# MICROSTRUCTURAL EVOLUTION MODEL FOR HOT STRIP ROLLING OF A Nb-Mo COMPLEX-PHASE STEEL

by

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#### ABSTRACT

A comprehensive study on the microstructural evolution of a new generation Nb-Mo microalloyed model complex-phase (CP) steel under hot strip rolling conditions has been conducted. The experimental investigation includes the austenite conditioning during reheating, work hardening and static softening of austenite during hot deformation, austenite decomposition to multiphase structure during run out table cooling operation and finally precipitation strengthening during coiling at downcoiler.

The flow stress and static softening behaviour of austenite is modeled by the physically based approaches of Kocks-Mecking and Zurob et al., respectively, whereas empirical approaches are employed to model recrystallized austenite grain size and grain growth after recrystallization. The start of ferrite formation is described by the early growth of corner nucleated ferrite. A limiting carbon concentration concept is postulated above which ferrite formation ceases. A semi-empirical approach based on the Johnson-Mehl-Avrami-Kolmogorov (JMAK) theory adopting additivity is employed to describe ferrite as well as bainite growth with individual parameters for each reaction. The present ferrite model includes the formation of the transformation stasis regime, where a critical driving pressure approach is adopted to describe the stasis initiation. Present research concludes that the same driving pressure approach is applicable to describe bainite start and the transition from stasis to bainite start occurs at 620°C. The effect of carbon enrichment in the remaining austenite after ferrite formation is included to describe bainite growth. Martensite + retained austenite volume fraction is calculated empirically as a function of carbon enrichment resulting from the ferrite formation. The isothermal aging kinetics is modeled by a modified Shercliff-Ashby approach, which is then extended for coil cooling path to predict the optimum coiling temperature range (580-610°C) to maximize the precipitation strengthening of microalloying elements. Finally the hardness of the material is expressed as a function of the volume fractions of various transformation products and the precipitation strength contribution. The overall model prediction is validated successfully by torsion simulation of the entire hot rolling and controlled cooling schedule. Current research suggests that fine multiphase structure is possible to achieve in the present steel through proper austenite conditioning and adopting complex cooling strategies.

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In Chapter 2 and 4, the complete experimental and modelling work is performed by me, whereas significant input has been provided during the laser-ultrasonic set-up and the experimental data analysis by Dr. Andre Moreau at the Industrial Materials Institute, Boucherville, Canada. Dr. Andre Moreau also provided valuable suggestions in preparing the manuscripts for Chapter 2 and 4.

In Chapter 3, 5, 6, I am the primary author of the text and the figures. However, I incorporated the suggestions made by my supervisors to give them the final shape for submission. With the exception of Chapter 1, all chapters have been published or accepted in journals and conferences for publication.

#### **CHAPTER 1: INTRODUCTION**

#### 1.1 Complex-phase steel

Steel continues to be an important engineering material for automotive and construction applications because of its attractive mechanical properties (a combination of formability and high strength) at comparatively low cost [1-2]. From an industrial point of view, automotive steels can be classified into two main categories; (a) low strength steels (carbon-manganese and microalloyed interstitial free steels) and (b) high strength steels (high strength low alloy steel, HSLA). Conventional high strength steels have found use in the automotive sector for many years, particularly for the production of automotive body parts. In addition to common alloying elements, such as carbon (C) and manganese (Mn), the composition of these steels are tailor-made by the controlled addition of various microalloying elements, such as niobium (Nb), titanium (Ti), vanadium (V), resulting in an improved steel mechanical properties [3-4]. The typical manufacturing process for these steels consists of controlled hot rolling, which may or may not be followed by cold rolling and coating to produce final product.

However, the increasing demand for improved fuel efficiency to address environmental concerns has forced the automotive sector to develop advanced high strength steels with improved properties and/or light metals such as aluminum alloys for use in automotive body parts [1-2, 5-6]. This trend in material has created a highly competitive automobile market which demands higher quality material at lower cost. To address this complex global issue, leading car manufacturers and steel companies from all over the globe (35 major steel producers from 18 countries) announced a novel concept, i.e. ULSAB (Ultra Light Steel Auto Body), in 1994-95. The main aim of the ULSAB project was to design a lighter automotive structure using commercially available highstrength steels to decrease the vehicle weight resulting in lower emissions at a competitive cost without compromising safety. Following this, a second ULSAB project proposed the development of a new generation of advanced high-strength steels (AHSS) to replace the existing conventional C-Mn and HSLA (high-strength low alloy) steels. These new steel grades include DP (dual phase), TRIP (transformation induced plasticity) and CP (complex phase) steels [3-4, 7-8], which combine high strength with adequate elongation and are associated with complex multiphase microstructures, i.e. an optimum combination of various phases, such as ferrite, bainite, martensite and retained austenite.

The strength of steel is an important criterion from an automotive designer's point of view, and currently available high strength steels are generally divided into two classes; (a) conventional high strength steel (HSS) and (b) Ultra high strength steels [3-4, 9]. Conventional high strength steels generally exhibit yield strengths ranging from 210-550MPa and subsequent tensile strengths between 270-700MPa. In contrast, ultra high strength steels (UHSS) have yield strengths greater than 550MPa and tensile strengths higher than 700MPa. Newly developed AHSS overlap the range of strengths between HSS and UHSS. Figure 1.1 shows conventional HSS (an example is HSLA steels) steels and new generation AHSS steels (such as DP, TRIP and CP). The main difference between conventional HSS and modern AHSS is that the latter show higher elongation values and better crashworthiness for a given tensile strength. This is achieved by a different final microstructure as compared to HSLA steels. Conventional HSLA steels generally have a ferrite or ferrite-pearlite microstructure, whereas AHSS have a multiphase microstructure, which is a mixture of two or more phases (ferrite, bainite, martensite and retained austenite). AHSS microstructures that combine ferrite, bainite and martensite in appropriate quantities exhibit improved properties in terms of strength and elongation [8, 10].

Complex-phase steels (CP) are a unique type of low carbon bainitic steels, often characterized by a multiphase microstructure consisting of a fine ferrite matrix and a significant volume fraction of hard constituents, such as bainite, martensite and small islands of retained austenite, which is further strengthened by fine precipitates [4, 8-11]. In addition to common alloying elements, such as manganese (Mn) and silicon (Si), CP steels have small quantities of niobium (Nb), titanium (Ti) or vanadium (V) to form fine strengthening precipitates. These microalloying elements exert a strong retarding effect on the austenite to ferrite transformation leading to the formation of bainite and martensite. The presence of a significant volume fraction of bainite in CP steels provides high strength levels (tensile strengths approximately 800MPa and above) with adequate elongation (approximately 10-15% total elongation) [12-13]. As compared to DP steels, the CP steels exhibit a higher yield strength at equal tensile strength of 800MPa or higher [4]. Typical usage of CP steels is in the high-energy absorption area of a car body such as bumpers and B-pillar reinforcements [4, 14]. In general, CP steels are produced via hot rolling and controlled cooling. The final hot rolled microstructure is produced on the runout table and a suitable coiling temperature must be chosen to achieve the desired microstructures associated with the target mechanical properties. However, cold rolled CP steels have recently been proposed, where the cold rolled steel is intercritically annealed followed by isothermal holding at a temperature higher than the martensite start temperature to control the bainite reaction [10, 12].



**Figure 1.1** – Schematics of AHSS (CP, DP and TRIP) steels as compared to low strength steels (IF) and conventional HSS (HSLA) [after ref. 4].

As mentioned above, the microstructure of the CP steels is complex in nature and their development is very sensitive to the processing conditions. Therefore, improved control of the process parameters is required to optimize the structure-property relationship. To achieve this, it is useful to develop microstructure models that can capture and predict the underlying metallurgical development of the CP steels, such as their hot deformation, recrystallization, and austenite decomposition behaviour. For example, the structure-property relationship for conventional HSS involves the proper consideration of ferrite grain size, grain shape and chemical composition of the steel. In contrast, during the structure-property quantification of the new generation complexphase steels, significant attention should be given to the (a) volume fraction of individual phases, (b) ferrite grain size, (c) mechanical stability of metastable phases and (d) local chemical composition [3].

The present work is intended to investigate the microstructural development of a model microalloyed complex-phase steel under hot strip rolling condition. Based on the experimental studies of the microstructural evolution in the selected steel and starting from the established approaches for low carbon steels, a complete microstructure model has been proposed that consist of individual sub-models for constitutive behaviour, static recrystallization and grain growth after recrystallization followed by austenite decomposition, precipitation strengthening and coiling simulation. Finally, the overall process model has been validated with torsion simulations of the entire hot strip rolling process.

#### **1.2** Literature review

#### 1.2.1 Process modelling of hot rolling

A significant amount of research in the past 30-35 years has focused on the study of hot strip mill rolling of various steel grades. From an industrial point of view, importance was given to the description of microstructural evolution during hot rolling. Figure 1.2 shows the expected microstructural evolution during hot rolling of multiphase steel. Several research groups have developed process models with a view to understand, predict and control the microstructure and mechanical properties of conventional hot rolled steel products [15-22]. An ideal hot strip mill process model should make good predictions regarding the effect of process variables on i) the temperature distribution, ii) the microstructural evolution of rolled materials, iii) the mechanical behaviour of steel during rolling and iv) the final product properties. In previously developed models [15-22], the hot rolling process is typically divided into five individual segments according to the unique microstructural development, (a) grain growth of austenite during reheating, (b) work hardening and softening behaviour of austenite during rough and finish mill rolling, (c) grain growth following recrystallization, (d) austenite decomposition during run-out table cooling and coiling and (e) microalloyed precipitation during coiling.



Figure 1.2 – Typical process layout of the metallurgical developments during hot strip rolling of a multiphase steel.

Process models developed during the 80s' and 90s' were typically empirical or semi-empirical in nature. As an example, previous models adopted the semi empirical Johnson-Mehl-Avrami-Kolmogorov (JMAK) approach [23-25] to capture the static recrystallization behaviour of the C-Mn steels during rough and finish rolling. The fraction recrystallized was described in terms of the time for 50% recrystallization ( $t_{0.5}$ ), which is expressed as a function of initial austenite grain size ( $d_0$ ), applied strain ( $\varepsilon$ ), strain rate ( $\dot{\varepsilon}$ ) and the activation energy of recrystallization ( $Q_{Rex}$ ). A summary of various JMAK equation based recrystallization models for C-Mn steels was presented by Sellars [26] (see Table 1.1). In all models, recovery and recrystallization were considered as separate mechanisms operating during the softening of steel. The recovery contribution to the total softening was considered as the initial 15-20% [26].

As shown in Table 1.1, each recrystallization model differs from the others in terms of the value of activation energy for recrystallization, pre-exponent factor and the grain size/applied strain/strain rate exponent. Similar differences in model parameters can be observed in the proposed models to predict the recrystallized grain size ( $d_{rex}$ ). These model predictions are presented in Figure 1.3, where significant differences can be observed in their predictions. In addition to the differences in the model fit parameters, significant difference can be observed between the formulations of the models (Table 1.1) by different groups that lead to the different temperature dependencies in Figure 1.3.

Although, the previously developed process models [15-22] have limitations in their applicability due to their empirical nature, they can still be employed to predict the microstructural evolution of steel under industrial hot rolling conditions. Adopting suitable fit parameters, these models are currently used to predict microstructural development in research and developments trials of C-Mn and HSLA steel. Therefore, in the following section, a brief summary will be presented on the complete process models proposed by various research groups in the last 20-25 years.

steels (after behals [20]).									
<i>R</i> =8.31Jmol <sup>-1</sup> K <sup>-1</sup> , initial grain size ( $d_0$ ) in $\mu$ m, $\varepsilon$ is equivalent strain, $\dot{\varepsilon}$ in s <sup>-1</sup> , T in K.									
Sellars et al. [27]									
$\varepsilon_{P}$ = Peak strain, $\varepsilon^{*}$ = Some critical strain condition to separate nucleation during dynamic									
or static recrystallization, Z= Zener-Holloman parameter									
Recrystallization model	Recrystallized grain size model								
$t_{0.5} = 2.5 \times 10^{-19} d_0^2 \varepsilon^4 \exp\left(\frac{300000}{RT}\right) \text{for} \varepsilon \le 0.8\varepsilon_p$	$d_{rex} = 0.5 d_0^{0.67} \varepsilon^{-1} \text{ for } \varepsilon \le \varepsilon^*$ $d_{rex} = 1.8 \times 10^3 Z^{0.15} \text{ for } \varepsilon \ge \varepsilon^*$								
$t_{0.5} = 1.6 \times 10^{-5} Z^{-0.6} \exp\left(\frac{300000}{RT}\right) for \varepsilon \ge 0.8\varepsilon_P$	$\varepsilon^* = 2.8 \times 10^{-4} d_0^{0.67} Z^{-0.15}$								
$\varepsilon_P = 4.9 * 10^{-4} d_0^{0.5} Z^{0.15} and Z = \dot{\varepsilon} \exp\left(\frac{312000}{RT}\right)$									
Choquet et al. [23	8]								
Recrystallization model	Recrystallized grain size model								
$t_{0.5} = 6.1 \times 10^{-13} d_0^{0.14} \varepsilon^{-4} \dot{\varepsilon}^{-0.28} \exp\left(\frac{270000}{RT}\right) \text{ where }$	$d_{rex} = 45d_0^{0.374} \varepsilon^{-0.6} \dot{\varepsilon}^{-0.1} \exp\left(\frac{-25000}{RT}\right)$								
$A = 0.95d_0^{0.22}$									
Yada et al. [29]									
$S_V = Surface area/vo$	lume								
Recrystallization model	Recrystallized grain size model								
$t_{0.5} = 2.86 \times 10^{-8} d_0^2 S_v^{-0.5} \varepsilon^{-2} \dot{\varepsilon}^{-0.2} \exp\left(\frac{150000}{RT}\right) \text{ where }$	$d_{rex} = 5S_V - 0.6\varepsilon^{-0.6}$								
$S_{\nu} = \left(\frac{24}{\pi d_{0}}\right) (0.491 \exp \varepsilon + 0.155 \exp - \varepsilon + 0.1433 \exp - 3\varepsilon)$									
Roberts et al. [30]									
Recrystallization model	Recrystallized grain size model								
$t_{0.5} = 5.1 \times 10^{-21} d_0^2 \varepsilon^{-4} \exp\left(\frac{330000}{RT}\right)$	$d_{rex} = 6.2 + 55.7 d_0^{0.5} \varepsilon^{-0.65} \exp\left(\frac{-35000}{RT}\right)$								

**Table 1.1**: Various empirical models to evaluate the 50% recrystallization temperature  $(t_{0.5})$  and recrystallized grain size  $(d_{rex})$  proposed by different research groups for C-Mn steels (after Sellars [26]).



**Figure 1.3** – Empirical model predictions (presented in Table 1.1) for  $t_{0.5}$  and  $d_{rex}$  by four different research groups on C-Mn steel as a function of temperature: (a) for  $t_{0.5}$  and (b) for  $d_{rex}$ . Significant differences are clearly visible between their predictions (after Sellars [26]).

#### 1.2.2 Overview of complete process models

Choquet et al. [15], and Hodgson and Gibbs [16] separately developed complete hot rolling process models for low C micro-alloyed (Mn-Ti-Nb-Mo) steels in the late 1980's to 1990's. The complete model framework was divided into various parts; (1) modelling of flow stress and recrystallization behaviour during multi-pass hot rolling, (2) grain growth after recrystallization, (3) austenite decomposition on the run-out table and (4) precipitation of micro-alloyed carbides. Finally the structure-property relationship was proposed in terms of yield stress, ultimate tensile stress and % elongation [15-16]. Hot torsion simulations were employed to investigate the material behaviour under hot rolling conditions. Choquet et al. [15] used the Kocks-Mecking model [31-32] to describe the work hardening behaviour of Nb and Mo containing microalloyed steels during deformation. During multipass rolling, static recrystallization was considered as the dominant softening mechanism that affects the austenite conditioning for phase transformation [15]. Recrystallization kinetics was described by the JMAK equation [23-25]. In contrast to Choquest et al. [15], Hodgson and Gibbs [16] considered the possibility of dynamic or post dynamic recrystallization during finish rolling. They argued [16] that due to the short interpass times during finish rolling, sufficient strain energy is accumulated that may lead to dynamic recrystallization. However, once the material is out

of any deformation zone (rolling stand), further softening takes place through metadynamic or postdynamic recrystallization. The recrystallized grain size is described empirically as a function of strain, initial austenite grain size and temperature. It is often possible during multi-pass rolling to achieve partial recrystallization between the successive two rolling passes. Hodgson and Gibbs [16] considered this situation and modelled the recrystallization kinetics assuming a single average microstructure with a total effective strain (i.e.  $\varepsilon_{eff} = \varepsilon_2 + \lambda(1 - X)\varepsilon_1$ ,  $\varepsilon_{eff}$  is the effective strain for rolling pass number 2, X is the recrystallized volume fraction,  $\varepsilon_1$  and  $\varepsilon_2$  are the applied strain for pass number 1 and 2 respectively and  $\lambda$  is a constant in the order of unity for C-Mn and C-Mn-Nb steels). Finally they modified the parabolic grain growth law for austenite grain growth as originally proposed by Burke and Turnbull [33] by a general power law equation. The driving pressure for the austenite grain growth was addressed by employing an activation energy term in the range of 430kJmol<sup>-1</sup> for all steels (both C-Mn and Nb/Ti/V containing microalloyed steels). However, the grain growth exponent (*m*) was varied in the range of 4-10 depending on the steel chemistry.

Choquet et al. [15] proposed a transformation model for the austenite decomposition to ferrite under the continuous cooling conditions, adopting an average cooling rate and the amount of undercooling. Ar<sub>3</sub> and Ar<sub>1</sub> temperatures were considered for the initiation of ferrite and pearlite formation. Both temperatures were expressed as a function of the chemical composition of microalloyed steel and the continuous cooling rate. Final ferrite grain size was then derived as a function of the initial austenite grain size, cooling rate, residual strain accumulated during deformation and steel composition. The final microstructure that was obtained for the microalloyed steel [15-16] consisted of ferrite and pearlite. Therefore, to evaluate the base strength of the steel in terms of yield and tensile strength, the contributions from i) solid solution strengthening, ii) the final ferrite grain size and iii) the ferrite and pearlite volume fractions were considered. The total material strength is then expressed as a summation of the base strength and the precipitation strength achieved from the microalloyed precipitates [15-16]. In a separate research work, Anelli [17] expressed the onset of austenite decomposition on the basis of Scheil's hypothesis [34]. Further the fraction of austenite that transformed to ferrite and pearlite or bainite was calculated according to the JMAK equation  $(X = 1 - \exp(-bt^n))$ ,

where X is the fraction transformed, b is the rate parameter, t is time and n is the JMAK exponent) [23-25] adopting additivity for the transformation range above the martensite start temperature ( $M_S$ ). An effective austenite grain size was considered during modelling to accommodate the effect of retained strain. Below  $M_S$ , the austenite transformed to martensite, where the Koistinen and Marburger equation [35] was used to predict the martensite fraction as a function of undercooling below  $M_S$ .

A large amount of research was carried out in Japan to develop complete process models for hot rolling of C-Mn and microalloyed steels [18-21]. In the 1990s', Senuma et al. [19] presented a complete process model of hot rolling of microalloved steels, where austenite grain growth during reheating of slabs was described by using a power law as proposed by Hillert [36]. A pinning parameter was introduced to accommodate the drag force exerted by the microalloyed precipitates during the austenite grain growth. However they argued that during hot strip rolling, where the total reduction is over 90-95%, the final microstructure is hardly influenced by the initial austenite grain size. In contrast, they gave importance to the austenite grain growth that can be observed during the rough and finish rolling regime. A grain growth exponent of 10 is assumed to describe the austenite grain growth after recrystallization. Their finding was similar to that proposed by Gibbs and Hodgson [16]. Both dynamic and static recrystallization was identified as the dominant softening mechanisms during and after deformation, respectively. However, they proposed that dynamic recrystallization is only effective at high operating temperature, i.e. during rough rolling. The static recrystallization behaviour was modelled in terms of the decrease in the dislocation density after deformation. The initial dislocation density was calculated from the stress-strain curve using the Taylor relationship [37], i.e., the flow stress is proportional to the square root of the dislocation density. They proposed that microalloying elements such as Nb, Ti, V have strong retarding effect on the softening kinetics of steel during hot rolling either by solute drag or by strain induced precipitation. Classical nucleation theory was adopted to describe the nucleation rate of strain-induced precipitation. The extent of the drag effect was assumed to depend on the rolling strain, operating temperature and the amount of microalloying content. With increasing microalloying additions, the chances of carbide/carbonitride

precipitation increases and thus precipitate pinning becomes the dominant retarding mechanism rather than solute drag.

The product microstructure that was obtained after the phase transformation was ferrite, pearlite and bainite [19]. To address the austenite decomposition, classical nucleation theory for heterogeneous nucleation was used to describe ferrite nucleation, whereas the ferrite fraction formed was determined by the JMAK equation assuming additivity [18-19, 23-25]. Following the ferrite transformation, pearlite formation was assumed to begin, when the carbon concentration in the austenite-ferrite interphase is higher than the critical carbon concentration required for the cementite formation. Finally, a composition dependent empirical equation is proposed for the bainite start temperature [19]. The overall strength of the steel was evaluated in terms of hardness, which was expressed as the summation of the individual contribution from various transformed phases. A similar approach to determine the hot rolled steel strength was suggested by Suchiro et al. [18]. However, the hardness contribution from ferrite and bainite was expressed as a function of the transformation start temperature of ferrite and bainite. They argued that with decreasing transformation start temperature, the microstructural features of ferrite and bainite became finer and could significantly increases the hardness of the product.

In the later 1990s' Militzer et al. [22] proposed a complete process model for hot strip rolling of various low carbon micro-alloyed steels containing Nb-Ti-V as the main microalloying elements. A parabolic grain growth law was employed to describe the grain growth during reheating and after complete recrystallization during roughing mill deformation. They observed a significant retarding effect from Nb and other microalloying elements (due to the dissolution of Nb rich carbide/carbonitride particles) in solution on austenite grain growth [22]. Supporting the argument originally proposed by Senuma et al. [19], the austenite grain growth after recrystallization was assumed to influence austenite conditioning during hot rolling. To model the softening behaviour, the initial 20% of the total softening was attributed to recovery and the remainder to recrystallization. Both metadynamic and static recrystallization was considered for micro-alloyed steels. To describe the occurrence of metadynamic recrystallization a limiting

Zener-Hollomon parameter (temperature-compensated strain rate,  $Z = \dot{\varepsilon} \exp\left(\frac{Q_{def}}{RT}\right)$ ,

where Z is the Zener-Hollomon parameter,  $\dot{\varepsilon}$  is the strain rate, T is the temperature in K and  $Q_{def}$  is the effective deformation activation energy) was considered. For Z<Z<sub>lim</sub> (limiting value of Zener-Hollomon parameter), interstand metadynamic recrystallization occurred. Similar to Senuma et al. [19] they proposed that metadynamic recrystallization is more probable during rough rolling, where the deformation temperature and strain are relatively high and the applied strain rate is low. In between roughing passes or especially in the early stages of finish rolling, static recrystallization was considered as the dominant softening mechanism and the primary reason for grain refinement. The JMAK equation [23-25] was used to model the static recrystallization kinetics The recrystallized grain size was then described as a function of initial austenite grain size, strain and temperature, where the grain boundary area of the deformed grains were considered as the primary source for the formation of recrystallized nuclei. They argued that a grain refinement limit could be attained, when the recrystallized grain size equals the austenite grain size achieved from the previous recrystallization.

The effect of cooling rate, initial austenite grain size and retained strain was considered on the austenite to ferrite transformation [22]. The temperature at which the 5% fraction transformed was complete was proposed as the transformation start temperature. The transformation start temperature model considered ferrite nucleation at the austenite grain corners and it was proposed that the early growth of ferrite nuclei was controlled by the carbon diffusion in austenite. In their model [22], a limiting carbon concentration in the remaining austenite was introduced above which the ferrite nucleation was inhibited. Subsequent ferrite growth was then modelled using the JMAK equation, assuming additivity to be valid during the ferrite formation regime. A transition temperature between polygonal and nonpolygonal ferrite formation was empirically established as a function of retained strain for Nb/Ti microalloyed steel. Finally, the ferrite grain size, the steel chemistry and the transformation start temperature as originally proposed by Suehiro et al. [18].

One important aspect of their process model [22] was to address the precipitation of microalloyed carbides/carbo-nitrides either in austenite during hot rolling as straininduced precipitation or in ferrite during the run-out table cooling and coiling. The microalloyed precipitation in ferrite was assumed to contribute to the strengthening of hot rolled steel. They argued that in a typical industrial hot strip rolling schedule, the total finish mill residence time of a hot rolled strip is in the range of 10s. This time may be short enough to avoid precipitation during finish rolling. In this situation, the precipitation of micro-alloying elements occurs primarily in the transformed phases during cooling [22]. For Al-killed plain carbon steel, the precipitation was related to the formation of AlN. The kinetics of AlN precipitation in hot rolled coils was described by the model proposed by Duit et al. [38]. However, for HSLA steels, the precipitation was related to the microalloyed carbides (such as in V, Ti and Nb steels) and aging tests in combination with hardness measurement were conducted to estimate the precipitation contribution. Aging results showed a temperature-independent volume fraction of strengthening precipitates that is consistent with the extremely low solubility of microalloying elements in ferrite [22]. To model the aging kinetics, the coarsening of the precipitates was assumed to be the rate controlling step during precipitation. This approach was originally proposed by Shercliff-Ashby [39] to model the aging kinetics in Al alloys considering the coarsening of the precipitates as the rate controlling parameters during aging. Finally, the isothermal precipitation strengthening model was extended to describe the precipitation strength developed during the coil cooling. Assuming a coil cooling rate of 30°Ch<sup>-1</sup>, they predicted an optimum coiling temperature range for the V and Nb microalloyed steels to achieve the maximum precipitation strength on the final hot rolled product. The model prediction of the normalized precipitation strength for Nb and V microalloyed steels is presented in Figure 1.4 as a function of coiling temperature. The complete hot strip process model was successfully applied to various C-Mn and micro-alloyed steels and was able to predict the microstructure-property evolution of the investigated steels under typical hot strip rolling condition.



**Figure 1.4** – Model prediction of normalized precipitation strength as a function of coiling temperature for Nb and V contained HSLA steels. A coil cooling rate of  $30^{\circ}$ Ch<sup>-1</sup> was considered (after Militzer et al. [22]).

As discussed above, previously proposed process models were aimed at the structure-property development for C-Mn and conventional HSLA steels. The hot rolled microstructures were relatively simple, consisting of polygonal ferrite and pearlite as the major transformed products. The microstructural development in a modern AHSS may exhibit similar trends to that of a conventional HSLA steel during rough and finish rolling (i.e. the softening, work hardening and grain growth behaviour may be very similar). However, significant differences are expected in the final hot band microstructure between the two generation steels. As an example, a conventional HSLA steel with a carbon content of approximately 0.1wt% generally produce a final microstructure consisting of polygonal ferrite and a few percent of pearlite. In contrast, the microstructure of a new generation AHSS consists of a combination of polygonal/nonpolygonal ferrite, pearlite, bainite and/or martensite/retained austenite. Therefore an improvement is needed in the previous process models to appreciate the formation of multiphase microstructures in the new generation AHSS.

One of the earliest approaches to model the simultaneous decomposition of austenite to ferrite, pearlite, bainite and martensite was proposed by Umemoto et al. [40]. They developed a thermodynamic program considering a 10 element system (Fe-C-Si-

Mn-Ni-Cr-Mo-V-W-Cu) to predict a free energy composition curve, phase diagrams, the evolution of free energy during ferrite, pearlite and martensite formation, the transformation start temperature for Widmanstätten ferrite and bainite, the parabolic rate constant and growth rate of Widmanstätten ferrite and bainite. The model was also able to accommodate the effect of retained strain on the austenite to ferrite transformation. Ferrite nucleation was described by the classical nucleation theory, whereas a parabolic diffusion controlled growth rate was proposed for the ferrite growth as originally proposed by Zener [41]. Considering ferrite nucleation at austenite grain surfaces, the overall isothermal transformation kinetics of ferrite formation was described by the model proposed by Cahn [42]. Pearlite formation was assumed to occur on the ferrite-austenite phase boundaries as the prior austenite grain boundaries were already occupied by proeutectoid ferrite. A critical ferrite-austenite interphase velocity was considered, below which cementite nucleation was possible. However, the average carbon concentration in the untransformed austenite should be higher than the equilibrium carbon content of austenite with respect to cementite (due to the rejection of carbon during ferrite formation) [40]. A critical driving pressure approach, as originally proposed by Bhadeshia [43], was adopted to describe the initiation of Widmanstätten ferrite and bainite. A diffusional approach [44] was considered to describe the growth of Widmanstätten ferrite and bainite. Finally, the  $M_S$  temperature was calculated by the equation proposed by Hsu and Hongbing [45]. At first, the models were developed for isothermal cooling condition and later extended to the continuous cooling path. The continuous cooling path was considered as the sum of the short time isothermal holding at successive temperatures.

In a separate work, Samoilov et al. [46] proposed a model framework to describe the austenite decomposition kinetics for DP steel. The model prediction was validated under industrial cooling condition at Voest-Alpine Stahl Linz Gmbh. Classical nucleation theory was used describe the polygonal ferrite nucleation, where austenite grain corners, edges and faces were considered as the potential nucleation sites. Carbon diffusion rate was assumed as the rate controlling parameter for the ferrite growth. Ferrite/austenite interfaces were believed to be the only sites available for the pearlite formation in a low carbon steel. It was proposed that the pearlite formation occurs at the later stage of austenite decomposition in a low carbon steel and thus the prior austenite grain boundaries were not available for pearlite formation. The pearlite growth rate was then described by the theory originally proposed by Hillert [47]. A diffusionless approach as proposed by Bhadeshia et al. [48-49] was employed to describe bainite formation. It was considered that the activation energy for nucleation of bainite is proportional to the driving pressure of transformation and a critical stored energy concept was used below which bainite growth is possible and above which austenite would transform to Widmanstätten ferrite [46]. They successfully validated their model for low C steels (carbon content varies from 0.05 to 0.15wt%) based on the industrial hot rolling conditions as presented in Figure 1.5.



