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The effect of processing parameters on the microstructure and mechanical properties of low-Si transformation-induced plasticity steels

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Abstract

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Keywords

effect, low, transformation, induced, plasticity, si, microstructure, steels, mechanical, parameters, processing, properties

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The Effect of Processing Parameters on the Microstructure and Mechanical Properties of Low Si Transformation Induced Plasticity Steels

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Abstract

A base low Si, high Al Transformation Induced Plasticity (TRIP) steel and one with 0.03Nb and 0.02Ti (wt.%) additions were subjected to thermo-mechanical processing (TMP) and galvanising simulations. The microstructure and mechanical properties were analysed using a combination of optical and electron microscopy, X-ray diffraction and tensile testing and the results compared with those from intercritically annealed – galvanised steels. The addition of Nb and Ti results in microstructure refinement and an increase in the amount of the retained austenite after TMP which in turn, leads to increases in the tensile strength (~750 MPa) and the total elongation (~29%). A deterioration in the volume fraction of retained austenite and the mechanical properties was noted in both steels after the additional galvanising simulation. For the base steel, all TMP and galvanised samples presented with continuous yielding during tensile testing. The Nb-Ti steel exhibited discontinuous yielding and extended Lüders banding when TMP was followed by a longer coiling time. Both steels returned discontinuous yielding after the intercritical annealing – galvanising treatment. The discontinuous yielding behaviour was associated with the much finer ferrite grain size in the intercritically annealed steels and the ageing processes that take place during galvanising.

Keywords: TRIP steel, thermo-mechanical processing, galvanising, intercritical annealing, tension, microstructure, mechanical properties, scanning electron microscopy (SEM), transmission electron microscopy (TEM).

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1. Introduction

Since the transformation induced plasticity (TRIP) effect was first characterised by Zackay *et al.* [1], an enormous amount of research has been undertaken to understand the mechanism in detail and to introduce further improvements in TRIP-assisted steels. The latter class of steels is distinguished by their high strengths and elongation; both of which are key material parameters of interest to the automotive industry [2-5]. A typical TRIP steel microstructure consists of polygonal ferrite (PF) as the major phase along with varying fractions of bainite (B) and retained austenite (RA) and a minor phase of martensite (M). It has been suggested that the most important prerequisite for obtaining a desired combination of mechanical strength and ductility in TRIP steels [6,5] is the formation of a significantly high volume fraction of RA with sufficient stability. The volume fraction and stability of the RA is controlled by varying the chemical composition of the steel and/or processing parameters such as the magnitude of deformation and the coiling temperature/time. For example, while coiling at 465 °C compared to 400 °C led to an increase in the volume fraction of the RA [7], an intermediate coiling time resulted in an even larger volume fraction of RA [8]. On the other hand, Takahashi and Bhadeshia [9] found that even with a 15% volume fraction of RA, the improvement in uniform elongation was only 2% such that the further gains in ductility were the direct result of the contribution and interaction between the other phases and/or microstructural constituents [10,11].

Silicon is an important element that enhances the TRIP effect. Its high solubility in ferrite and low solubility in cementite suppresses the formation of the latter phase [3,12]. Thus, Si leads to a further carbon enrichment of the RA and a subsequent lowering of the martensite formation temperature. Furthermore, Si addition increases the mechanical strength by solid solution strengthening and it is claimed that co-alloying with P multiplies this effect. Barbe *et al.* [12] investigated the influence of P on a base 0.19C-1.68Mn-0.48Si-0.84Al steel with additions of 0.015P and 0.066P (wt.%). The study observed that the P increases the volume fraction and stability of the RA during prolonged bainitic transformation.

Microalloying additions of carbide or carbo-nitride forming elements such as Nb or Ti are used for microstructure refinement and to gain even further increases in mechanical strength [13-15]. Nb not only refines and strengthens ferrite but also mechanically stabilises the RA by reducing its size [16] and increasing its volume fraction [17].

Since a high Si content in TRIP steel usually degrades the adhesion of Zn during automotive sheet galvanising by the formation of a thin surface oxide layer, the element is usually substituted with Al. While some studies have found that Al substitution further stabilises the RA at room temperature and results in mechanical property improvements compared to Si-only TRIP steels [18], other investigations have suggested that the TRIP effect of Al is less than that of Si [19]. However, since the galvanisability of high Al TRIP steels is far better than high Si TRIP steels [7,20], significant resources have been devoted towards the further development of high Al TRIP steels using cold rolling and intercritically annealed (IA) techniques.

The production of TRIP steels by controlled thermo-mechanical processing (TMP) has distinct advantages over cold rolled and intercritically annealed methodologies as it reduces the number of processing stages and accordingly, decreases the cost of production while simultaneously boosting productivity. Initial attempts to produce thermo-mechanically processed TRIP steels have resulted in very good mechanical property combinations [20-22]. In the case of high Si steels, it has been shown that 25% thickness reduction in the austenite recrystallisation region followed by 47% thickness reduction in the non-recrystallisation region above A_{r3} results in the highest amount of the RA with optimum stability leading to an enhancement of mechanical strength and ductility [22].

In order to characterise TRIP steel behaviour during forming operations, different empirical models have been proposed to describe the rate of work hardening during uniform straining. While the Hollomon model [23] is limited to interpreting the work hardening behaviour of single phase steels, other empirical equations such as the Ludwik [24], Voce [25], Swift [26] and Ludwigson [27] relationships have only been partially satisfactory in interpreting complex phase steels [2]. Studies on dual phase steels [28,29] have shown that the modified Crussard-Jaoul (C-J) model (which is based on the Swift relationship) successfully describes its two stage work hardening behaviour. In multiphase TRIP steels, the work hardening rate is controlled by the dislocation accumulation mechanism as well as by the strain-induced austenite transformation [10]. Thus, further model modifications have to account for the evolving volume fraction and the strength of the various phases at different strains [30,31]. Since the work hardening rate of TRIP steels is typically characterised by three stages [31], the dynamic composite model [30] has been found to be the more appropriate.

