TEMPERATURE AND STRAIN RATE DEPENDENCE OF THE MARTENSITIC
TRANSFORMATION AND MECHANICAL PROPERTIES IN
ADVANCED HIGH STRENGTH STEELS

by

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ABSTRACT

Third-generation advanced high strength steels (3GAHSS) use the deformation-induced phase transformation of austenite to martensite to enhance their properties during forming and in service. However, deformation conditions may vary during these processes, which is problematic because the martensitic phase transformation is intrinsically responsive to strain rate and temperature. This is known to influence the balance of strain accommodation and strengthening via transformation-induced plasticity and dislocation slip. This thesis explores the processing-property relationships that govern the performance of 3GAHSS created with the Quenching and Partitioning (Q&P) process, with the goal of better understanding the influence of deformation processing factors on the austenite stability and mechanical performance.

Two grades of Q&P steel, having different fractions of ferrite and ultimate tensile strengths of 980 and 1180 MPa, were mechanically tested over a range of temperatures from 22 to 250 °C and strain rates from $10^{-4}$ to $10^{3} \text{s}^{-1}$, while the martensitic transformation was tracked by a combination of X-ray diffraction (XRD) and electron microscopy. In some instances, XRD was performed ex-situ with a lab diffractometer, while in higher resolution experiments, XRD was performed in-situ using high-energy synchrotron light. After fracture, spatially-resolved electron backscatter diffraction kernel average misorientation (EBSD-KAM) analysis was performed to complement the bulk XRD data.

Several studies used ex-situ XRD to elucidate the effect of the martensitic transformation on the mechanical properties, as a function of tensile specimen orientation and strain rate, respectively. The orientation of tensile specimens relative to the rolling direction of the sheet appeared to strongly influence the extent of the martensitic transformation, which may have been caused by crystallographic texture that developed during cold rolling and Q&P heat treating. Meanwhile, increasing the strain rate appeared to suppress the martensitic transformation, but it was unclear whether deformation-induced heating or the strain rate increase was responsible. An additional experiment was performed using a thermal-mechanical simulator, to decouple the influences of deformation-induced heating and strain rate on the martensitic phase transformation. By the combination of ex-situ XRD and EBSD-KAM analysis, it was observed that deformation-induced heating suppressed the martensitic transformation, while increasing the strain rate promoted the transformation.

To resolve the martensitic transformation with greater spatial and temporal resolution, quasi-static elevated-temperature tensile tests and dynamic-rate room-temperature tensile tests were performed, while XRD data were collected in-situ using synchrotron light. Because the XRD was performed in-situ, the bulk
mechanical response, as well as the amount of deformation-induced martensite, the stresses/strains on individual phases, and the peak broadening due to the elastic strain fields of dislocations, were measured concurrently. A strong temperature dependence of the martensitic transformation was observed, and the highest quality fit against existing empirical models for the transformation was reached when the strain partitioning between the phases was incorporated. The temperature dependence may have been linked to both the driving force for the transformation and the number of available martensite nucleation sites. Meanwhile, in the high-strain rate experiment, a clear dependence of the martensitic transformation on the steel grade and strain rate was less obvious, because the fidelity of the measurements was limited by a relatively low signal-to-noise ratio.
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CHAPTER 1
INTRODUCTION

1.1 Background

Demand for lighter and more efficient vehicles has resulted in a need for stronger, yet ductile, sheet steels [1, 2]. Third generation advanced-high strength steels (AHSS) are candidate alloys for automotive structural components, due to their enhanced combinations of strength and ductility. The intent is to use AHSS grades in non-exposed high-strength structural components, such as in engine areas, pillar reinforcements, and longitudinal rails [3, 4]. By replacing conventional automotive grades with AHSS, structural components can be made thinner and lighter, with no compromise to performance in energy absorption or penetration-resistant crash zones.

AHSS use the deformation-induced martensitic transformation (DIMT) of metastable austenite to martensite to enhance toughness. Thus, it is critical to understand and control the transformation to predict and maximize the mechanical performance in service. Although property combinations of AHSS are frequently reported using quasi-static room temperature uniaxial tension tests, it is increasingly clear that DIMT is responsive to extrinsic deformation processing factors, such as strain state, strain rate, and temperature [5].

High-rate deformation behavior is relevant to both crash performance and sheet formability; for instance, strain rates in automotive collisions range between \(10^{-2}\) and \(10^{4}\) s\(^{-1}\) [6]. Furthermore, mechanical behavior at high strain rates is complicated by deformation-induced heating. Prior work at the Colorado School of Mines by Poling and Clarke began to address these effects [7–9]; however, it appears that additional investigations could augment our understanding of austenite response to different strain rates and temperatures, in order to more accurately predict the flow behavior during forming operations and the crash performance of finished parts.

1.2 Hypothesis and Research Questions

The overarching hypothesis for this work is that: Extrinsic deformation processing factors, such as sheet orientation, strain rate, and temperature, can be used to control the martensitic phase transformation in Q&P steels during stamping operations. Understanding these mechanisms will enable optimization of deformation processes to tailor the forming response and mechanical properties of stamped parts. To systematically evaluate this hypothesis, three research questions, and sub-hypotheses for each research question, were developed:
1. How are the deformation-induced martensitic transformation and the mechanical properties responsive to (a) sheet orientation and (b) strain rate?

(a) Preferred crystallographic texture, developed from cold rolling and subsequent Q&P processing, will influence the kinetics of DIMT during uniaxial tests of specimens oriented in different directions relative to the rolling direction. (b) Increasing strain rate will have the effect of reducing the kinetics of DIMT, because deformation-induced heating will reduce the driving force for transformation.

2. (a) Is the strain rate sensitivity of the martensitic transformation due to deformation-induced heating alone, or a combination of independent strain rate and temperature effects? (b) If there is a strain rate effect that is independent of temperature, then can this effect be measured at strain rates above the adiabatic threshold?

(a) Because the strength of ferrite/martensite is known to increase with increasing strain rate, strain will be preferentially partitioned to austenite as the strain rate is increased. This strain partitioning will lead to more rapid DIMT as a function of bulk applied strain. Thus, there will be an independent effect of strain rate on DIMT. (b) For strain rate increases above the adiabatic threshold, for instance while increasing the strain rate from $10^{-1}$ to $10^{3}$ s$^{-1}$, DIMT will be enhanced, while the deformation-induced temperature rise will remain constant.

3. (a) Is the martensitic transformation sensitive to temperature over the range of 25 to 250 °C? (b) If so, is there an argument for using warm forming to tailor the martensitic phase transformation for improved performance during sheet forming and in service?

(a) Increasing forming temperature from 25 to 250 °C will decrease the thermodynamic driving force for DIMT, thereby suppressing DIMT in uniaxial tension. (b) The temperature dependence of DIMT will influence the forming response. Increasing the temperature will reduce the strain hardening capacity of the steels, because austenite will be increasingly stable and therefore less available to facilitate TRIP.

1.3 Thesis Overview

This document is organized to address the research questions proposed above. First, a review of the literature is presented in Chapter 2. Next, Chapter 3 describes the sample preparation techniques that were used. Chapter 4 describes the way each experiment aimed to answer the research questions. Each of the five experiments conducted in this project are organized into separate Chapters, 5-9. Finally, the research questions are revisited, along with some conclusions and suggestions for future work in Chapter 10.
In order to guide the design of experiments associated with this thesis work, this chapter aims to clarify the state of the art in the design and processing of advanced high strength steels (AHSS), with the intention of enhancing performance during sheet forming and in service. The review begins with a discussion of the topical relevance of AHSS. Next, the complex relationship between the presence of metastable retained austenite and the mechanical properties of AHSS is discussed, considering the effects of temperature, strain rate, and strain state. Finally, modern methods to characterize the volume fraction of austenite ($V_\gamma$) and the dislocation density ($\rho$) in AHSS are introduced.

2.1 Strain Accommodation and Strengthening Mechanisms in AHSS

AHSS are sophisticated grades that employ multiphase microstructures and various strengthening mechanisms to achieve a range of strength and ductility combinations. Multiple phases contribute to increased strength and ductility in ways not attainable with conventional high-strength steels, such as bake hardenable (BH) and high-strength low-alloy (HSLA) steels. First and second-generation AHSS include the dual-phase (DP), complex-phase (CP), ferritic-bainitic (FB), martensitic (MS), transformation-induced plasticity (TRIP), hot-formed (HF), and twinning-induced plasticity (TWIP) grades [10].

In AHSS, enhanced mechanical properties are achieved through the presence of a hard phase in a more ductile matrix. Tanaka et al. suggested that the size, shape, and dispersion of hard and non-deforming particles increases the hardening behavior of metal by forming internal stress fields, due to the strain incompatibility between the hard and soft phases [11]. Although all AHSS grades employ multiphase microstructures to promote strength and ductility, some AHSS grades employ an additional strengthening mechanism known as the transformation-induced plasticity (TRIP) effect. The TRIP-effect is activated by the deformation-induced martensitic transformation (DIMT) of retained austenite to hard martensite. Therefore, TRIP steels leverage the formation of the hard martensite phase during deformation to increase the work hardening rate with increasing strain [10]. The TRIP-effect is rationalized by the concurrent selection of martensite variants (via the Magee effect [12]) and the generation of new dislocations in the adjacent untransformed austenite (via the Greenwood-Johnson effect [13]).

The activation of the TRIP-effect results in strain hardening properties that are generally improved over austenite-free AHSS grades, such as DP steels. For instance, Keeler et al. compared the tensile performance of a DP and TRIP steel having similar yield strengths, but the TRIP steel exhibited a
substantially enhanced work hardening rate (Figure 2.1) [10]. Because the stability of austenite in AHSS affects characteristics such as strain hardening, ultimate tensile strength, and ductility, it is prudent to understand how austenite stability is impacted by factors such as strain rate, strain path, ambient temperature, deformation-induced heating, alloying, microstructure, and thermal processing. One of the goals in developing third-generation AHSS is to control austenite stability and volume fraction, in order to optimize the mechanical performance [14].

Figure 2.1 TRIP 350/600 with a greater work hardening rate and total elongation than DP 350/600 and HSLA 350/450. The data used to generate this figure was replotted from Keeler et al. [10].

To illustrate the effect of austenite stability on strength-ductility combinations, Matlock et al. created hypothetical steels with varying austenite stabilities, based on the austenite stability function by Olson and Cohen [14, 15]. The four austenite stabilities used in modeling, with A being the most stable and D being the least stable, are presented in Figure 2.2(a). The modeling approach considered ferrite/martensite plus metastable austenite composites with initial austenite content varied between 0 vol pct and 85 vol pct. The microstructures lacking austenite resulted in ultimate tensile strengths below 400 MPa and uniform elongations of 35 pct. Microstructures with high amounts of austenite exhibited improved ultimate tensile strengths, while ductility depended on the austenite stability. The predictions, superimposed over the ultimate tensile strength and uniform elongation performance of traditional steel grades in Figure 2.2(b), suggest that the most effective strength and ductility combinations are achieved with high volume fractions of relatively stable austenite [16]. These predictions suggest that careful control of austenite stability and amount can produce sheet steels with mechanical properties comparable to those with costlier alloying approaches, such as austenitic stainless steels.
2.2 Thermodynamics of the Deformation-Induced Martensitic Transformation

In the previous section, the effect of DIMT on the mechanical properties of AHSS was discussed, considering the TRIP-effect. However, classic papers pertaining to the martensitic transformation were not discussed in detail. In this section, several fundamental papers are recalled.

The contribution of Bain to the understanding of the mechanism and kinetics of martensitic transformations is immense, and well documented by Paxton et al. [17]. In 1925, Bain reported that quench severity from the austenite phase field exhibited critical influence on the amount of retained austenite, with the amount of retained austenite decreasing with increasing quench severity [18]. This corroborated Bain’s theory that a lack of C “migration” was critical for martensite formation [19, 20]. Bain proposed that martensite transformation occurs with both a homogeneous strain and corresponding lattice invariant deformation (twinning or slip) [21, 22].

In 1930, Davenport and Bain “proposed the addition of a time factor to the Fe-C diagram”, with the curves presently known as Time-Temperature-Transformation (TTT) diagrams [23]. In 1939, Bain showed the effects of certain alloying additions on the austenite phase field (Figure 2.3) [24]. Alloying additions which expanded the austenite phase field, thereby lowering the equilibrium transformation temperature of austenite and ferrite, were labeled austenite stabilizers (e.g., Mn, Ni, and C). This is shown by the austenite phase field boundaries in Figure 2.3, where the dotted line represents a mixture of Fe-C, while
the solid lines represents Fe-C with increasing amounts of Mn. By reducing the temperature at which austenite and ferrite exist at thermodynamic equilibrium, the driving force for the martensitic transformation at ambient temperatures is reduced.

![Figure 2.3](image)

Figure 2.3 Mn additions expand the austenite phase field to lower temperatures in a carbon steel. This plot was reused from Bain [24].

A schematic of the Gibbs free energy ($G$) curves for austenite and martensite is shown in Figure 2.4(a) after Krupp et al. [25]. $G$ for both phases decreases with increasing temperature, with the slope of the $G_\gamma$ being steeper than the $G_\alpha$ curve in this regime, leading to stabilization of austenite at temperatures greater than $T_0$, and stabilization of martensite or ferrite at temperatures less than $T_0$. Upon cooling to below $T_0$, there is a driving force for the martensitic phase transformation ($\Delta G$). At the martensite start temperature ($M_s$), a small fraction of martensite is formed. Meanwhile, at the martensite finish temperature ($M_f$), all austenite is transformed to martensite. Shear deformation of the austenite increases $G_\gamma$, which increases the driving force for transformation, because the total driving force includes both a thermal component ($\Delta G_{thermal}$) and a mechanical component ($\Delta G_{mech}$). The deformation-induced martensite temperature ($M_d$) describes a temperature at which martensite forms under an applied stress ($\sigma$) or strain ($\varepsilon$). Increasing the size of the austenite phase field, for instance, by adding an austenite stabilizer to the alloy, would decrease the $G_\gamma$ curve, which would reduce the $M_f$, $M_s$, $M_d$, and $T_0$. For instance, in low carbon steels, $M_s \approx 500 ^\circ C$, but increasing the concentration of C decreases the $M_f$, $M_s$, and $T_0$ as shown by Porter and Easterling in Figure 2.4(b) [26].
2.3 The Quenching and Partitioning Heat Treatment Process

Quenching and partitioning (Q&P) steels are low-alloy steels which are processed using the Q&P heat treatment process. The Q&P process has the effect of creating highly refined martensite-based microstructures containing austenite and sometimes ferrite or bainite. Q&P microstructures are formed by partial formation of martensite during a quench, followed by enrichment of the remaining austenite with carbon during a partitioning step [27]. The Q&P process is shown schematically in Figure 2.5. After austenitizing, the steel is quenched to a quench temperature ($QT$) below the $M_s$ to form dispersed martensite. The fraction of martensite that is formed during the initial quench is determined by the $QT$ and $M_s$ [7]. A modified version of the Andrews equation has been used to predict the $M_s$ for Q&P steels [28, 29]. The resulting volume fraction of martensite in plain carbon steels is related to the $QT$ and $M_s$ by the Koistinen-Marburger relationship [30]. Modified equations for selecting the $QT$ for Q&P steels containing C, Mn, Si, Cr, and B were proposed by Seo et al. [31]. Following the quench, partitioning at elevated temperatures ($PT$) causes carbon to diffuse from the martensite to the remaining austenite, thereby stabilizing the austenite at room temperature. Partitioning thermal profiles are designed such that the diffusion of carbon is promoted, while the diffusion of substitutional elements such as Si and Mn is usually limited, leading to typical partition temperatures, $PT < 450 \, ^\circ\text{C}$, and times, $Pt < 1000 \, \text{seconds}$ [7]. Partitioning temperatures are selected on the basis of promoting carbon diffusion, while suppressing the decomposition of the retained austenite to bainite and the diffusion of larger alloying elements.
Besides modification of $QT$, $PT$, and $Pt$, additional levers for controlling the microstructure of Q&P steels exist. First, carbide formation must be suppressed to promote carbon diffusion into austenite to promote stability [27]. Suppression of carbide formation is often performed by alloying with Si and Al. Some consider Si to prevent the precipitation of transition carbides by modifying precipitation kinetics [32–35], but its exact contribution to microstructure development is under debate. Kim et al. observed that carbide precipitation occurred in both low and high-Si grades, but the addition of Si stabilized the austenite phase during the partitioning stage by favoring the partitioning of C from martensite to austenite grains [36]. Alloying with Al has been shown to suppress carbide formation at the sacrifice of reducing the austenite fraction and the tensile elongation [37]. In addition to suppressing carbide formation, compositions must be tailored to promote the stability and amount of austenite. Beyond C partitioning, Mn is often employed for this task, leading to traditional Q&P grades containing several wt pct Mn. Increasing the Mn content from 2.5 to 3.0 wt pct was observed to increase the uniform elongation at the expense of reducing the ultimate tensile strength, presumably due to an increased volume fraction of austenite and a reduced volume fraction of martensite [38].

2.4 Effects of Deformation Processing Factors on the Martensitic Phase Transformation

The design criteria for AHSS have historically been based around simple material properties, measured using quasi-static tensile tests at room temperature. For instance, material property combinations of AHSS are frequently discussed using an Ashby chart of strength and ductility, endearingly called the Banana Chart. A recent review by Hu and Thomas [5] observed that for first-generation AHSS (containing combinations of ferrite and martensite with no appreciable amount of austenite), deformation processing
factors like strain rate and temperature did not strongly influence the property combinations. Rather, the property combinations were controlled by the microstructure, e.g., the ratio of martensite to ferrite. As austenite-containing AHSS were developed to enhance properties via the TRIP-effect, material properties became increasingly responsive to deformation processing factors like strain rate, temperature, and strain state [5]. This section will explore the basis for these behaviors.

2.4.1 Temperature

Two variations of DIMT have been observed in steels, which are activated at various temperatures relative to $M^\sigma$ (Figure 2.6(a)), a material property related to the stability of the austenite existing in the steel. At temperatures below $M^\sigma$, DIMT is thought to be stress-assisted, where an applied stress enhances the kinetics of martensite formation on preexisting nucleation sites [39]. During stress-assisted transformation, yielding of the austenite progresses via the martensitic transformation, and can occur in the absence of dislocation slip. At temperatures above $M^\sigma$, DIMT is thought to be strain-induced, where nucleation sites formed during plastic deformation trigger nucleation. Richman and Bolling showed that the precise value of $M^\sigma$ can be determined by analyzing the nature of plastic yielding as a function of temperature, because stress-assisted DIMT is associated with yield drops and/or serrated yielding at the onset of plasticity [40].

![Figure 2.6](image-url)

Figure 2.6 (a) Schematic representation of martensite nucleation as a function of temperature, with $M^\sigma$ dividing the stress-assisted and strain-induced transformation regimes. (b) $M^\sigma$ temperature measurement for a Q&P candidate alloy. An $M^\sigma$ temperature of 10 °C was obtained. (a) and (b) were reused from Olson and Morris [41] and De Moor et al. [42] with permission (see Appendix F).

Because continuous and non-serrated yielding is desired for formability and performance, Q&P steels are designed so that the $M^\sigma$ is lower than the minimum temperature reached during service. For instance, De Moor et al. performed tensile tests at various temperatures, and showed that $M^\sigma$ was 10 °C for a
Since Q&P steels are designed to exhibit an $M_s^*$ that is nominally lower than the ambient temperature during service, strain-based kinetics models are most frequently used to capture the progression of DIMT during deformation processes. One prevailing model, proposed by Olson and Cohen (OC) [15], assumed that shear bands acted as the primary sites for martensite nucleation:

$$f^\gamma = f^\gamma=0[1 - \exp(-\beta[1 - \exp(-\alpha\varepsilon)]^n)]$$

(2.1)

Where $\alpha$ represents the rate of shear band formation, $\beta$ represents the likelihood of a potential nucleation site forming a martensite embryo, and $n$ is a material-dependent constant. Olson and Cohen reported that both $\alpha$ and $\beta$ should decrease as a function of increasing temperature, due to increases in the stacking fault energy and decreases in $\Delta G_{\text{thermal}}$, respectively. Figure 2.7 demonstrates the effect of changing each parameter on the amount of DIMT predicted by the OC model.

Less granular strain-based models have been proposed, which do not attempt to differentiate between the number of nucleation sites and the likelihood of forming an embryo. For instance, the empirical Burke-Matsumura (BM) model attempts to capture the DIMT kinetics with only one austenite stability parameter, $k$ [43, 44]:

$$f^\gamma = \frac{f^\gamma=0}{1 + p/[k\varepsilon f^\gamma=0]}$$

(2.2)

Where $p$ is a material-dependent constant that relates to the auto-catalytic propagation of strain-induced martensite [45]. An extensive review of the strain-based models was provided by Samek et al. [45].
acceptable agreement with each model was reported for various alloys. In a variety of TRIP-assisted steels, for the BM and OC models, \( k \), \( \alpha \), and \( \beta \) decreased with increasing temperature. Over the course of the investigation, Samek et al. showed that “the differences in the retained-austenite stability are due more to the difference in intrinsic stacking fault energy, rather than to the difference in chemical driving force” [45]. This suggested that the mechanistic explanation for strain-induced martensitic transformation, proposed by Olson and Cohen, was plausible. More importantly, Samek’s observation implied that the quantity of shear band interactions exercised a potent influence on the kinetics of DIMT as a function of temperature.

The temperature dependence of DIMT has been studied in several AHSS [7, 8, 46, 47]. For instance, for a Q&P steel with a nominal ultimate tensile strength of 980 MPa tested over a temperature range of -100 to 350 °C, Coryell et al. reported a minimization of DIMT and TRIP at approximately 150 °C, which corresponded to a decrease in the tensile mechanical performance [46]. The root cause of the trough-like behavior, where the austenite stability reached a maximum and mechanical properties reached a minimum, was unclear from the data presented. As a follow-on investigation from Coryell’s work, Poling et al. investigated the temperature dependence of DIMT in Q&P and Medium-Mn steels at the Colorado School of Mines [7, 8]. Poling interrupted tests at increments of several percent plastic strain for a range of temperatures from -10 to approximately 100 °C. The interrupted tests revealed that the DIMT kinetics of each steel were captured reasonably accurately by the OC model; in both steels, \( \alpha \) and \( \beta \) decreased with increasing temperature. One exception was that the OC model did not accurately capture the DIMT kinetics of the Q&P steel at one percent true plastic strain. For the Q&P steel, the retarded DIMT kinetics at elevated temperatures caused a reduction in the uniform elongation of the steel, which corroborated Coryell’s results. Hu and Thomas speculated that the minimization of DIMT at 150 °C, reported by Coryell and Poling, may have been due to the material having an \( M_d = 150 °C \). [5]. Hu and Thomas reported an enhancement in ductility for two Q&P steels (Q&P1000 and Q&P1200) tested at temperatures between 150 and 250 °C, which was attributed to an activation of dynamic strain aging (DSA) and a reactivation of DIMT [5, 48]. More detailed studies are needed to understand the role that each constituent phase exhibits in accommodating strain at high temperatures.

In a recent work, Tsuchida and Harjo proposed a potential pathway to tailor the properties of TRIP-assisted steels by changing the deformation temperature as a function of strain [49]. From true strains of 0 to 0.15, the deformation temperature was held constant at 100 °C, presumably to retard the DIMT kinetics (Figure 2.8(a)). Then, the specimens were unloaded, cooled, then reloaded at temperatures from -150 to 23 °C. The two-temperature interrupted test had the effect of greatly enhancing the total toughness, via a combination of large increases in the flow stress and a five percent increase in the uniform elongation (Figure 2.8(b)). It was clear based on the results of interrupted tests from the same experiment
(Figure 2.8(c)) that the imposed temperature reduction promoted more extensive DIMT at high true strains, leading to higher hardening rates that enhanced the toughness.

![Figure 2.8](image_url)

Figure 2.8 (a) Process schematic for the two-temperature interrupted tests. (b) Tensile mechanical performance for specimens initially loaded to $\varepsilon \approx 0.15$ at 100 $^\circ$C, then unloaded, cooled, and reloaded to failure at temperatures from -150 to 23 $^\circ$C. (c) Deformation-induced martensite fraction determined from the interrupted specimens. This figure was modified from Tsuchida and Harjo [49] under the terms of a Creative Commons Attribution license (CC BY 4.0).

Tsuchida and Harjo's work suggests that controlling deformation temperature during forming operations, e.g., by cryogenic treatment of the steel, could be used as a lever to control austenite stability and enhance the mechanical properties. Furthermore, it is clear that there may also be an argument for above-ambient temperature warm forming, in cases where it is desirable to impede the kinetics of DIMT. An additional mechanism by which temperatures evolve as a function of strain has been well documented in the literature; in the next section, the physical phenomenon of deformation-induced heating is further explored.

### 2.4.2 Strain Rate

Strain rate is known to affect DIMT behavior in two intrinsically coupled ways. First, as strain rate is changed from quasi-static to dynamic, the slip behavior of dislocations changes. Because the dislocation
slip behavior is notionally strain rate dependent in each phase [50], the strain partitioning between phases could be strain rate sensitive. Meanwhile, a change in dislocation motion within the austenite could change the number of shear band intersections, and thus the number of potential martensite nucleation sites. Furthermore, dislocation mobility affects the stresses achieved in each phase, and thus the mechanical driving force for transformation.

Second, at dynamic strain rates, deformation-induced heating increases the temperature of the steel. This heat originates as work from the process of plastic deformation, and can be computed from Equation 2.3, where $T_0$ is the temperature prior to the start of the test, and $C$, $\rho$, and $\beta_TQ$ are the specific heat capacity, density, and Taylor-Quinney coefficient of the steel, respectively.

$$T_{adiabatic} = T_0 + \frac{\beta_TQ \int_0^\varepsilon \sigma dz}{\rho C}$$

Although some uncertainty exists within the literature about the root cause of deformation-induced heating [51–53], it is well documented that approximately 90 percent of the work associated with plastic deformation ($\int_0^\varepsilon \sigma dz$) is converted to heat ($\beta_TQ = 0.9$). Plastic work is converted to heat at all strain rates. However, during deformation at quasi-static rates, the heat has ample time to dissipate into the environment. Meanwhile, as strain rates increase, the heat does not have time to dissipate. Within the literature, steels are known to reach adiabatic conditions, where heat dissipation is negligible, at strain rates around $10^{-2}$ to $10^{-1}$ s$^{-1}$, depending on the geometry of the specimen [9].

According to the classical interpretation of the OC model, increases in temperature as a result of deformation-induced heating would have the effect of stabilizing the austenite and retarding the DIMT kinetics. However, rising temperature would also increase the stacking fault energy of austenite, leading to smaller stacking fault widths, and thus a lower volume of potential stacking fault intersections that could be able to form a martensite embryo [15]. Furthermore, in the absence of a rise in temperature, strain rate could independently influence the dislocation slip, leading to an additional dependence of DIMT on strain rate. In a series of inventive papers, Hecker et al. discussed this complex problem manifested in a 304 austenitic stainless steel [54, 55]. In Part I of the classic paper [54], uniaxial and sheet punching tests were used in conjunction with magnetic measurement techniques to quantify DIMT and mechanical properties over a range of temperatures (−80 to 50 °C) and strain rates ($10^{-3}$ to $10^{3}$ s$^{-1}$). In Part II of the paper [55], the nucleation and growth of martensite was observed with transmission electron microscopy (TEM). TEM characterization substantiated Olson and Cohen’s postulation that $\alpha$'-martensite nucleates at the intersections of shear bands, containing faults, twins, and $\varepsilon$-martensite [15]. The number of shear band intersections at a given strain was observed to be higher at greater strain rates, suggesting that
forming at high temperatures and low strain rates could suppress DIMT.

