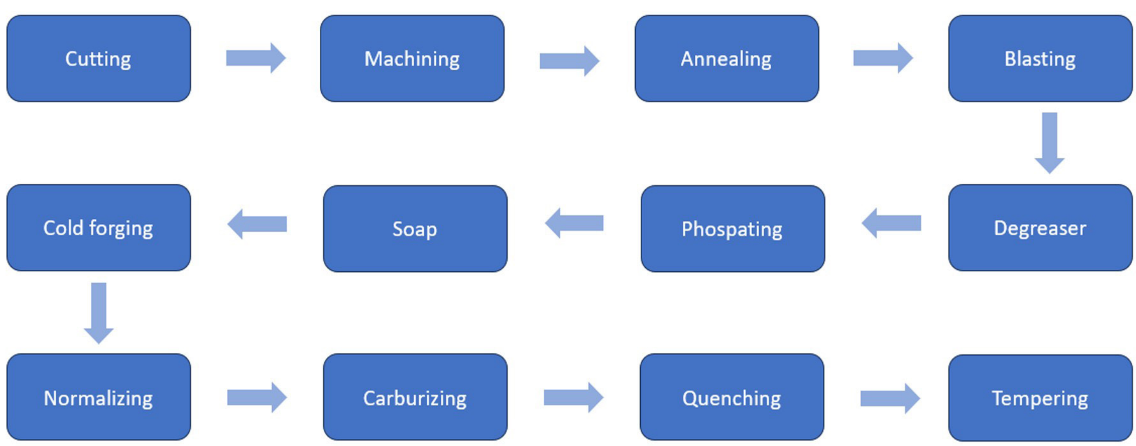

Figure 1. Production flow of a cold forged carburized automotive component.

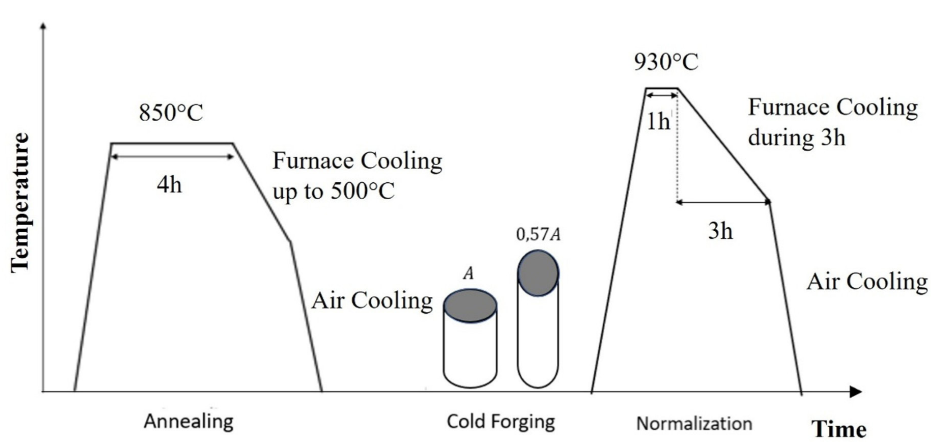

Figure 2. Summary of thermomechanical processing histories for studied steels.

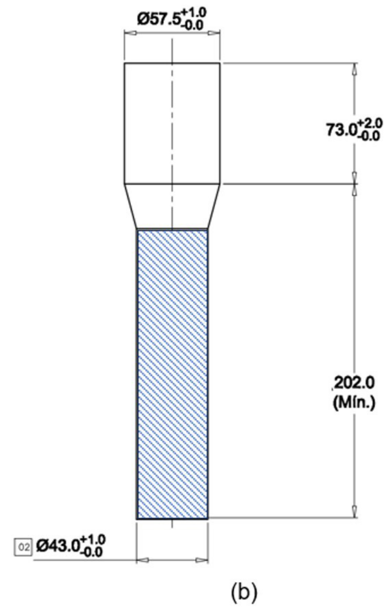

Figure 3. Dimensions of parts. (a) represents the blank (after cutting) and (b) the part after forging.

