Introduction of nanoprecipitation and transformation-induced plasticity in ultra-low carbon medium manganese quenching-partitioning-tempering steels

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ABSTRACT

Combined nanoprecipitation hardening and transformation-induced plasticity (TRIP) effects have been introduced in ultra-low carbon medium manganese steels for enhanced strength, plasticity and toughness. The mechanical properties and designed microstructure evolution were investigated after an innovative quenching-partitioning-tempering (QPT) treatment with different initial conditions, cold rolling (CR) and heat rolling (HR). The transmission electron microscopy (TEM) combined with 3D atom probe tomography (APT) observed plenty of block austenite (30%) with dispersed precipitation in CR-QPT steels, while less amounts of film austenite (18%) with higher density of nanoprecipitates in HR-QPT steels. The controlled multiphase microstructure evolution is based on the Mn diffusion and segregation process. The unloading-reloading tests reveals the respective roles of precipitation hardening and TRIP in the overall mechanical properties according to the Baushinger effect (BE): the nanoprecipitation results in a higher back stress strengthening, while the deformation-induced martensite transformation in a wide strain regime degrades the large stress concentration in grain boundaries, leading to a back stress softening but effective stress hardening in the later deformation stage.

Introduction

With manufacture and safety consideration, an increasing demand in automotive application is focused on medium Mn steels \cite{1–3} of lean alloy system designed with enhanced strength, ductility and toughness as the third-generation of advanced high strength steels (AHSS). The good plasticity mainly origins from a large fraction of ultrafine-grained austenite providing various transformation-induced plasticity (TRIP) and twinning-induced plasticity (TWIP) effect \cite{4–6}. However, the addition of austenite stabilizer element carbon is always limited due to the weldability performance and detrimental Portevin-LeChatelier (PLC) effect \cite{7}, which restricts the strength. Currently, one of the effective solutions \cite{8} is to adopt the combination of nanoprecipitation hardening and TRIP concepts to improve both strength and ductility simultaneously without the expense of massive alloying expense. The introduction of intragranular nanoscale precipitations act as strong obstacles to impede dislocation motion without initiating cracks at grain boundaries \cite{9}, thereby substituting for traditional strength enhancement such as grain refinement and cold deformation without compromising plasticity \cite{9,10}.

Intercritical annealing \cite{11} was demonstrated to be capable of obtain enough amounts of austenite by sufficient element partitioning into transformed austenite for stabilizing. A new approach of medium manganese steels quenching-partitioning-tempering; nanoprecipitation hardening; Baushinger effect; grain boundary embrittlement
precipitates [17] will decrease the concentration in solid solution and lower the stacking fault energy (SFE) of austenite, thus strongly influencing the TRIP effects. In particular, the precursor plays a crucial role in the microstructure evolution with Mn partitioning. The cold deformation prior to the austenite reversion treatment [18] has been adopted in medium Mn steels to obtain metastable austenite with a dispersed size distribution and varying micro-mechanical stability, which is also a consequence of non-uniform distribution [19] of Mn in austenite. Thus, the effects of hot and cold deformation before heat treatment are investigated with different number density of dislocations as nucleation sites for austenite reversion and precipitation.

On the other hand, although the combination of two strengthening and toughening mechanisms has been investigated in some alloy systems, the respective role of precipitation hardening and TRIP effect on the overall mechanical properties have not yet received much attention in medium Mn steels. The previous research [20] demonstrated that the large plasticity is unlikely predominantly caused by individual stimulation-induced martensite transformation because the newly martensite can also results in good strain hardening as strong inclusions [21]. The mechanical properties of multiphase are closely related to the deformation modes of constituent phases [22] and the stress partitioning resulting from the microstructural heterogeneity [23] during deformation. As reported in TRIP steels [24], long range internal stresses or back stresses arise from the plastic heterogeneity by the generation of geometrically necessary dislocations [25–27] (GND), contributing to a significant strain hardening and affecting the final ductility. As Baushinger effect [28–30] (BE) can provide a method for determine the contributions of the back stress and dislocation hardening to the flow stress, the kernel average misorientation [31,32] (KAM) can also show the deformation-induced local orientation gradients inside grains indicating the GND contribution. Consequently in this study, the respective roles of nanoprecipitation hardening and TRIP effect have been investigated by the evolution of flow stress components determined by the combination of the KAM characterization and modified BE methods.

