EFFECTS OF MICROALLOYING ON HOT-ROLLED AND COLD-ROLLED Q&P STEELS

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Third generation advanced high strength steels (AHSS) have been a major focus in steel development over the last decade. The premise of these types of steel is based on the potential to obtain excellent combinations of strength and ductility with low-alloy compositions by forming mixed microstructures containing retained austenite (RA). The development of heat treatments able to achieve the desired structures and properties, such as quenching and partitioning (Q&P) steels, is driven by new requirements to increase vehicle fuel economy by reducing overall weight while maintaining safety and crashworthiness.

Microalloying additions of niobium (Nb) and vanadium (V) in sheet products are known to provide strengthening via grain refinement and precipitation hardening and may influence RA volume fraction and transformation behavior. Additions of microalloying elements in Q&P steels have not been extensively studied to date, however. The objective of the present study was to begin to understand the potential roles of Nb and V in hot-rolled and cold-rolled Q&P steel. For that, a common Q&P steel composition was selected as a Base alloy with 0.2C-1.5Si-2.0Mn (wt. %). Two alloys with an addition of Nb (0.02 and 0.04 wt. %) and one with an addition of V (0.06 wt. %) to the Base alloy were investigated. Both hot-rolled and cold-rolled/annealed Q&P simulations were conducted.

In the hot-rolled Q&P study, thermomechanical processing was simulated via hot torsion testing in a Gleeble® 3500, and four coiling temperatures (CT) were chosen. Microstructural evaluation (including RA measurements via electron backscattered diffraction - EBSD) and hardness measurements were performed for all alloys and coiling conditions. The analysis showed that Nb additions led to overall refinement of the prior microstructure. Maximum RA fractions were measured at the 375 °C CT, and microalloying was associated with increased RA in this condition when compared to the Base alloy. A change in austenite morphology from lath-like to blocky with increasing CT was observed. Hardness generally increased with decreasing CT, consistent with the increased fraction of harder phases in the microstructure.

For the cold-rolled Q&P study, several combinations of quenching temperature (QT), partitioning temperature (PT), and partitioning time (t_p) were examined using heat treatments in salt baths. Uniaxial tensile tests and RA measurements via x-ray diffraction (XRD) were performed for all alloys and heat treatment conditions. Scanning electron microscope (SEM) imaging and EBSD were conducted for a few select conditions. In terms of microstructure, Nb promoted an extensive refinement of the prior austenite grain size. Additions of V and Nb also seemed to affect the morphology of the microstructural constituents. It was observed that V generally increased austenite fractions at lower t_p’s, and the Nb-
containing alloys had greater austenite fractions in most instances when compared to the Base alloy. Carbon content in austenite was usually increased or maintained with additions of Nb and V. In terms of mechanical properties, V slightly improved strength and elongation when compared to the Base alloy for most conditions. Niobium additions were somewhat more effective in improving ductility.
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CHAPTER 1: INTRODUCTION

Third generation advanced high strength steels (AHSS) have been a major focus in steel development over the last decade. The premise of this type of steel comes from the possibility to obtain excellent combinations of strength and ductility with low-alloy compositions by forming mixed microstructures containing retained austenite [1–3]. The development of heat treatments able to achieve the desired structures and properties came along with new requirements to increase fuel economy by reducing the overall vehicle weight while maintaining safety and crashworthiness [4–6]. These new regulations motivated research from government, and steel producers and users around the world to develop concepts and further improve the properties of the new generation of sheet steels for timely and widespread implementation in industry. Quenching and partitioning (Q&P), medium manganese (Medium Mn), and transformation induced plasticity (TRIP) aided bainitic ferrite (TBF) are new families of steels that are part of the 3rd generation of AHSS.

Microalloying additions of niobium (Nb) and vanadium (V) are known to provide strengthening via grain refinement and precipitation hardening, and may influence retained austenite (RA) volume fraction and transformation behavior in sheet products [3,7–16]. Additions of Nb and V in the 1st generation of AHSS have been studied and mostly focused on Nb in cold-rolled steels, like dual phase (DP), TRIP, and complex phase (CP) [7,10–13]. Vanadium is less widely implemented in cold-rolled sheet steels, and its strengthening effects are more pronounced in hot-rolled products [17]. These studies showed that additions of small quantities of V and Nb in hot-rolled and cold-rolled steels enhance mechanical properties when combined with appropriate processing.

Recent studies introducing additions of microalloying elements to 3rd generation AHSS have been published. Tan et al. [18–21] and Xu et al. [22] studied additions of V in hot-rolled and cold-rolled Q&P steels; Hashimoto et al. [23] additions of Nb in hot-rolled TBF steels; Hausmann et al. [24,25] and Sugimoto et al. [26–29], additions of Nb in cold-rolled TBF steels; and, Zhou et al. [30] and Zhong et al. [31,32], additions of Nb in cold-rolled sheet Q&P steels. Promising combinations of microstructures, mechanical properties and formability have been presented. These combinations have been made possible by decreased grain size, retention of large volume fractions of austenite, and improved strength and/or elongation with the addition of microalloying elements. The specific findings of these studies are presented in more detail in Chapter 2 (page 3).

The goal of the present study is to begin to understand the individual roles of Nb and V in hot-rolled and cold-rolled Q&P steel when compared to a base steel. Comparisons are made primarily by analysis of prior austenite grain size, influence on microstructural constituents and morphologies, stability
and fraction of RA, and mechanical properties, such as yield strength (YS), ultimate tensile strength (UTS) and uniform elongation (UE).
CHAPTER 2: BACKGROUND

In this chapter, basic concepts of hot-rolled Q&P (HR Q&P) and cold-rolled Q&P (CR Q&P) steels are presented. Relevant background on Q&P is discussed, as well as studies on the influence of Nb and V in HR and CR sheet steel products that contain RA. Such steels include TRIP and TBF steels.

2.1 Fundamentals of Q&P Steels

In this section, fundamentals of HR and CR Q&P are presented.

2.1.1 Cold-Rolled Q&P

A thermodynamic model, called “constrained carbon equilibrium” (CCE) [33,34], was developed to address the partitioning behavior of carbon from as-quenched martensite into untransformed austenite. Some assumptions presented in the CCE model were: a) all carbon partitions from the supersaturated martensite into the austenite; b) no competing reactions happen during carbon partitioning; c) fractions of martensite and austenite are constant during partitioning, which means that the martensite/austenite interface is constrained (immobile) and there are no short-range movements of iron or other substitutional atoms; and, d) the chemical potential of carbon is the same in both phases (martensite and austenite) at the end of partitioning [1,34].

Quenching and partitioning was a concept proposed by Speer et al. [33,35], based on the thermodynamic considerations of the CCE model. The Q&P heat treatment, shown in Figure 2.1, consists of austenitizing (full or intercritical annealing) followed by quenching to a temperature (QT) between the martensite start temperature (M_s) and martensite finish temperature (M_f). At the QT, the microstructure consists of martensite and untransformed austenite. The next step is partitioning, where carbon diffuses from supersaturated martensite to austenite, which will then stabilize the (retained) austenite and impede further transformation during the final quench to room temperature. Partitioning occurs either at the QT (one-step Q&P), or at a temperature higher than the QT (two-step Q&P) [36]. Figure 2.1a and Figure 2.1b present schematics of the fully austenitized Q&P heat treatment and the expected microstructure after each step of the process, respectively. The final microstructure generally consists of martensite with RA, but may include additional phases due to alternative mechanisms that can occur during the partitioning step, such as carbide precipitation, austenite decomposition, bainite formation, etc. [37].

During initial Q&P development, it was proposed that the amount of martensite and RA in the final microstructure is influenced by the QT [33]. The Koistinen-Marburger (K-M) relationship [38] gives the predicted martensite fraction (f_m) as a function of M_s and QT:

\[ f_m = 1 - e^{-1.1 \times 10^{-2} (M_s - QT)} \]
The $M_s$ temperature can either be estimated via empirically-derived equations in literature, or measured directly through dilatometric observation of the martensitic transformation. Equation 2.2, proposed by Mahieu et al. [39], provides an estimate for $M_s$ based on chemical composition:

$$M_s(\degree C) = 539 - 423C - 30.4Mn - 7.5Si + 30Al$$

where $C$, $Mn$, $Si$ and $Al$ are weight percent carbon, manganese, silicon and aluminum, respectively. Once an approximate value for $M_s$ temperature has been established, fractions of martensite and RA can be estimated using the K-M relationship or similar relationships [40,41].

![Figure 2.1](image)

(a) Schematic of the one-step and two-step Q&P thermal processing, considering full austenitization. AT, QT and PT are austenitizing, quenching and partitioning temperatures, respectively. $M_s$ and $M_f$ are martensite start and finish temperatures, and $t$ is time [36]. (b) Schematic illustration of the expected microstructures after each step of the Q&P heat treatment, where $C_\gamma$, $C_i$, $C_{\alpha'}$ are carbon concentration in austenite, initial carbon concentration and carbon concentration in martensite, respectively [42].

Optimum quenching temperature was another concept identified in early development of Q&P. Optimum QT is a temperature at which the maximum fraction of RA could theoretically be stabilized at room temperature (assuming full partitioning of carbon from martensite to austenite) and taking into consideration the final quench to room temperature [33]. Initial calculations for optimum QT were made for an alloy with composition of 0.19C-1.46Mn-1.96Al (wt. %) for a starting condition of 50 % intercritical ferrite, as shown in Figure 2.2 [43]. From Figure 2.2 it is noted that quenching below the optimum temperature will form a higher fraction of martensite than desired for maximum austenite retention. In this case, high levels of carbon are available for partitioning to the austenite, potentially making it more stable. On the other hand, quenching above the optimum temperature will result in greater amounts of austenite present at the QT. However, the decreased capacity for carbon partitioning from martensite results in insufficient austenite stability. The result, in this case, is the formation of additional martensite during the final quench to room temperature (so called “fresh” martensite) [43].
Calculations of the final fraction of austenite at room temperature were performed for the same alloy considering various fractions of intercritical ferrite, and the resulting curves are shown in Figure 2.3 [33]. The theoretical maximum fraction of RA after final quenching to room temperature is observed to be independent of intercritical ferrite fraction during annealing, as seen in Figure 2.3 [33], although the temperature associated with the maximum austenite fraction is shifted.

In later development of Q&P processing, authors reported that the theoretical RA fractions are not often achievable experimentally, as observed in Figure 2.4 [44]. These observations indicated that other competing factors, such as carbide precipitation, segregation of carbon to dislocations, and austenite decomposition into carbide-free bainite, for example, can occur during the partitioning step [45]. Work by Pierce et al. [46], using Mossbauer spectroscopy as a complimentary technique to transmission electron microscopy (TEM) and x-ray diffraction (XRD), showed that up to 41 % of the bulk carbon could be found in the form of η-carbides for a 0.38C-1.52Mn-1.48Si (wt. %) alloy. This amount of carbide precipitation helps to explain the variations of RA that is observed experimentally.
2.1.2 Hot-Rolled Q&P

The concept and feasibility of HR Q&P was first proposed by Thomas et al. [47–49]. In Thomas’s work [48], the main objectives were to apply the Q&P concepts to a hot-strip mill context and

Figure 2.3  Calculated final austenite fractions after partitioning and cooling to room temperature for Fe-0.19C-1.46Mn-1.96Al (wt. %), having 0, 50, or 75 % intercritical ferrite ($\alpha_{IC}$) [33].

Figure 2.4  Final austenite volume fraction as a function of quench temperature for different partitioning times for samples intercritically annealed at 820 °C for 180 s, quenched and held for 10 s, partitioned for 10, 30, 100 or 1000 s at 400 °C, and then water quenched to room temperature. Arrows highlight the behaviors exhibited with increasing partitioning time for each quench temperature. The final austenite volume fraction versus quench temperature is also shown (dashed line) calculated using the optimal quench temperature method [33,44].
understand if the non-isothermal partitioning (where carbon partitions from martensite to austenite during coil cooling) could be compared to the isothermal partitioning concept described in Section 2.1.1. For that, calculations based on the diffusivity of carbon were made and proved that coiling temperature (CT) could serve as the end of quenching and the onset of partitioning in a non-isothermal condition [48]. The schematic shown in Figure 2.5 was proposed, applying the non-isothermal Q&P theory to a hot-strip mill processing configuration.

In Thomas’s work [48] a variety of heat treatments (fully austenitized and intercritically annealed) in combination with a range of CTs (from 20 to 500 °C in 25 °C increments) followed by coil cooling simulation (up to 40 hours depending on the CT) were performed in a Gleeble® 1500. Deformation/rolling was not included in these simulations. After the heat treatments, microstructural analysis, RA measurements, and uniaxial tensile tests were performed for each condition. Figure 2.6 illustrates how the mechanical properties of non-isothermally treated HR Q&P conditions varied with CT (after intercritical annealing) and the types of microstructures that correlated to each CT range [48].

The mechanical properties obtained from Thomas’s work were compared and plotted against properties obtained by Clarke [44], as shown in Figure 2.7. Clarke’s work was based on isothermal partitioning and austempering conditions (related to CR Q&P). The overlap in properties between the two studies, as presented in Figure 2.7, shows that non-isothermal partitioning is viable and can produce similar properties and microstructures as obtained in CR Q&P steels.

![Figure 2.5 Schematic of a thermomechanical profile proposed to create Q&P microstructures directly from the hot-strip mill. PTi designates the initial partitioning temperature dictated by the coiling temperature (CT), which is also the quenching temperature (QT) [49].](image-url)
Figure 2.6 Mechanical properties as a function of coiling (quench) temperature for samples that were intercritically annealed and then quenched and subjected to a coil cooling profile. Ultimate tensile strengths (UTS) and yield strengths (YS) are plotted on the axis to the left. Uniform elongations (UE) are plotted on the axis to the right. The temperature range for each observed microstructure type is indicated qualitatively above the plotted data. The regions indicated are approximate, and can overlap [48].

Figure 2.7 Uniform elongation versus ultimate tensile strengths from Clarke’s work compared with data from Thomas’s work. Processing conditions employed by Clarke included Q&P, austempering, and direct quenching. Solid squares and solid diamonds represent data from Thomas’s hot-rolling simulations after intercritical annealing (IC) or full austenitization (F), respectively [48].
2.2 Other Studies on Hot-Rolled Q&P

Thomas’s work [47–49] pioneered hot-rolling simulations of Q&P processing and opened opportunities for further development of alloying and processing strategies to improve properties with a fuller understanding of the mechanisms associated with HR Q&P. Several studies have been published subsequently on HR Q&P, and results are presented in this section.

2.2.1 Direct Q&P

Somani et al. [50–52] investigated a thermomechanical processing (TMP), called direct quenching and partitioning (DQ&P), based on concepts initially developed by Speer et al. [33] and Thomas et al. [48]. The alloys used in these studies were denominated High-Si with composition of Fe-0.2C-1.5Si-2.0Mn-0.6Cr (wt. %) and High-Al with Fe-0.2C-0.5Si-1.0Al-2.0Mn-0.5Cr-0.2Mo (wt. %).

Dilatometric studies on a Gleeble® 1500 were conducted to determine parameters that were later used to simulate rolling in a laboratory mill. Austenite conditioning was also studied using a Gleeble® 1500. The same Q&P heat treatment was performed on specimens with two different austenite conditions: unstrained, and strained (three compression hits of 0.2 strain/pass and strain rate of 1 s⁻¹ at 850 °C). Based on analysis of the dilatometry and the Q&P experiments, laboratory rolling was performed with variations in finish rolling temperature (FRT) and QT. High FRT was used to simulate recrystallized (unstrained) austenite and low FRT was used to simulate deformed (strained) austenite. The partitioning step was done in a furnace by leaving the hot-rolled plates in the furnace for 10 minutes at the same temperature as the QT (similar to 1-step Q&P, where PT = QT) followed by water quenching. Coiling simulations (CS) – similar to Thomas’s experiment [48] - were also performed by switching off the furnace at the desired CT (which is also equivalent to QT) and lasted about 30 hours.

Results from the Q&P experiments performed in a Gleeble® indicated that prior strain in the austenite did not seem to show a large effect in these steels on the final fraction of RA or carbon content in the austenite. Straining the austenite prior to the Q&P heat treatment influenced the final microstructure, however. Microstructural analysis revealed refinement in the strained condition (smaller martensite packets/blocks), as observed in Figure 2.8.

Table 2.1 provides a summary of the mechanical properties of the laboratory hot-rolled steels obtained via DQ&P processing for the two alloys studied by Somani et al., including 0.2 % offset YS, UTS, UE, and 27 J transition temperature (T27J) from Charpy V-notch testing. The High-Si material with high FRT showed lower YS and higher impact transition temperature when compared to the same material with low FRT. The coiling simulation at 290 °C showed similar YS and UTS when compared to the same material that was isothermally partitioned at the same temperature. The property that was most influenced by FRT was the transition temperature. Small grain size (as observed in Figure 2.8b) greatly
influences impact and fracture toughness due to a more tortuous path for crack propagation. The High-Al alloy displayed slightly better overall properties at lower QT (270 and 320 °C). The low FRT was not as effective in improving mechanical properties for the High-Al alloy as it was for the High-Si. This was probably due to the presence of ferrite formed during rolling, which suggested that the FRT should be increased to avoid ferrite formation in the High-Al steel [50,51].

