Laser Ultrasonics Measurement of Ferrite Formation during Stepped Cooling

by

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Abstract

Dual phase steels consist of soft ferrite and hard martensite phases and have been the most used advanced high strength steels in automotive design. The control of the phase transformations during processing is essential to achieve their optimal mechanical properties. Thus, sensor technology which can in-situ monitor phase transformation is of great interest. Recently, laser ultrasonics (LUS) has been gaining attention as an in-situ monitoring technique for the microstructure evolution. In this work, phase transformation kinetics of two low carbon steels with potential dual phase chemistries has been investigated using LUS for thermal treatments conditions relevant for run-out table cooling in hot strip mills.

Comparing the ultrasound velocity changes during continuous cooling with conventional dilatometry, it was confirmed that LUS can successfully monitor the phase transformation in the present steels. For the industrially relevant stepped cooling transformation tests, the fraction transformed concluded from ultrasound velocity agrees well with the ferrite phase fraction in the final microstructure as obtained from ex-situ metallography. The hardness has a linear relationship with the fraction transformed, which is consistent with literature data for dual phase steels. The evolution of the normalized velocity change for a given isothermal holding temperature can be described by the JMAK approach with the exponent and the rate parameters being consistent with the literature data for the austenite-to-ferrite transformation. Depending on the heat treatment conditions, banded or non-banded microstructures were observed. FEM analysis confirmed that the geometric configuration of phases has a negligible effect on the LUS phase transformation measurement, indicating the robustness of the LUS method.
Lay Summary

To meet current and future demands of society, advanced steels and their processing are continuously developed. For example, advanced high strength steels are the key material for the automotive industry enabling modern vehicle designs with reduced weight and improved safety. The key metallurgical tool for tailoring the properties of these steels is engineering the microstructure by using the transformation from the high-temperature phase austenite to low-temperature phases including ferrite and martensite. Dual-phase steels with a ferrite-martensite microstructure are widely used in modern vehicles because of their attractive combination of high strength with good formability. To control the volume fractions of ferrite and martensite, sensor technologies for in-situ phase transformation monitoring during industrial processing are of great interest. In this study, laser ultrasonics was used in a laboratory setting to in-situ monitor phase transformations in dual-phase steels for conditions relevant for hot rolling. The measurements were validated with conventional ex-situ metallography.
Preface

All research work including experimental design, heat treatment, metallography, hardness measurement, thermodynamic calculation, analysis and interpretation of the data and writing the thesis were carried out at the Department of Materials Engineering at The University of British Columbia, Vancouver by the author under the supervision of Professor Matthias Militzer.

Samples were cast, rolled, and machined at Nippon Steel Corporation, Japan. The Gleeble experiments were conducted by the author with training from Brian Tran and Sabyasachi Roy. Thermodynamic calculations presented in Chapter 5 were carried out using Thermo-Calc software with the TCFE7 database.
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List of Symbols

$[c]_{iso}$ stiffness matrix of an isotropic continuum GPa

$\Delta V_{i,\text{norm}}$ normalized velocity change -

$\Delta V_i$ velocity change mm/μs

$\Delta V_{\text{max}}$ maximum velocity change mm/μs

$\bar{A}$ amplitude of a Ricker wavelet mm

$\gamma_{\alpha/\gamma}$ intersection between the carbon component ray which passes through $c_0$ and the PLE/NPLE transition line at.%

$c_{OE}^{\alpha/\gamma}$ carbon concentration on ferrite under ortho-equilibrium at.%

$c_{PE}^{\alpha/\gamma}$ intersection between the carbon component ray which passes through $c_0$ and the para-equilibrium two-phase boundaries of $\alpha$ side at.%

$c_{PE}^{\gamma/\alpha}$ intersection between the carbon component ray which passes through $c_0$ and the para-equilibrium two-phase boundaries of $\gamma$ side at.%

$c_0$ bulk carbon concentration at.%

$c_{i,j}$ stiffness constant GPa

$d_{avg}$ average diagonal length mm

$d_1$ difference between instantaneous normalized dilation and normalized dilation for FCC for a given temperature -

$d_2$ difference between normalized dilation for BCC and the one for FCC for a given temperature -

$f$ ultrasonic frequency Hz

$F$ load applied in microhardness measurement kgf
\( F_{tr,\alpha,d} \) fraction transformed measured from dilatometry
\( F_{tr,\alpha,LUS} \) fraction transformed measured from LUS
\( f^\alpha_{NPLE} \) volume fraction of proeutectoid ferrite at the end of ferrite growth under NPLE condition
\( f^\alpha_{PE} \) volume fraction of proeutectoid ferrite at the end of ferrite growth under para-equilibrium condition
\( f_\alpha(t) \) fraction transformed after a holding time \( t \)
\( F_p \) peak frequency in Ricker wavelet
\( f_\alpha,\text{max} \) maximum fraction transformed of alpha phase
\( h \) sample thickness
\( k \) JMAK rate parameter
\( k_{fit} \) JMAK rate parameter for fitting model
\( k_{raw} \) JMAK rate parameter for each measurement
\( M_s \) martensite start temperature
\( n \) JMAK exponent
\( n_{ave} \) averaged JMAK exponent
\( n_{raw} \) JMAK exponent for each measurement
\( t \) Time
\( T_C \) Curie temperature
\( t_m \) time at the local minima for the \( m^{th} \) echo
\( t_n \) time at the local minima for the \( n^{th} \) echo
\( V_l \) instantaneous ultrasound velocity
\( V_L^{Hill} \) longitudinal ultrasound velocity under Hill assumption \( \text{mm/μs} \)

\( V_{st} \) initial ultrasound velocity \( \text{mm/μs} \)

\( V_{TE} \) ultrasound velocity obtained by two-echo technique \( \text{mm/μs} \)

\( V_1 \) instantaneous ultrasound velocity change between the instantaneous velocity and the extrapolated FCC velocity \( \text{mm/μs} \)

\( V_2 \) reference velocity change between the BCC velocity and the extrapolated FCC velocity for the given temperature \( \text{mm/μs} \)

\( V_S^{Hill} \) shear ultrasound velocity under Hill assumption \( \text{mm/μs} \)

\( x \) length from the center in Gaussian distribution \( \text{mm} \)

\( X_i \) atomic fractions of alloying elements

\( \alpha \) alpha phase

\( \gamma \) gamma phase

\( \varepsilon \) constant in Ricker wavelet

\( \rho \) Density \( g/cm^3 \)

\( \tau \) incubation time \( s \)
## List of Abbreviations

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
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<tbody>
<tr>
<td>AHSS</td>
<td>Advanced High Strength Steel</td>
</tr>
<tr>
<td>API</td>
<td>American Petroleum Institute</td>
</tr>
<tr>
<td>ASTM</td>
<td>American Society for Testing and Materials</td>
</tr>
<tr>
<td>BCC</td>
<td>Body Centered Cubic</td>
</tr>
<tr>
<td>BCT</td>
<td>Body Centered Tetragonal</td>
</tr>
<tr>
<td>BH</td>
<td>Bake Hardenability</td>
</tr>
<tr>
<td>CI</td>
<td>Confidence Interval</td>
</tr>
<tr>
<td>CTE</td>
<td>Coefficient of Thermal Expansion</td>
</tr>
<tr>
<td>DIFT</td>
<td>Deformation Induced Ferrite Transformation</td>
</tr>
<tr>
<td>DP</td>
<td>Dual Phase</td>
</tr>
<tr>
<td>EBSD</td>
<td>Electron Backscatter Diffraction</td>
</tr>
<tr>
<td>EQAD</td>
<td>Equivalent Area Diameter</td>
</tr>
<tr>
<td>FCC</td>
<td>Face Centered Cubic</td>
</tr>
<tr>
<td>FE</td>
<td>Finite Element</td>
</tr>
<tr>
<td>FEM</td>
<td>Finite Element Model</td>
</tr>
<tr>
<td>FSV</td>
<td>Future Steel Vehicle</td>
</tr>
<tr>
<td>HAZ</td>
<td>Heat Affected Zone</td>
</tr>
<tr>
<td>HSLA</td>
<td>High Strength Low Alloy</td>
</tr>
<tr>
<td>HV</td>
<td>Hardness in Vickers</td>
</tr>
<tr>
<td>IQ</td>
<td>Image Quality</td>
</tr>
<tr>
<td>JMAK</td>
<td>Johnson-Mehl-Avrami-Kolmogorov</td>
</tr>
</tbody>
</table>
LE    Local Equilibrium
LUMet Laser Ultrasonics for Metallurgy
LUS    Laser Ultrasonics
NCAP   New Car Assessment Program
NPLE   Negligible-Partition Local Equilibrium
PLE    Partition Local Equilibrium
TRIP   Transformation-Induced Plasticity
TTT    Time-Temperature-Transformation
UFG    Ultrafine Grained
ULSAB  Ultra Light Steel Auto Body
ULSAB-AVC Ultra Light Steel Auto Body Advanced Vehicle Concept
UTS    Ultimate Tensile Strength
YS     Yield Strength
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To my wife Minami, my daughter Yuzuki, and my family
Chapter 1: Introduction

Escalating concerns about human-induced green-house gases have driven global legislators to pass more stringent vehicle emission regulations through 2020, while considering further, aggressive targets for the next ten years[1]. On the other hand, safety regulations and rating systems such as European and United States New Car Assessment Program, or NCAP, have been increasingly stringent, resulting that consumers are demanding safe cars, and governments are responding with new tests and standards[2]. In this context, carmakers are searching for new materials and engineering capabilities to meet often conflicting requirements. For example, structural parts require materials characterized by high strength and stiffness, often achieved with greater thickness. However, fuel economy and emissions are reduced by weight reduction, e.g., when component thickness is decreased.

To address these issues, steel producers and carmakers have proposed an ultra light steel auto body (ULSAB) concept in 1998[3], followed by ULSAB-AVC (advanced vehicle concept) in 2002[4, 5] and the Future Steel Vehicle (FSV) in 2012[6]. These car concepts had been based on by making extensive use of advanced high strength steels (AHSS), including dual phase and transformation-induced plasticity (TRIP) steels, to meet the seemingly conflicting demands of the automotive industry to build vehicles with reduced weight and improved safety at low cost. These novel steels show for a given strength higher formability and crashworthiness as compared to conventional high strength low alloy (HSLA) steels[7, 8].

Dual phase steel is the 1st family of AHSS that is manufactured by taking advantage of a specific phase transformation. Dual phase steels are composed of soft ferrite and hard martensite. The volume fraction of martensite determines the strength level of this type of steels. Therefore,
the volume control of ferrite/martensite in the final microstructure on the production line is essential to obtain the desired mechanical properties.

There are two different processing routes for dual phase steels: cold rolling route and hot rolling route. Producing dual phase steels through the cold rolling route deals with austenite formation in the intercritical region and its transformation into martensite upon quenching. The phase fraction depends mainly on the intercritical temperature[8]. On the other hand, in the hot rolling route, the ferrite-martensite dual phase microstructure is produced from austenite decomposition through the so-called “stepped cooling” process[9] on the run-out table. Since the volume fraction of ferrite/martensite determines the mechanical properties of dual phase steels, the microstructure evolution in both processes is carefully controlled using physically based, empirical, or hybrid models, respectively. However, the process parameters and information, which is connected to the microstructure evolution and available in the process, are limited (e.g., surface temperatures at limited locations in the process, heating, rolling, and cooling schedules, and so on). Thus, process monitoring techniques which can obtain information directly related to the microstructure evolution is of significant interest.

Laser ultrasonics (LUS) is a technique that generates and detects ultrasound waves by optical means, enabling non-contact rapid in-situ evaluation of materials at high temperatures. The ultrasonic velocity, which is related to the elastic constants of the tested material, can be used to evaluate any metallurgical phenomenon that affects elastic components and density, such as recrystallization[10], phase transformation[10-16], and precipitation[17, 18]. Owing to its non-contact nature, being available from a meters-away distance, LUS has been gaining attention as an in-situ monitoring technique in steel manufacturing lines[19-22]. Laser ultrasonics has been reported to successfully measure phase transformations in steels during continuous cooling as
verified with conventional techniques including dilatometry[10, 16, 23, 24]. On the other hand, few studies have been done for steels to validate the quantitative relationship between the fraction transformed from LUS and the phase fraction using ex-situ materials characterization, whereas some studies have been reported for titanium[11, 12].

The primary aim of the present thesis is to examine the possibility of LUS as an in-situ monitoring technique for microstructure evolution of dual phase steels in the production line. In particular, the measurement of the phase transformation kinetics during stepped cooling on the run-out-table of hot strip mills using LUS is of interest. A particular series of thermal laboratory treatments are designed to generate different ferrite phase fractions within the final microstructures. Ferrite phase fraction in the microstructure is quantified via optical microscopy and compared with that concluded from LUS measurements. The hardness of tested specimens is also quantified and correlated with the measured fraction transformed. The change of ultrasound velocity has been used to develop a Johnson-Mehl-Avrami-Kolmogorov (JAMK) model to describe the ferrite formation kinetics. Further, a 2D-finite element model (FEM) simulation is conducted to examine the effect of the geometric configuration of phases within dual phase microstructure on ultrasound velocity.
Chapter 2: Literature Review

2.1 Dual Phase steels

2.1.1 Introduction

Dual phase (DP) steels were the first family of advanced high strength automotive steels which are manufactured by taking advantage of a specific phase transformation[25]. Dual phase steels consist of a ferritic matrix containing 10 to 40 volume percent of a hard martensitic second phase. They range in ultimate tensile strength (UTS) from 450 to 1200 MPa. Dual phase steels derive their strength from the martensite phase and their ductility from the ferrite phase. The volume fraction of martensite determines the strength level of this type of steels. Their important advantages are very high strain hardening capability and better formability as well as higher strain rate sensitivity than HSLA steel grades[26], which leads to the improved crashworthiness because of increased energy absorption. Other unique properties of dual phase steels include low elastic limit, high tensile strength, high uniform elongation, high total elongation and additional high gain of the yield strength (YS) during paint baking of the finished parts (bake hardenability, BH). Dual phase steels have been the most commonly used advanced high strength steels in cars[27]. The following sections describe the typical processing path, the general mechanical properties and the applications as well as the microstructure evolution of dual phase steels.

