Effect of thermomechanical processing on microstructural evolution in precipitation strengthened ferrite steel

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Abstract

The present study is focused on processing simulation in Gleeble® to understand the effect of thermomechanical processing parameters, especially thin-slab casting rolling (TSCR) process, on the ferrite grain structure and precipitation in a nano-precipitation strengthened ferritic steel. The double-hit compression test confirmed that heavy deformation in the range of 900–850 °C can accumulate sufficient strain in deformed austenite that is required to achieve significant ferrite grain refinement. Subsequent simulation of conventional hot-strip mill processing route showed fully ferritic microstructure upon isothermal holding at and above 650 °C (for coiling simulation). Holding at lower temperatures (≤ 600 °C) generate pearlite and bainite, which are undesirable for formability. Finally a TSCR-type of processing schedule is applied maintaining a constant deformation temperature (870 °C) and isothermal holding temperature (650 °C) to understand the effect of equalization entry temperature (T_{eq}). A low equalization entry temperature (900 °C) was found to be beneficial to generate fine ferrite grains with fine precipitates during processing by TSCR route which is a novel finding.

Keywords Thermo-mechanical simulation · Ferritic steel · Thin slab casting and rolling · Equalization entry temperature · Precipitation strengthening

1 Introduction

The improvement in fuel efficiency by reducing the vehicle weight and the reduction in emission of greenhouse gases have been the driving force behind the development of high strength and advanced high strength steel (AHSS) grades such as dual phase, complex phase, transformation induced plasticity and bainitic-martensitic steels [1–3]. Depending on the design, the higher strength can ensure better fatigue resistance and improved crash performance. Besides high strength (yield strength preferably greater than 750 MPa), good ductility and formability are the essential requirements of AHSS [4, 5]. Better the formability lesser the requirement of welding in the manufacturing of automotive body parts. That is not only economical but

also beneficial for the structural stability as weld joints are the weak points of any structures [4, 5]. The steel grades mentioned above however, contain two or more microstructural constituents with different strength levels. Such a microstructure leads to the strain localization and the defect generation either along the interphase boundaries or within any of the constituent, affecting the formability [3–5]. A single phase ferrite microstructure is beneficial as it provides uniform deformation during forming ensuring satisfactory hole-expansion and stretch-flangeability properties. A uniform distribution of fine scale nanometer sized (typically less than 10 nm) microalloyed precipitates i.e. carbides and nitrides of V, Nb and Ti can increase the strength of ferrite by 200–300 MPa through precipitation strengthening, without hampering the formability [6–13].

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The alloy design of AHSS is certainly important but it is not the sole determining factor for obtaining the desired microstructure and properties. Thermomechanical processing parameters typically the soaking temperature, controlled rolling schedule, cooling rate and coiling temperature tailor the ferrite grain size as well as the size, distribution, frequency and morphology of the precipitates [6–13]. Industrial processing of high strength microalloyed ferritic steels is presently performed following either of the two different processing routes: Hot Strip Mill (HSM) route or Thin-Slab Casting and Rolling (TSCR) route.

Conventional HSM processing starts with reheating and soaking treatment (typically at 1200 ± 20 °C) of continuous cast slab, which is cold, thick and wide (> 200 mm thickness, up to 2.0 m width) [14–20]. Thickness reduction by rolling operation comprises of initial deformation in ‘reversible roughing mill’ (to 25–30 mm) followed by final deformation in tandem finishing mill (to 2–8 mm). After finish rolling at around 850–900 °C, the strip is accelerated cooled by water spraying (at 20–30 °C/s) and coiled over the range of 250–700 °C. As there is little scope for alternation in initial processing stages, the coiling temperature remains to be the primary controlling factor to tailor the microstructure, precipitation and the consequent mechanical properties of the industrial HSM strip. Several studies have been carried out on the microstructural evolution and precipitation during conventional HSM processing of high strength ferritic steels [5–20].

