INFLUENCE OF THE PROCESS PARAMETERS OF INTERCRITICAL ANNEALING IN PRODUCTIONS OF STEELS FOR HOT STAMPING *

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Abstract
Currently, continuous implementation of safety and CO2 emission standards in automotive vehicles has driven the search for weight reduction and safety enhancement solutions. In this context, development of steels high mechanical strength has been presented as the most applicable. Due to the strong elastic return after the stamping operations, parts that have geometric complexity and require greater mechanical strength are produced by hot stamping. The steels employed have addition of boron, such as 22MnB5. Parallel to the development of steels, researches related to the development of metallic coatings capable of withstanding mechanical stresses and heat treatment have been carried out. During the process of manufacturing the steels for the hot stamping some control points become extremely necessary. These points are distributed throughout the production chain, ranging from the control of the chemical composition, either by the elements responsible for temperability (C, Mn, B, etc.), as well as to the cold rolling and subsequent annealing and galvanizing processes. In the present work, a study was carried out on the effect of the process parameters of a continuous annealing and application of metal coating by hot immersion in the mechanical and microstructural characteristics of 22MnB5 steel for the hot stamping process. The samples were subjected to dilatometry simulations with fast cooling by helium injection. The results by simulation were compared to the industrial results, being evaluated by microhardness, uniaxial traction and metallographic analyzes
Keywords: Intercritical annealing; Dilatometry; Hot stamping.

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1 INTRODUCTION

In recent years, the implementation of new standards and regulations related to safety and emission of pollutants in automotive vehicles has driven the search for technological solutions capable of reducing vehicular weight and at the same time increasing levels of safety. Within this context, the development of Advanced High Strength Steel (AHSS) steels has been showing increasing demand from the automotive industries.

The main difference between AHSS steels and conventional steels is related to the microstructure [1]. The AHSS steels are multiphase steels that can present a microstructure consisting of ferrite, martensite, bainite, and / or retained austenite, depending on the added alloying elements and the process parameters used during the rolling and annealing. In AHSS steels the conventional cold stamping process is generally limited to the production of parts having relatively simple geometry and easy dimensional control.

For applications that present complexity geometric and require steels with higher mechanical strength, it was necessary to optimize hot stamping technology. The steels used in the hot stamping process generally have boron and / or chromium addition [2]. Obtaining suitable mechanical properties in high strength steels depends on both the chemical composition and the process parameters employed, so the operational variables gain a significant prominence due to the diversity of the hardening mechanisms involved (solid solution, precipitation, grain and density of discordances).

The use of a continuous annealing cycle employing intercritical annealing temperatures (temperatures between transformation temperatures A1 and A3) becomes an essential step for the production of steels with biphasic microstructure, especially for steels inserted in the AHSS group. In the specific case of steels for hot stamping, such as 22MnB5, the presence of martensite is not desired after continuous annealing since the final mechanical characteristics are obtained during the hot stamping process by the quenching heat treatment. The properties of a steel after hot stamping have a strong dependence on the initial microstructure [3,4]. This dependence is related to the size and morphology of the austenitic grains that are formed during the blanks austenitization process.

In turn, they are influenced by the initial microstructure of the material after the annealing process, which can present several microstructural constituents according to the process parameters used. This problem is directly related to the control of the cooling rate used during the steps of an intercritical annealing cycle, since high cooling rates favor the formation of a microstructure composed of martensite [5, 6].

High strength biphasic steels are produced using intercritical annealing cycles where the temperatures employed can range from 750 to 830°C depending on the chemical composition. The continuous annealing process for the production of biphasic steels follows the following process steps: heating to the intercritical temperature, soaking to allow nucleation and austenite growth and cooling for austenite transformation. The amount and morphology of the constituents formed in the microstructure of the steel depend on the cycles applied. Since the primary ferrite and austenite fractions formed depend on the heating and soaking temperature. The fraction of the ferrite / austenite in equilibrium can be calculated by applying the rule of the lever in the phase diagram in the Fe-C-M system, where M represents the effect of the alloying elements [7].

Through dilatometry simulations, the effect of the process parameters of an intercritical continuous annealing on the mechanical and microstructural characteristics of the 22MnB5 steel was evaluated. In order to determine a better process window for the production of this steel, the effect of the annealing temperature and the process speed employed was evaluated, thus obtaining different cooling rates. In the same way, two different designs of cooling cycles were tested, thus modifying the residence time of the steel in the equalization zone during the annealing process.The results obtained by dilatometry were compared to the industrial results, thus determining the best process window for 22MnB5 steel production.