Many studies about the strain rate sensitivity of DIMT have been performed for austenite-containing AHSS, including Q&P, TRIP, and Medium-Mn grades. In those studies, increasing the strain rate from quasi-static to the adiabatic threshold, $\sim 10^{-2} \text{ s}^{-1}$, suppressed DIMT. For example, Enloe et al. and Savic et al. reported strain rate dependent work hardening, elongation, and strength in TRIP and Q&P steels, respectively, with the modified deformation behavior being attributed to thermal stabilization of austenite and modified dislocation slip phenomena [56, 57]. Meanwhile, in studies of Q&P and TRIP steels by Poling and others, DIMT was less sensitive to strain rate shifts [7, 58–60]. Despite the mixed strain rate dependence of DIMT from quasi-static to intermediate rates, a different result has been reported for strain rates above the adiabatic threshold. For instance, Zou et al. and Wang et al. reported an acceleration of DIMT kinetics in Q&P steels at rates above $10^{-1} \text{ s}^{-1}$, which was attributed to an increased number of martensite nucleation sites [61, 62]. In many of the aforementioned studies, the authors noted that it was difficult to distinguish between the roles of strain rate and temperature in controlling DIMT kinetics, given the intrinsic link between strain rate and deformation-induced heating. Thus, experiments that aim to decouple the influences of deformation-induced heating and strain rate on DIMT are needed, to better understand the levers that control DIMT at high strain rates.

2.4.3 Strain State

While reporting property combinations of AHSS, mechanical properties are generally described for uniaxial tension [5]. This is because uniaxial tension testing is simple. Furthermore, tensile testing is repeatable, because it is generally insensitive to the specimen geometry prior to strain localization [63]. However, in engineering applications, including during sheet forming and in crash scenarios, AHSS are subject to a range of strain states beyond uniaxial tension. Thus, it is critical to understand how strain state affects the progression of DIMT, and correspondingly, the strengthening contribution of the TRIP-effect.

Sheet metal forming is a technique by which sheets of metal are plastically deformed into complex shapes while avoiding fracture or excessive thinning. Miles provides a useful review of formability testing, starting with a description of the fundamental forming operations [64]. Bending is the process by which metal is deformed around a radius, producing tensile stresses on the exterior of the bend and compressive stresses in the interior. In some cases, bent sections are straightened subsequently, imparting notable cold work, although net strains are small. Stretching is a process by which net elongation occurs in at least one direction of the sheet. In biaxial stretching, elongation occurs in both directions along the plane of the sheet, while the thickness is reduced. In plane strain stretching, elongation occurs along the principal axis,
with the normal strain in the plane of the sheet being equal to zero. Drawing causes elongation in one
direction at the consequence of compression in the perpendicular direction. These forming operations are
classified by the magnitude and direction of the strains along the two directions in the plane of the sheet
metal; these strains are known as the principal strains, $\varepsilon_1$ (major strain) and $\varepsilon_2$ (minor strain). In
conventional uniaxial tension testing, the major strain is positive. Meanwhile, the minor strain is negative,
and has a magnitude of one half of the major strain, assuming isotropic plastic deformation.

The forming limit diagram (FLD) describes the major and minor strains at which a material can no
longer be formed without necking or failure [65]. To create a useful FLD, sheets are clamped in-plane, then
a punch applying a force along the plane normal is used to stretch the specimens. In the FLD construction
process, multiple strain states must be tested, so a variety of sample widths are deformed, leading to
various amounts of constraint along the minor axis [66]. In FLD tests, $\varepsilon_1$ and $\varepsilon_2$ are frequently measured
by tracking the motion of speckle patterns using digital image correlation (DIC).

Strain state is known to critically influence the DIMT response, and thus the mechanical behavior, of
metastable austenitic steels. For example, Hecker et al. demonstrated that strain state influenced the
progression of DIMT in 304 stainless steel, with a greater number of shear band intersections, and more
rapid DIMT, occurring in biaxial tension relative to uniaxial tension [54, 55]. Hecker’s studies of 304
stainless steel have been invaluable in optimizing forming processes for steels containing retained austenite.
However, AHSS microstructures containing fractions of bainite, ferrite, and tempered martensite do not
behave identically to the fully austenitic microstructure of 304 stainless steel. Some formability studies
have been performed on first and second-generation AHSS grades [67–70]. For instance, in a TRIP steel,
Streicher observed that a greater fraction of martensite was formed in specimens deformed under plane
strain conditions, followed by biaxial tension and uniaxial tension, for a given effective strain [71, 72].

Integrating formability testing and integrated computational materials engineering (ICME) models into
the design of AHSS components has shown the potential for significant performance improvements. One
project demonstrated that with careful implementation of AHSS, augmented by atomistic, crystal
plasticity, and forming models, weight savings of 30 pct were achieved in an automotive subassembly [73].
Formability models for several AHSS grades were applied to predict the formability performance of those
grades in a T-shaped component, which contained features resembling the upper section of a side-structure
B-Pillar [73, 74]. In this forming operation, drawing, bending, and stretching forming modes were applied
to create the T-shaped component; forming evaluation was performed ex-situ with DIC and plotted using a
strain path diagram (SPD) in Figure 2.9. The ICME model’s ability to predict formed microstructure was
validated by comparing the measured retained austenite fraction with the modeled retained austenite
fraction. It was concluded that the model accurately predicted retained austenite volume fraction for
uniaxial tension. Furthermore, it was demonstrated that the amount of retained austenite after deformation was a function of both the effective strain and the strain path, with biaxial stretching reducing the volume of retained austenite by over 50 pct, in comparison to an 11 pct reduction for uniaxial tension [74].

Figure 2.9 Strain path diagram (SPD) of several locations, I, II, and III, on the T-shaped component created with a metastable austenite-bearing AHSS. The white circles annotate the regions of the T-shaped component represented on the FLD. Major and minor strain were measured using ex-situ digital image correlation. This figure was created using strain data from Wang et al. [74].

Despite the noteworthy contributions of Hecker, Streicher, Hector, and Wang, the forming response, and the associated DIMT kinetics, of third-generation AHSS have not been studied comprehensively. Numerous possibilities exist to enhance the forming community’s understanding of the TRIP-effect; for instance, possibilities for modifying forming processes exist, which could be developed given a clearer understanding of the link between DIMT and strain state, strain rate, and temperature. The path toward this understanding would be made smoother if a robust formability testing capability was developed at the Colorado School of Mines; efforts towards this goal are ongoing.

2.5 Methods to Measure Austenite Volume Fraction

Since the amount and stability of austenite are critical for dictating the potency of the TRIP-effect in AHSS, many studies attempt to measure the amount of austenite in the steel before, during, and after deformation. Several methods to quantify the fraction of the various phases/constituents in AHSS have
been adopted. For instance, for a AISI 304 austenitic stainless steel, Talonen et al. compared several methods, including optical microscopy (OM), magnetic measurements, X-ray diffraction (XRD), and density measurements [75]. In OM, contrast between phases is created by chemical etching. Subsequently, the area of each phase in an image is measured. In magnetic measurements, the ferromagnetism of α’-ferrite is assessed by placing the metallurgical specimen in a magnetic field. In XRD, the relative intensities of diffracted peaks are comparatively assessed [76]. Finally, with density measurements, density/volume changed associated with DIMT is detected. For AISI 304, Talonen et al. reported that OM was relatively insensitive to the small α’ particles. Meanwhile, magnetic measurements and density measurements performed better, with the limitation that the specimen dimensions influenced the results. Finally, XRD appeared to exhibit good accuracy, with the caveat that it was intrinsically sensitive to crystallographic texture (see Section 2.5.2) [75].

Metastable austenitic steels that undergo relatively extensive transformations to martensite are ideal for accurately measuring the kinetics of DIMT. For instance, an AISI 304 steel can transform nearly completely to martensite, given sufficient cold rolling reductions [75]. This means that comparative measurements of the fraction of austenite to martensite are simple, because the signal/contrast generated by each phase is relatively large, compared to the uncertainty associated with the measurement. In modern AHSS, the volume fractions of austenite are comparatively lower, and so better resolution is required. In reality, the density measurements and OM are insufficient for detecting DIMT in third-generation AHSS. The following sections describe the techniques that are presently used to assess austenite content in third-generation AHSS. The sections are separated into spatially-resolved and bulk techniques. In practice, a combination of both types of analysis is generally required for studies of the TRIP-effect.

2.5.1 Spatially Resolved Microscopy

Microscopy techniques, such as OM and scanning electron microscopy (SEM), are marginally capable of resolving the distribution and morphology of the phases/microconstituents in AHSS. For instance, viewed with an optical microscope, distinct contrast exists between the ferrite, martensite, and austenite in a Q&P steel that has been carefully metallographically ground, polished, and chemically etched [77]. However, the interpretation of such images is subjective, because OM lacks a direct measurement of the crystallography of each constituent. Furthermore, the presence of martensite formed by DIMT can be difficult to detect due to the limited resolution of OM [75]. The practical limit to the spatial resolution of each microscopy technique is on the order of the wavelength of the particle used for imaging. Because of the comparatively small wavelength of electrons in an electron microscope, the spatial resolution of SEM is vastly superior to OM. Thus, in SEM, phases/microconstituents within a steel can be identified not only by their imaging
contrast but also by their morphology and crystallography on the sub-µm scale.

A benefit of SEM is the emerging availability and capability of electron backscatter diffraction (EBSD) mapping [78]. In this technique, diffraction patterns are indexed for each pixel of the SEM image, which provides a spatially-resolved map of the crystallographic structure and lattice orientation. For instance, using EBSD, austenite is clearly differentiated from martensite because austenite exhibits a characteristic face-centered cubic diffraction pattern [77]. Since its inception in the 1990’s, the resolution and speed of EBSD analysis has improved with iterative enhancements in electron source and detector technology. For instance, the implementation of field emission guns, rather than W filaments, greatly reduced the minimum detectable feature size [79]. However, the utility of EBSD for characterizing the austenite in AHSS is still limited by both the scan rate and the practical spatial resolution. For instance, austenite existing with a fine lath morphology may not be properly indexed during EBSD analysis, because it is on the order of the same size as the EBSD beam interaction volume, which would have the effect of reducing the measured austenite fraction. Furthermore, EBSD is susceptible to bias error in steels with heterogeneous distributions of austenite (e.g., due to chemical and/or microstructural banding) [80].

2.5.2 Bulk Diffraction Analysis

Several bulk diffraction analysis techniques are available for phase volume fraction analysis. Such techniques, including neutron and X-ray diffraction, are advantageous because the sample preparation is simple and the interaction volume of the measurement is large. For example, particularly while characterizing fine-grained materials using purpose-built powder diffraction beamlines, statistically representative quantities of grains can be measured. Furthermore, these techniques can provide adequate temporal resolution for collecting phase volume fraction measurements in-situ during continuous (uninterrupted) deformation processes. Bulk diffraction studies using lab-scale diffractometers can also reach high levels of statistical rigor, but require more attention to sample surface preparation and scan parameter optimization. Generally, lab-scale diffractometers lack the temporal resolution and signal-to-noise ratio required to resolve the evolution of phase volume fraction as a function of strain, for uninterrupted loading experiments.

One limitation of bulk diffraction analysis is that it is not spatially resolved, and so it cannot distinguish between different microstructures having similar crystal structures. For instance, despite having unique microstructures, bainite, ferrite, and martensite share a nearly indistinguishable crystal structure and lattice parameter, i.e., body-centered cubic, \( a = 2.86 \text{ Å} \), which makes them exceedingly difficult to differentiate [81]. For this reason, bulk diffraction studies frequently do not attempt to distinguish between bainite, ferrite, and martensite, and instead treat each as “ferrite” (\( \alpha \)). Thus, the volume fraction of each
“phase” is given by Equation 2.4:

\[ f_\gamma + f_\alpha = 1 \]  

(2.4)

Where \( f_\gamma \) and \( f_\alpha \) are the volume fractions of austenite and ferrite, respectively. Fortunately, because \( \gamma \) and \( \alpha \) have dissimilar lattice parameters \( (a) \), the low-index reflections for each crystal structure do not overlap. Thus, the integrated intensities of the \( \gamma \) and \( \alpha \) peaks can be compared to measure \( f_\gamma \) using Equation 2.5 and 2.6 [76]:

\[
f_\gamma = \frac{\frac{1}{n} \sum_{i=1}^{n} \frac{I_i^\gamma}{R_i^\gamma}}{\frac{1}{m} \sum_{i=1}^{m} \frac{I_i^\alpha}{R_i^\alpha}}
\]  

(2.5)

\[
R_{hkl} = \frac{F^2 p LP e^{-2M}}{v^2}
\]  

(2.6)

Where \( F^2 \) is the structure factor, \( p \) is the multiplicity of the reflection, \( LP \) is the Lorentz-Polarization factor, which is equal to \((1 + \cos^22\theta)/\sin^2\theta\cos\theta\), \( \theta \) is the Bragg angle, \( e^{-2M} \) is equal to the temperature factor (provided in Table II of ASTM E975 [82]), and \( v \) is the volume of the unit cell [76, 82]. An additional limitation of bulk diffraction analysis is that the \( f_\gamma \) measured with Equation 2.5 is highly susceptible to bias error introduced by the presence of crystallographic texture [75, 81, 83], which is the preferred orientation of the crystal lattice relative to the axes of the bulk specimen. This problematic aspect of diffraction analysis has received some recent attention at the Colorado School of Mines [84].

### 2.6 Methods to Analyze Dislocation Content

Transformation-induced plasticity, dislocation motion, dislocation recovery, and interface plasticity have been identified as unique mechanisms that perhaps facilitate plasticity in TRIP-assisted steels at ambient or slightly elevated temperatures [85–87]. Thus, for the purpose of tailoring deformation behavior, it is prudent to characterize the extent and nature of dislocation content as a function of \( \varepsilon \). Several approaches are commonly applied to characterize dislocation content and microstructure, in order of increasing interaction volume: transmission electron microscopy (TEM), EBSD, and X-ray diffraction line-profile analysis (XRD-LPA).

#### 2.6.1 Transmission Electron Microscopy (TEM)

In TEM, dislocation density (line length per unit volume) can be directly measured in the bright field imaging mode, due to the contrast caused by the elastic strain fields around dislocations. For instance, Pešička et al. measured the dislocation density of several tempered martensitic chromium steels (X20 and
P91) with a combination of techniques, including TEM [88]. In this method, a grid of lines is superimposed on a TEM micrograph, then the number of intersections of dislocations with the grid lines is normalized by the grid line length. A micrograph demonstrating the technique is shown in Figure 2.10 after Pešićka et al. [88]. Despite the elegance of TEM-based dislocation density measurements, the technique is prohibitively time-consuming. Furthermore, the technique becomes increasingly difficult in complex microstructures with high dislocation densities, such as AHSS, and the method does not account for the effect of foil thickness on the perceived dislocation density.

Figure 2.10 TEM micrograph of X20 after the line intersection method was used to measure the dislocation density. This figure was reused from Pešićka et al. [88] with permission of the publisher (see Appendix F).

2.6.2 Electron Backscatter Diffraction (EBSD)

EBSD cannot directly resolve dislocations, because the technique operates at a magnification that is insufficient to resolve the contrast of individual elastic strain fields around dislocations. However, EBSD can estimate the density of geometrically necessary dislocations by comparing the orientations of neighboring “kernels” on an EBSD map (for an extensive review, see Wright et al. [89]). The principal benefit of implementing EBSD for strain analysis is the availability of concurrent local orientation and phase mapping. For instance, Figure 2.11 shows EBSD maps of a Medium-Mn steel after Sevsek et al. [90], where local strains in the body-centered cubic (α/α′) and the face-centered cubic (γ) phases are spatially resolved using kernel average misorientation (KAM) analysis.
Figure 2.11 EBSD maps of a Medium-Mn steel deformed in tension to $\varepsilon = 2\%$. Inverse pole figure (IPF) maps are plotted relative to the tensile axis for (a) ferrite/martensite ($\alpha/\alpha'$) and (b) austenite ($\gamma$). Kernel average misorientation (KAM) maps for (c) $\alpha/\alpha'$ and (d) $\gamma$. This figure was reused from Sevsek et al. [90] with permission of the publisher (see Appendix F).

For a Q&P steel, Adams applied integrated EBSD and SEM-DIC analysis to resolve strain partitioning and DIMT in-situ during tensile deformation of a Q&P steel [91]. This novel work led to the conclusion that austenite stability is heavily correlated to: (i) the Taylor factor, or the orientation of dislocation slip planes in retained austenite relative to the tensile axis; (ii) the quantity of geometrically necessary dislocations in the austenite (determined with KAM analysis); and (iii) alignment of the major axis of lath-type austenite grains with the tensile axis. Despite the strengths of EBSD, the technique is limited by the relatively small, near-surface interaction volumes and small scan areas (a characteristic of SEM).

2.6.3 X-Ray Diffraction Line Profile Analysis (XRD-LPA)

Conveniently, XRD-LPA can often be applied to powder diffraction scans collected for the purpose of measuring $V_\gamma$ (see Section 2.5.2). In XRD-LPA, a beam of primarily monochromatic X-rays impinges on a bulk sample, then diffracts when the Bragg condition is met [76]. As a result, XRD-LPA avoids some of the key weaknesses of EBSD and TEM as dislocation analysis techniques; XRD-LPA analyzes a larger cross section and depth of material, and is thus less sensitive to low grain statistics and surface roughness. The basis for strain analysis with XRD-LPA is that strain broadening is strongly $\{hkl\}$-dependent. Thus, the dislocation density can be approximated from the slope of the peak breadth ($\Delta K$) versus diffraction vector ($K'$) plot. In the literature, $\Delta K$ is taken as either the full width at half maximum (FWHM) or the
integral breadth [92]. For elastically isotropic materials with spherical-shaped cubic crystals, the linear function of $\Delta K$ versus $K$ is given by the conventional Williamson Hall (cWH) relation [93] in Equation 2.7:

$$\Delta K = \frac{0.9}{D} + K(0.263b\sqrt{\rho})$$

Where $D$ is the grain size, $b$ is the magnitude of the burgers vector, and the diffraction vector ($K$) is determined using Equation 2.8:

$$K = \frac{2\sin\theta}{\lambda}$$

Where $\lambda$ is the wavelength of the X-ray source in angstroms (Å). From inspection of Equation 2.7, it is apparent that the slope of the cWH plot is proportional to the square root of dislocation density. Despite its simplicity, the cWH relation generally does not generate an acceptable linear fit in cases of strong strain anisotropy [94], such as in martensitic microstructures. For instance, Pešička et al. performed XRD-LPA on several tempered martensitic chromium steels, and showed that the slope of the cWH plot was reduced after extensive tempering, but the broadening response as a function of $\sin\theta$ was not perfectly linear [88] (Figure 2.12).

![Figure 2.12](image)

Figure 2.12 (a) XRD line profile of the $\{310\}$ peak of P91 after austenitizing at 1100 °C and air cooling and after subsequent tempering at 750 °C for 0.25, 1, and 100 hours. Strain broadening was reduced after tempering. (b) Conventional Williamson-Hall (cWH) plots for the six lowest-index diffraction peaks in P91. Note that the slope of the plot is notionally proportional to the dislocation density of the steel, but strain anisotropy led to deviation from the linear cWH relation. This figure was adapted from Pešička et al. [88] with permission of the publisher (see Appendix F).

Several methods have been developed to account for the anisotropy-based deviation from the cWH relation. Generally, the methods implement a “scaling parameter” which is notionally based on the average dislocation character and determined by empirical fitting of the line broadening data. The most common
approach is the modified Williamson Hall (mWH) relation by Ungár et al. [95, 96]. With this approach, several experimentally determined factors are introduced to account for the dislocation character, including the dislocation contrast factor \((C_{ave})\) and the dislocation distribution parameter \((M)\). The modified relation is shown in Equation 2.9:

\[
\Delta K = \frac{0.9}{D} + bM \sqrt{\frac{\pi \rho}{2}} \left( C_{ave}^{1/2} \right)
\] (2.9)

For a full review of the mWH method, see Ungár et al. [95, 96] and HajyAkbary et al. [94]. It is noted that based on the outcome of the mWH empirical fitting approach, \(C_{ave}\) is strongly \{hkl\}-dependent and is also related to the edge/screw character of the dislocations. Despite its ability to accurately measure dislocation content, as proven by comparisons to direct measurement via TEM [97] and by correlation of the mWH-determined dislocation density to the flow stress via the Bailey Hirsch relationship [98], the mWH method is computationally cumbersome, and the correctness of microstructure-based terms (i.e., \(M\) and \(C_{ave}\)) is not verifiable without weighty TEM investigations.

The Diffraction Young’s Modulus Correction (DYMC) and Direct Fitting Williamson-Hall (DF-WH) methods were introduced by Takaki et al. to avoid the potentially arbitrary and indirect microstructure-based fitting procedures associated with the mWH method [99, 100]. Both methods propose that strain anisotropy can be corrected using the diffraction Young’s modulus ratio (\(\omega\)):

\[
\Delta K = \frac{0.9}{D} + \frac{\varepsilon_{DF} K}{\omega}
\] (2.10)

Where \(\omega\) is \{hkl\}-dependent and \(\varepsilon_{DF}\) is the average true microstrain contained within the X-ray interaction volume. In the DYMC method, \(\omega\) is calculated based on the known diffraction Young’s modulus of each peak (e.g., determined by the Kröner, Reuss, or inverse Kröner models [101]). As an extension of the DYMC method, the DF-WH method empirically determines \(\omega\) based on iterative evaluation of Equation 2.10 for \(0.5 \leq \omega_{h00} \leq 1.0\). Thus, the goodness of fit of Equation 2.10 is maximized without direct intervention of terms that describe the dislocation character. Both the DYMC and DF-WH methods have shown good agreement with the mWH method [100]. Because of the enhanced simplicity of the DF-WH approach, the method is compelling for high-throughput microstrain analysis.
3.1 Metallurgical Sample Preparation

This section provides an overview of the metallurgical specimen preparation techniques used in this work. Specific details about the nature of the deformation processing experiments are contained in the chapters that follow. An account of the specimen preparation techniques used for every results chapter is included in the following subsections.

3.1.1 Zinc Coating Removal

Prior to any metallurgical analysis, the Zn anti-corrosion coating was removed from the steels. A chemical removal procedure was used, where the steels were submerged in a solution of 50 percent water, 30 percent hydrochloric acid, and 20 percent hydrogen peroxide. During this step, care was taken to avoid excessive heating or bubbling of the solution; some heat was generated during the chemical removal process, particularly for large sections of steel. The Zn removal step was performed to improve edge retention during the subsequent grinding and polishing steps (see Section 3.1.2).

3.1.2 Microscopy Sample Preparation

Standard metallographic practices were used to resolve the microstructure of the AHSS studied in this work. Specimens were sectioned to a width and length no greater than one centimeter, then mounted in a compression hot mounting press using Bakelite with a curing temperature of 150 °C and time of four minutes. Subsequently, the mounted specimens were sequentially wet-ground using 240, 400, and 600-grit abrasive grinding paper, then sequentially polished using nine, six, three, and one µm diamond polishing compound. Finally, the specimens were vibratory-polished using a solution of 0.05 µm colloidal silica polishing compound. Between each preparation step, the specimens were cleaned using a combination of mechanical agitation, soap, and isopropyl alcohol, then rinsed with ethanol. Finally, after the series of grinding and polishing steps, the specimens were etched with 0.5 percent Nital solution for approximately 15 seconds, to create contrast between the constituents in the microstructure.

3.1.3 X-Ray Diffraction Sample Preparation

One inch square XRD samples were prepared in the normal plane using the metallographic practices described in Section 3.1.2. One difference was that the XRD specimens were not mounted in a metallographic mount, because their size would prohibit placement in the mounting press. Furthermore, it
was desired to avoid unnecessary heating of the specimens, which was thought to potentially temper the steel. To hold the specimens during the grinding and polishing steps, each specimen was adhered to a Bakelite puck using 3M Scotch permanent double-sided tape. After a mirror polish was achieved, the specimens were chemically thinned to a total thickness reduction of at least 20 µm, using a solution of 50 percent water, 30 percent hydrochloric acid, and 20 percent hydrogen peroxide. The purpose of the chemical thinning step was to reduce the effects of a polishing-induced deformation layer of the XRD-based measurements of the austenite phase fraction. To avoid oxide buildup on the specimens during chemical thinning, the solution was frequently replaced.

3.2 Light Optical Microscopy

Most optical micrographs were collected for the steels using bright-field microscopy. In selected cases, for instance, to distinguish ferrite from the other constituents in the microstructure, polarized light microscopy was used. All light optical micrographs (OM) were collected using an Olympus PMG 3 and an Olympus DSX500. Optical micrographs collected in either the transverse or rolling-direction plane were located at the nominal center of the thickness of the steel. Meanwhile, optical micrographs collected in the normal direction plane were located at an approximate distance of 200 µm from the surface of the sheets. This distance was selected to reduce the likelihood of a potential decarburization layer affecting the representativeness of the micrographs, while minimizing the required grinding and polishing depth.

3.3 Scanning Electron Microscopy

Scanning electron micrographs were collected in the secondary electron imaging mode using an FEI Quanta 600 environmental scanning electron microscope (ESEM) and a JEOL JSM7000F field-emission scanning electron microscope (FESEM). For imaging in the FESEM, a working distance of nine to ten millimeters was used.

For cases where additional crystallographic information was required, supplementary electron-backscatter diffraction (EBSD) analysis was performed using the FESEM. In the EBSD technique, the specimen was tilted to 70° with respect to the axis of the electron probe. Then, crystallographic information (in the form of Kikuchi patterns) was collected using an EDAX detector and OIM Analysis software, for maps having an approximate width of 31 µm, a square-grid step size of 0.1 µm, and a electron probe spot size of “Medium-11”. Subsequently, data visualization, phase volume fraction, crystallographic texture, and kernel average misorientation (KAM) analysis were performed using MTEX [102].
Reflection-mode XRD scans were collected on the steels as-received and after deformation at various effective strains, strain rates, and temperatures. Although the exact XRD scan parameters were chosen based on the needs of a given study, some general characteristics of the procedure were held constant.

Multiple X-ray sources were used in different studies, creating incident X-rays with different energies. Over the course of the thesis, Cu and Mo sources were used, having primary wavelengths of $\lambda_{\text{Cu}} = 1.54 \text{ Å}$ and $\lambda_{\text{Mo}} = 0.71 \text{ Å}$. The Mo source was expected to have a larger X-ray interaction volume at the consequence of a reduced Bragg angle ($2\theta$) range. A comparison of the instrument broadening profiles for each X-ray source is provided in Appendix B. In some cases, to represent the peak position by a term that was normalized by $\lambda$, the momentum transfer of scattering ($Q$) was computed:

$$Q = \frac{4\pi\sin\theta}{\lambda} \quad (3.1)$$

Scans were generally performed to cover a range of at least $2.75 < Q < 7.75$, which covered the four lowest-index reflections of each constituent: austenite $\gamma(111)$, $\gamma(200)$, $\gamma(220)$, and $\gamma(311)$, and ferrite/martensite $\alpha(110)$, $\alpha(200)$, $\alpha(211)$, and $\alpha(220)$.

XRD scans were collected on two instruments, a PANalytical X’Pert and a PANalytical Empyrean. Each diffractometer was equipped with interchangeable optics that allowed for tailoring the beam spot size. The beam spot size was controlled using a combination of incident beam slits and masks, with the goal of maximizing the beam spot size on the surface of the sample. In typical lab diffractometers, signal increases proportional to beam spot size, so a maximum spot size was preferred to enhance the signal-to-noise ratio of the scans. Prior to each scan, the specimen was placed on an low-background amorphous silicon wafer to reduce erroneous peak reflections from below the specimen. Then, the height of the specimen within the diffractometer was aligned with a dial gauge. Both diffractometers were used with relatively large area detectors, to enhance the signal-to-noise ratio of the scans. Scans were collected over a step size of 0.01 to 0.03 degrees, and scan time was selected to reach a minimum signal-to-noise ratio of at least 20, for the $\gamma(111)$ reflection.

After the XRD scans were collected, the diffraction patterns were fit using using HighScore Plus software by PANalytical, using a “Pseudo-Voigt” function. The integrated area of each peak, determined by the peak fits from HighScore Plus, were subsequently compared using the Cullity method [76] described in Chapter 2. To ensure maximum repeatability, the entire analysis process was automated by a combination of HighScore Plus macros and an external MATLAB script used to compare the integrated intensities. A sample MATLAB script that shows the process is outlined in Appendix D.2.
CHAPTER 4
EXPERIMENTAL DESIGN

4.1 Introduction

The materials and experimental design were selected on the basis of the research questions proposed in Chapter 1 and the literature review provided in Chapter 2. Deformation processing experiments, microstructure characterization, and DIMT models were employed to clarify the effects of strain rate and temperature on the DIMT response of Q&P steels.

