Effect of Microstructure on Strain-hardening Behaviour of a Ti-IF Steel Grade

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Thermomechanical processing parameters were adjusted during the processing of Ti-IF steel sheet to obtain microstructures with different grain sizes and precipitation states. The grain size and precipitation state were fully characterized for each specimen in order to investigate the effect of each on mechanical properties. Uniaxial tensile tests were performed at a strain rate of $2 \cdot 10^{-3} \text{ s}^{-1}$ at room temperature. Relationships between strain-hardening coefficient, n , and mechanical properties were analysed. Differences in measured n -values between the different specimens are associated to a change in yield strength resulting from hardening effects of precipitates and grain size at the beginning of plastic deformation. The role of grain size and precipitation state on strain-hardening behaviour is discussed in terms of their effect on dislocation structure evolution. A strain transition exists where dislocation tangles evolve towards well-defined dislocation cells. It is shown in the present study that the entangled dislocation density is very sensitive to the microstructure for the Ti-IF steel studied while the dislocation cell size appears to be insensitive to the microstructure.

KEY WORDS: strain-hardening coefficient; grain size; precipitates; dislocation structures.

1. Introduction

One requirement of the automotive industry is to deliver steel sheets with good stretchability. Ti-IF steels are ultra low carbon (ULC) steels with interstitial carbon and nitrogen completely trapped in Ti-containing precipitates. They are good candidates for applications requiring good formability because of their low yield strength and adequate non-ageing properties. Superior formability requires high values of strain hardening coefficient, n , and anisotropy coefficient, r . Appreciation of stretchability from the strain-hardening coefficient (n -value) is possible from stress-strain behaviour acquired during a simple tensile test. The deformation behaviour of ULC Ti-IF steel is commonly analysed with the Hollomon equation:

$$\sigma = k\varepsilon^n \quad \dots\dots\dots(1)$$

Where σ is the true stress, ε is the true strain, k is a strengthening factor and n is the strain-hardening coefficient. For materials which obey Eq. (1), n is equal to the homogeneous elongation, A_u , according to the criterion for instability:

$$\frac{d\sigma}{d\varepsilon} = \sigma \quad \text{where } \varepsilon = A_u \quad \dots\dots\dots(2)$$

where $d\sigma/d\varepsilon$ is the strain-dependent true strain hardening

rate. This condition is graphically illustrated in Fig. 1. The strain at instability, which is equal to the n -value, can be viewed as the sum of two terms according to D. K. Matlock¹⁾: the yield strength YS and the stress increment due to strain-hardening $\Delta\sigma_{\text{UTS-YS}}$:

$$\frac{d\sigma}{d\varepsilon}(\varepsilon = A_u) = \sigma_{\text{YS}} + \Delta\sigma_{\text{UTS-YS}} \quad \dots\dots\dots(3)$$

Fig. 1. Schematic diagram showing the instability condition, $\sigma = d\sigma/d\varepsilon$.

Table 1. Chemical composition in mass ppm of the Ti-IF steel.

C	N	S	Ti	Mn	Si	P	Al	Fe
22	25	93	700	1560	36	76	360	balance

In most cases, each term in Eq. (3) will vary with microstructure. In the case of the present study, the strain dependent strain-hardening behaviour of the Ti-IF steel grade, represented by $d\sigma/d\varepsilon(\varepsilon)$, is the same after a few percent deformation, typically 5 %, for several thermomechanical processes.²⁾ Therefore, increasing the n -value requires an understanding of how the microstructure influences each term in the right side of Eq. (3). The aim of this paper is an in depth analysis of a Ti-IF steel grade, including the effect of microstructure on each term in Eq. (3). This microstructural analysis is based on a thorough study of grain size measurement, precipitation state characterization, dislocation density assessment and dislocation arrangement study.

The originality of this paper appears in the fact that the studied Ti-IF steel grade was subjected to changes in thermomechanical processing parameters (slab reheating temperature (SRT), coiling temperature (CT), annealing treatment and cold reduction rate) in order to produce samples with different precipitation states and constant grain size, samples with different grain sizes and constant precipitation states and finally to distinguish each effect on n -value. Though many attempts were made to correlate the variation of n -value with microstructural parameters, such as the grain size,^{3,4)} so far a relevant analysis has not been made due to a lack of separation between the effect of each microstructural parameter.