**Figure 1.5** – Calculated (lines) and experimentally measured (symbols) ferrite start (5%), bainite start ( $B_s$ ), pearlite start ( $P_s$ ) and transformation end (95%) curves for the continuously cooled C-Mn DP steel (after Samoilov et al. [46]).

Recently, complete hot rolling models for new generation DP and TRIP steels have been developed by Liu et al. [50-51]. The JMAK approach assuming additivity was adopted to model the non-isothermal austenite decomposition to ferrite for a selected DP steel [50]. Carbon redistribution during the austenite to ferrite transformation was explicitly taken into account. The transformation rate parameter was then expressed as a separable function of temperature and fraction of ferrite transformed. Based on the resulting carbon enrichment in the remaining austenite, the martensite start temperature was calculated. The transformation model prediction was verified using experimental results obtained from selected stepped cooling tests designed to replicate industrial runout table cooling strategies. Satisfactory agreement was observed between the model predictions and the experimental results.

A more sophisticated industrial transformation model was proposed for a Mo-TRIP steel (0.2wt%C, 1.5wt%Mn and 1.6wt%Si) [51]. The model framework for ferrite growth was similar to that proposed for the DP steel mentioned above [50]. The model adopted a critical driving pressure concept for bainite start and a diffusional approach was used to describe the kinetics of the subsequent bainite growth. The authors employed the phenomenological Zener-Hillert approach [52] to calculate the maximum growth rate of bainitic ferrite plate as a function of transformation temperature and carbon content of the remaining austenite. Even though the model steel was designed to avoid carbide precipitation within bainite by adding silicon (Si), complete suppression of carbide formation could not be attained. With knowledge of the bainite fraction and the martensite start temperature, the authors employed a carbon mass balance approach to calculate the amount of carbon associated with the formation of cementite. A combination of the JMAK approach with additivity was considered to model the carbide formation during bainite transformation. Finally, the retained austenite fraction was determined by relating empirically the degree of cementite formation and the local distribution of carbon at the transformation temperature [52].

In summary, a limited number of hot strip mill process models have been proposed for DP and TRIP steels, whereas none is available for the CP steel. However, the CP steels are an integral part of the new generation AHSS and are already in use for the fabrication of selected automotive parts. The novelty of new generation CP steels depends on the development of the desired multiphase structures as a result of austenite decomposition during cooling on the run-out table. In contrast to DP and TRIP, CP steels have higher amounts of bainite in the transformed microstructure [4]. Therefore, modelling the evolution of transformed microstructures in CP steels requires further improvements in the bainite transformation model. In particular, it is necessary to critically delineate the transition from ferrite to bainite and martensite under the run-out table cooling operation. In addition, the structure-property predictions are expected to be more rigorous than those presented previously for the conventional steels. Thus it is imperative to develop a complete hot strip process model for the new generation CP steel that can satisfactorily describe the evolution of multiphase microstructure under the industrial hot rolling conditions.

### 1.3 Scope and objective

The overall objective of this work is to develop a microstructure model for hot strip rolling of a selected CP steel grade. The chemical composition of the steel is presented in Table 1.2. The material was lab cast and supplied by ArcelorMittal Dofasco in the form of forged bars.

Table 1.2: Chemical composition (in wt %) of the present CP steel.

Table 1.2. Chemical composition (in we 76) of the present Cr steer.										
C	Mn	Si	S	Р	Nb	Mo	Al	N	Fe	
0.05	1.88	0.04	0.007	0.005	0.049	0.49	0.05	0.004	balance	

The overall research work will be completed in two stages. First, fundamental knowledge needs to be created to understand the microstructural evolution of the steel during hot rolling. Based on the established theoretical knowledge from previous investigations on hot rolling of microalloyed steel, a series of experiments will be designed and carried out to study the microstructural development of the steel. During the experiments, the primary emphasis will be on i) the investigation of the austenite conditioning during hot deformation and ii) the subsequent austenite decomposition into multiphase structures, i.e. a combination of ferrite, bainite and martensite. In particular, the effect of processing parameters (cooling rate, retained strain, grain size etc.) on austenite decomposition will be studied in detail. Secondly, after the completion of these experiments, the results will be analyzed to develop the individual models to describe the metallurgical processes associated with the hot strip rolling operation. In detail, this will include:

- 1) Austenite conditioning during hot deformation in rough and finish rolling: The following three stages will be considered and modelled individually:
  - i) Constitutive behaviour
  - ii) Softening kinetics

- iii) Austenite grain size evolution after recrystallization.
- 2) Austenite decomposition to produce complex multiphase microstructure consisting of ferrite, bainite and martensite: The overall transformation process will be modelled and a particular emphasis will be given to the quantitative description of the transition conditions between the formation of different transformation products, such as from ferrite to bainite.
- 3) Aging kinetics of the transformed microstructure in the coil cooling range: A precipitation model will be developed for microalloyed carbides to predict the suitable coiling temperature range to achieve the optimum mechanical properties for the selected CP steel grade.
- 4) Material property evaluation after hot rolling: A structure-property correlation will be established for the current steel to relate final microstructure with the strength (in terms of hardness) of the material.

Once the complete process model is developed based on the experimental observations, the model will be verified by laboratory simulation of the industrial hot rolling process.

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#### CHAPTER 2: AUSTENITE GRAIN GROWTH\*

#### 2.1 Introduction

Austenite grain size plays an important role in determining the recrystallization of austenite and the subsequent phase transformation during processing of steels in a hot rolling mill. A fine austenite structure is desired to produce finer transformed products. In modern microalloyed steels, various microalloying elements such as niobium, vanadium and/or titanium are used in combination with suitable thermo-mechanical processing to obtain the austenite grain size distribution at elevated temperature that will lead to a desired microstructure after the phase transformation [1-3]. Hence, it is desirable to have reliable knowledge of the austenite grain size evolution during hot rolling of steel. At present, metallographic studies, such as conventional metallography, thermal grooving, oxidation, carburization, glass etching and ferrite/cementite delineation of prior austenite grain boundaries are the primary tools employed to investigate austenite grain growth behaviour [4]. Unfortunately, each method has its own limitations to quantify the grain size to an appreciable extent. Conventional metallography becomes difficult for low and ultra low carbon steels when direct quenching from high temperature to low temperature does not produce a completely martensite structure. Therefore, it becomes difficult to reveal the prior austenite grain boundaries for low and ultra low carbon steels. Other techniques, such as thermal grooving, glass etching, oxidation or carburization of austenite grain boundaries are limited to surface grains where the grain growth behaviour may demonstrate different characteristics than that of bulk grains [2, 4]. In addition, none of the above mentioned techniques is applicable to high temperature real time austenite grain size measurements.

Alternatively, laser-ultrasonics can be used to obtain real time information on austenite grain size evolution. Ultrasonic attenuation is a measure of decay of an ultrasonic wave as it propagates through the material. The attenuation process is known to be sensitive to material parameters such as grain size [5-6] and porosity [7]. The laser-

<sup>\*</sup> A version of this chapter was published in COM, 2006; S. Sarkar, M. Militzer, W. J. Poole and A. Moreau: Intl. Symp. on Advanced Steels, Eds: J. A. Szpunar and H. Li, Metall. Soc. of CIM, Montreal, QC, 2006, pp. 119-130.
ultrasonic technique relies on lasers for both the generation and detection of ultrasound waves and is non-contacting; this makes laser-ultrasonics suitable for high temperature measurements that are relevant to hot rolling operations [8-9]. At high temperature, the rate of attenuation of the ultrasonic signal can be correlated to the austenite grain size [10-12] and, to a lesser extent, to various internal friction mechanisms [13].

In previous studies [10-12], laser-ultrasonics has been used to monitor the austenite grain growth for various C-Mn and HSLA steels with carbon values mostly in the range of 0.05wt% to 0.7wt%. In the current research, this new technique is applied for the current low carbon steel, containing Nb and Mo as microalloying elements (detailed chemistry is presented in page 22). A calibration previously developed for various medium and high carbon grade steels [10-11] was used to quantify the grain size of the present steel grade for temperatures ranging from 900°C to 1250°C. The grain size values measured by laser-ultrasonics were then compared to the measurements made by conventional metallography for selected austenitizing conditions to investigate the effectiveness of the laser-ultrasonic method to measure austenite grain growth in a quantitative manner<sup>\*</sup>.

# 2.2 Experimental methodology

Rectangular strip samples for ultrasonic measurements having a dimension of  $200 \text{mm} \times 20 \text{mm} \times 3 \text{mm}$  were machined from the forged bars of the CP steel. The surfaces were machined to a smooth finish to minimize the effect of surface roughness on ultrasound attenuation. The laser-ultrasonic experiments were carried out using a Gleeble 3500 thermo-mechanical simulator equipped with a laser-ultrasonic system. Resistance heating is employed to heat up the samples and the temperature was monitored with a thermocouple (K type thermocouple for 900°C to 1000°C; R type thermocouple for 1050°C to 1250°C) spot-welded approximately 5mm away from the sample edge but on the same vertical line of the laser detection spot. A second thermocouple was placed approximately 1cm away from the first thermocouple to verify the temperature distribution over the sample. The variability of temperature was within 5°C over an area of 1x1cm<sup>2</sup> during the entire thermal process. The samples were heated at 5°Cs<sup>-1</sup> to various

<sup>\*</sup> A list of symbols used in this chapter is presented in appendix 1.

holding temperatures in the range of 900°C to 1250°C and held for approximately 15min to monitor austenite grain growth. At the end of the experiment, the Gleeble machine was shut off and the samples were allowed to cool to room temperature inside the machine.

The pulse-echo configuration (reflection mode) was used for attenuation measurement. During the whole thermal cycle, measurements were made as follows: a short (5ns) and energetic (150mJ) green (532nm) light pulse from a frequency-doubled Nd:YAG laser (Neodymium:Yttrium Aluminium Garnet laser) is focused on the sample to a circular area of 2mm diameter. This light pulse generates a wideband acoustic pulse by ablating a very thin surface layer of negligible thickness. A second laser (detection laser) with longer pulse (Nd:YAG infrared laser operating at 1064nm, with a pulse duration of 50µs and pulse energy of approximately (70mJ) was used for detection of ultrasonic displacement. The detection laser beam was focused on the sample into a uniform disk approximately 2mm in diameter that was superimposed onto the generation laser spot. A phase shift in the reflected infrared wave at the detection location is essentially proportional to the surface displacement and is picked up by the optical interferometric system attached to the signal processing system. The experimental set-up for the current ultrasonic measurement is presented in Figure 2.1. The measurements were made approximately once every 3 seconds for each thermal cycle.



Figure 2.1 – Schematic diagram of the laser-ultrasonic setup.

To verify the grain size results measured by ultrasonics, some samples were quenched to room temperature either employing helium gas or water quenching after completion of the grain growth experiments. In separate experimental work, selected rectangular samples (15mmx6mmx3mm) were subjected to grain growth treatments at 1050°C, 1150°C and 1250°C. The reheating cycle for the above samples replicated the same heat treatment that was mentioned for the laser-ultrasonic experiments but for a shorter soaking time and hence were interrupted by water quenching to room temperature. Saturated picric acid with a small amount of copper chloride and a wetting agent [14] solution was used to reveal prior austenite microstructure for all samples. All micrographs were taken using a Nikon EPIPHOT 300 series inverted microscope equipped with a digital camera. The individual equivalent area diameter (EQAD) of each grain was first measured using an image analyzer and the average EQAD was calculated for approximately 400-500 grains for each sample. However, the EQAD calculated after sectioning the samples (especially during metallography) can depend largely on how the samples were cut and virtually neglects the three-dimensional character of the grains [3]. Therefore, the volumetric austenite grain sizes, which are required for developing the microstructure evolution models were obtained by multiplying the measured EQAD with a factor of 1.2 [3]. Prior austenite grain size could not be revealed properly for grain sizes below 15µm, i.e. for temperatures of 1000°C and lower. In these cases, the quenched microstructures were found to be a mixture of bainite and martensite, i.e., the prior austenite grain boundaries was not easily visible.

## 2.3 Background theory

Ultrasonic attenuation is a measure of the decay in the ultrasonic wave amplitude and is caused by various microstructural parameters. In polycrystalline metals, grain scattering is usually considered to be the dominant mechanism. When a metal is hit by an intense laser pulse, two physical phenomena occur to generate an ultrasound pulse on the metal surface namely 1) thermal expansion and 2) ablation, including the generation of plasma at the sample surface. Either of these effects induces an ultrasonic wave in the test sample which propagates within the sample and is attenuated due to grain scattering. This scattering mechanism is frequency dependent and hence exhibits different attenuation rates at different frequencies. Generally, the attenuation measurements can be done in a narrowband system (measurement is carried out at a particular frequency) or can be carried out using a broadband system involving a large frequency domain. To adequately explain this relatively new technique, it is necessary to review the principles of grain size measurements by laser-ultrasonics. Ultrasonic grain size measurements are based on the grain size dependence of ultrasonic attenuation [5, 11, 15]. This may be written as:

$$\alpha_{sc}(f,T) = K(T)D^{n-1}f^{n}$$
(2.1)

where  $\alpha_{Sc}$  is the ultrasonic attenuation resulting from scattering by the grains, f is the frequency of the ultrasound wave, T is the operating temperature, D is the average grain diameter, and K(T) is a material parameter that depends on temperature. It is known from theoretical considerations that  $0 \le n \le 4$  depending on the ratio of acoustic wavelength to grain diameter. In the limit of wavelengths much larger than the grain diameter, n tends towards 4 whereas in the limit of wavelengths much smaller than the grain diameter n tends towards 0. In practice, at very long wavelengths, the total attenuation is small and difficult to measure. Conversely, at very short wavelength, the attenuation is so strong that the ultrasound is completely scattered by a few grains. Even though n varies in principle with frequency, in practice, it can be assumed constant (usually n is in the range of 1.5 to 3.5), even when a relatively large frequency range is employed as shown for stainless steel [16].

In addition, other important phenomena such as geometrical considerations, the associated self-diffraction of ultrasound, and internal friction [13] can affect signal amplitude and thus contribute to the total attenuation. When an ultrasound pulse travels some distance along the x direction, from  $x=x_1$  to  $x=x_2$ , the total attenuation may be expressed as:

$$\alpha(x_1, x_2, f, T) = \frac{20}{x_2 - x_1} \log \frac{A(x_1, f, T)}{A(x_2, f, T)} = \alpha_{IF}(f, T) + \alpha_{Sc}(f, T) + \alpha_D(x_1, x_2, f)$$
(2.2)

where,  $\alpha_{IF}$  and  $\alpha_D$  are the contributions to attenuation from internal friction and diffraction respectively and A(x, f, T) is the amplitude of a pulse that has traveled a distance x. In the previous work [11], the geometry was chosen such that the acoustic wave was a plane wave so that  $\alpha_D=0$ . In addition, the grain sizes were relatively large compared to the wavelength of the ultrasound wave and n was found to be 1.5. This implies that scattering by the grains was very intense such that the contribution of internal

friction could be neglected and the total attenuation was due to scattering. i.e.  $\alpha = \alpha_{Sc}$  as given in Eq.2.1.

An alternative measurement can be used when the geometry cannot be chosen such that  $\alpha_D$  is zero or when it is not known precisely. In such a case,  $\alpha_D$  can be estimated experimentally by using another sample having the same sound velocity and geometry and having zero (or negligible) internal friction and scattering. In previous work [10, 17], this reference sample was the same sample or a sample of the same shape and material, but with the measurements taken at room temperature, prior to heat treatment. Using the subscript "*ref*" to identify measurements made on the reference sample one has

$$\alpha_{ref}(x_1, x_2, f, T) = \frac{20}{x_2 - x_1} \log \frac{A(x_1, f, T_{ref})}{A(x_2, f, T_{ref})} = \alpha_D(x_1, x_2)$$
(2.3)

and Eq.2.2 becomes,

$$\frac{20}{x_2 - x_1} \log \left( \frac{A(x_1, f, T)}{A(x_2, f, T)} \frac{A(x_2, f, T_{ref})}{A(x_1, f, T_{ref})} \right) = \alpha_{IF}(f, T) + \alpha_{Sc}(f, T)$$
(2.4)

In general, the measurement sensitivity of laser-ultrasonics varies with temperature and surface condition of the sample as they affect the amplitude of the generated ultrasound pulse and the sensitivity of the detection interferometer. However for the ultrasound wave that has not yet propagated within the sample (i.e. the wave at x=0), it can be assumed that the ultrasound pulse amplitude, and not its shape, depends on temperature. This is a reasonable assumption if the reference sample is made of the same material as the materials to be tested. Therefore, the pulse amplitude at one temperature can be linearly related to the pulse amplitude at another temperature, i.e.

$$A(0, f, T) = c(T)A(0, f, T_{ref})$$
(2.5)

where c is a parameter that can vary with temperature and the sample condition. Implicit in Eq.2.5 is the assumption that the frequency dependence (pulse shape) of the generated ultrasound pulse is independent of temperature. Letting  $x_1=0$  and  $x_2=x$  in Eq.2.4, one has

$$\frac{20}{x} \log \left( \frac{A(x, f, T_{ref})}{A(x, f, T)} \right) = -\frac{20 \log c}{x} + \alpha_{IF}(f, T) + \alpha_{Sc}(f, T)$$
(2.6)

Eq.2.6 shows that only two measurements are needed: one at temperature of interest and the other at a reference temperature for the same propagation distance. As an

improvement to the method of Dubois et al. [11], where internal friction was assumed to be zero, it can be assumed that internal friction contributes an unknown but frequencyindependent amount to the total attenuation. Eq.2.6 combined with Eq.2.1 becomes

$$\frac{20}{x}\log\left(\frac{A(x,f,T_{ref})}{A(x,f,T)}\right) = a + \alpha_{sc}(f,T) = a + bf^{n}$$
(2.7)

where  $a = -\frac{20\log c}{x} + \alpha_{IF}(T)$  and b is related to grain size by

$$b = K(T)D^{n-1} \tag{2.8}$$

In Eq.2.7, the left side is measured experimentally. Then, it is fit to the right side of Eq.2.7 where, a, b and n are the fitting parameters. Although there are models to estimate K(T) and n, it is more practical to obtain them empirically. This amounts to a calibration procedure where b and D are measured simultaneously and n is assumed constant for some set of calibration samples. Such a calibration is available for austenite grain size in C-Mn and microalloyed steels where n = 3 was assumed [10]. In this previous work, the reference sample was chosen as a steel sample of low attenuation having the same dimensions, or as the same steel sample prior to being heated to high temperature (provided the scattering is sufficiently low prior to heating). For the present analysis, n=3was assumed and the value of K(T) was adopted from previous work [10].

# 2.4 Signal processing

The acoustic pulse created by the generating laser, bounces back and forth between the two parallel faces of the sample and is detected as a surface vibration by laser interferometry. Figure 2.2 shows a typical ultrasonic signal obtained from the present steel sample at 1050°C. The signal obtained in the first few hundreds of ns is caused by plasma generation at the sample surface and is not acoustical. Later, two acoustic echoes corresponding to the travel through twice and four times the sample thickness are observed. The ultrasonic attenuation can be measured between two corresponding echo signals at each frequency by comparing their amplitude after Fourier transforming each echo from the time domain to the frequency domain. Only the longitudinal part of the wave-front is considered for calculation as compared to the shear part (the small negative amplitude signal near  $2\mu$ s).

Finally, the attenuation is calculated by taking the ratio of the amplitude spectrum of the first echo with that of a reference echo and divided by twice the sample thickness to obtain an attenuation spectrum. This attenuation spectrum is then fitted to the power law for the corresponding selected suitable frequency domain. As shown in Eq.2.7, the power law is in the form of

$$\alpha = a + bf^n \tag{2.9}$$

As mentioned previously,  $\alpha$  is the total attenuation and f is the frequency in the selected frequency domain. The parameter a has no effect on determining the material grain size as shown previously. The parameter  $b \propto D^{(n-1)}$ , where D is a measure of grain size, i.e. here the average EQAD.



**Figure 2.2** – An example of a typical ultrasonic signal acquired during the grain size measurement at operating temperature for the present steel (for the present case the operating temperature was  $1050^{\circ}$ C).

Unfortunately, when the samples are thick (several to tens of mm) or when the grain size is large (hundreds of microns), the calculated attenuation based on the two-echo method is sometimes affected by noise and the accuracy of the measurement is reduced; especially the signal-to-noise ratio (SNR) can be very poor for the second echo. The other difficulty involving the conventional two-echo method is the requirement for correction of diffraction effects of the ultrasonic pulse, or the requirement that the measurement be done either in the near or far acoustic field, which present strong limitations on the

experimental geometry. Thus the present study employed a more recent technique for the calculation of the amplitude spectrum. In this technique, the amplitude spectrum of the test material is compared with that of a reference system for a single echo signal (generally the first echo is considered for calculation). The reference broadband ultrasonic pulse was obtained from the same sample at room temperature because room temperature attenuation is very low, which is a prime requirement for selecting the reference echo. Taking the ratio between amplitude spectrum at test temperature to that at room temperature for the same sample takes care to a great extent of the diffraction effect with respect to the broadband ultrasonic pulse because both measurements are made with the same experimental geometry, and in particular, for the same propagation distance. Finally, the room temperature signal shows excellent SNR value. Figure 2.3(a) shows an example of the amplitude spectrum of the test material at 1050°C with that obtained at room temperature (reference system) for a broadband frequency range, whereas, Figure 2.3(b) presents the ratio of the amplitude spectrums (in a frequency domain between 3-30MHz) shown in Figure 2.3(a).



(a) (b) **Figure 2.3** – (a) Example of a typical amplitude spectrum of the test object at  $1050^{\circ}$ C and at room temperature (reference spectrum), (b) ratio of two amplitude spectra presented in (a) or amplitude ratio for the particular trace measured at operating temperature,  $1050^{\circ}$ C.

Figure 2.4 exhibits a good fit of power law (Eq.2.9) with the attenuation spectrum for a frequency domain between 3MHz to 30MHz. Attenuation spectrum can be defined

as the amplitude ratio (Figure 2.3(b)) divided by the distance traveled by the ultrasound wave (here it is 2xthickness of the material).



**Figure 2.4** – Experimentally measured attenuation spectrum for the particular amplitude trace at  $1050^{\circ}$ C (presented in Figure 2.3) and its description by power law (Eq.2.9) within the selected frequency domain.

Generally three separate regimes are considered for the conventional analysis [2]:

Rayleigh regime ( $\lambda >>D$ )	$\alpha = K_R D^3 f^4$	(2.10a)
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Stochastic regime ( $\lambda \approx D$ )	$\alpha = K_s D f^2$	(2.10b)
Steenaone regime (// 2)		(2.100

Diffusion regime ( $\lambda << D$ )  $\alpha = K_D / D$  (2.10c)

where  $\lambda$  is the acoustic wavelength and  $K_R$ ,  $K_S$  and  $K_D$  are constants of the material. For the present research, the relationship between the ratio of the ultrasonic wavelength to the average grain size due to grain scattering lies between Rayleigh and Stochastic regimes.

# 2.5 Results and discussion

Figure 2.5 shows the austenite grain growth behaviour of the present steel grade as measured by laser-ultrasonics (based on the previously described calibration procedure) at selected temperatures (1050°C and 1150°C), which fall into different grain growth regimes. For each temperature two complete cycles of measured grain size data are presented in Figure 2.5. Although ultrasonic measurements show some scatter for any test

temperature, the grain size evolution as measured by laser-ultrasonics shows the reproducibility that is at least as good as that obtained from the traditional metallographic techniques. As can be seen from Figure 2.5, at 1050°C, the initial grain size is approximately 15 $\mu$ m, when the samples reached the test temperature (i.e. when t=0, t is the time of measurement). During further holding at 1050°C, a continuous austenite grain growth is observed for the present CP steel. In contrast to 1050°C, the initial grain size value measured at 1150°C is approximately 75µm. The variation in the initial grain size values between the test temperatures (here, at t=0, 15µm for 1050°C vs. 75µm for 1150°C) is because of the increase in the grain size value during the reheating stage, i.e. the time spending (20s) during increasing the temperature of the sample from 1050°C to 1150°C. During further soaking at 1150°C, grain sizes increase rapidly in the first two to three minutes followed by a much slower grain growth rate at longer holding time. Figure 2.5 also presents the metallography results at these temperatures. At 1050°C, laserultrasonic results show reasonable agreement with grain size data obtained from metallography. The situation is more complex for the grain size evolution at the higher test temperature of 1150°C. For short holding times (e.g. 2min at 1150°C) the apparent austenite grain size obtained by laser-ultrasonics is significantly larger than the grain size measured from conventional metallography, whereas at longer time (after 15min) both predictions are consistent with each other. Conventional metallography revealed bimodal grain structure for 2min holding at 1150°C. An example is shown in Figure 2.6, where a few large grains in the range of 90-100µm are surrounded by many small grains of average grain diameter 15-30µm. In comparison, Figure 2.7 shows the micrograph of the steel sample, which was soaked at 1150°C for 15min, where a more homogeneous distribution of grain sizes is observed.



**Figure 2.5** – Comparison of average grain sizes measured by laser ultrasonics (lines) and conventional metallography (symbols). For each temperature, two laser-ultrasonic measurements are presented (solid vs. dashed data lines). Grain sizes are presented as EQAD.



Figure 2.6 – Observed grain structure after 2min soaking at 1150°C as revealed by optical metallography.



Figure 2.7 - Observed grain structure after 15min soaking at 1150°C as revealed by optical metallography.

Figure 2.8 summarizes the mean austenite grain size evolution obtained for the present CP steel grade by laser-ultrasonic technique at seven austenitizing temperatures between 900°C to 1250°C for a soaking period of fifteen minutes. In a few operating temperatures, such as at 950°C, 1000°C, 1050°C, 1100°C, 1150°C and 1250°C, more than one measurements (2-4 measurements) were done to see the variability in the ultrasonic results. Deviations are observed (approximately 15-25% deviation from the average grain size evolution data) and thus average austenite grain size evolution results are presented at those temperatures (Figure 2.8). Ultrasonic measurements were made at 1250°C for 15min, but the analysis of the grain size data were restricted to the initial 300s because of the poor signal-to-noise ratio caused by high attenuation at longer soaking time. The laser-ultrasonics grain size data were smoothed by using best fit polynomial functions at each test temperature. As can be seen from Figure 2.8, the laser-ultrasonic measurements are consistent with what one would expect, i.e. the austenite grain size increases with temperature and soaking time.

As presented in Figure 2.8, the entire austenite grain growth phenomenon can be divided into three distinct grain growth regimes. At low temperatures (900°C to 1000°C), there is negligible grain growth probably due to the strong pinning effect on austenite grain boundary motion by precipitates of the microalloying elements. A previous study on the austenite grain growth in a Nb contained microalloyed steels [18] suggested that the dissolution of the Nb-rich precipitates starts at temperatures of 1100°C and above. The

larger grain growth rates observed at 1050°C and 1100°C as compared to lower testing temperatures (900-1000°C), probably reflect a growth stage when the pinning effect of precipitates on grain boundary motion is gradually becoming weaker. Complete dissolution of precipitates may not be possible at these temperatures. The situation becomes more complex at higher temperatures, such as at 1150°C. It appears that at 1150°C, there is rapid (more or less complete) dissolution of precipitates promoting abnormal grain growth (a situation when few bigger grains are surrounded by many smaller grains) for shorter soaking time (within first 300s of soaking at 1150°C), whereas longer holding time at 1150°C stabilizes normal grain growth of the coarse structure that emerges from the abnormal growth stage. These situations are verified by conventional metallography as presented in Figures 2.6 and 2.7. Grain growth at 1200°C is similar, i.e. rapid initial growth is followed by much slower growth rates at longer holding times.



**Figure 2.8** – Austenite grain growth behaviour at various test temperatures as obtained by laser-ultrasonics (grain sizes are in EQAD).

During the abnormal grain growth regime, large grains dominate the ultrasonic response. A simple explanation can be given if the material can be modeled as a mixture of two components, one with a distribution of large grain sizes, and one with a distribution of small grain sizes. Each volume fraction will contribute to the total attenuation according to Eq.2.9. Because the attenuation increases as the square of grain size (considering n=3), the volume fraction of large grain sizes will dominate the

attenuation behaviour. In contrast, a lower average grain size is obtained using conventional metallography and analysis. However, the bimodal grain size distributions that result from abnormal grain growth cannot be simply characterized by an "average" grain size; proper care has to be taken to give equal importance on the distribution of small and large grains. At much longer holding times and/or higher temperatures (e.g. 1200°C and 1250°C), the precipitates are readily dissolved and hence grain growth behaviour reverts back to a normal grain growth regime. However, at higher temperatures, such as at 1200°C and 1250°C, the number of grains in the analyzed volume is comparatively small so that the statistics are poor. This factor may then decrease the accuracy of the measurements both by laser-ultrasonics and metallography.

#### 2.6 Conclusion

Laser-ultrasonics employing first-echo processing is used to measure the austenite grain size for a low-carbon complex-phase steel containing 0.05wt%C. A previously developed calibration procedure for plain carbon steels with carbon levels ranging from 0.05 to 0.7wt% can also be employed to measure the grain size for the present low-C steel, as demonstrated by comparing the laser ultrasonic measurements with austenite grain sizes measured using conventional metallography. To a first approximation, grain size values obtained from the two different techniques match well with each other. However, during abnormal grain growth regime (at temperature range above 1100°C for shorter holding time), the laser-ultrasonic technique probably measures the grain size of the larger grains as opposed to the mean of the small and large grains. Similarly at much higher temperatures (1200°C and 1250°C), the attenuation is large and difficult to measure and the number of measured grains becomes relatively small. These two factors can limit the capability of the technique for measuring austenite grain size at higher temperatures.

However, the laser-ultrasonic technique has important distinct advantages as compared to other techniques. First of all, laser-ultrasonics is less time-consuming as compared to the conventional metallography technique. In addition, from an application point of view in industry, it has immense potential, especially in the high temperature metal industry. Being a non-contacting technique, there is an opportunity to use this approach for on-line high temperature grain size measurement in an industrial setting, e.g. as required during hot rolling of steel, and such on-line measurements are already being made [10]. The real-time determination of the austenite grain size could provide a tool to control microstructure in a closed loop controlled method during hot rolling of steel or other materials.

