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Microstructure and mechanical properties of C–Si–Mn(–Nb) TRIP steels after simulated thermomechanical processing

I. B. Timokhina, E. V. Pereloma, and P. D. Hodgson

Continuous and discontinuous cooling tests were performed using a quench deformation dilatometer to develop a comprehensive understanding of the structural and kinetic aspects of the bainite transformation in low carbon TRIP (transformation induced plasticity) steels as a function of thermomechanical processing and composition. Deformation in the unrecrystallised austenite region refined the ferrite grain size and increased the ferrite and bainite transformation temperatures for cooling rates from 10 to 90 K s⁻¹. The influence of niobium on the transformation kinetics was also investigated. Niobium increases the ferrite start transformation temperature, refines the ferrite microstructure, and stimulates the formation of acicular ferrite. The effect of the bainite isothermal transformation temperature on the final microstructure of steels with and without a small addition of niobium was studied. Niobium promotes the formation of stable retained austenite, which influences the mechanical properties of TRIP steels. The optimum mechanical properties were obtained after isothermal holding at 400°C in the niobium steel containing the maximum volume fraction of retained austenite with acicular ferrite as the predominant second phase. MST/4582

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Introduction

High strength, formable steels are used in the automotive industry for weight reduction. Since the late 1970s a new class of TRIP (transformation induced plasticity) steels has been proposed based on Fe–0.2C–1.5Si–1.7Mn (wt-%).^{1,2} The microstructure of these new TRIP steels is attractive because they contain a significant amount of retained austenite with a multiphase nature consisting of polygonal ferrite, pearlite, bainite, and acicular ferrite.³ Retained austenite is expected to increase the strength–ductility balance as a result of the TRIP effect during subsequent room temperature forming. The amount and stability of retained austenite controls the mechanical properties of TRIP steels.⁴ Two main factors that affect the final microstructure, in particular the quality and quantity of retained austenite, are the composition of the steel and the thermomechanical processing schedule.

The primary aim of thermomechanical processing is to refine the ferrite grain size.⁵ The first objective is to control the austenite microstructure at the point of transformation. The accumulation of retained strain in unrecrystallised austenite affects the transformation to ferrite by increasing the number of possible nucleation sites for ferrite grains. This leads to a progressive reduction of the ferrite grain size after transformation during cooling to room temperature with increased deformation in the un- or non-recrystallised region. The ferrite grain size also depends on the cooling rate and original austenite grain size.^{5,6}

In steel with bainitic microstructure, another objective of thermomechanical processing is to refine the bainite structure by deformation in the unrecrystallised austenite region to increase the toughness of the steels. It has been reported that deformed austenite transforms to a smaller quantity of bainite, which is more refined,^{5,7} due to the retarding effect of ‘pancaking’. The bainite microstructure can be significantly improved by deformation of more

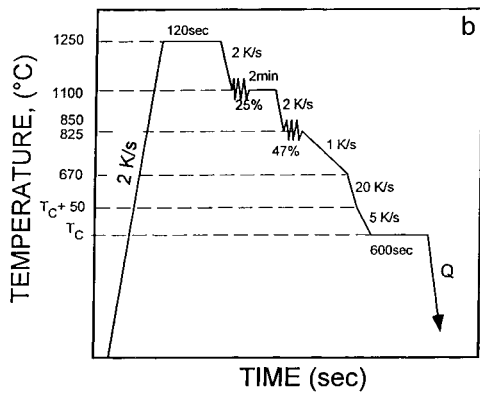
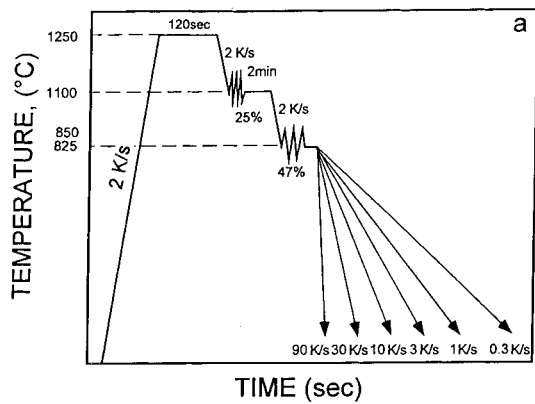
than 50% in the unrecrystallised region.⁸ Furthermore, thermomechanical processing influences the transformation behaviour leading to different morphologies of the second phase. The effect of this morphology is substantial. For example, acicular ferrite has been shown to result in a significant increase in the quantity and stability of retained austenite, which improves the room temperature mechanical properties of TRIP steels.⁸