2. Experimental Procedure

2.1. Material and Thermo Mechanical Treatments

Steel, with the chemical composition listed in **Table 1**, was vacuum melted and cold rolled to achieve a final thickness of a 0.75 mm. Several thermomechanical processing routes were used to change precipitation state including controlling Slab Reheating Temperature (1050°C and 1250°C), Coiling Temperature (570–600°C and 710–740°C) and the type of annealing (Continuous Annealing Line and Batch Annealing Furnace). Variation in grain size was obtained by controlling cold reduction rate (25 to 90 %) and CAL temperature (from 720 to 860°C).

2.2. Microstructural Investigations

2.2.1. Grain Size Measurement

Optical microscopy samples were prepared using standard metallographic procedures. Grain size was determined using the Linear Intercept Method specified by ASTM standard E112. Mean linear intercept grain size (m.l.i.) was determined and spherical grain morphology was assumed. The three dimensional grain diameter d is given by

$$d=3(\text{m.l.i.})/2 \dots\dots\dots(4)$$

2.2.2. Precipitate Analysis

The precipitation state was characterized by (scanning) transmission electron microscopy ((S)TEM) using carbon extraction replicas. A Jeol 2010 TEM operating at 200 kV

equipped with an EDX spectrometer coupled to an image analysis system quantitatively enabled chemical composition measurements of a large number of particles (typically around 160) per sample as well as particle size assessment.

2.2.3. Analysis of Dislocations

Thin foils were prepared and examined using a Philips CM30 TEM operating at 300 kV. The quantitative assessment of the dislocation density was done using the Ham's intercept method⁵⁾ from TEM micrographs. The dislocation density ρ is given by:

$$\rho = \frac{2 \cdot N}{L \cdot t} \dots\dots\dots(5)$$

where N is the number of intersections that dislocations make with a set of random lines with a total length L and t is the thin foil thickness. The LACBED (Large-Angle Convergent-Beam Electron Diffraction) technique was used to accurately determine the foil thickness.⁶⁾

A preliminary experimental survey showed that a total of five grains per specimen and four micrographs per grain were statistically needed to determine dislocation density with accuracy.

2.3. Tensile Testing

Tensile specimens with a gage length of 80 mm, width of 20 mm, and thickness of 0.75 mm were prepared by electro-sparking and then mechanically tested at room temperature. A Schenk UTS 20 kN electrical machine was used under control of the crosshead in order to obtain an average strain rate of $2 \cdot 10^{-3} \text{ s}^{-1}$ with the load axis perpendicular to the rolling direction of the sheet. Moreover, interrupted tensile tests were carried out at true strains of 0.02, 0.05, 0.10, 0.15 and 0.20, and thin foils were prepared for Transmission Electron Microscopy investigation.

Standard tensile properties, namely 0.2 % yield strength (YS), ultimate tensile strength (UTS) and uniform elongation (A_u), were determined from the load-elongation data obtained from these tests. Three samples were tested and average values are reported to account for scatter.

In this study, the strain-hardening coefficient (labelled n) was calculated between the true strains $\varepsilon=0.10$ and $\varepsilon=0.20$. Strain-hardening coefficient n is calculated using ASTM standard E646 for determination of tensile strain-hardening exponents of metallic sheet materials. The equation for the calculation is a linear regression using five points taken in geometrical progression between $\varepsilon=0.10$ and $\varepsilon=0.20$ and is:

$$n = \frac{5 \sum_{i=1}^5 (\log \varepsilon_i \log \sigma_i) - \left(\sum_{i=1}^5 \log \varepsilon_i \sum_{i=1}^5 \log \sigma_i \right)}{5 \sum_{i=1}^5 (\log \varepsilon_i)^2 - \left(\sum_{i=1}^5 \log \varepsilon_i \right)^2} \dots\dots\dots(6)$$

where, σ_i and ε_i are respectively the true stress and true strain at a level of plastic deformation of i .