2 MATERIAL AND METHODS

To determine the temperatures to be used in the intercritical annealing tests, it was necessary to know the transformation temperatures of the material under study (Ac1 and Ac3). For the determination of the temperatures Ac1 and Ac3 dilatometric tests were carried out and compared with the calculated temperatures, based on existing equations in the literature. Dilatometry simulations were performed on samples obtained in coils in the "Full Hard" condition, coil only as cold rolled. Samples were cut into small squares of approximately 5 mm for dilatometry simulations. The tests were performed using a Bahrs DT 1000 dilatometer with Argon cooling. The chemical composition of the studied steel (22MnB5) is shown in table 1.

Table1: Chemical composition of 22MnB5 steel used in this work (% by weight).

Elemento	C	Mn	Si	Al	Ti	P	B	Cr
%	0,240	1,800	0,276	0,050	0,038	0,019	0,005	0,190

Another important point in defining the thermal cycles to be tested is based on the Heating and Cooling rates of the continuous annealing line studied. The rates of heating and cooling have a dependence on the process speed as well as the length of the specific zones and are thus determined by means of approximate calculations. For the analysis of the effect of the intercritical annealing temperature on the mechanical and microstructural properties of the 22MnB5 steel, the use of two temperatures of 750 and 780 ° C, respectively, was stipulated. The process speed at which the coil travels throughout the galvanizing line has been kept constant and equal to 100 m / min. The maximum process speed targeted for adequate productivity is limited by the heating capacity of the annealing furnace. With the aim of evaluating the appropriate process speed to ensure the desired properties and at the same time a higher productivity, it was fixed at annealing temperature of 780 ° C and speed varied, 60, 100 and 150 m / m. Based on the length limitations of the cooling tower of the galvanizing line studied, simulations were performed for two specific cooling profiles: "Standard Cooling Profile" and "Short Cooling", use of forced cooling at the beginning of the cooling zone.

For samples obtained by dilatometry, mechanical and metallographic tests were carried out at five points defined during the annealing cycle, as shown schematically in figure 1 through the red cooling arrows. After the heat treatment, the samples were characterized with respect to the mechanical properties by means of the Vickers microhardness test in a Buehler Micromet 2100 electromechanical microdurometer, with a pyramidal indenter using a load of 1 kg. For each sample obtained, five measurements of microhardness were performed, being considered for comparative analyzes and represented in the following tables only the mean value. Likewise, for each defined step, metallographic analyzes were performed by optical and scanning electron microscopy. The samples were attacked by LePera chemical reagent to determine the volumetric fraction of martensite. In this way it was possible to estimate the percentage of austenite present in each stage of the thermal cycle and consequently to understand its transformation throughout the total cycle. For microstructural characterization, a Zeiss AXIO optical microscope (MO) and a scanning electron microscope JEOL 6360 (SEM) were used.

The industrial samples were collected only after the complete annealing represented by point 5 in figure 1. The sampling process was performed in three positions along the length of the coil. In order to better evaluate the results on an industrial scale, uniaxial traction tests were performed, and the results of the Yield Strength (YS) and Tensile strength (TS) and Total elongation (Telong) were evaluated. The results were compared statistically using the same method presented for calculations of microhardness values. The tensile analyzes were performed on a Universal Instron 5585 machine with a maximum load of 100KN, following the standard of DIN-EM 10002 in the longitudinal direction to the rolling direction at room temperature.

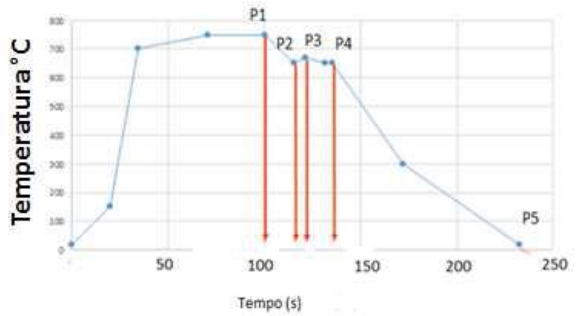

Figure1: Schematic representation of the annealing thermal profile highlighting the five sampling points and characterization throughout the process.