2.1.2 Processing path

There are two different processing routes for dual phase steels; cold rolling route and hot rolling route, as compared in Figure 2.1. In the cold rolling route, the starting material is thin cold-rolled sheets with typically a ferrite-pearlite microstructure. The sheet is heated up to the
intercritical temperature where both ferrite and austenite can exist. During “intercritical annealing”, ferrite-to-austenite transformation takes place. After the desired austenite fraction is achieved, the strip experiences a fast-cooling process to transform the austenite into martensite. Some of the austenite may transform into ferrite (epitaxial ferrite[28]), i.e., not all intercritical austenite necessarily transforms into martensite. The steel is either quenched to room temperature or the quenching is interrupted to galvanize the sheet in hot dip galvanizing lines. When processing in galvanizing lines, alloying elements are required to make sure that the “intercritical austenite” does not transform in the zinc bath.
Figure 2.1 Schematic drawing of (a) cold rolling and (b) hot rolling processing route to produce dual phase steels.
In the hot rolling route, the ferrite and martensite are created directly on the run-out table after finish rolling that typically takes place in the austenite region. First, the strip is cooled down from the finish mill exit temperature to just below the ferrite transformation start temperature by water cooling. Then, the water cooling stops, and the strip is air cooled in this temperature range to transform some austenite to ferrite. After the desired ferrite fraction is obtained, the strip is cooled again at high cooling rate to transform the remaining austenite to martensite. This typical cooling path on the run-out table is called “Stepped cooling”[9]. Thermomechanical processing in the intercritical region (intercritical rolling) and subsequent rapid cooling can be an option to produce dual phase microstructure in the hot rolling route as well[29]. This, however, results in strain hardened ferrite that impairs ductility of the ferrite matrix in the dual phase microstructure. Furthermore, fast cooling or quenching of deformed ferrite-austenite microstructure leads to a highly anisotropic banded or fibrous microstructure. Therefore, the deformation prior to run-out table cooling is usually performed in the fully austenite phase region. Since the ferrite formation is required to be within a short period of time, a faster generation of ferrite during cooling is promoted by low carbon content, heavy deformation of the austenite close to the Ar3 temperature and by refinement of the austenite grain size, i.e., by all factors reducing the hardenability of austenite and/or facilitating nucleation and growth of ferrite[30]. This approach promotes the enrichment of carbon into the remaining austenite by 5-6 times, ensuring the transformation to martensite during the subsequent cooling. Typically, additional alloying elements (higher Mn, but also Cr, Si, Mo etc.) are used in commercial DP steels in part to avoid bainite formation, resulting in mitigating the required degree of high cooling rates for the martensite transformation. For example, Avtar et al. reported that 0.06wt.%C-0.8wt.%Mn-1.0wt.%Si-0.4wt.%Cr-0.3wt.%Mo steel forms dual phase microstructures when coiled at the
temperature of 470 °C and below[31]. Recently, modern hot rolling mills have the capability to interrupt cooling at temperatures close to the “ferrite nose” in TTT diagrams to utilize the fastest formation of the target amount of ferrite, followed by fast cooling and coiling. In this case, the coiling temperature is set less than 200°C[32]. This process strategy allows using lean steel compositions which is more cost-effective. The cold rolling route can provide more options of thinner sheet thickness than the hot rolling route whereas the hot rolled dual phase steels can be produced more effectively with fewer processing steps.

2.1.3 **Properties and application of dual phase steel**

Dual phase steels offer high initial strain hardening rate as well as excellent elongation, leading to much higher UTS than conventional HSLA steels with similar YS[33]. In addition, dual phase steels exhibit higher strain rate sensitivity as compared to other high strength steels[34]. This contributes to the improved crash energy absorption (Figure 2.2)[35] which is of critical relevance for a variety of structural parts in vehicles such as inner and outer panels of autobody as well as safety cage components (B-pillar, floor panel tunnel, engine cradle etc.)[33, 36]. Examples of the hot rolled dual phase steels are wheel discs[37], wheel webs, longitudinal member and so on.
Figure 2.2: Relationship between average flow stress at a strain of 5% and the absorbed energy of a square tube which was axially crushed to simulate energy absorption in a car crash. Modified figure after [35].

A wide variety of UTS in dual phase steels is attributed to the change in volume fraction of the hard martensitic phase. Many studies confirmed a linear relationship between UTS and the volume fraction of martensite [39-41]. Figure 2.3 illustrates the effect of the volume fraction of martensite on the values of the yield strength, tensile strength, and their ratio (YS/TS ratio) in DP steels. It should be noted that an increase in both the tensile strength and the yield strength are accompanied by a significant increase in the YS/TS ratio. Numerous studies have been done to investigate the effect of the ferrite grain size on flow stress in dual phase steels [40, 42]. Son et al. produced ultrafine grained (UFG) dual phase steels containing different amounts of vanadium by equal angular pressing and subsequent intercritical annealing [43]. They showed that the strength of UFG dual phase steels was much higher than that of the coarse-grained counterpart, but
uniform and total elongation were not deteriorated. Mukherjee et al. applied deformation induced ferrite transformation (DIFT) on conventional DP600 steel chemistries containing 0.06wt.%C-1.9wt.%Mn-0.16wt.%Mo and varying Nb and V microalloying to produce UFG dual phase steels[44]. Ultrafine ferrite with the grain size of 1-2 μm was obtained by applying sufficient deformation (e.g. at the true strain of 0.6 or above) to an austenite microstructure with a grain size of 10-20 μm at 25-50°C above the austenite-to-ferrite transformation temperatures for the given cooling condition, followed by post-deformation cooling at cooling rates of around 100 °C/s to obtain the desired martensite volume fraction. In terms of industrial applicability of DIFT to produce UFG dual phase steels, they commented that the proposed processing schedule is beyond conventional hot strip mills, but that it provides an encouraging method to produce UFG dual phase steels without costly alloying elements.

![Figure 2.3: The effect of the volume fraction of martensite on the values of the yield strength, tensile strength, and their ratio in DP steels (0.1%C-2.1%Mn-0.7%Si-0.1%Mo)[39].](image-url)
Increased ductility at a given strength level is another advantage of dual phase steels. Total elongation in tension is the sum of the uniform elongation (strain before necking) and the elongation at the stage of localized deformation during necking that leads to failure. Fonstein et al. investigated the effect of volume fraction of martensite on uniform elongation of Cr-Mn-Si-B steels containing 0.07-0.15wt.% carbon[8]. They reported that uniform elongation decreases with increasing the volume fraction of martensite. Mazinani et al. investigated the effect of volume fraction and morphology (banded and more equiaxed) of the martensite phase on the mechanical properties by systematically varying the intercritical annealing temperature and the heating rate to this temperature for a 0.06wt.%C-2.0wt.%Mn-0.17wt.%Mo steel[45]. The experimental results show that the test samples with equiaxed martensite islands have around 10% higher uniform elongation compared to those with the banded morphology, whereas the martensite morphology has a negligible effect on yield and tensile strength.
Figure 2.4: Effect of volume fraction of martensite on uniform elongation of Cr-Mn-Si-B steels containing 0.07-0.15% carbon[8].

2.1.4 Microstructure evolution in hot-rolled dual phase steel

2.1.4.1 Ferrite transformation

Austenite can decompose into a variety of transformation products upon cooling, depending on the cooling rate, alloying elements, and their state as precipitates or in solution, prior austenite grain size and the state of stress in the austenite grains. Polygonal ferrite, pearlite, Widmanstätten ferrite, bainitic ferrite or martensite are the possible transformation products which can form.

Polygonal ferrite forms at high temperatures when austenite is cooled at a sufficiently slow cooling rate. Polygonal ferrite nucleates at austenite grain boundaries and grain corners, and grows into the grains[46]. It is characterized by very low dislocation densities and absence of substructure. At faster cooling rates austenite decomposition occurs at lower temperatures than for polygonal ferrite such that Widmanstätten ferrite forms. Its morphology is coarse elongated...
crystals of ferrite and it nucleates on prior austenite grain boundaries or from polygonal ferrite allotriomorphs[46, 47]. With increasing cooling rates, the austenite can transform to much finer ferrite crystals than described above and the whole microstructure consists of aggregates of plates of ferrite, separated by untransformed austenite, martensite or cementite[48]. Although the aggregates of plates are called bainite sheaves and the plates within each sheaf are the sub-units in the bainite microstructure, the most commonly used terms for the resulting ferritic microstructures are bainitic ferrite and acicular ferrite, respectively[46].

Since the solubility of carbon in ferrite is significantly lower than in austenite, long-range diffusion of carbon atoms from the newly formed ferrite to the parent austenite and the transfer of substitutional atoms across the austenite-ferrite interface control the growth of ferrite. Partitioning of substitutional alloying elements may cause significant reductions in growth rate of ferrite in low carbon steels[46]. Substitutional elements (M) such as Mn, Ni can considerably influence the kinetics since their diffusion coefficients are significantly lower than that of the interstitial carbon (C) atoms. For the quantitative prediction of alloying effects in real time scale, two models based on interfacial equilibrium conditions have been frequently used[49-52]: local equilibrium (LE) and para-equilibrium models. On the assumption of LE model, all the components of the α and γ phases have identical chemical potentials at the α/γ interface. The partitioning behaviors of M atoms in LE model can be classified into two modes. One is negligible-partition local equilibrium (NPLE), in which there is a spike of M accumulation in front of the α/γ interface, but no long-range partitioning of M atoms. Thus, in this mode, ferrite growth is controlled by carbon diffusion. The other mode is partition local equilibrium (PLE), also referred to as ortho-equilibrium, in which both M and C atoms redistribute between ferrite and austenite. Therefore, ferrite growth is controlled by the diffusion of M because the diffusion
coefficient of M is significantly lower than that of carbon, resulting in a slow growth rate that typically is not relevant in industrial processes (e.g., during run-out table cooling). Theoretically, a boundary, termed the PLE/NPLE transition line, separates the (α+γ) two-phase field into the NPLE and PLE regions (Figure 2.5). If an Fe-M-C ternary alloy which is fully austenitized at higher temperature is quenched into the NPLE region and subsequently isothermally held, proeutectoid ferrite will nucleate and then grow into the surrounding austenite under carbon diffusion control. Since long range partitioning of M does not take place under NPLE assumption, the average composition of the untransformed austenite simply follows a line parallel to the x-axis, which is known as the carbon component ray. However, once it reaches the PLE/NPLE transition line, M atoms are expected to start to redistribute, and then the ferrite growth will be controlled by slow M diffusion.
Figure 2.5: a) Schematic showing isothermal section of Fe-M-C ternary phase diagram, illustrating the relative positions of the PLE and NPLE regions. b) Corresponding M and c) carbon profile around the transformation interface when the bulk composition is below the PLE/NPLE transition line.

The para-equilibrium mode is a constrained LE where only carbon maintains the equality of the chemical potential at the $\alpha/\gamma$ interface whereas the alloying element M has the same atomic ratio to iron in ferrite and parent austenite. Ferrite growth is thus controlled by carbon diffusion without M redistribution and there is no M spike in front of the $\alpha/\gamma$ interface. Figure 2.6 illustrates schematically the para-equilibrium ($\alpha+\gamma$) two-phase region enclosed by the two para-equilibrium phase boundaries superimposed on the ortho-equilibrium two-phase region.
As more austenite transforms into ferrite, the carbon content within the remaining austenite increases because of the rejection of carbon from ferrite. Eventually, either the austenite reaches the eutectoid composition and pearlite formation can take place or the untransformed austenite becomes stabilized at the temperatures. As the temperature decreases, the driving force for austenite decomposition increases but the diffusivity of carbon decreases. Therefore, the ferrite transformation is the fastest at intermediate temperatures and the temperature for the fastest
ferrite transformation is called as ferrite “nose” in time-temperature-transformation (TTT) diagrams.

In practice, the selection of the holding temperature during stepped cooling for dual phase steel is dependent on the target mechanical properties and the alloying elements. Furthermore, since the available holding period during stepped cooling is limited to a number of seconds due to the industrial process window, it is common that the holding temperatures are selected for the condition where the ferrite formation is the fastest and the desired amount of ferrite fraction can be obtained during the available time periods\cite{9, 53-55}. Too high a holding temperature results in a decreased rate of ferrite formation, which leads to insufficient carbon enrichment producing bainite as second transformation product. Cooling below the optimum point may cause cementite formation at shorter timescales, resulting in a reduced volume of martensite, as the austenite grains have already a reduced carbon content\cite{54}. Although different steel chemistries have different optimal holding temperatures for the ferrite formation during stepped cooling on hot rolled dual phase steels, the holding temperatures range from 650°C to 720°C and the typical ferritic microstructure is polygonal ferrite\cite{9, 53-56}.

2.1.4.2 Martensite transformation

If the steel is quenched rapidly enough from the austenitic phase field, there is insufficient time for diffusion-controlled decomposition processes to occur, and the steel transforms into martensite, or in some cases, martensite with small amounts of retained austenite\cite{57}. Due to rapid cooling, the carbon atoms do not partition but instead are trapped in the octahedral sites of the body centered cubic (BCC) structure. The solubility of carbon in BCC structure at these temperatures is exceeded when martensite forms; hence, martensite has a body centered
tetragonal (BCT) structure[58]. This change in structure from face centered cubic (FCC) to BCT introduces shear in addition to an increase in volume. The temperature at which martensite starts to form is designated as the martensite start temperature ($M_s$). The $M_s$ reflects the amount of thermodynamic driving force required to initiate the shear transformation of austenite to martensite. The $M_s$ decreases significantly with increasing carbon content in Fe-C alloys and carbon steels. Carbon in solid solution increases the strength or shear resistance of the austenite and therefore, greater undercooling or driving force is required to initiate the shear for martensite formation in higher carbon steels[58]. It should be noted that $M_s$ depends on the composition of the remaining austenite and not the overall steel composition especially when prior transformation including ferrite, pearlite etc., has taken place. The temperature at which martensite starts to form can be calculated with the aid of empirical equations derived from a large body of experimental data[59]:

$$M_s(°C) = 539 - 423C - 30.4\text{Mn} - 17.7\text{Ni} - 12.1\text{Cr} - 7.5\text{Mo}$$ (Eq 2.1)

where the chemical composition is that of untransformed austenite in wt.%.