![Figure 2.8](image)

**Figure 2.8** Light optical micrographs of (a) unstrained and (b) strained samples of High-Si (Fe-0.2C-1.5Si-2.0Mn-0.6Cr wt. %) steel [51].

<table>
<thead>
<tr>
<th>Steel</th>
<th>FRT (°C)</th>
<th>QT/PT (°C)</th>
<th>0.2% YS (MPa)</th>
<th>UTS (MPa)</th>
<th>UE (%)</th>
<th>T27J (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>High-Si</td>
<td>800</td>
<td>290 CS</td>
<td>1029</td>
<td>1505</td>
<td>4.6</td>
<td>-108</td>
</tr>
<tr>
<td></td>
<td></td>
<td>320</td>
<td>1047</td>
<td>1391</td>
<td>3.2</td>
<td>-100</td>
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<tr>
<td></td>
<td>1000</td>
<td>320</td>
<td>953</td>
<td>1393</td>
<td>4.4</td>
<td>-44</td>
</tr>
<tr>
<td>High-Al</td>
<td>800</td>
<td>270 CS</td>
<td>1180</td>
<td>1495</td>
<td>3.9</td>
<td>-87</td>
</tr>
<tr>
<td></td>
<td></td>
<td>320 CS</td>
<td>1020</td>
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<td>890</td>
<td>310 CS</td>
<td>1116</td>
<td>1393</td>
<td>3.0</td>
<td>-67</td>
</tr>
</tbody>
</table>

### 2.2.2 Hot-Rolled Directly Q&P

Another processing schedule (also based on CR and HR Q&P concepts) named hot-rolled directly quenched and partitioned (HDQ&P) has been proposed. The HDQ&P process is the HR Q&P process adapted for TMP with ultra-fast cooling (UFC) technology (up to 200 °C/s) [18,22,53]. The partitioning step in HDQ&P processing can be performed either isothermally or dynamically (non-isothermally). It is
important to note that the dynamic partitioning in this case is different from the coil cooling simulations described in Section 2.1.2. In the HDQ&P case, water or air cooling was used to cool from QT to room temperature, which takes only a few minutes, in contrast to 20-45 hours to simulate coil cooling as previously described.

The first alloy studied was Fe-0.21C-1.67Si-1.65Mn-0.2V wt. % for the HDQ&P processing. Xu et al. [22] compared hot-rolled TRIP to the HDQ&P processing (with intercritical ferrite) with varying isothermal hold times (i.e. bainitic hold for the TRIP steel at 550 °C, and partitioning at 390 °C after quenching at lower temperature) for the same steel. Tan et al. present results for the same alloy. In the study by Tan et al. [18], comparisons were made between three processing routes (all rolling was done above Ar₃, so that no pro-eutectoid ferrite formed): (a) quench to room temperature after rolling (water or air cooling); (b) quench to an intermediate temperature (between Ms and Mf) followed by dynamic partitioning; and, (c) quench to an intermediate temperature (between Ms and Mf) followed by isothermal partitioning at a higher temperature, as seen in Figure 2.9. In a separate study with the same alloy, Tan et al. [20] compared processing types (a) and (b) from Figure 2.9, with conditions containing proeutectoid ferrite, as shown in Figure 2.10a. More recently, Tan et al. [19,53] introduced a new alloy to their study (without addition of V, with reduced Si content and a small P addition) with composition of Fe-0.22C-0.52Si-1.89Mn-0.079P wt. %. In [19], comparisons between the two alloys were drawn for processing with and without proeutectoid ferrite for isothermal partitioning (Figure 2.10b and Figure 2.10c). And finally, in [53], processing (a), (b) and (c) (from Figure 2.9) were performed for the Fe-0.22C-0.52Si-1.89Mn-0.079P (wt. %) alloy. The results from these processing are summarized in the following paragraphs.

Figure 2.9 Schematic thermal profiles of the HDQ&P process: (a) direct quenched, (b) non-isothermal, and (c) isothermal partitioning processes [18]. Quenching temperatures (QT) for process 3 and 4 were 275 and 280 °C, respectively. QT for 5, 6, 7 and 8 were 290, 285, 278 and 285 °C and tp were 1, 2, 5 and 15 minutes, respectively [18].
Figure 2.10  (a) Schematic thermal profiles of the HDQ&P process. Finish rolling temperature (FTR) was 900°C for all processes. Sheet 1 was air-cooled to 850 °C and directly quenched to room temperature (no formation of pro-eutectoid ferrite) after rolling. The other sheets were air cooled to 830 °C (#2) (no pro-eutectoid ferrite), 790 °C (5 % pro-eutectoid ferrite) (#3) and 720 °C (50 % pro-eutectoid ferrite) followed by quenching to the desired QT and air cooling to room temperature. QT = 302, 385 and 480 °C for sheets 2, 3 and 4, respectively [20]. Schematic thermal profiles of the HDQ&P process without (b) and with (c) pro-eutectoid ferrite (30 % volume fraction). For (b) FTR = 900 °C followed by quenching to QT (320 °C) and partitioning at 400 °C for 4 minutes. For (b), FTR = 850 °C followed by air cooling to 750 °C, quenching to QT (320 °C) and partitioning at 400 °C for 4 minutes [19].

The microstructures observed by Tan et al. [18], with processing schematics shown in Figure 2.9, showed the presence of lath martensite and interlath RA for all processing conditions, with increasingly coarser carbides as isothermal hold time was increased. Very small, spherical V-bearing carbides were observed in the microstructure via TEM, which were assumed to precipitate during cooling and believed to contribute to the high strength levels obtained in these processing routes. Stability of RA was also studied. Results showed that in the dynamically partitioned conditions (Figure 2.9b), the RA has lower carbon concentration. Decreased carbon saturation in RA allows it to transform more easily, influencing the TRIP effect in this alloy. The isothermally partitioned (Figure 2.9c) material exhibited greater carbon enrichment in the RA, making it more stable during deformation. In terms of mechanical properties, it was shown that the dynamically partitioned conditions had slightly better combinations of mechanical properties, with higher UTS, similar YS, and similar elongations when compared to the isothermally partitioned conditions. Ultimate tensile strengths ranged from 1300 to 1600 MPa, and total elongations from 14 to 18 %.

Additional work by Tan et al. [20], based on the processing schematic presented in Figure 2.10a, showed the effects of pro-eutectoid ferrite on HDQ&P processing. The formation of 5 % intercritical
ferrite seemed to increase the fraction of RA, while the formation of 50% ferrite substantially decreased the RA fraction. The authors attributed this difference mainly due to a decreased amount of untransformed austenite available and the fact that the sheet with 50% ferrite was quenched to 480 °C, which allowed formation of bainite and consumed more of the untransformed austenite [20]. Even with an increase in RA for condition #3 (Figure 2.10a), no improvement in mechanical properties was observed. Ultimate tensile strengths in the range of 1600 to 1700 MPa and elongations in the range 11.5 to 13% were observed for conditions #2 and #3. With an introduction of about 50% intercritical ferrite, tensile strength decreased by about 400 MPa, with an increase in elongation of approximately 2% [20]. Conditions 3 and 4 (from Figure 2.10a) were very different, however, which introduced a number of variations, making the analysis difficult.

Tan et al. [19] also investigated isothermal partitioning (Figure 2.10b and Figure 2.10c) in two alloys: a high-Si V-bearing alloy (Fe-0.21C-1.67Si-1.65Mn-0.2V wt. %), and a low-Si P-added alloy (Fe-0.22C-0.52Si-1.89Mn-0.079P wt. %) with and without intercritical ferrite. The High-Si V-bearing alloy reportedly showed more retention of austenite with higher austenite stability for both cases (with and without ferrite). Mechanical properties of the High-Si V-bearing alloy were improved in both situations as well, when compared to the Low-Si alloy. The differences in mechanical properties were attributed, by the authors, to the influence of precipitation strengthening from V precipitates formed upon cooling and solid solution strengthening of Si [19]. Note that there is a significant variation in Si in both alloys, therefore attributing the strengthening entirely to V may be improper without further analysis.

2.3 Microalloying in Hot-Rolled Sheet Steel

Additions of microalloying elements, especially Nb, in hot-rolled steels containing RA (such as TRIP and TBF steels) have been studied. The effects of Nb on hot-rolled sheet products are further explored in the next sections.

2.3.1 Hot-Rolled TRIP Steels

Several studies have been published on hot-rolled TRIP steels, comparing a reference alloy with and without additions of Nb to investigate different processing effects and compare prior austenite conditioning (recrystallized versus pancaked) [54–58]. Additions of Nb showed significant overall microstructure refinement in each study. In most cases, Nb seemed to improve mechanical properties (especially increased strength) while decreasing strain hardening. Nb-bearing alloys were also reported to increase the fraction of RA, and, in most cases, increase its stability [54–58].

Pereloma et al. [58] studied the effects of austenite conditioning, recrystallized versus pancaked, for two steels 0.2C-1.55Mn-1.5Si wt. % with and without the addition of 0.039 wt. % Nb. When comparing different austenite conditions, the pancaked austenite seemed to promoted a more uniform and
refined final microstructure with higher fractions of low transformation temperature constituents. Similar fractions and stabilities of RA were observed for both conditions. For the same austenite conditioning, the Nb-bearing alloy appeared to retain more austenite. Work by Feng et al. [55] also examined austenite conditioning for a 0.2C-1.5Mn-0.5Si-1.04Al-0.038Nb wt. % alloy. They showed refinement of microstructure and improved combination of mechanical properties for the pancaked condition. Pancaked austenite appeared to result in a larger fraction of RA for this study.

2.3.2 Hot-Rolled TBF Steels

TBF steels (also called carbide-free bainite, or CFB) are also being developed as part of the 3rd generation of AHSS. These steels are produced via isothermal holding in the bainitic region after fast cooling from a temperature above $A_3$. The general TBF steel microstructure consists of bainitic ferrite with dispersed regions of RA [59].

A systematic study by Hashimoto et al. [23] focused on the effects of Nb and Mo additions on hot-rolled TBF steels, with varying isothermal holding temperatures. Isothermal holds were performed at temperatures ranging from 300-500 °C with 50 °C increments with 10 minutes hold followed by air cooling. Five alloys were used in the study: a base alloy (B) with 0.2C-1.5Si-1.5Mn (wt. %), two alloys with Nb additions (0.02Nb (LN) and 0.05Nb (HN)) and two alloys with Nb and Mo additions (0.02Nb-0.1Mo (LNM) and 0.05Nb-0.2Mo (HNM)).

In the work by Hashimoto et al. [23], additions of Nb and Mo had a significant effect on the mechanical properties and the stability and morphology of the RA. Figure 2.11 is a summary of the mechanical properties (tensile strength x total elongation) for all five alloys based on the different CTs. From Figure 2.11, it can be noted that the microalloyed steels showed a better combination of properties for all CTs studied. Coiling at 400 °C seemed to be the optimum temperature for all steels except LNM and the HN and HNM showed a significant increase in combined properties at this temperature when compared to the other alloys. Fine complex carbides (NbC, in the Nb-containing alloys and (NbMo,C), in the Mo-containing alloys) were found, which were assumed to contribute to the additional strength when compared to the B alloy in most cases. Figure 2.12 presents EBSD image quality and phase maps showing how the addition of Nb affected the distribution and size of the RA. The addition of Nb influenced the RA morphology. The austenite is more evenly and finely distributed in the HN steel than the RA in the B steel, which is blocky and more heterogeneously distributed [23].

2.4 Microalloying in Cold-Rolled Sheet Steels

Several studies have been conducted on the effects of microalloying elements, especially Nb, on cold-rolled sheet products that contain RA. Initial studies focused on TRIP steel, which is part of the 1st
generation of AHSS, while later studies investigated microalloying additions on TBF and Q&P steels. Some results obtained in these studies are presented in the following sections.

Figure 2.11 Effects of alloying elements and coiling temperature (CT) on tensile strength (TS) and total elongation (El) of steels held for 10 minutes at the CT. B = 0.2C-1.5Si-1.5Mn, LN = B+0.02Nb, LNM = LN+0.1Mo, HN = B+0.05Nb and HNM = HN+0.2Mo (in wt. %) [23].

Figure 2.12 EBSD scans of steels B and HN coiled at 400 °C and held for 10 minutes. Top row shows the image quality map (IQ) and the phase map for steel B and bottom row, IQ map and phase map for steel HN. FCC-Fe (γ) is represented in red and BCC-Fe (α) in green [23]. (Color image – see PDF version)
2.4.1 Cold-Rolled TRIP Steels

The effects of Nb and V microalloying in cold-rolled TRIP steel have been studied by several authors. In general, the objectives of the microalloying strategies are to refine the austenite grain size, strengthen the steel matrix via precipitation hardening, and vary the phase transformation kinetics (which may affect phase fractions and stability).

Figure 2.13 shows the effects of Nb addition on TRIP steels with respect to RA fraction as a function of holding temperature and time. According to Hulka [60], the higher fraction of RA in Nb-bearing alloys can be explained by the grain refinement effect caused by the Nb additions. First, a finer austenite grain size formed in the austenite/ferrite two-phase region will promote ferrite nucleation and also retard bainite formation kinetics during cooling (from the intercritical annealing temperature to the austempering temperature), enhancing carbon enrichment of the remaining austenite (after bainite transformation). Second, the finer grain size may lower the $M_s$, which is directly related to the stability of the austenite at room temperature. The final austenite fraction is generally dependent on the various mechanisms that operate during transformation, which include carbon enrichment, grain refinement, and inhibition of martensite nucleation [61]. In terms of mechanical properties, additions of Nb in TRIP steels have been reported to slightly increase strength and elongation [62].

Vanadium additions in TRIP steels have also been studied. In work by Scott et al. [63], an increase in UTS of about 100 MPa was achieved with no significant loss in elongation with additions of 0.1 wt % V and 0.007 wt % N. In the same work, the addition of V did not seem to affect fractions or stability of RA. The design was to strengthen ferrite via precipitation of VN while maintaining sufficient levels of carbon in solution to stabilize the RA. Additions of V are widely used in hot-rolled HSLA steels, but are less common in cold-rolled products. This is because V precipitates are considered to coarsen faster than Nb precipitates during annealing, which can decrease the strengthening contribution [17].
2.4.2 Cold-Rolled TBF Steels

Systematic studies on the formability of TBF steels were conducted by Sugimoto et al., where the role of Nb was studied individually [26] and in combination with other alloying elements such as Si, Al, and Mo [27–29]. Hausmann et al. [24,25] also studied the effects of Nb additions on cold-rolled TBF steels.

The reference alloy for the study conducted by Hausmann et al. [24,25] was Fe-0.17C-0.78Si-2.6(Mn+Cr+Mo) in wt. %, to which three levels of niobium were added: 0.025, 0.045 and 0.090 wt. %.

Average prior austenite grain size (PAGS) was quantified, and the additions of Nb lead to substantial refinement of the austenite (from 12.1 µm in the reference material to 5.3-6.0 µm in the Nb-added steels). From the microstructural analysis, it was observed that the addition of Nb also modified the bainite morphology, from lath-type bainite in the Nb-free samples to a more globular or granular bainite in the samples with different Nb additions. The RA morphology also changed from lath to blocky with the addition of Nb. Figure 2.14 presents TEM micrographs showing the differences in the austenite and bainite morphology between the Nb-free and Nb-bearing samples. It was also observed that the amount of cementite in the Nb-bearing alloys was significantly reduced, allowing more carbon to stabilize higher fractions of austenite in these alloys, as shown in Figure 2.15. In terms of mechanical properties, greater ductility was observed (without any or with a small loss in strength) with the addition of Nb, which was associated with the increased RA fractions. On the other hand, Nb additions did not seem to have important effects on hole expansion formability when compared to the reference material.

Sugimoto et al. [26] used a very similar reference alloy, Fe-0.2C-1.5Si-1.5Mn, with four niobium additions: 0.020, 0.048, 0.078 and 0.109 (wt. %). The results reported from both groups have many similarities, including refinement of the PAGS, change in morphology of the bainite and austenite, reduced cementite fractions, and increased fractions of RA (in most cases) - without improving its stability - of the Nb-bearing steel when compared to the reference material. Nb did not substantially influence tensile strength, although it showed improvement in total elongation for some cases, which was also similar to results obtained by Hausmann et al. [24]. On the other hand, Sugimoto et al. [26–29] reported significant improvements to hole expansion strains with additions of Nb, while Hausmann and co-workers [25] reported little to no improvement.