3 RESULTS AND DISCUSSION

The results of simulation performed in the laboratory on 22MnB5 steel samples submitted to the different annealing parameters will be presented at three different points of approach. Following will be presented the results of the industrial tests, as well as the comparative results between simulation and industrial tests.

The values of the phase transformation temperatures obtained by calculations through equations in the literature [8,9,10,11] are shown to be close to the actual values obtained by dilatometry. The main divergences of results can be explained by the absence of some alloying elements present in the chemical composition of the steel used and which are not considered in the formulas used. The values of the transformation temperatures calculated and obtained through dilatometry tests can be visualized in table 2.

Table2: Values of the transformation temperatures calculated and obtained through dilatometry tests.

	Equação	Temperatura calculada (°C)	Temperatura por Dilatometria (°C)
Ac1 (°C)	Andrews – 3.1	722	735
Ac3 (° C)	Andrews - 3.2	834	855
Ms (°C)	Andrews - 3.3	399	400
Ms (°C)	Stevens – 3.4	405	400
Ms (°C)	Stuhlmann – 3.5	416	400
BS (°C)	Honeycombe – 3.6	645	-
BS (°C)	Li – 3.7	575	-

3.1 Effect of annealing temperature

Using the values of cooling rate and times in the annealing steps calculated, the samples were submitted to the simulation of heat treatment with intermediate tempering by dilatometry using annealing temperatures of 750 and 780°C, for each point analyzed, according to figure 1. The average values of microhardness (µ Hardness) and standard deviation (σµ Hardness) found are shown in table 3. The values of the austenite fraction through the measured martensite fraction and standard deviation (σaustenite) are also shown in table 3.

Table 3: Results of microhardness and volumetric fraction of martensite in samples treated by quench interrupted by dilatometry.

	Temp. °C	750°C - 100m/min				780°C - 100m/min			
Pontos Analisados		µ Dureza (HV_1)	$\sigma_{\mu Dureza}$	% Austenita	$\sigma_{austenita}$	µ Dureza (HV_1)	$\sigma_{\mu Dureza}$	% Austenita	$\sigma_{austenita}$
Encharque (P1)	750 /780	245,8	3,60	22,5	3,50	316,0	4,30	60,3	2,50
Resfriamento Rápido (P2)	650	237,6	3,40	19,7	2,20	279,2	3,20	37,4	4,20
Inicio Zona Equalização (P3)	670	226,8	1,50	9,1	1,90	246,6	2,60	29,6	3,60
Pote Revestimento (P4)	660	212,8	2,50	2,2	0,70	216,8	2,00	13,0	1,90
Ciclo completo (P5)	40	192,4	0,90	0	0,0	198,2	2,90	0	0,0

Analysis carried out in the first point, after the heating and soaking zones (Point 1), showed that at the temperature of 750 ° C the transformation of approximately 22.5%

of the initial matrix into austenite occurred. An increase of 30 ° C at the annealing temperature, 780 ° C, resulted in the addition of the austenite fraction of 60.3%. According to Mohanty et al. [12], higher temperatures provide the greater fraction of austenite, with the formation of austenite governed by the process of diffusional transformation that occurs by nucleation and growth. Consequently the microhardness value found at the annealing temperature of 780 ° C was approximately 70HV1 higher than the value found at 750 ° C (316.0 and 245.8 HV, respectively). When analyzing Point 2 (rapid cooling zone) it was observed that annealed samples at 750 ° C showed a 12% decrease in their initial austenite fraction (martensite). At the intercritical temperature of 780 ° C, the fraction of austenite (martensite) found in Point 2 was about 40% smaller than its initial fraction. The greatest difference found at the temperature of 780 ° C can be attributed to the sum of the annealing temperature and cooling rate effects that the samples were submitted (between the points P1 and P2).
The microhardness variations along the analyzed points are directly related to the austenite fraction present. The possible effect of recrystallization and grain size after soaking, as well as effect of the carbides present were not evaluated in this work.
From the points P4 and P5, region represented by the exit of the metal strip from the coating pot until the end of the cooling tower descent, the difference between the microhardness values found for the analyzed annealing temperatures becomes less significant. At the end of the process, point P5, the microhardness values are relatively similar for the two simulated temperatures. The similarity of microhardness results found can be explained by the absence of microstructural constituents with higher mechanical strength (martensite, bainite). For at the end of the process the material becomes only ferrite and perlite.