The morphology of martensite depends on the chemistry, especially the carbon content. Generally, in low carbon steels, the martensitic transformation product appears as laths. Each lath is a result of homogenous shear, and a bunch of parallel laths form a packet containing high dislocation density. Several packets exist within a single prior austenite grain. Plate martensite is observed in the case of carbon rich steel, commonly greater than 1 wt.% carbon and is characterized by non-parallel plates.

Since the ferrite transformation takes place prior to the formation of martensite in dual phase steels, the carbon atoms rejected from the new ferrite phase enrich in the remaining austenite. The effective carbon content in the remaining austenite is 5-6 times of the bulk after 0.8-0.85 of
volume fraction of ferrite has formed. Hence, the critical cooling rate for martensite transformation can be expressed as a function of the ferrite fraction[9].

### 2.1.4.3 Role of alloying elements

Alloying elements play various roles in microstructure evolution in dual phase steels. Carbon plays an important role to stabilize austenite during processing as well as to strengthen the martensite in the resultant microstructure. Typical dual phase steels contain 0.06-0.15wt.% C[60]. Manganese is known as austenite stabilizer promoting higher hardenability of austenite during cooling[8]. Typical dual phase steels contain 1.5-3.0wt.% Mn[60]. Increase in manganese moves the range of new ferrite formation to lower temperatures and slower cooling rates. Similarly, with increase in Mn the bainite transformation regions after full austenitization move to lower temperatures. Fonstein et al. report that, in 1wt.%Mn steels, however, the temperature range of bainite transformation during continuous cooling is approximately 100°C lower than in steels with 2wt.% and ~3wt.%Mn[8]. This can be explained by the higher volume fraction of new ferrite in low Mn steel and the corresponding enrichment of the remaining portion of austenite with carbon. Silicon is also a typical alloying element in dual phase steels and tends to inhibit carbide precipitation[61]; thus, the austenite can be enriched in carbon during ferrite formation, becoming significantly more stable, which is a beneficial effect for dual phase steels. Furthermore, combining with Mn, silicon can lower the critical cooling rate to obtain martensite because higher Si levels enhance Mn partitioning between ferrite and austenite[8, 62].

Aluminum as well as silicon are known as ferrite-forming elements, which facilitates ferrite formation during austenite decomposition, indirectly leading to the enhancement of the stability of the γ-phase, preventing the bainitic transformation and allowing for martensitic transformation
of the remaining austenite[8]. Molybdenum and chromium are also typical alloying elements in dual phase steels especially for the cold rolling route. They enhance the hardenability of austenite by suppressing pearlitic reaction, lowering the bainite transformation temperature. Molybdenum has among the common alloying elements the strongest effect on austenite stability. Irie et al. reported that its effect is 2.6 and 2 times stronger than that of manganese and chromium[63]. Adding molybdenum lowers the bainite start temperatures and/or extends the holding time for the start of bainitic transformation under quasi-isothermal conditions of zinc coating, thus allowing for avoiding of bainite in the final microstructure of coated cold-rolled DP steels.

2.2 Laser Ultrasonics

2.2.1 Introduction

Ultrasonics is a widely used non-destructive evaluation technique in industry for thickness gauging, flaw detection and to a limited but increasing extent for materials characterization[64]. Ultrasound velocities are related to the elastic constants of the tested material. Therefore, they can be used to evaluate any metallurgical phenomenon that affects elastic components and density, such as recrystallization[10], phase transformation[10, 11, 13-16, 65], and precipitation[17, 18]. The decrease in amplitude of the wave per unit length is called ultrasonic attenuation. Ultrasonic attenuation is mainly affected by grain scattering in anisotropic polycrystalline materials. The grain scattering is caused by the reflection and transmission of the wave at the grain boundaries and varies with the incident angle, the elastic mismatch between grains, and the size of the grains relative to the ultrasonic wavelength. Then, ultrasonic attenuation can be a measure of mean grain size.
Conventional contact ultrasonics is relatively simple to apply, the key component being a piezoelectric transducer often used for emission and reception. However, ultrasonics as it is usually applied, has several limitations, the main one being the need of direct contact or having a fluid medium for ultrasound coupling to the tested part. Further, there is a limited bandwidth of piezoelectric transducers and the difficulty of inspecting the parts with complex curvature. On the other hand, these limitations can be eliminated by the laser ultrasonic technique[64]. In this technique, since lasers are used to generate and detect ultrasonic pulses, this method allows one to perform measurements a meter-distant away from the probed material at elevated temperatures and to easily probe specimens of complex geometry. Due to the versatility stemming from the non-contact nature, laser ultrasonics provides a novel technique for real-time monitoring of microstructure evolution caused by different metallurgical phenomena such as recovery, recrystallization[10, 66-70], grain growth[69-73] and phase transformation[10, 11, 13-16, 65] during thermomechanical processing. The following sections describe examples of the application for velocity and attenuation monitoring.

2.2.2 Application of laser ultrasonics for materials characterization

2.2.2.1 Grain growth

The measurement of austenite grain size in steel via ultrasonic attenuation is one of the advantages of the laser ultrasonic technology in view of the challenges faced for the ex-situ austenite grain size measurements in low carbon steels. For the compressive wave propagating in the polycrystalline sample, there are generally three main sources of ultrasonic attenuation. The primary contribution is associated with the scattering by grains and is originated by the
dispersion of the wave energy due to the elastic mismatch between grains. The second
contribution arises from the diffraction of the pulse in the material. The third contribution of
attenuation is associated with the phenomena causing internal friction in the material.

Dubois et al. developed a first method to determine average grain size from ultrasonic
attenuation measurements for a series of C-Mn steels with carbon contents in the range of 0.003
to 0.17 wt.\%[14, 71]. They applied the two-echo technique. In this technique, Fourier transforms
on the first and second echoes for each ultrasound signal are applied, and then dividing the ratio
of these power spectra by-twice the sample thickness leads to the ultrasound attenuation
spectrum as a function of frequency, $f$. Figure 2.7-b represents the ultrasound attenuation
spectrum calculated from the two longitudinal echoes on A36 steel samples(0.17wt.\%C-
0.74wt.\%Mn).
They developed the temperature dependence of the ultrasonic attenuation for a single frequency (15-MHz) such as:

\[
\alpha_{15}(T) = \alpha_{15}(1100) - A_c \times (T - 1100)
\]  

(Eq 2.2)

where \(A_c = -2.26 \times 10^{-3}\) (dB/mm)/°C and \(\alpha_{15}(1100)\) is the attenuation at 1100°C.

Using the above relationship, the 15-MHz ultrasonic attenuation values measured at temperatures between 1050 and 1200 °C were converted to an ultrasonic attenuation at 1100 °C and compared with the austenite grain size measured by metallography (Figure 2.8). They also developed a parabolic fit of the grain size where the prediction is within approximately 10% of the average grain size larger than 100μm. The fit was calibrated for the 15-MHz ultrasonic attenuation, \(\alpha_{15}\), in terms of the equivalent area diameter (EQAD) for austenite grain size, and expressed as follows:
where $D$ is the austenite grain size (EQAD in $\mu$m) and $C = 8.31 \mu$m(dB/mm)$^2$.

Figure 2.8: Austenite grain sizes (EQADs) in A36 steel (0.17wt.%C-0.74wt.%Mn) measured by metallography vs. attenuation at 15 MHz converted to a temperature of 1100°C. The solid line is the parabolic fit[71].

Using Eq 2.3, laser ultrasonic attenuation values were converted to grain sizes. Figure 2.9 illustrates an example for a grain growth test at 1100 °C in A36 steel (0.17wt.%C-0.74wt.%Mn) comparing between the grain sizes from the laser ultrasonic technique and metallography. It is observed that the laser ultrasonic grain size agrees within approximately 10% of the metallographically measured values for each time point.
Figure 2.9: Comparison of austenite EQADs estimated from ultrasonic attenuation measurements with those measured by metallography at 1100°C in A36 steel samples (0.17wt.%C-0.74wt.%Mn) [71].

However, in the two-echo technique, the propagation distance for both echoes, i.e., the first and the second echoes, are different, resulting in a significant contribution from diffraction that is then difficult to extract from the grain scattering contribution. One alternative was therefore introduced to address this challenge by using the reference sample echo [70, 74]. In this second approach, 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 the propagation distance, here twice the sample thickness, to obtain an attenuation spectrum. The attenuation spectrum is fitted using the following equation:

\[ \alpha(f, T) = a + bf^n \]  

(Eq 2.4)
with a frequency dependent grain size contribution \( b \), and a frequency-independent parameter \( a \) which accounts for frequency-independent contributions such as those caused by internal friction as well as external factors associated with the variation of the laser source intensity and/or the reflectivity of the sample surface. \( n \) is taken to be 3[70, 74]. The grain diameter can be related to the parameter \( b \) using the following expression[70]:

\[
b = K(T)D^{n-1}
\]  
(Eq 2.5)

where \( K(T) \) is a parameter that depends on temperature and \( D \) is the average grain diameter.

The calibration curve shown in Figure 2.10 was generated for seamless tubes of various steel grades with a wide range of austenite grain sizes (20 \( \mu \)m to 300 \( \mu \)m), thickness (up to 30 mm) and temperature (900 to 1250°C). It can be seen that the fit applied to the data points is non-linear, suggesting that the scattering exponent is actually less than 3 in the studied steels.

![Figure 2.10: b parameter determined by the single-echo technique as a function of metallographic grain size for seamless tubes. Modified figure after [75].](image)

Figure 2.10: \( b \) parameter determined by the single-echo technique as a function of metallographic grain size for seamless tubes. Modified figure after [75].
This technique was used to study the evolution of austenite grain size in a laboratory steel (0.05wt.%C-1.9wt.%Mn-0.05wt.%Nb-0.5wt.%Mo) and X-80 microalloyed line pipe steels[72, 73, 76, 77]. Figure 2.11 verifies that the average grain size values determined by metallography and laser ultrasonics agree within the errors of measurements as shown for different holding times at 1050°C.

![Graph comparing grain sizes measured by laser ultrasonics and conventional metallography](image)

Figure 2.11: Comparison of measured average grain sizes by laser ultrasonics (lines) and conventional metallography (symbols) in 0.05wt.%C-1.9wt.%Mn-0.05wt.%Nb-0.5wt.%Mo steels at 1050°C.[73]

In terms of the on-line trial for the austenite grain size measurement, several studies were reported[19, 78]. Laser ultrasonics has been one of the methods for the on-line wall thickness measurement of seamless tubes. Using the same ultrasonic signals, Lévesque et al. investigated the austenite grain size and performed a comparison with the ones obtained by metallography on the same tubes after quenching[19]. Although the ultrasonic measurements and the metallography under on-line conditions are less accurate than in the laboratory, the ultrasonic grain sizes determined on-line have at least the same accuracy as those obtained from standard
metallography methods. Lim et al. installed a laser ultrasonics system in a hot rolling pilot plant and performed measurements of the ultrasound attenuation around 1100°C[78]. They reported that the austenite grain size calculated from the ultrasound attenuation was ASTM grain size No 6.2 (47.3μm in equivalent area diameter) whereas the ones measured by metallography was ASTM grain size No. 6.6 (41.2μm in equivalent area diameter).

2.2.2.2  Recrystallization

Recrystallization is usually associated with a change in texture. Starting from the deformation texture, the recrystallization texture develops. Using laser ultrasonics, Krüger et al. investigated the ultrasound velocity change during recrystallization in cold rolled samples of an Al-Mg alloy (AA5754) and an Al-Si-Mg-Cu alloy (AA6111)[68]. The recrystallized fraction, $X$, can be expressed as:

$$X = \frac{V - V_{coldworked}}{V_{recrystallized} - V_{coldworked}} \quad (\text{Eq 2.6})$$

where $V$, $V_{coldworked}$ and $V_{recrystallized}$ are the longitudinal sound velocities during, before and after recrystallization, respectively. Hutchinson et al. successfully monitored the progress of recrystallization in cold-rolled austenitic stainless steel sheets by laser ultrasonics[22]. During recrystallization, the overall texture change results in a significant decrease in ultrasound velocity and the recrystallization fraction was obtained by applying a lever rule on velocity vs. temperature curves. Militzer et al. studied the recrystallization kinetics of a 50% cold-rolled dual-phase steel (0.10wt.%C-1.86wt.%Mn-0.16wt.%Si-0.34wt.%Cr) during isothermal holding at various temperatures including 600, 625, and 650°C[77]. The recrystallization fraction
determined by the application of the lever rule method on the velocity data was consistent with the metallographically observations of recrystallization.