This paper focuses on the effect of thermo-mechanical processing and subsequent galvanising simulations on the microstructure-property relationships in a base low Si TRIP steel and another with Nb and Ti additions. Further comparisons between these steels have also been made with their intercritically annealed counterparts. Since information on the evolving volume fraction of each phase at different strains is not available during tensile testing, the present study applies the modified C-J equations to characterise the work hardening behaviour.

2. Experimental and analytical procedure

A base low Si TRIP steel and another with further Ti and Nb additions (Table 1) were received as 6 mm thick hot rolled plate from GIFT-POSTECH. The three processing schedules conducted on 8 (RD) $\times$ 20 (TD) $\times$ 6 (ND) mm³ samples (Figs. 1a, 1b and 2) using a Gleeble 3500 thermo-mechanical simulator are summarised as follows: (i) thermo-mechanical processing with a short (TMP-S) and, (ii) a long coiling time (TMP-L) and, (iii) TMP-L followed by a typical industrial galvanising treatment (TMP-LG).

In the case of the first and second schedules (Fig. 2a), the samples were heated at 2 Ks⁻¹ to 1250 °C, held for 120 s followed by cooling to 1100 °C where a 25% roughing reduction was applied. The samples were then held for 120 s in order to condition the recrystallised austenite and then

cooled down to the finish rolling temperature in the non-recrystallised austenite region ($T_{FR} = 850\text{ }^{\circ}\text{C}$ for the base steel and $875\text{ }^{\circ}\text{C}$ for the Nb-Ti steel). Following a second 47% finishing reduction, the samples were slow cooled at 1 Ks^{-1} to the accelerated cooling start temperature ($T_{AC} = 680\text{ }^{\circ}\text{C}$ for the base steel and $690\text{ }^{\circ}\text{C}$ for the Nb-Ti steel) to form $\sim 50\%$ polygonal ferrite. At the T_{AC} temperature, the cooling rate was further increased to 20 Ks^{-1} to avoid pearlite formation. Finally, the sample was held at $470\text{ }^{\circ}\text{C}$ coiling temperature (T_C) for different coiling times (t_c) to form bainite and then water quenched. When $t_c = 125\text{ s}$, the short coiling time samples are denoted as TMP-S whereas when $t_c = 1200\text{ s}$, the long coiling time samples are referred to as TMP-L.

In the case of the third industrial galvanising schedule (Fig. 2b), the TMP-L samples were reheated at 25 Ks^{-1} to $550\text{ }^{\circ}\text{C}$ and held for 25 s to allow for carbon segregation at the surface of hot rolled steel and avoid the formation of a silicon oxide layer [32,33]. The samples were then cooled at 5 Ks^{-1} to $T_C = 465\text{ }^{\circ}\text{C}$ (above the zinc melting point), held for 5 s and air cooled to room temperature.

In order to compare the efficacy of the TMP-S treatment, an intercritically annealed-galvanising (IA-G) schedule was also undertaken on the two steels at GIFT-POSTECH. As shown in Fig. 2c, cold rolled samples of both steels were reheated to a temperature within the intercritical (ferrite-austenite) region ($T_{IA} = 863\text{ }^{\circ}\text{C}$ for the base steel and $871\text{ }^{\circ}\text{C}$ for the Nb-Ti steel) and held for 120 s to form $\sim 50\%$ polygonal ferrite followed by rapid cooling at 30 Ks^{-1} to the isothermal bainite formation temperature ($T_C = 465\text{ }^{\circ}\text{C}$). The sample was held at this temperature for a total of 125 s in order to account for bainite formation during the first 120 s and thereafter, galvanising in a zinc pot for 5 s . This was followed by air cooling at 30 Ks^{-1} to room temperature.

From the above, it is understood that the isothermal bainite formation stages for the TMP-S and IA-G schedules are similar as holding was undertaken for 125 s at $T_C = 470\text{ }^{\circ}\text{C}$ and $465\text{ }^{\circ}\text{C}$, respectively.

The microstructure was characterised by a combination of optical microscopy and secondary and transmission electron imaging. The austenite and martensite phases were distinguished optically using Klemm's [34,35] colour etchant. Here the weak nature of the etched film [35] resulted in colour scheme inconsistencies in some samples. Secondary electron imaging of the Nb-Ti -rich precipitates was undertaken on a JEOL JSM-7001F field emission gun - scanning electron microscope at $3\text{-}15\text{ kV}$ accelerating voltage. The determination of the chemical composition of 20-25 precipitates after the TMP-L (base and Nb-Ti steels) and the TMP-LG (Nb-Ti steel) schedules was performed using an Oxford Instruments 80 mm^2 X-Max energy dispersive X-ray spectroscopy (EDS) detector and the AZtec software suite. In order to characterise the morphology of the various phases, bright-field imaging was undertaken on $\varnothing 3\text{ mm}$ electropolished discs of the TMP-L samples of both steels using a JEOL 2011 transmission electron microscope operating at 200 kV .