When evaluating the results found in the uniaxial traction test, it was possible to observe an increase in the values of YS and TS and consequently a reduction in Total elongation values when elevated the annealing temperature from 750 ° C to 780 ° C, Table 4.

Análise ANOVA - Temperaturas					
Propriedade	Temperatura	Médias	Desvio	Hipóteses	Valor -P
LE	750	480,24	10,45	Ho: μ 750°C = μ 780°C	0,372
	780	487,55	16,6		
LR	750	567,46	9,04	Ho: μ 750°C = μ 780°C	0,000
	780	608,24	12,16		
ALT	750	23,657	1,35	Ho: μ 750°C = μ 780°C	0,000
	780	21,91	1,89		

Table 4: Comparative statistical analysis applied between the averages of the mechanical tensile results in coils annealed at 750 ° C and 780 ° C.

The variation of results found was higher for the TS, approximately 40 MPa, than for the values of YS that were not higher than 10 MPa. Because the values obtained at the temperature of 750 ° C are within the range of deviation of the results obtained at the temperature of 780 ° C. For the values of TS and Total T.elong, no similarities were found between the average values for temperature of 750 ° C and 780 ° C. The values found for the significance factor (95% confidence) were lower than 0.05 (P-value).
The difference of mechanical properties found in the two temperatures tested can be explained through the metallographic analyzes. It was found that at the final point of the intercritical annealing process, the microstructure obtained presented only ferrite and perlite. It was not evidenced the presence of any more resistant phase coming from the transformation of austenite formed during the soaking stage. The samples from 750 ° C, it was found coarser perlite concentration in the ferritic matrix, distributed mainly in the ferritic grain boundary, when compared to the temperature of 780 ° C. It was not evidenced the presence of bainite in the regions of higher perlite concentration, as shown in figure 4.

The higher presence of coarse perlite tends to decrease the strength value, because it would be somewhat less carbon rich and thus more ductile [13]. The presence of a higher concentration of fine perlite at the temperature of 780 ° C is directly related to the cooling rates employed, especially during the fast cooling.

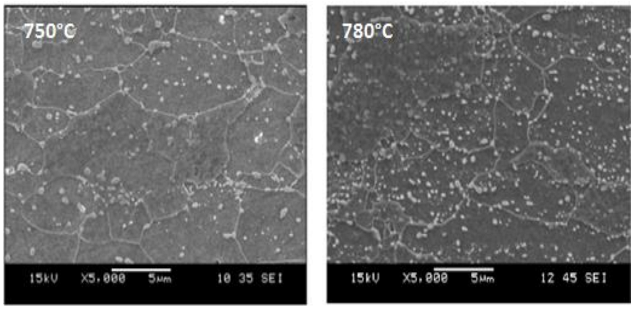

Figure4: Metallographic analyzes by SEM of samples annealed at 750 and 780 ° C.

3.2- Effect of process speed

The samples were submitted to the simulation by dilatometry at a temperature of 780 ° C, varying the process speed (60, 100 and 150 m / min) being cooled by f intermediate tempering. The average values of microhardness (μHardness) and austenite fractions (martensite) found were represented in the figures 5 and 6 respectively.

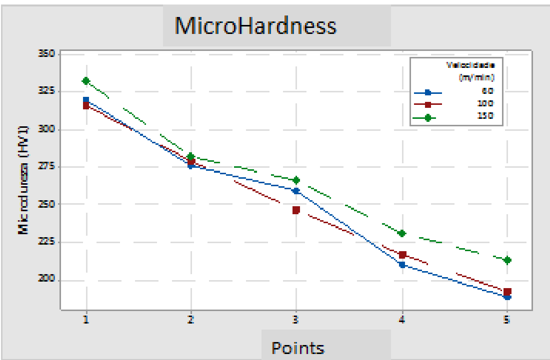

Figure 5: Representation of simulated austenite (martensite) microhardness for the process speeds of 60, 100 and 150 ° C.