2.4.3 Cold-Rolled Q&P Steels

Recent studies conducted by Zhou et al. [30] and Zhong et al. [31,32] have examined the effects of Nb and Mo additions in quenching-partitioning-tempering (Q-P-T) steels. The Q-P-T heat treatment is based on the 2-step Q&P process (described in Section 2.1.1), where the tempering step can be performed at the same (Figure 2.16a), lower (Figure 2.16b) or higher (Figure 2.16c) temperature than the PT. The
tempering step is believed to promote precipitation of complex carbides in steels that contain carbide-forming elements such as Nb, V, and Mo; or to promote the precipitation of transitional carbides in steels that do not contain such elements [64]. In practice, Q-P-T processing is often indistinguishable from Q&P processing, particularly as tempering temperature (TT) is often coincident with the PT.

Figure 2.14 TEM micrographs of a) 0, b) 0.025, c) 0.045, and d) 0.09 wt % Nb heat treated TBF steels (at overaging temperature and time of 400 °C and 600 s, respectively) [25].

Figure 2.15 Amount of RA for alloys with 0 (0Nb), 0.025 (25Nb), 0.045 (45Nb) and 0.090 (90Nb) niobium (in wt. %) as a function of overaging time for an overaging temperature of 400 °C [25].

Zhong et al. [31,32] analyzed a cold-rolled sheet steel with composition of Fe-0.2C-1.5Mn-1.5Si-0.05Nb-0.13Mo (wt. %). They compared the microstructures with those of a non-microalloyed steel (Fe-0.2C-1.5Mn-1.5Si) for one heat treatment (austenitizing at 900 °C, quenching to 280 °C and partitioning at 480 °C for 20 s). The microstructures obtained from that heat treatment are shown in Figure 2.17, and a clear refinement of the microstructure is observed in the microalloyed steel. The difference in PAGS reported by the authors is 20 µm for the non-microalloyed steel versus 8 µm for the Nb-bearing steel. The
heat treatments performed in the Nb-bearing alloy showed a good combination of mechanical properties, in the range of 1500 MPa tensile strength with 15 % elongation. Microstructures were composed of very thin laths of RA, martensite, and very finely dispersed Nb precipitates in the martensite laths, as shown in the TEM images of Figure 2.18. It was proposed that the high strength of the material came from the fine martensite laths with dispersed carbide precipitates in the matrix, and the high ductility and toughness came from the film-like interlath RA and the refined structure of the material [31,32].

![Figure 2.16](image)

Figure 2.16 Schematic of Q-P-T heat treatment cycles for (a) PT = TT, (b) PT higher than TT, and (c) PT lower than TT. Note: AT, QT, PT, TT and RT are austenitizing, quenching, partitioning, tempering (precipitation) and room temperature, respectively [64].

Work by Zhou et al. [30] also focused on additions of Nb in cold-rolled sheet steel treated by Q-P-T with composition of Fe-0.25C-1.5Mn-1.2Si-0.05Nb-1.5Ni. A substantial range of mechanical properties was reported, where tensile strengths ranged from 1200 to 1500 MPa and total elongations from 14 to 18 %. Thin laths of martensite and RA with finely dispersed Nb precipitates in the martensite laths were also reported in this study.

A study has been published on CR Q&P sheet steel with additions of V, although the role of V was not clearly elucidated through comparison with a reference material. The alloy used by Tan et al. [21] is the same as in the studies presented in Section 2.2.2, with composition of Fe-0.21C-1.67Si-1.65Mn-0.2V (wt. %). All heat-treatments were performed using one-step Q&P processing (presented in Section 2.1.1), with varying partitioning times. Ultimate tensile strengths were in the range of 1230 to 1330 MPa, and total elongations in the range of 11 to 15 %. Analysis of microstructure showed lath martensite and RA (in the form of laths and blocky morphology). Spherical V-bearing carbides were found via TEM with diameters between 20 and 50 nm. The V-bearing carbides were believed to form before the initial Q&P processing, since austenitizing temperature and annealing time were not sufficient to dissolve the precipitates, and not enough time was available for V precipitates to form during the partitioning step at 310 °C [21].
Figure 2.17 Optical micrographs of heat treated samples (a) plain carbon steel (Fe-0.2C-1.5Mn-1.5Si in wt. %) and (b) microalloyed steel (Fe-0.2C-1.5Mn-1.5Si-0.05Nb-0.13Mo in wt. %) after austenitization at 900 °C for 180 seconds, followed by quenching to 220 °C and partitioning at 480 °C for 20 seconds. [32].

Figure 2.18 TEM micrographs showing niobium carbide precipitation in the martensite matrix of the sample partitioned at 400 °C for 40 seconds. (a) Bright field and (b) dark field with SAD (selected area diffraction) pattern [31].
CHAPTER 3: EXPERIMENTAL PROCEDURES

This chapter presents the details of material selection and material processing, as well as details on characterization and analysis methods used throughout the study.

3.1 Material Selection

A common Q&P steel composition was selected for the base alloy, namely 0.2C-1.5Si-2.0Mn (wt. %). Silicon is added to these alloys to retard precipitation of cementite and thus enhance partitioning of carbon into the austenite [24]. Avoiding cementite (and transitional carbides) formation is of major interest in Q&P steels, since the formation of carbides consumes carbon and would result in less carbon to stabilize the austenite at room temperature [42]. Manganese enhances hardenability and helps to stabilize austenite by lowering its transformation temperature. Manganese may also increase carbon solubility in austenite, which enhances the possibility of carbon enrichment of austenite [42]. Additions of Nb and V were proposed based on literature and analysis of solubility (see Appendix A – page 90). The higher Nb addition of 0.045 wt. % is about the maximum effective content for 0.2 wt. % C and a reheat temperature of 1250 °C. Two alloys were then proposed with nominal additions of Nb, 0.020 and 0.045 wt. %. One alloy was designed with an addition of 0.06 wt. % V. The alloy compositions were designed so that the effects of Nb and V could be studied individually. Table 3.1 shows the composition of the four ingots that were air cast at AK Steel. Each ingot had approximate dimensions of 150 x 150 x 75 mm (width x length x thickness).

Table 3.1 – Chemical Composition of Experimental Steels Produced by AK Steel

<table>
<thead>
<tr>
<th>wt. %</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base</td>
<td>0.20</td>
<td>2.01</td>
<td>1.55</td>
<td>0.004</td>
<td>0.003</td>
<td>&lt;0.003</td>
<td>&lt;0.003</td>
</tr>
<tr>
<td>Low Nb</td>
<td>0.20</td>
<td>1.99</td>
<td>1.52</td>
<td>&lt;0.003</td>
<td>0.005</td>
<td>&lt;0.003</td>
<td>&lt;0.003</td>
</tr>
<tr>
<td>High Nb</td>
<td>0.20</td>
<td>1.99</td>
<td>1.56</td>
<td>&lt;0.003</td>
<td>&lt;0.003</td>
<td>&lt;0.003</td>
<td>&lt;0.003</td>
</tr>
<tr>
<td>V Alloy</td>
<td>0.20</td>
<td>1.97</td>
<td>1.52</td>
<td>&lt;0.003</td>
<td>&lt;0.003</td>
<td>&lt;0.003</td>
<td>&lt;0.003</td>
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</table>

<table>
<thead>
<tr>
<th>wt. %</th>
<th>Nb</th>
<th>V</th>
<th>Al</th>
<th>N</th>
<th>S</th>
<th>P</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base</td>
<td>&lt;0.003</td>
<td>&lt;0.003</td>
<td>&lt;0.003</td>
<td>0.0062</td>
<td>&lt;0.001</td>
<td>&lt;0.003</td>
<td>&lt;0.003</td>
</tr>
<tr>
<td>Low Nb</td>
<td>0.021</td>
<td>&lt;0.003</td>
<td>&lt;0.003</td>
<td>0.0120</td>
<td>&lt;0.001</td>
<td>&lt;0.003</td>
<td>&lt;0.003</td>
</tr>
<tr>
<td>High Nb</td>
<td>0.044</td>
<td>&lt;0.003</td>
<td>&lt;0.003</td>
<td>0.0075</td>
<td>&lt;0.001</td>
<td>&lt;0.003</td>
<td>&lt;0.003</td>
</tr>
<tr>
<td>V Alloy</td>
<td>&lt;0.003</td>
<td>0.060</td>
<td>&lt;0.003</td>
<td>0.0074</td>
<td>&lt;0.001</td>
<td>&lt;0.003</td>
<td>&lt;0.003</td>
</tr>
</tbody>
</table>

3.1.1 Presence of Inclusions

Oxide inclusions were observed in the as-received ingots as a result of the air casting process. Figure 3.1a shows an example of an area with a high density of inclusions and Figure 3.1b is a higher magnification image of the same inclusions. An energy-dispersive x-ray spectroscopy (EDS) system
coupled to a field emission scanning electron microscope (FESEM) was used to characterize the inclusions present in the alloys. Figure 3.1c is an EDS spectra showing the relative Si/Al levels in the oxide stringers. The presence of inclusions might have had detrimental effects on the final mechanical properties of the materials reported later in this thesis.

![Image](image1.png)

Figure 3.1 (a) Secondary electron micrograph showing high density of inclusions, (b) inclusions at higher magnification, and (c) EDS spectrum showing the composition of inclusion particles.

3.2 Material Processing - Part I: Hot-Rolled Q&P Simulated via Gleeble® Torsion Testing

The top half of each ingot that was air cast at AK Steel was sent to CSM, with approximate dimensions of 150 x 75 x 75 mm (width x length x thickness). Hot rolling was performed for all lab heats in a FENN mill with the objective of breaking down the cast structure and obtaining a thickness that was suitable to machine sub-sized Gleeble® torsion specimens.

Hot rolling consisted of first soaking the ingots in an air furnace for approximately three hours at 1200 °C. Once the ingots were homogenized, five rolling passes were performed in less than two minutes and the plates were then put back in the furnace at 1200 °C to reheat. The reheat step took approximately 40 minutes. Another five passes were completed in less than two minutes. After the ten total passes, each
plate was straightened in a hand press while it was still hot. The plates were then air cooled. The average reduction per pass obtained in the process was 15 % and the total reduction achieved was 80 %. The final thickness of the plates was approximately 14 mm. The microstructures and the hardness measurements of the as hot-rolled material are provided in Appendix B (page 92).

3.2.1 Hot Torsion Simulations in a Gleeble® 3500

The geometry of the sub-sized torsion specimen is shown in Figure 3.2. Sub-sized geometry was chosen due to the limited amount of material and so that higher cooling rates could be achieved as a result of the smaller gauge diameter when compared to the standard geometry (7.2 mm versus 10 mm). Specimens were machined with their longitudinal axis aligned with the rolling direction of the plates.

![Figure 3.2 Engineering drawing of sub-sized torsion samples machined from the hot-rolled plates. Dimensions in millimeters [65].](image)

The time-temperature schedule performed in the Gleeble® 3500 to simulate hot-rolled sheet processing is schematically presented in Figure 3.3. During processing, the machined torsion specimens were heated to 1250 °C at a rate of 15 °C/s and held at temperature for 10 minutes to allow microalloy precipitates to dissolve. After 10 minutes, the samples were cooled to 1100 °C, followed by a multi-pass rolling schedule, which was adapted from the work done by Nakata and Militzer [66].

To program the multi-pass schedule into the Gleeble® software (QuickSim2®) (screenshots of the torsion program are available in Appendix C – page 95), the use of equations to calculate the rotations based on the equivalent strain given was necessary. Equations 3.1 to 3.4 [67,68] (variables listed and defined in Table 3.2) allow transformation of torque-twist data acquired from the Gleeble® into true equivalent stress and true equivalent strain. The detailed rolling schedule is presented in Table 3.3. Pass 1 had a large strain to simulate the roughing process. Passes 2 through 8 had lower strains per pass and each one simulated a finishing pass [66]. The nominal FRT was 900 °C.
An important variable in the equations is the effective radius \( a^* \), which is a radial position chosen to represent the torsional deformation behavior, since shear strain varies significantly in the radial direction. For the present study, the radial position chosen was 72.4 % of the gauge radius. The rationale for the use of this specific \( a^* \) was developed by Barraclough et al. [67] and has implications for both mechanical and microstructural interpretations of hot torsion testing. In Barraclough’s study, it was shown that at 72.4 % of the gauge radius, the effects of strain rate sensitivity and strain hardening behavior were minimized [67,69]. The selection of an effective radius also allows for microstructural examination away from the surface where effects of oxidation and/or decarburization might happen at elevated temperatures [69].

\[
\gamma_{a^*} = \frac{a^* \theta}{l} \quad 3.1
\]

\[
\tau_{a^*} = \frac{3 \Gamma}{2\pi a^3} \quad 3.2
\]

\[
\sigma_a = \sqrt{3} \tau_{a^*} \quad 3.3
\]

\[
\varepsilon_a = \frac{\gamma_{a^*}}{\sqrt{3}} \quad 3.4
\]
Table 3.2 – Definition of Variables

<table>
<thead>
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<th>Variable</th>
<th>Definition</th>
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<tbody>
<tr>
<td>$a$</td>
<td>Radius (mm)</td>
</tr>
<tr>
<td>$a^*$</td>
<td>Effective Radius, $0.724\cdot a$ (mm)</td>
</tr>
<tr>
<td>$\theta$</td>
<td>Angle of Twist (rad)</td>
</tr>
<tr>
<td>$l$</td>
<td>Gauge Length (mm)</td>
</tr>
<tr>
<td>$\Gamma$</td>
<td>Torque (N·m)</td>
</tr>
<tr>
<td>$\gamma a^*$</td>
<td>Shear Stain</td>
</tr>
<tr>
<td>$\tau a^*$</td>
<td>Shear Stress (N/mm$^2$)</td>
</tr>
<tr>
<td>$\sigma a$</td>
<td>Equivalent True Stress (N/mm$^2$)</td>
</tr>
<tr>
<td>$\epsilon a$</td>
<td>Equivalent True Strain</td>
</tr>
</tbody>
</table>

Table 3.3 – Multi-Pass Rolling Schedule Used for Thermomechanical Processing Simulation (Adapted from [66])

<table>
<thead>
<tr>
<th>Pass number</th>
<th>1</th>
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<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
</tr>
</thead>
<tbody>
<tr>
<td>Equivalent Strain</td>
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<td>0.36</td>
<td>0.51</td>
<td>0.41</td>
<td>0.33</td>
<td>0.30</td>
<td>0.25</td>
<td>0.10</td>
</tr>
<tr>
<td>Shear Strain</td>
<td>1.732</td>
<td>0.624</td>
<td>0.883</td>
<td>0.710</td>
<td>0.572</td>
<td>0.520</td>
<td>0.433</td>
<td>0.173</td>
</tr>
<tr>
<td>Torsion Angle (°)</td>
<td>548.3</td>
<td>197.7</td>
<td>279.6</td>
<td>224.6</td>
<td>18a1.1</td>
<td>164.4</td>
<td>137.0</td>
<td>55.0</td>
</tr>
<tr>
<td>Temperature (°C)</td>
<td>1100</td>
<td>1041</td>
<td>979</td>
<td>955</td>
<td>934</td>
<td>919</td>
<td>908</td>
<td>900</td>
</tr>
<tr>
<td>Interpass time (s)</td>
<td>10.0</td>
<td>8.0</td>
<td>4.8</td>
<td>3.2</td>
<td>2.3</td>
<td>1.7</td>
<td>1.3</td>
<td>-</td>
</tr>
</tbody>
</table>

After the multi-pass rolling (torsion) simulation, the specimens were fast cooled with helium gas. One specimen of each alloy was quenched to room temperature immediately after the deformation steps (direct quench samples as shown in Figure 3.3) with the purpose to analyze the austenite condition (morphology) and grain size after the torsion steps. The other four samples of each alloy were quenched to a specific CT – which also represents the QT or the initial PT in the context of HR Q&P simulations [48,49]. The CTs chosen were based on Thomas’s work [48], and were nominally 275, 325, 375 and 425 °C. Once the CT was reached, the coil cooling simulation would start, meaning that these samples were cooled at a very slow rate. The total time of the coil cooling simulations was 24 hours and the cooling profiles were also based on Thomas’s work [48].

The typical practice for specimens that are heat treated or deformed in a Gleeble® is to spot weld thermocouples to the center of the gauge length, where maximum temperature can be captured. However in torsion tests, when the twists that simulate rolling passes are applied, the thermocouples often disconnect from the specimen if they are attached to the center of the gauge length. In most torsion experiments, the optical pyrometer (located outside the chamber) is thus preferred for temperature monitoring. In some cases, however, control of the temperature below the optical pyrometer limit (below 650 °C) is necessary. Sun [70] and Miller [65] have developed alternative methods to measure local temperature for the cases where low temperatures need to be recorded after torsion deformation. They proposed that the best method is to attach thermocouples to the shoulder of the specimen (at the very end
of the gauge section where there is no deformation) and account for the difference in temperature between
the center and the shoulder readings. Because low CTs (below the pyrometer capability) are of interest in
this work, the method proposed by Sun and Miller was used. An extensive study on the best location to
attach the shoulder thermocouples was conducted and the temperature reproducibility throughout the
gauge length was examined for the processing route proposed in Figure 3.3. Various tests were conducted
(without deformation) and the difference in temperature between the center and the shoulder
thermocouples was determined to be 5 ± 2 °C.