Table 2. Evolution of precipitation state with the slab reheating (SRT), coiling temperature (CT) and annealing treatment of the Ti-IF steel.

Specimen	SRT in °C	CT in °C	Annealing treatment	Precipitation state (population in %, average radius in nm)		
				Ti ₄ C ₂ S ₂	TiFeP	TiC
A	1050	710-740	CAL 750	(64; 79)	(16; 60)	/
B	1050	710-740	BAF high T	(26; 74)	(70; 37)	/
C	1050	710-740	BAF low T	(24; 89)	(66; 31)	/
D	1050	570-600	BAF low T	(13; 99)	(58; 33)	(28; 14)
E	1250	710-740	CAL 850	(51; 44)	(19; 33)	(24; 11)
F	1250	710-740	BAF high T	(4; 32)	(54; 40)	(39; 18)
G	1250	710-740	BAF low T	(14; 24)	(49; 37)	(35; 12)

3. Results and Discussion

3.1. Evolution of the Microstructure with the Thermo Mechanical Process

The first set of specimens (see **Table 2**) obtained by modifying thermomechanical processing parameters (SRT, CT, CAL and BAF) present different distribution and average size of precipitates but the grain size remained in the range of 22 to 28 μm .

Five primary types of Ti-containing precipitates are commonly observed in Ti-IF steels, as reported by several authors^{7,8)} including: Ti₄C₂S₂ titanium carbo-sulfides, TiFeP iron phosphides, TiC titanium carbides, some TiS carbo-sulfides and TiN titanium nitrides. The proportion and mean radius of each type of precipitate for all the specimens are reported in Table 2. From EDS-coupled TEM analysis, a population of one hundred and sixty precipitates was examined per specimen. It was observed that TiS and TiN represented less than five per cent of the precipitates analyzed; their sizes were much larger than the other types. Therefore, the effect on mechanical properties is expected to be negligible compared to smaller, more numerous types of Ti-containing precipitates. Thus, only Ti₄C₂S₂, TiFeP and TiC precipitates are assumed to have the largest effect on mechanical properties and strain-hardening behaviour.

The main effects of thermomechanical treatment on precipitation state change are reflected in the TiC carbides and Ti₄C₂S₂ carbo-sulfides due to dissolution of Ti₄C₂S₂ precipitates during reheating above 1 200°C.⁷⁾ SRT essentially acts on the size and composition of Ti-containing precipitates. Higher SRT resulted in reduced size of titanium carbo-sulfides. The average size of titanium carbo-sulfides is reduced by approximately two by increasing SRT as shown in Table 2. No relevant differences in precipitation state were observed in the specimens for both low and high batch annealing temperatures. Fine TiC particles were observed in specimens prepared with a SRT of 1 250°C, and in specimen D prepared with a different coiling temperature (570–600°C) and slab reheating temperature of 1 050°C as shown in Table 2. Precipitation of TiC occurs during both coiling and annealing operations. The proportion of fine TiC carbides ranged from 24 to 39 % in specimens prepared with a SRT of 1 250°C. Effects on mechanical properties were anticipated due to the presence of fine titanium carbides in some specimens. Indeed, the lower the precipitate size is, the higher the hardening effect is expected. The analysis of precipitation state showed that the average size

Table 3. Evolution of the grain size with the cold reduction and annealing treatment of the Ti-IF steel.

Specimen	Cold reduction rate in %	Annealing treatment (°C)	Grain size in μm
H	25	CAL 850	75.0
I	35	CAL 850-860	56.4
J	90	CAL 850	45.0
K	76	CAL 850	38.1
L	76	CAL 740	30.6
M	80	CAL 740	24.5
N	90	CAL 720	18.0

Table 4. Mechanical properties and n values of the Ti-IF steel with different precipitation states and different grain sizes.