4.2 Experimental Steels

Several commercially-produced Zn-coated C-Mn-Si Q&P steels were provided by Cleveland Cliffs Steel Corporation. The steels were created using a fully-integrated steelmaking process. Following hot rolling in a hot strip mill, the steels were coiled, cooled, then cold rolled to a nominal 50 percent thickness reduction. Subsequently, the steels were heat treated and Zn-coated. Some of the Q&P steels appeared to be intercritically annealed to form approximately 10 to 20 percent intercritical ferrite, while others appeared to have been fully austenitized, based on the lack of intercritical ferrite in the final microstructure. Subsequently, each steel was subjected to a Q&P process.

The Q&P processes were tailored to create target ultimate tensile strengths (UTS) of 980 and 1180 MPa. The Q&P steels of each strength level existed in two thicknesses. The thinner steel of each strength level was provided for limited use in one of the synchrotron studies, because the in-situ diffraction data were expected to be more reliable for the thinner steel. The thicker steel of each strength level was provided for all other experiments. Here, steels are designated based on strength level and thickness; for example, QP980-1.0 refers to a steel with a UTS of 980 MPa and an as-coated thickness of 1.0 mm. For the thicker steels of each strength level, two variants “A” and “B” were characterized. The variants of each strength level (e.g., QP980-1.4-A and QP980-1.4-B) were processed with a similar Q&P process and were removed from the same finished coil of steel. However, due to variations intrinsic in the Q&P process at commercial scale, the properties were slightly different. Thus, the steels were treated as unique, to better understand the role of small process variations on the microstructure and properties. A comparison of these variants is made in Chapter 5. A steel that was processed to create a dual phase (DP) microstructure of ferrite and martensite was also provided by Cleveland Cliffs Steel Corporation, to provide a control material with properties that would be similar to a Q&P steel lacking austenite.
Several key attributes of each steel are listed in Table 4.1, including the chemical composition; determined by LECO elemental analysis, austenite phase fraction; determined by XRD, and ferrite phase fraction; determined by OM and SEM. Because of the rigorous use of XRD (presented in the following chapters), the austenite fraction presented is believed to be accurate. However, there was some degree of uncertainty in the ferrite fractions tabulated in Table 4.1; despite the use of concurrent OM and SEM, a repeatable and robust method to quantitatively distinguish between bainite, martensite, and ferrite remains elusive within the literature. Here, a combination of polarized light microscopy and scanning electron microscopy was used to develop contrast between the ferrite islands, which showed the greatest amount of relief/depth after etching, and the remainder of the constituents. Based on image analysis of ten images of each steel grade, it is expected that the variability in the measurement and distribution of ferrite for each steel did not exceed five volume percent.

Table 4.1 Composition and Phase Fractions for Each Steel

<table>
<thead>
<tr>
<th>Steel</th>
<th>C (wt pct)</th>
<th>Mn (wt pct)</th>
<th>Si (wt pct)</th>
<th>Cr (wt pct)</th>
<th>Mo (wt pct)</th>
<th>Austenite (vol pct)*</th>
<th>Ferrite (vol pct)</th>
</tr>
</thead>
<tbody>
<tr>
<td>QP980-1.4-A</td>
<td>0.201</td>
<td>1.93</td>
<td>1.62</td>
<td>0.027</td>
<td>0.004</td>
<td>11.3 ± 0.5</td>
<td>23</td>
</tr>
<tr>
<td>QP980-1.4-B</td>
<td>0.201</td>
<td>1.93</td>
<td>1.62</td>
<td>0.027</td>
<td>0.004</td>
<td>11.3 ± 0.2</td>
<td>20</td>
</tr>
<tr>
<td>QP980-1.0</td>
<td>0.20</td>
<td>1.96</td>
<td>1.68</td>
<td>0.03</td>
<td>&lt;0.02</td>
<td>11.2 ± 0.4</td>
<td>26</td>
</tr>
<tr>
<td>QP1180-1.6-A</td>
<td>0.204</td>
<td>2.26</td>
<td>1.74</td>
<td>0.043</td>
<td>0.008</td>
<td>11.4 ± 0.4</td>
<td>22</td>
</tr>
<tr>
<td>QP1180-1.6-B</td>
<td>0.204</td>
<td>2.26</td>
<td>1.74</td>
<td>0.043</td>
<td>0.008</td>
<td>12.6 ± 0.2</td>
<td>17</td>
</tr>
<tr>
<td>QP1180-1.0</td>
<td>0.19</td>
<td>2.31</td>
<td>1.64</td>
<td>0.02</td>
<td>&lt;0.02</td>
<td>10.7 ± 0.3</td>
<td>0**</td>
</tr>
<tr>
<td>DP980-1.4</td>
<td>0.09</td>
<td>0.23</td>
<td>0.12</td>
<td>0.58</td>
<td>0.10</td>
<td>&lt;1***</td>
<td>42</td>
</tr>
</tbody>
</table>

*Error is the std. dev. of three separate orientations (RD, DD, TD) for one specimen.
**No intercritical ferrite was observed via OM or SEM.
***The austenite fraction was below the min. resolution (~1 vol pct) of the diffractometer.

Table 4.1 shows that each QP980 steel contained approximately 25 volume percent intercritial ferrite. Meanwhile, the QP1180 steels exhibited comparatively less ferrite. There was a large range of ferrite volume fractions in QP1180, with the QP1180-1.6-A and QP1180-1.6-B having a high amount of ferrite relative to QP1180-1.0 (22 and 17 volume percent versus almost none, respectively). The DP980-1.4 exhibited the highest amount of ferrite (42 volume percent), which was likely chosen to control the strength and to enhance the formability of the steel in the absence of any austenite that could contribute to strain hardening via the TRIP-effect. Each Q&P steel contained remarkably similar amounts of austenite; XRD revealed that each steel contained ~10-12 volume percent.

Base micrographs of each steel, collected using OM and FESEM, are presented in Figure 4.1 and Figure 4.2. Each microstructure was prepared based on the procedure given in Section 3.1.2 and imaged under consistent conditions in the Olympus DSX500 OM and JEOL 7000 FESEM. In each steel,
intercritical ferrite was revealed by the deep-etching regions, and generally has a low aspect ratio and a large grain size relative to the other constituents of the microstructure. The contrast between the phases was poor in the optical micrographs; this was because the complex and refined microstructures typical of AHSS approached the practical resolution of visible light. Furthermore, optical microscopy generally exhibits poor depth of field, although the optical micrographs in Figure 4.1 are composite images that were stacked over a 5 µm focal distance for enhanced depth of field.

Contrast between the phases was much clearer using the FESEM (Figure 4.2). For instance, in the inset view of QP980-1.4-A, both film and blocky austenite (γ) grains are clear in the regions with high signal, dark and smooth regions are ferrite (α), and the regions containing a dark matrix with light substructure are either bainite, martensite-austenite microconstituent, or martensite (α’). Complementary to Table 4.1 discussed above, the presence of intercritical ferrite is clear in each of the QP980 steels and in QP1180-1.6-A and QP1180-1.6.B. Meanwhile, the absence of intercritical ferrite in QP1180-1.0 is apparent. The prior austenite grain sizes of each Q&P steel appeared to be relatively consistent. Meanwhile, the ferrite grain structure of the DP980-1.4 appeared to be finer; this was consistent with the comparatively higher amount of intercritical ferrite within the microstructure, because the DP980-1.4 was likely annealed at a lower temperature relative to the other Q&P steels, which would have impeded grain growth during annealing. The grain size of DP980-1.4 was likely refined by the addition of 0.58 weight percent Cr, which is known to produce Cr-rich carbides that facilitate austenitization via the nucleation process, thereby creating comparatively fine austenite grains [103].

### 4.3 Overview of Experiments

Five tensile testing experiments (labeled A-E) were performed to address the research questions. A summary of the experiments is provided in Table 4.2. The main research question that each experiment intended to resolve is provided in the second column. Key details about the nature of the tension testing are also summarized, such as the strain rate and the orientation of the tensile specimens relative to the sheet axes \((i.e., \text{RD}=\text{rolling direction}, \text{DD}=\text{diagonal direction}, \text{and TD}=\text{transverse direction})\). An overview of the design of each experiment is provided in the following sections.
Table 4.2 Summary of the Tensile Testing Experiments

<table>
<thead>
<tr>
<th>Exp.</th>
<th>Research Question</th>
<th>Temp (°C)</th>
<th>Strain Rate (s⁻¹)</th>
<th>Orientation</th>
<th>XRD</th>
<th>Steel Grade</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>1(a)</td>
<td>22*</td>
<td>8.3 × 10⁻⁴</td>
<td>RD DD TD</td>
<td>Ex-situ at Mines</td>
<td>QP980-1.4-A QP980-1.4-B QP1180-1.6-A QP1180-1.6-B</td>
</tr>
<tr>
<td>B</td>
<td>1(b)</td>
<td>22**</td>
<td>10⁻⁴ 10⁻³ 10⁻² 10⁻¹</td>
<td>RD</td>
<td>Ex-situ at Mines</td>
<td>QP980-1.4-B</td>
</tr>
<tr>
<td>C</td>
<td>2(a)</td>
<td>22***</td>
<td>10⁻⁴ 10⁻¹</td>
<td>RD</td>
<td>Ex-situ at Mines</td>
<td>QP980-1.4-A</td>
</tr>
<tr>
<td>D</td>
<td>2(b)</td>
<td>22**</td>
<td>10³</td>
<td>RD</td>
<td>In-situ at APS-32ID</td>
<td>QP980-1.4-A QP1180-1.6-A</td>
</tr>
<tr>
<td>E</td>
<td>3(a,b)</td>
<td>25* 100* 175* 250*</td>
<td>2 × 10⁻⁴</td>
<td>RD</td>
<td>In-situ at APS-1ID</td>
<td>QP980-1.0 QP1180-1.0 DP980-1.4</td>
</tr>
</tbody>
</table>

*Tests were nominally isothermal.
**No external heating was applied. Adiabatic heating occurred due to the dynamic rates.
***Adiabatic heating was simulated with external heating in the DSI Gleeble.

4.3.1 Experiment A: Austenite Stability and Strain Hardening as a Function of Strength Level and Sheet Orientation

An initial study, which is presented in Chapter 5, was performed to measure how sheet orientation influences DIMT kinetics and strain hardening. Monotonic tensile tests were conducted for QP980-1.4-A, QP980-1.4-B, QP1180-1.6-A, and QP1180-1.6-B. After fracture, ex-situ X-ray diffraction (XRD) scans were performed to measure austenite content in the as-received steel and after deformation along the RD, DD, and TD. The extent of DIMT was compared to the strain hardening properties of each steel and sheet orientation.
Figure 4.1 Optical micrographs in the transverse plane of each steel. Each steel was etched in 0.5 percent Nital for 15 seconds.
Figure 4.2 Scanning electron micrographs in the transverse plane of each steel. Each steel was etched in 0.5 percent Nital for 15 seconds.
4.3.2 Experiment B: Strain Rate Dependent Ductility and Strain Hardening in QP980-1.4-B

Chapter 6 describes a study that was performed to evaluate the strain rate dependence of the bulk mechanical properties and DIMT response of QP980-1.4-B. Monotonic tensile tests were performed over a range of strain rates from $10^{-4}$ to $10^{-1}$ s$^{-1}$. Supplementary instrumented tensile tests were performed with an infrared camera and thermocouples, to measure the extent of deformation-induced heating. The measured heating for each strain rate was compared against a Taylor-Quinney model, which elucidated the critical strain rate above which the tests were ostensibly adiabatic. After fracture, the extent of DIMT was measured with XRD. By a combination of the XRD and mechanical testing data, the strain rate sensitive work hardening behavior of QP980-1.4-B was linked to the extent of DIMT. A discussion of the literature was performed to compare the deformation response of QP980-1.4-B to other AHSS variants.

4.3.3 Experiment C: Decoupling the Impacts of Strain Rate and Temperature on TRIP in QP980-1.4-A

Chapter 7 presents a follow-on investigation to the previous strain rate tests. In this study, the individual effects of strain rate and temperature on the strain hardening rate of QP980-1.4-A were examined. During quasi-static tests at $10^{-4}$ s$^{-1}$, resistive heating was used to simulate the deformation-induced heating that occurred during deformation at a strain rate of $10^{-1}$ s$^{-1}$. The deformation behavior of the resistive heated specimens was compared to that of the specimens deformed at $10^{-4}$ and $10^{-1}$ s$^{-1}$ without resistive heating, to isolate the effect of the deformation-induced heating and strain rate on the DIMT kinetics. For each thermal-mechanical condition, the DIMT kinetics were tracked using interrupted tensile tests. The interrupted specimens were characterized by a combination of XRD and electron backscatter diffraction kernel average misorientation (EBSD-KAM) analysis.

4.3.4 Experiment D: Resolving the Martensitic Transformation in-situ at Dynamic Strain Rates Using Synchrotron X-Ray Diffraction

Chapter 8 presents a study which aimed to resolve DIMT in-situ at dynamic strain rates relevant in crash scenarios, $\sim 10^3$ s$^{-1}$. QP980-1.4-A and QP1180-1.6-A were deformed using a fully-instrumented split-Hopkinson tensile bar (SHTB), while high-speed bulk radiography and diffraction videos were captured using synchrotron X-rays generated at the Advanced Photon Source (APS) 32-ID beamline at Argonne National Laboratory (ANL). The high-strain rate tensile mechanical behavior was compared to the in-situ radiography and diffraction data, to quantify the kinetics of DIMT and the stresses on the ferrite/martensite. EBSD maps of the deformed tensile bars were collected to further interpret the bulk diffraction data. The deformation behavior and DIMT kinetics were compared between the two steels, which contained different amounts of intercritical ferrite and otherwise similar microstructures.
4.3.5 Experiment E: Elucidating the Temperature Dependence of TRIP Using Synchrotron X-Ray Diffraction, Constituent Phase Properties, and Strain-Based Kinetics Models

In the final study, presented in Chapter 9, the DIMT kinetics and mechanical properties were resolved for quasi-static tensile tests at temperatures of 25, 100, 175, and 250 °C. Two austenite-bearing AHSS (QP980-1.0 and QP1180-1.0) were tested alongside a high-strength steel with a ferrite/martensite microstructure devoid of austenite (DP980-1.4). High-energy synchrotron X-rays generated at the APS 1-ID beamline at ANL were used to quantify DIMT, and to measure the stresses and strains on individual phases in the steels. The *in-situ* XRD data were used to validate several strain-based kinetics models for DIMT. From this exercise, a modification to the Olson-Cohen model was proposed, where the strain in the austenite, rather than the strain in the bulk, was used to fit the empirical model. Finally, fractography, EBSD-KAM, and EBSD texture analysis were used to further explore the complex relationship between temperature and austenite stability.
CHAPTER 5
EXPERIMENT A: AUSTENITE STABILITY AND STRAIN HARDENING AS A FUNCTION OF STRENGTH LEVEL AND SHEET ORIENTATION

Modified from a paper published in Metallurgical and Materials Transactions A
C.B. Finfrock, A.J. Clarke, G.A. Thomas, and K.D. Clarke

5.1 Abstract

Quenching and partitioning (Q&P) processing of third-generation advanced high strength steels generates multiphase microstructures containing metastable retained austenite. The deformation-induced martensitic transformation of retained austenite improves strength and ductility by increasing the instantaneous strain hardening exponent. This paper explores the influence of the martensitic transformation and strain hardening on tensile performance. Tensile tests were performed on steels with nominally similar compositions and microstructures (11.3 to 12.6 vol. pct retained austenite and 16.7 to 23.4 vol. pct ferrite) at 980 and 1180 MPa ultimate tensile strength levels. For each steel, tensile performance was generally consistent along different orientations in the sheet relative to the rolling direction, but the martensitic transformation kinetics were more rapid along the rolling direction. Neither the amount of retained austenite prior to straining, nor the total amount of retained austenite transformed during straining, could be directly correlated to tensile performance. It is proposed that stability of retained austenite, rather than the austenite volume fraction, greatly influences strain hardening rate, and thus controls strength and ductility. If true, this suggests that tailoring austenite stability is critical for optimizing the forming response and crash performance of quenched and partitioned grades.

5.2 Introduction

Advanced high strength steels (AHSS) are of rapidly increasing commercial interest [105], due to increasing global automobile production and heightened requirements for improved passenger safety and fuel efficiency [106]. Third-generation AHSS are attractive, particularly due to their enhanced mechanical properties relative to first-generation AHSS and lower cost than second-generation AHSS [16]. Due to their enhanced ductility and strength, third-generation AHSS can be used in thinner gauges, reducing body-in-white weight and consequently improving fuel efficiency. Unlike conventional automotive sheet steels, AHSS achieve improved ductility by suppressing strain localization with the transformation-induced

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plasticity (TRIP) effect [107–109]. In practice, a well-documented increase in tensile elongation occurs because of the deformation-induced martensitic transformation (DIMT) [41, 54, 55].

Quenching and partitioning (Q&P) processing, initially proposed by Speer et al. [27, 35, 110, 111], has been used to create steel microstructures that take advantage of TRIP by containing small fractions of retained austenite that are thermally stable at room temperature. In addition, these microstructures contain large-volume fractions of martensite, such that the yield strength is comparable to martensitic steels. Functionally, the yield strength is controlled largely by the presence of martensite, while work hardening and ductility are enhanced by TRIP.

Many Q&P compositions contain carbon [112] for austenite stabilization, manganese [38, 113] for austenite stabilization and solid solution strengthening, and silicon [34] to suppress the formation of carbides, which may otherwise reduce austenite stability by depleting carbon in austenite. By modifying either composition or Q&P heat treatment parameters, the microstructure and mechanical stability of retained austenite can be tailored, with the intention of optimizing deformation behavior.

The role of DIMT on strain hardening rate is of particular interest for suppression of strain localization [64, 114]. Strain hardening has been characterized for individual phases in Q&P steels, with the assumption that Q&P steels behave as composite materials [115–117]. In Q&P-processed steel, strain hardening behavior has been explored in martensite [118] and retained austenite [42]. Similar work has been performed for other AHSS, including manganese-enriched TRIP grades [119]. Deformation processing factors, [54, 55, 107, 120–122] such as strain rate, strain state, and temperature, as well as microstructure factors [123], such as distribution of carbon [33], presence of carbides [124, 125], phase volume fractions [126, 127], austenite grain size and morphology [128–131], and strain partitioning [132–135], have all been proposed as levers that affect the stability of retained austenite.

A simplistic design approach for AHSS that contain austenite for TRIP-enhanced ductility is to maximize the volume fraction of retained austenite; Wang et al. stated that strength/elongation product increases linearly with respect to austenite volume fraction [136]. However, an exhaustive review by De Moor et al. showed that no clear correlation exists between the amount of retained austenite before deformation and total elongation or strength/elongation product [137]. More recent works have investigated such behavior in TRIP steels [138, 139], Medium-Mn steels [140], and Q&P steels [141–143]. Most authors suggest that solely maximizing austenite fraction is inadequate for achieving the best possible tensile properties; rather, the austenite stability must be controlled by tailoring the microstructure by means discussed previously. The consensus in recent literature is that promoting progressive transformation of retained austenite during deformation is critical for achieving optimal mechanical properties; a microstructure which facilitates extensive transformation of austenite across a broad range of
strains will exhibit high work hardening rates that improve formability and suppress strain localization [119, 126, 144, 145]. It is hypothesized that a microstructure optimized for progressive transformation should contain austenite with a spectrum of stabilities.

This paper aims to further clarify the relationship between austenite stability, austenite volume fraction, and tensile mechanical performance in industrially produced Q&P steels [146–148]. Steels created with slightly different Q&P processing parameters and otherwise similar chemistries and microstructures are evaluated to study how small process variations, inherent in large-scale mill environments, can influence austenite stability and tensile performance. Orientation dependence of TRIP with respect to the rolling direction is examined, with the hypothesis that crystallographic texture and local austenite stability variations within the sheet could impact TRIP.

5.3 Materials and Methods

Q&P steels were manufactured on commercial scale production equipment. The steels were designed to have ultimate tensile strengths of 980 and 1180 MPa. Both strength levels of steel were intercritically annealed to form approximately 20 pct intercritical ferrite. Variants of the same strength level (e.g., QP980-1.4-A and QP980-1.4-B) exhibited nearly identical prior austenite grain size. After intercritical annealing, samples of each strength level were subsequently processed by two distinct Q&P processing routes; the resulting variants are referred to hereafter as QP980-1.4-A, QP980-1.4-B, QP1180-1.6-A, and QP1180-1.6-B. The specific Q&P processing parameters used to create these steels are proprietary and correspondingly omitted from this paper.

Chemical composition analysis was performed using LECO chemical analysis and by chemical spectroscopy. As shown in Table 5.1, the steels contain similar amounts of carbon and silicon, while the QP1180 steels contain a greater amount of manganese. The A and B variants of each steel have slightly different austenite and ferrite volume fractions. Since ferrite, retained austenite, and martensite are the predominant phases in the microstructures, it is inferred that the martensite occupies the remainder of the sample volume not occupied by either ferrite or retained austenite.

<table>
<thead>
<tr>
<th>Steel</th>
<th>Thickness (mm)</th>
<th>C (wt pct)</th>
<th>Mn (wt pct)</th>
<th>Si (wt pct)</th>
<th>Austenite (vol pct)</th>
<th>Ferrite (vol pct)</th>
<th>Martensite (vol pct)</th>
</tr>
</thead>
<tbody>
<tr>
<td>QP980-1.4-A</td>
<td>1.4</td>
<td>0.201</td>
<td>1.93</td>
<td>1.62</td>
<td>11.3 ± 0.5</td>
<td>23.4 ± 3.0</td>
<td>balance</td>
</tr>
<tr>
<td>QP980-1.4-B</td>
<td>1.4</td>
<td>0.201</td>
<td>1.93</td>
<td>1.62</td>
<td>11.3 ± 0.2</td>
<td>19.6 ± 2.0</td>
<td>balance</td>
</tr>
<tr>
<td>QP1180-1.6-A</td>
<td>1.6</td>
<td>0.204</td>
<td>2.26</td>
<td>1.74</td>
<td>11.4 ± 0.4</td>
<td>21.8 ± 5.7</td>
<td>balance</td>
</tr>
<tr>
<td>QP1180-1.6-B</td>
<td>1.6</td>
<td>0.204</td>
<td>2.26</td>
<td>1.74</td>
<td>12.6 ± 0.2</td>
<td>16.7 ± 2.3</td>
<td>balance</td>
</tr>
</tbody>
</table>
Sheet-type tensile specimens, with geometries conforming to ASTM E8 [149], were removed from each material in orientations of 0, 45, and 90° with respect to the rolling direction. The geometry of the tensile specimens is shown schematically in Figure 5.1. Four replicates of each sample condition were strained at a rate of 0.05 per minute (8.3 × 10^{-4} s^{-1}) to failure. A screw-driven MTS Alliance RT/100 tensile frame, equipped with an MTS 20,000 lbf load cell and a two-inch extensometer, was used to collect load-displacement data at a data acquisition rate of 15 Hz. The true instantaneous strain hardening exponent (n_i) was evaluated from 1 pct engineering strain until the onset of necking using Equation 5.1:

\[ n_i = \frac{d(ln\sigma)}{d(ln\varepsilon)} \] (5.1)

where \(\sigma\) and \(\varepsilon\) are true stress and true strain, respectively. To smooth the \(n_i\) versus engineering strain curves, the \(n_i\) values were averaged over sixty adjacent \(n_i\) values.

Characterization of the Q&P microstructure was performed post-mortem in the unstrained grip section of tensile bars removed from the rolling direction. A section of each steel was mounted and polished, revealing the transverse plane. Samples were then etched with 1 pct Nital solution for 10 seconds. These metallographic samples were viewed optically and with an electron microscope.

Optical microscopy (OM) was performed using an Olympus PMG 3 inverted microscope. Polarizing optics were used to increase color contrast between phases, such that ferrite appeared blue while martensite appeared orange or brown. ImageJ color thresholding was used to measure the area fraction of blue ferrite phase existing on ten images (100 times magnification) of each steel. Scanning electron microscopy (SEM) was performed using an FEI Quanta 600 environmental scanning electron microscope (ESEM) in the secondary electron imaging mode.

Retained austenite volume fraction was determined using X-ray diffraction (XRD), by comparing the integrated intensities of diffracted peaks of ferrite and austenite, using the two-peak method outlined in ASTM E975 [82] and theoretical intensity data [150]. A Malvern PANalytical Empyrean diffractometer, equipped with a molybdenum (Mo) X-ray source and a zirconium (Zr) K\(\beta\)-filter, was used to perform scans over a 2\(\theta\) range of 10 to 150° to identify present phases, followed by higher-resolution scans over the 2\(\theta\) range of 21 to 37° to record the \(\alpha\)(200), \(\alpha\)(211), \(\gamma\)(200), and \(\gamma\)(220) peaks.

XRD scans were performed on the fractured tensile bars in the unstrained grip section and in the uniformly-elongated section. The tensile bars were polished to mirror finish, then subsequently electrochemically thinned to remove any surface deformation layer. Scans were taken along three orientations (Figure 5.1), parallel, diagonal, and perpendicular, to the tensile axis, to mitigate effects of microstructural texture on measured retained austenite volume fraction [81, 151]. Intensities of ferrite and
austenite peaks from each orientation were subsequently compared to compute the retained austenite volume fraction.

Figure 5.1 (a) Schematic of the tensile specimen geometry with units in millimeters. (b) Location and orientation of the XRD scans and SEM metallographic samples removed from the fractured tensile specimens.

5.4 Results

In this section, microscopy, tensile mechanical behavior, and the extent of the martensitic transformation will be reported for each steel.

5.4.1 Microscopy of Q&P Microstructures

Optical microscopy reveals a Q&P microstructure containing light-etching ferrite and dark-etching martensite and retained austenite (Figure 5.2). Polarized light reveals ferrite as teal blue and martensite and austenite as brown. The area of blue ferrite regions was measured for each steel, with estimated volume percent ferrite shown in Table 5.1. Each steel contains approximately 20 pct intercritical ferrite, with marginally greater amounts of ferrite existing in the “A” conditions for both strength levels. Variability of several volume percent existed between images of each steel (Table 5.1). Scanning electron microscopy reveals a fine microstructure consisting of multiple phases, like those reported in the literature [77, 152–154]. Based upon detailed characterization by Navarro-Lopez [154], it is speculated that the dark featureless regions are ferrite (F), blocky regions exhibiting fine internal laths surrounded by white blocks are martensite—austenite islands (MA), and elongated white regions are film-like retained austenite (RA). The microstructures of each steel appear similar (Figure 5.3).
Figure 5.2 Optical micrographs of QP980-1.4-A, (a) in black and white, (b) photographed with polarized light, revealing ferrite as teal blue and martensite and austenite as orange, and (c) color thresholding overlay (red). The region in this image contains 24.0 area percent ferrite, while the average of ten images revealed 23.4 area percent ferrite.

Figure 5.3 SEM images of (a) QP980-1.4-A, (b) QP980-1.4-B, (c) QP1180-1.6-A, and (d) QP1180-1.6-B containing intercritical ferrite (F), martensite-austenite (MA) microconstituents, and dispersed retained austenite (RA).
5.4.2 Tensile Deformation and Strain Hardening Behavior

Tensile specimens from three orientations (0, 45, and 90° with respect to the rolling direction) of each steel were strained to failure. In all four steels, roundhouse yielding occurs at a high yield stress, due to the presence of tempered martensite in the microstructure. Following yielding, the instantaneous strain hardening exponent reaches a minimum at an engineering strain of approximately 0.02, then increases as tensile strain is increased.

Engineering stress and true instantaneous strain hardening exponent, \( n_i \), are plotted versus engineering strain in Figure 5.4. Although four replicates of each condition were tested, only one representative replicate of each condition is plotted here, to allow for direct comparison between the tensile deformation response and austenite transformation for each tensile bar. In QP980-1.4-A and QP1180-1.6-B, the stress–strain behavior and \( n_i \) curves are consistent regardless of orientation (0, 45, or 90°).

Correspondingly, each orientation had a very similar uniform elongation. However, in QP980-1.4-B and QP1180-1.6-A, work hardening rates for different orientations diverge significantly above an engineering strain of 0.10. This divergence led to greater amounts of uniform and total elongation in the 0° orientation for both QP980-1.4-B and QP1180-1.6-A. In some cases, the minimum instantaneous strain hardening exponent was often distinctive for different orientations, with 0° orientations of QP980-1.4-A and QP980-1.4-B material having lower strain hardening coefficients at low amounts of strain.

