Effect of microstructural refinement and intercritical annealing time on mechanical properties of high-formability dual phase steel

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ABSTRACT

The impact of the intercritical annealing time and refinement of microstructure upon the tensile properties and work-hardening capacity of a dual phase (DP) steel were evaluated. For the undeformed pre-intercritical microstructures, the sensitivity of the obtained DP microstructures on the time of annealing was low, and in all cases, a relatively coarse microstructure was obtained. It was shown that a fine-grained DP steel with chain-network martensite morphology can be readily obtained by carefully controlled intercritical annealing of a cold deformed martensitic microstructure. By continued intercritical annealing beyond the optimum value, the growth of ferrite grains and fading of the chain-network martensite morphology were found to be responsible for the lowering of work-hardening rate and obtained tensile properties. By optimization of the intercritical annealing condition, a DP300/600 steel with high work-hardening rate, low yield ratio, high tensile toughness, and good ductility was obtained, which exhibited significant enhancements compared with the conventional DP350/600 grades.

1. Introduction

One of the main targets of automobile manufacturers is to reduce the consumption of gasoline through the reduction of the weight. This can be achieved via thinner gauges of the parts, where, in return, increasing the strength is a vital requirement. However, the resulting deterioration of formability effectively retards the applicability of the used material. To address this problem, the advanced high strength steels (AHSS) [1-4], e.g. dual phase (DP) steels [5-10], have been developed to obtain high strength levels with maintaining ductility properties. Normally, low-carbon DP steels have a duplex ferritic-martensitic microstructure. The co-presence of martensite governs the good ductility-strength balance and high work hardening rate [11-13]; and the simplicity of the chemical composition is important from the standpoint of weldability [14].

The tensile properties of DP steels are determined by the adjustment of the individual constituents of the microstructure in terms of type, volume fraction, size, morphology, and spatial distribution [1,15-20]. Compared with their coarse-grained counterparts, the fine-grained DP steels have superior mechanical properties. Accordingly, various thermo-mechanical processing routes have been developed for microstructural refinement of DP steel [21-30]. Warm deformation followed by intercritical annealing was used by Calcagnotto et al. [23] for grain refinement. Nakada et al. [24] and Alibeyki et al. [25] have considered the intercritical annealing of the cold rolled martensite. The recrystallization of cold rolled martensite before intercritical annealing and its effect on the formation of chain-networked martensite grains was also discussed [24,26]. Development of fine ferrite/carbide aggregate during tempering of the cold deformed martensite was studied by Azizi-Alizamini et al. [27] and Mirzadeh et al. [18], where the development of fine DP microstructure after intercritical annealing was discussed. Cold deformation of DP steel followed by intercritical annealing was also found to be an effective grain refinement technique [28,29]. Finally, thermal cycling for grain refinement was taken into account by Ghaemifar and Mirzadeh [30].

Flat-rolled products are one of the largest category of high-formability low carbon steels, which are usually used in the cold-rolled and annealed condition. These steels have low carbon and manganese contents (< 0.10 wt% C and < 0.4 wt% Mn). The effect of martensite content on the tensile properties of a 0.035C-0.268Mn-0.035Si steel (all values are expressed in weight percent) has been systematically studied by the present authors [31] and it has been revealed that the resulting DP steels show relatively low strength (~ 418 MPa) but acceptable ductility (total elongation of ~ 30%). Therefore, microstructural modification of this steel is required to enhance its mechanical properties toward standard DP grades such as DP600. Accordingly, the influence

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of the intercritical heat treatment time and microstructure refinement on the tensile properties and work-hardening capacity of 0.035C-0.268Mn-0.035Si steel are evaluated in this study.

2. Experimental details

2.1. Processing

A st12 steel sheet with the chemical composition (wt%) of 0.035C-0.268Mn-0.035Si was received in the annealed state. The critical temperatures were estimated as $A_{1} = 736 \pm C$ and $A_{3} = 890 \pm C$ based on the equations developed by Trzaska and Dobrzanski [32]. The as-received material was austenitized followed by air cooling / water quenching to develop ferritic-pearlitic / dominantly martensitic microstructures, respectively (Fig. 1a). The resulting sheets were intercritically annealed at $850 \pm C$ for 5 and 15 min, which was followed by quenching in water medium to obtain DP steels. As shown in Fig. 1b, the water quenched sample was 50% cold rolled, and then, heated to the temperature of $850 \pm C$ at the rate of $\sim 30 \pm C/s$. After soaking at that temperature for 2.5, 5, and 15 min, the sheets were water quenched to obtain fine DP steels [24-26]. The processing routes shown in Fig. 1a and b will be referred as thermal treatments and thermomechanical treatments, respectively.

2.2. Characterisation

The obtained microstructures were etched by the LePera’s reagent and Nital solution. An optical microscope and a CamScan MV 2300 scanning electron microscope (SEM) were used for microstructural investigations. The JIS Z 2201 standard for tensile test specimens was used. A universal testing machine was used for tensile tests at room temperature with strain rate of $10^{-3} s^{-1}$. The reproducibility of results was evaluated by the repetition of the tests.