Based on the present ultrasonic experiment, the reheat conditions are considered for further softening and transformation study of the present steel. Care is taken during the selection of reheat conditions to avoid any soaking regimes that can result in a bimodal grain size distribution. The reheat conditions adopted for the present steel are soaking the steel sample at 950°C, 1000°C, 1050°C, 1100°C and 1250°C for 120s to achieve a wide volumetric austenite grain size distribution ranging from 8µm to 246µm.

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# CHAPTER 3: CONSTITUTIVE AND SOFTENING BEHAVIOUR OF AUSTENITE\*

## 3.1 Introduction

The evolution of austenite microstructure and flow stress during hot strip rolling is related to the softening of steel. In general, the deformation temperature, applied strain, strain rate and initial austenite grain size determine the work hardening and softening behaviour of steel during hot rolling [1-3]. During the roughing or finishing mill deformation, strain accumulated in the hot rolled strip and the softening process takes place during the period of interruption between each rolling passes in the mill [4-6]. Due to the longer interstand time during rough rolling, complete softening is possible for the steel strip, whereas deformed microstructure may not be recrystallized during the later stage of finish mill rolling [6]. In the latter situation, the strain acquired in the previous rolling pass accumulates progressively and significantly influences the flow stress behaviour of the rolled strip. Generally the softening process occurs in two steps; the initial part is dominated by the recovery of the material followed by recrystallization. However in previous hot rolling investigations, the importance of recovery has generally been ignored and the initial 10-20% of the softening has been assumed due to recovery [1, 7-8]. Although recrystallization is considered as the prime softening mechanism, there exist some doubts regarding the type of recrystallization (dynamic or static recrystallization) that primarily controls the microstructural evolution during the hot rolling of steel. It is believed that at high processing temperature and relatively lower strain rates, which are common to rough rolling, dynamic and metadynamic interstand recrystallization can occur and lead to significant softening, whereas static recrystallization becomes dominant during the finish rolling stage where higher strain rates and lower deformation temperatures are employed [3, 7, 9-12].

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From the product microstructure point of view, a fine deformed austenite grain structure associated with a considerable amount of retained strain, i.e. pancaked austenite, at the exit of finish rolling is a prerequisite to achieve a fine transformed microstructure of the processed steel. The formation of pancaked austenite is controlled by the softening of austenite during rolling [7, 12-14]. During modern hot strip rolling of AHSS, partial or no softening is observed during finish rolling as the interstand time decreases and deformation occurs below the so-called static recrystallization stop temperature [7, 13-16]. The austenite grain size decreases during rolling through recrystallization and austenite grains elongate when completion of recrystallization is not possible anymore. In addition, microalloying elements such as Nb, Mo, Mn and Ti delay the softening kinetics significantly either by solute drag or by strain induced precipitation such that pancaked austenite is often produced in microalloyed steels [7, 13-18]. Both decreasing the austenite grain size and/or elongating austenite grain increases the effective grain boundary area per unit volume such that the ferrite nucleation is promoted.

In the present research, the work hardening and static softening have been investigated for hot strip rolling condition that emphasizes multipass deformation in the austenite temperature range. Torsion tests have been conducted to simulate the entire hot rolling from reheating to finish rolling, whereas axisymmetric compression tests were carried out to understand the individual metallurgical phenomena, such as work hardening and softening. Finally existing physical based modelling approaches were considered to model the constitutive and softening behaviour of the present steel<sup>\*</sup>.

#### **3.2** Experimental methodology

To simulate the entire process of hot strip rolling similar to that experienced in industry (i.e. in terms of deformation temperature, deformation strain and interpass time), a hot torsion simulator (HTS100) was employed. The torsion specimens are 183mm in length, with a diameter of 14.3mm; the length of the working zone is 12.7mm, with a diameter of 10mm. There is feedback temperature control during reheating using two spot-welded thermocouples (one S type and another K type); while during multipass deformation an optical pyrometer is employed for this purpose. Prior to the test, the

<sup>\*</sup> A list of symbols used in this chapter is presented in appendix 2.

chamber was evacuated to a vacuum of approximately 5Pa and then back filled by argon to minimize surface oxidation and decarburization of the samples. A previously adopted standard hot rolling schedule consisting of one roughing pass (R1) and seven finishing passes (F1-F7) was simulated at an applied strain rate of  $1s^{-1}$  [7]. After deformation, He gas quenching was employed to cool the sample to room temperature. The details of the torsion simulation schedule are shown in Table 3.1. The primary aim of the standard torsion simulation was to compare the stress-strain behaviour of the present steel with three previously investigated C-Mn and microalloyed steels. The chemical compositions of these steels are presented in Table 3.2 (the current steel chemistry is found in page 18).

 Table 3.1: The schedule of the standard torsion experiment [19].

Soaking conditions: heating at 5°Cs <sup>-1</sup> to 1200°C and hold for 15minutes								
Parameters	R1	F1	F2	F3	F4	F5	F6	F7
Temperature (°C)	1100	1041	979	955	934	919	908	898
Equivalent strain	1	0.36	0.51	0.41	0.33	0.30	0.25	0.10
Interpass time (s)	10	8.0	4.8	3.2	2.3	1.7	1.3	-

Table 3.2: Chemical composition of previously investigated steels (in wt%) [7].

Steel	C	Mn	Si	Nb	V	Ti	Mo	Al	N
DQSK	0.038	0.3	0.009	-	-	-		0.04	0.005
Nb	0.08	0.48	0.045	0.036	-	-	-	0.024	0.0054
Nb/Ti 80	0.07	1.35	0.14	0.086	-	0.047	-	0.044	0.007

Subsequently, a revised hot rolling regime was simulated (Table 3.3) that reflects more accurately the processing conditions of a seven stand finishing mill except for the lower strain rates that have to be employed in the torsion simulations. Here, the sample was soaked at 1250°C for 30minutes and after 3 roughing passes (R1-R3 at a deformation temperature 1100°C) with a total true strain of 1 the 7 finishing passes were carried out between 1025°C and 900°C employing a strain rate of 1s<sup>-1</sup>.

**Table 3.3**: Modified torsion schedule simulating the hot strip rolling for the present steel.

Soaking conditions: heating at 5°Cs to 1250°C and hold for 30minutes								
Parameters	R1, R2 and R3	<b>F</b> 1	F2	F3	F4	F5	F6	F7
Temperature (°C)	1100	1024	982	958	950	922	910	900
Strain	0.33 (each pass)	0.35	0.50	0.40	0.30	0.30	0.25	0.10
Interpass time (s)	10	4	2.4	1.6	1.2	0.8	0.6	-

To evaluate the work hardening and softening behaviour of the present steel, a series of axisymmetric single and double stage compression tests were conducted on a Gleeble 3500 thermo-mechanical simulator using cylindrical samples (15mm in length and 10mm in diameter). The tests were conducted under high vacuum  $(1.3 \times 10^{-4} Pa)$ . Resistance heating was employed to heat the samples and the temperature was monitored with a thermocouple feedback system (K type thermocouple for soaking up to 1000°C: S type thermocouple for higher reheating temperature). The thermocouple was spot-welded at the centre of the sample. In selected tests, a second thermocouple was placed approximately at the edge of the cylindrical specimen to check the variability of temperature distribution over the sample. The maximum temperature variability observed between the centre and the edge thermocouple was approximately 5°C. Graphite foil in combination with nickel paste was used as a lubricant. The details of the single hit compression tests are presented in Table 3.4. An entire test consists of heating the sample to a soaking temperature at a heating rate of  $5^{\circ}$ Cs<sup>-1</sup> to achieve a desired austenite grain size. The selection of soaking temperatures was based on the austenite grain growth analysis described in Chapter 2. After soaking, the sample was cooled to the deformation temperature at a cooling rate of 10°Cs<sup>-1</sup>. Deformation to a selected strain at a constant strain rate was carried out after the sample temperature had been stabilized for approximately 5s.

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Soaking temperature	Deformation	Applied strain rate	Deformation
(°C)	strain	(s <sup>-1</sup> )	temperature (°C)
1250	0.5	0.1, 1.0,10.0	900-1100
1150	0.5	0.1, 1.0,10.0	900-1100
1050	0.5	0.1,1.0, 10.0	900-1000
950	0.5	0.1, 1.0,10.0	900

Table 3.4: Test matrix for the single hit tests.

The test matrix for the two stage compression tests is shown in Table 3.5. These compression tests were conducted to quantify the softening kinetics as a function of initial austenite grain size (given as volumetric grain size), applied strain, strain rate and processing temperature. During two stage compression tests, once the first stage of deformation is complete, the sample was held at the deformation temperature for various lengths of interhit-time (1-500s) followed by a second stage of deformation. Strain rate

was kept constant during the first and second stage of deformations in each test. After the completion of the second deformation stage, the power was shut off and the sample was allowed to cool inside the chamber. At least 5 measurements with various interhit-times were considered for each deformation condition.

Soaking	Austenite grain	Deformation	Applied strain	Deformation
temperature (°C)	size (µm)	strain	rate $(s^{-1})$	temperature (°C)
1250	246	0.2, 0.3, 0.5	0.1, 1.0,10.0	950-1100
1100	62	0.3	1.0	1000
1050	20	0.3	1.0	1000
1000	17	0.3	1.0	1000

Table 3.5: Test matrix for the double stage compression tests.

## 3.3 Results and modelling

#### 3.3.1 Stress-strain behaviour in multipass deformation

The stress-strain behaviour of the present steel in a standard hot strip mill simulation is compared in Figure 3.1 with that of the three previously investigated C-Mn and HSLA steels [7]. The results show that the flow stresses vary according to the level of Nb microalloying content. In particular, the present microalloyed steel flow stresses fall between those of the HSLA steels containing 0.086wt% and 0.036wt% Nb, respectively, thereby suggesting that previously developed concepts for work-hardening in austenite can be adapted for the new steel.

Figure 3.2 presents a more accurate hot strip mill simulation results (rolling schedule presented in Table 3.3) of the current steel. As can be seen from Figure 3.2, significant softening is observed during roughing rolling (between R1-R2 and between R2-R3) and in between roughing and finish rolling. In the later finishing passes (especially after the third finishing pass, F3), corresponding to rolling temperatures of around 950°C or below, the austenite work-hardens continuously. This suggests that little recrystallization can be attained for a typical finish mill operation for the present microalloyed steel approximately at 950°C or below.



Figure 3.1 - Stress-strain behaviour of the present steel during a standard hot strip mill simulation as compared to three previously investigated C-Mn and HSLA steels [7].



**Figure 3.2** - Stress-strain behaviour of the present steel in terms of equivalent stress-strain curves during typical hot strip mill simulation (Table 3.3).

#### 3.3.2 Flow stress behaviour

The flow stress behaviour of austenite is investigated in terms of the effect of deformation temperature  $(T_{def})$  and strain rate ( $\dot{\varepsilon}$ ). As expected the flow stress increases with decreasing deformation temperatures and increasing strain rates. Indirect evidence of dynamic recrystallization (existence of a peak in the stress-strain curve) is observed

(Figure 3.3) for deformation conditions involving higher deformation temperature, i.e. in the present case, 1100°C, higher soaking temperature (1150-1250°C) and slower strain rate (0.1-1.0s<sup>-1</sup>), which is typical of rough rolling. A similar observation regarding the existence of potential dynamic recrystallization was reported in the previous hot rolling investigations on C-Mn, HSLA, DP and TRIP steels [7, 9-11]. However, for the present scenario, the deformation conditions affected by dynamic recrystallization are neglected considering its' minor impact on the overall evolution of austenite microstructure during the entire hot deformation process [7, 9, 20-21].



Figure 3.3 – Flow stress curves for conditions involving dynamic recrystallization.

Previous research [7, 9] has shown that the reheating/soaking condition (i.e. the initial austenite grain size) has a negligible effect on the flow stress behaviour of austenite during hot deformation in the absence of dynamic recrystallization [20-21]. However, as shown in Figure 3.4(a), the observed stress-strain curves at 900°C ( $T_{def}$ ) exhibit the effect of initial soaking conditions; the main difference being the value of yield stress achieved for each stress-strain curve. At higher soaking temperatures (1150-1250°C) corresponding to an average austenite grain size (volumetric grain size ranging from 180-250µm), the overall flow stress curves look similar. In contrast, a significant rise in the yield stress values (from 40MPa to 65MPa) that ultimately raises the overall flow stress curves, is observed at lower soaking temperatures (950-1050°C, correspond to smaller austenite

grain sizes in the range of 8-20µm). Thus, to investigate the work hardening behaviour of the present steel, yield stress values are deducted from the total flow curves and presented in Figure 3.4(b). A possible explanation of the observed grain size dependencies on the yield stress values, could be the presence of microalloyed precipitates in the matrix at lower soaking temperature (950-1050°C), whereas partial or significant dissolution of precipitates is expected at higher soaking condition, i.e. during soaking at 1150-1250°C. The presence of higher precipitate volume fraction can increases the yield stress of the material for lower soaking conditions.



Figure 3.4 –Flow curves of the CP steel at various soaking temperatures: (a) including the yield stress value, (b) without the yield stress value.

To model the constitutive behaviour of steel during hot deformation, the Kocks-Mecking (KM) approach [22-23] has been used by many previous researchers [21, 24-26], due to its physical basis for modelling the stress-strain behaviour of FCC material. In this model, the flow stress of the FCC material can be expressed in two terms:

$$\sigma = \sigma_{YS}(T_{def}, \dot{\varepsilon}) + \sigma_{WH}(T_{def}, \varepsilon, \dot{\varepsilon})$$
(3.1)

where, the first term on the right hand side of Eq.3.1 is the temperature-strain rate dependent yield stress and the second term signifies the contribution from work hardening of the material. The work hardening increases with strain as the dislocation structure evolves due to the dislocation accumulation during deformation and rearrangement during dynamic recovery. In the present scenario, the work hardening behaviour of austenite is assumed to follow the Voce equation:

$$\sigma_{WH} = \sigma_{v} \left( 1 - \exp\left(-\frac{\theta_{0}}{\sigma_{v}}\varepsilon\right) \right)$$
(3.2)

where,  $\varepsilon$  is the true strain,  $\theta_0$  is the initial work hardening rate and  $\sigma_v$  is the scaling stress associated with the end of stage III work hardening. The temperature-strain rate dependency of the scaling stress can be expressed as follows [21]:

$$\frac{\sigma_{\nu}}{\mu} = \frac{\sigma_{\nu 0}}{\mu_0} \left( 1 - \left( \frac{k_B T_{def}}{g_0 \mu b^3} \ln \frac{\varepsilon_0}{\varepsilon} \right)^{\frac{1}{2}} \right)^2$$
(3.3)

In Eq.3.3,  $\sigma_{v0}$  is the scaling stress extrapolated to 0K,  $\mu$  is the temperature dependent shear modulus of pure iron [27],  $\mu_0$  is the value of the shear modulus at 0K,  $k_B$  is Boltzmanns' constant (1.381×10<sup>-23</sup>JK<sup>-1</sup>), *T* is the absolute temperature (in Kelvin), *b* is the Burgers vector that is related to the lattice constant of austenite ( $a_\gamma$ ) by  $b = \frac{a_\gamma}{\sqrt{2}}$  where

the temperature dependence of  $a_{\gamma}$  is taken into account [28],  $\dot{\varepsilon}_0$  is the strain rate normalizing factor and  $g_0$  is a material property, which depends on the stacking fault energy and the chemistry of the alloy. Eqs.3.2 and 3.3 contains four adjustable parameters that need to be determined, i.e.  $\theta_0$ ,  $g_0$ ,  $\dot{\varepsilon}_0$  and  $\sigma_{v0}$ . Previous study on TRIP steel [21] suggested a constant value of  $\mu/30$  and  $1 \times 10^7 \text{s}^{-1}$  for  $\theta_0$  and  $\dot{\varepsilon}_0$ , respectively, which is also in the range expected for FCC materials as proposed by Kocks and Mecking [22]. Therefore those values are adopted for the present analysis. Finally the scaling stress for each deformation condition (a combination of strain rate and temperature) is determined by plotting the experimentally observed work hardening rate  $(\frac{d\sigma}{d\varepsilon})$  in terms of flow stress after subtracting the 0.2% yield stress. In order to evaluate the  $\sigma_{v0}$  and  $g_0$ , plots of  $((\sigma_v - \sigma_{vS})/\mu)^{1/2}$  vs.  $(\frac{k_BT}{\mu b^3} \ln(\frac{\dot{\varepsilon}_0}{\dot{\varepsilon}}))^{1/2}$  as a function of temperature and strain rate are utilized as shown in Figure 3.5(a). Considering a  $g_0$  value of 1.0 [21], from the intersection of the plots a value of 1725MPa is obtained for  $\sigma_{v0}$ . As mentioned before, the first term on the right hand side of Eq.3.1 refers to the yield stress, which depends primarily on the Peierls stress for dislocation motion and solid solution hardening. To consider the yield stress, only the higher soaking conditions (1150-1250°C) are of importance as they closely replicate the condition of microalloyed precipitates (mostly in solution) during typical hot rolling operation. From a modelling point of view, this term is considered in the framework of thermally activated deformation as shown previously [21, 25], i.e.,

$$\frac{\sigma_{YS}}{\mu} = \frac{\sigma_{YS0}}{\mu_0} \left( 1 - \left( \frac{k_B T_{def}}{g_i \mu b^3} \ln \frac{\dot{\varepsilon}_0}{\dot{\varepsilon}} \right)^{\frac{1}{2}} \right)^2$$
(3.4)

where, the adjustable parameters are  $\sigma_{YS0}$  and  $g_i$  and evaluated from experimental data as shown in Figure 3.5 (b). Considering a  $g_i$  value of 1.0 [21], from the intersection of the plots a value of 595MPa is obtained for  $\sigma_{YS0}$ .



Figure 3.5 – Variations of saturation and yield stresses at different deformation temperatures and strain rates from various initial soaking conditions: (a) saturation stress variation, and (b) yield stress variation.

Finally the KM model results (solid lines) are presented in Figure 3.6 in comparison to the experimental data in terms of deformation temperature and applied strain rate. Soaking condition relevant to industrial hot rolling processing (i.e., 1250°C) is considered for the complete model prediction of the present steel. With the available model parameters the flow stress prediction of the present steel is extrapolated to higher

strain rate (100s<sup>-1</sup>) that is typical for a hot strip finish rolling operation. As can be seen from Figure 3.6, the model calculations and experimental results are in good agreement.



**Figure 3.6** – Comparison between the experimental (symbols) and model calculated results (solid lines) for the constitutive behaviour of the present steel: (a) effect of strain rate, (b) effect of deformation temperature.

### 3.3.3 Softening behaviour

Two typical double hit test results are presented in Figure 3.7. In this situation, the sample was soaked at 1250°C for 2min (volumetric austenite grain size of 246 $\mu$ m), cooled to 1000°C and then deformed with a strain rate of 1.0s<sup>-1</sup> up to a strain of 0.3. The interhit time employed were 10s and 15s, respectively.

To quantify softening from the two stage compression tests, changes in the yield stress are evaluated. The yield stresses are measured using an offset strain of 0.002 for the first hit ( $\sigma_0$ ) and the second hit ( $\sigma$ ), respectively. The yield stress ( $\sigma_0$ ) of the first stage of deformation is considered as the stress of the fully softened material, whereas  $\sigma$ represents the stress of the material other than the fully softened material. When the interhit time is zero (i.e. t=0s), the value of  $\sigma$  becomes  $\sigma_D$ , the as deformed stress of the first hit (Figure 3.7). During the compression tests it was observed that with increasing interhit time, there is a significant reduction in the yield stress ( $\sigma$ ) due to recovery and recrystallization consistent with the findings of previous studies on other steels (in Figure 3.7,  $\sigma$  decreases when the interhit time increases from 10s to 15s) [7, 9, 29]. By measuring the value of  $\sigma_0$ ,  $\sigma$  and  $\sigma_D$ , the fraction softening ( $F_S$ ) can then be calculated as follows:

$$F_s = \frac{\sigma_D - \sigma}{\sigma_D - \sigma_0} \tag{3.5}$$

An example is presented in Figure 3.8, where an initial volumetric austenite grain size of 246µm, an applied strain of 0.3 and a strain rate of 1.0s<sup>-1</sup> are considered for deformation in the temperature range from 950-1100°C. As expected, softening becomes faster with increasing deformation temperature. It is also important to observe that softening is completely inhibited at 950°C after approximately 30-40s. In addition, the experimental results confirm that softening rates become faster with increasing strain and strain rate and decreasing initial grain size. This finding is in agreement with the previously published softening results on microalloyed steel [7].



Figure 3.7 – Typical stress-strain curves obtained during the double hit tests for two different interhit times, 10s and 15s.

Previous models to describe the recrystallization kinetics during hot deformation were primarily based on the application of the Johnson-Mehl-Avrami-Kolmogorov (JMAK) approach [7, 10, 20-21, 24, 29-33], where the process of recrystallization was considered similar to a phase transformation. It was generally assumed that the initial 10-20% of total softening was due to recovery and thus recrystallization was considered as the dominant softening mechanism of the steel during hot deformation. The time for 50% recrystallization  $(t_{0.5})$  was expressed in terms of applied strain  $(\varepsilon)$ , strain rate  $(\dot{\varepsilon})$ , the average initial volumetric austenite grain size  $(D_0)$  and the deformation temperature  $(T_{def})$  as follows:

$$t_{0.5} = AD_0^p \varepsilon^q \dot{\varepsilon}^r \exp\left(-\frac{Q_{rex}}{RT_{def}}\right)$$
(3.6)

where, R is the universal gas constant (8.314Jmol<sup>-1</sup>K<sup>-1</sup>); A, p, q, r and  $Q_{rex}$  are fitting parameters that change with alloy composition. Finally the fraction recrystallized ( $F_x$ ) was expressed as

$$F_x = 1 - \exp(-0.693 \frac{t}{t_{0.5}})^n \tag{3.7}$$

where, n is the JMAK exponent that in theory depends on the nucleation and growth kinetics of recrystallization. This approach is simple and is suitable to industrial practise. However, the JMAK approach lacks the physical basis to understand the underlying mechanisms involved in the softening process. In addition, the model does not account for the recovery process after hot deformation.



**Figure 3.8** – Fraction softened calculated from double hit tests (effect of deformation temperature). Solid lines represent curve fitting. Softening process is completely inhibited at 950°C after approximately 40% softening, probably due to the strain induced microalloyed precipitation [13-18].

For the present analysis, a physically based softening model coupling recovery, recrystallization and precipitation previously developed for a Nb microalloyed steel [34] is employed to describe the softening behaviour. The model assumes recovery and recrystallization as two simultaneously operating softening mechanisms and may interact strongly with microalloyed precipitation depending on the deformation condition. In this model [34], recovery is considered as a thermally activated process and the rate of recovery depends only on the dislocation density (previously proposed by Guyot et al. [35]). The transition from recovery to recrystallization as the dominant softening mechanism is purely based on the dislocation density of the deformed state, which is considered as a function of time. The recrystallization model depends on three parameters: (1) the driving force for recrystallization, (2) the temperature dependent boundary mobility and (3) the number of recrystallization nuclei/volume.

In the present analysis, the value of the yield stress ( $\sigma$ ) was compared to  $\sigma_0$  (fully softened stress) and the difference of these two stress levels provides a measure for the amount of softening in between deformation steps. For simplicity and to be consistent with a typical hot strip rolling scenario, effects from precipitation are neglected in the present analysis since the majority of tests were performed after soaking at 1250°C, where almost complete dissolution of precipitates is expected. The rule of mixture is considered to express the yield stress evolution of the steel from the deformed to the fully softened state, i.e.,

$$\sigma = \sigma_o X_{\text{Re}x} + \sigma_{\text{Re}c} \left( 1 - X_{\text{Re}x} \right)$$
(3.8)

where,  $\sigma_{Rec}$  is the recovered stress of the material and  $X_{Rex}$  is the recrystallized fraction. The recovery kinetics are described as an internal stress relaxation phenomenon through thermally activated dislocation annihilation and rearrangement, i.e. [35]

$$\frac{d(\sigma_{\text{Rec}} - \sigma_0)}{dt} = -\frac{64(\sigma_{\text{Rec}} - \sigma_0)^2 v_D}{9M^3 \alpha^2 E} \exp\left(-\frac{U_A}{k_B T}\right) \sinh\left(\frac{(\sigma_{\text{Rec}} - \sigma_0)V_A}{k_B T}\right)$$
(3.9)

where the activation volume  $(V_A)$  can be expressed as,

$$V_{A} = \frac{\kappa M \alpha \mu b^{3}}{\left(\sigma_{D} - \sigma_{0}\right)}$$
(3.10)

In Eqs.3.9 and 3.10, M is the Taylor factor (3.1 for FCC material),  $\alpha$  is a geometric constant (0.3),  $v_D$  is the Debye frequency (10<sup>13</sup>Hz), E and  $\mu$  are the temperature

dependent Young's modulus and shear modulus of iron, respectively [27]. A value of 286kJmol<sup>-1</sup> that was previously used for Nb steel [34] is adopted here for the activation energy  $(U_A)$  and  $\kappa$  is employed as a fitting parameter [36]. The fraction recrystallized as a function of time (X(t)) was described by,

$$X(t) = 1 - \exp\left(-N_{Rx}M_{eff}^{3}\left(\int_{0}^{t}G(t)dt\right)^{3}\right)$$
(3.11)

Assuming site saturation conditions for nucleation of recrystallizing grains at grain boundaries, the potential nucleation density  $(N_{Rx})$  was taken to be proportional to the grain boundary area/unit volume  $(S_V)$  of deformed grains,

$$N_{Rx} = Kb^2 S_V \rho_D^2 \tag{3.12}$$

where K is an adjustable parameter and  $\rho_D$  is the initial dislocation density. The net driving pressure for recrystallization (G(t)) can be expressed as,

$$G(t) = \frac{1}{2}\rho(t)\mu b^{2}$$
(3.13)

Here, the dislocation density ( $\rho$ ) is obtained from the flow stress taking into account the recovery process using the Taylor relationship, i.e.

$$\rho = \left(\frac{\sigma_{\text{Rec}} - \sigma_0}{\alpha \mu b M}\right)^2 \tag{3.14}$$

where, the initial dislocation density ( $\rho_D$ ) is obtained by setting  $\sigma_{Rec} = \sigma_D$ . The effective grain boundary mobility ( $M_{eff}$ ) is adopted with an Arrhenius relationship as shown below,

$$M_{eff} = M_0 \exp\left(\frac{-Q_{eff}^M}{RT}\right)$$
(3.15)

where both  $M_0$  and  $Q_{eff}^M$  are adjustable parameters. Given that  $M_0$  can be combined in Eq.3.12 with the fit parameter K of  $N_{Rx}$ , it is useful to introduce  $K_M = KM_0^3$  as a single fit parameter (reduces the total number of fit parameters in the model). The model has then three fit parameters, i.e.  $\kappa$  for the recovery sub-model,  $K_M$  and  $Q_{eff}^M$  for the recrystallization sub-model. Analysing the present softening data,  $\kappa=0.05$ ,  $K_M=1.65 \times 10^3 \text{m}^{12} \text{J}^{-3} \text{s}^{-3}$  and  $Q_{eff}^M = 337 \text{kJmol}^{-1}$  have been determined. It is important to note that the employed approach reduces the number of fit parameters from 6 for the JMAK

model to effectively 4; the activation energy  $U_A$  has in general to be considered to vary with steel chemistry even though in the present analysis a value from the literature has been adopted. Figure 3.9 shows the experimental softening data and model description in terms of decrease in the difference of yield stresses ( $\Delta \sigma = \sigma - \sigma_0$ ) for a number of selected deformation conditions where complete softening is attained in less than 100s. Little recrystallization (less than 25%) is observed at lower deformation temperature (950°C) for inter-hit times up to 500s. The proposed model provides an adequate description of the softening kinetics and provides an attractive alternative to the traditionally employed but rather empirical JMAK approach [7].



**Figure 3.9** – Comparison of experimental results (symbols) and model fit (lines) for the softening behaviour of the CP steel: (a) effect of deformation temperature, (b) effect of applied strain, (c) effect of strain rate and (d) effect of initial austenite grain size.

### 3.4 Discussion

The present study investigated the constitutive and softening behaviour of the present steel under typical hot strip rolling conditions. Current research established that the present steel will experience significant softening during rough rolling or in-between rough and finish rolling, whereas negligible softening is expected at the later stages of finish rolling (Figure 3.2). This behaviour is consistent with expectations considering the effect of Nb and Mo in delaying recrystallization either by solute drag or due to strain induced precipitation. In particular, the effect of solute drag is of prime importance on recrystallization as soaking the steel at 1250°C for 30min will be sufficient to dissolve all microalloyed precipitates into solution. In addition, as discussed elsewhere [7], during a typical hot strip rolling operation, the interstand time may not be sufficient enough for strain induced precipitation to begin. Further, the double hit test results (Figure 3.8 and Figure 3.9) are in satisfactory agreement with the observations made in the torsion simulation (Figure 3.2). For example, softening times are in the range of 4-10s for a deformation temperature of 1000°C and above, when considering a suitable austenite grain size (i.e. larger austenite grain size value of 246µm at rough rolling regime and a smaller austenite grain size value of 17µm at finish rolling regime). Thus, for strip rolling at higher temperatures (approximately 1050°C and above), consistent with rough rolling or early stage of finish rolling, complete softening is possible. However, at lower temperatures (around 1000°C and below) partial or negligible softening may occur, in particular during the final passes of finish rolling, when the deformation temperature is low and the inter-pass times are short.

The current softening study revealed that little softening can be achieved when the sample was held at 950°C for times up to 500s (Figure 3.8). At this temperature (Figure 3.8), softening was slow at the beginning and almost stopped after 30-40s. Previous studies on Nb containing microalloyed steel suggested that approximately 10-30s is needed for strain induced precipitation of microalloyed carbides to begin at an operating temperature range of 850-950°C depending on the amount of microalloying elements in solution and the amount of retained strain in the hot rolled steel. Therefore a possible explanation for the no recrystallization situation observed at 950°C is that the strain-induced precipitation of the microalloying carbides/carbonitrides retards the softening.

However, the typical interstand time in an industrial hot rolling process around 950°C is approximately 1s or below (see, Tables 3.1 and 3.3). Therefore, the current study neglected the interaction between strain-induced precipitation and the material softening.