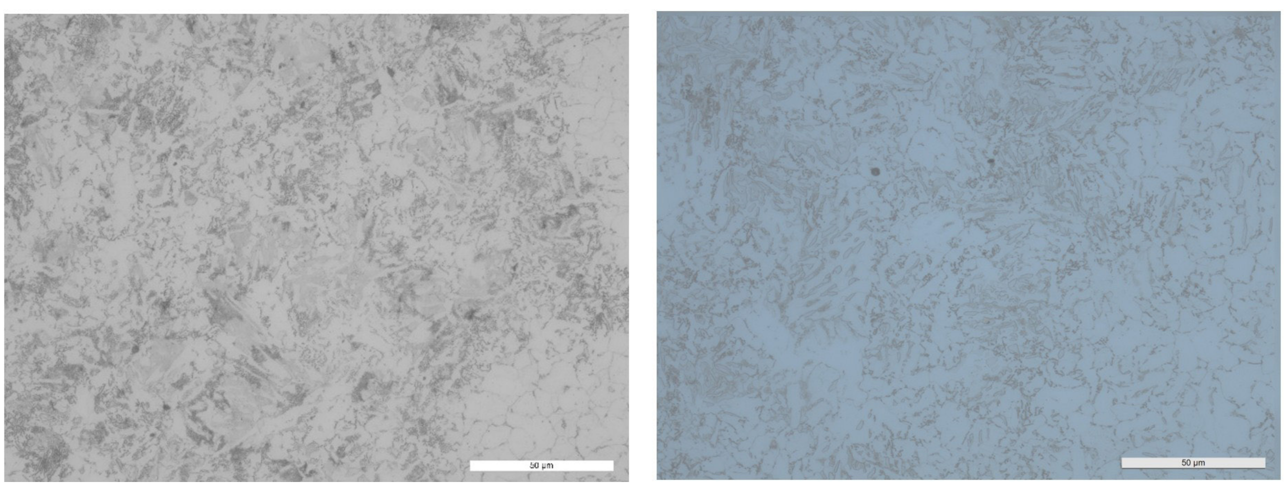

Figure 4. Cross-section microstructure using 2% Nital etching after annealing of 4320 STD steel (left) and 4320 Nb(1) steel (right). Magnification: 500x.

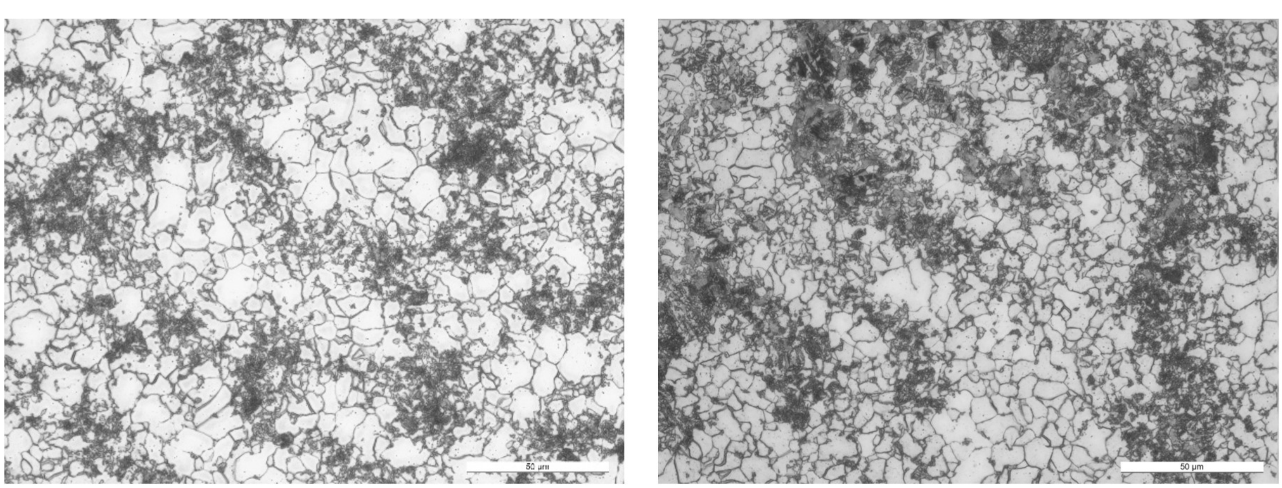

Figure 5. Cross-section microstructure using 2% Nital etching after normalizing of 4320 STD steel (left) and 4320 Nb(1) steel (right). Magnification: 500x.