Considering all four replicates, Table 5.2 presents a tabulated summary of ultimate tensile strength and uniform elongation along with the associated experimental uncertainties. The replicates exhibited very little variability; experimental errors were approximately 10 MPa for ultimate tensile strength and 0.5 pct for uniform elongation. For different orientations, there was an experimentally significant difference in the uniform elongation for QP980-1.4-A and ultimate tensile strength for QP980-1.4-B and QP1180-1.6-B.

Serrations in the strain hardening exponent curves were present to various degrees in every steel and orientation tested, particularly at higher amounts of strain. These serrations generally existed with an amplitude of 10 pct of the magnitude of the strain hardening exponent, but sometimes existed at a much larger amplitude. For example, large amplitude serrations were present in several cases for QP1180-1.6-B; large serrations were observed in zero of four 0° specimens, three of four 45° specimens, and two of four 90° specimens. Serrations in strain hardening curves detected in similar steel grades have been described as dynamic strain aging (DSA), caused due to interaction of diffusing solute atoms with dislocation cores [155–157]. De Cooman et al. documented such discontinuities, arising due to both TRIP and DSA, with the conclusion that Q&P greatly reduced the magnitude of DSA in comparison with conventional intercritical annealing treatments [157]. Based upon the greater number of large serrations in 45 and 90°
orientations of QP1180-1.6-B in comparison to 0°, it appears that microstructure orientation may influence DSA, because the amount of solute carbon should be identical for all three orientations.

Table 5.2 Summary of Tensile Performance and Martensitic Transformation Kinetics

<table>
<thead>
<tr>
<th>Steel</th>
<th>Orientation (deg)</th>
<th>UTS (MPa)</th>
<th>UE (pct)</th>
<th>RA_{ε=0} (vol pct)</th>
<th>RA_{ε=UE} (vol pct)</th>
<th>RA_{transformed} (vol pct)</th>
</tr>
</thead>
<tbody>
<tr>
<td>QP980-1.4-A</td>
<td>0</td>
<td>1017 ± 7</td>
<td>16.3 ± 0.4</td>
<td>11.8 ± 0.3</td>
<td>5.1 ± 0.5</td>
<td>6.7 ± 0.6</td>
</tr>
<tr>
<td></td>
<td>45</td>
<td>1015 ± 6</td>
<td>16.2 ± 0.2</td>
<td>11.2 ± 0.8</td>
<td>6.5 ± 0.9</td>
<td>4.8 ± 1.1</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>1028 ± 7</td>
<td>15.2 ± 0.1</td>
<td>10.9 ± 0.6</td>
<td>7.4 ± 0.9</td>
<td>3.5 ± 1.1</td>
</tr>
<tr>
<td>QP980-1.4-B</td>
<td>0</td>
<td>1028 ± 11</td>
<td>15.4 ± 0.8</td>
<td>11.4 ± 0.4</td>
<td>4.2 ± 0.7</td>
<td>7.2 ± 0.8</td>
</tr>
<tr>
<td></td>
<td>45</td>
<td>1008 ± 13</td>
<td>13.2 ± 0.1</td>
<td>11.4 ± 0.4</td>
<td>6.1 ± 0.8</td>
<td>5.3 ± 0.9</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>1047 ± 5</td>
<td>12.8 ± 0.1</td>
<td>11.1 ± 0.6</td>
<td>7.2 ± 0.5</td>
<td>3.9 ± 0.8</td>
</tr>
<tr>
<td>QP1180-1.6-A</td>
<td>0</td>
<td>1195 ± 7</td>
<td>13.5 ± 0.3</td>
<td>11.3 ± 0.7</td>
<td>5.3 ± 0.1</td>
<td>6.0 ± 0.7</td>
</tr>
<tr>
<td></td>
<td>45</td>
<td>1184 ± 6</td>
<td>13.1 ± 0.8</td>
<td>11.1 ± 0.5</td>
<td>5.4 ± 0.1</td>
<td>5.6 ± 0.5</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>1191 ± 6</td>
<td>12.4 ± 0.3</td>
<td>11.8 ± 0.6</td>
<td>4.9 ± 0.8</td>
<td>6.9 ± 1.0</td>
</tr>
<tr>
<td>QP1180-1.6-B</td>
<td>0</td>
<td>1225 ± 8</td>
<td>12.7 ± 0.2</td>
<td>12.9 ± 0.9</td>
<td>3.6 ± 0.7</td>
<td>9.3 ± 1.1</td>
</tr>
<tr>
<td></td>
<td>45</td>
<td>1182 ± 6</td>
<td>13.4 ± 0.4</td>
<td>12.4 ± 0.6</td>
<td>4.9 ± 0.0</td>
<td>7.5 ± 0.6</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>1190 ± 10</td>
<td>12.6 ± 0.1</td>
<td>12.6 ± 0.9</td>
<td>5.4 ± 0.6</td>
<td>7.2 ± 1.0</td>
</tr>
</tbody>
</table>

*Error is the std. dev. of tensile data from four replicate tensile specimens.
**Error is the std. dev. of RA in three separate orientations on one tensile specimen.
***Error is the propagation of uncertainty between RA_{ε=0} and RA_{ε=UE}.

5.4.3 Martensitic Transformation During Deformation

After straining to failure, XRD scans were performed to quantify retained austenite in both the unstrained grip section and in the uniformly-elongated gauge section. XRD scans for each steel and orientation are presented in Figure 5.5, with computed retained austenite superimposed on the curves. In the unstrained condition, austenite (200) and (220) peaks are easily distinguished. After straining to failure, the amplitude of austenite peaks is significantly diminished relative to the ferrite (200) and (211) peaks. A shoulder is present on the ferrite (211) peak in many conditions, due to inability to distinguish martensite and ferrite peaks because of the small difference in lattice spacings. Solute carbon has been shown to modify the shape of martensite peaks [158], suggesting that varied amounts of solute carbon in tempered and deformation-induced martensite could influence the shape of the peak shoulder. Slight peak broadening due to increased lattice strain was also observed after deformation, which may have smoothed the peak shoulders. Low-intensity peaks existing at 2θ values of 25.7° and 31.7° exist in all steels in both unstrained and strained conditions. These peaks are associated with the Mo-Kβ emission.
Figure 5.4 Engineering stress and true strain hardening exponent, $n_i$, plotted versus engineering strain for (a) QP980-1.4-A, (b) QP980-1.4-B, (c) QP1180-1.6-A, and (d) QP1180-1.6-B. Note that $n_i$ was calculated using true stress and true strain, according to Equation 5.1.
Figure 5.5 XRD scans of each tensile specimen in the unstrained grip section (labeled “Zero Strain”) and the uniformly-elongated gauge section after fracture for 0, 45, or 90° orientations of (a) QP980-1.4-A, (b) QP980-1.4-B, (c) QP1180-1.6-A, and (d) QP1180-1.6-B. The volume percent of retained austenite (RA), computed by the two-peak method, is superimposed on each scan.
The extent of DIMT is inferred by subtracting the retained austenite volume percent in the gauge from the retained austenite volume percent in the grip. For example, in the 0° orientation of QP980-1.4-A, the unstrained region contains 11.8 vol. pct retained austenite, while the gauge region contains 5.1 vol. pct retained austenite, suggesting that 6.7 vol. pct retained austenite transformed to martensite during uniform elongation. All four steels contained a similar amount of retained austenite prior to straining, on the order of 11 or 12 vol. pct (Table 5.2). However, the transformation exhibits significant variation for different orientations in the sheet. For QP980-1.4-A, QP980-1.4-B, and QP1180-1.6-B, the greatest amount of transformation occurred in the 0° orientation, while the least transformation occurred in the 90° orientation. Experimental errors associated with these measurements are presented in Table 5.2. Orientation dependence of the martensitic transformation was greater than the error for QP980-1.4-A and QP1180-1.6-B, but not for QP980-1.4-B. In the QP1180-1.6-A steel, the transformation was similar regardless of orientation; measured differences between the three orientations were well within the experimental error.

5.5 Discussion

It is understood that DIMT is instrumental for improving ductility and formability of AHSS [115, 159]. However, the data presented suggest that uniform elongation and UTS are not directly linked to total austenite content or the extent of DIMT in the Q&P steels investigated here. For example, although the QP980-1.4-A and QP980-1.4-B steels exhibited tensile strengths slightly greater than 980 MPa in all three orientations, highly variable amounts of DIMT occurred, ranging from 3.5 to 7.2 vol. pct, as shown in Figure 5.6(a). The 1180 MPa strength level exhibited near-identical behavior, except for a slightly greater amount of austenite transformation. Uniform elongation also showed no clear correlation with DIMT (Figure 5.6(b)). The discovery that DIMT is not directly proportional to mechanical performance complements the findings of De Moor et al. [137] and others [138–143]. Furthermore, these results suggest that the degree of progressive transformation of retained austenite to martensite critically influences the mechanical performance, as suggested by Mukherjee et al. [144].

In addition to the UTS and uniform elongation, the strain hardening exponent at the UTS was not directly related to the total martensitic transformation, as is shown in Figure 5.7(a). Meanwhile, the criterion for strain localization, \( \varepsilon = n_i \) [114], was clearly adequate for predicting the onset of necking, as is shown in Figure 5.7(b). Austenite stability models [16] are useful for explaining such behavior. Consider varying retained austenite stability in a Q&P microstructure. In a microstructure containing only unstable retained austenite, austenite will rapidly transform at low strains, so that at a high strain, TRIP will not provide adequate strain hardening to delay the onset of necking. Meanwhile, an optimal microstructure
containing a spectrum of austenite stabilities will contribute to strain hardening at the beginning of plastic deformation and effectively suppress necking [126]. In a microstructure containing exceedingly stable austenite, as was the case for all steels investigated here, some austenite did not transform, suggesting that it was not available to improve the ductility via the TRIP-mechanism. Because a substantial quantity of retained austenite remains after deformation, perhaps the uniform elongation could be further enhanced by tailoring austenite stability. Perhaps promoting additional carbide formation, either by modifying the heat treatment or reducing the Si content, could decrease austenite stability and increase the amount of austenite available for TRIP. Lower silicon grades processed with the non-isothermal partitioning process have been explored with some success [143, 159, 160].

Figure 5.6 (a) Ultimate tensile stress and (b) uniform elongation plotted versus retained austenite transformation to martensite (volume percent) during uniform elongation. All permutations of steel (orientation and heat treatment) for each strength level are plotted concurrently.

Comparing the transformation behavior among the experimental steels, it appears that some microstructures and orientations contained greater amounts of retained austenite that were available for TRIP at higher strains. These microstructures typically exhibited a strain hardening exponent curve which did not decrease in the final stages of deformation and generally reached a higher uniform elongation. Meanwhile, microstructures that contained less stable retained austenite experienced higher instantaneous strain hardening exponents at the beginning of plastic deformation, and a characteristic decrease in strain hardening near the UTS. Besides TRIP, the relative volume fractions and strain hardening characteristics of ferrite and martensite likely also influenced the strain hardening exponent in these steels. However, a categorical link between mechanical performance and ferrite and martensite volume fraction remains
elusive in these data, perhaps because the differences in volume fraction in each steel are only several volume percent. Findley et al. showed that the degree of martensite tempering, carbide precipitation, and carbon partitioning critically effects the small-strain work hardening behavior, and therefore impacts the tensile ductility [118]. Interestingly, significant variations in small-strain work hardening behavior were observed; for instance, QP980-1.4-A had a greater small-strain work hardening rate than QP980-1.4-B, perhaps due to differences in the degree of martensite tempering. Besides work hardening of martensite, multiphase microstructures containing ferrite and martensite exhibit very strong ferrite grain size sensitivity [161, 162], suggesting that small variations in intercritical ferrite grain size could influence work hardening behavior in Q&P steels. Although strain hardening was investigated in tension only, it follows that varied austenite stabilities due to microstructure orientation will affect formability. For materials with varied tensile properties along different orientations, we expect that formability specimen orientation will influence the shape of the forming limit diagram. The challenge of predicting deformation characteristics expands when considering non-monotonic deformation pathways (e.g., loading–unloading, bending– unbending, and stretching).

Figure 5.7 (a) Total fraction of retained austenite transformed to martensite and (b) tensile elongation plotted versus instantaneous strain hardening exponent, $n_i$, computed at the onset of necking.

To efficiently utilize TRIP behavior in Q&P microstructures, care must be taken to optimize both the stability and volume fraction of retained austenite. Tailoring the stability may involve designing microstructures that contain a spectrum of austenite stabilities, such that the austenite is available for transformation at all amounts of strain pertinent to sheet forming and crash scenarios. Furthermore, Q&P
steel must be characterized such that TRIP is predictable during forming, even while creating complex geometries, such as an automotive B-pillar [2, 67, 163], with varying strain rates, temperatures, and strain paths. This characterization should perhaps include interrupted mechanical testing [77] to elucidate the range of austenite stabilities that exist, formability testing [164] to clarify the role of strain state in TRIP phenomena, and elevated temperature testing [46], to quantify how retained austenite stability is influenced by die heating and processes, such as bake hardening, and perhaps deformation-induced heating during high-rate deformation [165–167].

5.6 Conclusion

Tension testing, optical and electron microscopy, and X-ray diffraction were used to analyze the role of martensitic transformation and strain hardening in the mechanical performance of various industrially-relevant quenched and partitioned C-Mn-Si steels. These results lead to the following conclusions:

1. In steels with nominally identical composition (e.g., QP980-1.4-A and QP980-1.4-B), relatively small variations in Q&P processing parameters influenced the transformation of austenite to martensite and the tensile properties.

2. In all steels, elevated instantaneous strain hardening exponents were achieved by TRIP at high amounts of plastic strain. In some cases, such as QP980-1.4-A and QP1180-1.6-B, the instantaneous strain hardening exponent was consistent regardless of the orientation with respect to the rolling direction. Meanwhile, the remaining steels showed differences as a function of orientation that may influence formability.

3. Neither the amount of retained austenite prior to straining nor the total amount of retained austenite transformed during straining was directly correlated to tensile mechanical performance. Meanwhile, in accordance with the instability criterion, the strain hardening rate at necking was directly correlated to uniform elongation. These data suggest that for optimizing formability, tailoring the stability of austenite near strain localization may be more effective than maximizing the volume fraction of austenite.

5.7 Acknowledgements

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CHAPTER 6
EXPERIMENT B: STRAIN RATE DEPENDENT DUCTILITY AND STRAIN HARDENING IN QP980-1.4-B

Modified from a paper published in Metallurgical and Materials Transactions A²

6.1 Abstract

Due to their high strength, formability and affordable cost, quenched and partitioned (Q&P) steels have shown the potential to reduce the mass of vehicles, thereby decreasing fuel consumption during service. Furthermore, because a lower mass of steel is used in each vehicle, energy consumption associated with the steelmaking process is also reduced. Q&P steels utilize the deformation-induced martensitic transformation (DIMT) of metastable retained austenite to enhance ductility and strain hardening. Accordingly, improvement of mechanical performance is contingent on the ability to precisely control the chemical and mechanical stability of austenite. Considering the multitude of factors that influence austenite stability, optimizing microstructures to delay necking or fracture is challenging, particularly as temperature and strain rate increase. Tensile tests of an intercritically annealed C-Mn-Si Q&P steel were performed over a range of strain rates ($10^{-4}$ to $10^{-1}$ $s^{-1}$) to evaluate effects on the DIMT and sheet tensile properties. As strain rates increased from $10^{-4}$ to $10^{-1}$ $s^{-1}$, the uniform elongation decreased from approximately 19 to 14 pct. This reduction in uniform elongation is associated with a decrease in the strain hardening exponent near the onset of strain localization. Based on experimental data from this study and review of previous research, it is postulated that the strengthening contribution of DIMT is controlled by competing effects of: (i) a decreasing chemical driving force for DIMT caused by deformation-induced heat accumulation at higher strain rates and (ii) an increasing number of martensite nucleation sites. This suggests that tailoring austenite stability for specific deformation conditions could enable further optimization of formability and vehicle crash behavior.

6.2 Introduction

Automobile manufacturers are seeking pathways to reduce the mass of new vehicles using design optimization and structural materials selection. This is important because the implementation of lightweighting technologies reduces the amount of energy used and greenhouse gases emitted during

²Reproduced under an open access license agreement, C.B. Finfrock, M.M. Thrun, D. Bhattacharya, T. Ballard, A.J. Clarke, K.D. Clarke, Strain Rate Dependent Ductility and Strain Hardening in Q&P Steels, Metallurgical and Materials Transactions A. 52A (2021) 928–942., see Appendix F.
manufacturing and driving of automobiles [168]. During manufacturing, reducing the mass of steel used in a vehicle can facilitate a proportional decrease in detrimental emissions generated by the steelmaking process (primarily from coke-making, blast furnace operation, and scrap reheating) [169]. In the use phase of the vehicle lifecycle, lightweighting can enhance driving efficiency by decreasing engine, gradient, braking, and rolling resistance losses; consequently, the potential efficiency gains associated with lightweighting are significant for conventional petroleum, hybrid electric, and all electric vehicles [168, 170, 171]. Considering a hypothetical mass reduction of 100 kg in a gasoline powered passenger car with a 200,000 km service life, Helms and Lambrecht reported a total energy savings of 23 GJ, leading to a savings of 700 liters of gasoline [168].

For sheet steels, alloy design for lightweighting has enabled mass reductions in two ways [169]. First, low-density steels containing high manganese and aluminum contents have been designed that reduce mass for a given thickness. Second, alloying and thermomechanical processing routes have been developed that create complex non-equilibrium microstructures with high strength and toughness. By enhancing the toughness and strength of a steel, the total sheet thickness can be reduced, with a corresponding decrease in vehicle mass.

In the context of emerging lightweighting strategies, automakers must maintain acceptable levels of passenger safety, sheet formability, and weldability. For instance, automobiles must retain the ability to absorb kinetic energy and prevent intrusion during a crash. By maximizing yield stress (YS) and ultimate tensile strength (UTS), crash performance can be preserved, despite a reduction in gauge thickness [172]. Meanwhile, fracture strain and strain hardening must be tailored to delay failures in bending and stamping operations. To meet these goals, third-generation advanced high strength steels (AHSS) offer improved toughness in comparison to first-generation steels at a lower cost than second-generation steels. Besides strength and formability, joinability of third-generation AHSS is an additional consideration for crash worthiness and vehicle durability; resistance spot welding parameter selection, dissimilar material joining, and liquid metal embrittlement susceptibility are areas of active research [173–176].

Quenched and partitioned (Q&P) steels, conceptualized by Speer et al. [27, 35, 110, 111], were designed to meet the strength and ductility targets of AHSS with relatively low alloy contents. The quenching and partitioning process creates a martensitic-based microstructure containing a controlled volume fraction of metastable retained austenite. During sheet forming, some or all of the austenite in the Q&P microstructure undergoes a deformation-induced martensitic transformation (DIMT). Additional DIMT may occur in the event of a vehicle crash, in cases when the austenite was not completely transformed to martensite during sheet forming and the crash-induced deformation conditions and austenite stability are suitable to facilitate additional transformation. The propensity for DIMT during a vehicle crash may also
depend on the susceptibility of the austenite to transform or decompose while subjected to various
temperatures during the automobile use phase (i.e., low temperatures during winter could induce the
transformation to martensite while high summer temperatures could decompose austenite to ferrite and
cementite). The DIMT causes the transformation-induced plasticity (TRIP) effect [177, 178], wherein the
volume expansion and distortions associated with the austenite to martensite transformation are
accommodated through plastic straining of the austenite surrounding the newly formed martensite
(Greenwood-Johnson effect [13]) and through selection of preferred martensite variants (Magee effect [12]).
The enhanced work hardening capacity due to DIMT is usually attributed to (a) increased dislocation
density in the austenite and (b) increased content of hard freshly formed martensite [42, 137], which
induces a composite-like strengthening effect due to the plastic incompatibility of martensite with the
surrounding softer matrix phases. Because the stability of retained austenite substantially influences
mechanical performance [16, 42, 119, 137], many studies intending to optimize alloying, heat treatment,
and resulting microstructure have been performed for Q&P steels [34, 68, 118, 146, 179, 180]. Deformation
factors such as strain state, strain rate, strain path, and temperature also affect the apparent stability of
austenite, which affect formability [54, 55, 71, 126, 181]. For example, for a medium manganese TRIP
steel, Wu et al. showed that strain path changes during forming can be manipulated to achieve a desired
retained austenite content and mechanical properties in stamped parts [182].

The deformation response of Q&P steels under nonlinear and dynamic loading conditions is of
paramount interest to the sheet forming community. This is because stamping operations and vehicle
 crashes generally impart deformation at high strain rates over complex pathways [183]. Typical ranges of
strain rates associated with sheet forming and vehicle crashes are approximately $10^{-1}$ to $10$ $s^{-1}$ and $10^2$ to
$10^3$ $s^{-1}$, respectively [58, 183, 184]. At rates in excess of $10^{-2}$ $s^{-1}$, deformation-induced heating is
significant in the workpiece [7, 9, 165, 185, 186], which is problematic because the driving force for DIMT is
intrinsically responsive to temperature [4, 26, 41, 107, 187, 188]. Furthermore, the number of shear band
intersections increases significantly as strain rates are increased from quasi-static to dynamic; this is
important because shear band intersections are considered to be the primary sites for martensite nucleation
during DIMT [15, 54, 55, 189, 190]. To inform models that forecast critical material performance, such as
surface strain distribution and the onset of necking, understanding DIMT in dynamic and nonisothermal
conditions is essential. However, at strain rates greater than 1 $s^{-1}$, mechanical testing becomes
increasingly challenging due to load frame limitations and ringing artifacts [184]. Accordingly, the test
matrix investigated in this paper was selected to resolve a range of strain rates from $10^{-4}$ $s^{-1}$, where
deformation-induced heating is negligible, to $10^{-1}$ $s^{-1}$, where heating is quasi-adiabatic. Additional testing
at dynamic strain rates (1 to $10^3$ $s^{-1}$) is warranted to fully evaluate the strain hardening and DIMT.
response of AHSS during vehicle crashes; see Chapter 8. With the goal of improving formability and final part properties and performance, this paper discusses changes in TRIP behavior and mechanical response as deformation rates are increased from quasi-static ($10^{-4} \text{ s}^{-1}$) to intermediate ($10^{-1} \text{ s}^{-1}$). Experimental data are presented for an intercritically annealed Q&P steel, where the microstructure contains intercritical ferrite with austenite and martensite typically found in Q&P microstructures.

### 6.3 Materials and Methods

A zinc-coated, 1.4 mm-thick 0.2C-1.9Mn-1.6Si (wt pct) steel was intercritically annealed to form intercritical ferrite before Q&P processing using commercial production equipment. The steel was designed to have an ultimate tensile strength of 980 MPa, leading to the designation “QP980-1.4-B”. A transverse section of the QP980-1.4-B was metallographically mounted, ground, polished, and etched with 1 pct Nital solution. A JEOL 7000 field emission scanning electron microscope (SEM) in the secondary electron imaging mode was used to reveal intercritical ferrite, retained austenite, and martensite in the microstructure (Figure 6.1).

![SEM micrograph of a transverse cross-section of QP980-1.4-B. Retained austenite blocks (RA), intercritical ferrite (F) and martensite-austenite microconstituent (MA) are visible in the microstructure.](image)

Figure 6.1 SEM micrograph of a transverse cross-section of QP980-1.4-B. Retained austenite blocks (RA), intercritical ferrite (F) and martensite-austenite microconstituent (MA) are visible in the microstructure.

The scale and morphology of the microstructure features appear consistent with previous characterization performed on QP980-1.4-B. In a previous study performed by the authors (see Chapter 3 and Chapter 5), quantitative optical and electron microscopy and image analysis revealed the microstructure contained approximately 20 vol pct intercritical ferrite and 12 vol pct retained austenite, with the remainder being martensite [104]. The retained austenite existed in both blocky and film type morphologies. The martensitic fraction of the microstructure was slightly tempered from the partitioning step of the Q&P thermal cycle. DIMT was observed to occur most completely when QP980-1.4-B was subjected to tensile strain along the rolling direction, rather than the transverse and diagonal directions.
This was primarily attributed to crystallographic texture in the ferrite, martensite, and austenite inherited from thermomechanical processing [104, 191]. Standard sheet-type tensile specimens conforming to ASTM E8 [149] were removed from the rolling direction of the QP980-1.4-B sheet. Interrupted and continuous tensile testing was performed using an MTS Alliance RT/100 load frame equipped with a two inch extensometer. Load-displacement data generated from each tensile test were normalized by the dimensions of each tensile specimen to compute true stress as a function of true strain, until the onset of strain localization. Ultimate tensile strength (UTS), 0.2 pct offset yield stress (YS), and uniform elongation (UE) were computed from engineering stress-strain data. Toughness was determined based upon the area under the engineering stress-strain curves from 0 pct strain until the onset of strain localization.

Variations in the strain hardening behavior of QP980-1.4-B were assessed by several numerical methods. Instantaneous strain hardening exponent \( (n_i) \) was calculated [114] after Equation 5.1, where \( \sigma \) and \( \varepsilon \) are true stress and true strain, respectively. In cases where \( n_i \) was plotted against true strain, the running average of \( n_i \) was computed with 50 adjacent values to smooth the curve. Average strain hardening exponent \( (\bar{n}) \) was computed as an average of \( n_i \) over an interval of strain, \( i.e., \bar{n}_{5-7.5} \) is the average of \( n_i \) from 5 to 7.5 pct true strain. To determine the various stages of strain hardening, the modified Crussard-Jaoul (C-J) analysis approach was used; in this method [192], changes in the slope of a \( \ln(d\sigma/d\varepsilon) \) vs. \( \ln\sigma \) plot can provide insights into the stages of strain hardening according to Equation 6.1:

\[
\frac{d(\sigma)}{d(\varepsilon)} = (1 - m)\ln(\sigma) - \ln(Cm)
\]  

where \( m \) is the C-J strain hardening exponent and \( C \) is a material constant.

For selected strain rates, surface temperature evolution during deformation was measured by thermocouples and infrared thermography (IRT). K-type thermocouples were spot welded at three arbitrary locations on each gauge section to measure surface temperature during tensile elongation. For IRT, after specimens were painted with a thin layer of matte black paint to mitigate reflections of IR light in the laboratory, a FLIR A325sc thermal camera was used to record a heatmap video of the tensile tests.

During interrupted tests at a strain rate of \( 10^{-4} \) \( s^{-1} \), the tensile specimens were removed from the tensile frame at increments of approximately 2 pct true strain. During each interruption, X-ray diffraction (XRD) was used to measure the phase volume fraction of austenite and martensite/ferrite in the center of the gauge length of the tensile specimen. The time elapsed between each interruption was on the order of 2 to 8 hours. Two scans were performed prior to plastic deformation: the first was performed on an as-received specimen (zero applied tensile strain); the second was performed on a specimen that was subjected to elastic loading to 50 MPa below the YS. For the monotonic testing, multiple samples were
strained until failure at strain rates of $10^{-4}$, $10^{-3}$, $10^{-2}$, and $10^{-1}$ $s^{-1}$. After fracture, XRD scans were performed in multiple locations on both the uniformly elongated gauge section and the grip section of two representative tensile specimens from each strain rate. Each XRD specimen was prepared by wet grinding, followed by polishing with diamond polishing compound to minimize the extent of grinding-induced deformation. As a result, XRD scans interrogated a region of the microstructure approximately 250 $\mu m$ beneath the zinc coating and 200 $\mu m$ beneath the steel surface. For interrupted and monotonic tests, XRD was performed using a PANalytical X'Pert and a PANalytical Empyrean, respectively. Both diffractometers were equipped with a Cu X-ray source. After reflection-mode scans were performed over a $2\theta$ range of 40 to 105 deg, the integrated intensities of austenite $\gamma(200)$, $\gamma(220)$, $\gamma(311)$, and ferrite $\alpha(200)$, $\alpha(211)$, and $\alpha(220)$ peaks were used to calculate the volume percent of austenite with the method outlined in ASTM E975 [76, 82] and in Chapter 3.4.

6.4 Results

6.4.1 Interrupted Tensile Testing

Interrupted tensile tests were performed at a quasi-static strain rate of $10^{-4}$ $s^{-1}$ to measure the transformation of austenite to martensite during deformation. The true stress-strain curves of QP980-1.4-B specimens loaded continuously and with interruptions for XRD analysis are plotted in Figure 6.2(a). First, the interrupted specimen was loaded to the nominal YS of QP980-1.4-B (750 MPa); loading to the YS led to the appearance of a slightly more pronounced yield point in the interrupted specimen relative to the continuously loaded specimen. Subsequently, the interrupted specimen was strained in increments of approximately 2 pct, until strain localization.