Considering the present results, it is now possible to apply the complete model (constitutive model + softening model) for predictions under industrial for a hot strip rolling conditions, which closely resembles the industrial rolling setup. As an example, two possible situations are considered below (Figure 3.10). In one case, a typical rough rolling situation was considered up to a deformation strain of 0.3, where an initial volumetric grain size of 200 $\mu$ m, a strain rate of  $10s^{-1}$  and a deformation temperature of 1100°C is chosen for deformation. The softening behaviour was then predicted based on the present model calculation. In the second situation, a typical finish rolling condition is adopted. An average volumetric austenite grain size of  $25\mu m$ , a strain rate of  $100s^{-1}$  and a deformation temperature of 950°C were considered for the second case (strain applied up to 0.3). In both deformation conditions, it is assumed that Nb and Mo are in solution and can only delay the softening through solute drag. As can be seen from Figure 3.10, the softening times are in the order of a few seconds for a deformation temperature of 1100°C such that recrystallization can readily occur during rough rolling (within 1s). However, for the high strain rate and low deformation temperature (e.g. 950°C) conditions in finish mill rolling, recrystallization times extend to approximately 5s. Consequently, finish mill rolling of the present steel will occur under partial or no-recrystallization conditions. This behaviour is consistent with the Nb microalloying content and the associated delay in recrystallization kinetics [7, 37-38].



**Figure 3.10** – Model predictions on the expected softening kinetics for the present steel in two different deformation situations; one applicable to rough rolling and the other typical for the later part of finish rolling condition.

## 3.5 Conclusion

The following conclusions can be drawn from the current experimental and modelling study.

- The current CP steel will achieve full softening during rough rolling. However little or negligible softening is expected during later stages of finish rolling corresponding to the shorter interstand time at lower operating temperatures due to significant solute drag effect from alloying and microalloying elements such as Nb, Mo and Mn. This finding established that the steel microstructure will consist of pancaked austenite at the end of finish rolling, which is a prime requisite for finer transformed product.
- The current research suggests that at deformation temperatures of approximately 950°C and below, the softening can be completely inhibited. One of the possible reasons can be the grain boundary pinning by strain-induced precipitation of microalloying elements (Nb and Mo), thus inhibiting the boundary motion.
- The Kocks-Mecking work hardening model was successfully applied to describe the flow stress behaviour of the present steel on a physical basis. Therefore, it is
possible to extrapolate the model prediction with confidence to the higher strain rates typically experienced in the finishing mill.

• A physically based softening model was employed to successfully describe the softening behaviour in terms of recovery and recrystallization. This model is an attractive alternative to the commonly adopted JMAK approach.

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# CHAPTER 4: RECRYSTALLIZED GRAIN SIZE AND SUBSEQUENT GRAIN GROWTH\*

### 4.1 Introduction

An outcome of recrystallization during hot rolling is the recrystallized grain size, which plays an important role in the evolution of the austenite grain structure [1-3]. It has been established that a fine recrystallized grain size is desirable for the subsequent austenite to ferrite transformation [3]. Although recrystallization plays the primary role in removing the internal energy stored during hot deformation, a further reduction of internal energy is caused by the reduction of the total austenite grain boundary area during grain growth [1-2, 4-5]. Grain growth following recrystallization of austenite generally occurs during rough rolling and in between rough and finish rolling when the deformation temperature is high and interpass time is long [6-7]. Previous research established that grain growth after recrystallization occurs unpinned in plain C-Mn steels, whereas grain growth is reduced in microalloyed steels containing niobium (Nb), titanium (Ti), or molybdenum (Mo) as grain refiners [6, 8-11]. Generally, these microalloying elements retard grain growth either by solute drag or by pinning due to precipitation. Given the significance of the austenite microstructure, it is important to have reliable knowledge of the austenite grain size evolution during hot deformation of steel.

As described in Chapter 2, the investigation of the austenite microstructure is challenging, as this microstructure is not present at room temperature. In addition, common metallographic tools to measure austenite grain size, such as conventional metallography, thermal grooving, oxidation, carburization, glass etching and ferrite/cementite delineation of prior austenite grain boundaries [12] can not be employed for real-time monitoring of austenite grain size at high temperatures.

In contrast, as shown in Chapter 2, laser-ultrasonics is an attractive alternative for real-time monitoring of grain size evolution in austenite. Ultrasonic attenuation, i.e. the decay of an ultrasonic wave as it propagates through the material, is sensitive to material parameters such as grain size [13-14, 16-17] and porosity [18]. Thus, in the absence of

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porosity, attenuation data can be directly correlated to the austenite grain size [13-14, 19-20]. Further, the change in relative velocity of the ultrasound wave during its propagation through the material is related to the evolution of texture and can be utilized to monitor recrystallization in-situ [13, 21-23].

Laser-ultrasonic measurements of the austenite grain size have so far emphasized conventional grain growth tests that involve reheating of a sheet sample [14, 19, 20, 24, Chapter 2]. Based on these investigations, calibration procedures were developed that relate the measured attenuation to the austenite grain size for plain carbon and microalloyed steels with carbon contents ranging from 0.05 to 0.74wt% [14, 19]. Further, the initial laser-ultrasonic measurements were made in combination with the stress relaxation technique (in terms of the evolution of dislocation density) to monitor *in-situ* recrystallization behaviour and recrystallized grain size for a C-Mn steel [23].

This chapter describes laser-ultrasonic measurements of austenite grain size evolution after deformation; i.e., a problem relevant to the commercial hot rolling of steel. For this purpose, modified measurement procedures were established to obtain information on recrystallization, recrystallized grain size and austenite grain growth. This approach offers insight into austenite grain growth after recrystallization that is considered in the framework of phenomenological microstructure evolution models<sup>\*</sup>.

# 4.2 Experimental methodology

Cylindrical samples (15mm in length and 10mm diameter) were machined from the current steel for axisymmetric compression tests using a Gleeble 3500 thermomechanical simulator equipped with a laser-ultrasonic system. The surfaces of the samples were machined to a smooth finish to minimize the effect of surface roughness on ultrasound attenuation. During the tests, a vacuum of approximately 7Pa (50millitorr) was maintained in the Gleeble chamber. A thermocouple was spot-welded to the centre of the sample to control the sample temperature; a K type thermocouple was employed for soaking the sample at 1000°C, whereas a R type thermocouple was used for higher soaking temperatures. Temperature gradients were evaluated with a second thermocouple placed near the end of the sample for some initial tests and were found to be in the order

<sup>\*</sup> A list of symbols used in the chapter in presented in appendix 3.

of 5°C. To reduce friction, graphite foil in combination with nickel paste was used as lubricant. The test matrix is shown in Table 4.1. The initial soaking conditions were selected to achieve a uniform austenite grain structure. Significant abnormal grain growth had been observed for the present steel during soaking at temperatures between 1100-1200°C (Chapter 2). Thus, these temperatures were avoided for the selection of initial soaking conditions. A heating rate of  $5^{\circ}Cs^{-1}$  was employed to heat the sample up to the soaking temperature. The sample was soaked there for 120s followed by cooling at  $10^{\circ}Cs^{-1}$  to the deformation temperature. After holding for 5s at the designated deformation temperature, the sample was compressed with applied strain and strain rates, as indicated in Table 4.1. After deformation, the sample was held at the deformation temperature for 10min before being rapidly cooled by He quench to room temperature. The fast cooling step was important to preserve the similar grain structure at the room temperature to that of operating temperature (i.e. the grain size at the operating and room temperature remains same). The current steel chemistry is given in page 18.

Soaking Temperature [°C]	Deformation temperature [°C]	Deformation	Strain rate
1250		<u></u>	10
	1000	0.2	1.0
		0.3	0.1
			1.0
		0.5	1.0
	1050		1.0
	1100	0.3	
	1150		
1100		0.3	1.0
1050	1000		
1000			

**Table 4.1**: Experimental matrix for the laser-ultrasonic tests.

The ultrasonic measurements were conducted from the beginning of deformation to the end of the experiment at room temperature. The primary reason to carry out ultrasonic measurements at the room temperature was to acquire a reference wave response on the same sample (see Chapter 2). The laser generating and detecting spots were set-up in such a way that after deformation, the thermocouple, generating laser and detecting laser remained on the same vertical plane. A schematic diagram of the

experimental set-up is shown in Figure 4.1. To generate the ultrasonic wave, a short (5ns), energetic (150mJ), green (532nm) light pulse from a frequency-doubled Nd:YAG laser was employed. This light pulse generates a wideband ultrasound pulse by ablating a thin surface layer. The laser interferometer employs a longer pulse Nd:YAG laser operating at 1064nm (infrared), with a pulse duration of 50us and pulse energy of approximately 70mJ. The detection laser beam was focused on the sample into a uniform disk approximately 2mm in diameter, i.e. roughly the same diameter as the source laser spot that was carefully aligned prior to the test on the opposite side of the cylindrical sample by monitoring the propagation delay of the ultrasound pulse. A confocal Fabry-Perot interferometer operating in reflection and with a detection bandwidth of 75MHz was employed. The acquisition rate was initially set to 10Hz to capture the rapid microstructure variations following the deformation, and gradually lowered to about 0.2Hz during the experiment to avoid any possible depression mark on the sample which could have occurred if an excessively large number of measurements had been taken. Although, ultrasonic measurements were carried out during and after deformation, the grain size evolution analysis was conducted when the piston stopped moving forward, i.e., the first time point was defined to occur as t=0.05s after the piston stopped. A smoothing function (10 point adjacent average method) was applied to the experimental data. The measured grain size data was multiplied by a factor of 1.2 to convert the EOAD grain size values to volumetric grain size values [24] that is required for modelling analysis.



Figure 4.1 - Schematic diagram of laser-ultrasonic experimental set-up inside Gleeble 3500.

### 4.3 Laser-Ultrasonics

### 4.3.1 Measurement geometry

Prior measurements of austenitic grain size were done either on sheet and plate samples [19, 25] or on tubes [14]. The ultrasound pulses were simple pressure pulses (longitudinal waves), reflected back and forth between the two free surfaces. However, the current study employs samples of cylindrical geometry that complicate measurements. When ultrasound waves are excited with a localized pulsed excitation, multiple acoustic waves propagate: a pressure pulse across the diameter of the cylinder, a surface wave around the circumference of the cylinder, and pressure pulses traveling in various directions that are reflected and mode converted to shear pulses by the curved surfaces of the cylinder [26-27]. This can lead to multiple pulses that are difficult to identify, or worse, that are partly superimposed.

The simplest possible geometry and propagation path was to locate the excitation and detection of the acoustic pulses on the opposite sides, across the diagonal of the cylinder. The acoustic pulse to be measured is simply the first pulse to travel across the diagonal. This pressure pulse is easy to identify because it has a high amplitude, the highest velocity (as compared to other waves), and the shortest path. In addition, the present work involves small enough generation and detection spots of the ultrasound (an area of less than 2mm diameter), so that they nearly behave as point-like transducers having smooth and well-behaved ultrasound radiation patterns.

#### 4.3.2 Calibration validation

As presented in sections 2.3 and 2.5, a previously developed calibration procedure based on the ultrasonic experimental results on C-Mn and microalloyed steels [14] has been employed successfully to monitor the austenite grain growth for the current steel using a simple rectangular strip geometry. Figure 4.2 shows the validation results for a austenite temperature of 1050°C, i.e. where the sample has a cylindrical geometry (the analysis procedure adopted for the present case is same as was described in section 2.4). The metallographic data and the ultrasonic grain size results are presented in the form of EQAD. The metallographic data were taken from water quenched rectangular austenite grain growth samples as mentioned previously (Chapter 2). As can be seen from Figure 4.2, these ultrasonic results (solid and dashed lines) are in good agreement with metallographic data confirming that the employed experimental procedure accounts adequately for the sample geometry. Although some scatter is observed in the ultrasonic results, the grain size evolution as measured by laser-ultrasonics shows reproducibility within the maximum error margin of approximately 25%.



**Figure 4.2** - Austenite grain size measured on the quenched samples by metallography (symbols) and in-situ at 1050°C by laser-ultrasonics (lines). Grain sizes are in EQAD.

### 4.4 **Results and modelling**

### 4.4.1 General observations

With the previously developed calibration procedure being validated, the measured attenuation data for the deformation tests can be represented in terms of the austenite grain size. Figure 4.3 presents the ultrasonic grain size evolution for various conditions in terms of temperature, deformation strain, initial grain size, and strain rate. The results of the individual measurements show three distinct stages:

i) Initially, the grain size remains almost constant for some time after deformation (varying from approximately 1s to 10s depending on the experimental condition). This suggests that this initial stage represents the time interval before the onset of recrystallization where recovery is the dominant softening process. The times for 5% recrystallization as predicted by the independently developed softening model (see

Chapter 3) are indicated by open symbols in Figure 4.3 and agree well with the end of the initial regime as measured by laser-ultrasonics.

ii) The second regime exhibits a marked decrease of grain size and coincides with recrystallization during this period. New grains begin to appear and in the process the average grain size becomes smaller. The time for 95% recrystallization predicted by the softening model (see Chapter 3) is indicated in Figure 4.3 by open symbols. These times are in agreement with the position of the minimum grain size measured by laser-ultrasonics at the end of this second period for 9 out of 10 deformation conditions as shown in Figure 4.3 (the one exception is for an applied strain rate of  $0.1s^{-1}$ , see Figure 4.3(c)). Thus, the minimum grain size is considered as the recrystallized grain size.

iii) In the final stage, the grain size increases for all deformation situations. This observation is consistent with grain growth after the completion of recrystallization.

#### 4.4.2 Initial grain size

The initial grain size results for the reheating at 1250°C was evaluated by averaging the grain size values measured by ultrasonics at the first measurement time point, i.e. when t=0.05s (see the initial grain size value at Figure 4.3); the result is shown in Table 4.2. As can be observed from Figures 4.3(a), 4.3(b) and 4.3(c), for reheating at 1250°C, there is a significant variation in the initial volumetric austenite grain size values (from 320µm to 180µm). Thus, an average initial grain size value of 246µm is determined for reheating at 1250°C (the maximum experimental deviation from the average value is approximately 25%). For each of the other reheating temperatures, i.e. for 1000, 1050 and 1100°C, only one experimental grain size value was measured (Figure 4.3). Therefore for the latter reheating conditions, the measured austenite grain size values are compared with the previously measured austenite grain sizes by laser-ultrasonics for the same steel grade (Figure 2.8 of Chapter 2). Both results agree with each other considering a maximum experimental deviation of approximately 25% from the average grain size value. Thus, initial volumetric austenite grain size values as measured in the present investigation, i.e. 62, 20 and 17µm for soaking temperatures of 1100, 1050, and 1000°C, respectively, are adopted for further analysis. The initial austenite grain size values are shown in Table 4.2.



(c)

(d)

**Figure 4.3** - Grain size evolution after deformation as measured by ultrasonics: (a) effect of deformation temperature, (b) effect of deformation strain, (c) effect of applied strain rate and (d) effect of initial grain size. Symbols in the graph represent the time to achieve 5% and 95% recrystallization of the present steel as predicted by the softening model (in Chapter 3). Grain sizes are presented in their volumetric values.

Soaking temperature (°C)	Austenite grain size (present study, µm)		
	EQAD	Volumetric	
1000	14	17	
1050	17	20	
1100	52	62	
1250	205	246	

Table 4.2: Adopted volumetric grain size values for modelling analysis.

#### 4.4.3 Recrystallized grain size

Figure 4.4 shows the volumetric recrystallized grain size values obtained from ultrasonic measurements. For a given deformation temperature, such as at 1000°C, the recrystallized grain size decreases with applied strain and increases with initial austenite grain size. Further, there is little effect of strain rate on recrystallized grain size. These trends are consistent with those reported previously based on metallographic analysis [6]. The situation is more complex when the variation with deformation temperature is considered. As shown in Figure 4.4(a), the recrystallized grain size exhibits minimal changes when the deformation temperature is increased from 1000°C to 1100°C, whereas the grain size increases significantly at higher deformation temperature (1150°C). As shown in Chapter 2, significant grain growth occurs at temperatures of 1150°C and above. Thus, one possible explanation for the significantly higher recrystallized grain size values at 1150°C is that recrystallization and substantial grain growth may overlap at these higher temperatures resulting in a significantly larger recrystallized grain size value when the material achieves the fully recrystallized state. This complex behaviour of the temperature dependence of the recrystallized grain size reflects the variety of relationships proposed in the literature that show either a temperature-independent or a weakly temperature-dependent recrystallized grain size [6, 28].

Frequently, an empirical relationship is employed to describe the recrystallized grain size  $(D_{rex})$  as a function of initial grain size  $(D_{\theta})$ , applied strain ( $\varepsilon$ ) and deformation temperature  $(T_{def})$  [6, 28-29]:

$$D_{rex} = \Lambda D_0^q \varepsilon^{-p} \exp(-\frac{Q_{grx}}{k_B T_{def}})$$
(4.1)

where  $\Lambda$ , p, q and  $Q_{grx}$  are adjustable parameters. In the present analysis  $Q_{grx}=0$  (i.e. the recrystallized grain size is independent of temperature) for  $T_{def} \leq 1100^{\circ}$ C and  $Q_{grx}=165$ kJmol<sup>-1</sup> for  $T_{def} \geq 1100^{\circ}$ C are adopted together with  $\Lambda=0.39\mu$ m<sup>1-p</sup> for  $T_{def} \leq 1100^{\circ}$ C and  $\Lambda=6.2 \times 10^{5} \mu$ m<sup>1-p</sup> for  $T_{def} > 1100^{\circ}$ C. In addition, p=0.82, q=0.84 are determined from the present analysis. For comparison, the model descriptions are included in Figure 4.4 and show satisfactory agreement with the experimental observation.



**Figure 4.4** - Dependence of the recrystallized grain size values as measured by laserultrasonics on (a) deformation temperature  $(T_{def})$ , (b) deformation strain  $(\varepsilon)$ , (c) strain rate  $(\dot{\varepsilon})$  and (d) initial austenite grain size  $(D_0)$ . Solid lines show model results. Grains sizes are presented in their volumetric values.

### 4.4.4 Grain growth after recrystallization

Figure 4.5 shows the grain growth behaviour after recrystallization when the sample was held at the deformation temperature for approximately 10min. The recrystallized values obtained from Figure 4.3 (also shown in Figure 4.4) are considered the initial grain size values in Figure 4.5 for each grain growth condition. Little grain growth is observed at lower temperatures, such as 1000°C and 1050°C (109 $\mu$ m to 165 $\mu$ m

at 1000°C and 100µm to 149µm at 1050°C within a time span of 10min). When the temperature is increased to 1150°C, substantial grain growth occurs, i.e. the grains grow from 154µm to 344µm within 5min. These grain growth trends are consistent with the austenite grain growth observations made on the current steel during reheating (see Chapter 2). The comparatively limited grain growth at lower temperatures is probably due to particle pinning and/or solute drag effects whereas these effects are much weaker at higher temperatures. Further, as observed, the applied strain and strain rate have little effect on subsequent grain growth. However, the present conditions are quite different from those of conventional grain growth kinetics, a more rigorous analysis of the data was performed using established model concepts for grain growth.

Burke and Turnbull [30-31] developed the original grain growth model by assuming that the pressure on the boundary arises from the curvature of the boundary. This leads to a parabolic grain growth law. However, many observations of grain growth, even in ultra-pure materials, suggest deviations from the parabolic growth behaviour such that frequently an empirical power law was proposed for grain growth:

$$D_r^m = D_0^m + Kt \tag{4.2}$$

where,  $D_0$  is the initial grain size, K is a temperature-dependent material parameter, t denotes time, and m is the grain growth exponent that is usually larger than 2. The present grain growth data suggests grain growth exponents in the range of 3.5-5. These deviations from the ideal parabolic grain growth behaviour have been attributed to the effects from solute drag and particle pinning [31]. Both aspects are important for the present steel as it contains Nb and Mo, which are strong carbide formers and, in addition, exhibit significant solute drag effects [3, 6-8, 29]. De-convolution of both mechanisms is challenging. Solute drag is usually described with a reduced effective mobility that, however, would not lead to deviations from m=2. To account for m>2 due to solute drag, a transition from a high to a low velocity branch during grain growth would have to be considered. In contrast, introducing a pinning factor is a well established procedure to explain m>2 [31]. Thus, here an approach for grain growth modelling that includes a pinning term (P) is selected. The generalized grain growth equation is then:

$$\frac{dD_{\gamma}}{dt} = M_{eff} \left( \frac{\alpha_{gg} \gamma_{gb}}{D_{\gamma}} - P \right)$$
(4.3)

where,  $D_{\gamma}$  is the average austenite grain size,  $M_{eff}$  is the effective grain boundary mobility,  $\alpha_{gg}$  is a geometrical constant and  $\gamma_{gb}$  is the grain boundary energy. For the present calculations,  $\alpha_{gg} = 0.5$  and a grain boundary energy value of 0.75Jm<sup>-2</sup> are used [30-31].



**Figure 4.5** - Grain growth behaviour measured by laser-ultrasonics (symbols) and model calculations (solid lines): (a) effect of deformation temperature, (b) effect of deformation strain, (c) effect of applied strain rate and (d) effect of initial grain size. Grain sizes are presented in their volumetric values.

The grain boundary mobility is expressed as:

$$M_{eff} = M_0 \exp\left(\frac{-Q_{eff}^M}{RT}\right)$$
(4.4)

where,  $M_0$  is the pre-exponential factor,  $Q_{eff}^M$  is an effective grain boundary activation energy, and R is the universal gas constant (8.3145Jmol<sup>-1</sup>K<sup>-1</sup>). In the present analysis,  $Q_{eff}^M = 337$ kJmol<sup>-1</sup> is adopted from the softening study as described in Chapter 3. Then, the pinning term P and  $M_0$  are employed as fitting parameters. Using this approach,  $M_0 = 5.3 \times 10^9 \text{m}^4 \text{J}^{-1} \text{s}^{-1}$  is determined and the values obtained for P are listed in Table 4.3. Figure 4.5 compares the model description for grain growth with the experimental data. The present model reflects the grain growth behaviour reasonably well when  $M_0$  is fixed and P is considered to depend on each grain growth situation.

Initial grain	Deformation	Deformation	Strain rate	Pinning
size [µm]	temperature [°C]	strain	[s <sup>-1</sup> ]	parameter [Jm <sup>-3</sup> ]
246	1000	0.2	1.0	$1.4 \times 10^{-3}$
		0.3	0.1	$2.0 \times 10^{-3}$
			1.0	$1.7 \times 10^{-3}$
		0.5	1.0	$3.9 \times 10^{-3}$
	1050	0.3	1.0	$2.4 \times 10^{-3}$
	1100			1.6x10 <sup>-3</sup>
	1150			$1.1 \times 10^{-3}$
62		0.3	1.0	9.5x10 <sup>-3</sup>
20	1000			$1.6 \times 10^{-2}$
17				$1.5 \times 10^{-2}$

**Table 4.3**: Values for fit pinning parameter (*P*) for the grain growth model.

### 4.5 Discussion

A new laser-ultrasonic technique was developed in the present research to measure *in-situ* austenite grain size evolution after deformation in a low carbon microalloyed steel. The present method establishes laser-ultrasonics as a grain size measurement technique, when the sample geometry is complex and/or changed due to plastic deformation. The accuracy of these measurements is similar to those made using simpler specimen geometries, i.e. tube or sheet [14, Chapter 2]. A previously developed calibration (developed on C-Mn and HLSA steels) has been adopted (without any additional corrections being required) either for the cylindrical geometry or for the

different steel chemistry. This further demonstrates the versatility of laser-ultrasonics as a continuous and non-destructive bulk measurement technique that can be employed at high temperature.

The current study suggests that attenuation measurements can be used for the measurement of i) grain size evolution and ii) the start (5%) and finish (95%) times of recrystallization, thus providing a new experimental tool to observe recrystallization and grain growth following plastic deformation. These attenuation measurements provide additional and different information than the laser-ultrasonic measurement of recrystallization using the velocity. The sound velocity is sensitive to the texture of the material and texture changes during recrystallization can be used to record the transformation kinetics [21-23]. The present study is an example where very little texture change is associated with recrystallization and the velocity data showed only small variations. On the other hand, attenuation measurements of recrystallization work well provided that there are measurable changes of the grain size during recrystallization. In particular, for hot rolling of steels, it has been observed that a limit of recrystallized grain sizes can be attained after repeated grain refinement in multi-stage deformation processes [6]. In this special case, the initial and recrystallized grain sizes are similar and the application of attenuation-based laser-ultrasonic measurements of recrystallization may not work. Further studies will be required to explore the potential of attenuation to also provide detailed information on the recrystallization kinetics. During recrystallization, a heterogeneous, i.e. partially recrystallized, microstructure is present. As shown in Chapter 2, during abnormal grain growth stages with a bimodal grain size distribution (a combination of large grains surrounded by cluster of smaller grains), the laser-ultrasonic result is dominated by the large grains in the distribution. Therefore, there may exist significant challenges in developing calibration procedures that permit to determine the recrystallized fraction between the recrystallization start and finish times using attenuation data.

An advantage that is further illustrated by the present study is that the ultrasonic technique can measure the austenite grain size evolution in low carbon steels, where it is very challenging to reveal the austenite microstructure using traditional metallography. In particular for recrystallized grain sizes, metallographic data are usually restricted to

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carbon levels of approximately 0.1wt% or higher as the required specimen geometry for deformation necessitates sufficient hardenability to succeed with metallographic techniques. Further, laser-ultrasonics may enable to measure the evolution of the austenite microstructure in interstitial free and other ultra low carbon steels that so far have remained elusive for quantitative austenite grain size measurements.

The absolute accuracy of the laser-ultrasonic grain size measurements has been determined to be approximately 25%. Precision, i.e. the ability to measure small changes in grain size during an experiment, is much better, approximately 5%. At first sight, the absolute accuracy may appear low. However it can more than be compensated by the general advantages of an in-situ technique and by the high precision. Further, the accuracy limit of metallographic investigations of austenite grain size is approximately 10% when the structure can be revealed with high quality. This accuracy drops rapidly with decreasing hardenability of the steels, i.e. with decreasing carbon content.

Evaluating the measurement accuracy for the initial grain size of the present study, there are a number of factors that may have contributed to increasing the spread of the data. For the reheating condition of 1250°C, the data (Figure 4.3) shows a variation of initial grain size immediately after deformation from 326µm to 182µm. This deviation could be related to the different operating conditions. Smaller grain sizes are observed when the recrystallization starts within a second after deformation at higher temperatures, such as at 1100°C (Figure 4.3(a)). Further, there is a general trend of decreasing grain sizes with increasing applied strain (Figure 4.3(b)). The possible reason could be the effect of deformation on the change of sample dimension (geometry effect) or the change in grain shape which may produce a second order effect on the ultrasonic measurement of grain size. Also, it must be noted that the ultrasonic measurement of grain size utilizes a calibration that has only been validated with recrystallized microstructures showing uniform (lognormal) distributions, whereas here, the measured initial grain sizes were made on deformed microstructures. Further, the possibility of grain size gradients on the measurement plane cannot be ruled out. For example, the strain gradient in the compression sample has been estimated to be approximately 7-10% from edge to centre. However, within the scatter that the calibration currently being used provides [14], it is reasonable to assume that the grain size gradient is a second order effect. This has been

confirmed by examination of recrystallized samples for higher carbon grade steel after compression testing using similar specimen geometry, where it was possible to metallographically reveal the austenite grain structure after quenching [32]. The structures showed no evidence of a non-uniform grain size distribution. Even so, there may be potential deviations that may be acceptable if one is aware of their possible existence and takes them into account in the analysis. For example, in the present study, laser-ultrasonics could unambiguously determine the transition between recovery, recrystallization, and grain growth regimes (see Figure 4.3). In addition, metallographic studies on quenched samples are time-consuming and only a few measurements are usually made to estimate a material's behavior.

Another important observation of the present research is that laser-ultrasonics provides quantitative insight into in-situ austenite grain growth after recrystallization for conditions that reflect those of industrial hot rolling, i.e. where reheating (soaking) leads to significant dissolution of precipitates. Comparing these soaking conditions with those for the lower reheat temperature where no or partial dissolution takes place, as frequently associated with conventional grain growth tests, permits to critically assess previously adopted descriptions.

In general, the recrystallized grain size is frequently described by a cubic relationship with the initial grain size that is present before the deformation step such that a grain size exponent (p) of 1/3 is assumed in Eq.4.1 [6, 33]. This relationship can be rationalized by assuming that new recrystallized grains form exclusively at grain boundaries and that the nucleation density on grain surface area is independent of grain size. In contrast, the present investigation reveals an apparent p value of 0.84. However, in this context, it is important to realize that the initial austenite grain size has been changed by applying different soaking temperatures and this also affects the density of precipitates. Then, the present finding suggests that different nuclei densities are present for different size distributions of precipitates. For significant dissolution, i.e. soaking at 1250°C, the nuclei density per grain boundary area is significantly lower than when precipitates are present. In the absence of precipitates, once a nucleus is formed it can readily grow along the grain boundary area, thereby consuming other potential nucleation

sites. However, when precipitates are present, they inhibit growth along the grain boundaries such that the additional nuclei can form.

Previously, the softening behaviour of the present steel was analyzed neglecting the effect of precipitates, i.e. using the assumption that nuclei density is independent of the precipitate state (Chapter 3). However, the adjustable parameter to fit the experimental data was the product of nuclei density and grain boundary mobility. This fit parameter appeared to be independent of the degree of particle dissolution (Chapter 3). In light of the present study the material behaviour may be interpreted as follows. For complete dissolution a low nuclei density is combined with a large effective mobility whereas for incomplete dissolution a high nuclei density pairs with a rather low effective mobility due to pinning.

This simplified approach to effectively account for pinning effects cannot be extended to describe grain growth following completion of recrystallization. Although the overall magnitude of grain growth could be replicated by introducing an effective mobility, it would also lead to parabolic grain growth behaviour, in contrast to the experimental observations. Thus, a pinning parameter must be considered explicitly to describe grain growth of the recrystallized austenite grain size. In addition, the pinning effects are much more pronounced for grain growth than for recrystallization since the driving pressures for grain growth are about three orders of magnitude lower than for recrystallization [31].