Austenite recrystallization in steels after hot deformation was first characterized by Smith et al.[79] and further pursued by other research groups[37, 67, 70, 76]. Smith et al. investigated the static recrystallization kinetics of hot deformed austenite in a steel with 0.19wt.%C-1.46wt.%Mn-0.44wt.%Si, combining the in-situ laser ultrasonic with stress relaxation measurements[79]. It was observed that the time change of measured ultrasound velocity reflects the metallurgical events during stress relaxation, i.e., recovery prior to recrystallization, as well as onset and completion of recrystallization. Sarkar et al. studied recrystallization of a low carbon complex phase steel (0.05wt.%C-1.9wt.%Mn-0.05wt.%Nb-0.50wt.%Mo) in the austenite region by applying uniaxial hot deformation on cylindrical samples. In this study, attenuation rather than velocity was used to identify start and finish time of recrystallization. Figure 2.12 illustrates the grain size evolution after deformation at 1000°C for various strain levels, i.e., 0.2, 0.3 and 0.4. Three distinct regimes during recrystallization and grain growth can be observed. Initially, the grain size remains approximately constant. During the occurrence of recrystallization, a significant decrease in grain size was measured and was followed by a stage of growth after completion of recrystallization. The end of the first step matches with the time for 5% recrystallization predicted by a softening model based on double hit tests[80]. In addition, the minimum grain size at the end of the second stage corresponds to the time of 95% recrystallization. Liang et al. also performed laser ultrasonic grain size measurement on C-Mn steels with two levels of niobium contents during recrystallization after hot deformation[67]. They reported that the onset and completion of recrystallization and the subsequent grain growth kinetics as estimated from laser ultrasonics corresponds to a physically-based model.
Figure 2.12: Effect of strain on grain size evolution after hot deformation of a model steel at the strain levels of 0.2, 0.3, and 0.4 (0.05wt.%C-1.9wt.%Mn-0.05wt.%Nb-0.50wt.%Mo). [70]

More recently, laser ultrasonic measurements were used for monitoring recrystallization and grain growth in cobalt-based superalloys [66, 81]. Keyvani et al. performed the real-time grain size measurement for an L605 cobalt-based superalloy (20wt.%Cr-15wt.%W and 10wt.%Ni) after hot deformation using laser ultrasonics [81]. Laser ultrasonic grain size evolution after deformation represents the aforementioned three regimes of recrystallization and matches with the ex-situ metallography within the measurement errors (Figure 2.13).
Figure 2.13: Evolution of the mean grain size in L605 cobalt-based superalloy after deformation at a) 1000°C, b) 1050°C, and c) 1100°C measured by laser ultrasonics (lines). Open circles represent the mean grain size obtained by metallography. The time scale refers to the time after the deformation [81].

2.2.2.3 Phase transformation

Dubois et al. measured the laser ultrasonic attenuation in hot-rolled A36 and interstitial free steels at temperatures between 500-1100°C [14]. They observed that the ultrasound attenuation reflects changes in grain size within microstructure during the phase transformation. Next, the same research group performed laser ultrasonic measurements of longitudinal velocity during continuous heating and cooling in the temperature range of 500–1000 °C for carbon steel samples [15]. They observed a reproducible hysteresis in dependence of the ultrasonic velocity versus temperature. (Figure 2.14) This hysteresis is attributed to the combined effects of the phase transformation and of the ferromagnetic-paramagnetic transition. The rate of change in velocity with temperature during heating suddenly diminishes at the Curie temperature.
Furthermore, in higher carbon content steels, the velocity behavior during cooling shows clearly start and end of phase transformation. It should be noted that the austenite decomposition which occurs below the Curie temperature can be captured by the ultrasonic velocity vs. temperature curve whereas the austenite formation which is expected above the Curie temperature is difficult to distinguish. They also investigated the longitudinal ultrasonic velocity in cold-rolled A366 steel samples (0.15wt.%C-0.60wt.%Mn) heated and cooled at 5°C/s three times (Figure 2.15). On the first heating, a drop of approximately 0.20mm/μs in ultrasonic velocity is observed between 800 and 900°C, where the structural phase transformation is expected whereas the velocity differences between the heating and cooling curves for the second and third curves are much smaller. This absence of drops in ultrasound velocity for subsequent heating can be attributed to the irreversible crystallographic orientation change of the textured grains caused by the phase transformation.
Figure 2.15: Ultrasonic velocity in the 500-1000°C temperature range measured in a A366 sample heated and cooled three times at 5 °C/s. For the sake of clarity, the velocity of the second and third thermal cycles are shifted downward by 0.2 and 0.4 mm/μs, respectively\[14\].

Krüger and Damm extensively investigated the longitudinal ultrasound velocity measured by the laser ultrasonic technique, comparing to dilatometry for the quantitative monitoring of austenite decomposition on low carbon steels\[16\]. They observed a similar temperature dependence of the ultrasound velocity with Dubois’s works and proposed applying the lever rule to the plot of velocity versus temperature, assuming that only two phases co-exist during the transformation progress. Figure 2.16 shows the general form of the temperature dependence of ultrasound velocity for austenite and ferrite. They mentioned that this method can make precise measurements if the velocities of the two phases are sufficiently different, i.e., below the Curie temperature. If the two phases have similar velocities, which can be observed above the Curie temperature, the lever rule cannot be used. It should be noted that the quantitative determination of each phase fraction will not be accurate if the parent or final phases are strongly textured due
to the aforementioned reason. Figure 2.17 compares the austenite fraction determined from velocity and dilatometry measurement during austenite decomposition. Both techniques show a similar pattern, although they differ quantitatively in the initial and final transformation stages.

Figure 2.16: Schematic showing of ultrasonic velocity for ferrite (solid line), austenite (dotted line) [16].

Figure 2.17: Austenite fraction determined by the velocity curve (solid line) and determined by dilatometry (squares) for a low alloy steel [16].
Several studies have been reported for laser ultrasonic phase transformation measurements of advanced high strength steels including transformation-induced plasticity (TRIP) steels. Krüger et al. measured the ultrasonic velocity on TRIP steel samples having different chemical compositions as well as different retained austenite fractions [65]. They reported a very good correlation between the ultrasonic velocity and the retained austenite measured by X-ray diffraction. This indicates that the laser ultrasonic technique is attractive especially for real-time measurements of TRIP steels in the laboratory and on production lines. In the same study, they also monitored the ultrasonic velocity during isothermal bainitic transformation at 300°C in a TRIP steel. Comparing to dilatometry, they reported some disagreements between the two techniques, such that dilatometry represents slightly slower transformation progress at initial stage and faster progress at later stage than laser ultrasonics. They mentioned the sensitivity of the dilatometric technique on the carbon enrichment of austenite as the reason for it. However, no metallographic measurement was provided in this work.

Militzer et al. investigated the microstructure evolution on thermal cycles for the heat affected zone (HAZ) in line pipe steels [10, 23]. They also reported the non-linear variation of ultrasonic velocity in ferrite during heating which has the abrupt change in slope around the Curie temperature corresponding to the magnetic transition (Figure 2.18-a). This transition is followed by a linear domain prior to the austenite formation. However, the transformation from ferrite to austenite around 900°C is not associated with a large velocity change. Upon cooling, starting from the high temperature, the velocity of all three samples increases initially linearly with decreasing temperature. At the transformation start temperature, the velocity leaves the linear temperature dependence of the austenite phase and gradually approaches the velocity value measured upon heating in the ferrite phase. Figure 2.18-b illustrates the quantitative analysis of
dilation measurement conducted simultaneously with the laser ultrasonics. For samples A and B, laser ultrasonics and dilatometer measurements are in agreement within the errors of measurements. For the lower transformation temperature of sample C, however, there is a systematic shift by about 40°C between the transformation temperatures obtained by the two techniques. This shift might be attributed to non-negligible temperature gradients during cooling conducted by helium gas.

Figure 2.18: a) Evolution of the ultrasonic velocity during heating (Solid lines) and cooling (Symbols) measured in sample A, B, and C of a low carbon line pipe steel (0.06wt.%C-1.49wt.%Mn-0.2wt.%Si-0.047wt.%Nb). b) Calculate fraction transformed during austenite decomposition upon cooling as obtained from dilatometer (Solid lines) and ultrasonic velocity (Symbols); Sample A, B: heated at 100°C/s up to 950°C, and 1150°C, respectively and held for 5min prior to a cooling at 3°C/s. Sample C: heated at 100°C to 1100°C and held for 5min prior to a cooling at 60°C/s[23].
Further, Militzer et al. simulated HAZ dual torch welding cycle in the API-X80 steel (0.06wt.% C - 1.65wt.% Mn - 0.24wt.% Mo - 0.034wt.% Nb - 0.012wt.% Ti - 0.005wt.% N), monitoring the laser ultrasonic velocity and the dilatometry onto the same cross-section, simultaneously[10]. Figure 2.19 displays the heat treatment cycle applied on the API-X80 steel and the measurement of fraction transformed from ultrasonic velocity and dilation during the cooling stages. The transformation temperatures from the two different techniques agreed within approximately 10 °C.

![Figure 2.19](image-url)  
Figure 2.19: a) Heat treatment cycle applied to the API-X80 line pipe steel to simulate dual-torch welding, b) Austenite decomposition for simulated HAZ dual torch welding cycle in the API-X80. Modified figures after [10].

Horn et al. applied Krüger’s ultrasonic velocity technique to investigate phase transformation kinetics at different quenching rates on state-of-art hot stamping steels containing complex
phases in different carburized conditions[24]. The effect of the carburization on the phase transformation kinetics was captured by laser ultrasonic measurements and supported by the corresponding hardness measurements. However, a quantification of the respective phase fraction by metallography was not undertaken.

More recently, several studies have been reported on laser ultrasonic measurement of the phase transformation kinetics in titanium alloys. Since conventional dilatometry to measure the phase transformation kinetics is challenging in titanium and its alloys, this technique has been gaining attention. Zamiri et al. monitored the phase transformation in a titanium alloy (Ti-6%Al-4%V) at high temperature using laser ultrasonic technique and observed a good sensitivity of the technique for such measurements[82]. Shinbine et al. measured the longitudinal velocity during cyclic heating and cooling in pure titanium using the laser ultrasonic technique and captured the phase transformation kinetics of the hcp to bcc structure[13]. They also performed electron backscatter diffraction measurements after the heat treatment and found that the texture before, and after, the heat treatment plays an important role in terms of the absolute values in ultrasonic velocity. Rodrigues et al. performed laser ultrasonic measurements to monitor the $\beta \rightarrow \beta+\alpha$ phase transformation kinetics in a Ti-5553 alloy during high temperature isothermal heat treatment[11]. The observed change in longitudinal wave velocities and the $\alpha$ phase fractions measured from electron microscopy were found to be in a linear relationship. Further, they reported that a partial TTT diagram built using the laser ultrasonics measurements and a JMAK modelling approach were consistent with the literature[83].
Chapter 3: Scope and Objective

The present research focuses on examination of the possibility of laser ultrasonics as an in-situ monitoring method of microstructure evolution for dual phase steels during stepped cooling. Two low carbon steels with potential dual phase chemistries and different Mn alloying are included in the proposed study that involves laboratory simulations with a Gleeble thermo-mechanical simulator equipped with a laser ultrasonics for metallurgy (LUMet) sensor. To accomplish this overall research goals, the following sub-objectives are designed:

1. To measure the phase transformation kinetics of dual phase steels during continuous cooling transformation tests by using LUMet and dilatometry.

2. To measure the phase transformation kinetics of dual phase steels during stepped cooling transformation tests by using LUMet.

3. To quantify the fraction of ferrite in the final microstructure via optical microscopy.

4. To determine the effect of Mn on the ferrite formation kinetics.

5. To measure the hardness in the final microstructure using a micro-Vickers hardness indenter.
6. To quantify the effect of the geometric configuration of phases within dual phase microstructures on laser ultrasonic velocity measurements using 2D finite element modelling simulations.
Chapter 4: Materials and Experimental Methodology

4.1 Materials

Two laboratory-grade steels were used for this study. The steels were cast as ingots and hot rolled into plates with the size of 750 × 130 × 25 mm at Nippon Steel Corp. (Chiyoda city, Tokyo, Japan). Chemical compositions of the two steels are summarized in Table 4.1. Compositions of the two steels are similar in terms of C, Si, Al. Two Mn content levels were investigated to determine the effect of Mn on phase transformation kinetics and microstructure. The \( \text{Ae}_3 \) temperatures were calculated using Thermo-Calc (TCFE7 database) and are reported in Table 4.1. Since Manganese is an austenite stabilizer, it reduces the \( \text{Ae}_3 \) temperature. [84]

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Al</th>
<th>N</th>
<th>( \text{Ae}_3 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.0%-Mn</td>
<td>0.096</td>
<td>0.48</td>
<td>1.97</td>
<td>0.0091</td>
<td>0.0015</td>
<td>0.030</td>
<td>0.0015</td>
<td>821.4°C</td>
</tr>
<tr>
<td>2.5%-Mn</td>
<td>0.094</td>
<td>0.48</td>
<td>2.45</td>
<td>0.0091</td>
<td>0.0015</td>
<td>0.029</td>
<td>0.0015</td>
<td>805.5°C</td>
</tr>
</tbody>
</table>

Steels were machined into sheet specimens of 60 × 10 × 3 mm from hot rolled plates for heat treatment as well as laser ultrasonic measurements (Figure 4.1). To not be affected by the macro-segregation of Manganese in the centre portion of the plate[85], the specimens were extracted from one fourth of the plate thickness. Note that the longitudinal direction of the specimens is perpendicular to the plate rolling direction. (Figure 4.2)
Figure 4.1: Sample geometry for Gleeble heat treatment.