The TSCR process is more recent and energy efficient as compared to HSM [21–33]. In TSCR the steel retains the heat during the continuous operation of successive processing stages involving thin-slab casting, equalization, rolling, cooling and coiling treatments. Cooling of the slab below the ferritic transformation is avoided until the coiling operation. Therefore the scope for alternation of processing parameters is even less in TSCR than that in HSM. Stringent process control is also necessary in TSCR due to the coarse initial austenite grain structure and limited scope for hot deformation [21–33]. Contrary to HSM, the hot and thin as-cast slab is directly inserted into the tunnel furnace for the equalization treatment typically performed over 1100–1200 °C. During industrial operation, while transforming the cast slab from the mould to the equalization furnace, its temperature drops to 900–1000 °C, which can be considered as the ‘equalization entry temperature’ (T_{eet}). Such a temperature drop in TSCR can be attributed to the thinner slab size (compared to HSM slab) with lesser heat content. If the

influence of this temperature drop on the microstructure and precipitation is well understood then the ‘equalization entry temperature’ can also be externally controlled for the process improvement. None of the previous studies investigated this aspect and it is addressed here.

In view of the microstructural requirements of high strength ferritic steel, i.e. fully ferrite microstructure with finest possible grain size and nano-precipitation strengthening, the first part of the study covered a double-hit hot compression testing and a conventional HSM-type processing simulation with the aim of selecting the suitable deformation temperature and isothermal holding temperature (for coiling simulation), respectively. Subsequently a TSCR-type processing simulation was applied based on the selected deformation temperature and isothermal holding temperature, varying the T_{eet} over the range of 900–1000 °C, before the simulation of equalization treatment which goes up to 1160 °C. The objectives of TSCR-type simulation study are twofold: (1) understanding the effect of T_{eet} on the ferrite grain structure and precipitation in TSCR-type processing and (2) preforming a comparative assessment of the microstructure and hardening obtained in the thermomechanically simulated samples following conventional HSM-type processing and TSCR-type processing. This simulation study may not exactly replicate the TSCR processing (where the as-cast thin slab cools down directly and rolled in a continuous process), however, is expected to identify the effect of the temperature drop to T_{eet} at the onset of the equalization process. Therefore instead of TSCR simulation, the applied schedule is referred as TSCR-type processing simulation.

2 Materials and methods

The chemical composition of the investigated steel is given in Table 1.

It is a low-C microalloyed steel containing V, Mo and Nb and a small amount of Ti. The steel is cast, subjected to a prolonged homogenization treatment to remove any segregation and hot-forged into 15 mm thick plate. Cylindrical samples (10 mm diameter and 15 mm height) were prepared from the hot-forged block and were subjected to thermomechanical processing simulation using Gleeble® 3800 simulator in Tata Steel R&D, Jamshedpur.

Table 1 Chemical composition (wt%) of the investigated steel

C	Mn	Si	S	P	V	Mo	Nb	Ti	Al	N
0.05	1.5	0.1	0.01	0.025	0.2	0.2	0.02	0.005	0.06	0.006

2.1 Thermomechanical simulation

In order to select the appropriate deformation temperature, the classical double-hit tests [34, 35] were performed in Gleeble® as shown in Fig. 1a. After reheating to 1200 °C, samples were cooled down to different deformation temperatures (850–1000 °C) and subjected to consecutive compressive deformation passes (ϵ per pass = 0.2) isothermally at different interpass intervals (1–1000 s) as in Fig. 1a. Flow curves, i.e. true stress versus true strain curves, for both the passes were analyzed to determine the softening fraction over the interpass time following an established method [34, 35]. Double-headed blue arrows in Fig. 1a indicate the extent of interpass softening primarily due to the static recrystallization of the hot deformed austenite. The softening fraction obtained at different deformation temperatures as the function of interpass interval is plotted in Fig. 1b.

It is evident from Fig. 1b that the interpass softening fraction reduced significantly with the decrease in deformation temperature to the range of 900–850 °C, which is expected due to the strain-induced Nb(C,N) or even V(C,N) precipitation [36–38]. Therefore 870 °C was selected as the deformation temperature. In view of the requirement of significant ferrite grain refinement for improving the strength and toughness, a heavy single-stage deformation (uniaxial compression) applying a true strain, $\epsilon = 1.0$, at a strain-rate, $\dot{\epsilon} = 1/s$, is selected for the subsequent simulation studies.