Based on the limitations related to temperature measurement during torsion testing, the
temperature control in the Gleeble® 3500 for the processing route proposed was done via optical
pyrometer at temperatures above 650 °C (upon heating, soaking and during the deformation steps) and it
was switched to shoulder thermocouples during the quenching step. The pyrometer was adjusted to
monitor the center of the specimen (Figure 3.4) and record the maximum temperature in the specimen.
The shoulder thermocouple was attached on the left side (fixed end) of the sample, 0.5 mm away from the
dge, according to the schematic shown in Figure 3.4.

![Schematic showing the pyrometer reading location (center of the sample) and the
shoulder thermocouple location. Note that the left side is fixed and the right side rotates.
The dashed line represents the axis of rotation. Drawing not to scale.](image)

All torsion tests were conducted under high vacuum to avoid decarburization and oxidation of the
surface of the material at high temperature. Vacuum would break once helium was inserted to the
chamber during quenching. For the coil cooling experiments, two flow rates of helium were used, a high
flow rate to rapidly achieve the desired CT and a very low flow rate so the heat from the grips and
specimen shoulders would not reheat the gauge section. The use of two flow gas rates was essential, as it
was noticed during trials that the temperature at the center would increase as much as 250 °C without the
application of a second gas flow.

The non-recrystallization temperature ($T_{nr}$) was calculated based on empirically derived equations
available in literature. The Fletcher equation [71] was the one that more closely related to the
experimental alloys used in the present study. The calculated $T_{nr}$ is presented in Table 3.4. Based on the
calculated values and considering that the FRT used in the Gleeble® processing was 900 °C, the High Nb
alloy would be expected to suppress recrystallization to a certain extent.
Table 3.4 – Non-Recrystallization Temperature (T_{nr}) Calculated via Empirically-Derived Equation [71]

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Base</th>
<th>Low Nb</th>
<th>High Nb</th>
<th>V Alloy</th>
</tr>
</thead>
<tbody>
<tr>
<td>T_{nr} (°C)</td>
<td>779</td>
<td>877</td>
<td>921</td>
<td>800</td>
</tr>
</tbody>
</table>

3.2.2 Hardness Measurements

Vickers hardness measurements were used to compare mechanical properties amongst the different alloys and coil cooling conditions in this part of the study due to sample geometry limitations. Figure 3.5 is a schematic illustration that shows how the torsion specimens were sectioned and the two views that were analyzed. The transverse cross-sections were obtained by cutting the specimen perpendicular to the torsional axis, at the center (where the temperature was controlled). The tangential cross-sections were obtained by sectioning the specimen parallel to the torsional axis at a distance near the effective radius followed by grinding (after mounting) to a depth representing the effective radius. The effective radius was identified based on a chord-length calculation, using the width of the ground cross-section to define the chord-length [69].

![Figure 3.5](image)

Figure 3.5 Torsion gauge length with arrows pointing to the two cross-sections (shaded gray) used for the analysis. Dashed lines represent the effective radius of interest. Drawing not to scale.

Samples were mounted in Bakelite, ground using 240, 320, 400 and 600 grit abrasive paper, followed by polishing with 6, 3 and 1 μm diamond slurry. After the samples were polished, Vickers microhardness indents were made using a LECO MHT Series 200 with 500 g load. The lengths of the two diagonals of each indent were measured with an Olympus PMG 3 light optical microscope (LOM) using PAX-it Image analysis software. All measurements of the indents were taken at a magnification of 1000x after proper calibration. Equation 3.5 [72] represents how the measurements obtained in the LOM were converted into Vickers Hardness (HV) values, where L is load in kgf and d is the average of the two diagonals of each indentation in mm.

\[
HV = 0.018544 \cdot \frac{L}{d^2} \tag{3.5}
\]
Figure 3.6 shows the two cross-sections of interest and the approximate location where the indents were made along the effective radius. For the transverse cross-section, eight indents were made 45° apart from each other. On the tangential cross-section, eight indents separated by 0.15 mm were performed, which was more than 2.5 times the indent diagonal (center to center) from each other, in accordance with the ASTM E384 standard [72].

![Figure 3.6](image)

(a) Transverse cross-section of the torsion specimen. Solid diamonds show approximate position of the microhardness indents that were separated by 45° along the effective radius (a*) (dashed line). (b) Tangential cross-section of the torsion specimen. Solid diamonds represent the approximate position of the indents that were separated by 0.15 mm along the length (dashed line). Drawing not to scale.

### 3.2.3 Imaging and EBSD on Gleeble® Tested Specimens

After microhardness measurements were made, samples were chemically etched with a 2 % nital solution (98 % ethanol and 2 % nitric acid) for 5 to 10 seconds. Samples were observed using a JEOL 7000 FESEM operated at 10 keV and 10 mm working distance. Images were taken at the effective radius (indents were used as reference) from both cross-sections for all alloys and conditions.

One EBSD scan was obtained, in the tangential cross-section, for each alloy and coiling condition in order to quantify the amount of RA. Each sample was prepared in a vibratory polisher with a 0.02 µm colloidal silica solution for four hours. Scans were made using the FESEM equipped with an EDAX DigiView camera and EDAX OIM-DC 5.2 collection software. Operating conditions were 70° tilt, 16 mm working distance, 60 nm step size, operated at 20 keV with scan areas of 30 x 35 µm.

EBSD scans were also obtained to reconstruct PAGS. Scans were performed in a FEI Helios NanoLab 600i focused ion beam (FIB) microscope equipped with a Hikori camera and EDAX OIM-DC 7.0 collection software. Operation conditions were 70° tilt (pre-tilted stage was used in this case), 8 mm working distance, 200 nm step size, 20 keV with scan areas of 80 x 80 µm.

Parent austenite grain reconstruction was performed on the EBSD data to assess the morphology of the parent phase grain structure, with assistance of Tuomo Nyyssonen. The reconstruction algorithm was written on Matlab™ using the MTEX texture and crystallography analysis toolbox developed by Bachmann et al. [73]. A grain map was assembled from the orientation pixel map with a 3° minimum
grain boundary threshold. The data were manually cropped to select an area containing martensitic orientations from a single PAG, from which an average orientation relationship between austenite and martensite was determined using the method by Miyamoto et al. [74]. The orientation relationship was used to reconstruct the PAG orientation map using the methodology of Cayron et al. namely by finding “nuclei” for PAGs and growing them (numerically) to full size on a neighbor-to-neighbor basis [75].

3.3 Material Processing – Part II: Laboratory Hot-Rolled Q&P Simulation

Laboratory HR Q&P simulation was also conducted using the remaining half of each ingot prior to cold rolling at AK Steel. Each ingot bottom (Figure 3.7a) was rough-rolled to about 15 mm (Figure 3.7b) and air cooled prior to the HR Q&P simulations. The rough rolled plates of each alloy were cut into three slabs (Figure 3.7c) so three different CTs (Figure 3.7d) could be simulated for each alloy during finish rolling. After sectioning, each part went through the same finish rolling procedure (except for CT), as shown in Figure 3.8.

For the hot rolling simulation, each slab was reheated to 1250 °C and held at temperature for three hours, followed by five rolling steps, with total reduction of about 80 %, and aim finishing temperature of 900 °C. The approximate final thickness of the sheets was 3.3 mm. After the last rolling step, each sheet was transported to a water tunnel, where water was sprayed from top nozzles, for 9 or 10 seconds (depending on the aim CT). The temperature of each sheet after the water quench varied from 380 to 450 °C (depending on the time that it was under the water). After water quenching, each sheet was air cooled to the final CT (between 10 and 20 seconds in air depending on the CT). The surface temperature was monitored with an infrared pyrometer gun. Once the CT was reached, each sheet was put in a furnace (set to the specific CT of interest) and the furnace power was turned off. Coiling simulation (furnace cooling) lasted about 48 hours. The three CTs chosen were 425, 375 and 325 °C, based on results of the Gleeble® simulations. A length of approximately 125 mm was cut from one end of each hot-rolled Q&P sheet and sent to CSM for testing and analysis.

3.3.1 Imaging of Laboratory Hot-Rolled Q&P Specimens

Metallography of the HR Q&P specimens involved examination of the longitudinal through thickness cross-section using coupons taken from each sheet as shown in the schematic of Figure 3.10. To characterize the microstructure of the materials, samples were mounted in Bakelite, ground, and polished to 1 μm diamond slurry. All samples were etched with a 2 % nital solution. A JEOL 7000 FESEM operated at 10 KeV accelerating voltage and 10 mm working distance was used for imaging. Light optical microscopy was also used to characterize the steels.
3.3.2 XRD on Laboratory Hot-Rolled Q&P Specimens

Sample preparation to determine RA fractions and Cγ via XRD was done by lightly grinding one face of each coupon (12 x 12 mm) using 240, 320, 400 and 600 grit abrasive papers, followed by
chemical thinning using a solution of 5 parts of deionized water, 5 parts of hydrogen peroxide and 1 part of hydrofluoric acid for about 20 to 30 minutes, which reduced the thickness by 15 to 30%. Retained austenite volume fraction was measured via XRD using nickel-filtered copper radiation in a Phillips X-pert diffractometer operating at 45 kV and 40 mA and equipped with an X’celerator detector and a 1° slit. Four austenite peaks, \{111\}, \{200\}, \{220\}, \{311\}; and four ferrite/martensite peaks, \{110\}, \{200\}, \{211\}, \{220\} were quantified. Samples were scanned through a 2-theta range from 40 to 105° with a step size of 0.02° and 120 second dwell time per step. The amount of RA was calculated according to the SAE method [76]. Carbon content in austenite was calculated using the peak position of austenite \{220\}, according to Equation 3.6:

\[
a_\alpha = 3.555 + 0.044C_\gamma
\]

where \(a_\alpha\) is the austenite lattice parameter (Å) and \(C_\gamma\) is the carbon content in austenite (wt. %) [77]. Figure 3.9 is an example XRD spectrum.

Figure 3.10 shows the locations from which the XRD coupons were cut. XRD 1 (top) and XRD 2 (bottom) were scanned on opposite faces relative to the hot-rolled sheets.

![Example of XRD spectrum obtained to determine retained austenite fractions and carbon content in austenite for Base alloy coiled at 425 °C.](image)

3.3.3 Tensile Testing of Laboratory Hot-Rolled Q&P Specimens

ASTM E8 sub-size [78], shown in Figure 3.11, was the specimen geometry chosen for uniaxial tensile testing of the laboratory hot-rolled steels, due to limited material availability. Figure 3.10 is a schematic illustration showing how the tensile specimens were taken from each HR Q&P sheet. The specimens were machined using the wire-EDM (electrical discharge machining) technique. Specimens
were machined along the rolling direction (RD) and labeled 1 through 5 as shown in the schematic. Five replicate tensile tests were performed for each alloy and condition. A MTS Alliance frame with load cell capacity of 9,000 kg was used for all tests. A 25 mm extensometer was attached to the gauge length (25 mm) and an engineering strain rate of $5 \times 10^{-4}$ s$^{-1}$ was used for all tests.

![Schematic illustration showing how the longitudinal tensile specimens (ASTM E8 sub size), and metallographic (M) and XRD coupons were taken from the hot-rolled sheet. Sheet thickness was approximately 3.3 mm. RD represents the rolling direction. All dimensions are approximate and in millimeters. Drawing not to scale.](image)

![Figure 3.11  ASTM E8 sub-size [78] specimen geometry. All units in millimeters.](image)

3.4 Material Processing – Part III: Cold-Rolled Q&P

Before cold rolling, the remaining parts of the 3.3 mm sheets (as shown in Figure 3.7d) were annealed for two hours at 600 °C, followed by air cooling. Cold rolling was performed at AK Steel and about 70 % cold reduction was achieved. The final thickness of the cold-rolled sheets were approximately 1 mm.
3.4.1 Dilatometry

Dilatometric measurements were obtained from the cold-rolled material to determine critical temperatures (Ac\textsubscript{3} and Ms) that were necessary to build the temperature matrix for the Q&P heat treatments. ASTM Standards A1033 [79] and E228 [80] were used as references for the dilatometry testing method and analysis procedure. A push-rod contact dilatometer with a LVDT (linear variable differential transformer) system was used in a Gleeble®3500 in a low-force setup. To obtain the critical temperatures of interest, ten specimens of the as-received cold-rolled sheet of each alloy were sheared with approximate dimensions of 75 x 6 x 1 mm (length x width x thickness). Specimens were heated to 1050 °C at a rate of 75 °C/s (to mimic the salt baths heating rate), held for 120 seconds and rapidly cooled to room temperature at an approximate rate of 70 °C/s (see Appendix C for a screenshot of the QuickSim2 program).

Figure 3.12 is an example result showing how the critical temperatures were determined from each dilatometric curve. To determine Ac\textsubscript{3}, a linear extension of the dilatometric curve (in the austenite region) was fitted and the critical temperature was determined at the point at which the curve started to deviate from linearity. To determine Ms, a method proposed by Yang and Bhadeshia [81] was used. This method uses an offset from the linear extension and the offset corresponds to the transformation strain due to 1 vol. % martensite. Ms is determined at the point at which the offset line overlaps with the dilatometric curve, as shown in Figure 3.12.

Critical temperatures were also calculated with equations available in the literature, for comparison. The critical temperatures measured via dilatometry and calculated via empirically-derived equations are summarized in Table 3.5.

It is noted that the two calculated values of Ac\textsubscript{3} using relationships from literature (from Hougardy, and Kasatkin and Vinokur) agree well with each other, with the difference between them being 13 °C or less for all alloys. The Kasatkin and Vinokur equation [82] includes Nb and V additions, while Hougardy’s [82] does not. The difference between the Ac\textsubscript{3} as calculated and measured via dilatometry is at least 50 °C for all alloys, however.

The Ms temperatures calculated with the Andrews and Mahieu equations [83] differ by 19 °C for all alloys. Neither equation accounts for any possible effects of microalloying. The differences between the measured and calculated Ms temperatures are relatively large, being over 30 °C for the Nb alloys and over 50 °C for the Base and the V alloys. The measured Ms for the two Nb-containing alloys differs from the Base and V alloys as much as 24 °C. The difference in Ms temperature between the experimental alloys might be influenced by PAGS. Work by Yang and Bhadeshia [84] and Kennett [85] showed that a difference in 50 µm in PAGS can vary Ms as much as 50 °C.
3.4.2 Determination of Austenitizing Temperature

Heat treatments were performed to confirm a suitable temperature to achieve full austenitization. For that, one coupon of each alloy (with approximate dimensions of 50 x 10 x 1 mm) was heat treated in salt baths at 880 °C (which is close to the maximum temperature allowed in the salt pot furnaces) for 120 seconds and immediately water quenched. The microstructures were analyzed to observe the final microstructure and determine whether or not the presence of primary ferrite was observed.

Figure 3.13 shows secondary electron micrographs of each coupon after austenitized and quenched. The observed microstructures seemed to be formed primarily of martensite. One EBSD scan was also collected for each coupon to confirm the absence of intercritical ferrite. Figure 3.14 shows the inverse pole figure and the image quality map for the Base steel as an example of the results obtained via EBSD, which were very similar for all alloys (see Appendix D for the EBSD scans of Low Nb, High Nb and V alloys). The presence of primary ferrite was not observed, which indicated that 880 °C was a high
enough temperature for full austenitization. This temperature for full austenitization is consistent with all empirically derived equations, but was somewhat lower than expected from dilatometry.

As a final confirmation of the suitability of 880 °C as the austenitizing temperature for the experimental alloys, dilatometry experiments were also conducted. Samples were heated to 880 °C at 75 °C/s and held for two minutes (to mimic the experiments done in the salt baths). After the two minute hold, specimens were further heated to 1050 °C at 10 °C/s to see if any additional transformation could be detected. Samples were fast cooled to room temperature once they reached 1050 °C. An example curve for this dilatometry experiment is presented in Figure 3.15 for the V alloy. The V alloy is presented here because it presented the highest measured $A_c_3$ (shown in Table 3.5). As observed in the dilatometry curve, no further transformation (change of slope) was observed, confirming that full austenitizing was achieved for the alloys.

![Figure 3.13](image)

Figure 3.13 Secondary electron micrographs of specimens heat treated in salt baths, held at 880 °C for 60 s followed by water quench. Etched with 2 % nital.
Figure 3.14  (a) Inverse pole figure and (b) image quality map of the Base steel heated in salt bath to 880 °C for 120 s followed by water quench. (Color image – see PDF version)

Figure 3.15  Dilation (µm) versus temperature (°C) for V alloy. Specimen geometry was 75 x 6 x 1 mm (length x width x thickness) with a free span of 15 mm.

3.4.3 Q&P Matrix

A Q&P process matrix for the cold-rolled material was proposed, as shown in the schematic of Figure 3.16. Three QTs were chosen, 275, 300 and 325 °C, based on optimal QT calculations [43] and analysis of the dilatometric curves obtained for each material (Section 3.4.1). Two partitioning
temperatures (PT), 400 and 450 °C, and five partitioning times (tp), 10, 30, 60, 120 and 300 seconds, were chosen based on literature and relevance to the industry. The austenitization temperature was 880 °C, as discussed in Section 3.4.2.

