The Developments of Cold-rolled TRIP-assisted Multiphase Steels. Low Silicon TRIP-assisted Multiphase Steels

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TRIP-assisted multiphase steels are newly developed steels exhibiting an improved balance of strength and ductility very well adapted for the automotive industry. In order to allow the retention of austenite, the steels studied up to now contained high levels of silicon incompatible with the industrial practice. This study shows how the combination of both a composite strengthening effect and the TRIP effect can improve the mechanical properties of a cold-rolled 0.16wt%C–1.3wt%Mn steel containing only 0.4 wt% of silicon. A complete characterisation of the bainite transformation and of the resulting microstructures, tensile properties and mechanical stability of retained austenite has allowed the determination of the factors responsible for the enhanced balance of strength and ductility of this steel well adapted for automotive applications.

KEY WORDS: high strength steels; retained austenite; TRIP and multiphase steels; bainite transformation; mechanical properties.

1. Introduction

Since the end of the 1980s, new formable steels with higher strength levels have been thoroughly studied.^{1–3)} These steels are well adapted to the requirements of the automotive industry for driving down the weight and improving the crashworthiness. Among these high-strength steel grades, the new and so-called ‘TRIP-assisted multiphase steels’ (that are related to the Dual Phase steels) present an improved balance of strength and ductility thanks to complex multiphase microstructures and the TRIP effect, *i.e.* a mechanically-induced martensitic transformation of retained austenite. The chemical composition and processing routes of these TRIP-assisted multiphase steels are designed for the retention of austenite within a multiphase microstructure. In the case of cold-rolled and annealed steels, the TRIP-aided steels are obtained at the end of a 2-stage heat-treatment. The first stage consists in an intercritical annealing (as for Dual-Phase steels) during which a controlled volume fraction of austenite is formed together with the intercritical ferrite. This intercritical annealing is followed by an isothermal holding stage in the bainite transformation temperature range. During this holding, part of the austenite transforms to bainite, whereas residual austenite (*i.e.* non bainitically transformed austenite) may become sufficiently stabilised by carbon rejection from bainitic ferrite as to not transform to martensite during the final quenching to room temperature. It is indeed well known that the bainite transformation is accompanied by the carbon redistribution from the bainitic ferrite to the surrounding residual austenite.⁴⁾

In order to promote the formation of carbon supersaturated austenite instead of cementite precipitation during the bainitic holding, TRIP-aided steels are alloyed with significant amounts of silicon (from 1 to 2 wt%).^{4,5)} Silicon inhibits cementite precipitation during bainite transformation and therefore promotes enrichment of residual austenite with carbon. As a consequence, studies dealing with the TRIP-assisted steels mainly focus on alloys containing rather high concentrations of silicon (from 1 to 2.5 wt%).

These high levels of silicon constitute the major drawback for industrial developments of the TRIP-assisted multiphase steels. Silicon is known to cause surface defects after casting and to form adherent hard oxides not compatible with the industrial surface cleaning practice prior to hot-rolling. Furthermore, steel compositions have to be kept at low silicon contents in order to allow galvanisation and spot welding.

Studies dealing with conventional high silicon TRIP-assisted steels have mainly focused on the maximisation of the TRIP effect. This effect is considered as the main cause responsible for the enhanced mechanical properties of these steels. The emphasis was thus to allow austenite retention in a wide range of bainite transformation conditions. Furthermore, it is sometimes believed that retention of austenite is almost impossible with silicon contents lower than 1 wt%.^{6,7)} Whereas it is well demonstrated that the TRIP effect plays a favourable role on the properties of TRIP-assisted multiphase steels, hardly anything has been reported on the effect of the other phases and on the mechanical properties of multiphase steels. However, a recent

study⁸⁾ has shown that high levels of austenite can be retained in a steel grade comparable to Dual Phase steels with a reduced silicon content.