After the annealing heat treatment, cementite spheroidization is expected to occur; however, the spheroidization rate is conditioned by the starting microstructure. When comparing a fine pearlitic matrix with a coarse one, fine pearlite has a greater spheroidization potential. In turn, when comparing martensitic, bainitic and pearlitic microstructural matrices, martensite has the greatest spheroidization potential [13]. Between pearlite and bainite, bainite shows a slight advantage. This aspect was analyzed by Kamyabi-Gol and Sheikh-Amiri [22], who showed that the levels of defects in martensite enhance the spheroidization rate, and extrapolated this characteristic to bainitic and pearlitic microstructures so that points with defects in the pearlite lamellarity have a greater spheroidization potential. As the steel studied was obtained through conventional rolling, there is a strong tendency for bainite to form during cooling after rolling which may explain the lack of formation of perfect cementite spheres (Figure 4).

Normalizing is often used as a preliminary treatment prior to other thermal processes (e.g., quenching and tempering) to ensure a suitable initial microstructure. This process promotes a more uniform distribution of alloying elements and carbon, eliminating segregations and heterogeneities that may occur during solidification [23].

As shown in Figure 5, the microstructure obtained after heat treatment is in the form of fine pearlite and ferrite. Grain size was evaluated using Picral etching and Leica's software based on 15 field's analysis that generate a grain size distribution plot (Figure 6). Annealing results indicate that the grain sizes for steel with and without Nb are similar, with average grain size of 10.9 and 10.4 for 4320 STD and 4320 Nb(1) steel respectively (both ASTM grain sizes). Nb was not expected to cause any grain size effect in this condition since the objective of the heat treatment was to coalesce the cementite to decrease material hardness. However, in the normalizing condition, average grain size for 4320 STD steel was 10.9 and for 4320 Nb(1), 10.7. Our data showed no significant difference in grain size that would justify the presence of Nb in the steel. One factor that may explain this issue is heat treatment temperature. When the normalizing temperature is in the γ (austenite) region, relatively homogeneous ferrite and pearlite grains can be obtained. With the increase of normalizing temperature, both the ferrite and pearlite grain size increase gradually, while steel strength and toughness first rise and then drop. In the literature, the excellent combination of mechanical properties was obtained at the normalizing temperature of 880 °C [15] which is a lower temperature than that used in the present study.

The mechanical characteristics were evaluated by analyzing tensile properties, impact resistance, and hardness to determine whether the addition of Nb influenced these properties. A statistical assessment of these attributes was performed using Student's *t*-test, the results of which are presented in Table 2 and Table 3 for annealing and normalizing conditions, respectively. A 0.5 significance level (α) was adopted to indicate the probability of rejecting the null hypothesis (H_0) that the mean values of the analyzed

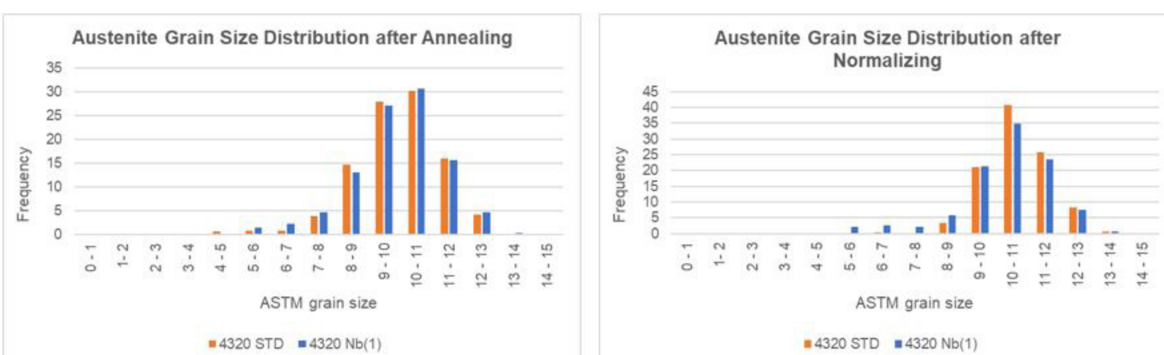

Figure 6. ASTM grain size distribution for both steels after different types of heat treatment.