Figure 3.16 Q&P heat treatment matrix for cold-rolled materials. AT = austenitizing temperature, tA = austenitizing time, QT = quenching temperature, tQ = quenching time, PT = partitioning temperature, tp = partitioning time.

3.4.4 Tensile Testing of Cold-Rolled Q&P Specimens

The ASTM E8 sub-size specimen geometry [78], shown in Figure 3.11, was used for uniaxial tensile testing of the CR Q&P study. Longitudinal specimens were machined using wire-EDM. Tensile testing for the cold-rolled material was performed after each heat treatment combination of QT, PT and tp (Figure 3.16). A MTS Alliance frame with load cell capacity of 9,000 Kg was used for all tests. A 25 mm extensometer was attached to the gauge length (25 mm) and a strain rate of 5x10^4 s^-1 was used for all tests. Two tensile tests were performed for each alloy and heat treatment combination, except for the 275 °C condition.

3.4.5 XRD on Cold-Rolled Q&P Specimens

Retained austenite volume fraction was measured via XRD using nickel-filtered copper radiation in a Phillips X-pert diffractometer operating at 45 kV and 40 mA and equipped with an X’celerator detector and a 1° slit. Sample preparation was done by lightly grinding one face of each coupon (10 x 15 mm) on 240, 320, 400 and 600 grit abrasive papers, followed by chemical thinning using a solution of 10 parts of deionized water, 10 parts of hydrogen peroxide and 1 part of hydrofluoric acid for
about 5 minutes, which reduced the thickness by approximately 40%. Samples were scanned through a 2-theta range from 40 to 105° with a step size of 0.03 and 100-second dwell time per step. The amount of RA was calculated according to the SAE method [76].

3.4.6 Imaging and EBSD on Cold-Rolled Q&P Specimens

Metallography of the CR Q&P involved examination of the longitudinal through thickness cross-section. To characterize the microstructure of the materials, samples were mounted in Bakelite, ground, and polished to 1 μm diamond slurry. All samples were etched with a 2% nital solution for 5 to 10 seconds. A JEOL 7000 FESEM operated at 10 KeV accelerating voltage and 10 mm working distance was used for imaging.

EBSD scans were run with the objective to better understand the microstructural features and also to reconstruct PAGS. The methodology for EBSD analysis and PAGS reconstruction are described in Section 3.2.3.

3.5 Summary

Figure 3.17 is a schematic that summarizes how the initial ingot of each designed alloy was divided and processed for each part of this project. The initial ingots, air cast at AK Steel, with approximate dimensions of 150 x 150 x 75 mm (width x length x thickness), were cut in half. Half of each ingot was sent to CSM and hot-rolling was performed in a FENN mill, with final thickness of approximately 14 mm. Sub-sized torsion specimens, adequate for the Gleeble® 3500 torsion setup, were machined from the hot-rolled plates. Thermomechanical processing HR Q&P simulations were conducted in the Gleeble® for all alloys with different CTs. Examination of microstructure, hardness and amounts of RA, obtained via EBSD, was conducted. The details of processing and analysis of Part I: HR Q&P Simulated via Gleeble® Torsion Testing were presented in Section 3.2 (page 22). Results and discussion of this study are presented in Chapter 4 (page 40).

The other half of the initial ingot was hot-rolled at AK Steel, and processing parameters similar to the earlier Gleeble® simulations were used. Three of the preferred CTs based on results of Part I were selected. The details of this part of the project (Part II: HR Q&P Simulated in Laboratory Rolling) were presented in Section 3.3 (page 29). Results of Part II are presented in Appendix E (page 100) and are not part of the main discussion because of the large variability observed in the results, due to processing variations (time in water tunnel, time in air, inhomogeneous cooling profile throughout the sheets). The results obtained did not seem to add to the overall understanding of the role of microalloying elements in HR Q&P steels, but are reported in Appendix E for completeness.

The last part of the project followed the more traditional processing for Q&P steels. After the HR Q&P simulations, the remaining material was annealed followed by cold rolling at AK Steel, and
Q&P processing in salt pots at CSM. The details of Part III: Cold-Rolled Q&P were presented in Section 3.4 (page 32). Results and discussion of Part III are presented in Chapter 5 (page 60).

Figure 3.17 Schematic illustration showing the division of the project and material into three parts. Dimensions shown are approximate and in millimeters. Drawing not to scale.
CHAPTER 4: RESULTS AND DISCUSSION – HOT-ROLLED Q&P SIMULATED VIA TORSION TESTING

In this chapter, the results of the HR Q&P study performed in a Gleeble® 3500 are presented and discussed.

4.1 Microstructural Analysis

Microstructural observations were performed for all steels and coiling conditions (CT = 275, 325, 375 and 425 °C) in the FESEM to observe how the change in CT affected the microstructures with the different microalloying additions. Figure 4.1 and Figure 4.2 show micrographs of the transverse cross-section at the effective radius position for all alloys and all the CTs tested. Recall that the range of CTs extends below and right around the Ms temperature, measured to be around 430-450 °C (depending on the steel). Images were also obtained from the tangential cross-sections, and with similar microstructures to the ones obtained from the transverse cross-section. One example micrograph for each alloy and processing condition is provided in Appendix F (page 109).

A transition from a finer acicular (lath type) morphology at lower CTs to a quasi-polygonal ferrite morphology with more blocky martensite/austenite (MA) islands at high coiling temperatures is observed for most conditions in Figure 4.1 and Figure 4.2. A mix of lath and quasi-polygonal morphologies is observed in the intermediate CTs (325 and 375 °C). The lath morphology remains prevalent in the V alloy, even at higher CTs resulting in coarser acicular microstructures. The range in processing temperatures near and below Ms results in complex mixtures of bainite, partitioned/tempered martensite, fresh martensite, RA, carbides, etc. EBSD analysis was therefore conducted to provide further insight.

4.2 EBSD Analysis

One EBSD scan of each alloy and condition was obtained with the objective to help interpret microstructures, and to estimate the amount of RA present in each condition. It is important to note that EBSD is not the most accurate way to measure bulk RA fractions, due to the limited scan areas and inability to resolve very thin laths. Thus, EBSD, in this case, provides local measurements of austenite content, rather than values averaged throughout the bulk of the material. Further, an underestimation of the total fraction of RA is expected. EBSD was the most appropriate technique for this part of the study, however, due to the torsion specimen used. There are significant gradients in strain and temperature throughout a torsion specimen, which makes only a very small volume of the specimen that undergoes the designed processing conditions available for microstructural analysis.
Figure 4.1 Secondary electron micrographs taken with a FESEM at 5000x in the transverse cross-section view of Base and V alloys at all coiling temperatures tested. Areas imaged are at the effective radius position. Etched with 2% nital.
Figure 4.2  Secondary electron micrographs taken with a FESEM at 5000x in the transverse cross-section view of Low Nb and High Nb alloys at all coiling temperatures tested. Areas imaged are at the effective radius position. Etched with 2% nital.
The EBSD results for each alloy and CT condition are presented in Figure 4.3 through Figure 4.18. These figures include an inverse pole figure map, an image quality map and a phase map. Figure 4.19 and Figure 4.20 are phase maps superimposed with image quality maps to show how the RA is distributed within the microstructure in the tangential cross-section. The images in Figure 4.19 and Figure 4.20 are cropped portions of the full scans, to facilitate the observation of small features.

The observations from the EBSD scans can be correlated to the observations made for the SEM micrographs. The transition from thin laths of both austenite and martensite to a more blocky type of structure at higher CT is noted. In the EBSD scan for the V alloy coiled at 425 °C, the more lath-like characteristics are evident when compared to the other alloys at the same CT.

Another observation from the EBSD scans shown in Figure 4.19 and Figure 4.20 can be made with regard to the increasing amount of fresh martensite with increase in CT based on image quality. The fresh martensite can be identified in the scans by the dark areas, which correspond to areas of low confidence index, due to high density of density of strain and dislocations [86]. The fresh martensite could have formed during cooling associated with the non-isothermal partitioning process, but some may also have formed during sample preparation. The change in morphology, from lath to blocky at higher CT, tends to decrease the stability of the RA [62], which leads to an increase in the amount of fresh martensite. Reduced stability is also associated with the blocky morphology due to longer paths for carbon to diffuse through large austenite islands during partitioning [87]. This in turn creates a carbon gradient within the austenite islands, where more carbon (more stable austenite) is found in the outside and less carbon in the inside. This behavior is noted at the 375 and 425 °C CTs, where some of the RA that is left in large MA islands is surrounded by fresh martensite, as observed in Figure 4.21. The increase in fresh martensite fraction at these temperatures probably indicates that the austenite is less stable following incomplete bainitic transformation. Blocky austenite tends to transform early in forming processes, and may be beneficial for enhancing ductility [88,89]. The hard martensite regions also tend to be more susceptible to boundary decohesion and crack initiation, which is less desirable during sheared edge forming processes such as hole-expansion [88].

The RA fractions obtained via EBSD are summarized in Table 4.1. Figure 4.22 shows how the measured RA varied with the addition of Nb and V when compared to the Base alloy for varying CTs. From Figure 4.22a, it is observed that the addition of Nb did not substantially affect the total amount of RA measured by EBSD for 275, 325 and 425 °C CTs. Niobium additions in the 375 °C condition exhibited the highest fractions of RA. This implies that Nb effectively stabilized RA at this CT. From Figure 4.22b, it is observed that V also did not substantially affect RA fractions for the 275 and 325 °C CTs. The V alloy in the 375 °C condition had the highest fraction of RA, which also implies that the addition of V effectively stabilizes austenite at this CT. At 425 °C, however, a decrease in RA with V is
observed. Since the results are based on EBSD analysis of a single region and the thin interlath austenite films are difficult to measure by EBSD, the uncertainty associated with these observations is not clear.

Table 4.1 – Measured Retained Austenite (in vol. %) via EBSD for Each Alloy and Condition

<table>
<thead>
<tr>
<th>Alloy / CT (°C)</th>
<th>275</th>
<th>325</th>
<th>375</th>
<th>425</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base</td>
<td>3.6</td>
<td>4.2</td>
<td>7.1</td>
<td>8.8</td>
</tr>
<tr>
<td>Low Nb</td>
<td>3.6</td>
<td>4.6</td>
<td>9.7</td>
<td>8.9</td>
</tr>
<tr>
<td>High Nb</td>
<td>4.6</td>
<td>4.1</td>
<td>10.2</td>
<td>8.6</td>
</tr>
<tr>
<td>V</td>
<td>4.1</td>
<td>4.7</td>
<td>9</td>
<td>6.6</td>
</tr>
</tbody>
</table>

Figure 4.3 (a) Inverse pole figure map, (b) image quality map, and (c) phase map, where red represents FCC-Fe (austenite) and green represents BCC-Fe, obtained via EBSD for Base alloy coiled at 275 °C. (Color image – see PDF version)

Figure 4.4 (a) Inverse pole figure map, (b) image quality map, and (c) phase map, where red represents FCC-Fe (austenite) and green represents BCC-Fe, obtained via EBSD for Base alloy coiled at 325 °C. (Color image – see PDF version)
Figure 4.5 (a) Inverse pole figure map, (b) image quality map, and (c) phase map, where red represents FCC-Fe (austenite) and green represents BCC-Fe, obtained via EBSD for Base alloy coiled at 375 °C. (Color image – see PDF version)

Figure 4.6 (a) Inverse pole figure map, (b) image quality map, and (c) phase map, where red represents FCC-Fe (austenite) and green represents BCC-Fe, obtained via EBSD for Base alloy coiled at 425 °C. (Color image – see PDF version)
Figure 4.7  (a) Inverse pole figure map, (b) image quality map, and (c) phase map, where red represents FCC-Fe (austenite) and green represents BCC-Fe, obtained via EBSD for V alloy coiled at 275 °C. *Color image – see PDF version*

Figure 4.8  (a) Inverse pole figure map, (b) image quality map, and (c) phase map, where red represents FCC-Fe (austenite) and green represents BCC-Fe, obtained via EBSD for V alloy coiled at 325 °C. *Color image – see PDF version*
Figure 4.9  (a) Inverse pole figure map, (b) image quality map, and (c) phase map, where red represents FCC-Fe ( austenite) and green represents BCC-Fe, obtained via EBSD for V alloy coiled at 375 °C. (Color image – see PDF version)

Figure 4.10  (a) Inverse pole figure map, (b) image quality map, and (c) phase map, where red represents FCC-Fe ( austerite) and green represents BCC-Fe, obtained via EBSD for V alloy coiled at 425 °C. (Color image – see PDF version)
Figure 4.11  (a) Inverse pole figure map, (b) image quality map, and (c) phase map, where red represents FCC-Fe (austenite) and green represents BCC-Fe, obtained via EBSD for Low Nb alloy coiled at 275 °C. (Color image – see PDF version)

Figure 4.12  (a) Inverse pole figure map, (b) image quality map, and (c) phase map, where red represents FCC-Fe (austenite) and green represents BCC-Fe, obtained via EBSD for Low Nb alloy coiled at 325 °C. (Color image – see PDF version)
Figure 4.13  (a) Inverse pole figure map, (b) image quality map, and (c) phase map, where red represents FCC-Fe (austenite) and green represents BCC-Fe, obtained via EBSD for Low Nb alloy coiled at 375 °C. (Color image – see PDF version)

Figure 4.14  (a) Inverse pole figure map, (b) image quality map, and (c) phase map, where red represents FCC-Fe (austenite) and green represents BCC-Fe, obtained via EBSD for Low Nb alloy coiled at 425 °C. (Color image – see PDF version)
Figure 4.15 (a) Inverse pole figure map, (b) image quality map, and (c) phase map, where red represents FCC-Fe (austenite) and green represents BCC-Fe, obtained via EBSD for High Nb alloy coiled at 275 °C. (Color image – see PDF version)

Figure 4.16 (a) Inverse pole figure map, (b) image quality map, and (c) phase map, where red represents FCC-Fe (austenite) and green represents BCC-Fe, obtained via EBSD for High Nb alloy coiled at 325 °C. (Color image – see PDF version)
Figure 4.17  (a) Inverse pole figure map, (b) image quality map, and (c) phase map, where red represents FCC-Fe (austenite) and green represents BCC-Fe, obtained via EBSD for High Nb alloy coiled at 375 °C. (Color image – see PDF version)

Figure 4.18  (a) Inverse pole figure map, (b) image quality map, and (c) phase map, where red represents FCC-Fe (austenite) and green represents BCC-Fe, obtained via EBSD for High Nb alloy coiled at 425 °C. (Color image – see PDF version)
Figure 4.19  Image quality maps superimposed with phase maps, where red represents FCC-Fe (austenite) and green represents BCC-Fe obtained via EBSD for Base and V alloys at all coiling temperatures tested. *(Color image – see PDF version)*
Figure 4.20 Image quality maps superimposed with phase maps, where red represents FCC-Fe (austenite) and green represents BCC-Fe obtained via EBSD for Low Nb and High Nb alloys at all coiling temperatures tested. (Color image – see PDF version)
Figure 4.21  Cropped EBSD scan that shows in detail the location of austenite ($\gamma$) – red – with respect to the fresh martensite ($\alpha'_\text{fresh}$). (Color image – see PDF version)

Figure 4.22  Retained austenite (vol. %) versus (a) wt. % Nb and (b) wt. % V for all coiling temperatures tested.

4.3 Prior Austenite Grain Analysis

Assessment of PAGS was of interest to characterize the structure of each steel immediately after the torsion steps and assess the influence of microalloying on the austenite condition prior to transformation. Various chemical etchants as well as tempering treatments before etching for PAGS were attempted for the experimental steels without success, perhaps due to the lack of impurities that segregate
to grain boundaries (such as P and S) in the materials. Thus, the EBSD reconstruction technique was attempted to observe the prior structure, instead of conventional chemical etching. The samples utilized for this analysis were direct quenched to room temperature, which promoted martensite formation and allowed prior microstructure observations. The schematic and details of the Gleeble® testing are described in Section 3.2.1 (page 23).

The EBSD scans were taken at the effective radius of the torsion specimens in the tangential view (see Figure 3.6), which is the view that would best allow observation of grain pancaking (or inclination angles) if it had occurred. The occurrence of recrystallization and shear deformation during multi-pass torsion testing is expected to produce a microstructure with variation in inclination angles, as illustrated in Figure 4.23a, based on recent work on microalloyed bar steel [69]. The morphological features of interest in this example are: (B) highly elongated austenite grains that have not recrystallized during processing and should have the same inclination angle as (A) MnS inclusions, that indicates the total amount of shear imparted; (C) elongated austenite grains at smaller angles than A and B, which are indicative that grains have recrystallized and then were subjected to additional stain; and (D) recrystallized austenite grain without strain accumulation, as presented in Figure 4.23a [69]. Figure 4.23b is an etched micrograph showing the features above described [69].