The volume fraction of the RA was estimated from X-ray diffraction (XRD) conducted on the TD-ND plane using the direct comparison method for $2\theta = 60\text{-}100^{\circ}$ [36] and integrating the intensity

of the $(220)_\gamma$, $(311)_\gamma$, $(200)_\alpha$ and $(220)_\alpha$ peaks. The carbon content (wt.%) in the RA was calculated as follows [37]:

$$a_\gamma = \left[0.363067 + \frac{0.0783}{1 + 0.2151 \left(\frac{100}{C} - 1 \right)} \right] + \left[25.92 - \frac{51}{1 + 0.2151 \left(\frac{100}{C} - 1 \right)} \right] 10^{-6} (T - 727) \quad (1)$$

where a_γ is the RA lattice parameter and T is temperature ($^{\circ}\text{C}$).

Tensile dog-bones were wire-cut according to: (i) the sub-size ASTM-E8M standard [38] for the TMP-S, TMP-L and TMP-LG samples (Fig. 1c) and, (ii) the EN 10002 (Type 2) standard [39] for the IA-G samples (Fig. 1d). The use of a sub-size sample geometry was necessary for all TMP conditions due to the initial sample size limitations for thermo-mechanical processing on the Gleeble TMP simulator. Uniaxial tensile testing was conducted on a minimum of two dog-bone samples per condition at a constant strain rate of 4.5×10^{-4} using: (i) an in-house modified 5kN Kammrath and Weiss GmbH tensile stage for all TMP samples at UOW and, (ii) a 50 kN Instron 5569 screw-driven tensile machine for the IA-G samples at GIFT-POSTECH.

The yield strength (YS) is defined as the 0.2% offset proof stress or the point after Lüders banding is completed in the case of continuous and discontinuous yielding, respectively. Thereafter, the work hardening rate $\Theta = d\sigma / d\varepsilon$ and the strain hardening exponent (n-value) were calculated from the true stress and true strain curves [40]. The strain hardening exponent is an indication of the uniformity of strain distribution during forming operation such that the n value increases with greater thinning resistance during forming operations. The instantaneous strain hardening exponent is independent of the any power law and can be defined as $n = \left[(\varepsilon / \sigma) \cdot \Theta \right]$ [41]

3. Results

The microstructures and mechanical properties of the base and Nb-Ti steels after the TMP-S, TMP-L, TMP-LG and IA-G schedules are presented in Figs. 3-8 and are discussed concomitantly as per their relevance. Due to the rather significant variation in the tensile sample geometries of the TMP and IA-G samples, the comparison of their mechanical properties is limited to only discussing their overall trends in terms of yielding (continuous versus discontinuous) and work hardening behaviour.

3.1 The effect of composition on the microstructure and mechanical properties after the TMP-S schedule

The microstructures of the base and Nb-Ti steels after the TMP-S schedule are shown in Figs. 3a, 3b, 4a and 4b. In both steels, the microstructure consists predominantly of ~45-48% polygonal ferrite (PF) and ~35-40% bainite (B); with the remainder comprising the austenite and the martensite phases. The PF and bainite phases appear as light and dark grey after nital etching (Figs. 3a and 4a). Alternatively, the PF, bainite, austenite and martensite phases appear as light blue/brown, dark brown, white and black after colour etching, respectively (Figs. 3b and 4b).

PF tends to be of approximately equiaxed shape with $12\pm 8\ \mu\text{m}$ and $10\pm 6\ \mu\text{m}$ grain size (Table 2) for the base and Nb-Ti steels, respectively. The bainite lath thickness ranges between 0.5 to 1 μm such that they are slightly coarser in the base steel compared to the Nb-Ti steel.

The $\sim 7\%$ volume fraction of the RA phase was similar in both, the base and Nb-Ti steels. The RA is present as blocky grains between the PF grains or at the interface between the PF and bainite or as fine layers between the BF laths. In both steels, the RA grains are ~ 0.1 to $0.3\ \mu\text{m}$ thick* such that they are slightly coarser in the base steel compared to the Nb-Ti steel. The majority of the martensite phase was found between PF grains and at the interface of the PF and bainite.

Both steels exhibit continuous yielding and smooth work hardening curves after the TMP-S treatment (Figs. 5 a-d). While the base steel recorded yield and ultimate tensile (UTS) strengths of $491\pm 35\ \text{MPa}$ and $745\pm 6\ \text{MPa}$, respectively, the Nb-Ti steel returned an $YS = 474\ \text{MPa}$ and a UTS $782\ \text{MPa}$. In both steels, the uniform (UE) and total (TE) elongations were similar at $\sim 20\%$ and 29% , respectively. As seen in Fig. 5e, the TMP-S base steel reached a maximum n-value of 0.19 at a higher strain of ~ 0.06 , fluctuated around this value until ~ 0.14 strain and then slightly declined to $n \sim 0.18$ at 0.17 strain. In the TMP-S Ni-Ti steel (Fig. 5f), a maximum n-value of ~ 0.24 was reached at a higher strain of ~ 0.1 and remained steady until it declined slightly to $n \sim 0.22$ at 0.15 strain, followed by a further decline to $n \sim 0.18$ at 0.18 strain.

3.2 The effect of extended coiling time on the microstructure and mechanical properties

The microstructures of the base and Nb-Ti steels after the long coiling time TMP-L schedule (Figs. 3c, 3d, 4c and 4d) are similar to those after TMP-S. The values of the volume fractions and the grain size of the ferrite phase after TMP-L remained similar to those obtained after the TMP-S treatment. As shown in the TEM micrographs (Fig. 6), two bainite morphologies were observed after TMP-L comprising fine lathes of bainitic ferrite (BF) and plates of granular bainite (GB).

In stark comparison to the TMP-S schedule, the TMP-L treatment showed that the volume fraction of the RA phase had decreased to $\sim 4\pm 1\%$ in the base steel and increased to $\sim 13\%$ in the Nb-Ti steel as a direct result of microalloying (Table 2). Based on the micrographs after colour etching, the fraction of martensite was also slightly higher in the base steel compared to the Nb-Ti steel (Figs. 3d and 4d).