Two different industrial processing schedules, one following conventional HSM route, Fig. 2a, and another

following TSCR-type processing route, Fig. 2b, were simulated in Gleeble®. A couple of K-type thermocouples attached to the samples (refer to the inset in Fig. 2a) monitored the temperature at ± 3 °C accuracy during Gleeble® testing.

In conventional processing simulation, Fig. 2a, the samples were reheated at 5 °C/s to 1160 °C, soaked for 3 min, cooled down to 870 °C at 10 °C/s and subjected to uniaxial compression. From the deformation temperatures the samples were rapidly cooled at 30 °C/s (simulating accelerated cooling) to different temperatures over the range of 550–700 °C (at an interval of 50 °C), isothermally held at that temperature for 1 h, followed by air cooling. Isothermal holding treatment was performed to simulate the industrial coiling, where the steel cools down very slowly from the coiling temperature. The purpose of this simulation study is to select the appropriate isothermal holding temperature at which fully ferrite microstructure with fine grain size and precipitates can be achieved.

In TSCR-type of processing simulation, Fig. 2b, the samples were soaked at 1200 °C for 3 min, cooled down to the typical range of T_{aet} of 900–1000 °C at 10 °C/s, reheated slowly to 1160 °C over 20 min at heating rate 0.13–0.20 °C/s (simulating the equalization treatment) and immediately cooled at 10 °C/s to the chosen deformation temperature of 870 °C. The temperature, strain, strain-rate for deformation and the subsequent cooling schedules applied in TSCR-type simulation were identical to the corresponding conventional processing simulation. The selection of the constant isothermal holding

Fig. 1 **a** Schematic diagram showing classical double-hit tests performed in Gleeble® along with the flow curves (stress–strain curves) at different temperatures. The extent of interpass softening on the

flow curves was shown by double headed blue arrow. **b** Variation of softening fractions with interpass time at different temperatures calculate from flow curves

Fig. 2 Schematic diagrams showing the thermomechanical processing schedules performed in Gleeble simulator following industrial **a** HSM and **b** TSCR route. The soaking/deformation temperatures and soaking times along with heating and cooling rate

are depicted in the figures. Inset in Fig. 1a showing dimensions of Gleeble sample along with the location of K-type thermocouples attached with the sample

temperature of 650 °C for the TSCR-type simulation (Fig. 2b) is based on the findings of the conventional processing simulation (Fig. 2a).

The continuous cooling-transformation diagram of the investigated steel was experimentally determined from the dilatometric study by cooling down the small cylindrical samples (4 mm diameter and 6 mm height) at different average cooling rates using BAHR® DIL 805A/D quenching and deformation dilatometer.

2.2 Microstructure and precipitate study

Microstructures were prepared from the thermocouple attached cross sections of the investigated samples following the standard metallographic techniques. Detailed microstructural studies were carried out using optical microscope with image analysis attachment and scanning electron microscope (SEM). From each sample near about 500 grains were measured to determine the average ferrite grain size following the equivalent circle diameter (ECD) method [39].

High resolution transmission electron microscopy (HR-TEM) was carried out on the thin foils (300 nm) prepared by ion milling technique from the thermocouple attached locations of the compression tested samples. JEOL® ZEM-2100 and Philips® CM200 microscopes having a resolution of 0.2 nm were used for the analysis with an operating voltage of 200 kV. Morphological and compositional studies of the precipitates were carried out with a converged beam diameter of 20 nm in order to collect maximum signal from the precipitates. Energy dispersive spectroscopy (EDS) analysis was performed using Oxford® detector that includes XPP correction scheme. The precipitates located

close to the perforation of the foils were only analyzed to minimize the compositional effect of the underneath matrix. The volume fraction of the precipitates was calculated by using the number density (number of precipitates per unit area), average size (average diameter and cord-length of spherical and cuboidal precipitates respectively) and the foil thickness (i.e. 300 nm). To characterize the precipitates ten bright field TEM images were analyzed from each sample. LV-700 model LECO® Vickers Hardness Tester was used for macrohardness measurements with a load of 20 Kg and dwell time of 15 s. The reported hardness values were based on the average of five readings taken from each sample.