Figure 4.2: Schematic drawing of the sample location cutting from the hot rolled plate

4.2 Processing set up

A Gleeble 3500 thermo-mechanical simulator (Dynamic System Inc., Poestenkill, NY, USA) equipped with a LUMet sensor was used for the heat treatment. To minimize surface oxidation, the sample chamber was evacuated down to a pressure of 0.002 Pa until cooling started. A set of low force jaws was used to conduct thermal treatments. A pair of Type-K thermocouples were spot-welded onto the centre of the length of specimens to monitor the temperature. Sample placement and set up of heat treatment in the Gleeble are shown in Figure 4.3.
The LUMet system is attached to the back door of the Gleeble chamber. The principles of the LUMet technique are shown schematically in Figure 4.4. The system uses two types of lasers, a generation laser, and a detection laser. The generation laser, a frequency-doubled Q-switched Nd: YAG laser with a wavelength of 532 nm, a duration of 6 ns and a maximum energy of 72 mJ, which results in an instantaneous power of 12 MW, generates a broad band ultrasonic pulse by ablation of a very thin layer of the sample surface. The generated pulse travels through the sample, reflects at the back wall, and returns to the generation surface. The arrival of the pulse generates a small surface displacement that is recorded with the detection laser, a frequency-stabilized Nd: YAG laser which has a wavelength of 1064 nm and a pulse duration of 90 μs. The detection laser light reflected on the sample surface is modulated by the surface displacement caused by the arrival of the ultrasound wave and demodulated in a photo-refractive crystal using a two-wave mixing interference method[86]. Both generation and detection laser beams are
collinearly aligned at the centre length of the sample on the opposite side of the thermocouple junction. The angle between the laser beam and the sample surface is 20° which is measured with a digital angle meter. The pulse generation rate can be as high as 50 Hz and can be adjusted during the experiments. Furthermore, the number of pulses per second is chosen in a way that the resulting surface damage does not affect the ultrasonic velocity measurement. The characteristics of the ultrasound pulse are representative of the average properties of the material over a volume created by the surface of the laser spot (diameter of 2 mm) multiplied by the sample thickness (here 3 mm).

![Figure 4.4](image.png)

**Figure 4.4:** Schematic showing of principles of the LUMet technique.

### 4.3 Thermal treatment

#### 4.3.1 Continuous Cooling Transformation Tests

Thermal cycles were designed to study the kinetics of austenite decomposition at constant cooling rates to compare the phase transformation measurements between the dilatometry and the laser ultrasonic velocity technique. Militzer et al. reported the agreement between the fraction transformed measured by conventional dilatometry and through the lever rule analysis of the velocity changes[10]. In that work, the cooling rate of 150 °C/s was selected and the austenite decomposition occurs in the range between 500 and 400 °C, where ultrasound velocities in
ferrite and austenite are significantly different. In the present study, to capture the austenite-to-ferrite phase transformation, cooling rates of 3 and 10 °C/s were selected.

Figure 4.5 shows the thermal cycle designed for continuous cooling transformation testing. The samples were heated at 50 °C/s to 950 °C, where they were isothermally held for 120 s. The samples were then cooled down to room temperature at the cooling rates of 3 and 10 °C/s. The cooling rate of 3 °C/s was attained under vacuum, but the cooling rate of 10 °C/s was achieved with helium gas. The LUMet system acquired ultrasound signals during heating and cooling. The sampling frequency during heating is 50 Hz and the one during cooling is 5 Hz.

4.3.2 Stepped Cooling Transformation Tests

This type of thermal cycles was designed to simulate the cooling path to produce the hot rolled dual phase steels on the run-out table of a hot strip mill. Figure 4.6 and Figure 4.7 show the thermal cycle designed for stepped cooling transformation testing. As is the case with continuous cooling transformation testing, the samples were heated at 50 °C/s to 950 °C, where they were
isothermally held for 120 s. To simulate the above cooling path, the samples were cooled down to the target temperatures at cooling rate of a 10 °C/s without helium to achieve uniform cooling within the sample and isothermally held there for the specific holding times, followed by rapid helium quenching. Based on the preparatory testing, it was confirmed that the cooling rate of a 10°C/s is possible without He if there is no phase transformation. In this study, two sets of stepped cooling transformation tests were conducted for each chemistry. For the first set, the samples were isothermally held at different target temperatures for 600 seconds. For the second set, the samples were isothermally held at a specific temperature of 650 °C for different holding times.

For the 2.0%Mn steel, the target temperatures of 700, 675 and 650 °C were selected for the first set and the holding times of 10, 20, 60 and 600 s for the second set (Figure 4.6). For the 2.5%Mn steel, the target temperatures of 650, 625, 600 and 575 °C were selected for the first set and the holding times of 600, 1800 and 7200 s for the second set (Figure 4.7). The LUMet system acquires ultrasound signals during cooling only until the isothermal holding is completed. The sampling frequency was changed by stages. For initial cooling, 5 Hz were selected. For isothermal holding, 1 Hz were selected for all the testing sets for 2.0%Mn and the first set for 2.5%Mn steels. For the second set for the 2.5%Mn steel, the LUMet signal acquisition was conducted at 5 Hz every 5 min for 1800 s holding condition and every 10 min for 7200 s. Cooling rate of 10 °C/s for initial cooling were not maintained once the transformation started due to the heat of transformation.
Figure 4.6: Schematics of thermal treatments for stepped cooling transformation testing on 2.0%Mn steel. a) different target temperature tests, b) different holding time tests.

Figure 4.7: Schematics of thermal treatments for stepped cooling transformation testing on 2.5%Mn steel. a) different target temperature tests, b) different holding time tests.
4.4 Dilatometry

The molar volume of austenite and the transformation products are different, which results in volume change during austenite decomposition. This change in volume causes a change in the dimension of the sample and is measured by a contact type dilatometer. As shown in Figure 4.3, the dilatometer is placed at the centre of the length of the sample on the same cross-section where the thermocouple is spot-welded. Figure 4.8 represents an example of the dilation data obtained during cooling, where the relative change of dilatometer is plotted against temperature. The relative change in dilatometer is calculated by normalizing the change in the width with respect to the initial width of 10 mm. The curve can be divided into two linear segments; one at higher temperature is due to thermal contraction of the austenite phase, and the other one at lower temperature is a result of the thermal contraction of the daughter phases. Austenite decomposition occurs in the non-linear region between these two linear segments.
The lever rule (ASTM 1033) was used to calculate the fraction transformed from the dilation data. Figure 4.8 shows how the lever rule is applied. At any given temperature, the fraction transformed is given by

\[ F_{tr,\alpha,d} = \frac{d_1}{d_2} \]  

(Eq 4.7)

where \( d_1 \) and \( d_2 \) are determined as shown in Figure 4.8. Applying the lever rule over the complete temperature range results in fraction transformed as a function of temperature, which is shown in Figure 4.9. To ensure the reliability of the tests, experimental thermal expansion coefficients (CTE) were compared against reported literature CTE values, i.e., \( 22.5 \times 10^{-6} \, ^\circ C^{-1} \) and \( 14.8 \times 10^{-6} \, ^\circ C^{-1} \) for austenite and ferrite, respectively. Tests with CTE values within a \( \pm 10\% \) margin were deemed valid.
4.5 Laser Ultrasonic analysis

Ultrasonic velocity is related to density and bulk elastic constants of the probed materials. It can be used to evaluate metallurgical phenomena that affects elastic constants and density, such as phase transformations[14-16].

The longitudinal ultrasound velocity is measured by use of two successive echoes. In the present study, this velocity was calculated using the CTOME analysis software [87] employing the so-called two-echo technique, in which two echoes in a single waveform were used for the calculation of the time delay between echoes. In this case, the velocity is given by:

$$V_{TE} = \frac{2(m - n)h}{(t_m - t_n)}$$  \hspace{1cm} (Eq 4.8)
where $V_{TE}$ is velocity obtained by the two-echo technique, $h$ is the sample thickness, $m$ and $n$ are the index numbers of the $m^{th}$ and $n^{th}$ echoes and $(t_m-t_n)$ is the time difference between local minima of the $m^{th}$ and $n^{th}$ echoes. In the present study, the 2$^{nd}$ and the 3$^{rd}$ echoes are used for the above calculation ($m=2$, $n=3$) (Figure 4.10) because the 1$^{st}$ echo is typically disturbed by the generation signal and the 4$^{th}$ and subsequent echoes have relatively low amplitudes caused by the attenuation.

![Diagram of ultrasound signal](image)

Figure 4.10: Example of ultrasound signal

Figure 4.11 shows an ultrasound velocity dependence on temperature for various phases. BCC-phase, including ferrite, bainite and martensite, has a non-linear relationship between velocity and temperature below the Curie temperature due to the transition from ferromagnetic to paramagnetic state, and, above the Curie temperature, the ultrasound velocity decreases linearly with temperature. On the other hand, FCC-phase, or austenite, always has a linear relationship between ultrasound velocity and temperature. The measured velocity curve under phase transformation is located between BCC and FCC curves below the Curie temperature. Therefore, the fraction transformed for a given temperature and measured velocity can be obtained via the
lever-rule[16]. Figure 4.11 shows how the lever rule is applied. At any given temperature, the fraction transformed is obtained by:

\[ F_{tr, \alpha, \text{LUS}} = \frac{V_1}{V_2} \]  \hspace{1cm} (Eq 4.9)

where \( V_1 \) is the instantaneous velocity change between the instantaneous velocity and the extrapolated FCC velocity during cooling and \( V_2 \) is the reference velocity change between the BCC velocity during heating and the extrapolated FCC velocity for the given temperature.

In this study, the BCC velocity curve is obtained from the velocities measured during heating. To minimize the surface damage due to ultrasound firing during heating prior to the subsequent cooling paths, three BCC velocity curves are measured and averaged to give a general reference curve for each steel because the heating process are common for each thermal treatment. The FCC velocity curve is extrapolated from the velocity measured during cooling at higher temperature for each measurement. The sample thickness was measured with a digital caliper at room temperature before the test and, by using the variation of the sample width with temperature in the dilation measurement, it was corrected at each tested temperature for thermal expansion[87]. The current experimental set-up measures the travel time of ultrasound to calculate the ultrasound velocity using the corrected thickness in the aforementioned way. To compensate the sample-by-sample thickness measurement error, the reference BCC velocity curve is shifted to match the ultrasound velocity curve measured during cooling in the high-temperature range.
Figure 4.11: Schematic drawing of the ultrasound velocity dependence with temperature obtained during continuous heating and cooling. $T_C$ and $T_{Ae3}$ are the Curie temperature and the Ae3 temperature for the 2.0%-Mn steel.

4.6 Microstructure characterization

4.6.1 Sample preparation

Sheet samples were sectioned at the centre of the length where the thermocouples were spot-welded. Samples for optical microscopy were hot mounted in a phenolic resin using a compressive hot mounting press. Each sample was ground and polished on a Buehler polishing machine. Grinding was carried out on progressively finer silicon carbide paper with grid size 240, 400, 600, 800, and 1200 with continuous water flow for lubrication. The samples were then polished using 6 μm followed by 1 μm diamond suspension and a lubricating agent. The samples were washed with liquid soap under running water, and then dried with denatured ethyl alcohol after each stage of grinding and polishing to avoid contamination.
For optical microscopy, the polished sample was then etched with 2% Nital solution for 15-20 seconds, rinsed with water and denatured ethyl alcohol and then dried.

4.6.2 Characterization by optical microscopy

A Nikon EPIPHOT 300 series inverted microscope attached with a digital camera was used to capture optical images. The multi-layer capture feature in the image capturing software, Clemex, was used to obtain focused images. Images were taken at magnifications of 200 and 500 times, respectively. The manual point counting method according to ASTM E562-89 [88], was applied using a square grid superimposed on micrographs. The ratio of the grid points falling onto a given phase to the total number of grid points provides the phase fraction. Figure 4.12 shows an example of a mixture of polygonal ferrite and martensite etched with 2% Nital. Black circles mark intersections that fall on regions comprising ferrite which are white. The yellow arrows indicate regions that fall on phase boundaries or mixed regions and might be characterized differently by different researchers. A minimum of 9 micrographs each containing 330 grid points was quantified for each sample to ensure statistic measurements of the present transformation products.
Figure 4.12: 2% Nital-etched micrograph showing a mixture of ferrite and martensite superimposed grid for point counting method. Black circles mark intersections that fall on onto ferrite; yellow arrows indicate phase boundaries and mixed regions.

4.7 Hardness measurement

Hardness was measured using a Micro-Vickers hardness indenter under 1kgf load, applied for a 10 second dwell time. The diagonal length of the indent made by the pyramid-shaped diamond indenter was measured. The hardness value is calculated from the average diagonal length according to ASTM E384 [89]

\[
Hardness (Hv) = \frac{1.8544 F}{d_{avg}^2}
\]  
(Eq 4.10)

where, \( F \) is the load applied in kgf and \( d_{avg} \) is the average diagonal length in mm. A minimum of 2.5 diagonal length was maintained between the indents to avoid incorrect hardness measurements because of work hardening caused by a previous indent.
Chapter 5: Experimental Results and Discussion

5.1 Continuous Cooling Transformation Tests

Figure 5.1 displays the relationship between ultrasound velocity and temperature in the 2.0\%Mn steel samples during heating and cooling. During heating the ultrasound velocity decreases as temperature increases. The velocity decreases quickly as temperature approaches 740 °C. Above this temperature, the relationship between ultrasound velocity and temperature gradually becomes linear. This characteristic change in the velocity-temperature curve has been reported in previous studies [15, 16] and is attributed to the Curie temperature, i.e. the temperature for the magnetic transition from ferromagnetism to para-magnetism. The Curie temperature in pure iron is reported to be at 770 °C [90]. Nishizawa et al. [91] proposed the following equation taking into account the effect of alloying elements on the Curie temperature such that

\[ T_C(K) = 1043 + 1020 X_{Co} - 1250 X_{Be} - 1500 X_{Mn} - 200 X_{Mo} - 400 X_{Ni} - 400 X_{Zn} - 1800 X_p - 100 X_{Cu} \]  

(Eq 5.1)

where \( T_C(K) \) is the Curie temperature, \( X_i \) are the atomic fractions of alloying elements. For the 2.0\%Mn steel, the Curie temperature is then calculated as 740 °C, which is consistent with the temperature where the prominent velocity inflection is observed in the present study. It should be noted that there is no visible velocity change for the ferrite-to-austenite transformation above the Curie temperature. Although the Ae3 temperature of 821°C is an important reference temperature, the velocity continuous to decrease monotonously.

On the other hand, during cooling, the velocity increases linearly as temperature decreases to the austenite decomposition start temperatures. At the cooling rate of 3 °C/s, the velocity starts to
increase faster around 680 °C with decreasing temperature, whereas at the cooling rate of 10 °C/s, this occurs at about 650 °C. The red heating curve reflects 100% ferrite whereas the dashed line, which is the low temperature extrapolation of the fully austenite velocity at higher temperature, reflects 100% austenite. Then, the cooling curve can be considered as the mixture of these two phases and the lever rule can be applied to the fraction transformed[10, 16].