The EDS analysis of the precipitates in the TMP-L base steel revealed the presence of 100-200 nm (Al,Si)(N,O) and (Mn,Cu)S particles that were randomly scattered throughout the sample volume (Fig. 7a). In the TMP-L Nb-Ti steel (Fig. 7b), three major types of precipitates were observed: (i) 60–150 nm sized (Ti,Nb)(C,N), (ii) $< 100\ \text{nm}$ Nb(C,N) and, (iii) $> 40\ \text{nm}$ NbC (Figs. 7e-g).

The representative stress-strain, work hardening and instant-n curves are shown in Fig. 5 while the average data is given in Table 2. After the TMP-L treatment, the average yield (YS) and

* These measurements were undertaken using a combination of secondary electron and bright field imaging via scanning and transmission microscopy, respectively.

ultimate tensile (UTS) strengths are 445 ± 44 MPa and 722 ± 34 MPa for the base steel and 476 ± 7 MPa and 749 ± 20 MPa for the Nb-Ti steel, respectively. The uniform and total elongations are 15% and 25% for the base steel and 18% and 29% for the Nb-Ti steel, respectively. The base steel exhibits continuous yielding behavior which returns a smooth work hardening curve and instantaneous n -values with a maximum of 0.2 at 0.05 strain followed by a gradual decrease to 0.15 at ~ 0.15 strain. On the other hand, the TMP-L Nb-Ti steel shows slight discontinuous yielding such that the work hardening curve steeply declines up to 0.01 strain and has a local maxima at 0.02 strain. The n -value reaches a maximum of 0.24 between strains of 0.05-0.08 followed by a gradual decrease to $n \sim 0.18$ at 0.17 strain.

3.3 The effect of galvanising after TMP-L on the microstructure and mechanical properties

Figs. 3e, 3f, 4e and 4f show the microstructure of the samples after the galvanising TMP-LG schedule. In the base and Nb-Ti steels, colour etching returned more martensite (black areas) and a small number of white grains of the RA phase. These results were confirmed via XRD which also showed that the volume fraction of RA had decreased from $4\pm1\%$ and $13\pm0\%$ after TMP-L to $2\pm1\%$ and $6\pm4\%$ after TMP-LG in the base and Nb-Ti steels, respectively (Table 2). In addition to (Ti,Nb)(C,N) and Nb(C,N) particles observed in the TMP-L Nb-Ti steel, the formation of Fe_3C particles (Fig. 7h, 7i) was detected in this steel after galvanising (TMP-LG).

The tensile test results (Figs. 5a and b) show that the base steel underwent continuous yielding whereas the Nb-Ti steel depicted Lüders banding. Compared to the TMP-L samples, the TMP-LG samples recorded an increase in the YS of both steels (from 445 ± 44 to 476 ± 6 MPa for the base steel and from 476 ± 7 to 499 ± 6 MPa for Nb-Ti steel) while the UTS was found to have reduced from 722 ± 34 to 699 ± 9 MPa in the base steel and from 749 ± 20 to 685 ± 32 MPa in the Nb-Ti steel. Correspondingly, the uniform and total elongations in both the steels was also seen to reduce from 15-18% and 25-29% after TMP-L to 12-13% and 24-26% after TMP-G treatment, respectively.

The work hardening and instantaneous n -value curves (Figs. 5c-5f) show marked differences between the TMP-L and the TMP-LG schedules. In the TMP-LG base steel, the n -value declines from a strain of ~ 0.06 at a faster rate and reaches its lowest value of 0.11 at ~ 0.11 strain. In the TMP-LG Nb-Ti steel, the n -value fluctuates around the maximum n -value of ~ 0.195 between strains of 0.05-0.12. Overall, the n -curve for the Nb-Ti steel after TMP-LG records its lowest values compared to all other schedules.

3.4 The effect of intercritical annealing on the microstructure and mechanical properties

The secondary electron images of the microstructures after TMP-S and IA-G are shown in Figs. 8a, 8b and 8c, 8d for the base (Figs. 8a, 8c) and Nb-Ti (Figs. 8b, 8d) steels, respectively. It is obvious that the two processing techniques result in completely different microstructures. While the images record the similar volume fractions of PF for both steels, the PF grain size after TMP-S is

approximately twice that after the IA-G schedule (Table 2). After TMP-S, the austenite, bainite and martensite phases exist within banded areas whereas after the IA-G treatment, the same phases are equiaxed and interspersed between the PF grains. Compared to the TMP-S condition, both steels after IA-G possess slightly larger volume fractions of RA with lower carbon content.

The tensile curves after the IA-G schedules record discontinuous yielding in both steels (Figs. 5a and 5b). Compared to the TMP-S condition, the YS after IA-G is ~ 131 and ~ 36 MPa lower in the base and Nb-Ti steels, respectively (Table 2). In contrast, if the UTS values after the two schedules are compared, the values are ~ 42 MPa higher in the base steel and ~ 72 MPa lower in the Nb-Ti steel. In the IA-G samples, a visible change in the gradient of the work hardening curves occurs at ~ 0.016 strain for the base steel and ~ 0.03 strain for the Nb-Ti steel (Figs. 5c, 5d). In contrast to all other conditions, further changed in the work hardening rate occurs at the strains of ~ 0.14 and ~ 0.153 in the Nb-Ti IA-G steel. In both steels, the shape of the instantaneous n -value curve is very different after the IA-G schedules (Figs. 5e, 5f). A rather narrow peak with maximum n -values of ~ 0.39 and ~ 0.34 at 0.03 and 0.035 strains is followed by sharp declines to ~ 0.175 and ~ 0.19 at 0.175 and 0.13 strains for the base and Nb-Ti steels, respectively.