In this paper, an ultra-low carbon medium Mn steel with enhanced strength and plasticity. The alloy system of Fe-Mn-Ni-Al-C was used in this study. The alloys were melted and cast to round billets of 1 kg in vacuum. After homogenization at 1473 K for 60 min, Hot-forged swaging was conducted in eight passes between 1000 and 1300 K and then water quenching to room temperature, resulting in hot-rolled plates size of 300 mm × 240 mm × 25 mm.

A one-step aging of partitioning and two-step aging of partitioning and tempering for various periods of time respectively marked with ‘QP’ and ‘QPT’ process, and then water quenching. In comparison, extra cold rolling for multiple passes for a total reduction of ~75% before QPT procedure applied to introduce more nucleation sites for austenite reversion. The nominal compositions and corresponding aging procedure in this study are listed in Table 1.

Plate-type sub-sized (the gage section 16 mm × 5 mm × 1.6 mm) tensile tests at room temperature were conducted on a Zwick (BTC-T1-FR020 TN.A50) universal testing machine, the tensile axis is parallel to the rolling direction, with a constant strain rate of 4 × 10⁻⁴ s⁻¹. The tensile load-unload-reload (LUR) tests were conducted. The condition for LUR tests was the same as that of monotonic tensile tests. Vickers hardness measurements were conducted under a 1000 g applied load and a dwell time of 15 s, and the average hardness from twelve different measurements was reported. Dilatometric samples (10 × 4 × 4 mm³) from the cold-rolled and hot-rolled samples were conducted a two-step partitioning-tempering treatment.

Interrupted tensile tests at RT were carried out on samples at different strains of 3%, 7%, 11%, 15% and 20% to evaluate the microstructure evolution during deformation. The microstructure morphology and austenite volume fraction were characterized using scanning electron microscopy (SEM), saturation magnetization (SM) measurement and X-ray diffraction (XRD), Transmission electron microscopy (TEM) with energy dispersive spectrometer (EDS) using a nanoscale electron beam and 3D atom probe tomography (APT) were carried out to study the size and spatial distribution of the precipitates. Deformed microstructures were characterized by electron backscattering diffraction (EBSD Oxford) to identify the GND distribution with microstructure evolution. EBSD specimens were electro-polished for about 35 s in a mixture of 10 vol. % perchloric acid and 90 vol. % glacial acetic acid at room temperature with an applied potential of 50 V. Thin TEM foils were prepared by a twin-jet electrochemical polisher at 40 V in a 7 vol. % HClO₄ acetic acid electrolyte at −20°C, and then investigated in a JEOL JEM 2100F field emission transmission electron
microscopy operated at 200 KV. APT was performed with a local electrode atom probe (LEAP 3000 HR). The testing temperature is around 50 K under ultra-high vacuum \((4.5 \times 10^{-11} \text{ Torr})\) with a pulse fraction of 0.2 and a pulse repetition rate of 200 kHz.

**Results and discussions**

**Mechanical properties of QPT steels**

Figure 1 shows the engineering stress-strain curves and corresponding strain hardening rate curves at room temperature for three pretreatment conditions. All the mechanical properties are presented in Table 2. At RT, all the yield strengths (YS) of QPT steels have improved from 650 MPa to 900 MPa compared to QP steels due to precipitation hardening effects, corresponding to the hardness results (Table 2), while it is surprising that the total elongation doesn’t drop but increases from 20% to more than 25%. Good combinations of ultimate strength (1 GPa) and total elongation (20–25%) are all obtained for two states. However, there exists a multistage stress-strain relationship (marked as ‘plateau’ and ‘strain hardening’) and an unexpected ‘convex’ phenomenon connected with multiple deformation mechanisms in CR-QPT steels. In contrast the HR-QPT steels almost show a stable strain hardening rate during deformation.

