Abstract

The effect of microstructural refinement and intercritical annealing on the mechanical properties and work-hardening response of a low carbon St12 steel was studied. It was revealed that intercritical annealing of the ferritic-pearlitic sheet results in the formation of a coarse-grained DP microstructure with discrete martensite islands normally formed in place of pearlitic colonies, which results in the minor enhancement of mechanical properties with disappearance of the yield-point elongation. On the other hand, a fine-grained DP steel with chain-network martensite morphology can be obtained by intercritical annealing of the cold rolled martensitic microstructure, which shows superior work hardening rate, low yield ratio, and high tensile strength. In this way, it is possible to enhance the mechanical properties of St12 steel toward those of DP300/600 steel. Compared with the conventional DP350/600 grade, a significant enhancement in the work-hardening behavior can be achieved with acceptable strength-ductility balance compared with the usual trend seen in steels. As a result, it was concluded that cold rolling of the initial martensitic microstructure before intercritical annealing is a viable approach for processing DP steels with enhanced mechanical properties for industrial applications.

Keywords: Dual phase steels; Grain refinement; Chain-network martensite morphology; Mechanical properties; Strain hardening rate.

1. Introduction

High strength generally incorporates a lower formability, which effectively retards broad applications of steel sheets. Therefore, a variety of advanced high strength steels (AHSS) were developed \(^1, 2\). Among them, the low carbon dual phase (DP) steels with a duplex ferritic-martensitic microstructure are considered as the most common ones. The co-presence of martensite governs the good strength-ductility balance and high work hardening rate \(^3-5\).

The mechanical properties of DP steels are determined by the adjustment of the individual microstructural constituents in terms of type, morphology, and in particular volume fraction, size and spatial distribution in the microstructure \(^6-8\). It has been shown that fine-grained DP steels have much better mechanical properties compared with their coarse-grained counterparts. As a result, several thermomechanical processing routes have been used for grain refinement of DP steels \(^9-12\).
Intercritical annealing of the cold rolled martensite was discussed by Nakada et al.\textsuperscript{9,10} and the importance of the chain-networked martensite grains formed on grain boundaries in the recrystallized ferrite matrix was also discussed. Development of fine ferrite/carbide aggregate during subcritical annealing of the cold rolled martensite was studied by Azizi-Alizamini et al.\textsuperscript{11}, where the effect of the subsequent intercritical annealing on the development of fine-grained DP steel was discussed. Finally, the intercritical annealing of cold rolled DP microstructures has been considered by Karmakar et al.\textsuperscript{12} and Mazaheri et al.\textsuperscript{3}.

The largest category of low-carbon steels is flat-rolled products (sheet or strip), usually in the cold-rolled and annealed condition. The carbon content for these high-formability steels is very low, less than 0.10 wt. % C, with up to 0.4 wt. % Mn (e.g. St12 steel). Typical uses are in automobile body panels, tin plate, and wire products. The DP steels produced from these steels generally show low strength (~ 400 MPa)\textsuperscript{13}, which precludes their utilization in industrial applications. Therefore, microstructural modification of these steels is required to enhance their mechanical properties toward standard DP grades such as DP600. Accordingly, the present work has been dedicated to study the effect of microstructural refinement on the mechanical properties and work-hardening response of St12 steel with the chemical composition (wt. %) of 0.035C-0.268Mn-0.035Si.

2. Experimental Details

A St12 steel sheet (1.0330 according to DIN EN 10130) with the chemical composition (wt. %) of 0.035C-0.268Mn-0.035Si was received in the normalized condition. The $A_x$ and $A_y$ temperatures were respectively estimated as ~ 736 °C and ~ 890 °C based on the Hougardy equations as shown in Eq. 1\textsuperscript{13}. As shown in Fig. 1, the as-received sheet was austenitized at 1050 °C followed by water quenching to develop a dominantly martensitic microstructure. The as-received sample was intercritically annealed at 850 °C followed by water quenching to obtain the DP2 steel. The heating rate to the intercritical annealing temperature in all cases was ~ 30 °C/s.