3 Results and discussion

3.1 Transformation behaviour and microstructure of thermomechanically simulated samples

The time–temperature–transformation (TTT) diagram of the investigated steel was predicted from JMatPro® software version 8.0, Sante Software Limited, Fig. 3a. The range of isothermal holding temperatures (T_{hold}) used in the conventional processing schedule are indicated by the dotted lines on the TTT diagram in Fig. 3a. TTT diagram indicates that primarily ferrite is expected to form at higher T_{hold} (650–700 °C) with a very small amount of pearlite or distributed iron-carbide particles. The decrease in T_{hold} is expected to increase the fraction of bainite and pearlite, following Fig. 3a. The CCT diagram in Fig. 3b, which also includes the cooling curves for different cooling rates, indicates that continuous cooling at 10–30 °C/s

Fig. 3 **a** TTT and **b** CCT diagrams of the investigated steel predicted from JMatPro software. Isothermal holding temperature and continuous cooling curves are marked by dotted and solid lines

respectively in the figures. The start and finish temperatures of different phases are also shown by colour legends

Fig. 4 **a–d** Optical micrographs of the Gleeble samples after processing via Conventional HSM route (Conv.) with different isothermal holding temperatures, as depicted in Fig. 1a. Distinct phases

are identified from high magnification SEM micrographs in the inset of the respective figures. *F* Ferrite, *P* Pearlite and *B* Bainite

is expected to generate predominantly ferrite–bainite type microstructures.

The optical micrographs of the conventionally processed samples for different T_{hold} are given in Fig. 4. To help in the identification of different microstructural constituents, high magnification secondary-electron SEM images are inserted into the optical micrographs. The microstructures of the conventionally processed samples comprised of predominantly ferrite phase, Fig. 4.

Ferrite grains, however, were not necessarily polygonal, rather often quasi-polygonal in shape especially at lower T_{hold} . Such ferrite grain structures can be expected as the

transformation occurred during isothermal holding at the temperatures (550–700 °C) much below the equilibrium ferrite transformation start—($A_{e3} \sim 860$ °C) and end temperatures ($A_{e1} \sim 710$ °C) based on the prediction of Thermo-Calc®.

The fraction of microstructural constituents and the average ferrite grain sizes of the conventionally processed samples are listed in Table 2.

The ferrite grain size increased with the increase in T_{hold} , Table 2. The SEM insets in Fig. 4 and the fractions of different microstructural constituents given in Table 2 verify the prediction of TTT diagram about the formation of pearlite and bainite at a lower T_{hold} , i.e. 550–600 °C. According to Fig. 3, the pearlite and bainite start temperature for the investigated steel can be as high as 670 °C and 620 °C, although bainitic transformation is kinetically favoured over pearlitic transformation. As a result higher fraction of bainite was found at the T_{hold} of 600 °C and 550 °C, than pearlite, Table 2. Typical bainitic sheaves penetrating into the ferrite grain can be observed in Fig. 4b. A complete ferrite matrix was found in all the simulated samples at T_{hold} of 650 °C and above. Therefore, 650 °C was selected as the isothermal holding temperature for TSCR-type processing simulation for understanding the effect of T_{eet} .

Table 2 Phase fractions and mean ferrite grain sizes with standard deviations of Gleeble® simulated samples for HSM schedule as the function of isothermal holding temperature

Isothermal holding temperature (°C)	Microstructural constituents (%)			Mean ferrite grain size (μm)
	Ferrite	Pearlite	Bainite	
550	92 ± 2	3 ± 1	6 ± 1	3.54 ± 2.69
600	95 ± 2	1 ± 0.3	5 ± 1	3.69 ± 2.63
650	100	–	–	4.01 ± 3.54
700	100	–	–	4.14 ± 3.12