![Figure 4.23 Example of grain pancaking and rotation expected during torsion deformation, when viewed on the tangential plane.](image)

(a) Schematic representation of the expected composite microstructure formed during hot shear deformation. Manganese sulfide inclusions (A) and some austenite grains (B) are expected to accumulate all imposed shear strain without recrystallizing. Other grains (C) predicted to deform in shear after recrystallizing, or (D) recrystallize following the final deformation passes. A reference line, parallel to the torsional axis, is constructed through a series of microhardness indentations used as fiducial markers. (b) Micrograph that shows the features and angles expected from (a) [69].
The PAGS reconstruction for each experimental alloy is presented in Figure 4.24 and Figure 4.25. From Figure 4.24, Base and V alloys seem to have similar PAGS and appear to have fully recrystallized austenite microstructures, indicating minimal strain accumulation. These observations are consistent with the fact that precipitation and recrystallization suppression is expected to occur at lower temperatures than the rolling temperature experienced during multi-pass hot torsion for the Base and V alloys. This is consistent with lower $T_{nr}$ expected for these alloy combinations (Table 3.4) [71,90]. When comparing the Base alloy (Figure 4.24) to the Nb-containing alloys (Figure 4.25), a change in austenite size and morphology is observed, with additional refinement accompanying the higher Nb content alloy. In the Nb-added alloys, grain inclination with respect to the torsional axis (austenite pancaking) is observed. Greater inclination angles are noted for the High Nb alloy, which is consistent with a higher degree of strain accumulation, due to its higher expected $T_{nr}$ when compared to the Base and the Low Nb alloys [69]. The calculated $T_{nr}$ for the Low Nb alloy (Table 3.4) was lower than the FRT used for this processing, but suppression of recrystallization and austenite pancaking was clearly observed for this alloy. This difference indicates that the equation used to calculate $T_{nr}$ for the experimental alloys was not completely accurate for the steels, but provides a qualitative comparison of the $T_{nr}$ for the different alloys. A greater number of small recently recrystallized grains seems to be present in the High Nb alloy, perhaps indicating partial recrystallization. However, some of these features in the reconstructions might not necessarily be grains and may be related to low confidence fitted grains. The low confidence fitted grains result from either poor image quality obtained in the EBSD scan and/or ambiguity in the algorithm used for the reconstruction. Nonetheless, the larger number of small features, especially in the High Nb steel, is believed to be indicative of newly recrystallized grains in this condition [91].

4.4 Vickers Hardness

Vickers hardness was used to compare mechanical properties of the steels due to constraints of the torsion specimen (temperature and strain gradients across the gauge, which limits the area of interest), as discussed in Section 4.2. Tensile ductilities were measured on larger specimens from laboratory rolling (Appendix E – page 100), but these results appeared non-systematic due to inconsistent processing.

The average Vickers hardness values (from 16 indentation measurements) obtained for all alloys and conditions are summarized in Table 4.2. Figure 4.26 shows how the average hardness varied with the addition of Nb and V when compared to the Base alloy. In general, quenching to lower CTs resulted in microstructures with higher hardness, consistent with the increase in the fraction of martensite at lower CTs. Variations in the amount of bainite, RA and fresh martensite may also play a role in the averaged hardness of each alloy and conditions, but it is difficult to quantify these effects since constituent phase fractions were not measured.
Figure 4.24 Prior austenite grain reconstruction via EBSD for Base and V alloys after undergoing a multi-pass torsion schedule and direct quenched to room temperature. Scans were taken at the effective radius position in the tangential cross-section. Reference line indicates the torsional axis and the position of the effective radius. Black lines are prior austenite boundaries while light green lines indicate austenite twin boundaries. Reconstructions were a courtesy of Tuomo Nyyssonen. (*Color image – see PDF version*)
Figure 4.25 Prior austenite grain reconstruction via EBSD for Low Nb and High Nb alloys after undergoing a multi-pass torsion schedule and direct quenched to room temperature. Scans were taken at the effective radius position in the tangential cross-section. Reference line indicates the torsional axis and the position of the effective radius. Black lines are prior austenite boundaries while light green lines indicate austenite twin boundaries. Reconstructions were a courtesy of Tuomo Nyyssonen. (Color image – see PDF version)
Comparing the Nb alloys to the Base alloy (Figure 4.26a), it is noted that for the 325 and 425 °C CTs that hardness is fairly constant amongst the alloys. In the 375 °C condition, a significant drop in hardness was observed from the Base to the Low Nb alloy. The hardness remained low and fairly constant with a further increased addition of Nb. This drop in hardness occurred while the RA fraction increased in the Nb steels at the 375 °C CT (Figure 4.22a). A significant reduction in hardness also occurred at the 275 °CT from the Base to the High Nb. The decrease in hardness also corresponded to an increase in RA observed under these conditions (Figure 4.22a).

Comparing the Base alloy to the V alloy, as shown in Figure 4.26b, the Base alloy hardness gradually decreases with increase in CT, while the V alloy behavior was different. For the V alloy, the hardness of the 275 and 325 °C conditions was rather similar and the hardness of the 375 and 425 °C was also very similar. These behaviors are not fully understood, but the increase in hardness with addition of V for the 425 °C condition is probably associated with the acicular morphology encountered at that CT only in the V alloy. Primary ferrite may have been present in isolated instances, influencing the hardness results for all alloys.

Table 4.2 – Average Vickers Hardness and Standard Deviation for Each Alloy and Condition

<table>
<thead>
<tr>
<th>Alloy / CT</th>
<th>DQ</th>
<th>275 °C</th>
<th>325 °C</th>
<th>375 °C</th>
<th>425 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base</td>
<td>554 ± 14</td>
<td>489 ± 14</td>
<td>451 ± 7</td>
<td>406 ± 17</td>
<td>339 ± 10</td>
</tr>
<tr>
<td>Low Nb</td>
<td>555 ± 20</td>
<td>470 ± 10</td>
<td>464 ± 14</td>
<td>373 ± 15</td>
<td>338 ± 12</td>
</tr>
<tr>
<td>High Nb</td>
<td>558 ± 15</td>
<td>447 ± 19</td>
<td>460 ± 18</td>
<td>368 ± 18</td>
<td>335 ± 14</td>
</tr>
<tr>
<td>V</td>
<td>546 ± 13</td>
<td>464 ± 11</td>
<td>462 ± 8</td>
<td>369 ± 5</td>
<td>369 ± 18</td>
</tr>
</tbody>
</table>

Figure 4.26  Vickers hardness versus (a) wt. % Nb and (b) wt. % V for all coiling temperatures tested. Error bars represent standard deviation.
In this chapter, results and discussion of the Q&P response of cold-rolled microalloyed steels are presented in terms of microstructure (RA fractions, C content in austenite - \( C_\gamma \), PAGS and SEM micrographs) and mechanical properties (primarily YS, UTS, UE, YS/UTS ratio, and UTS·UE). The present chapter focuses on the effects of V and Nb in CR Q&P when compared to a Base alloy (without V or Nb additions).

The proposed experimental matrix (Figure 3.16 — page 37) for this portion of the study consisted of a combination of three QTs (275, 300 and 325 °C), two PTs (400 and 450 °C) and five \( t_p \)'s (10, 30, 90, 120 and 300 s), giving a total of 30 heat treatments for each experimental alloy. In the following sections, each property of interest is plotted against \( t_p \) for each combination of QT and PT. The effects of V and Nb are then analyzed for each combination of processing. Duplicate tests were conducted for all conditions except the lowest QT condition (275 °C) because of sample availability and the somewhat poorer combination of properties obtained when compared to the other processing parameters observed in the first round of heat treatments. All of the results for the 275 °C QT condition are provided in Appendix G (page 112). A table with mechanical properties, RA and \( C_\gamma \) measurements for all alloys and conditions is available in Appendix H (page 116).

5.1 Microstructural Analysis

Prior austenite grain size analysis, SEM imaging and EBSD results are presented in the following sections.

5.1.1 Prior Austenite Grain Size

Prior austenite grain size was determined in order to evaluate the effects of V and Nb on the austenite grain size. One specimen of each alloy was heat treated in a salt bath for 120 s at 880 °C followed by a water quench. The EBSD data (image quality and inverse pole figure map) for these specimens were presented in Figure 3.13 and Appendix D. Figure 5.1 shows the PAGS reconstructions for the Base, V, and Nb-containing alloys. Grains are equiaxed in all alloys and grain refinement is observed with microalloying additions. A small degree of grain refinement is observed for the V alloy, with additional grain refinement evident for the Nb-containing alloys. PAGS quantification was not performed due to the small area available for such analysis and the presence of low confidence fitted features that could affect the quantification.
5.1.2 General Microstructure: FESEM and EBSD

Figure 5.2 and Figure 5.3 are example FESEM micrographs comparing microstructures of the Base, V, and Nb-containing alloys for the same heat treatment conditions (325 °C QT, 400 ° PT and 60 s $t_p$) at two different magnifications. The microstructures of all alloys contain martensite and retained austenite, typical of Q&P microstructures in fully austenitized steels. Some primary ferrite may also be present in the microalloyed steels. The Base alloy generally displays larger PAGS (consistent with the reconstructions presented in Figure 5.1) and larger martensite packets when compared to the V or Nb microalloyed steels, as seen in Figure 5.2. At a higher magnification (Figure 5.3), the Base alloy is primarily composed of lath martensite and mostly interlath RA. The V and Low Nb alloys display a combination of lath and blocky or equiaxed morphologies throughout the microstructure, while the High Nb alloy contains a greater fraction of the blocky/equiaxed-type of structure, as observed in Figure 5.3.
EBSD scans were performed in the same heat treatment condition to further explore the change in morphology observed in the FESEM images shown in Figure 5.2 and Figure 5.3. Figure 5.4 shows the IQ maps and the phase maps of each alloy. The Base alloy has the largest austenite grain size and larger martensite packets when compared to the microalloyed steels. The microalloyed steels presented a more equiaxed/blocky type of morphology and this type of structure is more obvious at the High Nb alloy. The change in morphology of the RA was not so evident when comparing the phase maps for all the alloys. All alloys present a combination of interlath RA and small blocky retained austenite (less than 1 µm). The microalloyed steels seem to contain a higher fraction of blocky RA when compared to the Base. Some ferrite might be present in the microalloyed steels (small white equiaxed grains observed in the IQ maps).

![Secondary electron micrographs of Base, V and Nb-containing alloys heat treated in salt baths. Quenching and partitioning temperatures were 325 and 400 °C, respectively, and 60 s partitioning time. Etched with 2 % nital.](image)

Figure 5.2

5.2 Retained Austenite Fractions and Carbon Content in Austenite

Retained austenite fractions and austenite carbon content were measured for all alloys and conditions via XRD. Fractions of RA as well as $C_\gamma$ were plotted against $t_p$ for all combinations of QT and PT. Figure 5.5 shows these results for the Base and V alloys for QT/PT combinations of 300 °C QT and 400 °C PT (Figure 5.5a), 300 °C QT and 450 °C PT (Figure 5.5b), 325°C QT and 400 °C PT
(Figure 5.5c), and 325°C QT and 450 °C PT (Figure 5.5d). Corresponding results for the Base and Nb-containing alloys are presented in Figure 5.6. It is evident from Figure 5.5 and Figure 5.6 that the RA fraction tends to decrease with \( t_p \) while the C content in austenite tends to increase at short \( t_p \)'s, and then stabilize or decrease at longer times (especially at the higher PT).

The addition of V to the Base alloy (Figure 5.5) generally increased RA fractions at low partitioning times (up to 30 s). Beyond that time, fractions of RA were not strongly influenced by V addition, as evidenced by the general overlapping of the curves for the two alloys. Austenite carbon content did not seem to change systematically with the addition of V, and the values were relatively similar for the two alloys. The addition of Nb to the Base alloy (Figure 5.6) increased RA fractions for most combinations of QT/PT and \( t_p \)'s for both Nb levels. The higher Nb steel contained the greatest austenite fraction at room temperature, in the majority of instances. The effects on C\(\gamma\) were also small, but Nb seemed to increase, or at least maintain, RA stability in most conditions when compared to the Base alloy.

![Secondary electron micrographs of Base, V and Nb-containing alloys heat treated in salt baths. Quenching and partitioning temperatures were 325 and 400 °C, respectively, and 60 s partitioning time. Etched with 2 % nital.](image)

**Figure 5.3** Secondary electron micrographs of Base, V and Nb-containing alloys heat treated in salt baths. Quenching and partitioning temperatures were 325 and 400 °C, respectively, and 60 s partitioning time. Etched with 2 % nital.
The increased RA fractions observed for the Nb alloys could be associated with the refinement in PAGS (observed in Figure 5.1). Smaller PAGS potentially lead to finer martensite laths, which might alter the competition between C partitioning into austenite, and carbide precipitation within the martensite (by reducing the effective distance over which C needs to diffuse to escape the martensite). Additional analysis techniques, such as TEM and Mossbauer Spectroscopy, would be necessary to confirm this hypothesis.

Comparing the results for the two different PTs at a given QT, greater RA fractions and higher austenite stability were observed in all alloys at short tp’s for the higher PT. A significant decrease in RA and Cγ is observed at long tp’s for the 450 °C PT but not the 400 °C PT; this decrease in RA and Cγ is consistent with austenite decomposition that may occur at long tp’s and high PT [92]. The results for the different QTs do not exhibit systematic variations.

The averages of two XRD measurements to characterize RA fraction and carbon concentration are presented for all processing conditions in Figure 5.7, for the Base and V alloys, and in Figure 5.8, for the Base and the two Nb-containing alloys, as a function of QT. The solid line represents the calculated RA fractions from the model using the K-M relationship discussed in Chapter 2 (page 3). No clear RA peak was observed in the experimental results. A larger fraction of austenite was retained at the 325 °C QT condition than predicted by the model, suggesting that the optimum QT may be higher than predicted. This shift in optimum QT (and RA peak) could be due to small inaccuracies in the experimentally measured Ms. For example, from Andrew’s equation [83], a decrease in bulk carbon content of 0.01 wt. % C increases Ms by 5 °C, and an increase in 5 °C in the Ms shifts the predicted curve by 5 °C to the right.

Other mechanisms that operate during partitioning also influence the comparison between measured and predicted results. For example, the peak predicted RA fraction is also greater than any of the experimentally observed values. Full partitioning of C from the supersaturated martensite to the untransformed austenite is assumed in the predictive model. Since full partitioning typically is not achieved (due to the formation of transitional carbides, austenite decomposition, carbon trapping by dislocations, etc.), the amount of C available in solution is very often lower than the total bulk C. Less of the C in solution in austenite after partitioning would lead to lower maximum theoretical RA fractions at room temperature, and shift the predicted austenite fractions to lower levels. Some recent studies have focused on these issues, developing new predictive models based on measured martensite transformation kinetics [40].
Figure 5.4  Image quality (IQ) maps and phase maps, where red represents FCC-Fe (austenite) and green represents BCC-Fe obtained via EBSD for all alloys austenitized at 880 °C, quenched to 325 °C, and partitioned at 400 °C for 60 s. (Color image – see PDF version)
Figure 5.5  Retained austenite and carbon content in austenite as a function of partitioning time for Base and V alloys for (a) 300 °C QT and 400 °C PT, (b) 300 °C QT and 450 °C PT, (c) 325°C QT and 400 °C PT, and (d) 325°C QT and 450 °C PT.
Figure 5.6  Retained austenite and carbon content in austenite as a function of partitioning time for Base and V alloys for (a) 300 °C QT and 400 °C PT, (b) 300 °C QT and 450 °C PT, (c) 325 °C QT and 400 °C PT, and (d) 325 °C QT and 450 °C PT.
Figure 5.7  Average measured retained austenite values as a function of quench temperature for various partitioning times (10, 30, 60, 120 and 300 s) and two partitioning temperatures (400 and 450 °C) for (a) Base and (b) V alloys. The solid line represents predicted amounts of retained austenite using the K-M relationship assuming full partitioning.
Figure 5.8  Average measured retained austenite values as a function of quench temperature for various partitioning times (10, 30, 60, 120 and 300 s) and two partitioning temperatures (400 and 450 °C) for (a) Low Nb and (b) High Nb alloys. The solid line represents predicted amounts of retained austenite using the K-M relationship assuming full partitioning.

5.3 Mechanical Properties

Strength (YS and UTS) and tensile UE were plotted against t_p for all combinations of QT and PT. Figure 5.9 shows these results for the Base and V alloys for the QT/PT combinations of 300 °C QT and 400 °C PT (Figure 5.9a), 300 °C QT and 450 °C PT (Figure 5.9b), 325°C QT and 400 °C PT (Figure 5.9c), and 325°C QT and 450 °C PT (Figure 5.9d). Corresponding results for the Base and Nb-containing alloys are presented in Figure 5.10. A general trend evident from Figure 5.9 and Figure 5.10 is
that YS increases while UTS decreases during partitioning up to about 60s. The UE decreases slightly with partitioning time up to 60 s and then remains relatively constant for longer \( t_p \)’s in most instances, over the range of partitioning times examined.