The values of work-hardening rate ($\sigma/d\varepsilon = (\sigma_{i+1} - \sigma_{i-1})/(\varepsilon_{i+1} - \varepsilon_{i-1})$) were used in the modified Crussard-Jaoul approach [17,18,33], where the latter is based on the relation of $\varepsilon = \varepsilon_{0} + k\alpha n$ ($n$, $k$, and $\varepsilon_{0}$ are constants). As a result, $\sigma = \sqrt{(\varepsilon - \varepsilon_{0})/k}$, then $d\sigma/d\varepsilon = \sigma/[n(\varepsilon - \varepsilon_{0})]$, and eventually $\ln(d\sigma/d\varepsilon) = (1-n)\ln \sigma - \ln (nk)$. As a result, the double-logarithmic $d\sigma/d\varepsilon - \sigma$ plots will be used. Tensile toughness was calculated based on the estimation of the area under the stress-strain curve: $Area = \sum (\varepsilon_{i} - \varepsilon_{i-1})(\sigma_{i} + \sigma_{i-1})/2$.

3. Results

3.1. Thermal treatments

The tensile properties of the sheets after thermal treatments and the obtained microstructures are depicted in Fig. 2. The normalized sheet (Fig. 2b) has a typical ferrite-pearlite microstructure, where its tensile curve shows an obvious yield point elongation regime [34] (Fig. 2a). The microstructure of the DP steel produced by intercritically annealing of the normalized sheet at $850 \pm C$ for 5 min (DPAC5) is shown in Fig. 2c. A ferritic-martensitic microstructure with $\sim 35$ vol% martensite can be seen (in fact the surface fraction as determined by image analysis). The martensite phase (austenite at the intercritical annealing temperature) has formed in the pearlite colonies and surrounding ferrite matrix and also on the grain boundaries of ferrite. Fig. 2a reveals that the yield point phenomenon vanished, which is caused by the unpinned dislocations in the vicinity of the martensite phase [27,35]. The high work-hardening rate of DP steel can be inferred from the higher slope of the tensile curve compared with that of the normalized sheet. Accordingly, this DP steel shows higher strength with maintaining relatively good total elongation. Fig. 2d depicts the microstructure of the DP steel obtained by 15 min intercritical annealing of the normalized sheet at $850 \pm C$ (DPAC15). Compared with Fig. 2c (DPAC5), the grains of ferrite and martensite particles have slightly grown: average ferrite grain size of 16.1 $\mu m$ vs. 17.4 $\mu m$ for DPAC5 and DPAC15, respectively. This is responsible for relatively inferior mechanical properties of this DP steel [18,25,36]. However, the differences are not large as can be seen in Fig. 2a.

Fig. 2e shows the microstructure of the as-quenched sheet. It can be seen that this sheet has a typical morphology of lath martensite [37]. During intercritical annealing, the martensitic substructure provides various nucleation sites for austenite (and then martensite after quenching), and hence, a different morphology of DP steels will be
obtained (Fig. 2f). However, as shown in Fig. 2a for DPWQ5, its tensile properties are similar to those discussed above. Thus, it can be inferred that it is not possible to effectively enhance the tensile properties of this steel based on the simple thermal heat treatments. To address this problem, the prior cold rolling will be considered.

3.2. Thermomechanical treatments

By consideration of the substructure of martensite, it can be deduced that the martensitic transformation is an effective grain subdivision process [37]. It has been shown in several research work [38–40] that after cold rolling, a lamellar structure in rolling direction (RD) will be formed as a result of the deformation of the lath martensite structure. Subsequent tempering of this microstructure can be used for obtaining an aggregate of fine ferrite grains and carbide particles [40]. Moreover, a fine DP microstructure can be produced via intercritical heat treatment of this microstructure [24,25]. The latter technique was used in this work (Fig. 1b) and the results are summarized in Fig. 3. By comparing Fig. 3a (DPWQ5: Water Quenched + 5 min at 850 °C) with Fig. 3b (DPCr2.5: Water Quenched + 50% Cold Rolled + 2.5 min at 850 °C), it can be seen that the grains of ferrite in the DPCR2.5 are much finer. In fact, the average size of ferrite grains for the DPWQ5 and the DPCR2.5 steels are ~ 21 and ~ 11.5 µm, respectively. Fig. 3c shows a SEM image of the DPCR2.5 steel at high magnification, where the presence of the chain-networked martensite morphology at grain boundaries of ferrite is evident (shown by arrows for a grain in Fig. 3c). This is consistent with the previous research [7,24], which showed that the cold-deformed martensite recrystallizes into ferrite upon heating. Then, the formation of a chain-like martensite is resulted from the nucleation of austenite on the grain boundaries [26].