specimen	YS MPa	UTS MPa	$\Delta\sigma_{\text{UTS-YS}}$ MPa	A_n %	d in μm	r	n
A	102	289	187	33	21.7	2.17	0.350
B	100	278	178	31	26.9	2.53	0.345
C	115	276	161	31	28.5	2.00	0.325
D	133	308	175	29	26.7	1.72	0.290
E	89	276	187	32	28.0	2.76	0.355
F	115	303	188	33	28.0	2.50	0.340
G	132	312	180	30	26.4	2.74	0.310
H	78	263	185	33	75.0	1.24	0.385
I	80	258	178	34	56.4	1.55	0.390
J	84	265	181	31	45.0	3.12	0.360
K	77	261	184	33	38.1	2.52	0.375
L	82	264	182	29	30.6	2.96	0.340
M	98	275	177	35	24.5	2.65	0.370
N	105	285	180	32	18.0	2.47	0.345

of TiFeP precipitates remained constant for all the specimens, regardless of slab reheating temperature and annealing treatment. Except for specimens A and E, the proportion of TiFeP ranged 50–70 % of total precipitate population in the specimens.

The second set of specimens (see **Table 3**) obtained by modifying thermomechanical processing parameters present different grain sizes from 18.0 to 75.0 μm ; precipitation state remained constant. Specimens contained essentially Ti₄C₂S₂ and TiFeP precipitates, as observed in specimen C (see Table 2).

3.2. Mechanical Properties

3.2.1. Tensile Testing Results

Uniaxial tensile test results are presented in **Table 4**.

Fig. 2. Evolution of the strain hardening coefficient n versus uniform elongation of a Ti-IF steel after thermomechanical process.

Fig. 3. Evolution of the strain hardening coefficient n versus yield strength of a Ti-IF steel after thermomechanical process.

Variations in mechanical properties as well as n -values are observed which correlate to microstructure of the Ti-IF steel grade with a constant chemical composition. The strain-hardening coefficient was ranging from 0.290 to 0.390 and the yield strength varied from 77 to 133 MPa. The higher the measured n -value was, the higher the uniform elongation (A_u) was (Fig. 2). Therefore, the ability of a steel to deform by stretching, which the strain-hardening coefficient is an assessment,^{9,10)} can be improved simply by modifying the microstructure. Low and high n -values were obtained with r -value above 2.00 (except for specimens D, H and I). It must be noted, however, that for materials presenting double- n behaviour, which is the case with Ti-IF steels,^{11,12)} n is not numerically equal to A_u but the relationship is maintained.

The stress increment $\Delta\sigma_{\text{UTS-YS}}$, expressed as the difference between the value of the ultimate tensile strength and the yield strength, does not significantly vary from one specimen to another one (except for specimen C). The average value of the stress increment $\Delta\sigma_{\text{UTS-YS}}$ for all the studied specimens is $180 (\pm 7)$ MPa. Therefore, microstructural changes obtained by thermomechanical processing do not seem to affect plastic behaviour during tensile deformation and therefore should affect essentially the yield strength, which influences n -value according to Eq. (3).

3.2.2. Relationships between Strain-hardening Coefficient and Yield Strength

Correlation of the strain-hardening coefficient n with mechanical properties is presented in Fig. 3, where the measured n values of the studied Ti-IF steel grade are plotted against the yield strength values. Higher yield strength results in lower strain-hardening coefficient for specimens that exhibit the same chemical composition (Table 1) and a similar stress increment $\Delta\sigma_{\text{UTS-YS}}$ (Fig. 4). The difference is only in precipitation state and grain size (see Tables 2 and 3). Therefore, the measured value of strain-hardening coefficient strongly depends on hardening effects from both grain size and precipitates acting at the beginning of plastic deformation. The yield strength of ULC Ti-IF steels is highly dependent on microstructure, in this case altered by variation in the thermomechanical parameters during specimen preparation.

Fig. 4. Evolution of $\Delta\sigma_{\text{UTS-YS}}$ (difference between the ultimate tensile strength UTS and the yield strength YS) versus yield strength of a Ti-IF steel after thermomechanical process.