The addition of V to the Base alloy resulted in an increase in strength and/or elongation in many instances. No significant decrease in YS, TS or UE was observed to result from the addition of V in any processing condition. The smaller addition of Nb to the Base alloy had only a small influence on YS and UTS, but clearly influenced the UE. In most instances, the Nb-containing alloys exhibited higher ductility. The High Nb addition increased the YS in some conditions and decreased it in several other conditions (to varying extents). Similar behaviors are noted in literature. In work reported by Hausmann et al. [25], for TBF steels, at 400 °C overaging temperature and varying overaging times, similar YS and UTS levels were observed when comparing the reference alloy to the Nb-containing alloy and an increase in a few percent UE was noted (1-4 %). Sugimoto et al. [26,28] also studied different levels of Nb in TBF steels. In both studies, Nb additions seemed to affect strength in a non-systematic way when compared to the reference steel, and ductility (only total elongation reported) seemed to be generally increased by a few to several percent (depending on the overaging temperature).

Comparing the results for the two different PTs at a given QT, a few percent increase in elongation is observed for each alloy with increasing PT to 450 °C, along with a small reduction in UTS. The YS is increased at higher PT for short \( t_p \)’s. The decrease in UTS and increase in YS at 450 °C PT at short \( t_p \)’s, when compared to the 400 °C PT condition, reflects a significant change in work hardening behavior at short \( t_p \)’s for both PTs. No systematic influence of QT is noted in Figure 5.9 and Figure 5.10.

The results presented in Figure 5.9 and Figure 5.10 indicated that work hardening behavior was highly influenced by the partitioning process, since the YS and UTS responded very differently with increased time. The YS/UTS ratio provides insight into work hardening behavior. A ratio close to 1 means that the difference between YS and UTS is small and there is less work hardening. Figure 5.11 shows the YS/UTS ratio as a function of \( t_p \) for the QT/PT combinations of 300 °C QT and 400 °C PT (Figure 5.11a), 300 °C QT and 450 °C PT (Figure 5.11b), 325°C QT and 400 °C PT (Figure 5.11c), and 325°C QT and 450 °C PT (Figure 5.11d) for the Base and V alloys. Corresponding results for the Base and Nb-containing alloys are presented in Figure 5.12. A general trend observed from Figure 5.11 and Figure 5.12 is that the YS/UTS ratios increased with increasing \( t_p \) up to 60 s.

The addition of V to the Base alloy resulted, in most cases, in either the same or a small increase in YS/UTS ratio. The smaller addition of Nb to the Base alloy generally resulted in lower YS/UTS ratios at short \( t_p \)’s (up to about 60 s) and higher ratios above 60 s when compared to the Base alloy. This behavior was accentuated in the High Nb alloy at the 400°C PT. At 450 °C PT, the High Nb alloy
exhibited a lower YS/TS ratio (greater work hardening) for almost all of the partitioning conditions, associated with the decrease in YS in this condition.

Comparing the results for the two different PTs at a given QT, a much narrower range of YS/UTS ratio is observed with increased PT. The lowest YS/UTS values at short \( t_p \)’s are not observed for each alloy at 450 °C, but may have resulted at shorter times than used in this study. A slight increase in work hardening (lower YS/UTS ratio) is observed for the 450 °C PT and long \( t_p \)’s (mostly at 300 s), independent of the QT. No systematic influence of QT is noted from Figure 5.11 and Figure 5.12.

![Graphs showing yield strength, ultimate tensile strength, and uniform elongation as a function of partitioning time for Base and V alloys for different conditions](image)

Figure 5.9 Yield strength, ultimate tensile strength, and uniform elongation as a function of partitioning time for Base and V alloys for (a) 300 °C QT and 400 °C PT, (b) 300 °C QT and 450 °C PT, (c) 325°C QT and 400 °C PT, and (d) 325°C QT and 450 °C PT.
Figure 5.10 Yield strength, ultimate tensile strength and uniform elongation as a function of partitioning time for Base and V alloys for (a) 300 °C QT and 400 °C PT, (b) 300 °C QT and 450 °C PT, (c) 325°C QT and 400 °C PT, and (d) 325°C QT and 450 °C PT.

The effects of partitioning on the YS/UTS ratio in the early stages of partitioning could be related to two possible phenomena. One likely contribution is associated with higher degrees of martensite tempering at 450 °C PT when compared to the 400 °C for the same partitioning time. Martensite tempering usually increases the YS through relaxation of internal stresses associated with the martensite transformation, or solute atmosphere pinning of mobile dislocations. Another mechanism that might be operating during early partitioning is associated with the reduced extent of carbon partitioning at short $t_p$’s. At shorter times and lower temperatures, the extent of carbon enrichment in the austenite is likely
reduced, thereby reducing austenite stability. If this behavior (low austenite stability) leads to stress-induced transformation of the austenite prior to yielding of the martensite, a reduced YS and increased UTS can result [93].

Figure 5.11  YS/UTS ratio as a function of partitioning time for Base and V alloys for (a) 300 °C QT and 400 °C PT, (b) 300 °C QT and 450 °C PT, (c) 325°C QT and 400 °C PT, and (d) 325°C QT and 450 °C PT.
From the earlier discussion of yield and tensile strengths and work hardening behavior, the variation in YS and YS/UTS ratio was considered potentially to be related to the degree of partitioning and the associated stability of the RA. To investigate this hypothesis further, the correlation between YS and $C_\gamma$ as a function of $t_p$ was examined. The results are plotted for the Base and V alloys for the QT/PT combinations of 300 °C QT and 400 °C PT (Figure 5.13a), 300 °C QT and 450 °C PT (Figure 5.13b),
325°C QT and 400 °C PT (Figure 5.13c), and 325°C QT and 450 °C PT (Figure 5.13d). Corresponding results for the Base and Nb-containing alloys are presented in Figure 5.14. A general trend evident from Figure 5.13 and Figure 5.14 is that the increase in C is accompanied by an increase in YS for all alloys. Short t_p’s generally resulted in a low YS and correspondingly low Cγ. As t_p increases, both YS and austenite stability generally increase, consistent with the proposed mechanism (wherein reduced austenite carbon enrichment and austenite stability lead to stress induced transformation at low stresses). Further confirmation of this mechanism would require detailed assessment of the martensite transformation kinetics during deformation.

Figure 5.13  Yield strength and carbon content in austenite as a function of partitioning time for Base and V alloys for (a) 300 °C QT and 400 °C PT, (b) 300 °C QT and 450 °C PT, (c) 325°C QT and 400 °C PT, and (d) 325°C QT and 450 °C PT.
Figure 5.14  Yield strength and carbon content in austenite as a function of partitioning time for Base and V alloys for (a) 300 °C QT and 400 °C PT, (b) 300 °C QT and 450 °C PT, (c) 325°C QT and 400 °C PT, and (d) 325°C QT and 450 °C PT.

Consideration of austenite stability helps clarify the difference in work hardening behavior for 400 and 450 °C PT at short $t_p$'s. Other factors, besides $C_\gamma$, might also influence the onset of plastic deformation, like the size and distribution of the RA in the microstructure, the internal stress distribution, the strain aging behavior, the carbon distribution within the RA, dislocation density, and the amounts of other possible softer phases present in the microstructure, such as ferrite, bainite, and tempered martensite. Other authors [94–96] have noted the similar work hardening behavior in Q&P steels with varying PT and similar reasoning was employed in discussing the results.
Engineering stress-strain curves were examined to further assess the properties, and especially the yielding behavior. At the low PT of 400 °C, Figure 5.15a presents example stress-strain curves for the Base and V alloy for 325 °C QT, and 60 s tp. Corresponding results for the Base and Nb alloys are presented in Figure 5.15b for 120 s tp. The results indicate continuous yielding in all alloys and conditions partitioned at 400 °C. Some of the general trends discussed earlier are also indicated: a small increase in YS, UTS, and UE associated with an addition of 0.06 wt. % V to the Base alloy, and an increase in ductility with Nb additions to the Base alloy. The effects of microalloying on stress-strain behavior were different at higher PT. Figure 5.16 shows stress-strain curves (with the yielding regime magnified) for all alloys quenched at 300 °C and partitioned at 450 °C for 120 s (Figure 5.16a) and 300 s (Figure 5.16b). Discontinuous yielding was observed in all of the microalloyed steels, but not in the Base steel, after long tp’s (120 and 300 s) at the highest PT (450 °C). The presence of discontinuous yielding was dependent on partitioning but independent of QT. The combination of a high PT and high tp’s likely results in significant fractions of tempered martensite with a relatively low initial mobile dislocation density, which is usually a requirement for discontinuous yielding [97].

While the origin of discontinuous yielding in the V and Nb Q&P steels is not fully understood at this time, it may be related to effects of finer austenite on the transformed microstructure and internal stress distribution, or effects of microalloy precipitates on the population of mobile dislocations [97]. Another possible mechanism that may have influenced yielding behavior could be related to a shift in the $M_s$ (temperature at which the TRIP behavior switches from stress-assisted to strain-induced transformation of the austenite into martensite)[98]. The change from stress-assisted to strain-induced is accompanied by a change in yielding behavior, from continuous to discontinuous [99]. The microalloying elements might influence this behavior by increasing the yield strength of the austenite (via precipitation hardening), possibly also affecting the $C\gamma$ and the RA morphology, which, consequently increases $M_s$. This mechanism has been proposed previously in microalloyed TRIP steel literature [99,100]. Figure 5.17 is a schematic illustration that represents the change in $M_s$ for cold-rolled TRIP steel microalloyed with titanium when compared to a reference steel [99].

Considering the overall balance of mechanical properties, Figure 5.18 and Figure 5.19 plot the UTS versus UE values obtained for the Base and V-containing steels, and for the Base and Nb-containing steels, respectively, for all processing parameters. There is considerable scatter in the data, but close observation suggests that a higher concentration of points is observed for the V and Nb-containing alloys relative to the base alloy in the upper-right region of the figures, indicating that overall the microalloyed steels exhibit the potential for better combinations of strength and ductility than the Base alloy. While these figures ignore the detailed effects of processing variations, they provide general insight on how the combined properties compare between the Base and the microalloyed steels.
Figure 5.15  (a) Representative engineering stress-strain curves for Base and V alloys processed with 325 °C QT, 400 °C PT and 60 s t_p. (b) Representative engineering stress-strain curves for Base, Low Nb and High Nb Q&P processed steels with 325 °C QT, 400 °C PT and 120 s t_p.

Figure 5.16  (a) Engineering stress-strain curve showing discontinuous yielding observed for all microalloyed steels partitioned at 450 °C. Example for (a) 300 °C QT and 120 s t_p, and (b) 300 °C QT and 300 s t_p for all alloys.
Figure 5.17  Schematic illustrating the effect of precipitation strengthening on the increase of the $M_s^{\sigma}$ temperature [99].

Figure 5.18  Combinations of ultimate tensile strength and uniform elongation obtained for all combinations of processing for Base and V alloys, as measured by quasi-static uniaxial tensile testing.
Figure 5.19 Combination of ultimate tensile strength and uniform elongation obtained for all combinations of processing for Base, Low Nb and High Nb alloys, as measured by quasi-static uniaxial tensile testing.
CHAPTER 6: CONCLUSIONS

This chapter presents the main observations and conclusions obtained from the HR and CR Q&P studies described in previous chapters.

6.1 Hot-Rolled Q&P Simulated via Torsion Testing

- SEM imaging and EBSD phase mapping showed a change in retained austenite morphology from lath-like to blocky following processing with increasing CTs. Morphological changes were accompanied by an increase in “fresh martensite” with increasing CT, indicative of lower stability of the RA at higher CTs.
- Refinement of PAGS was significant with additions of Nb. Comparing Low Nb to High Nb, the further addition of Nb led to additional refinement. Austenite grain elongation was observed for the Nb-added alloys after thermo-mechanical processing. The addition of V was not observed to influence the austenite grain size or morphology.
- The maximum austenite fraction was observed at 375 °C CT, and microalloying (both Nb and V) seemed to be effective in increasing RA fractions for this CT. Microalloying did not appear to influence the austenite fractions for the other conditions.
- A decrease in CT resulted in higher hardness, which is consistent with the increased fractions of harder phases in the microstructure.

6.2 Cold-Rolled Q&P

- Prior austenite grains were equiaxed in all alloys and significant grain refinement was observed with additions of Nb to the Base alloy.
- Generally, austenite fractions were higher for short $t_p$’s and were maintained or reduced with further increased $t_p$. Lower stability of austenite was observed at short times and higher (or maintained) stability with increased $t_p$. When compared to the Base alloy, V seemed to retain higher fractions of austenite at short $t_p$’s, while Nb increased the retained austenite fractions in most conditions. Results did not show systematic or strong influence of V and Nb on austenite stability, and the $C_\gamma$ was similar for the microalloyed and Base steels in most conditions.
- In terms of mechanical properties, the general behavior observed was that YS increased while UTS and UE decreased during partitioning up to 60 s and remained relatively constant at longer $t_p$’s. Vanadium seemed to increase strength and/or elongation in most heat treatments. Additions of Nb seemed to increase UE and had a small influence on UTS. Yield strength decreased in several instances for the High Nb alloy.
• The partitioning step was observed to play an important role in work hardening behavior. Partitioning at short times (up to 60 s) and high temperature caused a significant decrease in work hardening, associated with higher YS/UTS ratios.

• Discontinuous yielding was observed for all microalloyed steels that were partitioned at 450 °C for 120 or 300 s, independent of the QT. This behavior was not observed for the Base alloy in any instance.

• While the data exhibited considerable variation, microalloying additions improved the combination of UTS and UE when compared to the Base alloy.
REFERENCES


S. Hashimoto, S. Ikeda, K. Sugimoto, and S. Miyake, “Effects of Nb and Mo Addition to 0.2%C-1.5%Si-1.5%Mn Steel on Mechanical Properties of Hot Rolled TRIP-Aided Steel Sheets,” *ISIJ International*, vol. 44, no. 9, pp. 1590–1598, 2004.


B. D. Cullity, “Elements of X-ray Diffraction.”


91 T. Nyyssonen, “Private Communication, December 2015.”


APPENDIX A: SOLUBILITY ANALYSIS

The solubility of Nb and V in austenite was assessed for each experimental alloy designed for the present work (compositions available in Table 3.1 – page 21). The general solubility product equation is:

\[ \log [M]^x [X]^y = A - \frac{B}{T} \]  

where [M] is the concentration of metal atoms, [X] is the concentration of non-metal atoms, T is the absolute temperature (measured in K), and A, B, x and y are empirical constants [90]. The solubility parameters used for this study are presented in Table A.1 and were obtained from Turkdogan’s work [101] and Gladman’s book [90].

From Equation A.1 and Table A.1, solubility curves were plotted and are shown in Figure A.1 and Figure A.2 for Nb and V, respectively. The experimental alloy compositions are identified in the plots. The plotted isotherms were selected to represent important temperatures used in the HR Q&P processing of the alloys in a Gleeble® during hot torsion testing. The alloys were soaked at 1250 °C to allow full dissolution of precipitates (which can be visually confirmed on the plots of Figure A.1 and Figure A.2, or numerically, in Table A.1), 1100 °C was the temperature that samples were cooled to before the torsion (roughing) passes, and 900 °C was the finish rolling temperature.

Table A.2 presents the calculated temperature at which precipitation is expected to begin upon cooling (which also represents the temperature at which the precipitates are expected to be fully dissolved upon heating). Note that the Low Nb alloy had a significant amount of N in the initial composition, which elevated the precipitation temperature of NbN close to that of the NbC. Formation of complex second phases, such as Nb(C,N), can also occur upon cooling.

---

Table A.1 – Solubility Product Parameters for Various Precipitates

<table>
<thead>
<tr>
<th>log (k_s)</th>
<th>A</th>
<th>B</th>
<th>x</th>
<th>y</th>
</tr>
</thead>
<tbody>
<tr>
<td>log [Nb][C]</td>
<td>2.81</td>
<td>7020</td>
<td>1</td>
<td>0.87</td>
</tr>
<tr>
<td>log [Nb][N]</td>
<td>3.79</td>
<td>10150</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>log [V][C]</td>
<td>7.06</td>
<td>10800</td>
<td>1</td>
<td>0.75</td>
</tr>
<tr>
<td>log [V][N]</td>
<td>2.86</td>
<td>7700</td>
<td>1</td>
<td>1</td>
</tr>
</tbody>
</table>
Figure A.1  Solubility of NbC (a) and NbN(b) for the two experimental alloys containing Nb.

Figure A.2  Solubility of (a) V₄C₃ and (b) VN for the V-containing experimental alloy.