The interrupted and continuously loaded specimens exhibited similar ductility. The interrupted specimen exhibited rounded yield points from 0 to 6 pct true strain. Rounded yield points have been reported for Q&P steels during interrupted tests [193], and are morphologically consistent with the Haasen-Kelly effect [194, 195]. After 6 pct true strain, serrated yield point phenomena occurred during reloading. The serrated yield points could be caused by static strain aging, or pinning of mobile dislocations by interstitial carbon atmospheres [50]. Researchers have observed static strain aging in TRIP-assisted steels [156, 196–198]. Additionally, Portevin-Le Châtelier bands caused by dynamic strain aging have been observed at elevated temperatures (373 K to 523 K) by Min et al. for a Q&P steel with a similar microstructure to QP980-1.4-B [199]. Despite these reports, the discovery of yield point reemergence during consecutive incremental strains at room temperature is novel in the literature for Q&P steels. Because serrations consistent with strain aging appeared only after plastic tensile strains of several percent, it is theorized that static strain aging occurred in the strain-induced martensite. Strain-induced...
martensite likely has a solute carbon content sufficient to cause strain aging because: (a) the partitioning process increases the carbon content of the parent austenite above the bulk alloy composition and (b) the martensite has not been sufficiently tempered to form carbides. A recent study also revealed that that the average dislocation content in martensite formed by DIMT decreases with increasing strain amounts [200]; the reduced dislocation density in martensite might further explain the observed load drop or serrated yield point effect during interrupted tension tests. An extended discussion of the serrated yielding upon reloading is provided in Appendix A.

Figure 6.2 (a) True stress-strain curves for monotonic and interrupted tensile tests of QP980-1.4-B. During each interruption, the QP980-1.4-B specimen was removed from the tensile frame to measure the retained austenite content with XRD. (b) Retained austenite content (in volume percent) as a function of true strain, measured in interrupted tensile specimens. The retained austenite content diminished during tensile deformation. Regression analysis was performed to quantify the variability in XRD data (standard error = 0.42 vol pct).

To measure the extent of TRIP in the interrupted sample, retained austenite volume percent was measured using XRD at each strain increment. Shown in Figure 6.2(b), the transformation from austenite to martensite was exemplified by the reduction in retained austenite volume percent from approximately 10 to 5 pct at the end of uniform elongation. No DIMT was apparent during elastic loading; the volume fraction of retained austenite in the initial undeformed specimen was relatively similar to that in the elastically strained specimen. In contrast, measurable DIMT was detectable only after macroscopic yielding. Scatter in the retained austenite content of approximately one volume percent was evident, perhaps due to local variations in chemical composition and noise in the XRD scans. Despite the scatter, the presence of DIMT confirmed that the strain hardening of QP980-1.4-B was enhanced by the TRIP-effect. Furthermore, the DIMT response reported here was comparable to similar Q&P steels.
analyzed using synchrotron radiation by Abu-Fahra et al. [201] and Hu et al. [202]. In the same synchrotron-based investigation of a Medium-Mn steel, Abu-Fahra et al. observed that propagative instabilities (Portevin-Le Châtelier bands) led to inhomogeneous DIMT due to the nonuniform strain in the vicinity of the deformation bands [201]. For the present interrupted testing of QP980-1.4-B, the occurrence of serrated yielding suggests the brief progression of inhomogeneous deformation. Thus, based on the analysis by Abu-Fahra et al. [201], it is imperative to note that the authors of this study cannot rule out the possibility that DIMT progressed nonuniformly during serrated yielding. Further work is warranted to elucidate the effects of strain interruptions and strain aging on the extent and character of DIMT.

6.4.2 Deformation Response at Various Strain Rates

Figure 6.3 presents true stress-strain curves of QP980-1.4-B specimens continuously strained to failure at rates of $10^{-4}$, $10^{-3}$, $10^{-2}$, and $10^{-1}$ s$^{-1}$. Despite nearly identical roundhouse yielding behavior for specimens deformed at all strain rates, the specimens appeared to exhibit strain-rate dependent ductility; as strain rates were increased from $10^{-4}$ to $10^{-1}$ s$^{-1}$, the uniform elongation repeatably decreased from approximately 19 to 14 pct.

![Figure 6.3 True stress-strain response of QP980-1.4-B deformed at approximate strain rates of $10^{-4}$ s$^{-1}$ (blue), $10^{-3}$ s$^{-1}$ (green), $10^{-2}$ s$^{-1}$ (red) and $10^{-1}$ s$^{-1}$ (purple). Despite the comparable roundhouse yielding behavior of each specimen, increasing the strain rate significantly decreased the amount of uniform elongation.](image)

As a consequence of the instability criterion for uniaxial tensile tests, the onset of strain localization occurs when $n_i$ is equal to the true strain [203]. Accordingly, $n_i$ was computed from stress-strain data to assess how the strain rate dependent work hardening rate of QP980-1.4-B resulted in changes in uniform
elongation. In Figure 6.4, \( n_i \) is plotted as a function of true strain for each specimen. For all strain rates, \( n_i \) reached a minimum at approximately 2 pct true strain, then increased with additional strain. In the \( 10^{-4} \text{ s}^{-1} \) specimens, \( n_i \) continued to climb until failure. For the \( 10^{-3} \text{ s}^{-1} \) specimens, the shape of the curve was similar, although \( n_i \) was lower. Meanwhile, for the two higher strain rate tests (\( 10^{-2} \) and \( 10^{-1} \text{ s}^{-1} \)), \( n_i \) began to decrease at true strains above approximately 8 pct. It is notable that curves for \( 10^{-2} \) and \( 10^{-1} \text{ s}^{-1} \) appeared nearly identical. Because the higher strain rate specimens exhibited comparatively low strain hardening rates after approximately 8 pct true strain, those specimens formed necks at the lowest true strains, in agreement with the tensile instability criterion.

Figure 6.4 Instantaneous strain hardening exponent, \( n_i \), plotted as a function of true strain for every tensile specimen. Curves from every strain rate are plotted separately in (a through d) to enable comparison; curves from the labeled strain rate are plotted in color while curves from other strain rates are plotted in gray. For reference, the tensile instability criterion (\( \varepsilon = n_i \)) is also plotted.
Fundamental tensile properties, determined from the stress-strain behavior, are plotted as a function of strain rate in Figure 6.5. No clear trend is apparent between either UTS or YS and strain rate (Figure 6.5(a)); neither UTS nor YS appear to be strain rate sensitive in the tested strain rate regime. In Figure 6.5(b), $\bar{n}$ is plotted for increments of true strain from 2.5 pct until failure. For true strains from 2.5 to 10 pct, i.e., for $\bar{n}_{2.5-5}$, $\bar{n}_{5-7.5}$, and $\bar{n}_{7.5-10}$, the average strain hardening rate was marginally responsive to strain rate. However, at true strains above 10 pct, i.e., for $\bar{n}_{10-\text{failure}}$, the average strain hardening rate greatly diminished as the strain rate increased from $10^{-4}$ to $10^{-1}$ s$^{-1}$. The responses of UE and toughness to strain rate are denoted in Figure 6.5(c) and (d), respectively. Toughness was computed based on the area under the engineering stress-strain curve from the beginning of loading until the onset of necking. On average, increasing the strain rate from $10^{-4}$ to $10^{-1}$ s$^{-1}$ decreased UE by 29.9 relative percent and toughness by 31.2 relative percent. UE and toughness decreased by similar extents, because the flow stress of QP980-1.4-B was not strongly strain rate sensitive at any given strain value.

As shown in Figure 6.4 and Figure 6.5, the instantaneous strain hardening exponent changed as a function of both true strain and strain rate. To assess the stages of strain hardening at the lowest and highest rates, i.e., $10^{-4}$ and $10^{-1}$ s$^{-1}$, a modified C-J analysis was performed [192]. The linear regions of the $\ln(d\sigma/d\varepsilon)$ vs. $\ln\sigma$ curves in Figure 6.6 show that deformation at both strain rates induced multi-stage strain hardening behavior. While stages I, II, and III were present for both strain rates, stage IV was observed in the high-rate specimen only. Soares et al. and Skowronek et al. have provided speculation about origins of each strain hardening stage [204, 205]; stages I, II, and III are perhaps associated with the onset of plastic deformation near the yield stress, slip of mobile dislocations in intercritical ferrite, and strain accommodation by the TRIP-effect, respectively. Stage IV is associated with the exhaustion of the TRIP-effect and slip in the ferrite/bainite, martensite, and remaining retained austenite. The presence of stage IV hardening in the high-rate specimens suggests that DIMT was suppressed in the final stages of uniform straining, facilitating the rapid decrease in the instantaneous strain hardening exponent shown in Figure 6.4(d). Interrupted or in-situ testing aimed to resolve local strain partitioning and austenite phase volume fraction (see Salehiyan et al. [206]) would be useful to confirm if DIMT was suppressed during stage IV hardening.
Figure 6.5 (a) UTS and YS, (b) $\bar{n}$, (c) UE, and (d) toughness ($E = \int_0^\varepsilon \sigma \, d\varepsilon$) plotted as a function of engineering strain rate. $\bar{n}$, UE, and toughness appeared to be strain rate sensitive. The average strain hardening exponent at high strains ($\bar{n}_{10\text{-}\text{failure}}$) decreased at higher strain rates, facilitating a decrease in uniform elongation, in accordance with the tensile instability criterion.
Figure 6.6 \(\ln(\frac{d\sigma}{d\varepsilon})\) vs. \(\ln\sigma\) curves for the modified C-J analysis of representative specimens deformed at approximate strain rates of \(10^{-4}\) s\(^{-1}\) (blue solid line) and \(10^{-1}\) s\(^{-1}\) (purple dotted line). Four apparent stages of strain hardening are labelled (I, II, III, and IV).

### 6.4.3 Deformation-Induced Heating

Surface temperature was measured during tensile deformation for selected specimens to elucidate the contribution of deformation-induced heating to the strain hardening response of QP980-1.4-B. During the quasi-static tests (10\(^{-4}\) s\(^{-1}\)), the surface temperature remained within several degrees Celsius of room temperature. Although heat was necessarily generated by the mechanical work imparted during plastic deformation, the heat was allowed to dissipate into the air and grip fixtures during the approximately forty-minute tensile tests. Meanwhile, at strain rates of 10\(^{-3}\) s\(^{-1}\), Poling and Connolly et al. have shown that deformation-induced heating leads to temperature rises of approximately 15 °C [7, 207]; at this strain rate, only some of the deformation-induced heat was allowed to dissipate into the environment (i.e., the deformation was neither isothermal nor adiabatic). In this study, for the higher strain rate tests (10\(^{-2}\) and 10\(^{-1}\) s\(^{-1}\)), the tensile tests occurred over an average duration of 20 and 2 seconds, respectively. At such high rates of deformation, the surface temperature of the tensile specimens rose from room temperature to approximately 55 °C at the onset of localization. Surface temperature evolution for specimens deformed at of 10\(^{-2}\) and 10\(^{-1}\) s\(^{-1}\) is plotted as a function of true strain in Figure 6.7(a), which clearly indicates deformation-induced heating of specimens strained at high rates.

A simple model was applied to predict the temperature rise of a specimen deformed at a rate of 10\(^{-1}\) s\(^{-1}\), assuming no dissipation of deformation-induced heat (i.e., adiabatic conditions). For the purpose of the model, it was assumed that 90 pct of the mechanical work imparted on the tensile specimen was
available to evenly heat the specimen; expressed as a decimal, this term is known as the Taylor-Quinney coefficient [51]. The predicted temperature reached was computed [120] using Equation 6.2:

\[ T_{\text{adiabatic}} = T_{\text{RT}} + \frac{0.9 \int_0^\varepsilon \sigma \, d\varepsilon}{\rho C} \]  

(6.2)

where \( T_{\text{RT}} = 21.4 \, ^\circ\text{C}, C = 495 \, J/kg^\circ\text{C}, \rho = 7860 \, kg/m^3, \) and \( \int_0^\varepsilon \sigma \, d\varepsilon \) was computed empirically from the area under a representative tensile curve (10\(^{-1}\) s\(^{-1}\)) up to a given tensile strain and has units of MJ/m\(^3\). Note that temperature increases associated with the exothermic martensitic transformation are not incorporated into the model, but have been observed by Vázquez-Fernández et al. for austenitic stainless steels [208]. Vázquez-Fernández et al. reported that the extent of heat generation associated with the martensitic transformation can be incorporated into the model by changes in the Taylor-Quinney coefficient. For example, the Taylor-Quinney coefficient of a fully stable austenitic steel (AISI 316) was lower than a metastable austenitic steel (AISI 301), the latter of which presumably exhibited more DIMT during uniaxial tension testing and thus more deformation-induced heating [208]. Based on these findings, the study proved the necessity of simultaneous measurement of surface temperature and strain for validating deformation-induced heating models of steels containing metastable austenite. This experimental approach, coupled with \textit{in-situ} measurement of austenite volume fraction, would be highly useful for resolving DIMT and forming response under nonlinear strain paths, strain states including but not limited to uniaxial tension, and strain rates relevant to stamping (10\(^{-2}\) to 10\(^{-1}\) s\(^{-1}\)) and vehicle crashes (10\(^2\) to 10\(^3\) s\(^{-1}\)). For instance, such an approach could quantify the potential surface temperature inhomogeneity arising from the nonuniform deformation and DIMT reported by Abu-Fahra \textit{et al.} [201]. Furthermore, this approach would be useful for developing and validating damage-based models that incorporate the strain state, strain path, strain rate, stress triaxiality, Lode angle, and temperature dependence of DIMT for determining the damage locus (for example, see Kim \textit{et al.} [209]).

The temperature response projected by the adiabatic heating model is plotted alongside the actual thermocouple data in Figure 6.7(a). The model forecasted heating behavior that was nearly identical to the actual heating behavior for specimens deformed at the strain rates of 10\(^{-2}\) and 10\(^{-1}\) s\(^{-1}\); this equivalence suggests that both strain rates produced ostensibly adiabatic conditions. This finding was consistent with work by Clarke \textit{et al.}, who analyzed deformation-induced temperature rises both theoretically and experimentally over a range of strain rates and thicknesses for ferritic stainless steels [186]. Clarke \textit{et al.} determined that adiabatic conditions were reached more rapidly as specimen thickness increased; based on these findings, the onset of quasi-adiabatic conditions at a strain rate of 10\(^{-2}\) s\(^{-1}\) is realistic for the 1.4 mm-thick QP980-1.4-B investigated in this study. This suggests that for
strain rates higher than $10^{-2} \text{ s}^{-1}$, the temperature rise would not drastically change without a proportional shift in the stress-strain curve or DIMT.

In a previous paper [165], it was concluded that thermocouples were unreliable for measuring temperature evolution during post-uniform elongation, and IRT provided a robust method to measure the uneven heating that occurred after the onset of strain localization. Accordingly, IRT was used here to evaluate temperature rise in the region of the neck; a map of the surface temperature distribution for a specimen deformed to failure at a strain rate of $10^{-1} \text{ s}^{-1}$ is shown in Figure 6.7(b). Although the specimen reached a temperature of approximately 55 °C at the onset of localization, the neck subsequently reached a temperature of approximately 160 °C at fracture. The sharp rise in temperature was seemingly reasonable, because the region of the neck was subjected to drastically higher strain rates, effective strains, and true stresses than the rest of the tensile specimen. A thermal gradient adjacent to the two extensometer attachment pins was visible in the IRT data; this suggests that environmental conditions, such as geometry of the sheet-tool interface, may influence heat dissipation in deformation processing operations.

### 6.4.4 Deformation-Induced Transformation of Austenite to Martensite

XRD scans were performed to consider whether the reduction in strain hardening exponent at elevated strain rates could be attributed to a less dominant TRIP-effect. For a representative tensile specimen deformed at $10^{-3} \text{ s}^{-1}$, Figure 6.8(a) depicts example scan locations and XRD spectra used to calculate
retained austenite volume percent in the grip section (nominally zero strain) and the uniformly elongated
gauge section. Note that although only one scan is depicted in each region on the schematic, two scans
were performed in each region to account for possible local variations in austenite content. For each
QP980-1.4-B specimen strained at different rates, the integrated intensities of austenite peaks were
diminished in comparison to ferrite/martensite peaks, suggesting that austenite transformed to martensite
during deformation at all strain rates. The relative amounts of DIMT as a function of the applied strain
rate are shown in Figure 6.8(b). The greatest transformation of retained austenite occurred at the slowest
strain rate, suggesting that a greater volume fraction of retained austenite was available to facilitate TRIP.
Generally, higher strain rate specimens exhibited less DIMT. However, variability in local austenite content
prior to deformation caused some variability between replicates (see the error bars). Meanwhile, replicate
scans of the same region of the specimen exhibited nearly identical austenite contents, suggesting that
noise error from the XRD measurement technique was minimal.

Figure 6.8 (a) Representative XRD scans of QP980-1.4-B before deformation (zero strain) and after
uniform elongation at a strain rate of $10^{-3} \text{s}^{-1}$. (b) Relative amount of retained austenite transformed to
martensite during uniform elongation, plotted as a function of strain rate. Error bars are the standard
deviation from four XRD scans performed in the gauge sections of two representative tensile specimens
deformed at each strain rate.

6.4.5 Fractographic Analysis

The true stress-strain response of QP980-1.4-B presented in Figure 6.3 exemplified deformation
behavior consistent with AHSS of similar alloy content and processing. Furthermore, specimens deformed
at every strain rate exhibited significant post-uniform elongation (several percent engineering strain) prior
to fracture. Despite the exhibition of these principally ductile behaviors for all strain rates, specimens
deformed at higher strain rates generally exhibited reduced UE and toughness. In an attempt to identify the root-cause of the reduction in toughness, OM and SEM fractography was performed. Figure 6.9 shows representative OM and SEM images of the necks and fracture surfaces, respectively. At every strain rate, diffuse necking occurred prior to the formation of a localized neck. The shape and size of each diffuse neck appeared similar, with no evidence of a strain rate dependent shift in morphology. Viewed with SEM, the entirety of each fracture surface was decorated with ductile microvoids, suggesting that microvoid coalescence and growth preceded fracture during deformation at each strain rate. Watershed image analysis in previous work showed that dimple size and morphology did not change significantly between each strain rate [165], suggesting that a shift in microvoid accumulation in the microstructure was not a valid explanation for the reduction in toughness. After ruling out microvoid accumulation, a shift in the TRIP-effect is perhaps the most rational cause of the strain rate dependent ductility.

![Fractographs of QP980-1.4-B](image)

Figure 6.9 Fractographs of QP980-1.4-B deformed at rates of approximately (a) $10^{-4} \text{s}^{-1}$, (b) $10^{-2} \text{s}^{-1}$, and (c) $10^{-1} \text{s}^{-1}$. Viewed optically, specimens deformed at each strain rate exhibited macroscopically ductile diffuse and localized necking behavior. For scale of the inset images, the initial gauge width of each tensile specimen was 12.7 mm. Viewed at higher magnification with SEM, ductile microvoids were evident on the fracture surface of each specimen.

6.5 Discussion

6.5.1 Strain Rate Sensitive TRIP-Effect in QP980-1.4-B

Deformation-induced heat accumulation in tensile samples deformed at $10^{-2}$ and $10^{-1} \text{s}^{-1}$ led to a rise in surface temperature to approximately 53 and 160 °C at UTS and final fracture in the neck, respectively. Meanwhile, for samples deformed at $10^{-4} \text{s}^{-1}$, the surface temperature did not increase measurably. In a previous study of QP980-1.4-B [165], it was hypothesized that for elevated strain rate conditions, deformation-induced heat accumulation would increase the stability of the retained austenite, reduce the work hardening rate, and facilitate early necking. The DMT and strain hardening data presented here support this hypothesis; as the samples heated quasi-adiabatically, the TRIP mechanism became less active.
and the average strain hardening rate near failure (\(\bar{n}_{10-failure}\)) was reduced. However, at small strains (\(\bar{n}_{2.5-5}\) and \(\bar{n}_{5-7.5}\)) before deformation-induced heating significantly increased specimen temperature, the average strain hardening rate appeared to be relatively constant. This suggests that examining the entire instantaneous strain hardening exponent curve is necessary for assessing the mechanisms that dictate the extent of DIMT. Based on the data presented here, it is apparent that strain rate sensitivity of the TRIP-effect may facilitate substantial shifts in the work hardening behavior and consequently the toughness.

6.5.2 Mechanical Performance and DIMT in TRIP-Assisted AHSS

To contextualize the behavior of QP980-1.4-B, a literature review pertaining to the role of temperature and strain rate on TRIP and mechanical performance is justified. Generally, elevated specimen temperature increases strain rate sensitivity and decreases the work hardening rate and flow stress \([114, 203]\). Additionally, several mechanisms for temperature dependence of TRIP have been discussed in this paper. First, the chemical driving force (\(\Delta G^{\gamma\rightarrow\alpha}\)) for the austenite to martensite transformation decreases as specimen temperature rises \([4, 26, 41, 187, 188]\). Second, stacking fault energy is temperature dependent, which can control the number of shear band intersections (the primary nucleation sites of martensite) that form during deformation \([15, 54, 55, 189, 190]\). Hecker et al. demonstrated the contribution of both mechanisms in an austenitic stainless steel, by showing that the number of shear band intersections increased at higher strain rates, but the lowered chemical driving force for martensitic transformation reduced the transformation rate as deformation-induced heat accumulated in the specimens \([54, 55]\). Similarly, for a Q&P steel deformed isothermally at a quasi-static rate, Coryell et al. showed that DIMT reached a minimum at temperatures of approximately 150 °C, leading to a 30 pct reduction in tensile elongation \([46]\). The minimization of the TRIP-effect led to more predominant slip. The study also noted that austenite stability could be influenced by additional mechanisms during prolonged exposure to elevated temperatures, including additional carbon partitioning and bake hardening.

As was shown in this study and by others \([7, 186, 207, 210]\), for quasi-static to intermediate strain rates, increasing the strain rate increases the temperature reached during deformation. This is because the deformation-induced heat is given less time to dissipate during shorter duration mechanical tests \([186]\). However, there is a critical strain rate beyond which heating is quasi-adiabatic; in such conditions, virtually no heat dissipates into the environment. Zou et al. concluded that the adiabatic critical strain rate was reached for a 1.2 mm-thick Q&P steel around \(10^{-1} \text{ s}^{-1}\); this threshold strain rate was incorporated into a DIMT model with reasonable agreement with experimental data \([61]\). Here, for a 1.4 mm-thick sheet, the adiabatic critical strain rate was approximately \(10^{-2} \text{ s}^{-1}\). As discussed previously, specimen heating is also
influenced by the extent of the exothermic martensite transformation [211]. Therefore, deformation processing factors that influence DIMT (such as strain rate and strain state), could influence the Taylor-Quinney coefficient and the flow behavior, thereby controlling the temperature rise under adiabatic conditions. In cold stamping operations, several factors could further influence heat flow and consequently control DIMT. First, the nature of the sheet-tool interface could influence the flow of heat away from the specimen; in cases where the dies are cold, heat flow out of the specimen could be enhanced significantly. However, repeated stampings often cause dies to heat to above room temperature, potentially mitigating this effect. Besides heat flow from the workpiece to the dies, frictional heating at the sheet-tool interface could also manipulate the temperature of the sheet.

Based on Figure 6.4, Figure 6.5, and Figure 6.8, trends in ductility, strain hardening and DIMT appear to saturate at the adiabatic critical strain rate \(10^{-2} \text{ s}^{-1}\), for monotonically loaded QP980-1.4-B. However, because no tensile tests were performed at strain rates greater than \(10^{-1} \text{ s}^{-1}\), it is unclear if these trends would further evolve for QP980-1.4-B deformed at higher strain rates. The summary of literature reported below aims to resolve some potential shifts in strain hardening and DIMT for TRIP-assisted AHSS, including for rates greater than \(10^{-1} \text{ s}^{-1}\).

A summary of the strain rate dependence of DIMT and mechanical response for a variety of TRIP-assisted AHSS is provided in Table 6.1 and Table 6.2, respectively. For Q&P, TRIP, Medium-Mn, and austenitic and duplex stainless steels, many authors reported that increasing strain rates from quasi-static (\(\sim 10^{-4} \text{ s}^{-1}\)) to intermediate (\(\sim 10^{-1} \text{ s}^{-1}\)) suppressed DIMT, partially due to adiabatic heating. For example, Enloe et al. and Savic et al. reported strain rate dependent work hardening, elongation, and strength in TRIP and Q&P steels, respectively; in both studies, thermal stabilization of austenite and modified dislocation slip phenomena due to elevated strain rates exhibited potentially counteracting effects on the formation of martensite [56, 57]. Furthermore, in a study that incorporated synchrotron diffraction experiments and crystal plasticity modeling of isothermal and adiabatic conditions, Connolly et al. concluded that although temperature rise partially suppressed DIMT at intermediate strain rates (\(8 \times 10^{-2} \text{ s}^{-1}\)), the strain rate dependence of DIMT could not be attributed to temperature rise alone [207]. In some studies of Q&P and TRIP steels, DIMT did not vary significantly during deformation at quasi-static or dynamic rates [7, 58-60]. In general, for Q&P steel microstructures, the mechanical stability of austenite is high and austenite volume fractions are relatively small, so perhaps a change in transformation behavior occurred, but was not easily resolvable; accurate measurement of austenite fractions in these steels remains challenging [81, 83]. Another potential source of variability between studies arises due to evaluation of different austenitic microstructures; for example, austenite morphology and grain size can greatly affect the extent of DIMT because of the differences in alloy content, proximity
to lath martensite and intercritical ferrite, and dislocation density of the lath martensite [123, 130].
Although increasing strain rate from quasi-static to intermediate rates generally suppressed or did not drastically alter DIMT, a different behavior arose at strain rates above the adiabatic critical strain rate. For Q&P steels, Zou et al. and Wang et al. described an acceleration in DIMT at strain rates above the adiabatic threshold, likely due to an increase in the number of martensite nucleation sites [61, 62].

The TRIP-effect theory postulates that DIMT delays necking by locally increasing the strain hardening rate [177, 178]. If DIMT is suppressed at a given strain rate, then it is reasonable that the TRIP-effect will be less active. Therefore, the strain hardening rate will be decreased. This behavior was observed here for a Q&P steel deformed at quasi-static to intermediate rates. It is notable that a trough in ductility was reported at intermediate strain rates for multiple types of AHSS, including Q&P steels [7, 212]; the root cause is possibly related to a decreasing chemical driving force (due to adiabatic heating) and increasing mechanical driving force (due to larger stresses) for DIMT as strain rates increase. In addition to the TRIP-effect, some recent studies have shown that strain rate dependence of dislocation density in martensite and austenite affect strain hardening. For example, Hao et al. suggested that dislocation absorption by retained austenite (DARA [213]) is prominent at quasi-static rates and suppressed at dynamic rates [200]. Meanwhile, Wang et al. reported that despite more complete DIMT at dynamic strain rates, lower dislocation density in martensite reduced the work hardening rate [62]. Elucidating the root cause(s) of the trough in ductility should be of significant interest to the stamping community, because the trough is potentially problematic for predicting deformation behavior in stamping operations and vehicle crashes. Characterizing the ductility trough could allow stamping parameters to be tailored for specific material attributes, such as thickness and microstructure; for example, if a minimum ductility is reached at a strain rate of $10^{-1}$ s$^{-1}$, then press displacement rates could be selected to exceed this minimum strain rate in critical regions (i.e., areas of high strain), thereby avoiding the ductility loss. Strain path and strain state dependence of the ductility trough should also be explored; for instance, it is currently unclear if the ductility troughs reported in literature for tension also exist in other strain states (e.g., plane strain and balanced biaxial tension), where necking criteria and DIMT are different.