**Multiphase microstructure evolution associated with the Mn partitioning process**

To reveal the microstructure evolution process, the SEM phase maps of QP and QPT steels are represented in Figure 2. The HR-QP steels show a typical morphology of tempered martensite but enough ultrafine austenite has formed in the CR-QP steels during the one-step partitioning process. After the following tempering procedure, the morphology and volume fraction of austenite almost remain stable in CR-QPT steels while austenite in HR-QPT steels is found nucleating in the lath interfaces. Typical TEM microstructures of the two QPT steels are shown in Figure 3. In HR-QPT steels, there exists a large density of nanoparticles of B2-ordered structure distributed dispersedly in the bcc matrix, which can be identified as NiAl intermetallic. The selected area diffraction (SAED) reveals a film-like morphology of transformed austenite, exhibiting a Kurdjumov–Sachs (KS) orientation relationship with martensite, namely \([101]_g/\langle111\rangle_a\) and \((111)_g/\langle011\rangle_a\). However, the CR-QPT steels show microstructural constitution of block austenite and heterogeneous nanoprecipitations in ferrite, of which the number fraction is less than that in HR-QPT steels.

To characterize the precipitation behavior in two QPT steels, three-dimensional selected atom maps together with one-dimensional concentration profiles of the constituent elements in HR-QPT and CR-QPT steels are shown in Figure 4. It is indicated that the number density and average diameter of precipitates in HR-QPT steels are much larger than those in CR-QPT steels, corresponding to the TEM results. The Mn enrichment is also detected accompanied with NiAl precipitates. The average element contents of Ni, Al and Mn in the bcc matrix and precipitates are given in Table 1.

**Table 1. Nominal compositions and corresponding aging procedure of cryogenic steels [8].**

| Pretreatment | C (wt.%) | Mn (wt.%) | Ni (wt.%) | Al (wt.%) |
|--------------|----------|-----------|-----------|-----------|
| CR-QP        | 0.01     | 7         | 2.5       | 1.5       |
| CR-QPT       | 0.01     | 7         | 2.5       | 1.5       |
| HR-QPT       | 0.01     | 7         | 2.5       | 1.5       |

**Table 2. All the mechanical properties for three pretreatment conditions conducted at different temperatures [8].**

| Pretreatment | Yield strength (YS) (MPa) | Ultimate tensile strength (UTS) (MPa) | Total elongation (EL) (%) | Vickers hardness (HV) |
|--------------|--------------------------|--------------------------------------|--------------------------|-----------------------|
| CR-QP        | 647 ± 15                 | 928 ± 23                             | 20.3 ± 1.7               | 287 ± 5              |
| CR-QPT       | 971 ± 20                 | 984 ± 13                             | 27.9 ± 1.5               | 359 ± 4              |
| HR-QPT       | 902 ± 15                 | 998 ± 16                             | 24.8 ± 0.8               | 365 ± 5              |

CR: cold rolling; HR: hot rolling. QP: quenching-partitioning; QPT: quenching-partitioning-tempering.

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**Figure 1.** The engineering stress-strain (a) and corresponding strain hardening rate (b) curves at RT for three pretreatment conditions [8].
in Table 3. The proportion of element in nanoprecipitates shows the existence of Ni(Al, Mn) phases with Mn substituting mainly for Al \[33,34\] in all two steels. In general, the microstructure evolution process during the multistage heat treatment is closely connected with the partitioning of the alloying element such as strong austenite stabilizers Mn and Ni \[35\]. A quantitative analysis by means of dilatometry was carried out to determine the partitioning of Mn during QPT process. Figure 5 shows the dilatometric strain changes related to austenite formation and Mn partitioning process. It’s indicated that the strain contraction was observed during the partitioning treatment while the strain almost remained stable in the following tempering process, which is attributed to the available austenite reversion process only at high temperatures. Besides, the volume change of austenite reversion can be measured by the related strain \[35–38\]. Meanwhile, the onset of martensite transformation temperatures from $\gamma$ to $\alpha$ can be estimated from the reference formula \[39\]. In consequence, the contents of the partitioning elements Mn and Ni, and the

![Figure 2. SEM phase maps (a, b, c, d) and X-ray diffraction pattern (e) of QP and QPT steels [8].](image)

![Figure 3. TEM microstructures of the (a) HR-QPT and (b) CR-QPT steels [8].](image)
estimated \(M_s\) temperatures are listed in Table 4. It is obvious that the reverse austenite in the HR state is not enough stable for a poor partitioning degree. However in the CR state, the partitioning content of Mn element to austenite is so abundant that a considerable amount of reverse austenite has remained even directly quenched to room temperature, resulting from a higher driving force from cold deformation.