The pinning parameter to describe grain growth depends on reheat temperature and deformation condition. Although the justification of the absolute values of P is beyond the scope of this study, nevertheless their trends show a sensible pattern. As presented in Table 4.3, the pinning parameter falls into two regions depending on the reheating conditions, i.e. P is in the order of  $10^{-3}$ Jm<sup>-3</sup> for reheating at 1250°C and in the order of  $10^{-2}$ Jm<sup>-3</sup> for lower soaking temperatures. The dissolution of previously present precipitates is either negligible or partial at lower reheating temperatures (1000-1100°C), whereas almost complete dissolution of precipitates is expected for reheating at 1250°C. Thus, there is significantly more particle pinning present for the lower reheat conditions. From an industrial perspective, the studies for the higher reheat temperature are of particular interest, as these conditions are closer to industrial practices. Thus, for hot strip rolling, reheating at 1250°C is of relevance and then  $P=2x10^{-3}$ Jm<sup>-3</sup> can be adopted as a representative value to describe grain growth during and after rough rolling. Also, for this reheat condition; *P* varies systematically as a function of the process conditions. In particular, a marked increase of the pinning parameter is found when the applied strain is increased from 0.2 to 0.5. The increase in *P* with strain may be an indicator of strain-induced precipitation [6-7].

In addition to the pinning parameter, the grain boundary mobility was employed as a fitting parameter to describe grain growth. This implicitly reflects that both particle pinning and solute drag are affecting grain growth. To deconvolute both effects, information on the actual particle size distribution is required and could not be obtained with the current experimental approach. Extensive electron microscopy studies would be needed to quantify particle size distributions.

# 4.6 Conclusion

The following conclusions are made from the above experimental and modelling study.

- A comparatively new technique (laser-ultrasonics) was applied successfully to the in-situ study of grain size evolution following plastic deformation at austenitic temperatures relevant to hot strip rolling.
- The grain sizes measured by ultrasonics are accurate to within 20-25%. This accuracy is comparable to that of standard metallographic procedures when available. Moreover, grain size variations as small as about 5% arising from thermal processing can be detected with laser-ultrasonics.
- Grain size evolution data measured by ultrasonics can be employed to determine recrystallization start and finish times in-situ.
- The in-situ recrystallized grain size measurements depend weakly on deformation temperature below grain coarsening temperatures; the recrystallized grain size increases significantly with deformation temperatures when the deformation temperature is high enough so that substantial grain growth overlaps with recrystallization.

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# **CHAPTER 5: PHASE TRANSFORMATION**\*

### 5.1 Introduction

Austenite decomposition during cooling on the run-out table is of critical importance considering its influence on the final microstructure and mechanical properties of steel. Austenite decomposition in steel has been investigated extensively in the last 50-60 years and a good summary on experimental observations of austenite decomposition was provided by Zhao and Notis [1]. Pure iron has a face-centred cubic (fcc) structure in the temperature range of 912°C and 1392°C, which is known as austenite. Lowering the temperature below 912°C leads to the formation of the less dense body centred cubic (bcc) structure. Depending on the cooling rate, initial austenite grain size, deformation condition and alloy addition, various transformation products can form through different types of transformation mechanisms (as an example, diffusional and displacive transformation mechanisms) [2]. Polygonal ferrite (PF), acicular ferrite (AF), pearlite (P), bainite (B) and lath martensite (LM) are the main transformed products reported for hot rolled low C and microalloyed steels after austenite decomposition.

Extensive investigation on allotriomorph ferrite nucleation has demonstrated that ferrite nucleation occurs primarily at the prior austenite grain boundaries; the prominent ferrite nucleation sites being the grain corners followed by grain edges and grain surface [1-5]. Classical nucleation theory for heterogeneous nucleation has been used by many researchers to describe ferrite nucleation, whereas the growth of polygonal ferrite has frequently been described by the JMAK approach [6-8] assuming additivity [9-15] for various C-Mn and micro-alloyed steels. In addition to the JMAK approaches, numerous studies were devoted to describe ferrite growth from a fundamental point of view. Generally the austenite to polygonal ferrite transformation has been characterized as a diffusional transformation. During ferrite formation, as the transformation front moves, it rejects carbon to the parent austenite. The long-range diffusion of carbon within austenite was then considered as the rate-controlling step for the transformation [1, 16-17].

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However, in a low C steel, carbon diffusion alone cannot account for the transformation kinetics. Hence the lattice transformation from austenite (fcc) to ferrite (bcc) structure at the interface must be considered to describe the kinetics of transformation [1, 18-19]. Recently a more balanced approach [20-21] has been proposed to describe ferrite growth. This approach is termed as the mixed mode model and according to this model the ferrite growth shifts from interface controlled growth in the beginning to diffusional controlled growth at later stages.

The ferrite transformation is accelerated by the presence of finer and pancaked austenite grains, whereas, small amounts of solutes (Mn, Nb, V etc.) tend to delay the transformation to lower transformation temperatures and the morphology of ferrite changes from polygonal to acicular structure [22-24]. However with a combination of increasing alloying content (i.e. Nb) and significant retained strain that is expected during rolling, the drag force may reduce due to the precipitation of microalloyed carbide/carbonitrides [25]. Once the ferrite formation is complete, the final ferrite grain size can be described as a function of prior austenite grain size, cooling rate and retained strain, where higher cooling rate, higher strain and finer, deformed austenite grain sizes are favoured for ferrite grain refinement [10, 26-28].

Hot strip rolling of modern micro-alloyed AHSS is expected to produce a final microstructure that consists of primarily fine ferrite, bainite and martensite after austenite decomposition [29-30]. The start temperature for bainite is generally presented as chemistry dependent, i.e. the bainite formation shifts to lower temperature with increasing carbon content or carbon equivalent [31-32]. Rees et al. [33] investigated the effect of alloying elements on the bainite transformation temperature. Their work on low carbon Nb microalloyed steel with varying Nb contents (0.001- 0.035wt%), showed that similar to polygonal ferrite, bainite transformation is retarded with increasing Nb content in solution. Their study [33] also revealed that the initial austenite grain size has negligible effects on the bainite formation. The growth of bainite is still a subject of immense debate. A number of theories have been proposed for bainite formation kinetics and they are mainly classified as, either diffusional or displacive approaches [31, 34-38]. In addition to diffusive and displacive approaches, the JMAK approach has also been employed to describe the bainite formation kinetics [38-40].

Finally the formation of martensite is characterized as a diffusionless transformation, where austenite with a fcc crystal structure changes to martensite with a bct (body centered tetragonal) crystal structure. The formation of martensite occurs when the steel is cooled below the martensite start temperature ( $M_S$ ) and the transformation can continue up to 150K below  $M_S$  [41]. Over the years, various empirical equations have been proposed to predict the  $M_S$  temperature as a function of the chemical composition of steel or the carbon concentration of remaining austenite [3, 42]. The martensite volume fraction is widely accepted to be a function of temperature below  $M_S$  [43]. The formation of martensite occurs at the speed of sound, i.e. the detailed kinetics is not of practical significance.

Recently, with the arrival of new advanced steel products consisting of a multiphase microstructure, it is important to develop modelling approaches that can describe the simultaneous formation of more than one transformation product. Relatively, few studies have been proposed to capture the overall decomposition of austenite to various transformed products such as ferrite, pearlite, bainite and martensite under industrial cooling conditions. Separate work by Umemoto [44], Bhadeshia [45], Minote et al. [38], Samoilov [46] and Liu et al. [47], used the JMAK law to describe austenite decomposition to ferrite and pearlite and the required modifications were made to capture the simultaneous reactions, i.e. either diffusional or displacive approaches were proposed to describe the bainite formation. Most of these models had originally been developed for the isothermal transformation condition, but then extended to the continuous cooling condition.

The objective of the present work is to describe the phase transformation behaviour of the present steel under continuous cooling conditions in the cooling rates regime of  $1-100^{\circ}$ Cs<sup>-1</sup> with various initial austenite grain structures. To accommodate the effect of retained strain in austenite, the samples were pre-deformed below the norrecrystallization temperature up to a strain of 0.6. THERMO-CALC software using Fe2000 database was applied to determine the Ae<sub>3</sub> temperature of the steel, which is  $832^{\circ}$ C<sup>\*</sup>.

<sup>\*</sup> A list of symbols used in this chapter is presented in appendix 4.

# 5.2 Experimental methodology

Continuous cooling tests (CCT) were conducted to investigate the effects of initial grain size, retained strain and cooling rate on austenite decomposition behaviour. Details of the test matrix are shown in Table 5.1. To conduct CCT tests without prior deformation, tubular specimens of 20mm in length with an outer diameter of 8mm and a wall thickness of 1mm were employed. Using the Gleeble 3500 thermomechanical simulator, dilatometer measurements were carried out to measure the diameter change of the sample associated with phase transformation. During an entire test, the temperature was controlled by spot welding a thermocouple to the outer surface of the samples. K type thermocouples were used for soaking temperature  $\leq 1000^{\circ}$ C, whereas S type thermocouples were used for higher reheating temperatures. All the experiments were conducted under a vacuum of  $10^{-6}$  militorr (1.3x10<sup>-4</sup>Pa). To investigate the effect of retained strain on the austenite decomposition kinetics, specimens 6mm in diameter and 10mm in gauge length were deformed in axisymmetric compression prior to cooling. A single stage deformation was executed to a true strain of up to 0.6 at 875°C (applied strain rate ( $\dot{\varepsilon}$ ) of 1.0s<sup>-1</sup>), i.e. under the no recrystallization condition, as determined from the softening study (Chapter 3). A variety of controlled cooling conditions were applied to cool the sample from the reheating temperature; to achieve higher cooling rates (15-100°Cs<sup>-1</sup>), controlled gas (He) quenching has been used. Quantification of the phase transformation was done based on the difference between the molar volume of the parent and product phases. As an example, a transformation situation from austenite to polygonal ferrite formation can be considered. Austenite having a fcc crystal structure and the product phase ferrite with a bcc crystal structure, the sample volume increases during phase transformation from austenite to ferrite. This phenomenon is recorded and presented as the measured dilation vs. transformation temperature (T). A schematic illustration of determining the fraction transformed during continuous cooling tests is given in Figure 5.1. Applying lever rule [48] the total fraction transformed (X) can be written as:

$$X = \frac{d_{m}(T) - d_{Aus}(T)}{d_{Pro}(T) - d_{Aus}(T)}$$
(5.1)

where,  $d_m(T)$  is the measured dilation as a function of temperature,  $d_{Aus}(T) = A_1 + S_{Aus}T$ is the extrapolated dilation from the parent region (for the present study austenite is the parent phase) and  $d_{Pro}(T) = A_2 + S_{Pro}T$  is the extrapolated dilation from the product regime.  $A_1$  and  $A_2$  are constants, whereas  $S_{Aus}$  and  $S_{Pro}$  are the thermal expansion coefficients of austenite and product phases, respectively. From the experimental curve, the fraction transformed can be estimated.

Tuble 3.1. Experimental matrix for the continuous coomig tests.					
Soaking temperature	Austenite grain size	Deformation	Cooling rate		
(°C)	(μm)	strain	(°Cs <sup>-1</sup> )		
1250	246	0.4-0.6	1-40		
1100	62				
1050	20	0.0-0.6	1-100		
950	8				

Table 5.1: Experimental matrix for the continuous cooling tests.

The dilatometer measurements were supplemented with the metallographic analysis using 2% nital etching to reveal the final microstructures. Micrographs to measure ferrite volume fraction and ferrite grain size were taken using optical microscopy and scanning electron microscopy (SEM). Optical and SEM metallography were conducted on transformed samples etched with natal, to measure the polygonal ferrite fractions. Martensite + retained austenite volume fractions were measured from optical/SEM micrographs, which were pre-etched by 2% nital followed by LePera etchant [49]. ASTM E562-89, point counting method was employed to measure the polygonal ferrite fraction, whereas an image analyzer was used to measure martensite + retained austenite (MA) volume fraction. The method described above could not separate the martensite fractions from the retained austenite fractions. Once the ferrite and MA volume fraction are determined, the rest is considered as bainite. Polygonal ferrite grain sizes were measured on some selected samples, where at least 15-20% polygonal ferrite fractions were obtained in the transformed microstructure. The individual equivalent area diameter (EQAD) of each ferrite grain was first measured using an image analyzer and the average EQAD was calculated for approximately 400-500 grains for each sample. Hardness measurements were conducted on the transformed samples employing Vickers micro-hardness tester with a load of 1000gm. The fields of measurements were chosen

randomly and care was taken to avoid the sample surface which was partially affected by surface oxidation and decarburization.



**Figure 5.1** – Typical dilation curve obtained during continuous cooling transformation tests during austenite decomposition.

# 5.3 Results

### 5.3.1 General transformation trend

An example of the fraction transformed curve is presented in Figure 5.2. To present the fraction transformed curve, the metallographically measured MA fraction is deducted from the calculated total transformed fraction. As presented in Figure 5.2, the transformation curve exhibits three stages in the austenite decomposition process; (a) relatively slow transformation rates at the beginning of transformation (nucleation of product phases at the beginning of transformation), (b) fast austenite decomposition behaviour (growth of newly nucleated grains) and (c) finally a slower transformation kinetics at the later stage of transformation (impingement of growing grains with the neighbouring grains). The transformed microstructures consist of a combination of polygonal ferrite, acicular ferrite/bainite and martensite. Although four different austenite grain sizes (ranging from 8 to  $246\mu$ m) are considered for the present study, only the grain sizes relevant to finishing regime (i.e.  $8-62\mu$ m) are discussed in the following sections. In addition, the present chapter will stress on the transformed conditions involving retained

strain in the austenite region as heavily deformed pancaked austenite structure is expected at the end of finish rolling (see Chapter 3).



Figure 5.2 – Example of a fraction transformed curve as a function of transformation temperature.

#### 5.3.2 Effect of cooling rate

The effect of cooling rate ( $\phi$ ) on the continuous cooling austenite decomposition kinetics of the present steel is illustrated for an initial austenite grain size of 20µm for non-deformed and deformed conditions (Figure 5.3). As can be seen from Figure 5.3, with increasing cooling rate, transformation shifts towards lower temperature. This observation can be explained as follows. With increasing cooling rates, the ferrite nucleation gets suppressed and the transformation shifts to lower temperature. However, when the transformation occurs from the work hardened austenite ( $\varepsilon$ : 0.6), the effect of cooling rate, on the austenite decomposition decreases.

### 5.3.3 Effect of initial austenite grain size

The initial austenite grain size  $(D_{\gamma})$  has a strong effect on continuous cooling transformation kinetics as shown in Figure 5.4 for non-deformed and deformed condition of austenite. As can be seen from Figure 5.4, with decreasing austenite grain size, austenite decomposition shifts to higher transformation temperatures. With decreasing

austenite grain size, the grain boundary area per unit volume increases which acts as the potential nucleation site. Therefore nucleation is promoted and the transformation shifts to higher temperatures. It can be also observed from Figure 5.4 (b) that the effect of initial austenite grain size decreases significantly when the transformation starts from pancaked austenite ( $\varepsilon$ : 0.6). A similar trend is observed regarding the effect of initial austenite grain size with increasing applied cooling rate.



**Figure 5.3** – Selected CCT examples showing the effect of cooling rate ( $\phi$ ) for austenite grain size ( $D_{\gamma}$ ) of 20µm: (a) undeformed ( $\varepsilon$ : 0.0) and (b) deformed ( $\varepsilon$ : 0.6) conditions.



**Figure 5.4** – Selected CCT examples showing the effect initial austenite grain size  $(D_{\gamma})$  for an applied cooling rate  $(\phi)$  of 15°Cs<sup>-1</sup>: (a) undeformed austenite ( $\varepsilon$ : 0.0) and (b) deformed austenite ( $\varepsilon$ : 0.6).

### 5.3.4 Effect of retained strain

It is observed that austenite decomposition was shifted to higher transformation temperatures for similar initial austenite grain size and cooling rates when the austenite was deformed before the transformation. The result is shown in Figure 5.5 for the various initial austenite grain sizes (8, 20 and 62 $\mu$ m). With prior deformation, the grain boundary area increases that promote potential nucleation sites for transformed phases. As an example, for an austenite grain size of 20 $\mu$ m, with increasing deformation/retained strain ( $\varepsilon$ ) from 0.0 to 0.6, the transformation shifts to higher temperature for any given cooling rate.



**Figure 5.5** – Selected CCT examples showing the effect of strain ( $\varepsilon$ : 0.0 vs. 0.6): (a) for two different cooling rates ( $\phi$ ) and (b) two different austenite grain sizes ( $D_{\gamma}$ ).

#### 5.3.5 Transformation start temperature

The transformation start temperature  $(T_S)$  has been determined as the temperature for 5% austenite decomposition for all transformation conditions from the dilatometric response during transformation. Similarly transformation finish temperature is considered as the temperature where 95% of austenite is transformed to product phases. As shown in Figures 5.3 to 5.5, the transformation start temperatures increase with decreasing austenite grain sizes and cooling rates and with increasing the amount of retained strain. Experimental result on the relationship between the transformation start temperatures and the austenite grain sizes (20 vs.  $62\mu$ m), continuous cooling rates (1-100°Cs<sup>-1</sup>) and retained strain (0.0 vs. 0.6) is presented in Figure 5.6.



**Figure 5.6** – Transformation start temperatures as a function of cooling rates for different deformation conditions ( $D_{\gamma}$ : 20 vs. 62µm and  $\dot{\varepsilon}$ : 0.0 vs. 0.6).

### 5.3.6 Transformation stasis

One important observation in the transformation results is the formation of transformation stasis associated with the polygonal ferrite formation. During the formation of stasis, the ferrite transformation rate  $\left(\frac{dX_{\alpha}}{dt}\right)$  slowed down and in few cases (such as at  $\phi$ : 1°Cs<sup>-1</sup>) came to a complete growth cessation. At the end of stasis period, the transformation continued with the formation of nonpolygonal structure such as acicular ferrite or bainite. As an example, Figure 5.7 shows the transformation stasis observed in the non-deformed and deformed samples from three initial austenite grain sizes for an applied cooling rate of 1°Cs<sup>-1</sup>. As can be seen from Figure 5.7, during the stasis regime, the austenite to ferrite transformation exhibits a near horizontal plateau regime for approximately 50°C before resuming with the formation of bainite. However, it is important to mention that the observation of significant stasis period was prominent mostly at slower cooling rate (i.e. 1°Cs<sup>-1</sup>).

Observation of transformation stasis on molybdenum (Mo) containing steel has previously been reported [50-52], where the rate of ferrite formation decreases

significantly and the transformation comes almost to a complete halt within a certain temperature range, depending on the Mo and C concentration in the steel (it mostly depends on the Mo to C ratio in austenite). This is also known as incomplete transformation. Recently, it has been suggested that stasis can also be observed in high manganese (Mn) steel [52].



Figure 5.7 – Transformation stasis observed during austenite decomposition.

### 5.3.7 Transformed microstructure

The current steel exhibits predominantly three transformation products, i.e. polygonal ferrite, nonpolygonal ferrite/bainite and martensite + retained austenite. It is observed that the transformed microstructure becomes finer with decreasing initial austenite grain size, increasing cooling rate and retained strain. A predominantly polygonal ferrite microstructure is obtained when the austenite grain size is small and the cooling rate is low; pancaking austenite further promotes polygonal ferrite fraction. With increasing austenite grain size and increasing cooling rate, the austenite decomposition shifts to lower temperature such that the formation of polygonal ferrite is suppressed, resulting in the formation of acicular ferrite/bainite. The effect of initial austenite grain size, retained strain and cooling rate on the product microstructure is shown in Figure 5.8. Overall, Figure 5.8 produces three prominent transformed regions; a) polygonal ferrite (NPF)

structure (white, non elongated structure) and c) dark second phase structure (bainite (B) or MA; MA regions are separated from the other phases present in the final microstructure by using the LePera etched micrographs). The different phases are marked in Figures 5.8(a) and 5.8(d).

A detailed metallography analysis shows a mixture of 40% polygonal ferrite and acicular ferrite/bainite as the secondary transformation product forms, when cooling at 1°Cs<sup>-1</sup> from an equiaxed austenite microstructure (initial austenite grain size of 20µm) (Figure 5.8 (a)). Pancaking this initial microstructure (applied strain of 0.6) increases the polygonal ferrite fraction to approximately 70% (see Figure 5.8(b)) and decreases the ferrite grain size (EQAD) from 8.5 to 7 $\mu$ m. When the cooling rate is increased to 40°Cs<sup>-1</sup> (Figure 5.8(c)), the resulting microstructure is further refined and consists now of a predominantly acicular ferrite/bainite structure. Increasing the initial austenite grain size from 20µm to 62µm decreases the polygonal ferrite fraction to approximately 10% and the transformed microstructure becomes coarser (comparing Figure 5.8(d) with Figure 5.8(a)). Figure 5.8(e) shows the transformed microstructure from an initial austenite grain size of 62µm that was predeformed to a strain of 0.6. As compared to Figure 5.8(d), Figure 5.8(e) exhibits higher polygonal ferrite fraction (approximately 40%); however this value is significantly lower compared to the case where the initial austenite grain size was finer, i.e. 20µm (Figure 5.8(b)). Finally with increasing cooling rate to 40°Cs<sup>-1</sup> (Figure 5.8(f)), the structure becomes finer and polygonal ferrite fraction disappeared. very similar to the structure observed for the finer austenite grain size shown in Figure 5.8(c). This indicates that the transformed microstructures become independent of the initial austenite grain size when transformation happens at higher cooling rate from sufficiently pancaked austenite (here  $\varepsilon=0.6$ ).

Micrographs obtained after nital etching (as an example, Figure 5.8) are analyzed only to measure the polygonal ferrite volume fraction. Metallographic results on the polygonal ferrite fraction as a function of initial austenite grain size, retained strain and applied cooling rate is presented in Figure 5.9.

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(c) (f) **Figure 5.8** – Microstructural evolution during continuous cooling transformation of the present CP steel: (a)  $D_{\gamma}$ : 20µm,  $\varepsilon$ : 0.0,  $\phi$ : 1°Cs<sup>-1</sup>, (b)  $D_{\gamma}$ : 20µm,  $\varepsilon$ : 0.6,  $\phi$ : 1°Cs<sup>-1</sup>, (c)  $D_{\gamma}$ : 20µm,  $\varepsilon$ : 0.6,  $\phi$ : 40°Cs<sup>-1</sup>, (d)  $D_{\gamma}$ : 62µm,  $\varepsilon$ : 0.0,  $\phi$ : 1°Cs<sup>-1</sup>, (e)  $D_{\gamma}$ : 62µm,  $\varepsilon$ : 0.6,  $\phi$ : 1°Cs<sup>-1</sup> and (f)  $D_{\gamma}$ : 62µm,  $\varepsilon$ : 0.6,  $\phi$ : 40°Cs<sup>-1</sup>.


**Figure 5.9** – Metallographically measured polygonal ferrite volume fraction as a function of cooling rate for different deformation conditions ( $D_y$ : 20 vs. 62µm and  $\varepsilon$ : 0.0 vs. 0.6).

Figure 5.10 shows the dilation curves for an initial austenite grain size of  $62\mu m$  that have experienced a cooling rate of 1 and  $15^{\circ}Cs^{-1}$ . A clear deviation from the linearity can be observed in Figure 5.10 as shown by the dashed lines. The point of deviation from linearity can be considered as the beginning of MA phase in the transformed structure. To measure the MA fraction in the final microstructures, LePera etched optical or SEM micrographs are analyzed. As an example, a LePera etched optical micrograph is shown in Figure 5.11, where the white areas (marked by arrows in the figure) are identified as the MA fraction.

Optical and SEM metallographic investigation confirmed that with increasing polygonal ferrite fraction, the MA volume fraction increases marginally. It has been observed that the MA fraction varies from 0.095 to 0.12 depending on the fraction ferrite in the transformed microstructures. The measured MA volume fractions from metallography for some selected transformed samples are presented in Figure 5.12 as a function of polygonal ferrite fractions. As can be seen from Figure 5.12, the measured MA fractions are within the limit of error of the metallographic measurements ( $\pm 0.025$  in absolute value). However in the present analysis, a simple empirical correlation is

established between the MA fraction ( $X_{MA}$ ) and fraction ferrite ( $X_{\alpha}$ ) to calculate the MA fraction for all the continuous cooling conditions.

$$X_{MA} = 0.03X_{\alpha} + 0.095 \tag{5.2}$$

Once the MA fractions are calculated (from Eq.5.2) for each transformation conditions, the fraction transformed curves are normalized by  $(1-X_{MA})$ . Therefore, as presented previously (Figures 5.2-5.5), none of the fraction transformed curves reach a total fraction of 1.



**Figure 5.10** – Dilation curves: (a)  $D_{\gamma}$ : 62µm,  $\varepsilon$ : 0.6,  $\phi$ : 1°Cs<sup>-1</sup> and (b)  $D_{\gamma}$ : 62µm,  $\varepsilon$ : 0.6,  $\phi$ : 15°Cs<sup>-1</sup>.



**Figure 5.11** – Example of a LePera etched micrograph  $(D_{\gamma}: 62\mu m, \varepsilon: 0.6, \phi: 1^{\circ}Cs^{-1})$ . White regions (marked by arrows) are identified as martensite + retained austenite.



**Figure 5.12** –  $X_{MA}$  fraction obtained from optical metallography (symbols). The trendline  $(X_{MA} \text{ vs. } X_{\alpha})$  presented is adopted for the MA fraction calculation for all investigated transformation situations.

#### 5.3.8 Polygonal ferrite grain size

Polygonal ferrite grain size  $(D_{\alpha})$  was measured on selected transformation conditions, where at least 15% polygonal ferrite is observed in the transformed microstructure. Figure 5.13 shows the effect of cooling rate and initial austenite grain size on the resulting polygonal ferrite grain size from deformed and non-deformed austenite. As can be seen from Figure 5.13(a), with decreasing initial austenite grain size and increasing cooling rate, the resulting polygonal ferrite grain size becomes finer. With increasing retained strain (from  $\varepsilon$ =0.0 to 0.6), the ferrite grain size becomes even finer (Figure 5.13(b)). Metallography confirmed that the fine polygonal ferrite grain structure (ferrite grain size in the range of 2µm, EQAD) can be achieved by applying a cooling rate higher than 5°Cs<sup>-1</sup> from work hardened austenite. The number of available nucleation sites is an important requirement for the grain refinement during transformation. Considering the austenite grain boundaries as the potential nucleation sites for ferrite nucleation, nucleation density increases with the increasing austenite grain boundary area. Retained strain and the finer austenite grain size thus promote the density of potential nucleation sites for polygonal ferrite transformation.



Figure 5.13 – Measured average ferrite grain sizes (EQAD) as a function of applied cooling rates and austenite grain sizes (symbols): (a) without retained strain and (b) with retained strain of 0.6. Error in the measurement is approximately 10-15%.

### 5.3.9 Hardness

The measured hardness (H) values in VHN scale are presented in Figure 5.14 for all transformed products. As can be observed from Figure 5.14(a), with increasing austenite size and cooling rate, the hardness of the material increases. As an example, it can be seen from Figure 5.14(a), for an initial grain size of 8um, the hardness increases from 168VHN to 245VHN, when the cooling rate increases from 1°Cs<sup>-1</sup> to 100°Cs<sup>-1</sup>. Similarly, for a constant cooling rate of 40°Cs<sup>-1</sup>, the hardness of the material increases from 222VHN to 247VHN, when the initial austenite grain size increases from 8µm to 62µm. With increasing initial austenite grain size and applied cooling rate, the formation of non polygonal acicular/bainitic structure associated with the higher dislocation density is promoted and the transformed microstructure becomes finer. Therefore the hardness of the material increases. The situation becomes complex when the austenite was deformed before the transformation. The hardness results of the transformed structures from the predeformed austenite are shown in Figure 5.14(b). Transformation from a pancaked austenite results in an increase in the finer ferrite volume fraction (applicable for slower cooling rate of 1-5°Cs<sup>-1</sup>, at higher cooling rate polygonal ferrite fraction decreases significantly, see Figure 5.9), thus decreases the hardness of the transformed product (polygonal ferrite is a softer phase compared to acicular/bainite structure). In addition, the

effect of cooling rate and the austenite grain size on the hardness values decreases when the transformation occurs from pancaked austenite. As can be seen from Figure 5.14(b), there is little difference in hardness values (205VHN to 215VHN) for a cooling rate of  $15^{\circ}$ Cs<sup>-1</sup>, when the austenite grain size varies from 8 to 62µm.



**Figure 5.14** – Hardness values as a function of cooling rates and initial austenite grain sizes (symbols): (a) without retained strain and (b) with retained strain of 0.6. Error in the measurement is approximately 10VHN.

### 5.4 Modelling

### 5.4.1 Overview

Based on the continuous cooling transformation data, a model for the overall austenite decomposition kinetics has been developed that consists of five sub-models, i.e. (i) start of ferrite transformation, (ii) ferrite growth, (iii) bainite start, (iv) bainite transformation and (v) martensite/retained austenite fraction. As presented in section 5.3, the CP steel had polygonal ferrite as a transformation product when the austenite decomposition occurred from a fine pancaked austenite microstructure and the applied cooling rates were relatively low (less than  $40^{\circ}$ Cs<sup>-1</sup>). The first two sub-models describing the formation of polygonal ferrite are formulated starting from approaches previously proposed for low carbon and microalloyed steels [10]. To account for the austenite structure before transformation), an effective austenite grain size ( $D_{eff}^r$ ) is introduced [53],

$$D_{eff}^{\gamma} = D_{\gamma} \exp(-\varepsilon_{eff})$$
(5.3)

#### 5.4.2 Transformation start temperature

Polygonal ferrite nucleation occurs preferentially at grain corners [4-5, 54-55]. However, with increasing cooling rate, transformation shifts to lower transformation temperature and thus increases the driving pressure for nucleation. Therefore during faster cooling conditions, less favourable areas of austenite grains, such as grain edges and surfaces become the potential nucleation sites for ferrite formation [56]. Previously developed modelling approaches for ferrite nucleation [10, 56] proposed site saturated nucleation of ferrite grains, i.e. all the possible ferrite nucleation sites were consumed at the beginning of transformation (generally associated with a ferrite fraction of 0.05). Following nucleation, the ferrite formation is then governed by ferrite growth up to the point of ferrite cessation. Carbon diffusion in austenite during the ferrite formation is assumed as the rate controlling step, although solute drag from the substitutional elements such as Mn, Mo etc may be of importance. A possible carbon profile is shown in Figure 5.15 during ferrite formation (spherical ferrite nuclei).