Several studies have reported that the dependence of mechanical behavior on strain rate was contingent on the temperature of the specimen before deformation. For a Q&P steel, Frint et al. suggested that strain rate sensitive ductility was more pronounced at 200 °C than at room temperature [85]. In addition to dislocation slip, dislocation recovery, and DIMT, interface plasticity was proposed as a relevant mechanism at elevated temperatures. Recently, a greater understanding of interface plasticity in AHSS microstructures has been reached [86, 87], but has not yet been satisfactorily applied in formability studies.
Table 6.1 Strain Rate Dependence of DIMT in TRIP-Assisted AHSS (⋆ = This Study)

<table>
<thead>
<tr>
<th>Effect of Increasing Strain Rate on DIMT</th>
<th>Q&amp;P</th>
<th>TRIP</th>
<th>Medium-Mn</th>
<th>Austenitic Stainless</th>
</tr>
</thead>
<tbody>
<tr>
<td>DIMT was suppressed by increasing strain rate</td>
<td>⋆ [61, 207, 212]</td>
<td>[189, 214]</td>
<td>[7, 90, 210]</td>
<td>[54, 55, 190, 208, 215, 216]</td>
</tr>
<tr>
<td>DIMT did not vary significantly</td>
<td>[7, 58, 59, 217]</td>
<td>60, 218</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>DIMT was accelerated at dynamic strain rates</td>
<td>[61, 62, 212]</td>
<td>56</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Strain rate effects on DIMT were observed at low strains (prior to strong deformation-induced heat accumulation)</td>
<td>[61, 62]</td>
<td>56, 218</td>
<td>—</td>
<td>[54, 190, 208, 215]</td>
</tr>
</tbody>
</table>

Table 6.2 Strain Rate Dependence of the Tensile Response of TRIP-Assisted AHSS (⋆ = This Study)

<table>
<thead>
<tr>
<th>Effect of Increasing Strain Rate on Mechanical Performance</th>
<th>Q&amp;P</th>
<th>TRIP</th>
<th>Medium-Mn</th>
<th>Austenitic Stainless</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reduced work hardening rate at high strains</td>
<td>⋆ [57]</td>
<td>214, 219</td>
<td>[7, 184, 210]</td>
<td>[120, 190, 220]</td>
</tr>
<tr>
<td>Decrease in tensile elongation with increase in strain rate from quasi-static to intermediate. No high rate samples were tested.</td>
<td>⋆ [59]</td>
<td>—</td>
<td>—</td>
<td>[220]</td>
</tr>
<tr>
<td>As the strain rate was increased from quasi-static to intermediate, tensile elongation decreased. As strain rate increased from intermediate to dynamic, elongation increased, forming a ductility trough.</td>
<td>[7, 212]</td>
<td>60, 214, 218</td>
<td>[7, 90, 184]</td>
<td>[190, 215, 223]</td>
</tr>
<tr>
<td>Strain rate dependence was coupled with pre-deformation specimen temperature.</td>
<td>[85]</td>
<td>[224]</td>
<td>[7, 210]</td>
<td>[225]</td>
</tr>
<tr>
<td>Strain rate sensitivity was low at quasi-static to intermediate rates but increased significantly at dynamic rates.</td>
<td>[7, 181]</td>
<td>218, 222, 226</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Necking behavior changed</td>
<td>[61, 217]</td>
<td>—</td>
<td>[90]</td>
<td>—</td>
</tr>
</tbody>
</table>
6.5.3 Recommendations for Future Research

The results presented in this paper suggest that controlling austenite stability at various strain rates is critical for optimizing flow behavior in forming operations and the properties and performance of finished parts. Therefore, understanding how aspects of Q&P microstructures control the TRIP-effect could greatly improve the viability of Q&P steels. To reach a deeper understanding, the following are recommendations for areas of concentration in future research:

1. *Investigate the mechanism(s) that control the trough in ductility.*

   Despite extensive reports of a trough in ductility at intermediate rates for many TRIP-assisted AHSS (see Figure 6.5(c), for example), a mechanism for this behavior has not been fully established. It is theorized that the ductility trough may be caused by a competition between a reduced chemical driving force, an increasing number of martensite nucleation sites, and an increasing mechanical driving force for DIMT. Decoupling the influences of strain rate and strain-induced heat accumulation deserves some attention (see Chapter 7); for example, for a metastable austenitic stainless steel, Vázquez-Fernández et al. concluded that strain rate likely affects the extent of DIMT, perhaps by increasing the stress required for a partial dislocation to glide in a shear band [208]. The rapid development of *in-situ* phase volume fraction analysis capabilities may also be useful to clarify the role of DIMT and microstructure damage accumulation on strain hardening and tensile elongation [62].

2. *Expand test matrices to include more strain states, strain rates, and temperatures.*

   Several recent studies evaluated forming performance of Q&P steels by creating forming limit diagrams [228, 229]. Of particular interest, Liu et al. reported that the formability was reduced as the strain rate increased from $10^{-2}$ to $1 \text{s}^{-1}$ [230]. Three additional studies attempted to link formability to the stability of retained austenite for industrially relevant stamping geometries [74, 126, 182]. Chiriac et al. stamped B-pillar components using third-generation TRIP-assisted AHSS with similar chemical composition and UTS, but different phase volume fractions, austenite stabilities, and strain hardening behaviors; austenite stability appeared to control surface strain distribution at low to medium strains and strain localization at high strains [126].

   Despite these noteworthy contributions, additional testing is needed to elucidate the connection between DIMT and formability for a greater number of strain states, strain rates, and temperatures. Path sensitive deformation characteristics, such as the Bauschinger effect, should also be explored.

3. *Incorporate the strain rate dependence of DIMT into formability and crash models.*
The strain rate dependent DIMT and ductility of TRIP-assisted AHSS is not easily compatible with existing constitutive models for deformation at dynamic rates [231]. Because the TRIP-effect profoundly influences flow behavior in these steels, developing reasonable empirical models for DIMT that incorporate strain state, strain rate, and temperature rise during non-isothermal deformation will be crucial for informing microstructure design [7, 232–234]. Currently, modeling the role of the TRIP-effect on plasticity and fracture is of significant interest for optimizing microstructure design and predicting forming limits and crash performance [232–234]. However, these models have not yet incorporated the strain rate sensitivity of DIMT.

6.6 Conclusions

For a quenched and partitioned third-generation advanced high strength steel (QP980-1.4-B), these experiments demonstrated the necessity of concurrent strain, surface temperature, and retained austenite volume fraction measurements during formability testing. Based on characterization of the tensile stress-strain behavior, deformation-induced heating, and progression of the martensite transformation for QP980-1.4-B, the following conclusions can be made:

1. The transformation of retained austenite to martensite was observed during tensile deformation of QP980-1.4-B at every strain rate tested ($10^{-4}$ to $10^{-1}$ s$^{-1}$). DIMT was most pronounced at the slowest strain rate tested ($10^{-4}$ s$^{-1}$), likely due to a lack of deformation-induced heat accumulation.

2. At the two higher strain rates ($10^{-2}$ and $10^{-1}$ s$^{-1}$), the instantaneous strain hardening rate decreased dramatically near the UTS, resulting in a relative 30 pct reduction in uniform elongation and toughness. Based upon modified C-J analysis and surface temperature measurements, this was attributed to the suppression of DIMT by quasi-adiabatic heating. No evidence of a change in macroscopic or microscopic fracture behavior was observed.

3. The mechanical response and DIMT behavior for the QP980-1.4-B was reported and compared to other TRIP-assisted AHSS results in the literature. Suppression of the TRIP-effect by deformation-induced heating is well documented as strain rates are increased from low ($10^{-4}$ s$^{-1}$) to intermediate ($10^{-1}$ s$^{-1}$), although mechanistic explanations are still unclear. At high strain rates (greater than $\sim10$ s$^{-1}$), the TRIP-effect is increasingly active, despite quasi-adiabatic heating conditions, perhaps due to an increase in the number of martensite nucleation sites. Correspondingly, a trough in ductility at intermediate strain rates is reported for a broad variety of AHSS.
6.7 Acknowledgements

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6.8 Author Contributions

CF: conceptualization, formal analysis, investigation, writing - original draft, visualization. MT: investigation, writing - review and editing. DB: writing - review and editing, visualization. TB: investigation. AC: conceptualization, writing - review and editing, supervision. KC: conceptualization, writing - review and editing, supervision.
CHAPTER 7
EXPERIMENT C: DECOUPLING THE IMPACTS OF STRAIN RATE AND TEMPERATURE ON TRIP IN QP980-1.4-A


7.1 Abstract

The individual effects of strain rate and temperature on the strain hardening rate of a quenched and partitioned steel were examined. During quasi-static tests, resistive heating was used to simulate the deformation-induced heating that occurs during high strain rate deformation, while the deformation-induced martensitic transformation was tracked by a combination of X-ray and electron backscatter diffraction. Unique work hardening rates under various thermal-mechanical conditions are discussed, based on the balance between the concurrent dislocation slip and transformation-induced plasticity deformation mechanisms. The diffraction and strain hardening data suggested that the imposed strain rate and temperature exhibited dissonant influences on the martensitic phase transformation. Increasing the strain rate appeared to enhance the martensitic transformation, while increasing the temperature suppressed the martensitic transformation.

7.2 Introduction

Third-generation advanced high strength steels (3GAHSS) demonstrate excellent mechanical property combinations, such as ductility and strength, by leveraging multiple deformation mechanisms. In quenched and partitioned (Q&P) steels, deformation is accommodated by dislocation slip in ferrite, martensite, and austenite, as well as by volume expansion and distortion associated with the Transformation-Induced Plasticity (TRIP) effect in austenite. The TRIP effect is activated upon the transformation of metastable austenite to martensite during plastic deformation. The TRIP effect enhances the strain hardening rate by increasing the dislocation density in the untransformed austenite, while simultaneously increasing the volume fraction of the strong martensite. The balance of dislocation slip versus TRIP is controlled by characteristics of the microstructure (such as crystallographic texture, grain size, morphology, dislocation content, and alloy content), as well as by the imposed deformation conditions (such as the strain state, strain rate, and temperature) \([104, 235]\).

\(^3\)Reproduced under an open access license agreement, C.B. Finfrock, D. Bhattacharya, B.N.L. McBride, T.J. Ballard, A.J. Clarke, and K.D. Clarke, Decoupling the Impacts of Strain Rate and Temperature on TRIP in a Q&P Steel, JOM, 74(2) (2022) pp. 506-512., see Appendix F and reference [167].
During deformation in processing (e.g., sheet forming) and service (e.g., automobile crashes), the strain rates imposed upon Q&P steels can vary from quasi-static ($\sim 10^{-4} \text{ s}^{-1}$) to dynamic ($\sim 10^3 \text{ s}^{-1}$). For high strain rate deformation, there is an increased number of shear band intersections in austenite, which correspondingly increases the number of possible martensite nucleation sites [54, 55]. Concurrently, as strain rates increase, the heat generated during plastic deformation is allowed less time to dissipate from the steel; at high enough strain rates, sheet temperatures rise adiabatically [51]. Rising temperatures decrease the chemical driving force for the martensitic transformation, and thus the likelihood that martensite will nucleate [8, 46]. Strain partitioning between the phases within a Q&P steel may also be strain rate sensitive; because short range barriers to dislocation motion are more difficult to overcome in body centered cubic (BCC) versus face centered cubic (FCC) crystals [50], it is expected that the flow stresses of martensite and ferrite will increase as strain rates increase, leading to enhanced plasticity in austenite.

We have previously shown that increasing the strain rate from $10^{-4}$ to $10^{-1} \text{ s}^{-1}$ deleteriously decreased the strain hardening rate and ductility of a Q&P steel (see Chapter 6). It was hypothesized that a ductility trough existed at intermediate strain rates (approximately $10^{-1} \text{ s}^{-1}$), which was caused by the competing effects of adiabatic heating and strain rate on the deformation induced martensitic transformation. The experiment was limited in two ways. First, no interrupted tests were performed, so it was difficult to resolve the extent of the martensitic transformation as a function of the imposed true strain ($\varepsilon$). Second, temperature and strain rate are intrinsically physically coupled, so it was difficult to determine whether temperature, strain rate, or a combination of both variables, affected the work hardening behavior of the Q&P steel. In most high strain rate experiments, temperature is viewed as a response variable that is impossible to decouple from strain rate. However, several methods have been implemented to decouple the effects, including isothermal bath testing, strain rate jump testing, and thermal-mechanical simulation. Rana et al. performed tensile tests of a Medium-Mn steel at various strain rates in cooled ethanol and heated oil baths [210]. The heated and cooled baths accelerated the heat transfer between the tensile specimen and the environment, thereby reducing the effects of deformation induced heating on the mechanical properties. However, the temperature change could not be fully eliminated for the strain rates and temperatures of interest in the study, so strain rate and temperature effects were not fully decoupled. Isakov et al. performed strain rate jump tests to evaluate the strain rate sensitivity of the martensitic transformation in a metastable austenitic stainless steel [236] By deforming the steel to a predetermined strain at a quasi-static rate, then rapidly increasing the strain rate, the strain rate dependence of the martensitic transformation was evaluated without a significant temperature rise; in this case, measurements of the martensite fraction suggested that increasing the strain rate partially suppressed the martensitic transformation. One drawback was that the influence of strain rate history, and thus any strain
rate dependent microstructure evolution, was not resolved.

Thermal-mechanical simulation has emerged as a more promising approach; by simulating the deformation-induced temperature rise during quasi-static testing, thermal conditions can be held equivalent between tests with drastically different strain rates. Using this approach, Vázquez-Fernández et al. removed the influence of heat accumulation and showed that the martensitic transformation rates decreased under increasing strain rates for a metastable austenitic stainless steel [208]. Strain rate and temperature effects have yet to be decoupled for 3GAHSS with lean alloy additions. In this paper, thermal-mechanical simulations will be implemented to decouple the roles of temperature and strain rate in dictating the balance of dislocation slip and TRIP in a Q&P steel. Based on the thermal-mechanical simulations and complementary microstructure characterization, insights will be provided for tailoring the stability of retained austenite for enhanced performance over a range of strain rates.

7.3 Materials and Method

A commercially produced 1.4 mm-thick 0.2C-1.9Mn-1.6Si (all in weight percent) steel was intercritically annealed followed by Q&P processing. In a previous study using scanning electron microscopy (SEM), the microstructure was revealed to contain ∼20 volume percent ferrite formed by intercritical annealing, ∼70 volume percent martensite, and ∼12 volume percent retained austenite (see Chapter 5). During deformation under uniaxial tension at a quasi-static strain rate of $10^{-4}$ s$^{-1}$, the steel achieved an ultimate tensile strength greater than 980 MPa and a uniform elongation of 19 percent. Accordingly, the steel is referred to as “QP980-1.4-A” hereafter. QP980-1.4-A was revealed to exhibit strain rate sensitive ductility and strain hardening; as the strain rate was increased to $10^{-1}$ s$^{-1}$, the uniform elongation decreased to 14 percent [166]. This study aims to further investigate the strain rate sensitive ductility.

Tensile specimens with a gauge length of 25.4 mm and a width of 12.7 mm were machined parallel to the rolling direction of the sheet. Thermal-mechanical simulation was performed in a Dynamic Systems, Inc. Gleeble 3500 equipped with closed-loop control direct resistance heating and servo hydraulic actuation. Temperature was monitored using a K-type thermocouple welded to the center of every sample, while actuator displacement was used to compute strain. The springs-in-series reciprocal addition relationship was used to correct for elastic compliance of the hydraulic actuator. The compliance correction was based on an assumed elastic modulus of the steel samples (200 GPa), the initial dimensions of the tensile samples, and the assumption that the actuator and the samples initially deformed elastically in accordance with Hooke’s law. A description of the compliance correction process is provided in Appendix D.

Three thermal-mechanical conditions were investigated for the samples that were monotonically loaded until fracture. Two conditions were tested without external heating at strain rates of $10^{-4}$ and $10^{-1}$ s$^{-1}$. 

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The third condition was tested with external heating at $10^{-4}$ s$^{-1}$, where the thermal profile as a function of strain was programmed to mimic the deformation induced heat rise of the $10^{-1}$ s$^{-1}$ condition; a schematic of a tensile specimen subjected to the resistance heating is shown in Figure 7.1(a). The test conditions are referred to by their strain rate and thermal condition: $10^{-4}$ s$^{-1}$ (isothermal), $10^{-1}$ s$^{-1}$ (adiabatic), and $10^{-4}$ s$^{-1}$ (resistance heated). Comparing the $10^{-4}$ s$^{-1}$ (isothermal) and $10^{-4}$ s$^{-1}$ (resistance heated) samples will give an indication of effect of temperature rise on TRIP. Meanwhile, comparing the $10^{-4}$ s$^{-1}$ (resistance heated) and $10^{-1}$ s$^{-1}$ (adiabatic) samples will isolate the effect of strain rate rise on TRIP, while maintaining comparable temperature. Note that for the adiabatic and external resistive heating test specimens, thermal gradients likely existed as a result of specimen geometry and contact with the grip fixtures. However, for both tests, gradients along the length of the specimens were measured to be no greater than five degrees Celsius. For the monotonic tests to fracture, two replicates were tested for each thermal-mechanical condition, and no significant variability in the mechanical response was observed between the replicates.

To complement the complete tensile tests, where specimens were deformed until fracture, single specimens were interrupted in $\varepsilon$ increments of approximately 0.02, to evaluate the (untransformed) austenite volume fraction and microstructure evolution as a function of $\varepsilon$. On interrupted specimens, metallographic polishing to 1 $\mu$m was performed prior to X-ray diffraction (XRD) scans on the normal plane. Scans intended to determine the bulk austenite content were performed using a PANalytical X'Pert diffractometer equipped with a copper source over a range of 40-146 degrees 2$\theta$. Because seven to ten interrupted specimens were characterized at sequential $\varepsilon$ increments for each thermal-mechanical condition, the data presented resolves the variability in the measurement of austenite phase volume fraction as a function of the thermal-mechanical condition and $\varepsilon$. After XRD, selected samples were vibratory polished to 0.05 $\mu$m, prior to SEM electron backscatter diffraction (EBSD) mapping of the microstructure. EBSD maps were collected in a JEOL JSM7000F field emission SEM with a step size of 0.1 $\mu$m. Kernel average misorientation (KAM) analysis was performed for fifth order neighbors and a threshold of 10 degrees using MTEX [102], to gain insights into the lattice rotation and microstructure as a function of $\varepsilon$ and the thermal-mechanical condition.

### 7.4 Results and Discussion

The three thermal-mechanical conditions are represented with temperature versus $\varepsilon$ plots in Figure 7.1(b). The $10^{-4}$ s$^{-1}$ (isothermal) specimen exhibited a temperature rise of less than 2 degrees Celsius above room temperature, because the test duration (~40 minutes) was sufficient to allow for nearly complete heat dissipation to the environment.
Figure 7.1 (a) Schematic of the tensile specimen used for the thermal-mechanical simulations. Some specimens deformed at $10^{-4}$ s$^{-1}$ were resistance heated, to emulate the deformation-induced temperature rise at $10^{-1}$ s$^{-1}$. (b) Surface temperature, (c) true stress, and (d) strain hardening exponent ($n_i$) plotted as a function of true strain for two representative tests from each thermal-mechanical condition.
For the $10^{-1} \text{s}^{-1}$ (adiabatic) specimen, the surface temperature increased as a function of $\varepsilon$, up to approximately 40 degrees Celsius above room temperature at the end of uniform elongation. Based on previous work by the authors in Chapter 6 and reference [166], it is assumed that the strain rate of $10^{-1} \text{s}^{-1}$ is ostensibly greater than the adiabatic threshold strain rate for QP980-1.4-A. The thermal profile of the $10^{-4} \text{s}^{-1}$ (resistance heated) samples was chosen empirically, based on the temperature of the $10^{-1} \text{s}^{-1}$ (adiabatic) specimen. The temperature profiles of the $10^{-1} \text{s}^{-1}$ (adiabatic) and $10^{-4} \text{s}^{-1}$ (resistance heated) specimens were within two degrees Celsius for the duration of the tests.

The tensile mechanical responses for each condition are shown with $\varepsilon$ versus true stress ($\sigma$) and instantaneous strain hardening exponent ($n_i$) plots in Figure 7.1(c) and (d), respectively. To eliminate the influence of the load cell noise in $n_i$, $\sigma$ and $\varepsilon$ were smoothed by adjacent values. $n_i$ was calculated after Dieter [114], $n_i = d(ln\sigma)/d(ln\varepsilon)$. Each thermal-mechanical condition exhibited similar initial yielding behavior without any clear strain rate sensitivity. However, significant differences in the work hardening behavior (manifested by $n_i$) were apparent after the onset of plastic deformation. Comparing the two test conditions deformed at $10^{-4} \text{s}^{-1}$, the isothermal specimens exhibited a much higher instantaneous strain hardening exponent at all stages of plastic deformation. Meanwhile, comparing the $10^{-4} \text{s}^{-1}$ (resistance heated) and $10^{-1} \text{s}^{-1}$ (adiabatic) specimens, the higher strain rate samples exhibited a higher instantaneous strain hardening exponent at intermediate tensile strains, then the instantaneous strain hardening exponent for both conditions converged at $\varepsilon = 0.15$. Each sample necked in accordance with the tensile instability criterion ($n_i = \varepsilon$). Thus, the specimen deformed isothermally at $10^{-4} \text{s}^{-1}$ exhibited the highest ductility because it had the highest work hardening rate at $\varepsilon > 0.12$. Serrations in the strain hardening exponent curves were observed for specimens deformed at $10^{-4} \text{s}^{-1}$ for both thermal conditions; these are attributed to dynamic strain aging in ferrite and secondary martensite [157, 166, 201]. It is known that the diffusivity of carbon within the BCC matrix increases with increasing temperature. However, between the isothermal and resistance heated specimens, no difference in the severity of the serrations was observed. Meanwhile, no serrations were present in the specimen deformed adiabatically at $10^{-1} \text{s}^{-1}$, presumably because the carbon diffusion kinetics were insufficient to facilitate DSA for the higher average dislocation velocities associated with higher strain rates.

To measure the extent of DIMT, XRD characterization was performed. Representative XRD scans of QP980-1.4-A prior to deformation and after 17 percent tensile strain are shown in Figure 7.2(a). Four FCC peaks ({$111$}, {002}, {112}, and {022}) and six BCC peaks ({$011$}, {002}, {112}, {022}, {013}, and {222}) were indexed. After tensile deformation, the relative height of FCC peaks diminished significantly, indicating that the deformation induced transformation of austenite to martensite occurred. The progression of the martensitic phase transformation suggests that the TRIP effect influenced the
deformation response. Figure 7.2(b) shows the austenite content in the interrupted test specimens as a function of $\varepsilon$. The austenite fractions reported in Figure 7.2(b) were measured by comparisons of the integrated intensities of BCC and FCC peaks after Cullity [76]. Taken as a measure of the TRIP effect, the exponential decay of the austenite fraction in each test condition supports the strain hardening data. That is, higher work hardening rates were correlated to more extensive decreases in the austenite fraction. For instance, samples deformed adiabatically at $10^{-1} \text{s}^{-1}$ exhibited the highest work hardening rates and decreases in the austenite content during the initial stages of plastic deformation, suggesting that the TRIP effect was more active in the rapidly strained specimens. Conversely, the extent of the deformation-induced martensitic transformation was reduced in samples deformed (isothermally) at a strain rate of $10^{-4} \text{s}^{-1}$. For the deformation condition employing a strain rate of $10^{-4} \text{s}^{-1}$ along with resistance heating, the TRIP effect was further suppressed. Therefore, based on both the tensile data and the associated XRD characterization, strain rate and temperature independently influence the TRIP effect in Q&P alloys. Variations up to one volume percent were observed between the exponential decay functions and the experimentally measured austenite fraction. This variability is perhaps due to a combination of uncertainty in the XRD measurements, as well as local variations in austenite stability and volume fraction due to strain and chemical inhomogeneity.

Specimens labeled with star symbols in Figure 7.2(b) were selected for further characterization with EBSD. In Figure 7.3(a), image quality maps overlaid with phase maps indicate the presence of austenite (FCC), ferrite (BCC without substructure), and martensite (BCC with blocky substructure) prior to deformation and after deformation at a strain rate of $10^{-4} \text{s}^{-1}$ under isothermal conditions. The austenite phase in both maps is mostly visible in a blocky morphology and is generally located adjacent to ferrite grain boundaries. Film-morphology austenite also exists between martensite laths. However, it was not fully resolved because the interaction volume of the backscattered electrons was larger than the lath thickness. The EBSD and XRD scans of the undeformed QP980-1.4-A agree well; the XRD scan measured a slightly greater amount of austenite (12 versus 10 volume percent), suggesting the presence of several volume percent film-morphology austenite. Secondary martensite in the deformed samples, assumed to have transformed from austenite because of the tensile deformation, is labeled with the black oval in Figure 7.3(a). This feature was distinguished from the primary martensite and ferrite based upon its darker contrast in the EBSD image quality maps and high local misorientation in the KAM maps. Note that the (secondary) martensite in the deformed Q&P microstructure has a greater carbon content and dislocation density compared to primary martensite in undeformed specimens; both these factors lead to observable contrast differences in the EBSD image quality and KAM maps.
Figure 7.2 (a) XRD scans before deformation ($\varepsilon = 0$) and after deformation ($\varepsilon = 0.17$) at a strain rate of $10^{-4}$ s$^{-1}$ under isothermal conditions. During tensile deformation under this condition, the austenite content decreased from 11.9 to 3.8 volume percent. (b) Evolution of retained austenite versus true plastic strain for each thermal-mechanical condition. Plotted lines are exponential decay fits with $R^2$ values of 0.87, 0.93, and 0.90, for the $10^{-1}$ s$^{-1}$ (adiabatic), $10^{-4}$ s$^{-1}$ (isothermal), and $10^{-4}$ s$^{-1}$ (resistance heated) test conditions, respectively. The star symbols in (b) represent the specimens which were subsequently characterized using EBSD.

Short range rotation of the BCC and FCC lattices, caused by the presence of geometrically necessary dislocations, is revealed with KAM maps in Figure 7.3(a). Prior to deformation, the local misorientation was uneven in the BCC lattice; KAM was low in the ferrite and high in the martensite. After deformation at $10^{-4}$ s$^{-1}$ under isothermal conditions, KAM became more homogeneous in the BCC map. This suggests that ferrite accommodated a relatively large amount of strain with dislocation slip, relative to martensite. This makes sense because ferrite is known to exhibit a comparatively lower flow strength, due to its lower initial dislocation density and depleted alloy content (e.g., by carbon and manganese partitioning to austenite during intercritical annealing). KAM also increased in the austenite during deformation. This was attributed to dislocation slip in the austenite, as well as dislocation generation due to the TRIP effect [13].

KAM distributions of the undeformed QP980-1.4-A are plotted for the BCC and FCC phases in Figure 7.3(b). The inhomogeneous KAM within the BCC phases prior to deformation is evident, where the undeformed ($\varepsilon = 0$) profile contains a broad martensite shoulder at a KAM above 0.7 degrees. Comparing the undeformed BCC and FCC spectra, it appeared that austenite exhibited an initial KAM that was greater than ferrite and less than martensite.
Figure 7.3 (a) EBSD image quality + phase maps, kernel average misorientation (KAM) maps of BCC phases, and KAM maps of FCC phases, prior to deformation and after deformation, at a strain rate of $10^{-4}$ s$^{-1}$ under isothermal conditions. The black oval in the IQ + phase map and KAM in BCC map emphasizes a region of the microstructure which was suspected to contain secondary martensite that formed by the deformation-induced martensitic transformation. (b) KAM distributions of the BCC and FCC phases, respectively, for each thermal-mechanical condition.
It is sensible that KAM was relatively high in the austenite; the volume expansion associated with the martensitic phase transformation likely imposed significant compressive stresses on the surrounding austenite during the initial quenching step of the Q&P process. These stresses necessitate dislocation slip in austenite to maintain plastic compatibility at austenite-martensite interfaces.

After plastic deformation under every thermal-mechanical condition, the KAM distributions evolved similarly (variations in the distributions were within the estimated uncertainty associated with the EBSD scan size). This suggested that the extent of dislocation slip was ostensibly similar for each thermal-mechanical condition. This is rational because the strain hardening rates of ferrite-martensite dual phase steels, whose microstructures are like QP980-1.4-A that lacks austenite, are reportedly indistinguishable from $10^{-3}$ to $10^{-1}$ s$^{-1}$ [237]. Since the dislocation slip behavior was similar for each thermal-mechanical condition, it is concluded that the dissonant strain hardening responses shown in Figure 7.1(d) are solely due to variations in the potency of the TRIP effect.