The presence of microalloying elements in the steel can greatly enhance the microstructural control by precipitation before, during, or after the γ – α transformation.⁹ Niobium is often used to obtain a finer ferrite grain. Niobium in solid solution retards the recrystallisation of austenite, the phase equilibrium between austenite and ferrite, and the kinetics of the austenite to ferrite transformation.^{10,11} Also, niobium inhibits recrystallisation by a strain induced precipitation.^{12,13} Furthermore, the addition of niobium to TRIP steel increases the relative amount of retained austenite by approximately 25%, which can significantly improve the mechanical properties.¹⁴ Silicon refines the austenite microstructure, is a ferrite stabiliser,¹⁰ and accelerates the polygonal ferrite transformation.¹³ It also increases the volume fraction of retained austenite by retarding the precipitation of carbides during the bainite transformation.¹⁵

The main goal of the present work is to clarify the influence of the thermomechanical processing schedule and the composition on the transformation history and mechanical properties of TRIP steels.

Experimental

Two high silicon, high manganese TRIP steels with and without a small addition of niobium were investigated. The chemical compositions of the steels (designated Nb steel and Nb free steel respectively) are given in Table 1. The steels



a continuous cooling, unrecrystallised austenite condition; b discontinuous cooling, unrecrystallised austenite condition

1 Representative dilatometer schedules

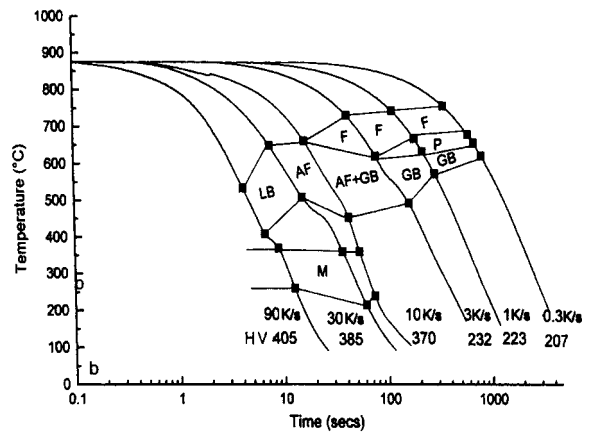
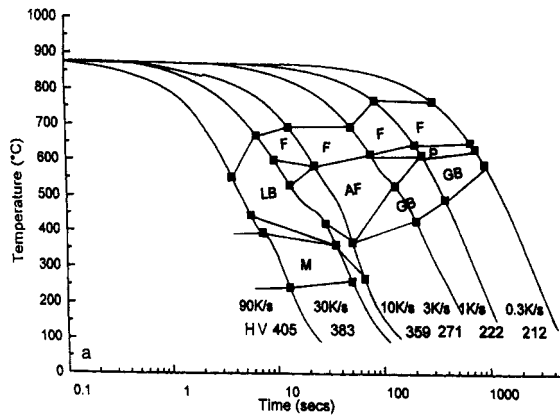
were received in the as rolled condition from BHP Research, Melbourne Laboratories. Cylindrical specimens, 8 mm in length and 4 mm in diameter, were machined from the plates with the longitudinal axis parallel to the longitudinal (rolling) direction. The experiments were performed using a MMC quench deformation dilatometer in a vacuum of 10 torr and the cooling rates were controlled by adjusting the helium gas flow. The experimental programme consisted of two studies as follows.