Figure 5.15 – Schematics of carbon profile expected during ferrite formation.

Considering site saturated nucleation at grain corners of parent austenite during continuous cooling, the transformation start temperature  $(T_S)$  is proposed as the temperature with the cessation of ferrite nucleation due to early growth of ferrite. The growth of ferrite nuclei under steady state conditions can be written as [10, 56]:

$$\frac{dr_{\alpha}}{dT}\frac{dT}{dt} = D_C \frac{c_{eq}^{\gamma} - c^{\circ}}{c_{eq}^{\gamma} - c_{eq}^{\alpha}} \frac{1}{r_{\alpha}}$$
(5.4)

where,  $r_{\alpha}$  is the radius of ferrite grains,  $D_C$  is the carbon diffusivity in austenite [57],  $c^0$  is the bulk concentration of carbon and the equilibrium concentrations of carbon in ferrite,  $c_{eq}^{\alpha}$ , and austenite,  $c_{eq}^{\gamma}$ , respectively, have been determined from Thermo-Calc using the Fe2000 data base assuming ortho-equilibrium. Integrating Eq.5.4 over a constant cooling path  $T=T_N-\phi t$ , yields,

$$r_{\alpha} = \sqrt{\frac{2}{\varphi} \int_{T}^{T_{N}} D_{C} \frac{c_{eq}^{\gamma} - c^{0}}{c_{eq}^{\gamma} - c_{eq}^{\alpha}} dT}$$
(5.5)

Here,  $T_N$  is the nucleation temperature of corner ferrite and  $\phi$  is the constant cooling rate between ferrite nucleation to the designated temperature T, over the cooling path. The ferrite nucleation cannot take place in the areas of austenite grain boundary which are already covered by ferrite grains or even in the proximity of growing ferrite nuclei due to the sharp rise in carbon concentration within the austenite grains close to the ferrite-austenite interface (Figure 5.15). This complex situation is captured by considering a limited carbon concentration,  $c^*$ , in the vicinity of the growing ferrite grain above which the ferrite nucleation is inhibited. The limiting radius,  $r^*$ , for nucleation can be quantified as a function of  $r_{\alpha}$  and ferrite nucleation at austenite boundaries ceases, when  $2r^{*2} = D_{\text{eff}}^{r^2}$ , which is equivalent to [56]

$$r_{\alpha} \ge \frac{c^{*} - c^{0}}{c_{eq}^{*} - c^{0}} \frac{D_{eff}^{*}}{\sqrt{2}}$$
(5.6)

The condition of nucleation site saturation is assumed to coincide with the measurable transformation start (5% transformed for the present situation and is determined from the literature study). For a constant cooling rate ( $\phi$ ), Eqs.5.5 and 5.6 yield,

$$\frac{c^* - c^0}{c_{eq}^r - c^0} = \frac{2}{\varphi^{\frac{1}{2}} D_{eff}^r} \sqrt{\int_{T_s}^{T_s} D_C \frac{c_{eq}^r - c^0}{c_{eq}^r - c_{eq}^\alpha} dT}$$
(5.7)

to determine the transformation start temperature  $T_s$ . Using this approach,  $T_N = 782^{\circ}$ C and a limiting carbon concentration of

$$c^* = \left(3.1 + \frac{22\,\mu m}{D_{eff}^{\gamma}}\right)c^0 \tag{5.8}$$

have been determined for the present steel. The present model has three fit parameters, i.e. the nucleation temperature,  $T_N$ , and the fit parameters associated with the determination of  $c^*$ . The  $T_N$  temperature is determined in the present analysis from the transformation start temperatures for the slowest continuous cooling rate conditions (i.e.  $1^{\circ}$ Cs<sup>-1</sup>), where a significant amount of polygonal ferrite is observed. The parameter  $c^*$  is then used to describe the transformation start for higher cooling rates as long as a measurable amount of polygonal ferrite forms. The strong dependency of  $c^*$  on the austenite grain size has been reported previously for Nb-Ti microalloyed steels [10].

Figure 5.16 shows the of the transformation start model results along with the experimental transformation start temperatures (5% fraction transformed) for three different initial austenite grain sizes (8-62 $\mu$ m) and different continuous cooling conditions (i.e. for various cooling rates). For the present steel, the experimental data is presented as the undercooling ( $\Delta T$ ) required to initiate the polygonal ferrite transformation. The analysis of transformation start has been restricted to cases where at least 15% polygonal ferrite is observed in the final microstructures.



**Figure 5.16** – Comparison between model fit (solid lines) and experimental results (symbols) for undercooling required to initiate ferrite transformation.

#### 5.4.3 Austenite to polygonal ferrite transformation

JMAK approach [6-8] adopting additivity has frequently been used [10, 15, 26-27, 44, 46-47] to describe polygonal ferrite growth. In the present research, a similar approach is employed. The present model was developed based on the transformation data which were associated with microstructures consisting of at least 15% polygonal ferrite fraction as measured from optical metallography. The differential form of the JMAK equation can be written as

$$\frac{dF(X)}{dt} = nk_x^{1/n} (1 - F(X)) \left[ -\ln(1 - F(X)) \right]^{\frac{n-1}{n}}$$
(5.9)

Here, b is a rate parameter, F(X) is the normalized polygonal ferrite fraction and n is the JMAK exponent. At first, the value of n was evaluated so that the rate parameter, b is independent of applied cooling rate. The best value of n is obtained at n=1.1, which is within the range of 0.8 to 1.2 as proposed previously for plain carbon and microalloyed steels [11-12]. The value of the rate parameter, b is then expressed as a function of transformation temperature (T) during the ferrite transformation range and the effective prior austenite grain size,  $D_{eff}^{\gamma}$  in µm, as shown below,

$$\ln(b) = B_1 + B_2 T + B_3 \ln D_{eff}^{\gamma}$$
(5.10)

Here,  $B_1$ ,  $B_2$  and  $B_3$  are fit parameters and the best fit is obtained adopting  $B_1=10.926$ ,  $B_2=-0.017^{\circ}C^{-1}$  and  $B_3=-1.365$ . The true polygonal ferrite fraction obtained during continuous cooling was normalized to the equilibrium ferrite fraction at each temperature increment, where the equilibrium ferrite fraction was calculated using THERMOCALC software based on ortho-equilibrium condition. A 6<sup>th</sup> order polynomial equation was used to best describe the equilibrium ferrite fraction for the present steel. The true ferrite fraction transformed,  $X_{\alpha}$ , is related to F(X) by

$$X_{\alpha} = \frac{c_{eq}^{\gamma} - c^{0}}{c_{eq}^{\gamma} - c_{eq}^{\alpha}} F(X)$$
(5.11)

Although the model can describe the early part of ferrite formation as obtained experimentally, it failed to describe the later portion of ferrite growth due to the occurrence of transformation stasis (see Figure 5.7). Therefore, the model was modified to accommodate the transformation stasis at the later stage of the ferrite formation regime.

#### 5.4.4 Transformation stasis

To describe the transformation stasis formation, selected ferrite transformation conditions associated with severe stasis formation are analysed. Selected examples are presented in Figure 5.7. As shown in Figure 5.7, just before reaching transformation stasis, ferrite growth slows down and forms a nearly horizontal plateau region, which constitutes stasis. The stasis continues for some temperature range before the transformation resumes with the bainite start. A detailed analysis is shown in Figure 5.17. Figure 5.17(a) shows a typical fraction transformed curve as a function of time and Figure 5.17(b) presents the derivative  $(dX_{\alpha}/dt)$  as a function of time (the present analysis includes only the polygonal ferrite and stasis regime). As can be seen from Figure 5.17(b),  $T_{SI}$  is the point of maximum ferrite formation rate in the time-transformation rate curve. Beyond this point the ferrite transformation rate decreases steadily and approaches zero at  $T_{S2}$ , which can be essentially considered as the beginning of the stasis. The corresponding positions of  $T_{S1}$  and  $T_{S2}$  points are also shown in Figure 5.17(a) that also indicates the equilibrium ferrite fraction that could have been achieved without the presence of transformation stasis.  $T_{SI}$  can be considered as the point where the gradual transition into the stasis regime begins.

In the present analysis, the values for  $T_{S1}$  and  $T_{S2}$  are established from the temperature-fraction transformed curves (the time-fraction transformed curve can also be represented as the temperature-fraction transformed curve) on selected cases, where their presences are clearly visible.

Then, the driving pressures at those temperature-fraction transformed points ( $T_{S1}$  and  $T_{S2}$ ) are calculated based on the ferrite fraction formed up to those temperature points. The driving pressure ( $\Delta G$ ) is calculated from the mole fraction of carbon in the remaining austenite using THERMOCALC software, such as

$$\Delta G = a_1 [C^2] + a_2 [C] + a_3 \tag{5.12}$$

where,  $a_1$ ,  $a_2$  and  $a_3$  are temperature dependent fit parameters and [C] is the mole fraction of carbon in the remaining austenite. The calculated driving pressures at  $T_{S1}$  and  $T_{S2}$  are presented as a function of temperature in Figure 5.18. In both cases the driving pressure calculated shows a linear dependency on temperature. To simplify the critical driving pressure approach, an average critical driving pressure ( $\Delta G_{stasis(average)}$ ) concept is formulated. This average critical driving pressure is then adopted to describe the beginning of transformation stasis. The stasis of the ferrite transformation is included in the model by relating the representative stasis temperature,  $T_{stasis}$ , to a critical, temperature-dependent driving pressure (in Jmol<sup>-1</sup>), i.e.

$$\Delta G_{stasis(average)} = \frac{\Delta G_{stasis(T_{S1})} + \Delta G_{stasis(T_{S2})}}{2} = 3784 - 5.14T$$
(5.13)

for the present steel where T is given in °C. The calculated average critical driving pressure trendline for stasis formation is shown in Figure 5.18 along with the driving pressure lines at  $T_{S1}$  and  $T_{S2}$ .



Figure 5.17 – Selection of two important temperature points on the transformation curve where severe transformation stasis is observed.

### 5.4.5 Polygonal ferrite growth (including stasis)

To include the critical driving pressure for stasis start into the ferrite growth model, the evolution of driving pressure ( $\Delta G_{\alpha}$ ) during the entire polygonal ferrite formation regime (i.e. from polygonal ferrite start, corresponding to 5% transformation temperature to the completion of polygonal ferrite + transformation stasis portion) is calculated using Eq.5.12 (as a function of carbon enrichment in the remaining austenite during ferrite formation). The evolution of driving pressure during ferrite growth is then compared with the critical driving pressure for stasis start ( $\Delta G_{stasis(average)}$ ) along the austenite decomposition path. A gradual transition to the condition of complete stasis, i.e. when  $dX_{\alpha}/dt=0$ , is facilitated by introducing a reduction factor,  $\lambda$ , for the ferrite formation rate that decreases linearly from 1 to 0 when the driving pressure of ferrite transformation is within 20% of the critical driving pressure for the stasis start value according to  $\Delta G_{stasis(average)}$ . The quality of the ferrite model including the above stasis formation criteria is illustrated in Figure 5.19 for two different cooling conditions from various initial austenite microstructures.



**Figure 5.18** – Adopted critical driving pressure lines for  $T_{S1}$  and  $T_{S2}$  experimental points. The dashed line represents the average critical driving pressure for the transformation stasis formation.



**Figure 5.19** – Ferrite model fit (solid lines) implementing the transformation stasis criteria for different transformation conditions: (a)  $\phi$ : 1°Cs<sup>-1</sup> and (2)  $\phi$ : 5°Cs<sup>-1</sup>.

#### 5.4.6 Ferrite grain size

Polygonal ferrite grain size  $(D_{\alpha})$  has frequently been expressed empirically as a function of initial austenite grain size, cooling rate and chemical composition of steel [10, 26-27, 58]. However, those approaches were not satisfactory for higher cooling rates even for a plain carbon steel. A modified approach [10] expressed the polygonal ferrite grain size (in terms of EQAD) values as a function of transformation start temperature, volume fraction of ferrite and initial austenite grain size as originally proposed by Suehiro et al. [28], i.e.,

$$D_{\alpha} = \left[ X_{\alpha} \exp\left(\beta - \frac{E}{T_{s}}\right) \right]^{\frac{1}{3}}$$
(5.14)

where,  $X_{\alpha}$  is the ferrite volume fraction as measured from optical metallography,  $T_S$  is the transformation start temperature in K. E is a fit parameter and  $\beta$  is expressed in terms of effective austenite grain size  $(D_{eff}^{\gamma})$  to accommodate the effect of retained strain prior to austenite decomposition,

$$\beta = p D_{eff}^{\gamma^q} \tag{5.15}$$

For the present analysis, a best fit is obtained by adopting p=30.67, q=0.04 and E=28031K. The model is developed based on the ferrite grain size data, where at least 15% polygonal ferrite fraction is obtained after austenite decomposition. Figure 5.20 compares the quality of model calculation with the measured ferrite grain size obtained from three pancaked austenite conditions with a retained strain of 0.6. The calculated and experimental ferrite grain size values are presented in terms of EQAD.

### 5.4.7 Bainite start temperature

As proposed previously [59], and verified for simulated run-out table cooling of a Mo-TRIP steel [60], the critical driving pressure concept can also be used to predict the bainite start temperature,  $T_{Bs}$ . In the present research, the non-polygonal ferrite and/or bainite start temperature is determined as the ferrite stop temperature from the experimental dilation curve based on the polygonal ferrite volume fraction (including stasis). To simplify the modelling approach, acicular ferrite and bainite are clustered together in one group and termed as bainite. Driving pressures at the individual bainite

start temperature are calculated as a function of carbon concentration (in terms of mole fraction) in the remaining austenite (after the polygonal ferrite formation including stasis). Eq.5.12 is employed to calculate the individual experimental driving pressures points (values are shown in Figure 5.21). As shown in Figure 5.21, when plotted, the calculated individual driving pressure for bainite start shows a linear dependency with the transformation temperature. Therefore a critical driving pressure trendline is established based on the experimental results to calculate the bainite start condition for the present CP steel.



**Figure 5.20** – Measured (symbols) and model calculated (lines) ferrite grain size values from three different initial austenite grain sizes with an applied deformation strain of 0.6.

Interestingly, as shown in Figure 5.22, to a first approximation, the derived critical driving pressure relationship for the stasis formation and bainite start are very similar and thus the relationship applicable to describe transformation stasis (i.e. Eq.5.13) can be used as the critical driving pressure for the onset of the bainite transformation. The transition to bainite instead of stasis occurs, when the transformation temperature is below 620°C. Therefore, the ferrite model works as a competitive driving pressure approach where the evolution of the transformation driving pressure of remaining austenite is continuously compared with the critical driving pressure of the bainite/stasis formation. Once the transformation temperature is below the critical/maximum bainite start temperature (i.e. 620°C) and the transformation driving pressure is below the driving pressure shown in

Eq.5.13, ferrite formation ceases and bainite starts to form. Further, along fast cooling paths where the austenite decomposition has not yet commenced when reaching the bainite start criterion,  $T_{Bs}$  represents also the overall transformation start temperature and no polygonal ferrite is present in the final microstructure.



**Figure 5.21** – Critical driving pressure for bainite start. Symbol represents the individual driving pressure at the experimental start temperature as determined from the optical metallography and dilation data.



Figure 5.22 - Critical driving pressure line for the stasis formation and bainite start.

#### 5.4.8 Bainite growth

To a first approximation, the growth of bainite can be described similar to that previously proposed for ferrite, i.e. using the JMAK approach adopting additivity as given in Eq.5.9. For the present study, a value of 0.85 is adopted for *n* and the rate parameter (*b*) for the bainite reaction is expressed as a function of the effective grain size of the remaining austenite ( $D_{eff}^{r_{rem}}$  in µm) and its carbon concentration ( $c_{eff}^{r_{rem}}$ ),

$$\ln(b) = C_1 + C_2 T + C_3 \left( C_{eff}^{\gamma_{rem}} * \ln(D_{eff}^{\gamma_{rem}}) \right)$$
(5.16)

Here, 12.3,  $-0.019^{\circ}C^{-1}$  and -15.405 are adopted for the fit parameters  $C_1$ ,  $C_2$  and  $C_3$  respectively. The remaining effective austenite grain diameter available for the bainite transformation can be expressed as,

$$D_{eff}^{\gamma_{rem}} = \sqrt[3]{(1 - X_{\alpha})D_{eff}^{\gamma^{-3}}}$$
(5.17)

where,  $X_{\alpha}$  is the ferrite fraction and  $D_{eff}^{\gamma}$  is the effective initial austenite grain size. The carbon concentration in the remaining austenite grain size (after ferrite formation) is expressed as,

$$c_{eff}^{\gamma_{rem}} = \frac{c^0}{1 - X_{\alpha}}$$
(5.18)

where,  $c^{\theta}$  is the bulk carbon concentration of the current steel. Figure 5.23 illustrates that this approach leads to a reasonable description of bainite transformation from finegrained, equiaxed or pancaked austenite in the present steel with initial austenite grain sizes of 20µm or below. However, for increasingly larger austenite grain sizes, the limits of this approach become apparent as indicated in Figure 5.23 for the results when  $D_{\gamma}=62\mu$ m. For larger austenite grain sizes sympathetic nucleation is expected to take an ever increasing role and this additional nucleation mode can significantly contribute to the non-additive character of the bainite transformation. Nevertheless, for the rather narrow austenite grain size range expected at the exit of the finish mill the proposed model provides a useful approximation for the bainite transformation kinetics. The normalized bainite fraction is related to the true bainite fraction,  $X_B$ , by

$$X_{B} = (1 - X_{a} - X_{MA})F(X)$$
(5.19)

where  $X_{MA}$  is the MA fraction that is empirically related to the ferrite fraction (see Eq. 5.2). This relationship (Eq.5.2) reflects the increased stability of austenite when it is enriched with carbon.



Figure 5.23 – JMAK model fit (solid lines) to describe the experimental bainite growth.

### 5.4.9 Model validation (stepped cooling regime)

Once the complete model is developed for austenite decomposition, run-out table simulations are conducted to compare the model predictions with the measured volume fractions of transformation products. The key processing step considered here is cooling on the run-out table where the phase transformation takes place and is tailored to result in a fine, multi-phase microstructure consisting of ferrite, bainite and martensite. For this purpose, stepped cooling regimes are employed as follows: rapid cooling at 40°Cs<sup>-1</sup> from 875 to 700°C, slow cooling ( $\phi_{slow}$ ) in the ferrite transformation range from 700 to 640°C with different cooling times varying from 4-12s followed by another rapid cooling step (40°Cs<sup>-1</sup>) to a coiling temperature of 600°C that has been selected to maximize precipitation strengthening (the basis of selecting 600°C as the coiling temperature is described in Chapter 6). Coiling simulations are conducted at 30°Ch<sup>-1</sup> to 510°C followed by air cooling of the sample to room temperature. Two different initial austenite grain sizes (8 and 20µm) those are relevant to finish rolling regime, are considered and the samples were deformed to a strain of 0.6 before the stepped cooling tests.

Examples of the observed microstructures are shown in Figure 5.24. As can be observed from Figure 5.24, for smaller austenite grain size and slower cooling rate regime, the microstructure is predominantly polygonal ferrite. With increasing cooling rate  $(\phi_{slow})$  from 5°Cs<sup>-1</sup> to 15°Cs<sup>-1</sup> in between 700-640°C, the polygonal ferrite fraction decreases (Figure 5.24(a) vs. 5.24(b)). Increasing the austenite grain size from 8µm to 20µm (Figure 5.24(b) vs. 5.24(c)), the polygonal ferrite amount further decreases and the structure becomes more acicular in nature. Table 5.2 provides a comparison of the predicted and measured fractions of transformation products. As presented in Table 5.2, the model provides a satisfactory description of the tendencies in the resulting microstructural features, especially the prediction of the polygonal ferrite volume fraction is excellent. However, predicted MA fractions are somewhat lower than those obtained from fraction metallography.

### 5.5 Discussion

The current research was intended to investigate the phase transformation behaviour of the present steel and to understand the cooling process required to obtain a multiphase microstructure, consisting of fine scale ferrite, bainite and martensite. As expected the present transformation study reflects the importance of the initial austenite grain size, retained strain and applied cooling rate, where ferrite formation is promoted with decreasing austenite grain size, decreasing cooling rate and increasing amount of retained strain. In addition, the present study reveals that the fine polygonal ferrite structure (in the order of 2-3µm) can be achieved when the transformation occurs from fine pancaked austenite employing a cooling rate in the range of 5-15°Cs<sup>-1</sup>. However, at higher cooling rates (>15°Cs<sup>-1</sup>), the effect of initial austenite grain size disappears and the structure becomes mostly nonpolygonal. In a traditional hot strip rolling mill, an average cooling rate of 40°Cs<sup>-1</sup> is generally experienced by a hot rolled strip in the run-out table. Therefore, considering the experimental results (Figures 5.3 to 5.7), it is now possible to establish that a combination of fine pancaked austenite and a complex/stepped cooling regime is a prerequisite to achieve the desired multiphase microstructure, i.e. a combination of fine polygonal ferrite, bainite and martensite + retained austenite.



(c)

**Figure 5.24** – Microstructures obtained from pancaked austenite ( $\varepsilon$ : 0.6) after stepcooling operations with slow cooling from 700 to 640°C: (a)  $D_{\gamma}$ : 8µm,  $\phi_{slow}$ : 5°Cs<sup>-1</sup>, (b)  $D_{\gamma}$ : 8µm,  $\phi_{slow}$ : 15°Cs<sup>-1</sup> and (c)  $D_{\gamma}$ : 20µm,  $\phi_{slow}$ : 15°Cs<sup>-1</sup>.

$D_{\gamma}$	$\phi_{slow}^*$ , (°Cs <sup>-1</sup> )	Predicted			Measured			
(µm)		Xα	X <sub>B</sub>	X <sub>MA</sub>	Xα	$X_B$	X <sub>MA</sub>	
8	5	0.64	0.25	0.11	0.61	0.22	0.17	
8	10	0.48	0.41	0.11	0.53	0.28	0.19	
8	15	0.40	0.49	0.11	0.45	0.36	0.19	
20	5	0.33	0.576	0.10	0.40	0.40	0.20	
20	10	0.21	0.69	0.10	0.21	0.59	0.20	
20	15	0.17	0.73	0.10	0.17	0.61	0.22	

**Table 5.2**: Predicted and measured volume fractions of different transformed phases after the stepped cooling tests.

\* $\phi_{slow}$  is applied between 700-640°C.

For the first time, both the growth of polygonal ferrite and bainitic is described by the JMAK approach adopting additivity. From a modelling point of view, application of the JMAK approach to appreciate the polygonal ferrite growth is not new. However, in the present transformation situations, the polygonal ferrite formation is associated with the formation of transformation stasis. Thus, the model for the ferrite growth is modified to appreciate the stasis formation (section 5.4.3, 5.4.4 and 5.4.5). The stasis model is based on the consideration of an average critical driving pressure condition for stasis formation. When incorporated in the overall ferrite growth model, the transformation stasis regime is captured to a satisfactory extent. In addition, considering the experimental accuracy, the critical driving pressure relationship that is adopted to describe the stasis formation can be applied to predict the bainite start condition.

The present study concludes that the JMAK approach assuming additivity can adequately describe the bainite growth when a fine pancaked austenite (initial  $D_{\gamma}=20\mu m$ ) is considered as its initial structure (Figure 5.23). However, increasing the initial austenite grain size limits the applicability of the JMAK model. As mentioned before, the current research is inconclusive to differentiate between the different nonpolygonal ferrite structures and thus a simple approach is considered to cluster all irregular shaped ferritic phases as bainite. This approach is simple but fails to appreciate the nucleation mechanism of different types of non polygonal phases. For smaller austenite grain size, the formation of bainite is at relatively high temperature, i.e. approximately at 600°C or above, whereas with increasing austenite grain size, the bainite formation shifts to lower temperature (close to 550°C). With increasing austenite grain size, the sympathetic nucleation is expected to take a significant role and this additional nucleation mode can contribute to the non-additive character of the bainite formation. However, considering the evolution of austenite grain structure during industrial hot rolling for austenite decomposition, the larger grain sizes are of less importance.

One important aspect of the present research is establishing the relationship between the polygonal ferrite fraction and the MA fraction as shown in Figure 5.12 i.e. the MA volume fraction increases with increasing the polygonal ferrite volume fraction. This observation reflects the increased stability of austenite with increasing carbon content. In the present study, dilation data shows (Figure 5.10) that the martensite start

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temperatures were around 275°C. However, in most cases the martensite start temperatures were difficult to measure from the dilation data as they all exhibit little deviation from linearity, probably due to the presence of low martensite fraction in most cases. Thus, to calculate the MA fraction an empirical relationship (Eq.5.2) is proposed based on the metallography results. This approach is simple but fails to accurately predict the MA fraction obtained after stepped cooling regime as shown in Table 5.2. Further investigations are required to improve the predictive capabilities of the proposed transformation model for the secondary transformation products (i.e. bainite and MA).

Finally, the present study is inconclusive regarding the effect of microalloying elements on phase transformation behaviour. Especially the effect of Nb in solution on overall transformation kinetics can be important. As mentioned elsewhere [23-25, 48], due to the segregation of solute Nb on the austenite-ferrite grain boundary, Nb exerts a strong solute drag force on the moving boundary and thus, retards ferrite growth. However, when Nb is allowed to precipitate, the transformation becomes faster. A similar observation was reported for bainite transformation [33]. In a typical hot strip rolling, most of the microalloying elements are expected to be in solution (except Ti) during the austenite decomposition and thus a strong solute drag force is expected from them. However, in the present transformation study, the selected reheating temperature to achieve finer austenite grain size (i.e., soaking at 950-1100°C) may encourage little dissolution of Nb. Therefore most of Nb is expected to be in the matrix as microalloyed precipitates and may have no or negligible retarding effect on the overall phase transformation.

# 5.6 Conclusion

The following conclusions are made from the above experimental and modelling study.

• The austenite grain size, retained strain and applied cooling rate have strong influence on the austenite decomposition kinetics. The transformation shifts to higher temperatures with decreasing austenite grain size and cooling rates and with increasing retained strain. However, with increasing retained strain and cooling rate the effect of austenite grain size is diminished. To achieve a fine multiphase transformed microstructure with a desired polygonal volume fraction, a combination of heavily deformed fine austenite structure and complex/stepped cooling regime is required.

- The transformation start temperature model previously developed for predominantly polygonal ferrite structure is successfully employed for the present steel, even though the transformed conditions results in lower polygonal ferrite volume fraction (approximately 15%). The model is fundamentally based on nucleation of ferrite at grain corners and early growth of these ferrite grains. However, during continuous cooling conditions a more realistic model may be desirable to capture the allotriomorphic ferrite nucleation at grain edges and surfaces. In addition, the model does not separately capture the solute drag effect of microalloying elements on transformation start temperature.
- The present research suggested a fine polygonal ferrite grain size in the order of 2-3µm is achievable under industrial cooling condition. A semi-empirical approach has been adopted to describe the polygonal ferrite grain size. Comparison between model prediction and experimental data showed reasonable agreement.
- Both polygonal ferrite and bainite growth are successfully modelled with the JMAK approach adopting additivity. An effective austenite grain size concept is employed to incorporate the effect of retained strain during modelling. Considering a critical driving pressure approach the ferrite model can successfully accommodate the formation of transformation stasis between ferrite and bainite transformation regime. Interestingly the same relationship can also be applied to describe the bainite formation. However, bainite model prediction gradually deviates from the experimental observation with increasing austenite grain size.
- The present models do not account for the solute drag effect by microalloying elements on austenite decomposition, which can significantly delay the polygonal ferrite formation and thus promotes more non polygonal structures.
- The total model prediction when compared with the stepped cooling results showed an excellent agreement for ferrite formation regime. However, the experimentally observed MA fraction is generally higher than that predicted by

the model. Additional investigations are needed to appreciate the formation of MA fraction and hence to improve the overall model prediction.

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# **CHAPTER 6: COILING SIMULATION AND PROPERTY EVALUATION**\*

# 6.1 Introduction

The primary aim of modern thermomechanical processing is to achieve a fine scale microstructure that results in an optimum combination of strength and elongation in a hot rolled coil. In addition to thermomechanical processing the chemical composition of the steels can be suitably altered by microalloying additions such as Nb, Ti and V (0.01-0.1wt%) that aid the formation of finer product structure and strengthen the steel by precipitation hardening [1-3]. The final product strength of steel can then be characterized by the volume fraction and scale of various transformed phases, ferrite grain size and the strengthening by solutes and microalloyed precipitates [4-6]. In modern AHSS, the final microstructure consists of fine ferrite, bainite and martensite, which is further strengthened by fine carbides/carbonitrides of microalloying elements [7-8].

Previous studies suggested that the precipitation of microalloying elements such as Nb, Ti and V can occur at three different stages of hot rolling, namely during deformation in the fully austenite region, i.e. strain induced precipitation [1, 9-12], during the austenite to ferrite transformation, i.e. interphase precipitation [13-15] and after transformation in the supersaturated ferrite/transformed phases [3, 5-6]. Carbides may form both in austenite and ferrite depending on the processing conditions and the chemistry of the steels. In contrast, Mo is believed to be mostly soluble in austenite but can precipitate in ferrite depending on the presence of the other carbide forming elements [16-17]. However, the prior precipitation of microalloyed carbides in austenite during deformation or during phase transformation has been found not to strengthen the ferrite significantly [18]. In addition, considerable research suggests that in a typical hot strip rolling scenario (i.e. comprising of short interpass times during finish rolling (5 to 0.5s) and accelerated cooling that delays the austenite decomposition to lower transformation

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temperatures on the run-out table), the microalloying precipitation occurs in the supersaturated ferrite predominantly during coiling of the hot rolled products [3, 5-6]. This precipitation in ferrite and other transformation products is believed to contribute significantly to the hardening of steels. These particles form in the supersaturated ferrite either during prolonged heat treatment after transformation [19-20] or during aging of quenched materials [6, 7, 21] and in the industrially processed under-aged hot strip coils during coiling [3]. The observed particles are either spherical or needle-like in shape and generally are observed in regions of high dislocation density.