4. Discussion

4.1 The effect of processing parameters on the microstructure-mechanical property relationships

It should be kept in mind that in the TMP-S, TMP-L and TMP-LG schedules the parameters for austenisation and the formation of PF were kept constant; which also resulted in the formation of approximately similar (50%) volume fractions of PF. The isothermal bainite transformation temperature (= the coiling temperature, T_c) during simulated coiling was also kept similar (465-470 °C) such that only the coiling time (t_c) was varied between 125 s and 1200 s in the case of TMP-S and TMP-L, respectively. During the TMP-LG schedule, an additional galvanising treatment was employed only after the TMP-L treatment was completed.

The above variations in the processing schedules have a pronounced effect on the microstructure and the mechanical properties of the two steels (Table 2). For example, the YS of the base steel decreases with longer coiling time followed by an increase in strength after the TMP-LG treatment. On the other hand, the UTS values decrease with increasing coiling time in both steels such that an even further lowering of the UTS occurred after the TMP-LG schedule. With respect to ductility, both steels record a decrease in their UE and TE values with higher coiling times. In this case, the base steel was more severely affected with longer coiling times resulting in even smaller ductility values. From the above, it is clear that longer coiling times leads to an overall deterioration of the strength-ductility balance in both steels.

Although the PF grain size in the base steel after TMP-S could be regarded as only slightly coarser ($12 \pm 8 \mu\text{m}$) than the Nb-Ti steel ($10 \pm 6 \mu\text{m}$), the YS of the base steel after TMP-S is higher compared to the Nb-Ti steel. This disparity could be related to the complexity of the microstructure in

our multiphase steel and the dominant effect of other microstructural parameters such as the morphology of the bainite. The base steel consists predominantly of fine 0.35 μm sized plates of granular bainite (GB). On the other hand, the Nb-Ti steel mostly comprises significantly coarse 0.81 μm sized bainitic ferrite (BF) laths. Thus, it is entirely plausible that the variation in the dislocation mean free path between GB and BF is primarily responsible for the differences in the macroscopic mechanical properties of the two steels after the TMP-S treatment. Generally speaking, the combination of strength and elongation after the TMP-S and TMP-L schedules is slightly better in the Nb-Ti steel compared to the base steel. This is ascribed to the finer microstructure of the Nb-Ti steel and the additional solid solution and/or precipitation strengthening via alloying with Nb and Ti. While the above is in agreement with previous reports for high Si, Nb-containing steels [42], other variations between the two steels are detailed below.

During the longer coiling times, the formation of bainite is accompanied by the carbon enrichment of the RA which is located in-between bainitic ferrite laths or plates. If bainite transformation is completed with the steel remaining at a high temperature, the next stage of phase transformation is initiated wherein: (i) the carbon-enriched austenite starts to decompose with the formation of the ferrite phase and carbides and, (ii) in the remainder BF which is supersaturated with carbon, the segregation of carbon to dislocations and/or the formation of fine carbides takes place. The latter observations were noted previously during the bake hardening of high-Si TRIP steels [43].

It is clear that the bainite transformation was completed sometime between 125 s and 1200 s in the base steel, whereas this amount of transformation time was insufficient for the Nb-Ti steel. This is ascertained by the fact that the volume fraction of the RA decreases between the TMP-S and TMP-L treatments in the base steel whereas it continues to increase in the Nb-Ti steel (Table 2). Specific to the Nb-Ti steel, the significance of the interaction between the other phases in dictating the overall mechanical response is also highlighted here. The increase in the RA volume fraction helps the Nb-Ti steel to maintain its YS and TE levels while reducing the rate of decrease in the UTS and UE values after the TMP-S and TMP-L schedules.

However, the effect of a nearly doubled volume fraction of the RA phase content after the TMP-L schedules in the Nb-Ti steel underlines the more crucial effect of the alloying elements on the kinetics of the bainitic transformation. In the presence of Nb, the bainitic transformation is more sluggish as the prior austenite grain size is reduced [16] and this results in a low volume fraction of stable austenite; *i.e.*- a higher volume fraction of untransformed austenite with lower chemical stability such that it readily transforms to martensite upon tensile deformation.

During the TMP-LG industrial galvanising schedule, a further decomposition of the RA takes place; as evidenced by the low volume fractions of RA in both steels (Table 2). While both steels recorded increases in yield strength between the TMP-L and TMP-LG conditions, the UTS, UE and TE values were reduced. Since a larger volume fraction of RA pre-existed in the TMP-L Nb-Ti steel, the degradation in its mechanical properties after the TMP-LG schedule is more severe than that in the

base steel. In addition, while carbon segregates to dislocations and grain boundaries, it also forms carbides in the PF and BF phases after the TMP-L and TMP-LG treatments (Fig. 7b-7h). In particular, carbon segregation could take place in the areas of high dislocation density surrounding the martensite crystals that are newly formed upon quenching from the coiling temperature [43]. The small amount of martensite formed on cooling will also undergo a typical tempering process; with the formation of intermediate carbides or cementite during the TMP-LG schedule [44,45]. Thus, all these changes will contribute to the interaction of dislocations with carbon atoms and with precipitates during tensile testing such that they will result in an increase in YS and a reduction in ductility values.

An important difference in the stress-strain curves between the base and Nb-Ti steels is that the former shows continuous yielding up to the TMP-LG schedule (Fig. 5a) whereas the latter presents with discontinuous yielding after the TMP-L and TMP-LG treatments (Fig. 5b). Similar strain hardening behavior was observed for dual-phase and IA TRIP steels after pre-straining and bake hardening [46,42]. Discontinuous yielding via Lüders banding is typically evidence of dislocations locking either by Cottrell atmospheres and/or by fine precipitates and their subsequent unlocking or, the formation of new mobile dislocations [40].