1. Continuous cooling transformation from the unrecrystallised austenite structure (Fig. 1a): specimens were solution treated at 1250°C for 120 s, cooled at 2 K s⁻¹ to 1100°C, deformed to 25% compressive strain, and held for 120 s to produce a uniform recrystallised austenite structure. Further deformation (47% in compression) was performed at 850°C for the Nb steel and 825°C for the Nb free steel in the unrecrystallised region. Then, the specimens were continuously cooled at 0.3–90 K s⁻¹.

2. Discontinuous cooling transformation after deformation in the unrecrystallised region (Fig. 1b): as for study 1, above, but after deformation in the unrecrystallised region, specimens were cooled at 1 K s⁻¹ to 670°C to form ~50% ferrite. This was followed by accelerated cooling at 20 K s⁻¹ to 50 K above the simulated coiling temperature T_c, after which the specimens were cooled at 5 K s⁻¹ to T_c (T_c=550, 500, 450, 400, and 350°C), where specimens were held for 600 s to undergo isothermal transformation to bainite, followed by quenching to room temperature. The

Table 1 Chemical composition of steels used, wt-%

Steel	C	Si	Mn	Mo	Ni	Cu	N	Nb	Fe
Nb	0.22	1.45	1.55	0.004	0.019	0.021	0.0057	0.039	Bal.
Nb free	0.21	1.55	1.55	≤0.01	0.009	0.003	0.0035	0.005	Bal.



a Nb steel; b Nb free steel

2 Continuous cooling transformation diagrams for steels in unrecrystallised austenite condition: F ferrite, AF acicular ferrite, P pearlite, GB granular bainite, LB lower bainite, M martensite

lower cooling rate to T_c was used to avoid undershoot in the holding temperature.

The continuous cooling transformation diagrams were constructed for each steel (Fig. 2). The start and finish transformation temperatures were determined from the dilation curves. Specimens were sectioned parallel to the deformation direction for Vickers hardness testing and optical metallography. The preparation of samples for optical examination involved standard metallographic procedures. The samples were etched with 2% nital to reveal the microstructure. Micrographs were taken along the deformation direction. Vickers hardness tests were performed with a 10 kg load and five indentations in the centre of the samples.

The volume fraction of retained austenite was ascertained by a tint etching technique and X-ray diffraction measurements. The colour etchant was a mixture of 4% HNO₃ + 7% (NO₂)₃C₆H₂OH + saturated Na₂S₂O₃.^{15,16} Quantitative analysis of retained austenite was carried out using VTHSCSA image analysis software. X-ray diffraction measurements of retained austenite were performed using a Philips PW1130 diffractometer (40 kV, 30 mA) to examine the integrated intensities of (200)_z, (211)_z, (200)_y, (220)_y diffraction of Cu K_α radiation.

Optical microscopy was supplemented by transmission electron microscopy (TEM) using a Philips CM20 microscope at an accelerating voltage of 200 kV. The foils for TEM were prepared by twin jet polishing using a solution of 5% perchloric acid in methanol at -30°C and an operating voltage of 60 V.

Room temperature mechanical properties were determined by a shear punch technique. This technique has been developed by Lukas *et al.*¹⁷ and is used when only a small

amount of the material is available and it is not possible to utilise standard mechanical testing techniques, as in the case of the present dilatometer specimens. A manufactured in-house rig similar to that of Lukas *et al.*¹⁷ was attached to an Instron tensile testing machine. A 5 kN load cell with 0.5 mm min⁻¹ crosshead speed was used in the tests. Samples were taken from the dilatometer specimens, parallel to the deformation direction in the range 350–650 µm thickness and 3 mm diameter punch. The test was instrumented to provide punch load–displacement data to determine tensile strength using the following empirical equation¹⁷

$$\sigma_{\text{eff}} = \frac{P - F}{2\pi r t} = C\sigma \quad \dots \dots \dots (1)$$

where P is the maximum load (N), σ is the corresponding uniaxial stress (MPa), F is the friction load (N), r is the punch radius (mm), t is the specimen thickness (mm), and C is a constant.

The correlation coefficient between the shear punch load–displacement curves and uniaxial tensile data had been determined in a previous investigation.¹⁸

The following empirical equation was used to calculate the total elongation¹⁷

$$El_{\text{total}} = d_f / t \quad \dots \dots \dots (2)$$

where El_{total} is the total elongation and d_f is the displacement at failure.