As summarized, in the HR-QPT steels, the reverse austenite easily transforms to martensite again due to a low partitioning level during the high temperature aging stage. In the following tempering process, the secondary austenite reversion occurs meanwhile a large density of NiAl nanoprecipitates nucleate around the defects in ferrite. The element Mn will segregate in the austenite/ferrite interfaces and precipitates for Ni(Al, Mn) substitution with the aging time increasing. However, the CR-QPT steels show a higher transformation rate due to high density of dislocations as nucleation sites, leading to large amounts of reverse austenite while the precipitation kinetics are poor owing to low element contents in the matrix. Therefore, the multiphase microstructure evolutions, including the number density of precipitates, the morphology of austenite and its mechanical stability during deformation strongly depend on the Mn diffusion process during QPT treatment.

### Table 3. The average element contents (at. %) of Ni, Al and Mn in the bcc matrix and precipitate respectively [8].

| Average element content (at. %) | Matrix | Precipitate |
|--------------------------------|--------|-------------|
|                               | Mn     | Ni          | Al       | Mn     | Ni          | Al       |
| HR-QPT                         | 6.74   | 1.95        | 0.27     | 15.76  | 46.55       | 32.76    |
| CR-QPT                         | 2.94   | 0.35        | 1.88     | 12.09  | 40.69       | 30.74    |

### Figure 4. Three-dimensional atom maps of Ni, Al and Mn with corresponding one-dimensional concentration profiles of the constituent elements in (a, b) HR-QPT and (c, d) CR-QPT steels [8].

#### Figure 5. Dilatometric strain changes related to austenite formation and the associated Mn partitioning during QPT treatment [8].

#### Load-unload-reload stress-strain behavior and KAM characterization during deformation

To probe the various deformation mechanisms originating from such designed multiphase microstructure in two QPT steels, LUR tests were conducted as shown in Figure 6(a,b). Typical features of hysteresis loop and unload yield effect are evidence of the BE in unload-load cycles (Figure 6(b)). Interestingly, there exists a distinction of stress-strain response after 10% strain between CR-QPT and HR-QPT steels. Obvious necking stage is observed in HR-QPT steels while a renewed strain hardening process occurs in CR-QPT steels, which demonstrates that there exists a transition of deformation mechanism in the later deformation stage. What's more, the macroscopic stress can be divided into the effective stress required locally for...
dislocation move and back stress associated with long-range stress on mobile dislocations, which all have been schematically defined and expressed previously [23]. The evolutions of the back and effective stresses in three pretreatment conditions are presented in Figure 6(c–e). In CR-QP steels, all the two stresses have just increased a little during deformation, and then decrease in the necking stage. Compared with the QP steels, the QPT steels all exhibit a significant back stress strengthening but the effective stress component almost remains stable in the previous deformation stage. However, with the strain exceeding to 10%, it is indicated that an obvious back stress softening and effective stress hardening occur in the CR-QPT steels while the back stress still maintains a high value even in the later deformation stage in the HR-QPT steels. Figure 6(f) shows the volume fraction changes of austenite in CR-QPT and HR-QPT steels after interrupted tensile tests. It is demonstrated that the austenite has almost transformed totally after 10% strain in the HR-QPT steels while the TRIP effect occurs over a wide strain regime, specifically at high strains in the CR-QPT steels.

Furthermore, EBSD characterization was conducted for the two QPT steels in deformed states with the engineering strain $\varepsilon = 10\%$. The KAM maps and values which reveal the evolution of the substructure in bcc with deformation are shown in Figure 7. The initial austenite in two QPT steels exhibits a block-like and film-like morphology respectively. What’s more, negligible amount of austenite has remained at 10% strain in the HR-QPT steels. As shown in Figure 7(e,f), although both steels exhibit similar average KAM values at the initial states, the bcc structure in HR-QPT steels shows a higher value of misorientation than that in CR-QPT steels at equivalent high strains, resulting from different neighboring austenite transformation process.