Fig. 5 a–c Optical micrographs of the Gleeble samples after processing via TSCR route with different equalization entry temperatures, as depicted in Fig. 1b. Distinct phases are identified from high magnification SEM micrographs in the inset of the respective figures. F Ferrite

Table 3 Microstructural characterization of Gleeble® simulated samples for TSCR schedule as the function of isothermal holding temperature

Heat treatment condition (°C)	Microstructural constituents	Mean ferrite grain size (μm)
900–650	Ferrite	3.50 ± 2.21
950–650	Ferrite	3.91 ± 3.08
1000–650	Ferrite	4.23 ± 3.54

The optical micrographs (with SEM insets) of the simulated samples for TSCR-type of processing, for different T_{eet} are displayed in Fig. 5. The corresponding microstructures and the average ferrite grain sizes are listed in Table 3.

The selection of isothermal holding temperature (650 °C) was appropriate as all the TSCR-type processed samples had completely ferrite microstructure with average ferrite grain sizes similar to that of the conventionally processed samples. The ferrite grains were quasi-polygonal in shape and the average grain size increased with the increase in T_{eet} Fig. 5.

3.2 Hardness of thermomechanically simulated samples

The variation in Vickers macrohardness as the function of T_{hold} in conventional processing schedule and as the function of T_{eet} in TSCR-type schedule in Fig. 6a, b shows interesting trend.

In conventional processing, hardness increased initially with the decrease in T_{hold} from 700 °C (205 HVN), reached a peak at 600 °C (270 HVN) and subsequently dropped at 550 °C (230 HVN), Fig. 6a. In TSCR-type processing, hardness increased continuously with the decrease in T_{eet} from 1000 °C (235 HVN) to 900 °C (265 HVN). Such comparison of hardness between conventional- and TSCR-type

processing schedules along with the hardness variation in those schedules depending on the processing parameters have not been reported earlier.

Considering the effects of ferrite grain size and microstructural constituents on hardness, the hardness is expected to increase continuously with the decrease in T_{hold} in conventional processing and with the decrease in T_{eet} in TSCR-type processing. However, in conventional processing, hardness reached at peak at a T_{hold} of 600 °C. In TSCR-type processing, the increase in hardness was more severe at lower T_{eet} range (i.e. from 950 to 900 °C) as compared to higher T_{eet} range (i.e. from 1000 to 950 °C). Analysis of the microalloy precipitates is necessary to address these findings.

3.3 Precipitation in thermomechanically simulated samples

3.3.1 Thermodynamic prediction of precipitation

Thermo-Calc® prediction of the nature and stability of the microalloyed precipitates in the investigated steel is given in Fig. 7. Figure 7a shows the precipitation temperature and the increase in precipitate mole fraction with the decrease in temperature for different precipitates. In spite of the low Ti content of the steel, due to the strong affinity of Ti for N, TiN is predicted to form at ~ 1250 °C. V-rich complex precipitates are expected to appear below 1100 °C and reach a high fraction upon cooling, whilst Nb-rich precipitates form below 900 °C and reach a low fraction, Fig. 7a. The internal compositions of V- and Nb-rich precipitates, as predicted by Thermo-Calc®, are shown in Fig. 7b, c, respectively.

At higher temperatures (> 900 °C) V-rich precipitates are of (V, Nb)CN type, containing significant amount of Nb and N. As temperature drops below A_{e3} (indicated by red dotted line in Fig. 7a), the precipitate become (V, Mo)C type,

Fig. 6 Variation in Vickers microhardness with **a** isothermal holding and **b** equalization entry temperatures after following **a** HSM and **b** TSCR route in Gleeble simulator

Fig. 7 **a** Thermo-Calc® software prediction of equilibrium precipitate stability in the investigated steel. Variation in the internal composition of **b** V-rich and **c** Nb-rich precipitates with temperature in the sample as predicted from Thermo-Calc® software

by the enrichment of Mo and C at the expense of Nb and N. As these precipitates form at high fraction at lower temperatures (during austenite to ferrite transformation), they are expected to contribute high precipitation hardening. Nb-rich precipitates are anticipated to be primarily simple NbC type and their formation below 980 °C is accompanied by a decrease in Nb content of V-rich precipitates.