### 7.5 Conclusions

The data presented here quantified the independent effects of strain rate and deformation induced temperature rises on the martensitic phase transformation. By externally heating without increasing the strain rate, it was shown that deformation induced temperature rises greatly impede the martensitic transformation and reduce the strain hardening contribution of the TRIP effect. Meanwhile, by comparing samples deformed at $10^{-4}$ and $10^{-1}$ s$^{-1}$ that were heated externally and adiabatically, increasing the strain rate appears to accelerate the martensitic transformation and enhance the work hardening. This result clarifies the root cause of the ductility and martensitic transformation trough that was reported for 3GAHSS previously [166]; because the temperatures reached by deformation induced heating saturate at the adiabatic threshold strain rate, adiabatic temperature rise no longer causes shifts in the TRIP effect for strain rate variations above approximately $10^{-1}$ s$^{-1}$. Rather, for strain rate shifts at dynamic rates, e.g., for a shift from 1 to $10^3$ s$^{-1}$, the martensitic transformation kinetics are controlled by an increase in the number of martensite nucleation sites and an increased mechanical driving force [57, 188].

This revelation was quite challenging to reach using conventional tensile tests. Thus, thermal-mechanical simulation is a promising method to decouple the influences of temperature and strain rate on the deformation behavior of structural metals; the technique will be useful for tailoring the microstructures of 3GAHSS, and other alloy classes that take advantage of the TRIP or TWinning Induced Plasticity (TWIP) effects for strain accommodation and strengthening.
7.6 Acknowledgements

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7.7 Author Contributions

CF: conceptualization, formal analysis, investigation, writing - original draft, visualization. DB: writing - review and editing. BM: writing - review and editing, visualization. TB: investigation, writing - review and editing. AC: conceptualization, writing - review and editing, supervision. KC: conceptualization, writing - review and editing, supervision.
CHAPTER 8
EXPERIMENT D: RESOLVING THE MARTENSITIC TRANSFORMATION \textit{IN-SITU} AT DYNAMIC STRAIN RATES USING SYNCHROTRON X-RAY DIFFRACTION

Modified from a short manuscript intended for publication in a letters-style journal.

The anticipated author list is:


8.1 Abstract

The dynamic deformation response of two quenching and partitioning (Q&P) steels was investigated using a high strain rate tension pressure bar and \textit{in-situ} synchrotron radiography and diffraction. This allowed for concurrent measurements of the martensitic transformation, the elastic strains/stresses on the martensite and ferrite, and the bulk mechanical behavior. The steel with the greater fraction of ferrite exhibited greater ductility and lower strength, suggesting that dislocation slip in ferrite enhanced the deformability. Meanwhile, the kinetics of the martensitic transformation appeared similar for both steels, although the steel with a greater ferrite fraction retained more austenite in the neck after fracture.

8.2 Introduction

The transformation-induced plasticity (TRIP) effect is increasingly being used to enhance the mechanical properties of engineered materials [178]. The TRIP effect is observed when a metastable phase undergoes a deformation-induced martensitic transformation (DIMT), and a combination of martensite variant selection [12] and dislocation generation [13] causes strain accommodation and hardening. The TRIP effect is of particular interest for high-toughness materials designed for high-strain rate deformation. For instance, the TRIP effect is being explored for steels used in vehicle crash structures [14] and in blast or penetration-resistant armor [238].

Quenching and partitioning (Q&P) steels [27, 33, 104], an emerging class of TRIP-assisted advanced high strength steels (AHSS), are of particular importance because of their affordability and high strength. Q&P steels are candidates for use in vehicle crash structures, so it is necessary to understand the kinetics of DIMT so that the TRIP-effect can be tailored for enhanced crash performance. DIMT responds to increases in the strain rate by several mechanisms. First, shear band intersections, which act as potential nucleation sites for martensite, tend to increase in number at high strain rates [54, 55]. Second, as strain
rates increase, the heat produced during deformation has less time to disperse into the environment, and so the temperature of the steel rises [51]. Rising temperatures reduce the driving force for DIMT, and thus reduce the propensity for martensite to nucleate at a potential nucleation site [8, 46]. In a previous work by the authors, it was shown that the mechanical properties of a commercially available “QP980” steel were sharply strain rate sensitive; increasing the strain rate from $10^{-4}$ to $10^{-1}$ s$^{-1}$ decreased the strain hardening rate, and thus the ductility [166]. In a subsequent investigation, it was shown that adiabatic heating and strain rate variations exhibited dissonant effects on the DIMT kinetics in QP980; that is, increasing the strain rate appeared to enhance the martensitic transformation, while adiabatic heating appeared to suppress the martensitic transformation [167]. A limitation of the previous studies was that the maximum strain rate tested was $10^{-1}$ s$^{-1}$; meanwhile, vehicle crashes impart deformation at much higher strain rates, up to approximately $10^{3}$ s$^{-1}$ [56, 184]. Thus, it is also prudent to study mechanical properties and DIMT at dynamic rates.

Some details about microstructure evolution and DIMT kinetics in AHSS have been resolved using interrupted high-rate tests; for instance, Yang et al. showed that the progression of DIMT was more pronounced at $10^{3}$ than at $10^{-3}$ s$^{-1}$ [58]. However, with interrupted tests, it is difficult to measure microstructure and phase fraction evolution with sufficient strain resolution, which obstructs fitting the measured DIMT data to empirical kinetics models [15]. Furthermore, the strain path dependence of DIMT could bias the data; for instance, Du et al. showed that strain incompatibility between the phases in a Q&P steel can cause reverse plastic strains upon unloading of interrupted test specimens [239], which could cause further DIMT during unloading. The ex-situ X-ray diffraction technique also lacks the ability to measure stresses/strains on individual phases, which is problematic because this information could be useful to further tailor the DIMT behavior.

To inform better microstructure designs for metastable alloys, experimental configurations are needed that concurrently resolve the bulk mechanical properties, the constituent phase volume fractions, and the stresses on each phase. For quasi-static tests, these tools have become increasingly available at synchrotron and neutron radiation facilities. However, until recently, the scientific community has lacked the capability to perform such experiments at high strain rates. Here, we use a state-of-the-art synchrotron-based tool [240–242] developed at the 32-ID beamline at the Advanced Photon Source (APS) at Argonne National Laboratory (ANL), to collect radiography and diffraction videos of Q&P steels deformed at dynamic strain rates using a high-rate pressure bar. For the first time, high-speed radiography and high-energy X-ray diffraction (HEXRD) videos were collected in-situ, while the Q&P steels were deformed at $10^{3}$ s$^{-1}$, to elucidate the link between DIMT kinetics and mechanical performance at dynamic rates.
8.3 Methods and Materials

Two commercially produced, Zn-coated C-Mn-Si steels were selected for this study. Each steel was intercritically annealed to form approximately 10 to 20 volume percent intercritical ferrite. Subsequently, the steels were subjected to a Q&P process, leading to UTS values of 980 and 1180 MPa, respectively, for the microstructures containing higher and lower fractions of ferrite. Here, steels are designated based on strength level; for example, “QP980” refers to a steel with a UTS of 980 MPa. The chemical composition and thickness of each steel is listed in Table 8.1. In a previous paper, QP980 and QP1180 were characterized by a combination of optical microscopy, scanning electron microscopy (SEM), lab-scale X-ray diffraction (XRD), and tensile testing at quasi-static rates [104]. With XRD, it was determined that each steel contained approximately 12 volume percent austenite, with intercritical ferrite and martensite occupying the remainder of the microstructure. The austenite existed in both blocky and film-type morphologies. The martensitic fraction of the microstructure appeared to be slightly tempered from the partitioning step of the Q&P thermal cycle [104]. Tensile specimens, with a gauge length of 4 mm and a width of 1 mm, were machined parallel to the rolling direction of QP980 and QP1180 using wire electron discharge machining. Subsequently, they were metallographically ground and polished to a final thickness of approximately 200 µm, to minimize the X-ray absorption by the samples.

The Sector 32-ID beamline at the APS is equipped with several high-rate tension and compression pressure bars, along with imaging equipment capable of resolving fracture and phase evolution with sub-microsecond temporal resolution. Details of the beamline configuration are documented by Hudspeth et al. [240, 241]. A polychromatic undulator pink beam was used due to its relatively high intensity and pulse frequency. The beam had a nominal first harmonic energy of 24.65 keV (created with a 13 mm undulator gap) and was adjusted using slits to a width of 2 mm and a height of 1 mm. Radiography videos were recorded using a Photron FASTCAM SA-Z equipped with a LuAG:Ce scintillator at ~50,000 frames per second. Simultaneously, diffracted rings were recorded in the transmission mode with a Shimadzu HPV-X2 equipped with a LSO:Ce scintillator at ~200,000 frames per second. A modified tension bar equipped with a fast-response load cell was used for the experiments [242]. A schematic of the tension bar, the radiography and diffraction cameras, and the synchrotron beam are shown in Figure 8.1(a).

Table 8.1 Composition of Each Q&P Steel

<table>
<thead>
<tr>
<th>Steel</th>
<th>C  (wt pct)</th>
<th>Mn (wt pct)</th>
<th>Si  (wt pct)</th>
</tr>
</thead>
<tbody>
<tr>
<td>QP980-1.4-A</td>
<td>0.201</td>
<td>1.93</td>
<td>1.62</td>
</tr>
<tr>
<td>QP1180-1.6-A</td>
<td>0.204</td>
<td>2.26</td>
<td>1.74</td>
</tr>
</tbody>
</table>
8.4 Results and Discussion

Four replicate samples for each test condition were deformed at a strain rate of approximately $10^3 \text{s}^{-1}$. To exemplify the *in-situ* data that were collected during the experiments, representative radiography and diffraction frames at various stages of deformation are shown in Figure 8.1(b and c). In Figure 8.1(b), radiography images for QP1180 are shown during uniform elongation, necking, and fracture. For each specimen, diffuse necking occurred first, followed by localized thinning at the final location of fracture. In Figure 8.1(c), a diffraction image for the same specimen prior to deformation is shown; HiSPoD, a MATLAB-based diffraction analysis toolbox developed at the 32-ID beamline [243], was implemented to radially integrate the diffraction image, leading to the one-dimensional “lineout” shown in Figure 8.1(c). The theoretical locations of the BCC(002), BCC(112), BCC(022), FCC(022), FCC(113), and FCC(222) peaks are labelled in the diffraction image and the lineout.

Engineering stress-strain curves for QP980 and QP1180, deformed at $10^3 \text{s}^{-1}$, are shown in Figure 8.2(a). Undulations in the stress-strain curves of both steels were evident; these undulations may have been due to the dynamic nature of the experiment, where the deformation was not homogeneous due to stress wave propagation along the length of the sample. More specifically, the undulations are thought to be caused by elastic compliance in the vicinity of the load cell [244]. The QP980 generally exhibited a higher elongation and a lower yield stress, relative to QP1180. Interestingly, the measured stresses were similar to those observed previously at strain rates of $10^{-4}$ to $10^{-1} \text{s}^{-1}$; this suggests that a strain rate dependence of the flow stress was not obvious [166].

Based on the stress-strain curves, it was apparent that the total elongation was large relative to the uniform elongation, especially for QP1180. This suggested that most of the strain was localized in the neck. Meanwhile, the radiography videos showed that diffuse and localized necking occurred for both steels. To quantitatively access the bulk ductility, several measurements of the fractured specimens were collected. True uniform strain was measured based on the cross section of the sample, prior to deformation and after fracture in the uniformly-elongated region of the gauge section. True fracture strain was calculated based on the area of the fracture surface after Dieter [114]. Neck width was measured using the radiography stills after fracture. Each steel exhibited relatively little uniform elongation (Figure 8.2(b)). Meanwhile, trends in the neck width (Figure 8.2(c)) and true fracture strain (Figure 8.2(d)) suggested that QP980 had a higher necking strain relative to QP1180. This confirmed that the structure-property relationships that exist in the steels at quasi static strain rates also prevail at high rates. That is, the larger amount of ferrite in QP980 enhanced the ductility at both quasi static and dynamic rates.
Figure 8.1 (a) Configuration of the tensile specimen relative to the X-ray beam axis, radiography camera, and diffraction camera. The cross-section of the X-ray beam (1 × 2 mm) is shown schematically with the orange rectangle in the center of the tensile specimen. (b) Representative radiography images recorded during uniform elongation, necking, and fracture of a QP1180 specimen using an acquisition rate of 50,000 images per second. (c) Representative diffraction data of the same specimen prior to deformation, collected using an acquisition rate of 200,000 images per second. The predicted locations of various peaks are superimposed on the image. Each diffraction image was radially integrated using HiSPoD software; an example lineout for QP1180 prior to deformation is shown.
To measure the evolution of austenite content as a function of deformation time, HiSPoD was used to
create HEXRD lineouts at time increments of five microseconds. Despite the use of synchrotron light,
which is on the order of one quadrillion-times brighter than a common lab diffractometer, the minuscule
exposure times used in this experiment greatly limited the HEXRD signal that was collected by the
detector. Three steps were taken to improve the signal-to-noise ratio of the lineouts. First, the diffracted
intensities detected by each pixel were smoothed by pixels existing in adjacent Q-space. Second, the
lineouts were also smoothed over adjacent time increments. Finally, the lineouts for each of the four
replicates from each steel were summed. After this processing, the integrated intensities of the three
lowest-index peaks captured in the experiment, BCC(002), FCC(022), and BCC(112), were plotted over
time in Figure 8.3(a). Meanwhile, the FCC(113), FCC(222), and BCC(022) peaks were omitted because
their height was barely above the background; the low signal of those peaks was likely due to their
comparatively low structure factor. The average onset of straining and fracture are annotated with a black
horizontal line, while the peak position prior to straining is plotted with a red vertical line.

Changes in the intensity of the peaks were observed over time, which suggested that the number of
grains that met a diffraction condition changed during deformation. For instance, the BCC(002) peak
appeared to slightly diminish in intensity for both steels, which could be due to small changes in
crystallographic texture during plastic deformation, thus rotating crystals out of a diffraction condition.
The FCC(022) appeared to strongly diminish in intensity for both steels. DIMT was likely responsible for
this shift; DIMT could have facilitated a reduction in the total volume fraction of austenite, and thus a
decrease in the diffracted intensity of FCC(022) over time. Finally, the BCC(112) appeared to exhibit
relatively consistent intensity throughout the deformation process.

Deviations from the peak position (Q) prior to straining were observed for each steel and diffraction
peak. These peak shifts were due to the elastic lattice strains associated with mechanical loading of the
tensile specimens. After the d-spacing of each family of planes \(d_{hkl}^{\varepsilon}\) was determined with Bragg’s law, the
elastic lattice strains \(e_{hkl}^{\varepsilon}\) were calculated using Equation 8.1:

\[
e_{hkl}^{\varepsilon} = \frac{d_{hkl}^{\varepsilon} - d_{hkl}^{\varepsilon=0}}{d_{hkl}^{\varepsilon=0}}
\] (8.1)

The evolution of \(e_{002}^{\varepsilon \text{BCC}}\) and \(e_{112}^{\varepsilon \text{BCC}}\) are plotted in Figure 8.3(b,c), respectively. There was no obvious
difference in the lattice strains between QP980 and QP1180, which suggested that any difference was less
than the practical spatial resolution of the diffraction setup. A similar analysis was performed for the
FCC(022) reflection, but the signal-to-noise ratio of that peak, along with the decreasing peak height due
to DIMT, made the analysis unreliable.
Figure 8.2 (a) Stress-strain curves for both steels. Measures of bulk ductility for both steels: (b) true uniform strain, (c) neck width, and (d) true fracture strain.
The stresses nominally parallel to the tensile axis for the BCC(112) peak, $\sigma_{BCC}^{112}$, were estimated based upon Hooke’s law using Equation 8.2 [245]:

$$\sigma_{BCC}^{112} = E_{BCC}^{112} e_{BCC}^{112}$$

(8.2)

Where $E_{BCC}^{112}$ was assumed to be equal to 210 GPa after Harjo et al. [245]. $\sigma_{BCC}^{112}$ appeared to be relatively comparable for each steel; the maximum $\sigma_{BCC}^{112}$ was equal to 1030 ± 100 MPa for both steels. Interestingly, the stresses measured by diffraction appeared to be less sensitive to ringing artifacts than the stresses measured by the tension bar. Perhaps this was because the bulk measurement of stress via HEXRD was less sensitive to the non-homogeneous strain wave propagation.

After the beamtime, electron backscatter diffraction (EBSD) maps in the normal direction plane were collected in the grip, gauge, and neck. The EBSD maps were used to evaluate the microstructure development, and to confirm that DIMT was the root cause of the diminished intensity of the FCC(022) reflection. Figure 8.4 shows EBSD image quality (IQ) + austenite (FCC) phase maps overlaid for each steel. In the grip, the starting microstructure of each steel is evident. Three phases were present: ferrite, which formed during the intercritical annealing process and indexed as BCC with a relatively high IQ and minimal substructure; martensite, which indexed as BCC and exhibited a substantial lath substructure; and austenite, which indexed as FCC and existed in small blocky and film shapes. Prior to deformation, both microstructures looked similar, with the key difference being that the QP1180 appeared to contain less ferrite and more martensite.

In the uniformly-elongated section of the gauge, the IQ slightly decreased, which was especially noticeable in the ferrite. Although not shown here, this decrease in the IQ was accompanied by an increase in the lattice rotation in large ferrite grains, which was manifest by larger orientation deviation from grain center to edge. Relative to the scans in the grip section and in the uniformly-elongated gauge section, the scans collected in the neck of both steels exhibited a strong refinement of the substructure in every phase, along with a large deterioration in IQ. The austenite content changed in a similar manner; no strong change in austenite content was observed between the scans in the grip and in the uniformly-elongated gauge section, but the austenite content in the neck of both steels decreased extensively. Together, the observations of strong evolution of IQ and austenite content during necking suggested that both the TRIP-effect and dislocation slip accommodated the necking strains, despite the high effective strains and temperature rises associated with neck formation at dynamic strain rates.
Figure 8.3 (a) Intensity change over time measured *in-situ* for the BCC(002), FCC(022), and BCC(112) peaks. (b,c) Elastic strains on the BCC(002) and BCC(112) peaks, respectively. (d) Elastic stress on the BCC(112) peak, calculated based on Hooke’s law and an assumed elastic modulus ($E_{112}^{\text{BCC}}$) of 210 GPa [245]. The steps in each curve (b-d) are indicative of the minimum spatial resolution of the diffractometer, which was controlled by the pixel density of the diffraction camera.
With a true fracture strain of approximately one, EBSD analysis showed that several volume percent austenite remained untransformed within the neck in QP980, which suggested that the surviving austenite was exceedingly stable during high-rate deformation. Meanwhile, the DIMT behavior of QP1180 diverged, where a smaller fraction of austenite existed after necking, despite a lower true fracture strain of approximately 0.6. This suggested that the austenite in QP1180 was less stable under similar deformation conditions. This could have been caused by the higher ratio of martensite to ferrite in QP1180, which could have promoted strain partitioning to the austenite. This insight gained by EBSD analysis was not supported by the in-situ bulk diffraction data. Because the bulk diffraction analysis was qualitative in nature due to the low signal-to-noise ratio, it was impossible to compare the measured austenite fractions existing with phase amounts less than several volume percent. A greater signal-to-noise ratio would be required to quantify the DIMT kinetics in heavily deformed microstructures containing only a few percent austenite. In the future, a greater signal-to-noise ratio may be achieved with the development of higher sensitivity high-rate cameras and higher brightness beamline configurations.

Figure 8.4 EBSD image quality + austenite (FCC) phase maps for each steel, with progressively increasing applied tensile strain. From left to right: in the grip (zero strain), the uniformly-elongated gauge section, and the necked region.
8.5 Conclusions

The maturation of bulk diffraction tools with excellent temporal resolution has provided new opportunities to understand the deformation of materials at high strain rates. Here, the dynamic tensile mechanical properties of QP980 and QP1180 were measured, while DIMT was characterized with a combination of in-situ diffraction imaging and ex-situ EBSD. The QP980, which contained a higher fraction of soft ferrite, exhibited a lower yield strength and a higher ductility. During deformation, the strains and stresses on the BCC-indexed phases were tracked, and they appeared to be similar between the two steels, to within the practical spatial resolution of the diffraction detector. Meanwhile, the austenite content in both steels appeared to decrease similarly, prior to the formation of a neck. After a neck formed, EBSD showed that the austenite in QP980 appeared to exhibit a slightly greater stability as a function of true strain. Continued experiments in this field, coupled with enhancements in resolution and signal-to-noise ratio in bulk diffraction experiments, will elucidate the fundamental relationships between microstructure development and mechanical performance during deformation at dynamic strain rates.

8.6 Acknowledgements

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CHAPTER 9
EXPERIMENT E: ELUCIDATING THE TEMPERATURE DEPENDENCE OF TRIP USING SYNCHROTRON X-RAY DIFFRACTION, CONSTITUENT PHASE PROPERTIES, AND STRAIN-BASED KINETICS MODELS

Modified from a manuscript intended for publication as a journal article.

9.1 Abstract

Understanding the deformation-induced martensitic transformation (DIMT) is critical for interpreting the microstructure-property relationships that govern the performance of TRIP-assisted steels. However, modern TRIP-assisted steels often exhibit DIMT kinetics that are not easily captured by existing empirical models based on bulk tensile strain. We address this challenge by combined bulk uniaxial tensile tests and in-situ high energy synchrotron X-ray diffraction, which resolved the phase volume fractions, stress-strain response, and microstructure evolution of each constituent phase. A modification of the Olson-Cohen model is proposed, where the strain in austenite estimated with synchrotron diffraction is used, rather than the bulk tensile strain. The improved DIMT kinetic model is used as a framework to clarify the root cause of an insufficiently understood toughness trough reported for TRIP-assisted steels during deformation at elevated temperatures. The importance of the temperature-dependent toughness is discussed, based on the opportunity to modify deformation processes to tailor the DIMT kinetics and mechanical properties during forming and in service.

9.2 Introduction

Martensitic phase transformations are increasingly utilized to obtain enhanced mechanical property combinations in engineered materials, particularly under severe deformation conditions. Possibilities for toughness enhancements have been identified for several material classes, including zirconia ceramics [246], high entropy alloys [247–249], titanium alloys [250, 251], and advanced high strength steels (AHSS) [104, 178]. The transformation-induced plasticity (TRIP) effect is thought to be the principal mechanism by which martensitic phase transformations strengthen such materials. For instance, in blast and penetration resistant steels for protective structures, the TRIP effect is known to suppress
plastic instability and shear banding [238]. Meanwhile, in steels designed for automotive crash structures, the TRIP effect is used to enhance the formability and toughness.

In AHSS, the TRIP effect increases formability by several mechanisms. First, dislocations are generated during the deformation-induced martensitic transformation (DIMT) to maintain plastic compatibility between constituents (e.g., at austenite-martensite interfaces), which results in strain hardening. Second, depending on the strain state, the volume expansion associated with DIMT can provide an additional pathway for strain accommodation. Third, constituent phases in AHSS offer distinctive properties, and the phases deform in a composite manner within the bulk. For example, ferrite is soft and ductile, while martensite and metastable austenite are hard [252]. As a result, bulk property combinations can be tailored by adjusting the individual-phase properties and the fractions of each constituent. During DIMT in Quenching and Partitioning (Q&P) steels, new/untempered martensite progressively forms at the expense of austenite, which increases the bulk strength because untempered martensite is approximately 30 pct. harder than austenite [252]. Although it is understood that constituent phase properties are critical for tuning and maximizing property combinations, it is difficult to measure them during bulk mechanical testing. For instance, individual-phase stresses are relieved upon unloading, so it is impossible to measure such values with common ex-situ tools. Bulk in-situ characterization techniques have emerged to solve this problem; they can show how complex microstructures accommodate strain with a combination of dislocation slip and TRIP. In particular, high energy X-ray diffraction (HEXRD) using a synchrotron source can provide outstanding spatial and temporal resolution of the phase volume fraction [201], plastic strain accumulation [253], and elastic stresses for each constituent phase [245] during bulk mechanical testing.

The concept of austenite stability has been used to describe the kinetics of DIMT as a function of the applied strain [45, 254]; less stable refers to austenite which undergoes DIMT more rapidly. The study of austenite stability is of topical interest to enhance the properties of TRIP-assisted steels, because austenite stability decisively influences properties during forming and in service [104]. Strain-based models for DIMT have been proposed in the literature, such as the Olson-Cohen (OC) and Burke-Matsumura (BM) models (see Table 9.1). A review of the relevant models is provided by Samek et al. [45]. Generally, these empirically-derived models correctly capture the DIMT kinetics in fully austenitic steels, where the austenite grains are relatively chemically and morphologically homogeneous. However, the models struggle to capture the DIMT kinetics of steels with complex microstructures, where austenite grains exist with a spectrum of compositions, sizes, and shapes, and strain is partitioned unevenly between the constituent phases.

The complexity of austenite stability in multi-phase steels has motivated targeted efforts within the literature. Recently, He divided the factors that influence the stability of austenite into “intrinsic” or
“extrinsic” with respect to an individual austenite grain [255]. Intrinsic factors include dislocation density [181], alloy partitioning, and the composition, morphology, size, and orientation of austenite [91]. Extrinsic factors include strain rate [166], temperature [8, 46, 47], strain state [54, 55], and surrounding microstructure [179]. Increasing the deformation temperature decreases the chemical driving force for DIMT, which is represented by the austenite stability parameters, $k$ and $\beta$, in the BM and OC models, respectively. For Q&P steels, Poling et al. and Feng et al. showed that $\beta$ decreased with increasing deformation temperature up to 100 °C [8, 47], thereby suppressing DIMT kinetics. Temperature can also influence the austenite stacking fault energy, which can affect the formation of shear band intersections, the primary sites for martensite embryo nucleation [41]. In the OC model, a martensite nucleation parameter, $\alpha$, is used to capture this phenomenon. For fully austenitic steels, Olson and Cohen showed that $\alpha$ decreased with increasing temperature [15], while a similar trend was shown for Q&P steels up to 100 °C by Poling et al. [8]. Strain rate is of particular importance, because stamping operations and vehicle crashes generally impart deformation at high enough strain rates to raise sheet temperatures on the order of 50 to 100 °C above-ambient conditions [120, 165]. Furthermore, in Chapter 6 and Chapter 7, it was shown that strain rate exhibited an independent effect on DIMT kinetics that was not attributed to adiabatic heating, over a range of $10^{-4}$ to $10^{-1}$ s$^{-1}$. Rather, an acceleration of DIMT was observed, likely due to a positive correlation between the strain rate and the number of stacking fault intersections that were available to nucleate martensite, along with higher stresses on the austenite [166, 167].

### Table 9.1 Common Strain-Based Models for DIMT

<table>
<thead>
<tr>
<th>Equation</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>$f_\gamma^e = f_\gamma^{\varepsilon=0} [1 - \exp(-\beta [1 - \exp(-\alpha \varepsilon)])^n]$</td>
<td>Olson-Cohen (OC) [15]</td>
</tr>
<tr>
<td>$f_\gamma^e = f_\gamma^{\varepsilon=0}/(1 + p/[k_\varepsilon f_\gamma^{\varepsilon=0}])$</td>
<td>Burke-Matsumura (BM) [43, 44]</td>
</tr>
</tbody>
</table>

A source of inaccuracy in the empirical models for DIMT is that austenite stability is linked not only to the properties of the austenite (e.g., composition, size, and morphology), but also the properties of the surrounding phases within the microstructure. For instance, the flow stress of body centered cubic (BCC) crystal structures, that is, of ferrite, bainite, and martensite, is known to be strongly temperature and strain rate sensitive because of the thermal activation of dislocation slip in BCC crystal structures [50]. Because dislocation slip activation is less temperature sensitive in face centered cubic (FCC) crystal structures [50], (for example, in austenite,) there exists the possibility of variations in stress partitioning as a function of temperature and bulk strain. Furthermore, dynamic strain aging, dislocation recovery, or
tempering of martensite or bainite at elevated temperatures could also influence the stress partitioning between phases [118], leading to different effective stresses and strains in the austenite.