Table 2. Mechanical properties and p-values from Student's t-test after annealing condition

Property	4320 STD	4320 Nb(1)	p-value
Yield strength (MPa)	332.9±9	368.9±9	0.000
Ultimate strength (MPa)	577.9±5	597.5±6	0.003
Elongation (%)	32.22±2.7	27.27±2.2	0.015
Reduction of area (%)	55.77±1.7	56.60±5.5	0.761
Absorbed energy (J)	128.86±3.1	122.16±9.7	0.156
Hardness (HB)	151±2	173±2	0.000

Table 3. Mechanical properties and p-values from Student's t-test after normalizing condition

Property	4320 STD	4320 Nb(1)	p-value
Yield strength (MPa)	408.2±21	438.6±6	0.031
Ultimate strength (MPa)	639.4±36	601.7±18	0.124
Elongation (%)	28.71±3.7	31.10±1.6	0.234
Reduction of area (%)	51.00±10.7	58.33±5.4	0.219
Absorbed energy (J)	94.51±3.5	115.79±8.7	0.001
Hardness (HB)	154±7	171±2	0.048

characteristics are identical between the different steels. If the p-value is greater than α, H_0 is not rejected, indicating no significant difference between the steels for that property. If the p-value is less than or equal to α, H_0 is rejected.

After annealing, based on the values obtained, there are no significant differences between the steels regarding area reduction and absorbed energy as indicated by their p-values, all of which are greater than the significance level. In contrast, yield strength and hardness show a significant difference between the 4320 STD and 4320 Nb(1) steels, with a p-value of 0.000.

This indicates that Nb addition a statistically significant effect on these properties, as 4320 Nb(1) exhibits substantial improvement. Likewise, ultimate strength has a p-value of 0.003, further confirming the impact of Nb. Although elongation has a p-value that ensures a difference between steels, this was the only property in which the standard steel had a higher value outcome, indicating that Nb presence did not improve this property. This result is directly linked to the grain size values found—the grain averages were quite similar, with the standard steel presenting a slightly higher value, which may justify the greater elongation.

As for the normalizing condition, we observed no significant differences between the steels regarding yield strength, ultimate strength, elongation, and reduction of area as indicated by their p-values, all of which are greater than the significance level. Energy absorbed and hardness are the properties with statistically significant differences between the steels. In this case, the temperature used in the heat treatment may have influenced the lack of differentiation.

The Nb effect of Nb under different heat treatment conditions appears to be moderate in the initial manufacturing steps of a carburized automotive component. While the differences in properties analyzed were not statistically significant, further investigation of its effects in the case-hardened state could yield valuable insights. Examining Nb influence on fatigue life may reveal additional enhancements in material properties, underlining the potential benefits of Nb in steel formulations for automotive applications.

4 Conclusions

Steel strength is significantly influenced by its chemical composition; however, steelmaking processes employ several strategies to increase strength, including alloy design, controlled cooling, and precipitation hardening. The expected benefits of adding Nb to steel include improvements in mechanical properties, specifically increased strength, and toughness. The manufacturing process of a carburized automotive component requires several processing steps to ensure in the final product properties suitable for automotive applications.

Statistical analyses indicate that Nb addition between 200 and 500 ppm in 4320 steel results in increased yield strength by 7%, ultimate tensile strength by 3%, and hardness by 10% after annealing. For normalization, absorbed energy increased by 18% and hardness, 10%. Austenitic grain size showed very similar averages between the steel with and without Nb in both heat treatment conditions. For all properties, in cases where there was no significant improvement with Nb, they showed similar values for steel with and without Nb.

Acknowledgements

Study conducted at Gerdau S.A. in Pindamonhangaba, SP, in partnership with the Laboratory of Mechanical Behavior of Metals at Federal University of São Paulo and Federal University of ABC. The authors would like to thank FINEP, FAPESP, CAPES, and CNPq for their financial support.

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Received: 14 Jan. 2025
Accepted: 11 Mar. 2025

Editor-in-charge: Sabrina Arcaro