3.3.2 Observation and characterization of precipitates

The bright field and dark field TEM images with EDS and SAED (selected area electron diffraction) analysis of the thermomechanically simulated samples for conventional processing and TSCR-type processing are given in Figs. 8 and 9, respectively. The TEM images of the conventionally processed and TSCR-type processed samples show the presence of numerous fine and spherical precipitates within the ferrite matrix, Figs. 8 and 9. In case of conventional processing, the precipitates were coarser (average size of 14 ± 1 nm) at higher T_{hold} of 700 °C and finer at T_{hold} of 550–600 °C (5–7 nm). The precipitate density and volume fraction were highest at the T_{hold} of 600 °C as shown in Fig. 8f. The SAED analysis indicated that the precipitates had FCC crystal structure with lattice parameters of 4.10–4.16 Å which cover the lattice parameters of VC and

V(C,N) precipitates [1]. The ferrite matrix also appeared to be more strained at lower T_{hold} of 550–600 °C.

Fine precipitates observed in TSCR-type processing were of the similar nature and size of the precipitates as observed in conventional processing, Fig. 9. The decrease in T_{eff} from 1000 to 900 °C (for a constant T_{hold} of 650 °C) refined the average precipitate size from 9 to 5 nm and increased the precipitate fraction from ~0.004 to ~0.006. Besides fine, spherical precipitates, coarser (up to 100 nm) and cuboidal TiN precipitates were occasionally found in both conventional and TSCR-type processing simulated samples as predicted from Thermo-Calc®.

3.4 Discussion on the effect of grain size and precipitation on hardness

The presence of different microstructural constituents is already discussed in Sect. 3.1. It is interesting that in terms of final ferrite grain size there was no significant difference between conventional and TSCR-type processing simulations. Basically the same starting material, i.e. homogenized forged block used in both the schedules. The same cooling schedule and T_{hold} (650 °C) were used in both the schedules. Besides that a high strain ($\epsilon = 1.0$) was applied at 870 °C in both the schedules for achieving a significant grain refinement. Such a heavy deformation is preferred in

TSCR to refine the coarse austenite grain size of the as-cast thin-slab. The deformation of austenite at 870 °C generated crystalline defects such as serrated grain boundaries, deformation bands, twins etc. As the deformation temperature was sufficiently low, the deformed austenite could not recrystallize before it transformed into ferrite (as indicated by the low softening fraction in Fig. 1). Presence of the defects provided sufficient nucleation sites for ferrite and refined the ferrite grain size, irrespective of the processing schedule. As austenite to ferrite transformation occurred during isothermal holding, the ferrite grain size in the conventional processing schedule decreased with the decrease in T_{hold} due to the increase in driving force for ferrite nucleation and the decrease in driving force for ferrite grain growth. In TSCR-type schedule, the austenite grain growth resulted in larger grain size at higher T_{eet} (i.e. 1000 °C as compared to 950 or 900 °C) as a result of longer exposure at higher temperature.

The fine, spherical precipitates in ferrite matrix generated {111} ring pattern in SAED, indicating FCC structure of the precipitates. Such crystal structure with lattice parameters of 4.10–4.16 Å suggests that those were V-rich carbides, nitrides or carbo-nitrides [1]. Exact precipitate composition can only be determined by high end spectroscopic analysis in TEM like electron energy loss spectroscopy (EELS). Thermo-Calc® prediction indicates that those precipitates also contained Mo, i.e. (V,Mo)C. A number of earlier studies reported the formation of complex (V,Mo)C with FCC structure in place of separate FCC- V_4C_3 and HCP- Mo_2C precipitates [40–44]. (V,Mo)C has the same space group and lattice parameter as V_4C_3 (4.16 Å). Although Mo atom size is larger than V, the size difference is compensated by a decrease in C under stoichiometry [40–44].