Results and discussion

EFFECT OF UNRECRYSTALLISED AUSTENITE CONDITIONS ON TRANSFORMATION BEHAVIOUR OF CONTINUOUSLY COOLED C–Si–Mn (–Nb) STEELS

The continuous cooling transformation diagrams are shown in Fig. 2. It is well known that deformation in the unrecrystallised region refines the ferrite grain size, increases the ferrite volume fraction, and raises the transformation start temperature.^{5,7,8} Therefore, the specimens were deformed in both the austenite recrystallisation region, to refine the austenite and subsequently the ferrite grain size, and the unrecrystallised region, to study the further refinement of the ferrite grain size. For all steels deformed in the unrecrystallised region the thickness of the ‘pancaked’ austenite grains decreased from 100–250 to 20–40 µm.¹⁹ Ferrite grain refinement was also due to the formation of additional nucleation sites inside the austenite grains (deformation bands, twin bands, and dislocation arrays).¹³ The hardness increased from ~200 HV10 at the slow cooling rate (0.3 K s⁻¹) to ~400 HV10 at the fastest cooling rate (90 K s⁻¹). Moreover, both the kinetics of the transformation start and the progress of the transformation were affected by the amount of deformation. The ferrite transformation temperatures for unrecrystallised austenite conditions were from ~10 (0.3–3 K s⁻¹) to 50 K (10–90 K s⁻¹) higher than for the recrystallised austenite conditions for both steels.¹⁹ Hence, the ferrite transformation is accelerated depending on the amount of deformation, in agreement with previous work.⁵ Furthermore, it was observed that the temperatures at which the unrecrystallised austenite transformed to bainite at fast cooling rates (10–30 K s⁻¹) were ~30 K higher than for the recrystallised austenite condition.¹⁹ This behaviour was not clearly observed at slow (0.3–3 K s⁻¹) cooling rates. Deformation in the unrecrystallised region also reduced the bainite packet size; thus refining the bainite structure by increasing the density of nucleation sites and retarding bainite growth by deformation ‘debris’ in the austenite.⁷