As reported previously, new dislocations are generated in the ferrite phase either: (i) upon cooling after isothermal holding at 470 °C due to the volume change associated with the phase transformation of the unstable austenite to martensite or, (ii) during tensile testing when soft ferrite regions are adjacent to hard martensite or austenite crystals [43,46]. Since the Nb-Ti steel consistently returns higher volume fractions of RA than the base steel for corresponding conditions (Table 4), it could be expected that a higher number of mobile dislocations is generated in this steel as a result of the austenite to martensite transformation during straining. This could be one of the contributing factors to the pronounced yield point phenomena observed in the Nb-Ti steel after TMP-L and TMP-LG processing.

In addition, the difference in the yielding behaviors between the base and Nb-Ti steel can be further explained by considering the interaction of carbon with dislocations and the ability of the Nb, Ti and Fe atoms to form carbides. In the base steel, the interaction of carbon atoms with dislocations would be preferred mechanism as the binding energy between dislocations and carbon is 0.75 eV; which in turn, is higher than the 0.5 eV binding energy between the Fe and C atoms to form Fe₃C [47]. On the other hand, and to serve as an example, the binding energy between the Nb and C atoms to form NbC is 2.3 eV [42]. Thus, it could be speculated that the amount of C available in the base steel is sufficient to form saturated Cottrell atmospheres at dislocations. Contrarily, in the Nb-Ti steel, some of the carbon would be used up to form Nb-Ti carbides; thus leading to the formation of much weaker Cottrell atmospheres that are more easily unlocked on loading.

The appearance of Lüders strain in IA TRIP steels has also been previously attributed to the localisation of deformation within the ferrite phase; with whom the static ageing of solute carbon is also associated [48,49]. Thus, in the case of Nb-Ti steel, it can also be said that the discontinuous

yielding behavior is the combined effect of the finer ferrite grain size (leading to a localisation of the deformation within this phase) [50] and the ageing that occurs during the TMP-LG schedule.

To this end, the work hardening behavior of the steels was analysed using the modified C-J analysis [28,29]. Representative $\ln(d\sigma/d\varepsilon)$ versus $\ln(\sigma)$ plots are shown in Fig. 9 with the results from fitting presented in Table 3. Since the modified C-J analysis was unable to fit the flow curve over the entire uniform strain range region, the work hardening curves were divided into a maximum of three stages and different m -values were determined for each work hardening stage (Fig. 9).

If Fig. 9 is used as representative example, the behaviour of the Nb-Ti steel during each of the three stages is seen to vary rather significantly after the TMP-S (Fig. 9a) and TMP-L (Fig. 9b) schedules. This can be ascribed to the amount and the morphology of phase constituents; which in turn, control the behaviour during the various stages of work hardening. During Stage 1, the deformation of the soft ferrite phase leads to an accumulation of mobile dislocations in the regions near the interface between the ferrite and the harder phases such as RA and martensite. In Stage 2, the RA transforms to martensite due to the accumulation of strain energy in the RA phase. During Stage 3, the deformation of ferrite and martensite continues while the work hardening rate continuously decreases.

In Stage 1, the rate of dislocation accumulation is strongly linked to the ferrite grain size. In this regard, the influence of the finer ferrite grain size lead to the smallest m_1 -values being recorded after the IA-G schedule in both steels. Previous reports have also claimed that high m_1 -values are related to the larger volume fraction of martensite present in the initial microstructure [29]. However, the influence of this factor is not clear from the present study as the highest the m_1 -values were returned after the TMP-L and TMP-LG schedules for the base and Nb-Ti steels, respectively (Table 3).

Stage 2 behaviour that is more similar to dual phase steels [10] is returned after the TMP-S, TMP-L and TMP-LG schedules in the base steel and after the TMP-S schedule in the Nb-Ti steel (Fig. 9a). On the other hand, and as depicted in Fig. 9b, Stage 2 behaviour that is typically associated with the TRIP effect was noted after the TMP-L and TMP-LG treatments in the Nb-Ti steel. During Stage 2, the accumulation of strain energy in the RA phase occurs several times over and results in a gradual transformation of the RA to martensite. In turn, the phase transformation increases the work hardening rate (leading to a slope change) by inhibiting the dislocation glide process. Consequently, the differences in the behavior and slope of the Stage 2 hardening in the studied steels could be explained by rate of transformation of the RA during the uniform straining. This above phenomenon has been previously proven by observing areas of high dislocation density surrounding the newly formed martensite crystals [43].

The differences in the shape of strain hardening exponent curves over the period of uniform elongation allude to variations in the rate of strain-induced austenite transformation (Figs. 5e, 5f). In general, more gradual increases and sustained n -values are correlated with a slower transformation of the RA to martensite over longer strain ranges such that the TRIP effect then tends to contribute more

significantly towards extending the overall ductility of the steel. It should be noted that the authors had previously reported on the stability of RA during the uniaxial tensile testing of the base and Nb-Ti steels after TMP-L processing [51]. In that study, Electron Back-Scattering Diffraction was used to track the changes in the area fraction of the RA up to the UTS value. While the base steel recorded a more gradual change in the area fraction of RA from 5% (initial, $\varepsilon=0\%$) to 1.2% (at UTS, $\varepsilon=15\%$), the Nb-Ti steel exhibited a more rapid transformation of the RA fraction from 9.4% (initial, $\varepsilon=0\%$) to 1.4% (at UTS, $\varepsilon=18\%$).