3.2.3. Hall-Petch Parameters in the Studied ULC Ti-IF Steel Grade

The effects of grain size on mechanical properties in iron and low carbon steels have been extensively studied^{13,14)} over the last decades. However, this work originates by the fact that the effects of grain size on yield strength were analyzed while an attempt was made to control all other variables during thermomechanical processing, *i.e.* mainly precipitation state and texture. Indeed, the analysis of the precipitation state of some specimens presenting different grain sizes has shown similar precipitation states in the specimens *i.e.* mainly composed by titanium carbosulfides $\text{Ti}_4\text{C}_2\text{S}_2$ and iron phosphides TiFeP as in specimens B and C in Table 2. The influence of grain size on yield strength is commonly described by the Hall-Petch relationship given as follows:

$$\sigma_y = \sigma_0 + k_y d^{-1/2} \dots \dots \dots (7)$$

where σ_y is the yield strength, σ_0 is the “friction stress” and k_y the strengthening coefficient. The values of yield strength for grain diameters d that vary from 18.0 to $75.0 \mu\text{m}$ are plotted as a function of $d^{-1/2}$ in Fig. 5. The k_y value is $7.3 \text{ MPa} \cdot \text{mm}^{-1/2}$ and the σ_0 value is 48 MPa. These values are in good agreement with experimental values reported in literature for ULC IF-type steels.^{14,15)} The weak

value of k_y observed in the present study can be attributed to the weak amount of the dissolved C and N atoms, since C and N are trapped in precipitates in order to avoid segregation in the grain boundaries, and to the fact that the material presents preferred crystallographic orientations as showed by the OIM analysis²⁾ and supported by the measured high r -value. Therefore, the efficiency of the grain boundaries to block dislocations is reduced.

3.3. Analysis of the Role of Microstructure on Dislocation Structures

The evolution of dislocation structure with deformation

Fig. 5. Evolution of the yield strength *versus* grain diameter of a Ti-IF steel after thermomechanical process.

has been studied using TEM after interrupted tensile testing in order to understand the role of the Ti-IF microstructure. All the specimens show the same evolution of dislocation structures as shown in **Fig. 6**, where an example of the evolution of dislocation structures in specimen A is presented. This N specimen exhibits an intermediate value of yield strength and n -value, 115 MPa and 0.340 respectively. Tangling characterizes the dislocation structure up to a true strain of 0.05 whereas well-defined dislocation cells are observed above this level of deformation. The change of dislocation structure occurring around $\epsilon=0.05$ is typical of steels that exhibit a double n behaviour.^{11,12,16)} Indeed, for many steels, a single n value cannot represent an entire $\ln \sigma$ vs. $\ln \epsilon$ curve, regardless the microstructure.

It is now interesting to analyse the effects of precipitates and grain size on dislocation arrangements and dislocation structure evolution during tensile deformation to understand the role of microstructure on the strain-hardening coefficient. Therefore, dislocation density and dislocation cell size evolution with deformation were followed in different specimens presenting microstructural changes. Two sets of specimens presenting a high and a low n -value were selected to represent the range of the effects resulting from different precipitation state (specimens B and G) and different grain size (specimens I and N).

The results of dislocation density and dislocation cell size measurements are summarized in **Table 5**. Before testing, at $\epsilon=0$, the dislocation density is quite similar for all

Fig. 6. TEM micrographs of dislocation structures in the specimen A strained at a deformation of: $\alpha=0$, $\beta=0.02$, $\chi=0.05$, $\delta=0.10$, $\phi=0.15$ and $\gamma=0.20$. All micrographs are taken with the same vector of diffraction $[1\ 0\ -1]$.

Table 5. Measured dislocation density and cell size according to the strain.

specimen	<i>n</i> -value	Average dislocation density (Standard Deviation) 10 ⁸ cm ⁻²			Average cell size (Standard Deviation) μm		
		$\varepsilon = 0$	$\varepsilon = 0.02$	$\varepsilon = 0.05$	$\varepsilon = 0.10$	$\varepsilon = 0.15$	$\varepsilon = 0.20$
B	0.345	5.3 (2.7)	16.8 (3.8)	36.8 (9.9)	1.2 (0.3)	1.1 (0.2)	1.4 (0.4)
G	0.310	3.1 (0.8)	24.0 (7.7)	65.1 (4.5)	1.6 (0.4)	1.3 (0.3)	1.4 (0.3)
I	0.390	7.4 (2.0)	27.6 (9.4)	55.0 (16)	1.1 (0.2)	0.9 (0.3)	1.0 (0.3)
N	0.345	5.3 (1.0)	36.9 (5.9)	62.1 (15)	1.0 (0.3)	1.2 (0.2)	1.4 (0.5)