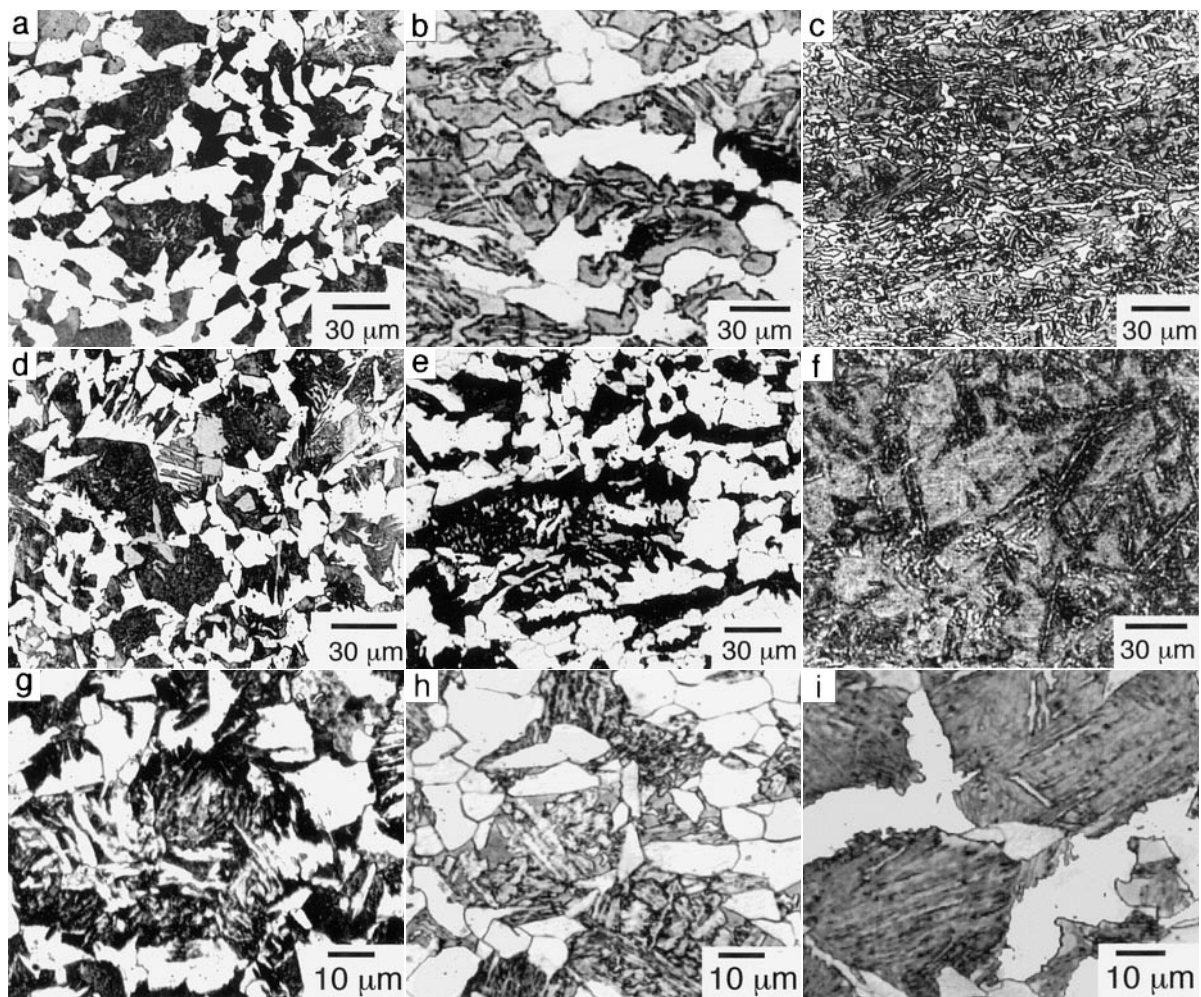
Representative microstructures for both steels for the different thermomechanical simulation tests are shown in Fig. 3. It was found that in the Nb free steel the recrystallised prior austenite grain size was about twice that in the Nb steel and the ferrite grains formed on transformation were also coarser. Hence, niobium retards the recrystallisation process by a solute drag mechanism on the recrystallised grain boundary.^{11,13} Furthermore, the addition of niobium can also refine the ferrite grains due to niobium carbide precipitates that form in ferrite and control the ferrite grain size.⁹ The volume fraction of ferrite gradually decreased with increasing cooling rate from 0.3 to 30 K s⁻¹. The ferrite transformation start temperatures for the Nb steel were ~20 K higher than for the Nb free steel, which suggests that niobium accelerated the ferrite transformation, most probably through the decrease in the austenite grain size before pancaking. The formation of ferrite in the Nb steel still occurred up to 30 K s⁻¹, whereas in the Nb free steel it formed only up to 10 K s⁻¹. The volume fraction of Widmanstätten ferrite was higher in the Nb steel, because niobium in solution favours the formation of Widmanstätten ferrite by reducing the transformation rate of proeutectoid ferrite formation through solute drag.¹¹

For all steels, a bainite microstructure was obtained as the second phase over a wide range of cooling rates (0.3–30 K s⁻¹). The bainite morphology changed from upper bainite, granular bainite, and acicular ferrite at low cooling rates to acicular ferrite and lower bainite at higher cooling rates. Microstructures consisting of granular bainite and acicular ferrite are characterised by the absence of carbides, the presence of isolated regions of austenite and martensite between crystals of bainitic ferrite, and ferrite crystals, which may, or may not, have a lath or plate morphology.^{20–22} Upper bainite consists of lath or plate shaped ferrite units arranged in packets and interlath carbide precipitates.²³ Lower bainite is characterised as a microstructure in which carbide particles are found within the bainitic ferrite plates.²⁴ However, there were also significant amounts of other phases such as polygonal ferrite and pearlite formed at 0.3–3 K s⁻¹ and martensite at 10–90 K s⁻¹ (Fig. 3). The addition of niobium increased the bainite transformation temperatures by ~20 K. The microstructure of acicular ferrite in the Nb steel was already formed at 1 K s⁻¹ and still occurred at 30 K s⁻¹, whereas in the Nb free steel acicular ferrite was observed only at 3–30 K s⁻¹. The above results confirm the findings reported in the literature that niobium in solution increases the ferrite and bainite start transformation temperatures but delays the transformation kinetics by solute drag.^{11,13}