This study aims at enlightening the improvements of the mechanical properties of a laboratory or industrially processed cold-rolled 0.16C–1.33Mn–0.38Si steel (*i.e.* a typical cold-rolled Dual-Phase steel composition) thanks to the combination of a composite strengthening effect and the TRIP effect. Despite a much lower silicon content than for the conventional TRIP-assisted steels, up to 10% of austenite can be retained in the microstructure of this steel which can be industrially produced and heat-treated. The laboratory developed low-silicon multiphase steel was compared with (i) conventional high silicon TRIP-aided steels, (ii) some processing attempts on industrial lines and (iii) conventional deep-drawable steels, from the viewpoints of uniaxial tensile testing, microstructure and stability of retained austenite. This study intends to show which factors govern the improved balance of strength and ductility obtained with these steels.

2. Experimental Procedure

The chemical compositions of the investigated steels are given in **Table 1**. 100 kg cast ingots of alloys HSiI and HSiII together with the industrially produced steel LSi were hot-rolled following classical practice before being 75 to 82% cold-rolled to 1 mm in thickness.

For the laboratory studies, heat-treatments were carried out on samples of steels HSiI, HSiII and LSi oriented in the rolling direction. Samples were first intercritically annealed in a fluidised bed furnace and then transferred to a lead bath furnace for the bainitic holding. A complete description of the heat-treatment conditions can be found elsewhere.⁸⁾ The heat-treatment schemes were also simulated in a dilatometer in order to characterise completely the formation and transformation of austenite.⁹⁾

The steel designated by 'IND' corresponds to a newly developed multiphase steel industrially produced by Usinor-Cockerill-Sambre. As shown in Table 1, its chemical composition is quite identical to steel LSi. After hot-rolling following classical route and 69% cold-rolling to a thickness of 1.25 mm, the steel was continuously annealed on an industrial line of Usinor-Cockerill-Sambre following classical industrial practice. The coil was first reheated at 760°C. The steel was then cooled to around 400°C by using the HOWAQ[®] process (which ensures a constant cooling rate of 50°C/sec) for the bainitic holding. The steel was finally cooled to room temperature by jet cooling before coiling.

Table 1. Chemical compositions (10^{-3} wt%) of the investigated steels.

(10^{-3} wt%)	C	Mn	Si	P	S	Nb	Al	N
HSiI	130	1420	1500	13	9	0	27	7.9
HSiII	290	1420	1410	12	20	0	40	8
LSi	160	1300	380	13	12	13	30	6.3
IND	160	1390	420	19	6	10	29	5.1
REFI	65	380	<30	<15	<8	50	35	5.2
REFII	30	200	<30	<15	10	0	40	3.5

The two last steel grades, REFI and REFII, are conventional steel grades presently used by the automotive industry. They correspond to classical HSLA and AIK DDQ steels.

Tensile specimens were prepared according to the European standard EN 10002-1. The initial gauge length was 50 mm and the width was 12.5 mm. Tensile testing was done at a crosshead speed of 2 mm/min. Measured loads and elongations were converted to true stress–true strain curves. Yield strength (σ_y), true stress at maximum load (σ_{TS}) and true uniform strain (ϵ_u) (*i.e.* the maximum uniform true strain) were measured from these tensile curves. The yield strength was defined either as the conventional 0.2% yield strength ($\sigma_{0.2}$) or as the true stress corresponding to the Lüders deformation plateau for samples with continuous or discontinuous yielding behaviours, respectively. Strain-hardening was characterised by the incremental strain-hardening exponent ($n_{incr.}$) defined as

$$n_{incr.} = \frac{d \ln \sigma}{d \ln \epsilon} \dots\dots\dots (1)$$

Microstructures were studied by light microscopy, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) after particular preparation routes described elsewhere.¹⁰⁾ Retained austenite contents were measured by Mössbauer spectroscopy.⁸⁾ X-ray diffraction was used to determine the lattice parameter (a_0) of retained austenite and therefore its carbon content by making use of the relationship¹¹⁾:

$$a_0 (\text{\AA}) = 3.578 + 0.033 (\text{wt}\% \text{C}) \dots\dots\dots (2)$$

3. Results and Discussion

3.1. Heat-treatment and Microstructure

Figure 1 sketches the map giving the evolution of the phases present in the room temperature microstructure of steel LSi after increasing bainite transformation times. The bainite transformation was conducted at 370°C after intercritical annealing for 5 min at 730°C (*i.e.* 10°C above A_{c1}). As represented in Fig. 1, the intercritical annealing brings about an initial volume fraction of austenite of 25%. Depending on the bainite transformation conditions, the nature of the non intercritical ferrite phases can be adjusted. **Figure 2** shows typical microstructures obtained after bainitic transformation times of 1 min and 30 min, respectively. The microstructures consist in a matrix of ferritic grains with a dispersion of bainite–austenite–martensite grains located at the ferrite grain boundaries. As shown in these 2 figures, up to 10% of austenite can be retained after very short holding times (from 15 sec to 3 min). The retention of such a large volume fraction of austenite was not anticipated when considering previous studies dealing with low silicon steel grades.^{6,7)} For longer holding times, the bainite transformation goes nearly to completion and only bainite can be found in the microstructure, as shown in Fig. 2(b). As predicted by the laboratory study on steel LSi, the microstructure of the industrially produced low silicon multiphase steel (IND) also consisted in a ferritic matrix with a dispersion of bainite and retained austenite as second phas-

Fig. 1. Transformation map of steel LSi giving the evolution of the room temperature microstructure with the bainitic holding time at 370°C. Steel LSi has previously been intercritically annealed at 730°C for 5 min.

Fig. 3. Transformation map of steel HSiII giving the evolution of the room temperature microstructure with the bainitic holding time at 360°C. Steel HSiII has previously been intercritically annealed at 760°C for 6 min.

(a)

(b)

Fig. 2. SEM micrographs of the typical microstructure of steel LSi intercritically annealed at 730°C for 5 min and bainitically transformed at 370°C for 1 min (a) and 30 min (b) (α : intercritical ferrite; α_b : bainite; α' : martensite; γ : retained austenite).

(a)

(b)

Fig. 4. SEM micrographs of the typical microstructure of steel HSiII intercritically annealed at 760°C for 6 min and bainitically transformed at 360°C for 3 min (a) and 15 min (b) (α : intercritical ferrite; α_b : bainite; α' : martensite; γ : retained austenite).

es.

A similar transformation map given in **Fig. 3** was obtained for steel HSiII after intercritical annealing at 760°C (*i.e.* 15°C above A_{c1}) for 6 min and bainitic holding at 360°C. Due to a higher carbon content, the volume fraction

of intercritical austenite is in this case larger despite similar annealing conditions (*i.e.* approximately the same temperature difference with respect to A_{c1}). Micrographs shown in **Fig. 4** were obtained after bainitic transformation times of 3 and 15 min, respectively. In the case of steel HSiII, a volume fraction of 16% of austenite can be retained with bai-

nite and ferrite for longer bainitic holding times than for steel LSi.

For steel HSiI, the same behaviour as for steel HSiII can be observed during the bainite transformation, *i.e.* the behaviour of a high silicon steel. However, because of a lower carbon content, a lower volume fraction of austenite can be retained. The maximum volume fraction of austenite is about 8–10% in the case of steel HSiI.

Figure 5 summarises the evolution of the volume fraction of retained austenite as a function of the bainitic holding time at 360°C for steels HSiI and HSiII and at 370°C for steel LSi while **Fig. 6** presents the evolution of the carbon content of this retained austenite. Figure 5 shows that the bainite transformation of intercritical austenite is accompanied by the stabilisation of austenite in each steel grade. However, the behaviours of the high and low silicon steels are not identical. In both steels HSiI and HSiII, the amount of austenite retained at room temperature first increases (at the expense of martensite) to reach a maximum when martensite completely disappears from the microstructure. This level of retained austenite is then maintained even for longer bainitic holding times. A much larger volume fraction of austenite (~20%) can be retained in steel HSiII due to a higher carbon content of the grade. However, steels HSiI and HSiII present identical evolutions of the carbon enrichment of austenite with the bainitic holding time. Indeed, the austenite carbon content increases in both steels up to a level of about 1 wt% that corresponds to the T_0 -curve (*i.e.* the upper bound for the carbon enrichment of austenite during a diffusionless phase transformation^{9,12,13}). Once this upper bound is attained, the bainite transformation cannot further proceed (the transformation is said to be ‘incomplete’) as shown in Fig. 6. As already reported elsewhere,^{14,15} this behaviour corresponds to the typical behaviour of high silicon steels in which cementite precipitation is totally inhibited. The TEM micrograph of **Fig. 7** confirms that only bainitic ferrite without cementite can be found together with retained austenite in steels HSiI and HSiII.