the specimens taking into account the accuracy of measurement.⁵⁾

When the tensile deformation starts and up to $\varepsilon=0.05$, the dislocation density depends on both precipitation state and grain size. Dislocation density is higher for specimen G than specimen B because of the presence of fine titanium carbides TiC (see Table 2). This difference in dislocation density is a result of different precipitation states achieved by changing thermomechanical processing parameters. Fine TiC particles increased the yield strength value (see Table 4) by the way of Ashby–Orowan type dislocation-precipitate interactions.¹⁷⁾ A similar effect on dislocation density due to grain boundary strengthening is observed in specimens I and N. As shown in Table 5, decreasing the grain sizes results in higher dislocation densities.

The experimental measurements of dislocation cell sizes presented in Table 5 show that the average dislocation cell size does not significantly differ between all the specimens, regardless of microstructure. Indeed, the dislocation cell size remains quite constant, around 1.2 μm. This result is in good agreement with the D. Kuhlmann-Wilsdorf's theory of Low Energy Dislocation Structures¹⁸⁾ (LEDSS) which considers the cellular structure as the most stable one. Indeed, the Orowan stress in precipitation-hardened materials does not increase with strain as long as the particle spacing $\bar{L}$ is large compared to the average dislocation spacing $\bar{l}$ (of the order of $\bar{l} \approx 1/\sqrt{\rho}$), which is the case after a few percent of deformation in Ti-IF steel grade. Therefore, the stress increment $\Delta\sigma_{\text{UTS-YS}}$ due mainly to dislocation work hardening remains constant in all the specimens (except in specimen C).

4. Conclusions

A Ti-IF steel has been subjected to several thermomechanical processing routes in order to produce controlled changes in the microstructure. The precipitation state and the grain size, which affect the strain hardening coefficient *n*, have been investigated.

From this work, the following conclusions can be made:

- (1) The yield strength directly affects the strain-hardening coefficient, *i.e.* *n* increases when Y.S decreases
- (2) A strain transition ($\varepsilon=0.05$) exists below which dislocations are entangled and above which a cellular structure forms. The dislocation structures below $\varepsilon=0.05$, are strongly dependent on the microstructure (precipitation and grain boundaries), but not the dislocation structures above $\varepsilon=0.05$. This thorough experimental survey showed that:
  - The stress increment $\Delta\sigma_{\text{UTS-YS}}$ is independent of microstructure for the Ti-IF steel grade studied.
  - The *n*-value is controlled by dislocation-precipitate and dislocation-grain boundary interactions occurring at the first percent of deformation.

- The *n*-value is controlled by dislocation-precipitate and dislocation-grain boundary interactions occurring at the first percent of deformation.

Therefore, this study showed that for a given chemical composition of a Ti-IF steel, the highest *n*-value is obtained with a slab reheating temperature of 1 050°C, a coiling temperature within 710–740°C.

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Nomenclature

ULC :	Ultra low carbon
SRT :	Slab reheating temperature (°C)
CT :	Coiling temperature (°C)
CAL :	Continuous annealing line (°C)
BAF :	Batch annealing furnace (°C)
<i>d</i> :	Grain size (μm)
ρ :	Dislocation density (cm ⁻²)
<i>r</i> :	Anisotropy coefficient
ε :	True strain
σ :	True stress (MPa)
<i>n</i> :	Strain-hardening coefficient calculated between $\varepsilon=0.10$ and $\varepsilon=0.20$
YS :	Tensile strength (MPa)
UTS :	Ultimate tensile strength (MPa)
$\Delta\sigma_{\text{UTS-YS}}$:	Difference between UTS and YS (MPa)
<i>A_u</i> :	Uniform elongation (%)

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