Fig. 4 shows the resulting tensile stress-strain curves. By comparing the tensile curves of the DPWQ5 and the DPCR2.5 steels, a significant enhancement in tensile properties can be seen: ~ 65 MPa and ~ 175 MPa enhancement respectively in yield and tensile strength with
maintaining the same total elongation value (See the bar charts). These increments in tensile properties can be linked to the microstructural refinement [21–30] and also to the formation of chain-like networked martensite [24,26,41] in the DPCR2.5 steel. The average grain size of ferrite for the DPCR is also fine (~13 µm) as shown Fig. 3d, which resulted in good mechanical properties. However, as can be deduced from Fig. 3e, by increasing the time of intercritical annealing, the average size of ferrite grains increases (~15 µm), and as a consequence, chain-like networked structure starts to vanish as can be clearly seen for DPCR15 (Fig. 3f). As a result, as shown in Fig. 4, for DPCR15, there is an obvious decrease in the ultimate tensile strength and total elongation. These observations can be related to change in work-hardening behavior as discussed in the following.

4. Discussion

In the previous section, it was revealed that the cold rolled martensitic structure is an appropriate one for grain refinement of DP steel, and by applying proper intercritical annealing time, it is possible to obtain a fine-grained ferrite with the chain-networked martensite morphology. As a result, the strength-ductility balance can be effectively enhanced, which needs to be discussed by consideration of the work hardening rate, which is shown in Fig. 5. The yield-point elongation of the normalized sheet is responsible for the observed weak initial work-hardening rate. An opposite trend can be seen for the DP steels in Fig. 5 due to the presence of the already available unpinned dislocations as discussed above. Stage I is a transient regime caused by the glide of mobile dislocations, Stage II is related to the deformation of ferrite, and Stage III appears as a consequence of the deformation of hardened ferrite and martensite [18,33].

In Fig. 5, the curve of DPCR2.5 is located above DPWQ5 indicating the better work-hardening response of the former. This reveals that producing a fine-grained DP microstructure is advantageous. Moreover, the work-hardening curve corresponding to DPCR15 falls below that of DPCR2.5, which implies that the presence of the chain-networked martensite morphology is in favor of enhancing the work-hardening behavior. In fact, it prevents the propagation of strain localization to an adjacent ferrite grain, and hence, it may contribute to the maintaining of ductility at high strength levels [24,26].

For DP steels, it has been revealed that the work-hardening rate \( (d\sigma/de) \) is dependent on the average diameter \( (d_M) \) and volume fraction \( (f_M) \) of martensite islands based on the equation of \( \sigma_M/d_M \) [18,36]. The values of \( d_M \) for DPWQ5, DPCR2.5, DPCR5, and DPCR15 was respectively measured as 8.39, 8.58, 7.57, 8.9 µm, which are nearly the same in these samples. Since the \( f_M \) is also the same, the work-hardening rate can be controlled based on the grain size of ferrite and also the morphology of martensite. The latter is in accordance with previous research [42,43], which showed the striking property of enhancing work-hardening rate with refining ferrite grain size in contrary to the trend seen in conventional steels.

Fig. 6a shows the values of tensile toughness. It can be seen that tensile toughness of DPWQ5 is lower than that of the Normalized steel. However, the tensile toughness of DPCR2.5 is much higher, which indicates that the fine-grained DP steel with chain-networked martensite morphology is conductive toward better strength-ductility balance. However, by increasing the intercritical annealing time beyond the optimum value, the tensile toughness sharply decreases, which is related to the grain growth of ferrite and fading of the chain-networked martensite morphology.

Fig. 6b shows the strength-ductility diagram of steels, where the results of the present study are also indicated. It can be seen that by prior cold rolling and proper intercritical annealing time (e.g. DPCR2.5), superior strength-ductility balance can be achieved compared with the conventional trend seen is steels.

The tensile properties of DPCR2.5 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 (experimentally 0.48). 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 more common DP350/600 steel with the yield ratio of ~0.58.

5. Conclusions

The influence of the time of intercritical heat treatment and microstructural refinement on the tensile properties and work-hardening capacity of a DP steel were evaluated. The following conclusions can be drawn from this work:

(1) The tensile properties of DP steels produced based on the simple thermal heat treatments without prior cold deformation showed low sensitivity on the time of intercritical annealing and pre- intercritical microstructure. In all cases, a coarse DP structure was obtained. In this way, the tensile properties were slightly enhanced.

(2) A fine DP steel with chain-network martensite was obtained by carefully controlled intercritical annealing of a cold rolled martensitic microstructure. It showed high tensile toughness and work-hardening behavior with low yield ratio. As a result, it can be considered to be a DP300/600 steel, which exhibits significant enhancements compared with the conventional DP350/600 grades.
By increasing the intercritical annealing time beyond the optimum value, the growth of ferrite grain and fading of the chain-network martensite morphology were found to be responsible for the declining of the work-hardening rate and final tensile properties. This unveils the necessity of close control of the intercritical heat treatment for the enhancement of tensile properties.

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Data availability

The raw data required to reproduce these findings are available to download from Mendeley Data [https://data.mendeley.com/datasets/wv2yhm4d4p/draft?fa=c4a5da31-e19e-4b24-bbeb-3e9f18324b27].

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