Microstructural evolution during continuous annealing of a 980 MPa cold rolled steel grade

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Abstract. The development of advanced high strength steels (AHSS) is motivated by automotive industry demands for higher strength steels with better formability and specific stretch-flange formability to avoid fractures during parts forming. In response to this, the new steel grades must have special microstructures in terms of constitution, morphology, distribution and hardness. Thus, a better understanding of formation mechanisms and the characterization of these microstructures has shown to be of fundamental importance for the development of new steels. In this study the influence of heating rate and intercritical temperature on microstructural evolution and hole expansion ratio of a cold rolled 980 MPa AHSS grade was evaluated, using a Dilatometer and a Gleeble machine. The stretch-flange formability was evaluated by hole expansion tests and the microstructures were characterized using scanning electron microscopy, electron backscatter diffraction (EBSD) and nanohardness measurements. The increase of heating rate resulted in an overlap between ferrite recrystallization and austenite formation, changing the morphology of austenite from random to fibrous distribution. On the other hand, higher intercritical annealing temperature resulted in more homogeneous microstructures and higher hole expansion ratios. This microstructural homogeneity, evaluated by nanohardness, suggested that the difference in hardness between ferrite and second-phase affects micro-void formation and crack propagation during hole expansion test.

1. Introduction

The development of advanced high strength steels (AHSS) is motivated by automotive industry demands for higher strength steels with better formability and specific stretch-flange formability to avoid fractures during parts forming. In response to this, the new steel grades must have special microstructures in terms of constitution, morphology, distribution and hardness. In this way, the best choice for the chemical composition and the schedule processing of the cold rolled steel sheet, especially in the continuous annealing stage, becomes important to achievement of desired microstructure.

The continuous annealing process includes heating to an intercritical temperature, followed by an isothermal holding and a subsequent slow cooling to a quench temperature, from which the material is rapidly cooled to an isothermal overaging temperature. Few studies have been reported on the microstructure evolution of cold rolled dual phase (DP) steels during continuous annealing under conditions similar to those used in industrial lines [1-2]. They suggest that ferrite recrystallization
during heating has a small effect on the subsequent austenite formation and decomposition. However, Huang et al. [3] have recently shown that the interaction between ferrite recrystallization and austenite formation can significantly affect the microstructure produced by the annealing. According to the authors, the heating conditions applied, specially the heating rate and the intercritical temperature can change the austenite formation kinetics and the resulting microstructure.

Thus, a better understanding of the austenite formation mechanisms and the characterization of these microstructures have shown to be of fundamental importance for the development of new steels. In this study the influence of the heating rate and the intercritical annealing temperature on the microstructural evolution and the final hole expansion ratio of a cold rolled 980 MPa AHSS grade was evaluated using a Dilatometer and a Gleeble machine.

2. Procedure
DP 980 (0.11C-2.3Mn wt. pct.) steel sheets received as industrially hot rolled material (HR) was used for this study. The HR steel was cold rolled from 3.3 mm to 1.6 mm (50 pct reduction) using a pilot scale rolling mill. For the austenite formation studies, dilatometry tests were conducted using test pieces of 4 x 10 x 1.6 mm cut out from the cold rolled sheets (CR) with the longitudinal direction of the test pieces aligned with the rolling direction. Continuous heating tests were performed with heating rates of 1°C/s and 100°C/s until 950°C. To analyze the microstructure during heating, samples were taken from interrupted heating at 760°C and 800°C and quenched with He gas. The cooling rate was approximately 230°C/s to ensure the complete austenite to martensite transformation during quenching.

In order to evaluate the influence of annealing temperature on microstructure and mechanical properties (tension, hole expansion and nanohardness), samples with 50 x 150 x 1.6 mm dimensions were taken from the CR sheets and subjected to continuous annealing cycles on a Gleeble thermomechanical simulator, with intercritical temperatures (IT) of 760°C, 800°C and 820°C. Figure 1 shows a schematic diagram of the thermal cycle applied.

![Figure 1. Schematic diagram of the thermal cycle simulated on Gleeble. IT: intercritical temperature](image)

Standard metallographic techniques were used to prepare samples for the characterization of the microstructure using a field emission scanning electron microscope (FESEM) and an optical microscope (OM). The samples were etched with LePera and 2 pct. Nital for OM and FESEM, respectively. Electron backscatter diffraction (EBSD) measurements were used to generate band slope maps using the Oxford HKL acquisition and analysis software. The FESEM for the EBSD measurements was operated at 20 kV and a step size of 0.15 μm.

In order to quantify the strength of the individual phases in the microstructure, nanoindentation tests were performed using a Hysitron TriboScope TS75 coupled to an atomic force microscope (SPM9600). A 5 x 9 array of indents (in total 45) spaced 3 μm apart was performed using a piezo-automated method. Indents were performed with a Berkovich tip under load-control to 7500μN using a 10 sec. linear loading, 3 sec. holding and 10 sec. linear unloading. The hardness values were generated by the machine software using the unloading portion of the load vs. displacement curves.
Each indent must be categorized on the etched microstructure as corresponding to ferrite or second phase.