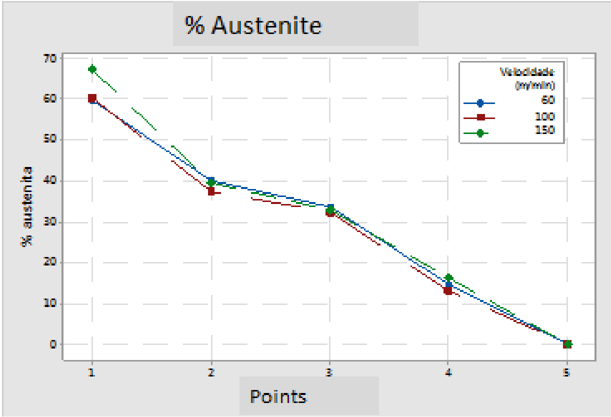

Figure 6: Representation of simulated volumetric fraction values for the process speeds of 60, 100 and 150 ° C.

Although the values of austenitic fraction similar to the end of the process (complete cycle), at the speed of 150 m / min the values of microhardness found at points P4 and P5 are slightly higher than the values found for the velocities of 60 and 100 m / min. This behavior can be attributed to the difference between the percentages of ferrite and perlite formed for the three lines speeds tested. Although the tested samples are submitted to the same process temperature, there is a difference in the cooling rate employed, thus promoting a difference in the formation of the ferrite and perlite fractions at the end of the process.

Comparing the industrial results obtained through uniaxial traction, it was observed that for the three speeds analyzed, the mechanical results obtained in the three speeds tested (Ho: μ60 = μ100 = μ140) showed an increase in the values of YS and TS (consequently a reduction in Total Elongation values) when the process speed is increased.

Figure 7 shows the microstructures of 22MnB5 steel produced at three different speeds, 60, 100 and 140m / min. It is observed the presence of thin perlite diluted in a ferritic matrix. When comparing the microstructures obtained at the speed of 60 / min and 100m / min it is possible to observe the existence of a slight difference in the perlite quantity.

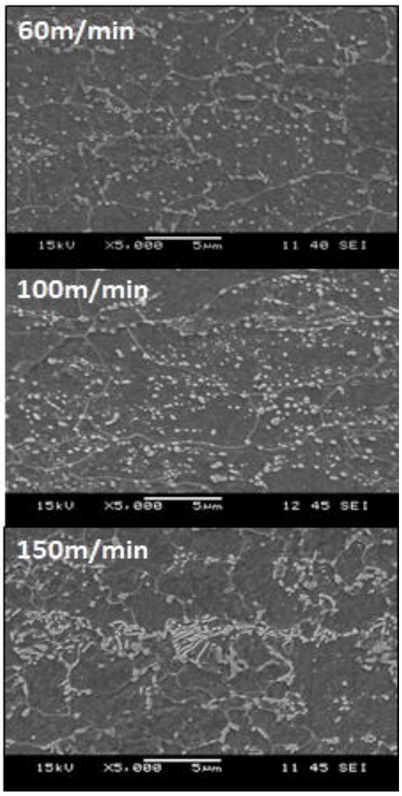

Figure7: Metallographic analyzes by SEM in samples annealed at 780 ° C, lines speeds of 60, 100 and 150m / min.

This difference observed in the two velocities analyzed was sufficient to increase the values of YS and TS [8]. The increase in the value of YS was considered not significant, since the average of the values obtained was considered equal by the statistical analyzes. For the TS, the increase caused by the percent of perlite was approximately 14 MPa.

For the process speed of 150m /min it was possible to verify the presence of bainite islands in an isolated way in the matrix. The presence of these bainite islands promoted an increase in the mechanical properties when compared to the velocities of 60 and 100m/min.

The presence of bainite islands is due to the higher cooling rate employed during the continuous annealing process. For the speed to 150m / min the cooling rate was 22 ° C /s, thus allowing the formation of bainite during the transformation of the austenite formed at the temperature of 780 ° C.

3.2- Effect of the cooling profile

Through the previous simulations the effect of the intercritical annealing temperature and the effect of the process velocity on the final microstructure as well as the mechanical behavior were verified. In all combinations of parameters analyzed, the presence of the austenite fraction higher than 10% at point P4 (point after the coating pot) was observed. As a consequence, the use of the standard cycles tested may limit the cooling capacity of the tower due to latent heat of transformation [14].

New simulations were performed based on the use of a forced cooling at the beginning of the cooling zone. Such a modification in the cooling profile, "short cooling", tends to increase the length of the equalization zone.In this process, the latent heat of transformation was sufficient to ensure that the metal strip enters the coating pot at the process temperature.