The volume fraction of acicular ferrite in the Nb steel was noticeably higher than in the Nb free steel. The intragranular formation of carbide free acicular ferrite is accompanied by an increased concentration of carbon in retained austenite, enhancing its stability. However, it is well known²¹ that the amount of acicular ferrite formed decreases with the reduction in prior austenite grain size due to the elimination of a number of the intragranular nucleation sites. This causes the formation of less acicular ferrite in unrecrystallised austenite compared with recrystallised austenite.¹⁹ The addition of niobium reverses this trend, leading to the higher volume fraction of acicular ferrite in Nb steel deformed in the unrecrystallised region compared with Nb free steel. This may be due to the increase in the number of intragranular nucleation sites formed in austenite owing to the presence of niobium in the steel. However, further clarification of this effect is required.

A martensitic microstructure was observed at 10 K s⁻¹ in both steels.

Hence, the transformation behaviour can be controlled by deformation in the unrecrystallised region. However, the



Nb steel continuously cooled at *a* 1 K s^{-1} , *b* 3 K s^{-1} , and *c* 30 K s^{-1} ; Nb free steel continuously cooled at *d* 1 K s^{-1} , *e* 3 K s^{-1} , and *f* 30 K s^{-1} ; Nb steel discontinuously cooled to *g* $T_c=550^\circ\text{C}$, *h* $T_c=450^\circ\text{C}$, and *i* $T_c=350^\circ\text{C}$

3 Typical microstructures of steels continuously or discontinuously cooled from unrecrystallised austenite

influence of the alloying elements on the transformation behaviour also needs to be considered.

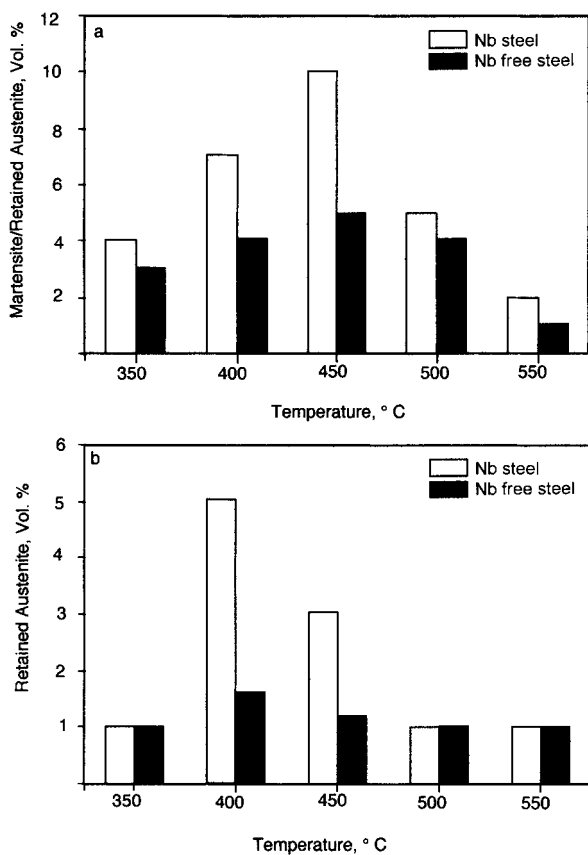
EFFECT OF BAINITE ISOTHERMAL TRANSFORMATION TEMPERATURE ON FINAL MICROSTRUCTURE OF DISCONTINUOUSLY COOLED C-Si-Mn(-Nb) STEELS IN UNRECRYSTALLISED AUSTENITE CONDITION