As discussed above, both composition and the processing conditions influence the precipitation behaviour. The present work is focused on simulating the entire hot rolling process including reheating the steel at 1250°C (to achieve a large initial austenite grain size in the range of 250µm and dissolving all microalloying elements), deforming in the range of 1150-900°C associated with short interpass time (10-0.5s from rough rolling to finish rolling) followed by rapid cooling to a designated coiling temperature employing a hot torsion system. Hardness measurements were conducted to evaluate the aging characteristics of the material. The resulting precipitation kinetics in the as-quenched materials has been investigated to propose optimum coiling conditions<sup>\*</sup>.

# 6.2 Experimental methodology

The simulation of the hot strip rolling process of the present steel has been carried out in a DSI HTS100 hot torsion tester. The hot torsion samples were machined from forged bar and the working zone of the sample was 10mm in diameter and 12.7mm in length. Prior to the test, the chamber was evacuated to 4-5Pa and back filled with commercial purity (99.95%) argon. During the entire simulation operation, the temperature of the sample was monitored by a S type (Pt/Pt-Rh) thermocouple. In addition, the temperature during the multipass deformation was monitored by an optical pyrometer (working range of 613-1400°C). The sample was soaked at 1250°C for 30minutes and after 3 roughing passes (R1 to R3, total equivalent strain of 1.0), 7 finishing passes were carried out between 1040°C and 900°C employing a constant strain rate of 1.0s<sup>-1</sup>. The details of the torsion schedule are presented in Table 6.1. Accelerated

<sup>\*</sup> A list of symbols used in this chapter is presented in appendix 5.

cooling on the run-out table was simulated by employing He gas cooling. However, instead of terminating the quench at the coiling temperature, the samples were cooled to room temperature and subsequently reheated to the potential coiling temperatures in the range of 530 to 630°C. The samples were held isothermally for up to one month at 530-630°C (aging treatment). This procedure was employed to facilitate a systematic study of the associated age hardening response and thus precipitation strengthening kinetics. A tube furnace with argon purging facility was employed for all aging tests.

Soaking conditions: heating at 5°Cs <sup>-1</sup> to 1250°C and hold for 30minutes												
Parameters	R1, R2 and R3	F1	F2	F3	F4	F5	F6	F7				
Temperature (°C)	1100	1024	982	958	950	922	910	900				
Strain	0.33 (each pass)	0.35	0.50	0.40	0.30	0.30	0.25	0.10				
Interpass time (s)	10	4	2.4	1.6	1.2	0.8	0.6	_				

Table 6.1: Torsion experiment schedule simulating hot strip rolling for the CP steel.

A Vickers micro-hardness tester with a load of 1000gm was employed to measure hardness of the torsion samples to investigate the precipitation response of the microalloying elements. The area of hardness measurements was kept close to the surface of the torsion samples, i.e. within 1mm from the edge of the sample as the effect of applied strain is highest at the sample edge (zero at the centre of the sample). Approximately 5-9 measurements were done for each aging condition (as a function of temperature and time) and the average values are taken for subsequent analysis. To investigate the variability in the hardness values from sample to sample, two torsion samples were considered, which have experienced the same prior thermomechanical treatment. Optical metallography was done employing a Nikon EPIPHOT 300 series inverted microscope equipped with a digital camera on the sectioned torsion samples (section, where the thermocouple was attached) before any aging treatment using Nital or Marshall reagent [22].

# 6.3 Results and modelling

### 6.3.1 As-quenched structure

The microstructure of the as-quenched material after torsion simulation is presented in Figure 6.1 along with the family of stress-strain curves measured from the

torsion simulation. As can be seen from Figure 6.1(b), the microstructure consists of fine acicular ferrite and carbon-rich residual phases (dark areas in the micrograph, probably bainite and martensite). Clarification of the exact nature of the secondary transformation products requires further investigations that will include transmission electron microscopy. However, negligible polygonal ferrite fraction was observed suggesting that more sophisticated cooling regimes are required to form the desired complex-phase microstructure.



**Figure 6.1** – Torsion test result: (a) Stress-strain behaviour of the CP steel during torsion simulation as per Table 6.1, (b) as-quenched (to room temperature) microstructure after torsion simulation.

# 6.3.2 Aging behaviour

The strength of the present microalloyed steel can be increased by precipitation of carbonitrides that readily form with the microalloying elements (Nb/Mo in the present case) in the transformed phases. The measured hardness of the as-quenched torsion sample was approximately 210VHN, i.e. approximately a tensile strength of 630Mpa (assuming that the tensile strength value is approximately 3 times the hardness value) that is below the target strength of CP steels. However, the quenching procedure produces severely underaged samples without any significant precipitation strength. Isothermal aging at three different simulated coiling temperatures ( $630^{\circ}$ C,  $580^{\circ}$ C and  $530^{\circ}$ C) indicates that the strength can be increased approximately by 36HV such that the overall tensile strength can indeed reach levels close to 750Mpa. This hardness change,  $\Delta H$ , can

be attributed to microalloyed precipitation and thus is termed as  $\Delta H_{ppt}$  and the maximum precipitation hardening contribution,  $\Delta H_{peak}$ , is 36HVN in the present steel. Figure 6.2 shows the variability in the hardness measurements of two as-quenched torsion samples those were given same hot rolling simulation as presented in Table 6.1. As can be seen from Figure 6.2, little variability is observed in the hardness results from the two investigated samples. Figure 6.3 exhibits the average hardness results (average hardness of two as-quenched torsion samples) at three proposed coiling temperatures.



Figure 6.2 – Variability in the hardness data from sample to sample, which were given the same hot rolling treatment (error in the measurement is approximately  $\pm 10$ VHN from the average).

Significant strengthening is observed during all aging treatments (Figure 6.3) as compared to the as-quenched materials. As expected, peak hardness time increases with decreasing aging temperature i.e. it is lowest for  $630^{\circ}$ C (approximately 600s) and highest for  $530^{\circ}$ C (around  $1.2\times10^{5}$ s). Further, the peak hardness is relatively independent of the aging temperatures, suggesting a temperature-independent volume fraction of strengthening precipitates. This is consistent with the observed low solubility of the microalloying elements in ferrite and replicates previous findings in HSLA steels [3, 5]. It is also important to notice that the increase in the hardness curve is due to the precipitation of the microalloying elements, whereas the decreasing part of the curve may be a combined effect due to coarsening of precipitates and the softening of the ferrite matrix.



Figure 6.3 – The aging response of the CP steel for three aging temperatures.

To determine the optimum coiling temperature for the present steel that will maximise the effect of precipitation strengthening, the approach originally proposed by Shercliff and Ashby [23] is employed in the present study with few modifications. This original model framework [23] has been developed to describe the aging response of commercial aluminium alloys (Al-Cu and Al-Mg-Si), based on the dislocation-precipitation interaction and the kinetics of precipitation. Further, the model [23] is assumed that a single kinetic equation can be applicable for a single peak aging curve and the evolution in the precipitates size can be modelled based on a single coarsening law. However, their model based on a cubic coarsening law (i.e.  $r_p^3 \propto t$ ) [24-25], could not describe the experimental data for the current steel as shown in Figure 6.4, especially during the underaging and overaging regime. A similar deviation in the model predictions was reported earlier on microalloyed steels with quasipolygonal structure as well as in the interstitial-free (IF) steels containing 1.4wt% Cu as precipitation strengthener [26-27]. Therefore a modified approach is considered for the present modelling purpose.



**Figure 6.4** – Precipitation prediction from model based on the Shercliff-Ashby approach [23] considering coarsening of precipitates as the rate controlling step.

Results for a Fe-12Ni-6Mn marageing steel [28] suggested that a parabolic growth approach can satisfactorily capture the precipitation behaviour at the early stage of precipitation hardening. However, at the later stage of precipitation (i.e. at the overaging regime), a shift may be necessary from growth based approach to a coarsening based approach to model the overall precipitation kinetics. For the present research, a particle growth approach as proposed previously [28] is considered to describe the overall aging curves presented in Figure 6.3. The growth of a mean precipitates size  $(r_p)$  with time (t)can be given by the Zener parabolic relationship [28-30].

$$r_p = C_1 \sqrt{D_{eff} t} \tag{6.1}$$

where,  $C_1$  is the constant related to solubility of microalloying elements in precipitates and matrix and  $D_{eff}$  is the effective diffusivity of microalloying elements (Nb/Mo for the present steel grade).

Considering,

$$D_{eff} = D_0 \exp\left(-\frac{Q_{ppt}}{RT}\right)$$
(6.2)

where,  $D_0$  is constant,  $Q_{ppt}$  represents the effective activation energy for diffusion of the microalloying elements forming the precipitates, R is the universal gas constant, T is the

aging temperature. A temperature-corrected time (P) can be introduced to characterize the precipitation during aging as

$$P = \exp\left(-\frac{Q_{ppt}}{RT}\right)t \tag{6.3}$$

Thus, mean precipitates size,  $r_p$ , becomes a function of P by

$$r_p = C_1 \sqrt{D_0} \times \sqrt{P} = C \sqrt{P} \tag{6.4}$$

where, C is a constant. The precipitation strengthening increment is a function of P, with the maximum strength being realised at  $P_p$ . A plot of the peak times,  $t_p$ , in terms of  $\ln(t_p)$ 

vs. 1/T results in a linear curve where the slope characterizes  $\frac{Q_{ppt}}{R}$  and the intercept is given by  $P_p$ , as illustrated in Figure 6.5. The value of the effective activation energy for the current steel is found to be 300kJmol<sup>-1</sup> with the  $P_p$  value being  $2.3 \times 10^{-15}$ s. These parameters are in a similar range to that for other Nb microalloyed steels [3, 5]. Master curves for precipitation strengthening can then be constructed by introducing a normalized temperature corrected time ( $P^* = \frac{P}{P_p}$ ) and a normalized precipitation strength

contribution  $\left(\frac{\Delta H_{ppt}}{\Delta H_{peak}}\right)$  as shown in Figure 6.6.



Figure 6.5 – Relationship between aging temperatures and the peak aging times.

As proposed by Shercliff-Ashby [23], considering the shearing and bypassing regime as the dominant mechanisms of precipitation strengthening and assuming a negligible contribution of solute solution hardening of the precipitate forming elements [6], the modified model results in the following age hardening kinetics:

$$\frac{\Delta H_{shear}}{\Delta H_{peak}} = \frac{2}{0.94} \left[1 - \exp\left(-2P^{*1.25}\right)\right]^{\frac{1}{2}} \left(P^{*\frac{1}{4}}\right) \text{ (Shearing regime)}$$
(6.5)

$$\frac{\Delta H_{bypass}}{\Delta H_{peak}} = \frac{2\left[1 - \exp\left(-2P^{*1.25}\right)\right]^{\frac{1}{2}}}{0.94P^{*\frac{1}{2}}}$$
(Bypassing regime) (6.6)

Finally the Harmonic mean of shearing (Eq.6.5) and bypassing (Eq.6.6) regime is taken to describe the entire precipitation behaviour. However, as previously shown to describe the precipitation behaviour in V and Nb containing microalloyed steels [31] a small correction has been made to ensure that peak strength occurs when  $P^*$  is equal to 1. In the present work,  $P^*=1.2P/P_p$  is proposed to describe the overall aging behaviour of the CP steel. The final equation is as given below,

$$\frac{\Delta H_{ppt}}{\Delta H_{peak}} = \frac{0.94}{2\left[1 - \exp\left(-2P^{*1.25}\right)\right]^{1/2}} \left(P^{*-1/4} + \sqrt{P^*}\right)$$
(6.7)

The model calculation is shown in Figure 6.6. The measured precipitation strength  $(\Delta H_{ppt})$  is adequately described when there is significant precipitation strengthening, i.e.  $\Delta H_{ppt} \ge 0.25 \Delta H_{peak}$ . For underaged material,  $\Delta H_{ppt}$  is somewhat underpredicted as solution strengthening and details of the nucleation kinetics of precipitates are not considered in the model. For overaging, a slower decrease of  $\Delta H_{ppt}$  is predicted than observed since recovery of the highly dislocated bainitic ferrite matrix is not taken into account.


Figure 6.6 – Master curve constructed from the experimental results, solid line shows the model calculation.

#### 6.3.3 Coiling prediction

To appreciate the precipitation strength during coil cooling, Eq.6.3 is modified to accommodate the continuous cooling path that is experienced by the hot strip during coiling. To estimate the precipitation strength developed during coil cooling, the value of  $P^*$  is determined from

$$P^* = \frac{1}{P_p} \int_0^t \exp\left(\frac{-Q_{ppt}}{RT(t)}\right) dt$$
(6.8)

along the coiling/aging time-temperature path. Based on the results shown in Figure 6.6, 90% of the maximum strength is realized when  $0.75 < P^* < 2.5$ . This provides a criterion to determine a suitable coiling temperature window. The coiling prediction for the present steel is shown in Figure 6.7 assuming a coil cooling rate of  $30^{\circ}$ Ch<sup>-1</sup> over a possible coiling temperature range. The present steel is then predicted to be coiled in the temperature range of 585-610°C to develop at least 90% of its precipitation-strength potential. The maximum precipitation strength can be achieved at 595°C.



**Figure 6.7** – Coiling prediction for the present steel to appreciate the precipitation strengthening from microalloyed precipitates.

#### 6.3.4 Property evaluation

To a first approximation the mechanical properties (here the hardness values, H) can be described as a function of the fraction of the constituent phases in the final microstructure and the contribution from precipitation strengthening assuming a linear addition law, i.e.

$$H = H_{Base} + \Delta H_{ppt} \tag{6.9}$$

where,  $\Delta H_{ppt}$  is the hardness contribution from the microalloyed precipitates and  $H_{Base}$  can be further expressed in terms of the volume fractions of transformation products, i.e. in the present situation in terms of ferrite, bainite and martensite/retained austenite. To evaluate the base property of the present steel, Vickers' hardness measurements were carried out on the continuously cooled transformed samples with initial austenite grain sizes of 8-62µm, i.e. samples, which have been soaked in the temperature range in between 950-1100°C. It was assumed that for these soaking regimes, there will be little or no dissolution of previously present precipitates. After the complete thermomechanical treatment, these large precipitates probably have negligible contribution on the overall hardness of the material. The hardness results as function of transformation start temperatures  $(T_S)$  are presented in Figure 6.8. As expected, the hardness values increase with a steady decrease in the transformation start temperature.



**Figure 6.8** – Average hardness values of the transformed materials as a function of transformation start temperatures (error associated with each measurement is approximately 10VHN).

For the present steel, the base strength is expressed in terms of its constituent phases. This approach is similar to those previously proposed to model structure-property relation in multiphase steels consisting of mainly ferrite/pearlite structures [32-33]. As a first approximation, the base strength/hardness ( $H_{base}$ ) is presented as follows

$$H_{Base} = H_{\alpha} X_{\alpha} + H_{B} X_{B} + H_{MA} X_{MA} \tag{6.10}$$

The parameters  $H_i$  (*i*: ferrite ( $\alpha$ ), bainite (*B*) and martensite + retained austenite (*MA*) indicate the relative strength contributions of the different transformation products. Knowing the individual phase volume fraction from metallography (Chapter 5), the best fit is obtained when  $H_{\alpha}=122$ ,  $H_B=710-0.81T_{Bs}$  and  $H_{MA}=406$  in VHN is adopted. The strength contribution for bainite is expressed as a function of the bainite transformation start temperature (in °C) as the feature size of bainitic ferrite is refined when the bainite reaction takes place at increasingly lower temperatures. For ferrite and MA, these relative strength contributions are taken as independent of scale and composition. Figure 6.9 shows the relationship between the experimental hardness values and the bainite start temperature ( $T_{Bs}$ ) of selected transformed samples consisting of predominantly bainite

structure (fraction bainite, i.e.  $X_B$  approximately 0.5 or above).  $T_{Bs}$  is determined from Chapter 5.



**Figure 6.9** – Base hardness values as a function of bainite start temperatures for predominantly bainitic samples ( $X_B$  is higher than 0.5).

Finally the model calculation is shown in Figure 6.10. As can be seen from Figure 6.10, the current empirical model is able to describe the base strength of the present steel to a satisfactory level.

#### 6.4 Discussion

The aim of the present chapter is to address the precipitation strengthening of microalloying elements and propose a suitable modelling approach to describe the structure property correlation. Previous work on the microalloyed steels predominantly focused on the precipitation behaviour in ferrite [3]. In contrast, the torsion simulation adopted in the current research produces a predominantly fine scale acicular ferrite/bainite in the as-quenched microstructure (see Figure 6.1(b)). This structure is complicated due to the heterogeneous distribution of dislocation density, which affects precipitate nucleation and matrix recovery [6]. However, assuming a growth based modified Shercliff-Ashby approach, the present model can capture the precipitation strengthening behaviour, to a satisfactory extent.



Figure 6.10 – Model fit for the base strength of the present CP steel. Both experimental data and model fit are in good agreement.

As proposed here, a particle growth based approach indeed describes the aging response of the microalloyed precipitation for most part of the curve (Figure 6.6). A small deviation is observed at the beginning and at the end of the aging curve, i.e. at  $P^* < 0.1$  and  $P^* > 30$ . The present model assumes a negligible contribution of solute strengthening from microalloying elements and in addition, the model completely ignores the possible effect of heterogeneous nucleation sites on precipitation. This possibly explains the limitation of the present model in describing the early stage of the aging curve. At the later part of the overaging regime ( $P^* > 40$ ), the model over predicted the precipitation strength. The faster drop in strength at longer aging time probably involves both softening of the matrix and coarsening of the precipitates. For a detailed investigation of softening of the matrix and the precipitation morphology and distribution, TEM study would be required.

The present model predicts a coiling temperature of 595°C to achieve maximum precipitation strength from microalloyed carbides/carbonitrides considering 30°Ch<sup>-1</sup> as the coil cooling rate. This optimum coiling temperature is slightly lower than that proposed for other Nb containing steels (620°C) and substantially lower than for the V containing steel (675°C) [5]. If the steel is coiled at higher temperatures, precipitation hardening will be reduced due to the coarsening of the precipitates. On the other hand,

coiling at lower temperatures will keep the ferrite in a supersaturated state and hence the maximum precipitation potential would not be realized in the hot rolled coil.

Previous models [4] predicted the strength of the material based on the ferrite grain size, ferrite and/or pearlite volume fraction in addition to the strengthening contribution from solid solution strengthener. These considerations were valid for the investigated HSLA steels as the product microstructure was predominantly ferrite-pearlite. However, the current steel shows a transformed microstructure (Chapter 5) that is more complex in nature, being a combination of fine polygonal ferrite, predominantly bainite and martensite phases. Therefore the strength contribution from bainite and MA on the overall material strength is considered. In addition, in most transformed microstructures there is little presence of polygonal ferrite network. Thus the strength of ferrite cannot be represented in terms of the ferrite grain size.

A justification of the absolute values for the strength contribution from various phases is beyond the scope of this study, nevertheless their trends show a sensible pattern, i.e. the strength contribution from martensite is higher than that of bainite, which in turn is higher than that of ferrite. In addition, the increase in strength of bainite with decreasing  $T_{Bs}$  is reasonable; as the bainite transformation moves to lower temperature, the inter-lath spacing within bainite decreases and the structure becomes finer, raising the strength of bainite. For the future, it would be of interest to examine the mechanical properties of steel, such as the yield strength, tensile strength and total elongation.

#### 6.5 Complete model validation

#### 6.5.1 Experimental overview

The overall model prediction (from reheating to coiling) was validated by conducting a torsion simulation. The complete rolling simulation includes five pass of rough rolling (R1-R5) in the temperature range in between 1150-1100°C and seven pass of finish rolling (F1-F7) in the temperature range in between 1050-875°C. Reheating of torsion specimens was carried out for 15 minutes at 1250°C to bring all Nb in solution. Previously developed constitutive, softening and grain growth models (Chapters 3 and 4) are employed to determine the austenite conditioning during the entire hot rolling regime that will provide the initial condition for austenite decomposition. In Table 6.2, two different rolling schedules are presented with varying amounts of accumulated strain, i.e. an accumulated (retained) strain ( $\varepsilon_{eff}$ ) of 1.6 and 2.3, respectively, as predicted by the model. The applied strain rate ( $\dot{\varepsilon}$ ) was varied from 0.6s<sup>-1</sup> to 1.0s<sup>-1</sup> to achieve different amounts of accumulated strain. This was done to investigate the effect of accumulated strain on the final ferrite volume fraction. For this rolling schedule (see Table 6.2, Schedule: A), the model predicted a volumetric austenite grain size of  $52\mu m$  (45 $\mu m$  for Schedule: B, Table 6.2) before the first finish rolling pass, F1. However, during deformation in the finishing passes (in between F1-F7), the austenite is expected to be severely deformed, resulting in a pancaked austenite microstructure after F7.

The key processing step during the torsion experiments is the cooling simulation on the run-out table, where the phase transformation takes place and can be tailored to result in a fine, multiphase microstructure consisting of polygonal ferrite (PF), bainite (B) and MA. For this purpose, stepped cooling as described in Chapter 5 was employed, i.e.: rapid cooling at 40°Cs<sup>-1</sup> from 875 to 700°C, slow cooling in the ferrite transformation range from 700 to 640°C with different cooling times ranging from 2 to 12s followed by another rapid cooling step (40°Cs<sup>-1</sup>) to a coiling temperature of 600°C and 550°C. According to the precipitation model (section 6.3), a coiling temperature of 600°C should result in achieving the full precipitation potential, whereas coiling at 550°C is expected to decrease the precipitation strength (coiling at 550°C results in approximately 35% of the total precipitation potential) but results in a refinement of the second phases. Coiling was simulated with a slow cooling rate ( $\phi$ ) of 30°Ch<sup>-1</sup> for three hours followed by air cooling of the sample to room temperature. The first series of tests (strain rate of  $0.6s^{-1}$ ) was designed to investigate the role of cooling time during the ferrite transformation range whereas the second test series (strain rate of  $1.0s^{-1}$ ) emphasized the role of coiling temperature. All torsion simulated specimens were characterized by metallography and hardness measurements.

Deformation	Temperature	(Sched) Total retaine Strain ra	ule: A) d strain (1.6) te: 0.6s <sup>-1</sup>	(Schedule: B) Total retained strain (2.3) Strain rate: 1.0s <sup>-1</sup>	
pass		Equivalent strain	Interpass time (s)	Equivalent strain	Interpass time (s)
R1	1150	0.30	10	0.52	10
R2	1150	0.29	10	0.46	10
R3	1100	0.26	10	0.40	10
R4	1100	0.26	10	0.40	10
R5	1100	0.26	10	0.40	10
F1	1050	0.36	2.5	0.59	3.6
F2	1020	0.32	1.9	0.52	2.3
F3	980	0.29	1.4	0.45	1.6
F4	950	0.24	1.1	0.39	1.1
F5	920	0.22	0.9	0.33	0.8
<b>F</b> 6	895	0.19	0.7	0.29	0.6
F7	875	0.16	-	0.25	-

**Table 6.2**: Simulated torsion schedule for validation of the hot strip model.

An example of the family of stress-strain curves during the multipass deformation is presented in Figure 6.11 for the tests with a strain rate of 0.6s<sup>-1</sup> (total retained strain of 1.6). As can be seen from Figure 6.11, little if any recrystallization is observed during finish rolling of the present steel; especially after the second finish mill pass (F2). This observation is independent of the details of the finish mill rolling schedule as shown before (see Table 6.1 and Figure 6.1). An increase in the applied strain rate to 1.0s<sup>-1</sup> (Schedule B) results a similar trend in the calculated stress-strain curves. Complete softening was observed during rough rolling and in between the rough and finish rolling. During finish rolling, significant softening (approximately 50%) is only observed between finish rolling passes F1 and F2 and after F2, the material work hardens continuously resulting in a higher level of retained strain compared to the lower strain rate simulation, i.e. retained strain of 2.3 instead of 1.6.



**Figure 6.11** – Hot deformation response of the present microalloyed steel during the hot torsion simulation (Table 6.2 (Schedule: A), total retained strain ( $\varepsilon_{eff}$ ): 1.6 and the applied strain rate ( $\dot{\varepsilon}$ ): 0.6s<sup>-1</sup>).

#### 6.5.2 Effect of slow cooling regime

Figure 6.12 illustrates selected examples of the complex-phase microstructures produced during the torsion simulation using two different slow cooling rates, i.e.  $\phi_{slow}=5$  and 10°Cs<sup>-1</sup> through the ferrite formation temperature range. In all the simulated materials, the final microstructures are a homogeneous mixture of fine polygonal ferrite, acicular ferrite/bainite and MA. A MA phase fraction of approximately 0.19 is observed in the final microstructures. The MA islands are comparatively large with sizes in the range of 5-10µm. Table 6.3 presents the measured and the predicted volume fractions of transformation products obtained after torsion simulations. As expected, slower cooling in the ferrite formation regime resulted in a small increase in the ferrite volume fractions agree well with the model predictions. However, the experimental ferrite fractions are approximately two times higher than the predicted MA fraction (Table 6.3). This finding is similar to that reported for the stepped cooling tests (Chapter 5, Table 5.2).

Further investigations are required to improve the predictive capabilities of the proposed transformation model for secondary transformation products (i.e. bainite and MA). For simplicity, the present model calculated the MA fraction based on the carbon enrichment resulting from the ferrite formation. The proposed empirical relationship has been developed based on the observations made in the CCT tests (Chapter 5). However, for a more advanced model one would have to track the martensite start temperatures taking into account the details of bainite formation and carbide precipitation.



**Figure 6.12** – Final microstructures obtained after hot torsion simulations of the CP steel using different cooling scenarios: (a)  $\phi_{slow}$ : 5°Cs<sup>-1</sup> in between 700-640°C and (b)  $\phi_{slow}$ : 10°Cs<sup>-1</sup> in between 700-640°C (Schedule: A).

$D_{\gamma}$	$\phi_{slow}^{**}$	Accumulated strain	Predicted			Measured		
(µm)	(°Cs <sup>-1</sup> )	$(\mathcal{E}_{eff})$	$X_{\alpha}$	X <sub>B</sub>	X <sub>MA</sub>	$X_{\alpha}$	X <sub>B</sub>	X <sub>MA</sub>
52	5	1.6	0.34	0.56	0.10	0.33	0.50	0.17
52	10	1.6	0.24	0.66	0.10	0.25	0.55	0.20

Table 6.3: Measured and predicted fractions for the hot torsion\* simulated rolling.

\*Coiling temperature: 600°C, \*\* $\phi_{slow}$  is applied between 700-640°C

#### 6.5.3 Effect of total accumulated strain

Table 6.4 presents the measured volume fraction of the various transformation products after torsion simulation with different deformation schedules but employing the same run-out table cooling strategies, i.e. a slow cooling rate of  $10^{\circ}$ Cs<sup>-1</sup> in the ferrite formation regime and a coiling temperature of 600°C (Schedule A vs. Schedule B, Table 6.2). The schedule B predicts an austenite grain size of 45µm before finish rolling instead

of 52µm as predicted by Schedule A. The total accumulated strain in austenite increases from 1.6 to 2.3 (see Table 6.2); thus refines the effective austenite grain size after finish rolling from 10 to 5µm (schedule A vs. Schedule B), resulting in a marginal increase in the measured polygonal ferrite fraction from 0.25 to 0.30 (see Table 6.4). This difference in the measured ferrite fraction is essentially within the error of the metallography measurements (an error in the range of 0.05 fractions in terms of absolute value). The model predictions taking an effective accumulated strain of 1.6 and 2.3 are also presented in Table 6.4. As can be observed from Table 6.4, model prediction and the experimental ferrite fraction are in agreement for an accumulated strain of 1.6 (X<sub>a</sub>: 0.24 vs. 0.25, respectively) whereas the model over-predicts the ferrite fraction for an accumulated strain of 2.3 ( $X_{\alpha}$ : 0.51 vs. 0.30). Surprisingly, the ferrite model calculation assuming 1.6 as the accumulated strain (from an austenite grain size of 45µm) predicts a polygonal ferrite fraction of approximately 0.32 (measured 0.30) in the final microstructure as presented in Table 6.4. It is worth noting that the effect of retained strain on the ferrite nucleation and growth model was characterized by compression tests where the maximum retained strain was approximately 0.6-0.7. Thus, the use of the model for the larger retained strains typical of realistic hot rolling practices involves a substantial extrapolation of these results. It is reasonable to assume that the effect of increasing levels of retained strain on ferrite formation decreases at large strains, i.e. once the grains are sufficiently pancaked, further pancaking of the grains has little additional effect on the subsequent ferrite formation. Based on the limited experimental data in this study, an accumulated strain of 1.6 is proposed as the saturation strain and above which the austenite decomposition is assumed to be independent of further pancaking of austenite grains. This assumption is consistent with the observations that the ferrite fraction, ferrite grain size and the MA fraction are similar for retained strains of 1.6 and 2.3, i.e. the measured volumetric ferrite grain size was 3-4µm and the MA volume fraction was approximately 0.18. Further investigations are required to confirm the proposed selection of 1.6 as the appropriate value for the saturation strain level.

D <sub>v</sub>	Osion **	<ul> <li>Accumulated strain</li> </ul>		Predicte	d	Measured		
(µm)	(°Cs <sup>-1</sup> )	$(\mathcal{E}_{eff})$	Xα	$X_B$	X <sub>MA</sub>	Xα	X <sub>B</sub>	X <sub>MA</sub>
52	10	1.6	0.24	0.66	0.10	0.25	0.55	0.20
45		2.3	0.51	0.38	0.11	0.30	0.51	0.19
45		1.6	0.32	0.58	0.10	0.30	0.51	0.19

Table 6.4: Measured and predicted fractions for the hot torsion\* simulated rolling.

\*Coiling temperature: 600°C, \*\* $\phi_{slow}$  is applied between 700-640°C

#### 6.5.4 Effect of coiling temperature

Decreasing the coiling temperature to  $550^{\circ}$ C resulted in a minor change in the final microstructure. As measured by metallographic studies, the volume fraction of various phases remains similar to what has previously been achieved, when a coiling temperature of 600°C was considered (Table 6.5). However, the MA islands in the transformed microstructure are finer when the sample was coiled at 550°C. Average size of the MA islands is in the range of 4-6µm as compared to 5-10µm for coiling at 600°C. The model prediction is also presented in Table 6.5.