Figure 5.1: Ultrasound velocity as a function of temperature in a 2.0%Mn steel sample heated at 50 °C/s (red line) and cooled at 3 (blue line) and 10°C/s (green line). Dashed line indicates the ultrasound velocity in FCC extrapolated from high temperatures.

Figure 5.2 shows the fraction transformed as a function of temperature in 2.0%Mn steel specimens cooled at 3 and 10 °C/s, calculated from both dilatometry and ultrasound velocity measurement. It can be observed that both techniques give very similar phase transformation kinetics. Owing to transformation temperatures being sufficiently lower than the Curie
temperature, the phase transformation kinetics can be captured by the laser ultrasound velocity technique in the present steel. Similarly, for the 2.5%Mn steel, the transformation start temperatures at the cooling rates of 3 °C/s and 10 °C/s are measured around 640, and 610 °C, by the laser ultrasonic velocity technique. The dilatometry measured them as 643 and 604 °C, respectively. This indicates that the laser ultrasonic velocity technique for monitoring the phase transformation kinetics can be applied to the other steel as well.

Figure 5.2: Fraction transformed as a function of temperature in 2%Mn steel samples cooled at 3 °C/s (blue) and 10 °C/s (green). Solid lines are obtained by dilatometry, symbols from the laser ultrasound velocity technique.

5.2 Stepped Cooling Transformation Tests

5.2.1 Laser ultrasonic measurements

The proeutectoid ferrite within a dual phase microstructure, which is the main objective to be measured by laser ultrasonics in this study, is formed during a stepped cooling path on the run-
out table of a hot strip mill. The ultrasound velocity was measured up to the exit of isothermal holding in the stepped cooling paths with different holding temperatures. Figure 5.3 and Figure 5.4 show the ultrasound velocities as a function of temperature during stepped cooling for a 1st set of stepped cooling transformation tests in 2.0%Mn and 2.5%Mn steels. Figure 5.3 and Figure 5.4 are the representative velocity curve for each condition out of three repeat tests. During initial cooling down to the target holding temperatures, the velocity increases linearly as temperature decreases. At 650 °C for the 2.0%Mn and 575 °C for the 2.5%Mn steels, the velocity deviates from the straight FCC-velocity curve extrapolated from higher temperatures before reaching the target temperatures. This indicates that the phase transformation already started above the target temperatures. During isothermal holding at different holding temperatures, the velocity increases with time.

The velocity differences between the alpha and gamma at 550°C are 0.28 mm/μs for the 2.0%Mn steel, 0.23 mm/μs for the 2.5%Mn steel, respectively. It could potentially be related to different transformation products as well as texture difference. Further studies are required for clarification.
Figure 5.3: Ultrasound velocity as a function of temperature during stepped cooling with holding temperatures of 700, 675, and 650 °C for the 2.0%Mn steels.
Figure 5.4: Ultrasound velocity as a function of temperature during stepped cooling with holding temperatures of 650, 625, 600, and 575 °C for the 2.5%Mn steels.

Figure 5.5 and Figure 5.6 display the fraction transformed obtained by the ultrasound velocity technique for the 1st set of stepped cooling transformation tests on 2.0%Mn and 2.5%Mn steels, respectively. For both steels, the fraction transformed increases more quickly and reaches a higher value as the holding temperature decreases. This behavior is also observed in the velocity evolution with time. In terms of the start of the phase transformation, the transformation start times, which were quantified at 5% transformed, were 2.6 and 2.0 s for 700 and 675 °C in the 2.0%Mn steel, respectively. In the 2.5%Mn steel, they were 5.2, 1.0, and 0.1 s for 650, 625, and 600 °C, respectively. For 650°C holding in the 2.0%Mn steel, 10% transformed was already achieved when reaching the target temperature, whereas 16% transformed was achieved for 575 °C in the 2.5%Mn steel.
Figure 5.5: Fraction transformed during isothermal holding at 700, 675, and 650 °C for the 2.0%Mn steel.

Figure 5.6: Fraction transformed during isothermal holding at 650, 625, 600, and 575 °C for the 2.5%Mn steel.
Based on the dilatometry during continuous transformation tests, 10% transformed is achieved around 600 °C in the 2.0%Mn steel at the cooling rate of 10 °C/s, which is inconsistent with the results for the 650 °C holding condition. This might be attributed to whether helium gas is used for the initial cooling. On continuous cooling transformation tests at 10 °C/s, the helium gas is always blown on the same surface which the thermo-couple is attached to whereas no gas is applied for the initial cooling during stepped cooling transformation tests. Since the monitored surface on continuous cooling transformation tests has the lowest temperature through thickness and the Gleeble system introduces electric current to control the temperature based on the information from the thermo-couple, the helium gas introduces a steeper thermal gradient within the specimen through thickness than no gas condition does. Thus, the actual average temperature through thickness when the transformation start is detected might be higher than the measured surface temperature during continuous cooling transformation tests. As the measured velocity or the subsequent fraction transformed obtained from the laser ultrasonic technique are representative of the average properties of the material over a volume created by a laser spot (2mm diameters) multiplied by the thickness, the temperature homogeneity should be considered during the measurement. Then, the straight FCC-velocity curve extrapolated from higher temperatures should be created by the measurement where homogenous cooling is achieved. In the present study, in order to simulate the application of the laser ultrasonic velocity technique for monitoring the phase transformation kinetics in real production line, the temperature homogeneity is maintained by cooling without helium gas up until the exit of the isothermal holding.

Figure 5.7 and Figure 5.8 illustrate the fraction transformed obtained by ultrasound velocity technique for the 2nd set of stepped cooling transformation tests for 2.0%Mn and 2.5%Mn steels,
in which the holding temperature is fixed as 650°C and the holding time is varied. The transformation curves for different holding times were reproducible and consistent for each steel. The final fractions transformed at the exit of the isothermal holding were 0.42, 0.57, and 0.69 for 10, 20, and 60s, respectively, in the 2.0%Mn steel whereas they were 0.44, and 0.40 for 1800, and 7200 s in the 2.5%Mn steel. For both chemistries, the scatter of data points grows with time because of the surface damages due to laser firing. The unrealistic drop of the fraction transformed at the later stages for the longest holding times for the 2.5%Mn steel may be attributable to the surface damage.
Figure 5.7: Fraction transformed obtained from the ultrasound velocity technique at the holding temperatures of 650 °C for 10, 20, 60, and 600s in the 2.0%Mn steel.

Figure 5.8: Fraction transformed obtained from the ultrasound velocity technique at the holding temperature of 650 °C for 600, 1800, and 7200s in the 2.5%Mn steel.
Table 5.1 and Table 5.2 represent the fractions transformed after 10, 30, 100, 300, and 600 s of the isothermal holding at each temperature in both steels, respectively. Here, the sampling (statistical) uncertainty was expressed as the 95% confidence interval, which was calculated from three experiments for each holding time at each temperature in both steels. For 10, 30, 100, 300s holding, single values of the fraction transformed after the target holding time were used from each test. For 600s holding, the fraction transformed for the last 10 second were averaged to calculate the representative value from each test.

Table 5.1: The fraction transformed after the isothermal holding in the 2.0%Mn steel

(Ave ±95%CI)

<table>
<thead>
<tr>
<th>Holding temp</th>
<th>10s</th>
<th>30s</th>
<th>100s</th>
<th>300s</th>
<th>600s</th>
</tr>
</thead>
<tbody>
<tr>
<td>700°C</td>
<td>0.17 (±0.01)</td>
<td>0.38 (±0.06)</td>
<td>0.50 (±0.07)</td>
<td>0.53 (±0.02)</td>
<td>0.54 (±0.04)</td>
</tr>
<tr>
<td>675°C</td>
<td>0.32 (±0.02)</td>
<td>0.49 (±0.01)</td>
<td>0.57 (±0.01)</td>
<td>0.58 (±0.12)</td>
<td>0.62 (±0.07)</td>
</tr>
<tr>
<td>650°C</td>
<td>0.43 (±0.05)</td>
<td>0.63 (±0.05)</td>
<td>0.72 (±0.05)</td>
<td>0.75 (±0.06)</td>
<td>0.75 (±0.10)</td>
</tr>
</tbody>
</table>
Table 5.2: The fraction transformed after the isothermal holding in the 2.5%Mn steel

(Ave ±95%CI)

<table>
<thead>
<tr>
<th>Holding temp</th>
<th>Holding time</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10s</td>
</tr>
<tr>
<td>650°C</td>
<td>0.06 (±0.09)</td>
</tr>
<tr>
<td>625°C</td>
<td>0.17 (±0.01)</td>
</tr>
<tr>
<td>600°C</td>
<td>0.22 (±0.03)</td>
</tr>
<tr>
<td>575°C</td>
<td>0.37 (±0.05)</td>
</tr>
</tbody>
</table>

5.2.2 Metallographic analysis

Figure 5.9 shows the optical micrographs for the 1st set of stepped cooling transformation tests on the 2.0%Mn steel. The microstructure consists of the light white region, which is considered as the proeutectoid ferrite, and the dark grey region, which formed during quenching after isothermal holding and is assumed to be martensite. Generally, the area of ferrite region gets larger with decreasing holding temperatures. At 700 and 675 °C holding conditions, banded microstructures are observed where the ferrite grows along the horizontal direction which corresponds to the rolling direction. On the other hand, at the 650 °C holding condition, the ferrite banding cannot be observed. Figure 5.10 displays the optical micrographs for the 1st set of stepped cooling transformation tests on the 2.5%Mn steel specimens for the holding temperatures of 650, 625, 600, and 575°C. Similarly, it comprises the white ferrite region and the dark grey martensite, and the area of the ferrite region within the microstructure increases with
decreasing holding temperatures from 650°C to 600 °C. The banded microstructure is also
observed. The microstructure obtained at the 575°C holding condition is finer than for other
conditions and no banded microstructure is observed. The contrast between the transformation
products formed during the isothermal holding and the subsequent quenching is less significant
than for the other conditions. The acicular products at 575°C can be categorized as bainitic
ferrite. However, it is difficult to perform the quantitative measurement by applying the point
counting method to differentiate the phases for this condition.

Figure 5.9: Optical microstructure for the 2.0%Mn steel after the 1st set of stepped cooling
transformation tests at different holding temperatures (a: 700°C, b: 675°C, c: 650 °C).
Figure 5.10: Optical microstructure for the 2.5%Mn steel after the 1\textsuperscript{st} set of stepped cooling transformation tests at different holding temperatures (a: 650°C, b: 625°C, c: 600 °C, d: 575°C).

Figure 5.11 shows the optical micrographs for the 2\textsuperscript{nd} set of stepped cooling transformation tests on the 2.0%Mn steel samples with a holding temperature of 650 °C. It can be observed that the area of the proeutectoid ferrite increases with increasing holding time and that the ferrite nucleates along the prior austenite grain boundaries and gets thickened with time. Figure 5.12
displays the optical micrographs for the 2nd set of stepped cooling transformation tests on the 2.5%Mn steel specimens for the same holding temperature of 650 °C. Similarly, the area of the proeutectoid ferrite increases with increasing holding time. It can be observed that the ferrite banding structure seems to get thickened with holding time. Compared with the 625 and 600 °C holding conditions, the microstructures for the longer holding conditions at 650°C developed more distinct banded structure instead of interconnecting between ferrite bands. This might support the aforementioned mechanism such that the regions with lower manganese content do not transform into ferrite at this temperature. This should be verified by future measurements of the alloying content across the sample thickness.
Figure 5.11: Optical microstructure for the 2.0% Mn steel after the 2\textsuperscript{nd} set of stepped cooling transformation tests at 650 °C for different holding times (a: 10s, b: 20s, c: 60s).
Figure 5.12: Optical microstructure for the 2.5%Mn steel after the 2nd set of stepped cooling transformation tests at 650 °C for different holding times (a: 1800s, b: 7200s).

Table 5.3 and Table 5.4 summarizes the area fraction of ferrite obtained from the optical microstructure measured by the point counting method. Here, the sampling (statistical) uncertainty was expressed as the 95% confidence interval, which was calculated from at least nine micrographs for each holding time at each temperature in both steels, according to ASTM E562-89[88]. For the 575 °C holding condition in the 2.5%Mn steel, where the microstructure is primarily bainitic, it is difficult to differentiate metallographically between the bainitic ferrite transformed during holding and the other formed during quenching. Therefore, it requires further study to establish a quantitative relationship between the fraction transformed during isothermal holding as obtained from the laser ultrasonic velocity technique and the bainite fraction within the microstructure.
<table>
<thead>
<tr>
<th>Holding temp</th>
<th>Holding time</th>
<th>Area Fraction Ave (±95%CI)</th>
</tr>
</thead>
<tbody>
<tr>
<td>700°C</td>
<td>600s</td>
<td>0.56 (±0.05)</td>
</tr>
<tr>
<td>675°C</td>
<td>600s</td>
<td>0.70 (±0.03)</td>
</tr>
<tr>
<td>650°C</td>
<td>600s</td>
<td>0.78 (±0.04)</td>
</tr>
<tr>
<td>650°C</td>
<td>10s</td>
<td>0.47 (±0.03)</td>
</tr>
<tr>
<td>650°C</td>
<td>20s</td>
<td>0.53 (±0.04)</td>
</tr>
<tr>
<td>650°C</td>
<td>60s</td>
<td>0.73 (±0.03)</td>
</tr>
</tbody>
</table>

Table 5.4: Area fraction of ferrite within microstructure for stepped cooling conditions in 2.5%Mn steels (ASTM E562 Point counting)

<table>
<thead>
<tr>
<th>Holding temp</th>
<th>Holding time</th>
<th>Area Fraction Ave (±95%CI)</th>
</tr>
</thead>
<tbody>
<tr>
<td>650°C</td>
<td>600s</td>
<td>0.38 (±0.05)</td>
</tr>
<tr>
<td>625°C</td>
<td>600s</td>
<td>0.55 (±0.06)</td>
</tr>
<tr>
<td>600°C</td>
<td>600s</td>
<td>0.57 (±0.03)</td>
</tr>
<tr>
<td>650°C</td>
<td>1800s</td>
<td>0.43 (±0.03)</td>
</tr>
<tr>
<td>650°C</td>
<td>7200s</td>
<td>0.46 (±0.08)</td>
</tr>
</tbody>
</table>
5.2.3 Comparison between ultrasound velocity technique and metallography

Figure 5. 13 displays the comparison between the fraction transformed calculated by ultrasound velocity technique and obtained from optical metallography. It represents a good agreement between these two different quantification techniques. This indicates that the ultrasonic velocity technique can be an option to measure the proeutectoid ferrite fraction in dual phase steels during phase transformation progress.