In practice, most hot rolling operations would be followed by cooling to a certain temperature followed by a hold or slower cooling. In hot strip mills, the strip is rapidly cooled by water on the run-out table followed by coiling where the strip cools very slowly. The discontinuous schedule in the present work approximated the hot strip mill procedure. Both steels were subjected to a discontinuous thermomechanical processing schedule (Fig. 1). The evolution of the microstructure obtained at the different isothermal holding temperatures for the Nb steel is shown in Fig. 3. For both steels, TEM studies confirmed the presence of polygonal ferrite, granular bainite, acicular ferrite, and martensite/retained austenite constituent. All specimens, after cooling at 1 K s^{-1} to 670°C contained approximately 45–50% polygonal ferrite. Ferrite formation was followed by accelerated cooling at 20 K s^{-1} to avoid pearlite. The bainite isothermal transformation time of 600 s was chosen because the shorter time leads to the low carbon content in retained austenite, which decreases its stability. A longer time leads to the supersaturation of the austenite phase with

carbon and subsequent formation of carbides, resulting also in a reduction in the volume fraction of retained austenite.^{3,12,16}

For both steels, the microstructure after the isothermal hold at 350°C consisted predominantly of lower bainite and blocky martensite and/or retained austenite in addition to polygonal ferrite. For the Nb steel, acicular ferrite was the main second phase at 400°C with lower bainite and martensite and/or retained austenite, whereas for the Nb free steel, lower bainite was still the main phase with acicular ferrite and martensite and/or retained austenite. For both steels isothermally held at 450 and 500°C , granular bainite was the major second phase with islands of martensite and/or retained austenite within bainitic ferrite. The other phases observed were acicular ferrite and upper bainite. Increasing the isothermal holding temperature to 550°C led to a more conventional bainite microstructure such as upper bainite with the addition of pearlite, with the volume fraction of pearlite higher for the Nb free steel. Hence, the final microstructure strongly depends on the bainite isothermal transformation temperature.

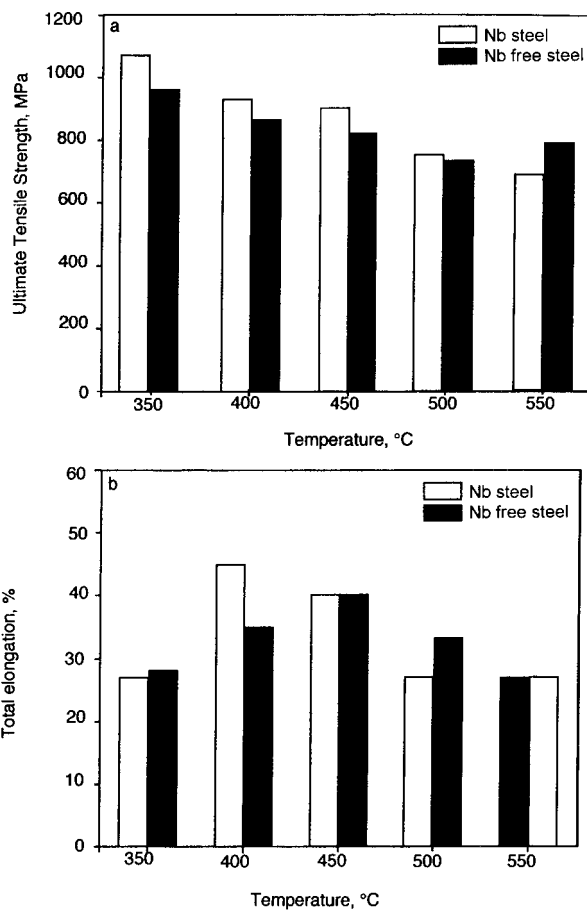
The formation of the carbide free acicular ferrite and granular bainite structures was stimulated by isothermal holding at 400°C for the Nb steel and at 450 and 500°C for both steels. At these temperatures, the time lag of carbide precipitation was too long for carbides to form in the microstructure and this would enhance the stability of retained austenite. The volume fraction of martensite/retained austenite constituent for both steels (Fig. 4a)