To this end, the following examples highlight the importance of both, the volume fraction and the carbon content of the RA phase in dictating the overall shape of the strain hardening exponent curves of both steels. In the base steel, the gradual transformation of the highest amount of the RA with the highest carbon content after the TMP-S schedule was responsible for the observed n-value behavior and good combination of strength and ductility. In the case of the Nb-Ti steel, the most gradual transformation of the RA during tensile straining was obtained after the TMP-S schedule. This could be due to the carbon content of the RA after TMP-S being slightly higher than that obtained after the TMP-L schedule when the highest volume fraction of the RA was returned. Since the volume fraction and the carbon content (chemical stability) of the RA were the lowest after the TMP-LG treatment, both steels presented with sharp rises in the strain hardening exponent which in turn, is correlated with the rapid transformation of the RA to martensite during the early stages of straining. It should be noted that while the effect of chemical stability was used to explain the overall stability of the RA, the difference in the carbon content after TMP-S and TM-L is not significant. Thus, the overall stability of the RA could also be affected by other parameters such as the mechanical stability of RA.

4.2 Comparison of thermo-mechanically processed and intercritically annealed steels

Although both the steels in the TMP-S and IA-G conditions contained $\sim 50\%$ PF in the microstructure, the morphology and distribution of the phases varied significantly. During the TMP-S treatment, the formation of the PF grains takes place predominantly at the boundaries of the elongated prior austenite grains upon cooling at 1 Ks^{-1} such that the process is interrupted when cooling is further accelerated to 20 Ks^{-1} . As a direct result of the accelerated cooling, the bainite transformation then occurs in-between the rows of the PF grains during the isothermal holding step ($T_c = 465\text{ }^\circ\text{C}$ for $t_c = 125\text{ s}$ and/or 1200 s). Consequently, the majority of the RA and martensite crystals are located within the bainite regions. Due to such clearly developed directionality in the microstructure, significant anisotropy in the mechanical properties could be expected between the longitudinal and transverse directions of the samples.

On the other hand, the microstructure in the IA-G steels first developed as the reverse transformation of the cold rolled microstructure into a mixture of austenite and ferrite such that the subsequent formation of bainite occurred during isothermal holding. This resulted in a more homogeneous distribution of equiaxed PF grains and bainitic areas.

The main distinctive feature of the IA-G stress-strain curves of both steels is the rather pronounced yield point elongation and a continuous reduction in n-value with greater tensile strain. These results are similar to the behavior of tempered dual-phase steels [52] and of C-Mn-Si steels subjected to the quenching and partitioning treatment [53]. The yield point phenomenon indicates that during IA-G treatment, some dislocation pinning by carbon atoms took place in the ferrite phase and that based on the SEM observations, small carbide particles were also present in the areas of decomposed austenite (Fig. 8d inset). As discussed previously, the finer ferrite grain size combined with the ageing processes that take place during the IA-G schedule [49,50] could be responsible for the pronounced Lüders strain and the increase in the Lüders elongation.

In both IA-G steels, the work hardening behavior during Stage 2 was similar to that of TRIP steels (Fig. 9b), whereas in the TMP-S steels it was similar to the one of dual-phase steels (Fig. 9a), as discussed in Section 4.1. On the other hand, the presence of a single maximum n-value early on the tensile test in both IA-G steels indicates that a large amount of the RA transformed to martensite upon initial straining. This can be correlated to the fact that the RA is less stable in the IA-G steels compared to the TMP-S steels. This lower stability could be due to several factors among which slightly lower carbon content of the RA in IA-G steels and also the presence of refined and rigid bainite plates which could assist in an earlier load transfer and the transformation to martensite of adjacent RA layers [54].

5. Conclusions

The microstructure-property relationships in a base low Si, high Al TRIP steel and one with 0.03Nb and 0.02Ti (wt.%) additions were analysed after different thermo-mechanical processing schedules such that:

1. Thermo-mechanical processing of the base steel with a short coiling time of 125 s at 470°C resulted in the highest amount of the retained austenite phase and the best combination of mechanical strength and elongation. All thermo-mechanically processed base steel samples recorded continuous yielding behavior.
2. The addition of Nb and Ti refined the final microstructure of the steel after thermo-mechanical processing but led to an increase in the time required for the development of a high volume fraction of the stable retained austenite phase. Compared to the base steel, the strength-ductility balance in the Nb-Ti steel was slightly improved due to a combination of microstructure refinement, a higher volume fraction of the RA and precipitation strengthening. The Nb-Ti steel exhibited discontinuous yielding behavior after thermo-mechanical processing when a long coiling time of 1200 s was undertaken.
3. The intercritically annealed samples of the base and Nb-Ti steels showed discontinuous yielding with long Lüders elongation. A steep reduction in the instantaneous n-value from its maximum at a small value of tensile strain was noted. This was explained by the refinement of the polygonal ferrite grains and the ageing processes that take place during galvanising.

4. The modified C-J analysis showed three stages of work hardening in the studied steels. However, the Stage 2 behavior was found to differ with the adopted processing schedule and was associated with the different rates of the RA transformation.

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Figures list

Figure 1: Schematic of the sample geometry and dimensions (a) before and (b) after thermo-mechanical processing. Tensile dog-bones (c) wire-cut from the RD/TD plane along the TD direction of the TMP-S, TMP-L and TMP-LG samples and, (d) of the IA-G samples.

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Figure 4: (a, c, e) Nital and (b, d, f) colour etched microstructures of the Nb-Ti steel after (a, b) TMP-S, (c, d) TMP-L and (e, f) and TMP-LG schedules.