\begin{equation}
\text{Hougardy} \Rightarrow A_x = 739 - 22C - 7Mn + 2Si + 14Cr + 13Mo - 13Ni
\end{equation}

\begin{equation}
\text{Hougardy} \Rightarrow A_y = 902 - 25C - 11Mn + 19Si - 5Cr + 13Mo - 20Ni + 55V
\end{equation}

Etching in the LePera’s reagent (1g Na$_2$S$_2$O$_3$ in 100 ml H$_2$O + 4 g picric acid in 100 ml ethanol) followed by 2% Nital solution was used to reveal microstructural features for optical microscopy (Olympus Vanox) and scanning electron microscopy (CamScan MV 2300 SEM). The tensile specimen was prepared according to JIS Z 2201 standard. The tensile tests were performed at room temperature using a universal testing machine under strain rate of 0.001 s\textsuperscript{-1}. Tensile tests were repeated once to insure the reproducibility of results. The work-hardening rate ($d\sigma/d\varepsilon$) was calculated based on the central difference approach expressed as $d\sigma/d\varepsilon = \sigma_{\varepsilon_1} - \sigma_{\varepsilon_2})/(\varepsilon_{\varepsilon_1} - \varepsilon_{\varepsilon_2})$, where $\sigma_{\varepsilon_1}$, $\varepsilon_{\varepsilon_1}$, $\sigma_{\varepsilon_2}$, and $\varepsilon_{\varepsilon_2}$ are constants. Thus, $\sigma = \sqrt{\varepsilon_0}\sigma' = \sigma_{\varepsilon_1} + k\varepsilon'$, where $\varepsilon_0$ is a constant and $n$ is a constant. Then, $d\sigma_{\varepsilon_2}/d\varepsilon_{\varepsilon_2} = \sigma/(n(1-n))$, and finally $\ln(d\sigma/d\varepsilon) = (1-n)\ln\sigma - \ln(nk)$. Therefore, double-logarithmic plots of $d\sigma_{\varepsilon_2}/d\varepsilon_{\varepsilon_2}$ versus $\sigma$ can be used to analyze the work-hardening behavior.

3. Results and Discussion

The microstructure of the normalized sheet is shown in Fig. 2a, which exhibits a typical ferritic-pearlitic morphology of low carbon steels. The water quenched microstructure is shown in Fig. 2b. It can be seen that
this sheet has a typical morphology of lath martensite \(^{15}\). After cold rolling, a lamellar structure in rolling direction (RD) has formed as shown in Fig. 2c as a result of the deformation of the lath martensitic structure. It has been shown that this microstructure can provide the substrate for the formation of very fine ferrite grains during subsequent thermal processing \(^{16}\) and formation of fine-grained DP steel after intercritical annealing \(^{9}\). Therefore, the as-received and the cold rolled sheets were used as the initial sheets for intercritical annealing as shown in Fig. 1.

The tensile stress-strain curves of the as-received sheet, DP1, and DP2 are shown in Fig. 3. The yield point elongation for the as-received sheet can be easily seen, in which after the upper yield point, dislocations are released from the Cottrell atmospheres and the stress drops to the lower yield point, and local plastic deformation spreads grain to grain, producing the yield-point elongation \(^{17}\). After that, the normal work hardening regime resumes. However, the DP steels do not show the yield point elongation, which is related to the presence of free dislocations in ferrite around the martensite particles \(^{18}\). In fact, the shear and volume changes associated with the transformation of austenite to martensite (during quenching from the intercritical region) generates some unpinned dislocations in ferrite, which can inhibit the occurrence of yield-point phenomenon. Fig. 3 reveals that DP steels show much better work hardening behavior compared with the as-received ferritic-pearlitic sheet. However, the most significant aspect of Fig. 3 can be realized by comparing DP1 and DP2, where the tensile properties of DP2 are much better: ~ 190 MPa difference in tensile strength with nearly the same total elongation value. These differences can be directly related to the microstructure of these sheets as discussed below.