4 Effect of isothermal holding temperature on *a* volume fraction of martensite/retained austenite constituent and *b* volume fraction of retained austenite

measured using colour etching, showed a maximum at 450°C corresponding to the maximum content of granular bainite in the microstructure. However, the volume fraction of martensite/retained austenite constituent for the Nb steel was significantly higher than for the Nb free steel. X-ray analysis showed that the highest volume fraction of retained austenite was achieved at 400°C in the Nb steel (Fig. 4b), which contains acicular ferrite as a main phase with retained austenite in the form of thin interlayers, enclosed between bainitic ferrite laths. Hence, it is possible to achieve a microstructure having a significant volume fraction of martensite/retained austenite constituent and stable retained austenite by varying the thermomechanical processing parameters and by the addition of niobium. It is important to remember that this was with the same volume fraction of ferrite, although the distribution of ferrite in the two steels would be different because of the different recrystallised austenite grain size.

The mechanical properties of the Nb steel and the Nb free steel at different isothermal temperatures are shown in Fig. 5. The ultimate tensile strength increased with decreasing holding temperature for both steels, due to the changes in bainite morphology and volume fraction of martensite/retained austenite constituent. However, the ultimate tensile strength of the Nb free steel was significantly lower than for the Nb steel due to the finer bainitic microstructure in the Nb steel and Nb(C,N) precipitation strengthening of the ferrite (and potentially other phases). The maximum elongation was observed at 400 and 450°C for both steels. At these temperatures, the two steels contained the maximum volume fraction of retained austenite and carbide free bainitic structures, such as granular bainite and acicular ferrite, implying an enhanced stability of retained austenite due to the higher



5 *a* ultimate tensile strength and *b* total elongation for Nb steel and Nb free steel after isothermal holding at different temperatures

carbon content when no carbides form in the bainite. The addition of niobium increased the volume fraction of retained austenite and consequently promoted the TRIP effect (Fig. 4). However, the elongation at 450°C was similar for both steels (Fig. 5). Thus, the greater amount of retained austenite in the Nb steel did not lead to a greater elongation. It seems that even if the volume fraction of retained austenite increased, the stability of retained austenite and corresponding elongation were reduced possibly due to the low carbon content in the retained austenite at the maximum volume fraction of retained austenite and also a significant presence of martensite in the microstructure.²⁵ On the other hand, the maximum uniform elongation at 400°C in the Nb steel, at the maximum volume fraction of retained austenite and amount of acicular ferrite was higher than in the Nb free steel. Therefore, it is suggested that the morphology of acicular ferrite stabilised the retained austenite and improved the elongation. Thus, considering the UTS × El, the optimum mechanical properties were at a coiling temperature of 400°C corresponding to acicular ferrite as the predominant phase with maximum volume fraction of stable retained austenite at this temperature.

In summary, the Nb and Nb free TRIP steels contain a complex mix of ferrite and bainitic phases, which affect the nature, distribution, and stability of martensite/retained austenite constituent that is present after cooling to room temperature. This phase mixture and morphology is also affected by the nature of the austenite at the point of transformation.

It is still not clear how the various morphologies and volume fractions of retained austenite affect the final

mechanical properties. To investigate this will require a systematic study of the TRIP effect for the steel that formed the optimum level of retained austenite.

Conclusions

1. The effect of niobium on the kinetics of phase transformation, final microstructure, and mechanical properties of C–Si–Mn(–Nb) steels has been studied. The results have confirmed that niobium promotes the formation of acicular ferrite. Reduction of the prior austenite grain size and also an increase in the number of ferrite nucleation sites due to the second deformation in the unrecrystallised austenite region increases the ferrite transformation start temperatures (by up to 20 K) and bainite transformation temperature at high cooling rates ($10\text{--}90\text{ K s}^{-1}$) for both steels. With increasing cooling rate the microstructure changes from predominantly polygonal ferrite, pearlite, and granular bainite to acicular ferrite and martensite.

2. The addition of niobium extends the range of isothermal holding temperatures leading to the formation of carbide free bainite structures such as acicular ferrite and granular bainite. The volume fraction of martensite/retained austenite constituent and retained austenite is higher in the Nb steel.

3. The results have shown that not only is the amount of retained austenite present important for optimum mechanical properties, but also its morphology and stability.

Acknowledgements

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