Table 6.5: Measured and predicted fractions for the hot torsion\* simulated rolling.

$D_{\gamma}$ Coiling@		Predicted ( $\varepsilon_{eff}$ : 1.6)			Measured (accumulated strain: 2.3)		
(µm)	(°C)	Xα	X <sub>B</sub>	X <sub>MA</sub>	$X_{\alpha}$	X <sub>B</sub>	X <sub>MA</sub>
45	600	0.22	0.59	0.10	0.30	0.51	0.19
42	550	550 0.32 0.58		0.10	0.29	0.54	0.17

\*  $\phi_{slow}$ : 10°Cs<sup>-1</sup> is applied between 700-640°C

#### 6.5.5 Property validation

Table 6.6 presents the model prediction of base strength of the present steel considering the stepped cooling regime (see section 5.4) and is compared with the measured hardness results those were obtained from experiments. It can be observed that the predicted base hardness is somewhat lower than the measured hardness values. This is consistent with the metallographic observation, as the predicted MA fractions are somewhat lower than the measured values (Table 5.2) and consequently the predicted hardness values are approximately 5-10% lower than those measured.

$D_{\gamma}(\mu m)$	<i>ø<sub>slow</sub></i> , (°Cs <sup>-1</sup> )**	Predicted (base strength)	Measured (base strength)
8	5	176	198
8	10	192	207
8	15	201	218
20	5	207	224
20	10	221	237
20	15	225	244

Table 6.6: Measured and predicted hardness values of base strength after the stepped cooling operations\*.

\*Coiling temperature: 600°C, \*\* $\phi_{slow}$  is applied between 700-640°C

The predicted strengths of the torsion samples (base strength + precipitation strength) are presented in Table 6.7 and are compared with the measured hardness values. As shown in Tables 6.3 to 6.5, the model consistently underpredicted the MA fraction and thus is expected to under predict the overall material hardness as compared to the measured hardness data. However, the overall predicted hardness results are in agreement with that of the measured data when a coiling temperature of 600°C is considered. One possible explanation can be the over prediction of precipitation strength in the present steel. During the quantification of the precipitation strength, microalloyed precipitation happened in a predominantly bainitic structure (see Figure 6.1), where precipitation kinetics and may be precipitation strengthening is aided by the presence of a highly dislocated structure. However, the torsion simulations resulted in an approximately 25-30% polygonal ferrite fraction, which has a much lower dislocation density phase as compared to acicular ferrite/bainite. Therefore the precipitation hardening response of polygonal ferrite is expected to be lower than that of the bainite structure.

However, as compared to the model prediction, the measured hardness is higher (i.e. 250VHN as compared to the predicted hardness of 236VHN), when a coiling temperature of 550°C was considered. Changing the coiling temperature from 600°C to 550°C, reduces the amount of precipitation strengthening (only 35% of the total precipitation potential is achieved when coiling is done at 550°C). Similar to what has been observed for the CCT samples the hardness is somewhat underpredicted since the predicted MA fraction is lower than the measured MA fraction (Table 6.6).

The measured hardness values suggest tensile strength levels of approximately 750Mpa that fall short of the target range of 800Mpa for complex-phase steels [34]. Therefore a further reduced coiling temperature (in the range of 500°C or below) needs to be considered, where refinement of microstructure, especially the refinement of bainite and MA islands can increases the strength of the steel above 800Mpa, even if one sacrifices the precipitation potential of microalloying elements.

	\$slow	Coiling@	T T	Predicted	1	Measured
$D_{\gamma}(\mu m)$	(°Cs <sup>-1</sup> )	°C	H <sub>Base</sub>	$\Delta H_{ppt}$	Н	(H, total strength)
52	5	600	208.5	35.5	244	248 ( <i>ɛ</i> : 1.6)
52	10	600	218	35.5	253.5	249 ( <i>ɛ</i> . 1.6)
45	10	600	210.5	35.5	246	245 ( <i>ɛ</i> : 2.3)
45	10	550	223.5	12.5	236	250 (ɛ: 2.3)

**Table 6.7**: Measured and predicted total hardness of the torsion specimens. Predicted volume fractions are calculated based on  $\varepsilon_{eff}$ : 1.6, which determines the  $H_{Base}$ .

#### 6.6 Conclusion

Based on the above experimental and modelling results following conclusions can be made.

- Significant strengthening is observed during aging at specified coiling temperatures. This can be attributed to the precipitation of microalloyed elements (here, Nb/Mo) in the as-quenched structure. The peak hardness is independent of the aging temperature. However, the present study is inconclusive on the chemical composition and distribution of the precipitates within the matrix. Further TEM studies are required to identify the composition and morphology of the precipitates.
- Assuming a particle growth based approach, the modified Shercliff-Ashby model results in a satisfactory description of the aging curve.
- Assuming a coil cooling rate of 30°Ch<sup>-1</sup>, the model predicts a coiling temperature between 585 to 610°C to realize at least 90% of its precipitation strength potential.
- The structure-property model successfully describes the base strength of the present steel as a function of various constituent phases. However, the model needs improvement; especially the strength of MA islands should be included as a

function of its carbon content or its formation temperature. In addition, the property model should be modified to predict the material property in terms of yield and tensile strength and percent elongation.

- In general the predictive capability of the model is satisfactory. The total model validation shows that the ferrite model prediction is excellent with the measured ferrite fraction after torsion simulation, when a total retained strain of 1.6 is considered. However, the model consistently underpredicts the MA constituents' fraction.
- The torsion simulation results shows that increasing the amount of retained strain over 1.6 or so creates little difference in the final microstructure and thus no apparent improvement in the product strength is observed. However the refinement of the transformed structure (especially in bainite) can lead to an improvement of the product strength that may be achievable by decreasing the coiling temperature of the present steel below 500°C.

#### 6.7 **Bibliography**

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#### **CHAPTER 7: OVERALL CONCLUSION AND FUTURE WORK**

#### 7.1 Conclusion

The aim of the current research was to achieve a detailed understanding of the microstructure evolution of a selected complex-phase steel under hot strip rolling conditions. To accomplish this goal, the individual metallurgical phenomenon was investigated from reheating to coiling at relevant hot strip rolling temperatures. Each microstructural phenomenon such as austenite grain growth, austenite conditioning during deformation, multiphase transformation from austenite and precipitation of microalloyed carbides on the transformed phases was studied. Laser-ultrasonics was used to study the grain size evolution during austenite conditioning before and after deformation. Following hot deformation, the development of the transformed multiphase microstructure was investigated considering various cooling strategies, which are applicable to run-out table cooling operations. Based on the laboratory simulations, a complete microstructure evolution model was proposed starting from the established approaches for the microalloyed steels to predict the structure-property relationship. The current research concludes:

• Laser-ultrasonics can be employed to measure the austenite grain size evolution before and after deformation. This technique is continuous, remote, non-destructive and suitable for steels where measuring the austenite grain size is a challenge using conventional techniques. This technique has the potential to be used for online monitoring of austenite grain size in industrial settings. The absolute accuracy of the laser-ultrasonic grain size measurements has been determined to be approximately 25%. The austenite grain growth results confirm that 1150-1200°C is the approximate dissolution temperature for Nb microalloyed precipitates. The current study finds that austenite grain growth is significantly affected by the temperature. At lower temperatures, i.e. between 900-1000°C, little grain growth is observed; whereas at higher temperatures, such as >1100°C, significant grain growth occurs. However, care should be taken in analysing the ultrasonic grain size results during abnormal grain growth stages with a bimodal

grain size distribution, as the laser-ultrasonic result is dominated by the large grains in the distribution.

The present investigation confirms that the calibration procedure for the ultrasonic measurements that was developed based on the recrystallized grain structure can be extended to measure the deformed, pancaked austenite grain size. In addition, the present study suggests that the attenuation measurements can be used for the measurement of i) grain size evolution and ii) the start (5%) and finish (95%) times of recrystallization, thus providing a new experimental tool to observe recrystallization and grain growth following plastic deformation. Further investigation will be required to explore the potential of attenuation based measurements to also provide detailed information on the recrystallization kinetics.

- The softening study concludes that complete softening is possible only during • rough rolling (rolling temperature in the range of 1150-1100°C); whereas partial or negligible softening is expected during finish rolling (rolling temperature around 1000°C or below). Therefore a heavily deformed pancaked austenite grain structure is expected to evolve at the end of finish rolling. Controlled stepped cooling (combination of fast and slow cooling procedure) on work hardened austenite can successfully produce a multiphase microstructure consisting of fraction of fine polygonal ferrite, bainite and MA. This CP steel shows significant precipitation potential during aging. Further, the peak hardness due to precipitation is independent of the aging temperature, suggesting a temperatureindependent volume fraction of strengthening precipitates. To utilize the precipitation strength over 90%, the present CP steel should be coiled between 585 to 610°C. However the results obtained from the torsion simulations suggested that a lower coiling temperature (500°C or below) may be desirable to refine the transformed bainite structure and thus increase the strength of the steel.
- The overall microstructure evolution model for hot strip rolling of the investigated steel is summarized in appendix 6 along with important adjustable parameters. Established physical based approaches are used to model the material behaviour in austenite during deformation. The austenite decomposition model is

of primary importance for the overall process model. A modified JMAK approach adopting additivity is successfully employed to describe the ferrite transformation including stasis and bainite transformation. Overall model validation concludes that model prediction for ferrite fraction is excellent, whereas improvement is needed to develop a more detailed model for the bainite transformation that will enable an accurate tracking of the martensite transformation. For simplicity, the present model calculates the MA fraction based on the carbon enrichment resulting from the ferrite formation. The proposed empirical relationship had been developed based on the observations made in transformation tests. However, for a more advanced model one would have to track the martensite start temperature by taking into account the details of bainite formation and possible carbide precipitation.

#### 7.2 Future work

The present model framework for transformation study is useful as an industrial process model and can capture the austenite decomposition behaviour to a satisfactory level. However a detailed study is needed to appreciate the formation of various types of bainite constituents and martensite. With the current, rather empirical approach the fractions of bainite are overpredicted whereas those for the MA are underpredicted for simulated run-out table cooling and coiling. A more fundamental approach is sought in predicting the fractions of the secondary transformation products, i.e. bainite and MA. For this purpose, different types of bainite constituents and carbide precipitation reactions will have to be taken into account.

In addition, the present transformation model does not account for the solute drag effect of the microalloying elements when they are in solution, which is more relevant to industrial hot rolling conditions. The role of microalloying elements depends on their distribution, i.e. either they are in solution or out of solution. The present transformation studies focussed on situations when Nb or Mo are mostly present as precipitates; especially after austenitizing at 950-1100°C, which are too low to dissolve most Nb in solution. However, during industrial operation a higher soaking temperature such as 1250°C will bring all Nb back in solution. Being in solution, Nb can exert a significant

solute drag effect to delay the ferrite formation and may even shift the bainite formation to lower temperature. Thus the present model approach needs to incorporate a solute drag term that can describe the ferrite or bainite transformation during run-out table cooling more accurately.

A detailed TEM study is required to appreciate the precipitation strengthening; especially detailed knowledge can then be obtained regarding the size, shape and the distribution of precipitates. The current steel microstructure is a heterogeneous structure, where the dislocation density can vary significantly from polygonal ferrite to acicular ferrite and bainite. Therefore the precipitate distribution and their contribution on strengthening can vary from phase to phase. In addition, the present precipitation model does not include the solid solution strengthening effect and also the nucleation regime of precipitates. Those situations should be considered and be implemented to improve the precipitation model. Finally the overall process model prediction should be validated under industrial rolling conditions rather than torsion simulation. This will require investigating a steel grade suitable for producing bainitic or complex multiphase structures under hot strip rolling, but a chemistry that is more practical for commercial products. The investigated steel grade contains a high level of Mo that is more suitable for laboratory investigation as the presence of Mo clearly separates different transformation stages. However, because of the higher cost of Mo, this chemistry is not particularly attractive for steel makers.

Symbol	Description	Unit
α	Total attenuation	-
$\alpha_{IF}$	Ultrasonic attenuation due to internal friction	-
$\alpha_D$	Ultrasonic attenuation due to diffraction	-
$\alpha_{ref}$	Ultrasonic attenuation of reference wave	-
$\alpha_{Sc}$	Ultrasonic attenuation due to grain scattering	-
λ	Ultrasound wavelength	μm
a	Fit parameter relates to internal friction	-
A	Amplitude	a.u
b	Parameter proportional to grain size	Hz <sup>-n</sup>
С	Temperature dependent function parameter	-
D	Average austenite grain size (in EQAD)	μm
f	Frequency	Hz
$K_D$	Material parameter (Diffusion regime)	Hz <sup>-n</sup> µm <sup>1-n</sup>
$K_R$	Material parameter (Rayleigh regime)	Hz <sup>-n</sup> µm <sup>1-n</sup>
$K_S$	Material parameter (Stochastic regime)	Hz <sup>-n</sup> µm <sup>1-n</sup>
K(T)	Material parameter (temperature dependent)	Hz <sup>-n</sup> µm <sup>1-n</sup>
n	Grain size exponent	-
<u>t</u>	Time	S
<u> </u>	Operating temperature	°C
Tref	Reference temperature	°C
<i>x</i>	Travelling distance of ultrasound wave	μm

### List of symbols used in Chapter 2 (Austenite grain growth).

Symbol	Description	Unit
α	Geometrical constant	-
ε	Applied strain	-
Ė	Applied strain rate	s <sup>-1</sup>
Ė	Strain rate normalizing factor	s <sup>-1</sup>
μ	Shear modulus	Gpa
$\mu_0$	Shear modulus at 0K	Gpa
ρ	Dislocation density	m <sup>-2</sup>
$\rho_D$	Dislocation density of as-deformed material	m <sup>-2</sup>
σ	Flow stress of the material	Мра
$\sigma_0$	Yield stress of the fully recrystallized material	Mpa
$\sigma_{D}$	Yield stress of the as-deformed material	Mpa
$\sigma_{Rec}$	Yield stress of the recovered material	Mpa
$\sigma_v$	Scaling stress	Mpa
$\sigma_{v0}$	Scaling stress extrapolated to 0K	Mpa
бин	Stress due to deformation (work hardening)	Mpa
$\sigma_{YS}$	Yield stress of the material	Mpa
σγςο	Yield stress extrapolated to 0K	Mpa
$\frac{d\sigma}{d\varepsilon}$	Work hardening rate	Mpa
$\theta_0$	Initial work hardening rate	Мра
A	Fit parameter (Eq.3.6)	$s^{(1+r)}um^{-p}$
	Lattice parameter of austenite $(\gamma)$	μm
b	Burgers' vector	μm
Ē	Youngs modulus	Gpa
F <sub>S</sub>	Fraction softening	-
$F_x$	Fraction recrystallized	-
go	Adjustable parameter	-
g_i	Adjustable parameter	-
G	Driving pressure for recrystallization	Mpa
<u>к</u>	Fit parameter (Eq.3.10)	
$k_B$	Boltzmann's constant	JK <sup>-1</sup>
<u>K</u>	Adjustable parameter	
K <sub>M</sub>	Fit parameters	m <sup>12</sup> J <sup>-3</sup> s <sup>-3</sup>
<u>M</u>	Taylor factor	
	Pre-exponential factor (fit parameter)	m <sup>4</sup> J <sup>1</sup> 's <sup>-1</sup>
M <sub>eff</sub>	Effective grain boundary mobility	m <sup>+</sup> J <sup>-</sup> 's <sup>-</sup> '
<u>n</u>	JMAK exponent	
$N_{Rx}$	Nucleation density	m <sup>-</sup>

List of symbols used in Chapter 3 (Constitutive and softening behaviour of austenite).

<i>p</i>	Fit parameter (Eq.3.6)	-
q	Fit parameter (Eq.3.6)	-
$Q_{e\!f\!f}^{\scriptscriptstyle M}$	Effective activation energy for grain boundary mobility	kJmol <sup>-1</sup>
Qrex	Activation energy for recrystallization	kJmol <sup>-1</sup>
r	Fit parameter (Eq.3.6)	-
R	Universal gas constant	Jmol <sup>-1</sup> K <sup>-1</sup>
$S_V$	Grain boundary area/volume	m <sup>-1</sup>
t	Time	s/min
t <sub>0.5</sub>	Time for 50% recrystallization	s/min
T	Temperature	°C/K
T <sub>Def</sub>	Deformation temperature	°C/K
$U_A$	Activation energy for recovery	kJmol <sup>-1</sup>
v <sub>D</sub>	Debye frequency	Hz
V <sub>A</sub>	Activation volume	μm <sup>3</sup>
X <sub>Rex</sub>	Fraction recrystallized	-

Symbol	Description	Unit
$\alpha_{gg}$	Geometrical constant during grain growth	-
ε	Deformation strain	-
Ė	Applied strain rate	s <sup>-1</sup>
γgb	Grain boundary energy	Jm <sup>-2</sup>
Λ	Fit constant (Eq.4.1)	μm <sup>1-p</sup>
$D_{0}$	Initial austenite grain size	μm
$D_{\gamma}$	Average austenite grain size	μm
D <sub>rex</sub>	Recrystallized grain size	μm
k <sub>B</sub>	Boltzmanns' constant	JK <sup>-1</sup>
K	Temperature dependent parameter	s <sup>-1</sup>
m	Grain growth exponent	-
$M_0$	Pre-exponential factor (fit parameter)	$m^4 J^{-1} s^{-1}$
M <sub>eff</sub>	Effective grain boundary mobility	$m^4 J^{-1} s^{-1}$
<i>p</i>	Strain exponent (Eq.4.1)	
P	Pinning parameter (fit parameter)	Jm <sup>-3</sup>
q	Grain size exponent (Eq.4.1)	-
$Q_{e\!f\!f}^{\scriptscriptstyle M}$	Effective activation energy for grain boundary mobility	kJmol <sup>-1</sup>
Qgrx	Activation energy for recrystallized grain size	kJmol <sup>-1</sup>
R	Universal gas constant	Jmol <sup>-1</sup> K <sup>-1</sup>
t	time	s/min
<u> </u>	Operating temperature	°C/K
T <sub>def</sub>	Deformation temperature	°C

List of symbols used in Chapter 4 (Recrystallized grain size and subsequent grain growth).

Symbol	Description	Unit
α	Ferrite region	-
β	Grain dependent parameter	-
$\Delta T$	Undercooling amount	°C
Е	Retained strain	-
Ė	Strain rate	s <sup>-1</sup>
$\mathcal{E}_{eff}$	Effective applied strain	-
φ	Cooling rate	°Cs <sup>-1</sup>
γ	Austenite region	-
λ	Reduction factor in ferrite formation rate (stasis formation)	-
$a_1, a_2, a_3$	Temperature dependent fit parameters (driving pressure calculation)	Мра
$A_1, A_2$	Constants	μm
AF	Acicular ferrite	
b	Rate parameter during ferrite/bainite growth	s <sup>-n</sup>
В	Bainite	-
$B_1, B_2, B_3$	Fit parameters (ferrite growth)	-
$c^0$	Bulk concentration of carbon	-
c*	Limiting carbon concentration (fit parameter)	-
$C_{eq}^{\gamma}$	Equilibrium concentrations of carbon in austenite	-
$c_{eq}^{lpha}$	Equilibrium concentrations of carbon in ferrite	-
$c_{e\!f\!f}^{\gamma_{rem}}$	Carbon concentration in remaining austenite	-
[C]	Mole fraction of carbon	-
$C_1, C_2, C_3$	Fit parameters (bainite growth)	-
d <sub>Aus</sub>	Calculated dilation data for austenite	μm
$d_m$	Measured dilation data	μm
$d_{Pro}$	Calculated dilation data for product	μm
$dX_{\alpha}/dt$	Ferrite formation rate	s <sup>-1</sup>
$D_{\alpha}$	Ferrite grain size	μm
$D_C$	Carbon diffusivity in austenite	m <sup>2</sup> s <sup>-1</sup>
γ	Initial austenite grain size	μm
$D_{e\!f\!f}^{\gamma}$	Effective austenite grain size (after deformation)	μm
$D_{e\!f\!f}^{\gamma_{rem}}$	Grain size of the remaining austenite	μm
E	Fit parameter (ferrite grain size model)	K
$F(X_i)$	Normalized fraction transformed (i= ferrite or bainite)	-
$\Delta G_i$	Driving pressure (i=ferrite/stasis formation or bainite start)	MPa
H	Hardness value	VHN

#### List of symbols used in Chapter 5 (Phase transformation).

LM	Lath martensite	-
M <sub>S</sub>	Martensite start temperature	°C
n	JMAK exponent for ferrite and bainite	-
NPF	Non polygonal ferrite	-
р	Fit parameter (ferrite grain size model)	μm <sup>-q</sup>
P	Pearlite	-
PF	Polygonal ferrite	-
9	Fit parameter (ferrite grain size model)	-
r*	Limiting ferrite radius (Transformation start model, ferrite)	μm
$r_{\alpha}$	Ferrite radius	μm
SAus	Thermal expansion coefficients of austenite	µm°C <sup>-1</sup>
Spro	Thermal expansion coefficients of product	µm°C <sup>-1</sup>
t	Time	s
T	Temperature	°C/K
$T_{Bs}$	Bainite start temperature	°C
$T_N$	Nucleation start temperature	°C
$T_S$	Transformation start temperature	°C
$T_{SI}$	Temperature when ferrite formation rate is maximum	°C
$T_{S2}$	Temperature when ferrite formation rate is minimum	°C
T <sub>stasis</sub>	Stasis temperature	°C
X	Fraction transformed	-
$X_{\alpha}$	Fraction ferrite	-
X <sub>B</sub>	Fraction bainite	-
X <sub>MA</sub>	Fraction martensite + retained austenite	-

# <u>APPENDIX 5</u>

List of symbols used in Chapter 6 (Colling simulation and property evaluation).			
Symbol	Description	Unit	
Eeff	Accumulated retained strain		
Ė	Strain rate	<u>s<sup>-1</sup></u>	
φ	Cooling rate	°Cs <sup>-1</sup>	
<i>\$slow</i>	Slow cooling rate	°Cs <sup>-1</sup>	
В	Bainite	-	
С	Constant	-	
$C_{I}$	Fit parameters (Eq.6.1)	-	
$D_{0}$	Pre-exponential factor for diffusivity	$m^2 s^{-1}$	
$D_{eff}$	Effective diffusivity	$m^2 s^{-1}$	
$D_{\gamma}$	Average austenite volumetric grain size	μm	
H	Total hardness	VHN	
H <sub>Base</sub>	Base hardness of the material	VHN	
$\Delta H_{bypass}$	Hardness contribution in bypass regime	VHN	
Hi	Hardness contribution of transformation product ( $I =$ ferrite,	VHN	
	bainite or MA)		
$\Delta H_{peak}$	Peak hardness value	VHN	
$\Delta H_{ppt}$	Hardness due to precipitation	VHN	
$\Delta H_{ppt} / \Delta H_{peak}$	Normalized precipitation strength contribution	-	
$\Delta H_{shear}$	Hardness contribution in shearing regime	VHN	
MA	Martensite + retained austenite	-	
P	Temperature-corrected time	S	
P*	Normalized temperature-corrected time	-	
$P_p$	Peak temperature-corrected time	S	
PF	Polygonal ferrite	-	
$Q_{ppt}$	Effective activation energy for precipitation (Nb/Mo)	kJmol <sup>-1</sup>	
$r_p$	Mean particle size	μm	
R	Universal gas constant	Jmol <sup>-1</sup> K <sup>-1</sup>	
t	Time	S	
T	Temperature	°C/K	
$T_S$	Transformation start temperature	°C/K	
$T_{Bs}$	Bainite start temperature	°C	
$X_{lpha}$	Fraction ferrite	-	
X <sub>B</sub>	Fraction bainite	-	
X <sub>MA</sub>	Fraction martensite + retained austenite	-	

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Metallurgical phenomenon	Model and important parameters
Constitutive behaviour	$\sigma = \sigma_{YS} \left( T_{def}, \hat{\varepsilon} \right) + \sigma_{WH} \left( T_{def}, \hat{\varepsilon}, \hat{\varepsilon} \right)$ $\sigma_{i} = 595 \text{MPa } \& g_{i} = 1.0 \text{ (Yield stress model)}$ $\sigma_{WH} = \sigma_{v} \left( 1 - \exp \left( -\frac{\theta_{0}}{\sigma_{v}} \varepsilon \right) \right)$
Softening behaviour	$\sigma_{v0} = 1/25 \text{MPa & } g_0 = 1.0 \text{ (Flow stress model)}$ $\sigma_{vs} = \sigma_o X + \sigma_{\text{Re}c} (1 - X_{\text{Re}x})$
Recovery	$\frac{d(\sigma_{\text{Rec}} - \sigma_0)}{dt} = -\frac{64(\sigma_{\text{Rec}} - \sigma_0)^2 v_D}{9M^3 \alpha^2 E} \exp\left(-\frac{U_A}{k_B T}\right) \sinh\left(\frac{(\sigma_{\text{Rec}} - \sigma_0)V_A}{k_B T}\right)$ $V_A = \frac{\kappa M \alpha \mu b^2}{(\sigma_D - \sigma_0)}$ $U_A = 286 \text{ kJmol}^{-1} \text{ and } \kappa = 0.05$
Recrystallization	$X_{Rex}(t) = 1 - \exp\left(-N_{Rx}M_{eff}^{3}\left(\int_{0}^{t}G(t)dt\right)^{3}\right)$ $M_{eff} = M_{0}\exp\left(\frac{-Q_{eff}^{M}}{kT}\right), N_{Rx} = Kb^{2}S_{V}\rho_{D}^{2}, G(t) = \frac{1}{2}\rho(t)\mu b^{2}$ $Q_{eff}^{M} = 337 \text{kJmol}^{-1} \text{and} K_{M} = KM_{0}^{3} = 1.65 \times 10^{3} \text{m}^{12} \text{J}^{-3} \text{s}^{-3}$
Recrystallized grain size	$D_{rex} = \Lambda D_0^q \varepsilon^{-p} \exp(-\frac{Q_{grx}}{kT_{Def}})$ $\Lambda = 6.2 \times 10^5 \mu m^{1-p}$ and $Q_{grx} = 165 k \text{Jmol}^{-1}$ for $T_{def} > 1100^{\circ}\text{C}$ , $\Lambda = 0.39 \mu m^{1-p}$ and $Q_{grx} = 0$ for $T_{def} \le 1100^{\circ}\text{C}$ p = 0.82 and $q = 0.84$
Grain growth	$\frac{dD_{\gamma}}{dt} = M_{eff} \left( \frac{\alpha_{gg} \gamma_{gb}}{D_{\gamma}} - P \right)$ $\alpha_{gg} = 0.5, \ \gamma_{gb} = 0.75 \text{Jm}^{-2} \text{ and } P = 2.0 \times 10^{-3} \text{Jm}^{-3}$
Transformation start	$D_{eff}^{\gamma} = D_{\gamma} \exp(-\varepsilon_{eff}), \ \varepsilon_{eff} = \varepsilon_{eff} \text{ if } \varepsilon_{eff} \leq 1.6 \text{ or } 1.6 \text{ for } \varepsilon_{eff} > 1.6$ $\frac{dr_{\alpha}}{dT} \frac{dT}{dt} = D_{C} \frac{c_{eq}^{\gamma} - c^{\circ}}{c_{eq}^{\gamma} - c_{eq}^{\alpha}} \frac{1}{r_{\alpha}} \& c^{*} = \left(3.1 + \frac{22\mu m}{D_{eff}^{\gamma}}\right) c^{\circ}$ $T_{N} = 782^{\circ} \text{C}$

Complete process model summary of the investigated CP steel.

Metallurgical phenomenon	Model and important parameters
	$\frac{dF(X)}{dt} = nk_x^{1/n} (1 - F(X)) \left[ -\ln(1 - F(X)) \right]^{\frac{n-1}{n}} \&$
Ferrite growth	$X_{\alpha} = \frac{c_{eq}^{\gamma} - c^{0}}{c_{eq}^{\gamma} - c_{eq}^{\alpha}} F(X)$
	$\ln(b) = B_1 + B_2 T + B_3 \ln D_{eff}^{\gamma}$
	$n=1.1, B_1=10.926, B_2=-0.017^{\circ}C^{-1}$ and $B_3=-1.365$
Ferrite grain size	$D_{\alpha} = \left[ X_{\alpha} \exp\left(\beta - \frac{E}{T_s}\right) \right]^{\frac{1}{3}} \& \beta = p D_{eff}^{\gamma^q}$
	E=28031K, $p=30.67$ µm <sup>-q</sup> and $q=0.04$
Critical driving pressure for transformation stasis and bainite start	$\Delta G_{stasis(average)} = 3784 - 5.14T \text{ in } (\text{kJmol}^{-1})$
	$\frac{dF(X)}{dt} = nk_x^{1/n}(1 - F(X))\left[-\ln(1 - F(X))\right]^{\frac{n-1}{n}} \&$
Bainite growth	$X_B = (1 - X_{\alpha} - X_{MA})F(X)$
	$\ln(b) = C_1 + C_2 T + C_3 \left( C_{eff}^{\gamma_{rem}} * \ln(D_{eff}^{\gamma_{rem}}) \right)$
	$n=0.85, C_1=12.3, C_2=-0.019^{\circ}C^{-1}$ and $C_3=-15.405$
MA fraction	$X_{MA} = 0.03 X_{\alpha} + 0.095$
	$\Delta H_{ppt} = 0.94$ (P* <sup>-1/4</sup> + (P*))
	$\Delta H_{peak} = \frac{1}{2 \left[ 1 - \exp(-2P^{*1.25}) \right]^{1/2}} \left( 1 + \sqrt{1} \right)$
Precipitation strength	$P^{*} = \frac{1}{P_{p}} \int_{0}^{t} \exp\left(\frac{-Q_{ppt}}{RT(t)}\right) dt$
	$Q_{ppt}$ =300kJmol <sup>-1</sup> and $P_p$ =2.3×10 <sup>-15</sup> s
	$H = H_{Base} + \Delta H_{ppt}$
Material strength	$H_{Base} = H_{\alpha}X_{a} + H_{B}X_{B} + H_{MA}X_{MA}$
	$H_{\alpha}$ =122VHN, $H_{B}$ =710-0.81 $T_{Bs}$ VHN and $H_{MA}$ =406VHN