![Graph showing comparison between ultrasound velocity technique and metallography](image)

Figure 5. 13: Comparison between the fraction transformed obtained from the ultrasound velocity technique and the optical metallography in the present steels; error bar indicates the 95% confidence interval.

5.2.4 Hardness

Hardness measurement is another way to characterize the resultant microstructure. Table 5.5 and Table 5.6 summarize the number of tests, the mean and the 95% confidence intervals of the results for all the conditions in both steels.
Table 5.5: Hardness Measurement on 2.0%Mn steels

<table>
<thead>
<tr>
<th>Holding temp</th>
<th>Holding time</th>
<th>Number of tests</th>
<th>Hardness (in HV) Mean (±95%CI)</th>
</tr>
</thead>
<tbody>
<tr>
<td>700°C</td>
<td>600s</td>
<td>35</td>
<td>227 (±8)</td>
</tr>
<tr>
<td>675°C</td>
<td>600s</td>
<td>21</td>
<td>212 (±9)</td>
</tr>
<tr>
<td>650°C</td>
<td>600s</td>
<td>21</td>
<td>203 (±7)</td>
</tr>
<tr>
<td>650°C</td>
<td>10s</td>
<td>20</td>
<td>247 (±9)</td>
</tr>
<tr>
<td>650°C</td>
<td>20s</td>
<td>20</td>
<td>225 (±6)</td>
</tr>
<tr>
<td>650°C</td>
<td>60s</td>
<td>20</td>
<td>215 (±5)</td>
</tr>
</tbody>
</table>

Table 5.6: Hardness Measurement on 2.5%Mn steels

<table>
<thead>
<tr>
<th>Holding temp</th>
<th>Holding time</th>
<th>Number of tests</th>
<th>Hardness (in HV) Mean (±95%CI)</th>
</tr>
</thead>
<tbody>
<tr>
<td>650°C</td>
<td>600s</td>
<td>44</td>
<td>276 (±11)</td>
</tr>
<tr>
<td>625°C</td>
<td>600s</td>
<td>28</td>
<td>264 (±11)</td>
</tr>
<tr>
<td>600°C</td>
<td>600s</td>
<td>21</td>
<td>244 (±11)</td>
</tr>
<tr>
<td>575°C</td>
<td>600s</td>
<td>35</td>
<td>252 (±10)</td>
</tr>
<tr>
<td>650°C</td>
<td>1800s</td>
<td>63</td>
<td>263 (±10)</td>
</tr>
<tr>
<td>650°C</td>
<td>7200s</td>
<td>47</td>
<td>246 (±14)</td>
</tr>
</tbody>
</table>

Figure 5.14 displays the relationship between the average hardness within the microstructure and the fraction transformed measured by laser ultrasonic technique. Generally, it can be...
observed that the average hardness decreases with the increasing fraction transformed measured by the laser ultrasonic technique. This can be attributed to the decrease in the fraction of the hard martensitic phase as the ferrite fraction increases. Therefore, the hardness measurement in the present study also supports the possibility that the laser ultrasonic technique can be applied for monitoring the microstructure evolution of the ferrite/martensite dual phase steels during the transformation progress.

In the present study, within the scatter of the data in a first approximation, a linear relationship can be proposed. The observed slope and intercept of the average hardness per fraction transformed are -165 [HV], and 333 [HV], respectively. The intercept which corresponds to the hardness of the fully martensitic microstructure is consistent with the literature [84].
Figure 5.14: Relation between hardness and fraction transformed obtained from the laser ultrasonic velocity technique for the investigated steels; error bar indicates the 95% confidence interval.

5.2.5 Effect of phase distribution on ultrasound velocity

5.2.5.1 Introduction

In the present study, some stepped cooling transformation conditions result in a banded microstructure, which is also often observed in real production lines. Then, the potential effect of phase distribution within the microstructure on the ultrasound velocity might be a concern. To evaluate this aspect, a series of finite element (FE) simulations of ultrasound wave propagation were conducted with different phase distributions. This modelling approach is intended to
evaluate the role of the geometric configuration of phases as well as phase fraction on the ultrasound velocity.

5.2.5.2 Domain setting

Explicit, 2-D finite element modelling (FEM) simulations were performed on Abaqus CAE version 6.13. Each grain assigned as one phase was assumed to be elastically isotropic and defined by its respective density and elastic stiffness constants, where here the available values for $\alpha$ and $\gamma$ iron were used. The temperature was fixed at 650 °C for the series of simulation.

The 2-D meshed specimen had a thickness of 2 mm, a length of 10 mm, and a mesh size of 4 $\mu$m in the small meshed region (4 mm by 2mm) in the centre of the specimen length. Figure 5.15 schematically illustrates the mesh template. Simulation domains were created by periodic repetition of a MICRESS®-generated sub-domain until it met a desired mesh template size. The mesh consisted of four-node bi-linear quadrilateral plane strain (CPE4R) elements. The simulated domain is sub-divided into a centered region with a fine mesh size of 4 $\mu$m and a coarse mesh on each side. The larger mesh structure outside the small meshed region play a role to dissipate transverse components of the wave front, so only the longitudinal component that returned to the monitoring region would be accounted for within the simulation time. In order to fix the sample geometry in space during the wave propagation, additional boundary conditions are defined for the elements of the mesh located along the left and right vertical edges such that no rotation and translation are permitted, i.e. $U_1=U_2=U_3=U_{R1}=U_{R2}=U_{R3}=0$ (Abaqus boundary condition: Mechanical-Encastre). $U_i$ and $U_{Ri}$ are respectively displacement components and rotational displacement components, and the subscript $i=1, 2, 3$ corresponds to the $X$, $Y$, and $Z$ dimensions in Abaqus. The model assumed linear elasticity for large displacements.
Figure 5.15: (a) 2D generated mesh template with thickness of 2mm, length of 10 mm and mesh size of 4 μm in the small mesh region (4 mm × 2 mm). (b) horizontal, (c) quasi-random, (d) vertical distribution condition with 50% of ferrite (blue) and austenite (yellow)

Since the elastic stiffness constants of γ-iron available in the literature are limited to higher temperatures (above 1000 °C), they were adjusted such that the simulated ultrasound velocities with 100% α-iron and 100% γ-iron match with the measured ultrasound velocities with fully ferrite and fully austenite phases, as follows: First, the ultrasound velocity with fully ferrite at 650 °C in the 2.0%Mn steel was determined as 5.41 mm/μs in the velocity-temperature curve in Figure 5.3 as well as the ultrasound velocity with fully austenite at 650 °C in the 2.0%Mn steel was calculated as 5.24 mm/μs from the extrapolation at higher temperature.

Second, the corresponding elastic stiffness constants were selected. The stiffness matrix of an isotropic continuum ([c]_{iso}) only contains the two independent elastic stiffness constants \( c_{11} \) and \( c_{44} \), and is defined as:
The \([c]_{iso}\) matrix is invariant upon rotation, meaning that if rotated by an arbitrary direction, the stiffness matrix will not change. Thus, the combinations of \(c_{11}\) and \(c_{44}\), which provide the ultrasound velocities closest to the measured ones, i.e., 5.41 mm/μs and 5.24 mm/μs, were needed. In the present study, the data set of the longitudinal and the shear ultrasound velocities of \(\gamma\)-iron and \(\alpha\)-iron varying with temperature under the Hill averaging assumption\[92\] were obtained from the CTOME software. The combinations of \(c_{11}\) and \(c_{44}\) can be calculated from the longitudinal and the shear ultrasound velocities by following equations:

\[
[c]_{iso} = \begin{bmatrix}
c_{11} & c_{11} - 2c_{44} & c_{11} - 2c_{44} & 0 & 0 & 0 \\
c_{11} & c_{11} - 2c_{44} & c_{11} - 2c_{44} & 0 & 0 & 0 \\
c_{11} & c_{11} - 2c_{44} & c_{11} & 0 & 0 & 0 \\
c_{44} & 0 & 0 & c_{44} & 0 \\
\text{symm} & \end{bmatrix}
\]  
(Eq 5.2)

\[
V_{L}^{Hill} = \sqrt{\frac{\langle c_{11} \rangle^{Hill}}{\rho}}
\]  
(Eq 5.3 11)

\[
V_{S}^{Hill} = \sqrt{\frac{\langle c_{44} \rangle^{Hill}}{\rho}}
\]

where \(V_{L}^{Hill}\) and \(V_{S}^{Hill}\) are the longitudinal and the shear ultrasound velocities of \(\gamma\)-iron under Hill assumption and \(\rho\) is the density. Here, the data set is used to compute the combinations of elastic constants to match the ultrasound velocities in 100% \(\alpha\)-iron and 100% \(\gamma\)-iron under isotropic assumption with the measured velocities. Finally, the elastic stiffness constants which provide the longitudinal ultrasound velocities close to the measured ones are selected for each
phase. Table 5.7 summarizes the parameters determined for the simulation including the ultrasound velocities, the elastic stiffness constants, and densities for the two phases.

<table>
<thead>
<tr>
<th></th>
<th>Density (g/cm$^3$)</th>
<th>Ultrasound Velocity (mm/μs)</th>
<th>$c_{11}$ (GPa)</th>
<th>$c_{44}$ (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\alpha$–iron</td>
<td>7.67</td>
<td>5.41</td>
<td>237.6</td>
<td>66.4</td>
</tr>
<tr>
<td>$\gamma$–iron</td>
<td>7.79</td>
<td>5.24</td>
<td>218.2</td>
<td>46.2</td>
</tr>
</tbody>
</table>

As shown in Figure 5.15, three different geometric arrangements of the two phases were considered. The plate structures were created with different phase fraction of $\alpha$-iron elements. The purpose of the creation of the horizontal and vertical plate geometries as well as the quasi-random dispersed element structure was to evaluate the effect of the banded microstructure on the resulting ultrasound velocity. In this simulation, 500μm × 500μm of the quasi-random dispersed element structure was generated repeatedly over the mesh template. Further, the current simulation has the limitation with the length scales that are much smaller than in the experiments. For example, the band width in the horizontal and vertical arrangements is about 35 μm but could be nevertheless useful to capture the effect of the geometric configuration on the relationship between the velocity and the fraction transformed for the first approximation.
5.2.5.3 Wavelet generation and wave propagation

The simulations consist of an ultrasound pulse generation step followed by a propagation step. During the first step, an initial displacement is created in the form of a double Ricker wavelet. An initial displacement was applied to a region 2 mm in size located at the center of the top surface of the fine meshed part (Figure 5.15-a). The amplitude of a Ricker wavelet is given by [93]

$$A(t) = \sum_{i=1}^{2} \left( 1 - 2\pi^2 (F_p)^2 (t - \varepsilon_i)^2 \right) e^{-\pi^2 (F_p)^2 (t-\varepsilon)^2}$$  \hspace{1cm} (Eq 5.4)

where $F_p$ is the peak frequency in MHz, $t$ is time in seconds, and $\varepsilon$ is a constant. The double Ricker wavelet amplitude was distributed across the 2 mm generation surface using a Gaussian function given by

$$\bar{A}(x) = \bar{A}(t) e^{-\left(\frac{x^2}{2(\sigma)^2}\right)}$$  \hspace{1cm} (Eq 5.5)

where $x$ is in the dimension of mm and, here $\sigma$ is set as 0.5 mm. The Gaussian distribution of amplitude reduces the stress discontinuity between nodes in the generation region and their adjacent nodes outside the generation region. Lasers generally are considered to emit beams with a Gaussian intensity profile. As the ultrasound pulse in the LUMet system is generated by laser ablation, the spatial profile of the generated displacement may not follow the one of the incident laser pulse due to the nonlinear nature of the vaporization process. Nevertheless, the Gaussian spatial distribution profile may be considered as a meaningful first approximation for the simulations.

During the second simulation step, the generation surface nodes are released, and ultrasound pulse propagation is simulated for a duration of 2000 ns. The displacement data was averaged along the nodes opposite of the 2mm generation surface (i.e. the centered 2mm length region
along the bottom surface of the simulation domain), and was processed to compute the time
delay and the subsequent ultrasound velocity in the same way as the LUMet measurement
signals were processed using the CTOME software. The plate thickness in simulation is 2mm
which is different from the current experimental condition. However, it has no impact on the
evaluation of the results as it has simply different propagation distances.