The highest volume fraction and small size of the precipitates obtained at an intermediate T_{hold} of 600 °C in the conventional processing schedule can be explained as follows:

- Higher T_{hold} (700 °C) provided adequate thermal activation for the diffusion of V and Mo, allowing the precipitate growth and coarsening, increasing the precipitate size.
- Lower T_{hold} (550 °C) certainly increased the driving force for precipitate nucleation and refined the precipitate size. However, lack of thermal activation for diffusion restricted the precipitate growth and affected the precipitation kinetics.
- Best combination of precipitate nucleation and growth was obtained at T_{hold} of 600 °C, resulting in a high volume fraction of fine precipitates.

As compared to the conventional processing, the precipitation in TSCR-type of processing schedule, especially

considering the effect of T_{eet} is more complex. In order to understand the effect of T_{eet} on microstructure and precipitation, selected samples were water quenched from the intermediate stage of TSCR-type schedule as shown by the dotted lines in Fig. 2b. Figures 10 and 11 present the TEM images of the precipitates (with SAED and EDS analysis) observed in the samples quenched from 1000 to 900 °C, respectively.

Cuboidal TiN particles up to 100 nm size were observed in both the samples, Fig. 10a, b. Coarse (up to 150 nm) and complex precipitates containing Ti, Nb and V (expected as (Ti,Nb,V)(C,N)) were only found in 1000 °C quenched sample, Fig. 10c. Although not predicted by Thermo-Calc®, formation of such complex precipitate upon long exposure at high temperature has been reported earlier [45, 46]. For T_{eet} of 1000 °C, sufficiently long thermal exposure allowed the formation of complex carbonitrides which could not dissolve during the equalization treatment. Such complex precipitates tied up the substitutional (Nb and V) and interstitial solutes (C and N) and imposed a negative effect on (1) the strain-induced Nb(C,N) precipitation during deformation and (2) the precipitation of V_4C_3 and (V,Mo)C during transformation.

In case of T_{eet} of 900 °C, the precipitates formed during the temperature drop were relatively finer (less than 30 nm), containing high amount of Nb, with or without Ti, Fig. 11a–c. Very fine precipitates of FCC structure having lattice parameter of around 4.45 Å, typical to NbC [1], were also found to have formed on the dislocations, Fig. 11d, e.

The precipitation of NbC below 980 °C is also predicted by Thermo-Calc® for the investigated steel. Thus, instead of coarse and complex precipitates finer and simpler precipitates (TiN, NbC and V-rich precipitates) formed separately as the temperature dropped to lower T_{eet} . Dissolution of NbC and V-rich precipitates during the subsequent equalization treatment was an easier process and therefore, alloying elements like Nb, V, C and N predominantly remained dissolved in austenite. Consequently strain-induced Nb(C,N) precipitation during deformation and the precipitation of V_4C_3 and (V,Mo)C during transformation were encouraged. Availability of more N in solution could also have promoted the formation of V-rich nitrides than carbides during isothermal holding. Better coarsening resistance of the nitrides, compared to the carbides [1, 40] retained the fine precipitate size in the final microstructure for T_{eet} of 900 °C.

In steels microalloyed with Nb, Mo and/or V, one of the important aspects is the formation of acicular ferrite. The influence of Nb content in the steel as well as Nb (C/CN) precipitates on the formation of acicular ferrite in the final microstructure have been depicted by several authors [47–49]. It was observed [47] that sufficient holding time at austenite enhances the formation of the Nb precipitates

Fig. 8 **a–e** Bright field (BF) and dark field (DF) transmission electron micrographs (TEM) and the corresponding SAED pattern analysis of the precipitates in the ferrite matrix of the investigated steel following Conventional HSM route for different isothermal holding temperatures as mentioned on the micrographs. **f** Variation in average precipitate size and volume fraction with isothermal holding temperature. Diffracted spots marked by solid circles in the SADP of **a** and **c** are responsible for generation of the DF images in **b** and **d** respectively