In the case of the low silicon steel LSi, the bainitic holding first leads to a very fast increase of the retained austenite content. Bainitic holding longer than approximately 50 sec then induces a monotonous decrease of the retained austenite content. Contrarily to steels HSiI and HSiII, this maximum of the retained austenite content occurs when the major part of the second phases is martensite and when the carbon enrichment of austenite is not maximum. As a consequence, it can be postulated that in the case of steel LSi, the stabilisation of austenite is not only due to the carbon enrichment of austenite but also to a partial martensitic transformation. As already reported by Xie *et al.*,¹⁶ a partial transformation of the austenite during quenching stabilises the remaining austenite against the further progress of the transformation.

Furthermore, if carbon enrichment of austenite during the bainitic transformation can be observed (Fig. 6) and has a stabilising effect, cementite precipitation also occurs in steel LSi. **Figure 8** presents a TEM micrograph of an austenite grain of steel LSi after 3 min of bainitic holding at 370°C. Together with the retained austenite, cementite particles can be found between the laths of bainitic ferrite.

Fig. 5. Evolution of the volume fraction of retained austenite with the bainitic holding time for steels LSi held at 370°C; and steels HSiI and HSiII held at 360°C.

Fig. 6. Evolution of the carbon content of retained austenite with the bainitic holding time for the three investigated steels.

Fig. 7. TEM micrograph (bright field) of steel HSiII after 6 min of bainitic holding at 360°C (intercritical annealing at 760°C for 6 min) showing the absence of cementite with the bainitic ferrite plates and retained austenite. (α_b : bainitic ferrite; γ : retained austenite).

This cementite precipitation can explain why no incomplete reaction phenomenon is observed in steel LSi. While bainite transformation leads to a carbon enrichment of austenite, cementite precipitation brings about a carbon weaken-

ing of austenite so that the upper bound for the carbon content of austenite dictated by the T_0 -curve cannot be attained before the complete transformation of residual austenite.⁹⁾

3.2. Tensile Properties

Figure 9 presents the values of true uniform strain vs. true stress at maximum load for all combinations of heat-treatment parameters (*i.e.* intercritical annealing temperature and time and bainite transformation temperature and time) of steels HSiI, HSiII and LSi as well as for the industrially produced multiphase steel (IND) and the 2 conventional steels REFI and REFII. We can see that better combinations of strength and ductility are obtained with the multiphase steels in comparison with steels REFI and REFII currently used in the automotive industry. Furthermore, this figure shows that each steel grade leads to particular and well-defined levels of strength and ductility.

For steels HSiI and HSiII, true stress at maximum load varies between 850 and 1050 MPa and 1000 and 1200 MPa, respectively, while true uniform strain ranges between 0.12 and 0.27 for both of them. These 2 conventional TRIP-assisted multiphase steels present the best mechanical properties among the investigated steels. Steel HSiII with its

much larger content of retained austenite leads to further enhancements of the combinations of strength and ductility. For the laboratory developed low silicon steel LSi, true stress at maximum load varies between 700 and 900 MPa, while true uniform strain ranges from 0.10 to 0.19. These performances are quite remarkable for such a steel composition when compared to solid-solution or precipitation hardened high-strength steels,¹⁷⁾ or even to conventional TRIP-aided steels with a lower carbon content (~ 0.1 wt%).¹⁸⁾ The mechanical properties of the industrially produced multiphase steel IND corresponds to a mean true stress at maximum load of 735 MPa with a true uniform strain of 0.15, *i.e.* comparable to the mean values of the laboratory processed identical grade. It is noteworthy that this combination of properties is much larger than the ones of the presently produced steels for the automotive industry.