5.2.5.4 FEM results

Figure 5.16 shows the simulated ultrasound velocity as a function of the fraction transformed
(i.e. the phase fraction of α-iron) for three different phase distributions under isotropic conditions
as well as the measured ultrasound velocity for the 2.0%Mn steel at 650°C holding with the 95%
confidence interval for each phase fraction. Overall, all the simulated velocities agree with the
measured ultrasound velocity within the measurement accuracy. The linear relationship between
the velocity and the phase ratio was successfully reproduced for the three different phase
distributions. All three different distributions result in the same velocities within the margins of
experimental errors. Therefore, the present simulations indicate that the effect of phase
distribution on ultrasound velocity is negligible.
5.2.6 Equilibrium ferrite fraction

Figure 5.5 and Figure 5.6 clearly show the effect of the temperature as well as the effect of manganese content on the transformation kinetics. Substitutional elements such as Mn, Ni can considerably influence the kinetics since their diffusion coefficients are significantly lower than that of the interstitial carbon atoms. In order to perform quantitative prediction of alloying effects in the real production time scale, NPLE and para-equilibrium ferrite fraction are calculated in both steels. Figure 5.17 represents the phase diagram of Fe-C-Mn-0.5wt.%Si system at the temperature of 650°C with mass percent of carbon in x-axis and u-fraction of Mn in y-axis,
illustrating the relative positions of the PLE and NPLE regions. The figure is a cut for a constant Si level, i.e., 0.5wt.%Si. If the bulk carbon content is \( c_0 \), the volume fraction of proeutectoid ferrite at the end of ferrite growth under NPLE condition \( f_{\text{NPLE}}^{\alpha} \) can be calculated by[49]

\[
f_{\text{NPLE}}^{\alpha} = \frac{c_{\text{NE}}^{\gamma/\alpha} - c_0}{c_{\text{NE}}^{\gamma/\alpha} - c_{\text{OE}}^{\alpha/\gamma}}
\]

(Eq 5.6)

where \( c_{\text{NE}}^{\gamma/\alpha} \) is the intersection between the carbon component ray which passes through \( c_0 \) and the PLE/NPLE transition line, and \( c_{\text{OE}}^{\alpha/\gamma} \) is the theoretical carbon concentration in ferrite under ortho-equilibrium.

Figure 5.17: Phase diagram of Fe-C-Mn-0.5wt.%Si system at 650°C illustrating the relative positions of the PLE and NPLE regions. Solid circles indicate the present steel chemistries.

Figure 5.18 illustrates the para-equilibrium \((\alpha+\gamma)\) two-phase region enclosed by the two para-equilibrium phase boundaries superimposed on the ortho-equilibrium two-phase region of Fe-C-
Mn-0.5wt.%Si system at the temperature of 650°C. Again, the figure is a cut for a constant Si level, i.e., 0.5wt.Si. Similarly, outside of the para-equilibrium two-phase region, Mn redistribution is expected. Thus, the volume fraction of proeutectoid ferrite at the end of ferrite growth under para-equilibrium condition ($f_{PE}^\alpha$) can be calculated by\[49\]

$$f_{PE}^\alpha = \frac{c_{PE}^{\gamma/\alpha} - c_0}{c_{PE}^{\gamma/\alpha} - c_{PE}^{\alpha/\gamma}}$$

(Eq 5.7)

where $c_{PE}^{\gamma/\alpha}$ and $c_{PE}^{\alpha/\gamma}$ are the intersection between the carbon component ray which passes through $c_0$ and the para-equilibrium two-phase boundaries of $\gamma$ side and $\alpha$ side, respectively.

Figure 5.18: Phase diagram of Fe-C-Mn-0.5wt.%Si system at 650°C illustrating the para-equilibrium phase boundaries. Solid circles indicate the present steel chemistries.

In the present study, NPLE/PLE transition line is assumed as the straight line connecting the $\alpha/\gamma$ two-phase boundary of ferrite on the Fe-Mn side with the one of austenite on the Fe-C side.
Para-equilibrium two-phase boundaries are assumed as the straight lines connecting the point of equal Gibbs free energy in $\alpha$ and $\gamma$ on the Fe-Mn side for the temperature with the ortho-equilibrium diagram boundaries of the Fe-C side. All the thermodynamic data was calculated using Thermo-Calc (TCFE7 database).

Figure 5.19 displays the comparison between the fraction transformed obtained by laser ultrasonic technique at the exit of the isothermal holding and the equilibrium ferrite fraction under two constrained equilibrium conditions. In all cases, the measured fraction is below both constrained equilibrium ferrite fraction. It is more pronounced for the 2.5%Mn steel, but to lesser degree also for the 2.0%Mn steel where NPLE is almost reached. For the 2.0%Mn steel, NPLE assumption can be useful to estimate in a first approximation the steady-state ferrite fraction in the present time scale, i.e., 600s.
5.2.7 JMAK model

Analysis of the transformation kinetics was made using the Jonhson-Mehl-Avrami-Kolmogorov (JMAK) theory to describe the ferrite fraction transformed as a function of time [94-97]. By adapting the JMAK equation for isothermal conditions, the $\gamma \rightarrow \alpha$ phase transformation kinetics can be modeled using:

$$\frac{f_\alpha(t)}{f_\alpha^{\text{max}}} = y = 1 - \exp(-k(t - \tau)^n)$$  \hspace{1cm} (Eq 5.8)

where $f_\alpha$ is the fraction transformed after a holding time $t$, $k$ is the reaction rate parameter, $n$ is the JMAK exponent, $\tau$ is the incubation time, $f_\alpha^{\text{max}}$ is the maximum (or the steady-state) $\alpha$ fraction
transformed, which is determined from the fraction transformed measured by the laser ultrasonic technique at the exit of the isothermal holding, and \( y \) is the normalized fraction transformed. At each holding temperature, the ultrasound velocity increases with the holding time during transformation progress in the present study. Then, the velocity change \( \Delta V_i \), which is defined by

\[
\Delta V_i = V_i - V_{st}
\]

(Eq 5.9)

where \( V_i \) is the instantaneous velocity and \( V_{st} \) is the initial velocity for each holding condition, is considered directly to be correlated to the transformation kinetics. Therefore, by using the maximum velocity change \( \Delta V_{max} \) for each holding condition, the normalized velocity change \( \Delta V_{i,norm}(= \frac{\Delta V_i}{\Delta V_{max}}) \) was assumed to be \( y \), here. Eq 5.8 can be rearranged into a linear form in which the slope \( n \) and the intercept \( \ln(k) \) can be found by plotting \( \ln(\ln(1/(1-y))) \) against \( \ln(t-\tau) \).

Such plots are shown in Figure 5.20 and Figure 5.21. Here, the normalized fraction, \( y \), considered in the fit was from 0.10 to 0.90. \( \tau \) was taken from the experimental data only for the 650°C holding condition for the 2.5%Mn steel whereas the transformation starts essentially immediately when reaching the holding temperature in all other conditions, resulting in setting \( \tau \) as zero.
Figure 5.20: Plot of $\ln(\ln(1/(1-y)))$ versus $\ln(t-\tau)$ showing the best linear fit for each temperature in the 2.0% Mn steel.

Figure 5.21: Plot of $\ln(\ln(1/(1-y)))$ versus $\ln(t-\tau)$ showing the best linear fit for each temperature in the 2.5% Mn steel.

Table 5.8 and Table 5.9 summarize the value of the incubation time, the JMAK exponent, and the rate parameter for each isothermal condition for the 2.0% Mn and 2.5% Mn steels,
respectively. The JMAK exponent represents similar values at each holding temperature for each chemistry. Then, the representative JMAK exponents for the 2.0%Mn and the 2.5%Mn steels can be determined as 0.89, and 0.78, respectively, by averaging. These values are within the reasonable ranges of the JMAK exponent of ferrite transformation in low carbon steels [76, 98-100]. By using the average JMAK exponents, the rate parameter $k$-fit with the same unit for each chemistry can be determined. Figure 5.22 shows a clear linear relationship between the holding temperature and the logarithm of the rate parameter $k$-fit in both chemistries. This leads to the temperature-related expression of the rate parameter for each chemistry shown in the tables. It also indicates that adding 0.5% Mn has a significant influence on the rate parameter. By substituting the incubation times, the average JMAK exponents, and the temperature-related rate parameters into Eq 5.8, the JMAK transformation kinetics can be calculated at each isothermal holding for both steels. Figure 5.23 and Figure 5.24 display the comparison between the normalized fraction measured by the laser ultrasonic technique and the fraction calculated with the JMAK models using the average n-values and associated temperature dependence of the rate parameter. As can be seen, the JMAK theory is suitable to describe the ultrasound velocity evolution in the present steels. This indirectly supports that the application of the current ultrasound velocity technique for the real-time monitoring of the phase transformation kinetics is possible as it successfully captures the well-known rate parameter change with temperature regarding phase transformation[98, 101, 102].
Table 5.8: JMAK exponent (n) and rate parameter (k) in 2.0% Mn steels

<table>
<thead>
<tr>
<th>Temp. (°C)</th>
<th>T(s)</th>
<th>n\text{raw}</th>
<th>k\text{raw}(s^{n})</th>
<th>n\text{ave}</th>
<th>k\text{fit}(s^{n})</th>
</tr>
</thead>
<tbody>
<tr>
<td>700</td>
<td>0</td>
<td>0.89</td>
<td>0.047</td>
<td></td>
<td></td>
</tr>
<tr>
<td>675</td>
<td>0</td>
<td>0.91</td>
<td>0.089</td>
<td>0.89</td>
<td>\text{exp}(-0.0223 \times T+18.761)</td>
</tr>
<tr>
<td>650</td>
<td>0</td>
<td>0.86</td>
<td>0.145</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 5.9: JMAK exponent (n) and rate parameter (k) in 2.5% Mn steels

<table>
<thead>
<tr>
<th>Temp. (°C)</th>
<th>T(s)</th>
<th>n\text{raw}</th>
<th>k\text{raw}(s^{n})</th>
<th>n\text{ave}</th>
<th>k\text{fit}(s^{n})</th>
</tr>
</thead>
<tbody>
<tr>
<td>650</td>
<td>2.3</td>
<td>0.89</td>
<td>0.024</td>
<td></td>
<td></td>
</tr>
<tr>
<td>625</td>
<td>0</td>
<td>0.66</td>
<td>0.077</td>
<td>0.78</td>
<td>\text{exp}(-0.0175 \times T+12.750)</td>
</tr>
<tr>
<td>600</td>
<td>0</td>
<td>0.76</td>
<td>0.079</td>
<td></td>
<td></td>
</tr>
<tr>
<td>575</td>
<td>0</td>
<td>0.82</td>
<td>0.102</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Figure 5.22: The rate parameter $k$-fit in the JMAK model with temperature for both steels.

Figure 5.23: The comparison between the normalized velocity change from the laser ultrasonic technique and the JMAK model in the 2.0%Mn steel.
Figure 5.24: The comparison between the normalized velocity change from the laser ultrasonic technique and the JMAK model in the 2.5%Mn steel.
Chapter 6: Conclusion

6.1 Summary

The objective of the current work is to examine the possibility of the laser ultrasonics technique as an in-situ monitoring method of microstructure evolution for dual phase steels during stepped cooling. Austenite-to-ferrite phase transformation kinetics during stepped cooling were recorded using LUMet. Microstructure characterization was carried out using optical microscopy and hardness measurements. The important results found in this work are summarized below:

1. For the investigated transformation conditions the changes of laser ultrasonic velocity can be correlated with a law of mixture, i.e., in a linearized way, with the fraction transformed.

2. Phase transformation kinetics measured from laser ultrasonic velocity and from dilatometry during continuous cooling transformation tests are in an agreement within 5°C of transformation temperatures, i.e., within the experimental errors. This indicates that the laser ultrasonic technique can capture the phase transformation kinetics in the present steels.

3. Fraction transformed measured from laser ultrasonic velocity during stepped cooling transformation tests agrees well with the ferrite phase fraction of the dual phase microstructures obtained from metallography in both steels.
4. Measured hardness, which is another way to characterize dual phase microstructure, has a linear relationship with fraction transformed consistent with literature data for dual phase steels.

5. The evolution of the ferrite fraction for a given isothermal holding temperature can be described by the JMAK approach. The JMAK exponent and the rate parameters are consistent with literature data for the austenite-to-ferrite transformation[76, 98-101].

6. Depending on the heat treatment conditions, banded or non-banded dual phase microstructures were observed. FEM analysis confirmed that the laser ultrasonic velocity depends on the phase fractions, but the geometric configuration of phases has a negligible contribution, further indicating the robustness of the laser ultrasonic method.

7. NPLE assumption can be a reasonable estimate for the final ferrite fraction for the 2.0%Mn steel during the present stepped cooling paths whereas neither NPLE nor para-equilibrium were suitable for the 2.5%Mn steel where the additional Mn alloying leads to a more significant delay of ferrite formation.

6.2 Future work

The following future studies are proposed as improvements, compliments, and extensions of the present work.
1. During the stepped cooling transformation test on the 2.5%Mn steel bainitic ferrite forms for the holding temperature of 575°C. This transformation can be recorded with laser ultrasonics as well, but validation of the fraction transformed with optical microscopy was not possible as isothermal transformation products and those formed during quenching could not be distinguished. Thus, it is suggested to employ electron backscatter diffraction (EBSD) characterization as an alternative to quantify the phase fraction formed during the isothermal holding. Due to the high dislocation density and distortion of the atomic lattice resulting from the martensite transformation, the image quality (IQ) of martensite, which is a measure of the perfection of the crystal lattice, is generally low. Several studies can be found in the literature where the IQ is related to distortions caused by a high dislocation density to distinguish martensite from other phases[103, 104]. However, several other factors influence the IQ including sample preparation and EBSD settings. Thus, it is necessary to establish the appropriate procedure for the normalization and the quantification of phases.

2. Some holding temperature conditions present banded dual phase microstructures. The microstructure observation in the 2nd set of stepped cooling transformation tests shows that the proeutectoid ferrite nucleates and grows within the preferred regions under lower driving pressure for phase transformation, resulting in the banded microstructure at the 650°C holding condition in the 2.5%Mn steel whereas the nucleation of ferrite becomes denser and the banded microstructure is not observed for higher driving pressures, for example at the 650°C holding condition in 2.0%Mn steel. This indicates that the manganese content level in the thickness direction may influence the microstructure evolution, resulting in banded microstructure. This, however, is necessary to be confirmed by measurements of the alloying
content across the sample thickness. These investigations may also be of relevance to further evaluate the discrepancies between the final ferrite fraction during isothermal treatments and the expected equilibrium fractions (either NPLE or para-equilibrium).
Bibliography


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