which invariably acts as sites for nucleation of acicular ferrite during cooling. Also, the formation of acicular ferrite depends on processing conditions like reheating temperature, isothermal holding, cooling rate etc. [49]. However, in the present investigation no traces of acicular ferrite were found in the thermo-mechanically simulated samples. In both the processing schedules, the austenite was heavily deformed below non-recrystallized temperature domain (i.e. 870 °C) which led to severe pancaking to the austenite grains along with other crystalline defects. This effectively increased the grain boundary area per unit volume and promoted the nucleation of polygonal ferrites from austenite grain boundaries. Moreover, processing schedule

followed in the present investigation particularly deformation schedule followed by faster cooling to higher isothermal holding temperature is conducive to polygonal ferrite formation as discussed previously [12, 13]. This hardly allows sufficient time to form strain induced precipitates of Nb and/or V. Therefore, the unavailability of sufficient intragranular nucleation sites in austenite (i.e. smaller grain size and strain induced precipitates); higher transformation temperature hindered the formation of acicular ferrite and encouraged the polygonal ferrite formation in the current scenario.

Finer ferrite grain structure provided higher grain boundary strengthening following Hall–Petch relationship [50]. Finer size and higher volume fraction of precipitates significantly contributed to precipitation strengthening following Ashby Orowan equation [51] (for incoherent precipitates) and its modified form for coherent precipitates [52]. As a result, 600 °C T_{hold} sample in conventional processing schedule (where small bainite and pearlite content also contributing to hardness) and 900 °C T_{et} sample in TSCR-type processing schedule achieved high hardness, as compared to the other samples.

Fig. 9 **a–c** Bright field (BF) and dark field (DF) transmission electron micrographs (TEM) and the corresponding SAED pattern analysis of the precipitates in the ferrite matrix of the investigated steel following TSCR route for different equalization entry temperatures as mentioned on the micrographs. Diffracted spot marked by solid circles in the SADP of **a** is responsible for generation of the DF images in **b**

Fig. 10 Bright field TEM micrographs of coarse Ti-rich precipitates after water quenched from the intermediate stage ($T_{\text{eet}}=1000\text{ }^{\circ}\text{C}$) of TSCR schedule. The chemical composition and crystal structure

of the precipitates are confirmed by EDS and SAED analysis in the inset of the figures

4 Conclusions

1. Thermomechanical simulation of the conventional Hot Strip Mill (HSM) processing schedule in Gleeble[®] showed that fully ferritic microstructure can be obtained at and above $650\text{ }^{\circ}\text{C}$ coiling temperature, simulated as the isothermal holding temperature (T_{hold}). Some amount of pearlite and bainite can also form at lower coiling temperatures ($\leq 600\text{ }^{\circ}\text{C}$).
2. In conventional processing simulation, peak hardness was achieved at the isothermal holding temperature of $600\text{ }^{\circ}\text{C}$. However, considering the requirement of high strength along with fully ferritic microstructure, use of $650\text{ }^{\circ}\text{C}$ coiling temperature can be recommended.
3. The Gleeble[®] simulation of thin-slab casting direct rolling (TSCR) type processing using a T_{hold} of $650\text{ }^{\circ}\text{C}$, developed fully ferrite microstructure with ferrite grain size in the range of $3.5\text{--}4.2\text{ }\mu\text{m}$. Heavy deformation in the range of $850\text{--}900\text{ }^{\circ}\text{C}$ can significantly refine the ferrite grain size of TSCR-type schedule as indicated by the double-hit compression test.
4. The maximum hardness of simulated sample processed following the TSCR-type processing route (265 HVN) having completely ferrite microstructure was very close to that obtained from the conventional processing route (270 HVN), where small amount of pearlite and bainite was present, apart from ferrite. Hence TSCR-type of processing can also be very effective in developing nano-precipitation strengthened ferritic steels.
5. Low equalization entry temperature of $900\text{ }^{\circ}\text{C}$ in TSCR-type processing develops fine precipitates which dissolve completely during the equalization treatment and fresh precipitation occurs during deformation and transformation.
6. In case of higher equalization entry temperature of $1000\text{ }^{\circ}\text{C}$, long exposure at high temperature forms coarse and complex $(\text{Ti,Nb,V})(\text{C,N})$ precipitates which partially dissolve during equalization treatment and affect the precipitation during deformation and ferritic transformation.

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