Figure 10 shows the yield strength as a function of the true stress at maximum load for all combinations of heat-treatment parameters tested in this study. For the 3 laboratory studied steels, *i.e.* HSiI, HSiII and LSi, the yield strength can be varied in a large range globally comprised between 200 and 600 MPa. As indicated on Fig. 10, this yields to σ_Y/σ_{TS} ratios between 0.3 and 0.8. The industrially produced multiphase steel IND presents a mean yield strength of 540 MPa and therefore a σ_Y/σ_{TS} ratio comprised between 0.7 and 0.8 (which is almost comparable with the σ_Y/σ_{TS} ratio of the steel REFI).

Figure 11 presents the true stress vs. true strain curves (Fig. 11(a)) as well as the n_{inc} vs. true strain curves (Fig. 11(b)) of particular specimens of steels HSiI, HSiII and LSi presenting the best strength-ductility balance for each steel grade. Furthermore, **Fig. 12** shows the evolution of the retained austenite content of these specimens as a function of true strain. Specimens of steels HSiI and LSi present the same initial amount of retained austenite ($\sim 8\%$) with different carbon contents (0.95 wt% for steel HSiI and 0.73 wt% for steel LSi). Specimen of steel HSiII contains twice as much austenite than the two other specimens with also a high carbon content (0.97 wt%). This carbon content influences the transformation rate during straining: a progressive transformation all along straining for steels HSiI and HSiII and a predominant transformation in the early part of plastic straining for steel LSi.

Fig. 8. TEM micrograph (bright field) of steel LSi after 3 min of bainitic holding at 370°C (intercritical annealing at 730°C for 5 min) showing that cementite can be found between the plates of bainitic ferrite (α_b : bainitic ferrite; θ : cementite).

Fig. 9. Map of true uniform strain vs. true stress at maximum load for all combinations of the investigated heat-treatments parameters on the different studied steels.

Fig. 10. Map of yield strength vs. true stress at maximum load for all combinations of the investigated heat-treatments parameters on the different studied steels.

Fig. 11. True stress-true strain (a) and work-hardening exponent ($n_{incr.}$) curves (b) of the specimens of each steel grade optimising the performance index $\epsilon_u * \sigma_{TS}$.

As also shown in Ref. 19), this transformation rate of austenite influences in a large way the strain-hardening behaviour expressed by the $n_{incr.}$ curves. Specimens of steels HSiI and HSiII presents an initially low $n_{incr.}$ value that increases with plastic straining, thus allowing larger true uniform strains to be reached before the fulfilment of the Considère's criterion and the onset of necking. On the contrary, steel LSi presents a decreasing $n_{incr.}$ curve reflecting

Fig. 12. Evolution of the retained austenite content during plastic straining of the specimen of each steel grade optimising the performance index $\epsilon_u * \sigma_{TS}$.

the large austenite transformation in the early stage of plastic strain followed by its levelling off. Nevertheless, steel LSi presents an improved balance of strength and ductility thanks to the presence in the initial microstructure of thermal martensite (*i.e.* martensite formed during the quench to room temperature). The simultaneous occurrence of the TRIP effect and the strengthening due to the martensite is responsible of the mechanical performance of steel LSi. These results show that the best combinations of strength and ductility obtained with steels HSiI and HSiII steels can be attributed to the TRIP effect and its beneficial influence on the mechanical properties. In the case of LSi, both the TRIP effect and a 'composite strengthening effect'²⁰⁾ contribute to the improvement of mechanical properties.

4. Conclusion

This study investigates how a low silicon cold-rolled steel compatible with the industrial practice can be heat-treated in order to generate multiphase microstructures containing retained austenite. It is shown that excellent tensile properties in comparison with presently used steels or even high-Si TRIP-aided steels can be obtained thanks to the combination of a composite strengthening effect and a

TRIP effect.

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