The microstructure of DP1 steel is shown in Fig. 4a. It can be seen that ~ 35 vol% martensite has formed in the matrix of ferrite. The martensite phase (in fact austenite at the intercritical annealing temperature) has formed in the pearlitic colonies and surrounding ferritic matrix and also on the ferrite grain boundaries. The average ferrite grain size of DP1 steel is ~ 17 µm. The microstructure of DP2 steel is shown in Fig. 4b that is finer (~ 11 µm), which is a result of the recrystallization of the cold-rolled martensite before partial austenitization at the intercritical annealing temperature.
temperature as schematically shown in Fig. 5 9). The presence of the chain-networked martensite morphology at grain boundaries of ferrite is evident in the SEM image of the DP2 steel (Fig. 4c), which is consistent with the intercritically annealed schematic shown in Fig. 5. In fact, the cold-deformed martensite completely recrystallizes into ferrite upon heating 9), and then, austenite nucleates at the ferrite grain boundaries that leads to the formation of a chain-like structure. The grain refinement and formation of chain-network structure directly affect the work-hardening behavior, which is the basis for the significant enhancement of properties. These aspects will be discussed in the following.

The work-hardening plots based on the modified Crussard-Jaoul analysis are shown in Fig. 6. It can be seen that the as-received sheet shows a low work-hardening rate at the start of plastic deformation, which is related to the occurrence of the yield-point phenomenon. For

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**Fig. 4.** Representative micrographs of the DP steels processed in this work.

**Fig. 5.** Schematic representation of the recrystallization of cold rolled martensite and formation of the chain-network martensite morphology after intercritical annealing 9).
the DP steels, the initial work-hardening rate is much higher due to the presence of unpinned dislocations that interact and produce a high initial strain hardening rate. Generally, for DP steels, three stages of work-hardening can be detected: The transient Stage I represents the glide of mobile dislocations in ferritic areas present near the martensitic regions, Stage II belongs to the deformation of the constrained ferrite, and Stage III is related to the concurrent deformation of martensite and hardened ferrite 19-21).

For a major part of the work-hardening curves, it can be seen that the work-hardening curve corresponding to DP2 is located above DP1 at each given flow stress, which shows that the work-hardening response of DP2 is better. This reveals that producing a fine-grained DP microstructure with the chain-networked martensite morphology is in favor of enhancing the work-hardening behavior. It has been reported that chain-networked martensite grains surrounding ferrite grain prevent a propagation of strain localization to an adjacent ferrite grain, and thus, the chain-networked structure of fine martensite grains may contribute to maintaining adequate elongation even at high strength level 9, 10). This chain-network martensite morphology is not present in the microstructure of the DP1 steel with the consequent inferior work-hardening behavior and tensile properties.

Figure 7 shows the strength-ductility diagram of steels, where the results of the present study are also indicated. It can be seen that the DP1 steel shows inferior tensile
properties. By contrast, based on the prior cold rolling of martensite (DP2 steel), it is possible to obtain enhanced strength-ductility balance compared with the conventional trend seen in steels. The mechanical properties of DP2 steel reveals that this steel can be considered to be a DP300/600 steel with yield stress of ~ 300 MPa and tensile strength of ~ 600 MPa, which resulted in the nominal yield ratio of ~ 0.50. The presence of chain-network morphology of martensite and fine ferrite grain size resulted in the enhancement of work-hardening in this steel when compared with the conventional DP350/600 steel with the yield ratio of ~ 0.58.

4. Conclusions

The effect of microstructural refinement and intercritical annealing on the mechanical properties and work-hardening response of a low carbon St12 steel was studied. The following conclusions can be drawn from this study:

- Intercritical annealing of the ferritic-pearlitic sheet results in the formation of a coarse-grained DP microstructure with discrete martensite islands normally formed in place of pearlitic colonies, which results in the minor enhancement of mechanical properties with disappearance of the yield-point elongation.
- A fine-grained DP steel with chain-network martensite morphology can be obtained by intercritical annealing of the cold rolled martensitic microstructure, which shows superior work hardening rate, low yield ratio, and high tensile strength. In this way, it is possible to enhance the mechanical properties of St12 steel toward those of DP300/600 steel. Compared with the conventional DP350/600 grade, a significant enhancement in the work-hardening behavior can be achieved with acceptable strength-ductility balance compared with the usual trend seen in steels.
- It can be realized that cold rolling of the initial martensitic microstructure before intercritical annealing is a viable approach for processing DP steels with enhanced mechanical properties for industrial applications.

References