Although the overall tensile properties of CR-QPT and HR-QPT steels are roughly the same, there exhibits a multistage stress-strain curve including the plateau and a renewed strain hardening process in the

| Partitioning process | $X_\text{Mn}$ (%) | $X_\text{Ni}$ (%) | $X_\text{Mn}$ (%) | $X_\text{Ni}$ (%) | Ms (°C) |
|----------------------|------------------|------------------|------------------|------------------|---------|
| HR                   | 14.8             | 8.5              | 4.52             | 85.2             | 1.95    | 252.41 |
| CR                   | 33.3             | 15.04            | 6.29             | 67.7             | 2.94    | 0.35   | 22.30  |

Table 4. The contents (at. %) of the partitioning elements Mn and Ni, and the estimated $M_s$ temperatures of CR and HR samples during the partitioning process [8].

Figure 6. Load-unload-reload stress-strain behavior (a, b) and the effective stress and back stress evolution with plastic strain for (c) CR-QP, (d) CR-QPT and (e) HR-QPT steels [8].
CR-QPT steels. Indeed, the back stress and effective stress evolutions can be divided into several stages during deformation. In stage 1, compared to the CR-QP and CR-QPT steels, the higher density of nanoprecipitates in HR-QPT steels contribute to a more significant back stress strengthening in the early deformation stage, which is consistent with a higher work hardening rate value (Figure 1(b)). On the other hand, a large amount of geometrically necessary dislocations \[40,41\] will be generated at phase boundaries to accommodate the strain gradient \[42\], leading to the dislocation pile-ups around the precipitates and the interfaces. During further straining up to the stage 2, significant deformation-induced martensite transformation dominates to accommodate for the large stress concentration in GBs. In CR-QPT steels, the improved strain hardening can be explained through an effective stress hardening with pronounced dislocation accumulation in grain interiors. Especially an unexpected ‘convex’ phenomena in the strain hardening curves is attributed to the transition of deformation mechanism from precipitation hardening to martensite transformation prevailing in a wide strain regime \[6\]. However, sustaining high back stress can’t be effectively relieved in HR-QPT steels due to insufficient TRIP effects after 10\% strain, corresponding to a higher average KAM value of ferrite in HR-QPT steels that CR-QPT steels (Figure 7). Furthermore, the higher internal stress in GBs cause grain boundary decohesion and damage-crack formation, which becomes a potential for the final necking process in stage 3. Both two stresses decrease in this deformation stage.

Generally, the introduction of nanoprecipitation will result in a significant back stress strengthening with lower dislocation generation in the matrix and constrained GNDs in the ferrite/austenite interface, while the deformation-induced martensite transformation in a wide strain regime degrades the large stress concentration in grain boundaries, resulting in a back stress softening and effective stress hardening in the later deformation stage. The cooperative multiple deformation mechanisms contribute to a good combination of strength and plasticity.

Conclusions

In the present study, combined nanoprecipitation hardening and transformation-induced plasticity effects have been introduced in ultra-low carbon medium Mn steels by an innovative QPT treatment with different initial conditions, cold rolling (CR) and hot rolling (HR). The designed multiphase microstructure evolution was determined by the Mn diffusion process. What’s more, the respective roles of nanoprecipitation hardening and TRIP effect have been investigated. Following conclusions can be made.

(1) Combined nanoprecipitation hardening and transformation-induced plasticity effects can be controlled by the innovative quenching-partitioning-tempering process. It was found that block austenite (30\%) with plenty of nanoprecipitates formation in CR-QPT samples while less amounts of film austenite (18\%) but higher density of nanoprecipitation in HR-QPT materials.

(2) The introduction of nanoprecipitation hardening results in higher back stress strengthening, while the stimulation-induced martensite transformation in a wide strain regime in CR-QPT steels degrades the large stress concentration in grain boundaries, resulting in a back stress softening and effective stress hardening in the later deformation stage. However, sustaining high internal stress in GBs of HR-QPT steels also make it easy for damage-crack formation, which becomes a potential for the final fracture.
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Disclosure statement

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