The simulations were performed using a speed of 100m / min, where the effects of temperatures of 750 and 780 ° C were compared in a standard cooling cycle and in a new cooling cycle.

In new tested configuration, the fast cooling zone is now considered to be only 4.40 meters and the equalization zone becomes approximately 16.95 meters. Different from the standard configuration we have a cooling zone of 15.40 meters and overageing with 5.95 meters. In this way the cooling rate applied to the new thermal configuration becomes higher, thus allowing the formation of a larger fraction of phases more resistant to the end of the fast cooling.

As expected, the samples submitted to the new cooling profile showed an increase in the austenite fraction (martensite) at the end of the fast cooling (Point2). However, for the annealing simulations at 750 ° C, the two cooling cycles tested showed a similar austenite fraction after immersion in the coating pot (point 4). For the annealing temperature of 780 ° C, the new cooling cycle ("short cooling") presented a fraction of austenite less than the standard cycle for the same point analyzed. This observation is explained by the increase of the equalization zone, since even with a higher fraction of austenite (martensite) at the end of the cooling, the increase in the length of the equalization zone provided a longer time for the transformation of austenite into ferrite and perlite.

The mechanical properties values obtained for the two different cooling profile showed the similar behavior. In this way it can be affirmed that the modification of the cooling profile in the first two zones did not interfere in the mechanical results at the end of the annealing process. When applying the "Short" cooling method, the fraction of austenite present at the end of the fast cooling zone is slightly larger when compared to the standard cooling cycle.

However, during the other stages of the annealing cycle, this difference did not influence the formation of more resistant phases in order to affect the mechanical properties values at the complete annealing cycle.

The metallographic analyzes performed in the samples submitted of the different cooling profile were not shown a significant increase in the perlite fraction at both annealing temperatures, as shown in Figures 8 and 9. Thus, it was confirmed that the modification of the cooling profile did not promote the formation of more phases resistant to the end of the annealing cycle.

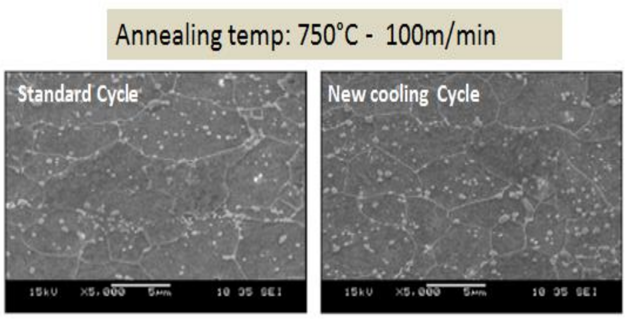

Figure8: SEM metallographic analyzes of annealed samples at 750 for the two thermal cooling profiles tested.

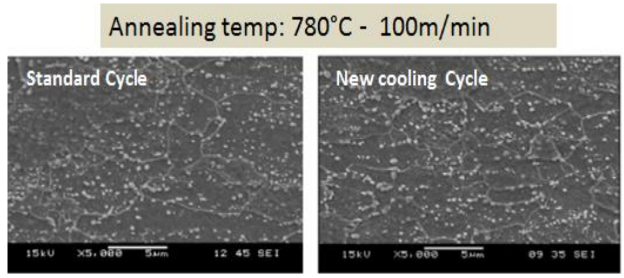

Figure9: SEM metallographic analyzes of annealed samples at 780 for the two thermal cooling profiles tested.

4 CONCLUSION

The results of mechanical properties obtained for the two annealing temperatures tested (speed of 100m / min) showed that for coils annealed at 780 ° C the mechanical properties were higher than 750° C. The variation of mechanical results is directly related to the presence of more refined perlite for samples annealed at 780 °C.

For results obtained in the three processes speeds tested (780 ° C), it was identified that coils produced at a speed of 150m / min showed values of mechanical properties slightly higher than the values obtained for the velocities of 60 and 100m / min. The difference found was related to the presence of more resistant phases (bainite) in samples produced at a speed of 140m / min. The presence of bainite after the complete annealing cycle can be attributed to a higher cooling rate applied when compared to the other simulated speeds.

The values of mechanical properties obtained for the two thermal cooling profiles tested were similar, regardless of the annealing temperature used. No changes were observed in the microstructure or in the phase fractions present in the samples after the complete annealing cycle.

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