Tensile tests were performed according to ASTM A370 (sub-size specimens) [4]. The mechanical properties were determined as an average of three specimens tested along the rolling direction. Stretch flangeability was measured by conical hole expansion tests (HET) carried out in an Erichsen formability testing machine (model 145-60, 1.000 kN tensile force) according to ISO 16630:2003 [5]. The HET consists of forcing a conical punch at 15 mm/min into a pre-punched hole until any crack extends through the test piece thickness. The blank holder force used for both the hole punching and expansion was 200 kN. The expanded hole diameter (Di) was measured in two perpendicular directions and the limiting hole expansion ratio (HER) is calculated in relation to the original hole diameter (Di) as described in the follow equation:

\[
\text{HER} (\%) = \frac{D_f - D_i}{D_i} \times 100
\]

3. Results

3.1. Dilatometry tests - Influence of heating Rate on Austenite formation

Figure 2 shows the first derivate of dilation curves for the continuous heating at 1°C/s and 100°C/s in black and gray lines, respectively. The stages prior to austenite formation, such as the lattice expansion in ferrite-perlite structures and recovery of deformed structures, take place at heating with similar behavior for both heating rate. A first clear sharp drop for both heating rates which refers to pearlite to austenite transformation. The austenite formation start temperatures (Ac1) were similar for both heating rates. However, the heating rate increase shifts up the austenite formation finish temperature (Ac3) by approximately 14°C.

![Figure 2](image_url)

**Figure 2.** First derivate of dilation curves during continuous heating with different heating rates.

Figure 3 shows the microstructure observed at the specimens quenched at 760°C and 800°C for the heating rates evaluated. The increase in the heating rate delayed the recrystallization process, changing ferrite from fully to partially recrystallized. As can be seen in the Figure 3c, for the 100°C/s heating rate the ferrite grains are still recrystallizing while austenite starts to nucleate and grow within the pearlite colonies. At the slow heating rate, a few austenite particles are observed at the ferrite grain boundaries, which grow in competition with the austenite grains nucleated in the pearlite. As a result, the austenite morphology changes from a random to a fibrous distribution as the heating rate increase. The total austenite fraction (martensite after quenching) increases from 36 to 52 pct. as the temperature increases from 760°C to 800°C, respectively. At 760°C the austenite fraction is smaller.
for the high heating rate (100°C/s). At 800°C the difference in the total austenite fraction obtained at the two heating rates becomes insignificant [6].

![Figure 3. OM and FESEM micrographs with LePera OM and 2 pct Nital, respectively, with different heating rates.](image)

**Figure 3.** OM and FESEM micrographs with LePera OM and 2 pct Nital, respectively, with different heating rates.

### 3.2. Influence of annealing temperature on mechanical properties and microstructure

Figure 4 shows the measured yield strength (YS), ultimate tensile strength (UTS), total elongation (TE) and hole expansion ratio (HER) for the conditions evaluated. It is evident that raising IT will increase the strength (YS, UTS) and decrease the ductility (TE) of the steel, while the HER increases.

![Figure 4. Mechanical properties (a) YS, (b) UTS, (c) TE e (d) HER with different intercritical temperature (IT).](image)

**Figure 4.** Mechanical properties (a) YS, (b) UTS, (c) TE e (d) HER with different intercritical temperature (IT).
As shown in Figure 5 at 760°C the microstructure consists of ferrite (F) and only martensite (M) as the second phase. For higher temperatures (800°C and 820°C) the microstructure shows a greater amount of second phase that includes M and bainite (B). These microstructures are in agreement with YS, UTS and TE results shown in Figure 4. On the other hand, the increase of HER for the condition of higher strength and lower ductility can be explained by the homogeneity of the microstructures. According to previous papers [7-8], a more homogeneous microstructure with a lower hardness difference between the constituents is appropriate to prevent the premature occurrence of voids and microcracks in the interfaces of the soft and hard phases.

![Figure 5](image)

**Figure 5.** Microstructural at different intercritical temperature (IT). (a) 760°C, (b) 800°C and (c) 820°C.

In order to better evaluate the microstructure morphology, band slope maps were obtained by EBSD scans for the 760°C and 820°C intercritical temperatures (Figure 6). The band slope value is proportional to the sharpness of the Kikuchi Pattern, which is related to the presence of the crystalline defects. A highly dislocated lattice is expected to have a low band slope value and a high nanohardness [9]. The band slope values are graded on a gray scale, with 0 being the darkest and 255 being the lightest. Thus, the degree of misorientation can be associated with the present phases, with ferrite being the constituent with the highest band slope value, and martensite the lowest value. For the samples at 820°C, the highest volume of second phase formed for higher intercritical temperatures was easily perceived by band slope maps (fig. 6b), as well as their distribution shifted to lower BS values (fig. 6c).

![Figure 6](image)

**Figure 6.** Band slope maps for (a) IT = 760°C, (b) IT = 820°C and (c) distribution curves of BS values in grey scale.
Average nanohardness and the standard deviation bars for ferrite and second phase constituents at 760°C and 820°C are shown in Figure 7. It can be seen that the ferrite nanohardness does not change significantly with the temperature increase. However, the nanohardness average and spread for the second phase for 820°C is lower than for 760°C. The ratio between second phase hardness and ferrite hardness is believed to influence the degree of strain partitioning in the microstructure [8] which explains the higher HER for the samples treated at 820°C.

![Figure 7. Average nanohardness values of the ferritic (F) and second phase (SP). Each error bar represents the standard deviation.](image)

4. Conclusion
In this study the influence of heating rate and intercritical annealing temperature on the microstructure and the stretch-flangeability of a cold rolled 980 MPa DP grade was evaluated.

The increase of the heating rate shifts the finish austenite transformation (Ac3) to higher temperatures and resulted in an overlap between ferrite recrystallization and austenite formation, changing the morphology of austenite from a random to a banded distribution.

On the other hand, a higher intercritical annealing temperature resulted in a more homogeneous microstructure and higher hole expansion ratio. The microstructural homogeneity, evaluated by nanohardness and EBSD techniques, suggested that the difference in hardness between ferrite and second phase affects micro-void formation and crack propagation during hole expansion test.

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