<table>
<thead>
<tr>
<th></th>
<th>T (°C)</th>
<th>Base</th>
<th>Low Nb</th>
<th>High Nb</th>
<th>V Alloy</th>
</tr>
</thead>
<tbody>
<tr>
<td>NbC</td>
<td>-</td>
<td>1104</td>
<td>1197</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>NbN</td>
<td>-</td>
<td>1101</td>
<td>1123</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>V₄C₃</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>877</td>
</tr>
<tr>
<td>VN</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>966</td>
</tr>
</tbody>
</table>
APPENDIX B: AS HOT-ROLLED MATERIAL CHARACTERIZATION

The as hot-rolled material was characterized in terms of hardness and microstructure. The processing details are presented in Section 3.2 (page 22). The characterization results are presented in the following sections.

B.1 Macrohardness

A minimum of ten macrohardness indents, Rockwell C, were performed in the material that was hot-rolled and air cooled at CSM. The average hardness and standard deviation of each experimental alloy are presented in Table B.1.

Table B.1 – Average HRC Hardness of the as Hot-Rolled Material

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Base</th>
<th>Low Nb</th>
<th>High Nb</th>
<th>V</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average HRC</td>
<td>19 ± 1</td>
<td>26 ± 1</td>
<td>26 ± 2</td>
<td>24 ± 2</td>
</tr>
</tbody>
</table>

B.2 Microstructure

Light optical micrographs were recorded from the longitudinal through thickness cross-section of the as hot-rolled material. Figure B.1 and Figure B.2 show the micrographs at two different magnifications.

The microstructure of the Base and V steels seems to be composed of proeutectoid ferrite, fine pearlite and coarse bainite. The microstructure of the Nb-bearing steels seems to be mostly composed of proeutectoid ferrite and bainite. The bainite contains large interlath MA islands. From Figure B.1 and Figure B.2, it is noted that the addition of Nb refined the overall microstructures and seemed to have suppressed the formation of pearlite.
Figure B.1 Light optical micrographs of as hot-rolled condition. Etched with 2 % nital.
Figure B.2 Light optical micrographs of as hot-rolled condition. Etched with 2% nital.
APPENDIX C: QUICKSIM2 PROGRAMS

Screenshots of the torsion and the dilatometry programs used in a Gleeble® 3500 are presented below.

C.1 QuickSim2 Program for Torsion Testing

Figure C.1  QuickSim2 program for torsion testing. Part 1 of 2.
Figure C.2 QuickSim2 program for torsion testing. Part 2 of 2.
### C.2 QuickSim2 Program for Dilatometry

![QuickSim2 program for dilatometry](image)

Figure C.3 QuickSim2 program for dilatometry.
APPENDIX D: EBSD SCANS TO HELP DETERMINE SUITABLE AUSTENITIZING TEMPERATURE FOR CR Q&P STUDY

Heat treatments were performed to determine a suitable temperature to achieve full austenitization of the cold-rolled experimental alloys used in this project. As described in Section 3.4.2 (Page 34), one coupon of each alloy was heat treated in salt baths at 880 °C for 120 seconds and immediately water quenched. One EBSD scan was collected for each coupon to determine whether full austenitization was achieved, or, in other words, to confirm the absence of intercritical ferrite. In Section 3.4.2 (page 36), the inverse pole figure and the image quality map for the Base steel was shown and the presence of primary ferrite was not observed, which indicated that 880 °C was a high enough temperature for full austenitization. The EBSD scans for Low Nb, High Nb and V alloys are presented in Figure D.1, Figure D.2 and Figure D.3, respectively, and they also do not seem to show the presence of primary ferrite.

Figure D.1  (a) Inverse pole figure and (b) image quality map of the Low Nb steel heated in salt bath to 880 °C for 120 s followed by water quench. (Color image – see PDF version)
Figure D.2  (a) Inverse pole figure and (b) image quality map of the High Nb steel heated in salt bath to 880 °C for 120 s followed by water quench. (*Color image – see PDF version*)

Figure D.3  (a) Inverse pole figure and (b) image quality map of the V alloy heated in salt bath to 880 °C for 120 s followed by water quench. (*Color image – see PDF version*)
APPENDIX E: RESULTS FROM LABORATORY HOT-ROLLED Q&P SIMULATIONS

This appendix presents concise results of the laboratory HR Q&P simulations described in Section 3.3 (page 29). Non-systematic variations occurred during processing of the material, such as variations in FRT, time in water tunnel, time in air, inhomogeneous cooling profile throughout the sheets, that may have affected the overall microstructure and properties of the steels. The variability in processing was largely associated with accurate control of cooling rate and CT, and the non-systematic results presented challenges in interpretation. Results are reported here for completeness, however. The details of material processing, testing and characterization can be found in Section 3.3 (page 29).

E.1 Microstructural Characterization

Microscopy and XRD were performed on the laboratory simulated HR Q&P specimens with the objective of understanding the microstructures and the resulting effect on mechanical properties. Table E.1 provides a summary of the RA volume fractions obtained from XRD for all alloys and CTs. The results do not show clear systematic variations of RA with alloy/processing and exhibit significant differences between specimens XRD 1 and XRD 2 (which were prepared from opposite faces of the sheet). These variations might be explained by heterogeneity through thickness in the microstructure due to temperature gradients during processing, associated with the challenges encountered in controlling cooling rate and CT. With this heterogeneity, the average RA fraction may not represent the bulk microstructure well, and introduces complications in understanding the mechanical behavior that is later presented.

<table>
<thead>
<tr>
<th>Alloy and CT</th>
<th>RA Volume (%)</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>XRD 1</td>
<td>XRD 2</td>
<td></td>
</tr>
<tr>
<td>Base CT = 325°C</td>
<td>2.5</td>
<td>2.8</td>
<td></td>
</tr>
<tr>
<td>Base CT = 375°C</td>
<td>10.9</td>
<td>13.1</td>
<td></td>
</tr>
<tr>
<td>Base CT = 425°C</td>
<td>6.3</td>
<td>14.6</td>
<td></td>
</tr>
<tr>
<td>Low Nb CT = 325°C</td>
<td>1.5</td>
<td>11.2</td>
<td></td>
</tr>
<tr>
<td>Low Nb CT = 375°C</td>
<td>1.6</td>
<td>12.6</td>
<td></td>
</tr>
<tr>
<td>Low Nb CT = 425°C</td>
<td>1.7</td>
<td>11.0</td>
<td></td>
</tr>
<tr>
<td>High Nb CT = 325°C</td>
<td>0.0</td>
<td>1.9</td>
<td></td>
</tr>
<tr>
<td>High Nb CT = 375°C</td>
<td>0.5</td>
<td>2.5</td>
<td></td>
</tr>
<tr>
<td>High Nb CT = 425°C</td>
<td>15.5</td>
<td>19.4</td>
<td></td>
</tr>
<tr>
<td>V Alloy CT = 325°C</td>
<td>4.4</td>
<td>12.4</td>
<td></td>
</tr>
<tr>
<td>V Alloy CT = 375°C</td>
<td>9.2</td>
<td>11.1</td>
<td></td>
</tr>
<tr>
<td>V Alloy CT = 425°C</td>
<td>11.9</td>
<td>15.1</td>
<td></td>
</tr>
</tbody>
</table>
To understand the variability in RA obtained via XRD and the possible cause of such behavior, LOM images of the microstructures from the sheets were examined near the surfaces (top and bottom) as well as in the center of each material (through thickness). Figure E.1 and Figure E.2 show LOM micrographs of each alloy and CT taken at mid-thickness and near the surface, respectively. The images were recorded at low magnification to provide a general perspective of the microstructures in comparison to each other and at different CTs. Figure E.3 shows higher resolution/magnification micrographs of the same material, taken from mid-thickness, so that smaller features could be observed and interpreted.

For the Base steel coiled at 325 °C, the Low Nb and High Nb steels at all CTs, and the V steel coiled at 325 °C, fine primary ferrite is found to have formed along the prior austenite grain boundaries. Elongated (pancaked) prior austenite is apparent for the Nb alloys, while more equiaxed prior austenite is observed for the Base and V alloys, consistent with observations for the HR Q&P simulated in the Gleeble®.

The images presented in Figure E.2 show some differences in microstructure when compared to the mid-thickness micrographs shown in Figure E.1. A greater presence of primary ferrite is generally observed near the surface, possibly formed upon cooling during the transportation of the sheets from the rolling mill to the water tunnel. Overall, the microstructure appeared homogeneous in the center half of each specimen. A microstructural gradient was observed from the surfaces (top and bottom) to the quarter thicknesses of the sheets. Depending on the amount of chemical thinning for each XRD coupon (ranged from 15 to 30 %), each XRD scan could have sampled a slightly different microstructural region (closer to the surface, in a transition microstructure or nearer to the center), which helps explain some of the variability observed in the XRD results (Table E.1).

In general, the microstructures include martensite, RA, and primary ferrite along the grain boundaries in some instances. In Figure E.3, the size and morphology of the martensite, ferrite and MA islands can be identified for each alloy and CT. The presence of primary ferrite is observed in several conditions, but in different amounts. The Base alloy showed coarse granular bainite, especially for 375 and 425 °C coiling conditions.

E.2 Mechanical Properties

The tensile mechanical properties (YS, UTS and UE) obtained from the laboratory HR Q&P sheets are summarized in Figure E.4 and Figure E.5. Figure E.4 shows how the mechanical properties varied from the Base steel (no Nb) to the Nb-added steel with 0.02 (Low Nb) and 0.04 (High Nb) wt. %, for the three different CTs tested (325, 375 and 425 °C). Figure E.5 shows the same variations comparing the Base alloy (no V) to the V-added steel (0.06 wt. %).
Figure E.1  Light optical micrographs of HR Q&P steels. Images taken at mid-thickness. 
CT = coiling temperature. Etched with 2 % nital.
CT = 325 °C

CT = 375 °C

CT = 425 °C

Base

Low Nb

High Nb

V Alloy

Figure E.2 Light optical micrographs of HR Q&P steels. Images taken near the surface. CT = coiling temperature. Etched with 2% nital.
Figure E.3  Secondary electron micrographs of HRQ&P steels. Images taken at mid-thickness. CT = coiling temperature. Etched with 2% nital.
Figure E.4a and Figure E.4b show how strength (YS and UTS, respectively) varied with CT. The general influence of CT on YS and UTS follows the expected behavior; strength increases with a decrease in CT, due to a higher fraction of hard phases that form at lower temperature. Figure E.4c shows how UE varied with CT, which generally follows the expected trend of decreasing elongation with decreasing CT (increased strength). The addition of Nb generally did not systematically affect strength when compared to the Base alloy. Figure E.4d shows the product of UTS and UE. From Figure E.4d, it is noted that Nb additions increased the product of UTS and UE when the sheets were coiled at 425 °C, but did not seem to improve the combination of mechanical properties at the 325 and 375 °C CTs. In the V-containing alloy (Figure E.5), the same general trend was observed, where YS and UTS increased while UE decreased with a decrease in CT. An increase in strength for the V alloy for 375 and 425 °C CT while a slight decrease at 325 °C was observed. The addition of V seemed to slightly decrease the UE. As shown in Figure E.5d, V did not seem to affect the overall combination of strength and elongation. Strengths were not well correlated with hardness values measured on Gleeble specimens processed with similar aim temperatures (Section 3.3). The influence of microalloying and processing remain unclear in light of concerns related to variability.

The tensile properties obtained from the laboratory HR Q&P sheets were compared to data published in literature using similar processing, and the results are presented in Figure E.6. Note that all data presented in Figure E.6 relate to non-isothermal partitioning and thus are compared to those obtained in this study. Uniform elongation is compared (instead of total elongation) in order to minimize potential differences caused by variations in specimen geometry.

The alloys used by Somani et al. [50] were Fe-0.2C-1.5Si-2.0Mn-0.6Cr and 0.2C-0.5Si-1.0Al-2.0Mn-0.5Cr-0.2Mo (wt. %). Four coiling simulations were conducted with these alloys, where coiling was simulated for 30 hours by switching off the furnace that was initially set at the desired CT (270, 290, 310, or 320 °C).

In Thomas’ [48] study, a commercial cold-rolled TRIP sheet steel was used with composition 0.19C-1.59Mn-1.63Si-0.036Al-0.013P-0.011N (wt. %). In this work, a wide range of CTs was studied (from 100 to 500 °C in 25 °C increments), some fully austenitized (FA) and some intercritically annealed (IC). No deformation steps were conducted, only heat treatments to understand the concept of non-isothermal partitioning. The duration of coiling simulations varied depending on the coiling temperature selected, since the range studied was substantial.

The results of the present work, for the two lower CTs (325 and 375 °C) are similar to the results of Thomas’ study using fully austenitized specimens, with high strengths (in the range of 1000-1300 MPa) and low UE (varying from 2-6 %). The present alloys simulated at 425 °C CT showed a combination of properties that also correlates with some of Thomas’s results from specimens that were
intercritically annealed – lower UTS and higher UE, which can be explained by a higher fractions of softer phases in both cases. Compared to both the work by Thomas and the present work, Somani’s work showed higher strengths (above 1400 MPa) and low UE (below 5 %).

Figure E.4 Mechanical properties: (a) yield strength, (b) ultimate tensile strength (UTS), (c) uniform elongation (UE), and (d) UTS UE of the Nb containing steels (0.02 and 0.04 wt. % Nb) compared to the Base steel (0 Nb) for the three coiling temperatures tested (275, 325 and 425 °C). Error bars indicate standard deviation.
Figure E.5 Mechanical properties: (a) yield strength, (b) ultimate tensile strength (UTS), (c) uniform elongation (UE), and (d) UTS UE of the V containing steel (0.06 wt. % V) compared to the Base steel (0 Nb) for the three coiling temperatures tested (275, 325 and 425 °). Error bars indicate standard deviation.
Figure E.6  Uniform elongation and ultimate tensile strength data from different studies on Q&P simulation with non-isothermal partitioning. CS = coiling simulation, IC = intercritical annealing, FA = full austenitizing. [48,50]
APPENDIX F: ADDITIONAL FESEM MICROGRAPHS OF HR Q&P SIMULATED VIA GLEEBLE®

Figure F.1 and Figure F.2 show example secondary electron images from the tangential cross-section, for all alloys and conditions tested in the HR Q&P simulated via torsion testing in a Gleeble® 3500 (Chapter 4 – page 40). Note lower magnification than in Figure 4.1 and Figure 4.2 (pages 41 and 42).
Figure F.1 Secondary electron micrographs taken with a FESEM at 2500x in the transverse cross-section view of Base and V alloys at all coiling temperatures tested. Areas imaged are at the effective radius position. Etched with 2 % nital.
Figure F.2  Secondary electron micrographs taken with a FESEM at 5000x in the transverse cross-section view of Low Nb and High Nb alloys at all coiling temperatures tested. Areas imaged are at the effective radius position. Etched with 2% nital.
APPENDIX G: ADDITIONAL PLOTS FOR THE EFFECTS OF VANADIUM AND NIOBIUM ON CR Q&P STUDY

This appendix presents the results for the 275 °C QT condition in the same order as presented in Chapter 5 (page 60) for Base, V and Nb-containing alloys. Only one specimen was tested for each condition at this QT.

Figure G.1  Retained austenite and carbon content in austenite as a function of partitioning time for Base and V alloys for (a) 275 °C QT and 400 °C PT, and (b) 275 °C QT and 450 °C PT.

Figure G.2  Retained austenite and carbon content in austenite as a function of partitioning time for Base, Low Nb and High Nb alloys for (a) 275 °C QT and 400 °C PT, and (b) 275 °C QT and 450 °C PT.
Figure G.3  Yield strength, ultimate tensile strength and uniform elongation as a function of partitioning time for Base and V alloys for (a) 275 °C QT and 400 °C PT, and (b) 275 °C QT and 450 °C PT.

Figure G.4  Yield strength, ultimate tensile strength and uniform elongation as a function of partitioning time for Base, Low Nb and High Nb alloys for (a) 275°C QT and 400 °C PT, and (b) 275 °C QT and 450 °C PT.
Figure G.5  YS/UTS ratio as a function of partitioning time for Base and V alloys for (a) 275 °C QT and 400 °C PT, and (b) 275 °C QT and 450 °C PT.

Figure G.6  YS/UTS ratio as a function of partitioning time for Base, Low Nb and High Nb alloys for (a) 275 °C QT and 400 °C PT, and (b) 275 °C QT and 450 °C PT.
Figure G.7  Yield strength and carbon content in austenite as a function of partitioning time for Base and V alloys for (a) 275 °C QT and 400 °C PT, and (b) 275 °C QT and 450 °C PT.

Figure G.8  Yield strength and carbon content in austenite as a function of partitioning time for Base, Low Nb and High Nb alloys for (a) 275 °C QT and 400 °C PT, and (b) 275 °C QT and 450 °C PT.
APPENDIX H:  TABULATED MECHANICAL PROPERTIES AND AUSTENITE MEASUREMENTS FOR THE COLD-ROLLED Q&P STUDY

Table H.1 – Average Mechanical Properties and Austenite Measurements for QT = 325 °C

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PT = partitioning temperature, t_p = partitioning time, YS = 0.2 % offset yield strength, UTS = ultimate tensile strength, UE = uniform elongation, RA = retained austenite fraction, Cγ = carbon content in austenite