Figure 5: The variation in (a, b) the engineering stress versus strain, (c, d) the work hardening rate versus true strain and, (e, f) the instantaneous n-value versus true strain in the (a, c, e) base and (b, d, f) Nb-Ti steels.

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Figure 9: Representative work hardening behaviors shown using examples of Nb-Ti steel after (a) TMP-S and (b) TMP-L schedules. Based on the modified C-J analysis, three distinct stages of work hardening can be seen.

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(a)

(b)

(c)

Figure 2: Schematics of the thermo-mechanical processing schedules denoted as (a) TMP-S and TMP-L for short and long coiling times ($t_c = 125$ and 1200 s), (b) TMP-LG where an additional galvanising treatment is undertaken after TMP-L and, (c) IA-G involving intercritical annealing – galvanising treatment. T_{FR} = finish rolling temperature; T_{AC} = start of accelerated cooling temperature, T_C = coiling temperature; T_G = galvanising temperature; T_{IA} = intercritical annealing temperature.

(a)

(b)

(c)

(d)

(e)

(f)

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(a)

(b)

(c)

(d)

(e)

(f)

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(a)

(b)

(c)

(d)

(e)

(f)

Figure 5: The variation in (a, b) the engineering stress versus strain, (c, d) the work hardening rate versus true strain and, (e, f) the instantaneous n-value versus true strain in the (a, c, e) base and (b, d, f) Nb-Ti steels.

Figure 6: Representative bright field micrographs of the various bainitic morphologies seen in the base steel after the TMP-L schedule. PF = polygonal ferrite; BF = bainitic ferrite; M = martensite and RA = retained austenite.

Figure 7: Representative secondary electron micrographs of the (a) TMP-L base, (b) TMP-L Nb-Ti and, (c) TMP-LG Nb-Ti steels. (d-i) Example EDS spectra of (d) an $\text{Al}(\text{O,N})+\text{Cu}$ particle from the TMP-L base steel, (e) $(\text{Ti,Nb})(\text{C,N})$, (f) $\text{Nb}(\text{C,N})$ and (g) NbC particles from the TMP-L Nb-Ti steel, (h) Fe_3C particles in and, (i) the matrix of the TMP-LG Nb-Ti steel.

(a)

(b)

(c)

(d)

Figure 8: Representative secondary electron micrographs after (a, b) TMP-S and (c, d) IA-G processing of the (a, c) base and (b, d) Nb-Ti steels. In (d), the inset shows the initial decomposition of the RA phase.

(a)

(b)

Figure 9: Representative work hardening behaviors shown using examples of Nb-Ti steel after (a) TMP-S and (b) TMP-L schedules. Based on the modified C-J analysis, three distinct stages of work hardening can be seen.

Tables list

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Table 1: The nominal chemical composition of the two low Si TRIP steels in wt.%.

Steel	C	Mn	Si	Al	P	Nb	Ti
Base	0.15	2.00	0.30	1.00	0.05	-	-
Nb-Ti	0.15	2.00	0.30	1.00	0.01	0.03	0.02

Table 2: A summary of the simulation processing parameters, the phase statistics and the mechanical properties of the base and Nb-Ti steels.

Steel	Schedule	Processing parameters				Phase statistics				Mechanical properties			
		T _{AC}	T _C	t _c	T _G	Polygonal ferrite		Retained austenite		YS	UTS	UE	TE
						VF	GS	VF	C				
		°C	°C	s	°C	%	µm	%	wt.%	MPa	MPa	%	%
Base	TMP-S	680	465	125	--	48±1	12±8	7±1	1.3±0	491±35	745±6	20±3	29±2
	TMP-L	680	470	1200	--	47±1	12±6	4±1	1.2±0	445±44	722±34	15±0	25±4
	TMP-LG	665	470	1200	465	46±3	12±8	2±1	1.1±0.1	476±6	699±9	12±2	24±1
	IA-G	--	465	125	--	50	5	10	1.1	360	787	19	26
Nb-Ti	TMP-S	690	465	125	--	45±1	10±6	7	1.2±0	474	782	20	29
	TMP-L	690	470	1200	--	49±3	8±4	13±0	1.1±0	476±7	749±20	18±0	29±0
	TMP-LG	690	470	1200	465	47±1	11±7	6±4	1±0.1	499±6	685±32	13±2	26±2
	IA-G	--	465	125	--	50	5	8	1.1	438	710	20	30

Legend: T_{AC} = accelerated cooling start temperature; T_C = coiling temperature; t_c = coiling time; T_G = galvanising temperature; VF = volume fraction; GS = grain size; C = carbon content; YS = 0.2% proof stress; UTS = ultimate tensile strength; UE = uniform elongation; TE = total elongation.

Table 3: Comparison of the modified Crussard–Jaoul (C-J) analysis with the experimental results at the different stages of work hardening in the base and Nb-Ti steels.

Steel	Schedule	Modified C-J			Experimental
		Stage 1 m_1^*	Stage 2 m_2	Stage 3 m_3	
Base	TMP-S	7.66	4.05	4.91	6.18
	TMP-L	12.96	2.87	4.18	7.05
	TMP-LG	7.25	3.89	6.47	10.03
	IA-G	1.99	1.41	5.02	6.06
Nb-Ti	TMP-S	10.20	3.53	4.11	5.46
	TMP-L	13.69	-0.98	4.03	5.97
	TMP-LG	21.25	0.66	4.95	7.91
	IA-G	8.48	2.05	5.42	6.09

*1- m_{1-3} = slopes at Stages 1 to 3

$^{\dagger}m = 1/(\varepsilon_u - \varepsilon_y)$; where ε_u = maximum uniform true strain, ε_y = strain at 0.2% offset proof stress or the strain where Lüders banding is completed.