Several recent studies have aimed to resolve how the balance of dislocation slip and TRIP influences the mechanical properties of AHSS, while considering strain rate and temperature. For instance, Liu et al. and Finfrock et al. both observed that inhibited DIMT kinetics led to a ductility trough at strain rates around $10^{-1} \text{s}^{-1}$ at 25 °C [166, 212]. Likewise, Coryell et al. discovered an analogous ductility trough for tensile tests at various temperatures under quasi-static strain rates. In Coryell’s work, a minimum ductility at 100 °C coincided with the suppression of DIMT and a high local lattice misorientation, which was attributed to strain accommodation primarily via dislocation slip [46]. The governing mechanism for both effects may be related; for instance, Frint et al. observed that the strain rate sensitivity of ductility was promoted at elevated temperatures [85]. Previous work has not established a clear explanation for the behavior based solely on the chemical stability of austenite [46, 85, 166, 212]. Fortunately, temperature can be adjusted during sheet metal forming with die heating or cooling. Likewise, strain rate can be adjusted during forming by manipulating forming press speeds. Thus, if the root cause(s) of the temperature and strain rate dependent ductility troughs can be established, there exists a possibility to modify deformation processes for improved mechanical response during forming and performance in service.

Here, we resolve the individual-phase deformation responses and DIMT kinetics for several Q&P steels with different microstructures, along with a dual phase (DP) steel to serve as a baseline for properties in the absence of austenite. Bulk mechanical testing at ambient and above-ambient temperatures, along with in-situ HEXRD data, are combined with post-mortem spatially-resolved electron backscatter diffraction (EBSD) to elucidate a stress partitioning mechanism that strongly impacts DIMT kinetics and TRIP strengthening. For the first time, stress partitioning, considered in combination with the traditional understanding of the chemical stability and martensitic embryo nucleation, is proposed as a principal mechanism by which a DIMT trough exists at above-ambient temperatures in Q&P steels. Furthermore, dislocation density measurements in the BCC phases are used to relate the DIMT kinetics to the ductility trough [46, 85, 166, 212]. The concurrent implementation of diffraction tools along multiple length scales (from sub-µm to mm) revealed insights about stress partitioning and TRIP kinetics that have been impossible to reach using ex-situ tests alone.

9.3 Experimental Procedure

Bulk tensile tests at various temperatures were conducted with in-situ HEXRD at the 1-ID beamline at the Advanced Photon Source (APS) at Argonne National Laboratory. The tests elucidated the roles of temperature and microstructure on deformation in several advanced high strength steels (AHSS). Using the
**in-situ** HEXRD technique (Figure 9.1(a,b)), we coupled bulk mechanical testing data with high-temporal resolution measurements of the tensile stresses, strains, and volume fractions of individual constituent phases. Subsequently, we augmented the HEXRD and mechanical testing data with spatially-resolved ex-situ EBSD maps, to clarify how local lattice rotation and crystallographic texture was correlated to bulk properties.

**9.3.1 Experimental Materials**

Three commercially-available lean-alloy AHSS were provided by Cleveland Cliffs Steel Corporation: QP980-1.0, QP1180-1.0, and DP980-1.4. The sheet steels were fully finished and Zn-coated on an industrial mill. Each steel was manufactured using distinctive alloy contents and heat treat schedules, which were intended to generate different property combinations by tuning the microstructure, phase constituents, and strengthening mechanisms. The steels are named based on the heat treat approach used: “QP” and “DP” refer to “quenching and partitioning” and “dual phase”, respectively. The steels are also named based on their intended minimum ultimate tensile strength (that is, 980 and 1180 MPa). The thicknesses of QP980-1.0, QP1180-1.0, and DP980-1.4 were 1.0, 1.0, and 1.4 mm, respectively. The chemical composition of each steel was determined with a combination of several analytical techniques including inert gas fusion, combustion, and inductively coupled plasma mass spectrometry. The chemical compositions are shown in Table 9.2. The two Q&P steels contained additions of C and Mn for increasing the stability of austenite and the hardenability, as well as Si which is known to suppress the formation of carbides that could deleteriously limit the amount of carbon that partitions to austenite. The carbon content of austenite \( C_\gamma \) was estimated using HEXRD to be 1.16 and 1.13 weight percent for QP980-1.0 and QP1180-1.0 (see Appendix C for details). Meanwhile, the DP980-1.4 contained a lower amount of C, Mn, and Si, as well as additions of Cr and Mo for hardenability enhancements.

Representative SEM micrographs of each steel etched with one pct. Nital are shown in Figure 9.1(c-e). QP980-1.0 contained \( \sim 20 \text{ vol. pct.} \) ferrite (\( \alpha \)) formed during an intercritical annealing step. During a subsequent quenching and partitioning process, \( \sim 12 \text{ vol. pct.} \) austenite (\( \gamma \)) was retained, with the remainder being a mix of tempered and untempered martensite (\( \alpha' \)). The QP1180-1.0 was not intercritically annealed, and so the microstructure did not contain substantial amounts of ferrite. The QP1180-1.0 also retained \( \sim 12 \text{ vol. pct.} \) austenite during Q&P processing, with the remainder martensite. The DP980-1.4 steel exhibited a ferrite-based microstructure with dispersed islands of martensite and negligible austenite (\( i.e., f_\gamma < 1 \text{ vol. pct.} \)). Each steel was observed to contain a fine prior austenite structure, and the structure was further-refined during the formation of the martensitic matrix. The intercritically-annealed steels, QP980-1.0 and DP980-1.4, exhibited ferrite grain diameters on the order of 5
and 2 µm, respectively. The approximate volume fraction of intercritical ferrite and austenite in each steel is listed in Table 9.2, with the balance being martensite.

![Diagram](image1.png)

Figure 9.1 (a) Schematic of the high-energy X-ray diffraction (HEXRD) geometry at the 1-ID beamline at the Advanced Photon Source. Debye-Scherrer rings are shown for the DP980-1.4 steel prior to deformation. (b) Example of a one-dimensional lineout for the QP980-1.0 steel. The lineouts were created by integrating around the azimuthal angle (ϕ) using GSAS-II software. Note that to show the high signal-to-noise ratio of the low intensity reflections, the full height of the α(110) reflection is not shown in (b). (c-e) Normal-direction SEM micrographs of QP980-1.0, QP1180-1.0, and DP980-1.4, respectively. The rolling and loading direction are to the right in the micrographs.

### 9.3.2 Mechanical Testing

Tensile specimens of each steel were machined with the loading direction (LD) parallel to the rolling direction (RD) of the sheets. The gauge sections of the specimens were 6 mm long and 1.4 mm wide. The as-received sheet thickness was used. Before testing at the APS, the Zn-coating was chemically removed
with a mixture of 20 pct. hydrochloric acid, 30 pct. hydrogen peroxide, and 50 pct. water.

An MTS servo hydraulic load frame was used to monotonically load each specimen until fracture in uniaxial tension, using a nominal engineering strain rate of \(2 \times 10^{-4} \text{ s}^{-1}\). A load cell was used to determine the stress. Crosshead displacement was used to calculate strain, and elastic deflection in the frame was removed from the crosshead displacement using the reciprocal springs-in-series relationship. A description of the compliance correction process is provided in Appendix D. After calculating true stress (\(\sigma\)) and true strain (\(\varepsilon\)), Equation 9.1 was used to calculate the instantaneous strain hardening exponent (\(n_i\)) [114]:

\[
n_i = \frac{d(ln\sigma)}{d(ln\varepsilon)} \quad (9.1)
\]

Specimens were deformed at temperatures of 25, 100, 175, and 250 °C. An infrared furnace was used to heat the specimens over a period of \(~5\) minutes. The furnace temperature was controlled using a K-type thermocouple attached directly to the gauge section of each sample. Only one specimen of each material and temperature was tested due to time constraints. Trends in the mechanical testing data as a function of steel and temperature were verified externally in separate tensile tests.

<table>
<thead>
<tr>
<th>Steel</th>
<th>C wt pct</th>
<th>Mn wt pct</th>
<th>Si wt pct</th>
<th>Cr wt pct</th>
<th>Mo wt pct</th>
<th>Austenite vol pct</th>
<th>Ferrite vol pct</th>
</tr>
</thead>
<tbody>
<tr>
<td>QP980-1.0</td>
<td>0.20</td>
<td>1.96</td>
<td>1.68</td>
<td>0.03</td>
<td>&lt;0.02</td>
<td>11.2</td>
<td>26</td>
</tr>
<tr>
<td>QP1180-1.0</td>
<td>0.19</td>
<td>2.31</td>
<td>1.64</td>
<td>0.02</td>
<td>&lt;0.02</td>
<td>10.7</td>
<td>0</td>
</tr>
<tr>
<td>DP980-1.4</td>
<td>0.09</td>
<td>0.23</td>
<td>0.12</td>
<td>0.58</td>
<td>0.10</td>
<td>&lt;1</td>
<td>42</td>
</tr>
</tbody>
</table>

9.3.3 In-Situ High Energy X-Ray Diffraction (HEXRD)

**HEXRD Experimental Configuration**

Haas et al. and Zhang et al. provided a comprehensive overview of the wide angle X-ray scattering diffraction configuration at the 1-ID beamline at APS [256, 257]. For this experiment, a monochromatic beam of X-rays at an energy of 71.68 keV (\(\lambda = 0.1731 \text{ Å}\)) with a cross section of 130 × 130 \(\mu\text{m}\) was used. The beam transmitted through the center of the gauge section of the specimens, with the beam propagation direction normal to the plane of the sheet. Considering the fine grain structure of each steel and the large beam interaction volume, \(i.e.,\) the beam cross section times the sheet thickness, the synchrotron beam interacted with hundreds of thousands of individual grains. Debye-Scherrer rings were detected using the “Hydra” detector array, which was composed of four GE Angio area detectors [258]. Each detector in the array was arranged to capture diffracted rings from either the LD or transverse direction (TD) of the tensile specimen. Figure 9.1(a) shows a schematic of the in-situ HEXRD geometry. HEXRD data were captured
prior to and throughout deformation to fracture. However, to isolate the moments where strain was
nominally homogeneous, only the data collected up to the end of uniform elongation are presented here.

**HEXRD Line Profile Analysis**

The GSAS-II software package was used to reduce the two-dimensional data from each detector into
one-dimensional “lineouts” [259]. First, the position of each area detector was calibrated using CeO$_2$
(NIST SRM 674b). Next, the intensity data from each detector were radially integrated. Two sets of
integration parameters were used for determining (i) phase volume fractions and (ii) stresses on
individual-phase constituents. For (i), all four detectors were integrated over a scattering vector
(*i.e.,* $Q = 4\pi\sin\theta/\lambda$, where $\theta$ is the Bragg angle in degrees) of 2.3 to 7.8 Å$^{-1}$ and an azimuth angle ($\phi$)
range of 50$^\circ$ about the LD or TD. For (ii), only the two detectors containing diffraction rings from the LD
were integrated, using a wider $Q$ range of 2.3 to 8.6 Å$^{-1}$ and a narrower $\phi$ range of 10$^\circ$ about the LD. In
both cases, the integration was performed over a range of $\phi$, rather than over the entire cone of the
diffraction space (*i.e.,* $0 < \phi < 360$) because the gaps between the four flat panel detectors would have
biased the comparison in the heights of peaks with various $Q$ positions. For instance, for reflections at
lower $Q$ positions, the peak encountered a lesser fraction of detector relative to the gap or bezels between
the detectors, which would have the effect of reducing the measured peak intensity.

Figure 9.1(b) shows an integrated lineout of QP980-1.0 using the (i) integration parameters. Two
crystal structures were indexed from the integrated lineouts of QP980-1.0 and QP1180-1.0:
FCC ($a_\gamma \approx 3.55$ Å) and BCC ($a_\alpha \approx 2.86$ Å). The FCC peaks were created by austenite, while the BCC
peaks were created by a combination of several phases/microconstituents including ferrite, martensite, and
perhaps bainite, as each have similar lattice parameters and cannot be resolved in the setup utilized here.
To determine the austenite volume fraction at various stages of deformation, the integrated intensities of
the BCC and FCC peaks were compared after Cullity [76]. The amount of new martensite that was formed
because of DIMT ($f_{\alpha'}^\varepsilon$) was calculated based on the austenite content before deformation ($f_{\gamma}^\varepsilon=0$) and after
deformation ($f_{\gamma}^\varepsilon$) (Equation 9.2):

$$f_{\alpha'}^\varepsilon = f_{\gamma}^{\varepsilon=0} - f_{\gamma}^\varepsilon$$  (9.2)

The lineouts created using the (ii) integration parameters were used to determine the lattice strains and
stresses on individual-phases relative to the loading direction. First, the lattice strains ($\epsilon_{\varepsilon}^{hkl}$) were
calculated based on the lattice spacing ($d_{\varepsilon}^{hkl}$) using Equation 9.3 [76, 245]. The lattice spacing prior to
deformation ($d_{\varepsilon=0}^{hkl}$) was determined empirically from the HEXRD data of an unloaded specimen:

$$\epsilon_{\varepsilon}^{hkl} = \frac{d_{\varepsilon}^{hkl} - d_{\varepsilon=0}^{hkl}}{d_{\varepsilon=0}^{hkl}}$$  (9.3)
Due to the HEXRD diffraction geometry and the (ii) integration parameters, the calculated lattice strains were nominally parallel to the LD, i.e.: $e_{11}^{hkl}$. Elastic stresses normal to the LD for individual planes of FCC and BCC-indexed phases, i.e., $\sigma_{\gamma,11}$ and $\sigma_{\alpha,11}$, were estimated based upon Hooke’s law using Equation 9.4 and Equation 9.5 [245]:

$$
\sigma_{\gamma,11} = E_{\gamma} e_{\gamma,11}^{311} \quad (9.4)
$$

$$
\sigma_{\alpha,11} = E_{\alpha} e_{\alpha,11}^{211} \quad (9.5)
$$

The $\gamma\{311\}$ and $\alpha\{211\}$ peaks were selected for analysis because they are known to exhibit strains similar to the bulk strains of BCC and FCC materials [245, 260, 261]. The diffraction elastic moduli of austenite and ferrite, $E_{\gamma}$ and $E_{\alpha}$, were assumed to be equal to 200 and 210 GPa, respectively [245].

To quantify lattice microstrain, which is known to be proportional to the total dislocation density [93], the (ii) lineouts were analyzed using X-ray diffraction line profile analysis (XRD-LPA). The basis for XRD-LPA is that lattice strain broadening is strongly $\{hkl\}$-dependent. Thus, the lattice microstrain or dislocation density can be assessed based on the slope of the peak breadth, $\Delta K_{hkl}$, versus the diffraction vector, $K_{hkl} = 2\sin\theta/\lambda$. For elastically isotropic materials, the relation of $\Delta K_{hkl}$ versus $K_{hkl}$ is given by the *Conventional* Williamson Hall (CWH) equation [93, 94], where $D$ is the crystallite size, $b$ is the magnitude of the Burgers vector and $\rho$ is the dislocation density:

$$
\Delta K_{hkl} = \frac{0.9}{D} + K_{hkl}(0.263b\sqrt{\rho}) \quad (9.6)
$$

Prior to XRD-LPA analysis, the instrumental broadening associated with the 1-ID diffraction geometry was removed from the experimental peak breadths using Gaussian correction [262]. The peak breadth appeared to increase nonlinearly as a function of $K_{hkl}$. Nonlinear $\{hkl\}$-dependence is commonly associated with strain anisotropy [94–96, 99], and it is suggested that the *Direct Fitting* Williamson-Hall (DF-WH) approach, proposed by Takaki [99], could enhance the reliability of the strain measurements. The DF-WH method proposes that strain anisotropy can be corrected using the diffraction Young’s modulus ratio ($\omega_{hkl}$), which relates to the diffraction Young’s modulus of each reflection according to Equation 9.7:

$$
\Delta K = \frac{0.9}{D} + \frac{\varepsilon_{DF} K_{hkl}}{\omega_{hkl}} \quad (9.7)
$$

where $\varepsilon_{DF}$ is the average true microstrain contained within the X-ray interaction volume. The DF-WH method determines $\omega_{hkl}$ based on iterative evaluation of Equation 9.7 to determine the closest linear fit; for this study, 500 iterations were performed over the bounds proposed by Takaki *et al.*[99]: $0.5 < \omega_{h00} < 1.0$, where $\omega_{hkl}$ was computed from Equation 9.8 [99]:

$$
\frac{1}{\omega_{hkl}} = 3.83(1 - \frac{1}{\omega_{h00}})H^2 + \frac{1}{\omega_{h00}} \quad (9.8)
$$
The orientation parameter, $H^2$, was determined by Equation 9.9 [94]:

$$H^2 = \frac{(h^2k^2 + k^2l^2 + l^2h^2)}{(h^2 + k^2 + l^2)^2}$$

For a more thorough description of the XRD-LPA, an example analysis is provided in Appendix B. The data for that appendix were created at Colorado School of Mines using a lab diffractometer, rather than the HEXRD data generated at the 1-ID beamline at APS. However, the DF-WH analysis procedure is similar.

### 9.3.4 Post-Fracture Characterization

To complement the bulk diffraction and mechanical testing data, spatially-resolved EBSD maps of QP980-1.0 were recorded using a JEOL JSM-7000F Field Emission Scanning Electron Microscope (FESEM). Quantitative analysis of the EBSD data was performed using the MTEX software package (version 5.4.0) [102]. Crystallographic texture in the BCC phases was assessed with inverse pole figures plotted relative to the loading direction (LD-IPFs). To assess local lattice rotation associated with geometrically necessary dislocations (GNDs), kernel average misorientation (KAM) analysis was performed for fifth-order neighbors with a threshold of 10 degrees. Quantitative KAM analysis was not performed on the austenite, because the austenite volume fraction was too low after deformation to achieve satisfactory grain statistics.

SEM fractographs were recorded for every material in the secondary electron imaging mode with an FEI Quanta 600 environmental scanning electron microscope (ESEM). Using low-magnification fractographs, reduction in area was measured using ImageJ [263]. For high-magnification fractographs, “watershed” image analysis was performed to determine the size distribution of ductile dimples using the ImageJ Interactive Watershed plugin [264]. Approximately 500 to 1200 dimples were measured per test condition.

### 9.4 Results

In this section, bulk tensile testing is coupled with *in-situ* HEXRD to isolate the roles of various phases in dictating phase evolution and mechanical properties over a range of temperatures. First, the bulk tensile mechanical properties of QP980-1.0, QP1180-1.0, and DP980-1.4 are discussed. Next, the HEXRD data are used to resolve the strains and stresses on individual constituent phases, leading to an improvement of the OC model. The potency of TRIP as a strengthening mechanism is linked to DIMT kinetics though the application of DF-WH lattice microstrain analysis. Finally, lattice rotation and damage due to microvoid coalescence are quantified *ex-situ* using electron microscopy.
9.4.1 Bulk Mechanical Response

True stress ($\sigma$) versus true strain ($\varepsilon$) curves for QP980-1.0, QP1180-1.0, and DP980-1.4 deformed at temperatures of 25, 100, 175, and 250 °C are plotted in Figure 9.2(a-c). The tensile behavior of QP980-1.0 and QP1180-1.0 appeared to be strongly temperature dependent. In the QP980-1.0 and QP1180-1.0 steels, the specimens deformed at 250 °C exhibited the highest uniform elongation (UE) and toughness (calculated from the area under each engineering stress-strain curve, $E = \int_{\varepsilon_{eng}}^{\varepsilon} \sigma_{eng} \, d\varepsilon_{eng}$), while those deformed at 100 °C exhibited the lowest toughness. The tensile behavior of the DP980-1.4 steel was similar regardless of test temperature. The tensile behavior (UTS, UE, and E) is tabulated for each steel and deformation temperature in Table 9.3. The yield stress of each steel was qualitatively observed to be insensitive to temperature, and was on the order of 600, 710, and 1045 MPa for the QP980-1.0, DP980-1.4, and QP1180-1.0 steels, respectively.

Table 9.3 Tensile Mechanical Performance for Each Steel and Temperature

<table>
<thead>
<tr>
<th>Steel</th>
<th>Temperature °C</th>
<th>UTS MPa</th>
<th>UE pct.</th>
<th>$E = \int_{\varepsilon_{eng}}^{\varepsilon} \sigma_{eng} , d\varepsilon_{eng}$ MJ m$^{-3}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>QP980-1.0</td>
<td>25</td>
<td>955</td>
<td>18.9</td>
<td>168</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>904</td>
<td>15.6</td>
<td>131</td>
</tr>
<tr>
<td></td>
<td>175</td>
<td>1015</td>
<td>24.0</td>
<td>228</td>
</tr>
<tr>
<td></td>
<td>250</td>
<td>1136</td>
<td>25.8</td>
<td>260</td>
</tr>
<tr>
<td>QP1180-1.0</td>
<td>25</td>
<td>1281</td>
<td>8.9</td>
<td>107</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>1288</td>
<td>6.1</td>
<td>72</td>
</tr>
<tr>
<td></td>
<td>175</td>
<td>1364</td>
<td>7.8</td>
<td>98</td>
</tr>
<tr>
<td></td>
<td>250</td>
<td>1471</td>
<td>17.0</td>
<td>229</td>
</tr>
<tr>
<td>DP980-1.4</td>
<td>25</td>
<td>1088</td>
<td>7.9</td>
<td>78</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>1101</td>
<td>9.6</td>
<td>96</td>
</tr>
<tr>
<td></td>
<td>175</td>
<td>1105</td>
<td>10.8</td>
<td>110</td>
</tr>
<tr>
<td></td>
<td>250</td>
<td>1125</td>
<td>9.8</td>
<td>99</td>
</tr>
</tbody>
</table>

To understand the temperature dependence of the ductility and toughness of QP980-1.0 and QP1180-1.0, Figure 9.2(d-f) shows $n_i$ plotted against $\varepsilon$. DP980-1.4 exhibited an $n_i$ that decreased as a function of $\varepsilon$, which was consistent with materials that do not show the TRIP-effect. Meanwhile, QP980-1.0 and QP1180-1.0 exhibited an $n_i$ that generally increased as a function of $\varepsilon$; this behavior has been attributed to the activation of TRIP [104, 113, 178]. Interestingly, for the QP980-1.0 and QP1180-1.0 steels, $n_i$ appeared to be greatly enhanced at 250 °C. The plots in Figure 9.2(d-f) also show the tensile instability criterion, $\varepsilon = n_i$. In accordance with the instability criterion, the onset of strain localization (necking) occurred approximately when the $\varepsilon$ versus $n_i$ curves intersected with the $\varepsilon = n_i$ line. For this reason, the UE was maximized when $n_i$ was relatively large at high true strains. For instance, the maximum UE and toughness was achieved for the QP980-1.0 and QP1180-1.0 at 250 °C because $n_i$ was
Some serrations in the $n_i$ curves were apparent for every steel. Similar serrations have been attributed to a combination of intermittent TRIP and dislocation slip (e.g., by dynamic strain aging) [166, 198, 265].

Figure 9.2 Tensile-mechanical properties as a function of temperature (25, 100, 175, and 250 °C) for each steel. (a-c) True stress and (d-f) instantaneous strain hardening exponent ($n_i$) versus true strain curves for QP980-1.0, QP1180-1.0, and DP980-1.4, respectively. The dotted line in (d-f) represents the instability criterion, $\varepsilon = n_i$.

9.4.2 Resolving Martensitic Transformation Kinetics as a Function of Bulk Plastic Strain

It was hypothesized that the temperature-dependent strain hardening in the QP980-1.0 and QP1180-1.0 steels could be attributed to a shift in DIMT kinetics, and thus a shift in the strengthening contribution of the TRIP-effect. To investigate this phenomenon, the austenite content in the QP980-1.0 and QP1180-1.0 steels was measured with HEXRD. Figure 9.3(a,b) shows the austenite content as a function of true plastic strain. HEXRD revealed that each steel contained approximately 12 volume pct. austenite prior to plastic deformation ($f_{\varepsilon=0}$). As the QP980-1.0 and QP1180-1.0 steels were deformed at each temperature, the austenite volume fraction as a function of true strain ($f_{\varepsilon}$) decayed. The kinetics of DIMT appeared to be connected to the deformation temperature. For instance, DIMT appeared to be minimized at 100 °C and maximized at 250 °C.

To quantitatively assess the temperature dependence of DIMT, the HEXRD data were fit to the OC and BM models using bulk plastic strain as the independent variable. Both fitting approaches appeared to fit well for the QP980-1.0 and QP1180-1.0 steels at lower test temperatures, but not at higher
temperatures. Because a poor fit was achieved for both models at elevated temperatures, the BM model fits are illustrated in Figure 9.3(c-g), to lessen the possibility of “overfitting” the experimental data with an excessive number of iterated variables (that is, two variables for BM: \( p \) and \( k \); three variables for OC: \( \alpha \), \( \beta \), and \( n \)). The quality of the fits was assessed based on the maximum deviation between the model and the experimental data at a given tensile strain; a fit exhibiting a deviation greater than approximately 0.5 vol. pct. was considered poor. Interestingly, for another Q&P steel, Poling also encountered poor fit quality at elevated temperatures while using bulk strain in the OC model [7].

Upon primary iterations of the BM model fitting process, \( p \) converged for each test around one. This result corroborated the work of Samek et al., who argued that the auto-catalytic propagation of strain induced martensite in multiphase TRIP-assisted steels is negligible (\( p = 1 \)) [45]. Accordingly, during the subsequent iterations of the fitting process, \( p \) was fixed at a value of one, which led to the solutions of \( k \) shown in Figure 9.3(g). For the QP980-1.0 and QP1180-1.0 steels, the variation of \( k \) as a function of temperature suggested that the maximum stability of austenite occurred at a deformation temperature of 100 °C. In other words, the kinetics of DIMT reached a local minimum at 100 °C.

Because of the relatively long times associated with the in-situ HEXRD tests, the variation of \( k \) with temperature could be related to other austenite decomposition mechanisms, such as thermal decomposition to bainite [266], being active alongside of DIMT. To rule out the possibility of isothermal austenite decomposition as a mechanism for reducing the austenite content, particularly at the highest test temperature, the austenite content was measured over time in a QP980-1.0 specimen that was not deformed but simply held at 250 °C. The investigation indicated that austenite decomposition was minimal after 30 minutes at 250 °C in the as-received QP980-1.0; see Appendix C for details. However, the extent of isothermal decomposition in plastically-deformed austenite was not investigated, and would be an interesting area for future studies.

9.4.3 Individual-Phase Stresses and Stress Partitioning in the Context of the Martensitic Transformation

The observation that the stability of austenite in Q&P steels, and thus the toughness, is coupled to temperature with a trough-like response is not novel; for instance, Coryell et al. first revealed a similar trend for a different “QP980” steel [46]. However, to our knowledge, a robust mechanism to explain this behavior has not been confirmed in the literature [166].Previously, stress partitioning between phases has been identified as a mechanism by which DIMT kinetics can be controlled, particularly by tuning the hardness of ferrite and martensite [267]. Thus, it was prudent assess the individual stresses in the BCC and FCC phases as a function of temperature. This section aims to elucidate stress and strain partitioning.
using the HEXRD data, with the eventual goal of enhancing the strain-based DIMT kinetics models.

Shifts in the lattice parameter, determined by HEXRD, were used to determine the stresses on individual-phases along the LD during deformation. Figure 9.4(a-c) shows the stresses on each phase in the three steels. For every temperature, the BCC phases yielded at a lower bulk true strain and phase stress than the austenite. Furthermore, after the onset of plasticity in each phase, the individual stresses in the austenite were higher than in the BCC phases. This observation suggested that the austenite bore an outsized share of the bulk tensile stress. For both phases in every steel, increasing the temperature appeared to increase the apparent phase stress. This apparent increase occurred because the diffraction elastic moduli of austenite (\(E_\gamma\)) and ferrite/martensite (\(E_\alpha\)) were assumed to be constant as a function of temperature for the Hooke’s law calculation of the phase stresses. However, both moduli presumably decreased by nearly identical degrees as a function of increasing temperature, leading to overpredictions of the phase stress at above-ambient temperatures. Because the diffraction elastic modulus is difficult to measure experimentally, the weighted-average stress contribution technique after Harjo et al. [245] was used to calculate the normalized fraction of the stress borne by each phase. In this approach, the contributed stresses, \(\sigma_{i,\text{cont}}\), for each phase were individually calculated based on the phase volume fraction and the phase stresses (for example, \(\sigma_{\gamma,\text{cont}} = f_\gamma \sigma_{\gamma,\text{HEXRD}}\)). Then, the ratio of the contributed stresses for each phase relative to the sum of the contributed stresses for both phases, \(\sigma_{\gamma,\text{cont}} / (\sigma_{\gamma,\text{cont}} + \sigma_{\alpha,\text{cont}})\), was plotted in Figure 9.4(d,e). The relative stress contribution of each phase was related to the DIMT kinetics for each steel and deformation temperature. As the austenite content decreased after yielding, the stress contribution from austenite decreased. For instance, for the QP980-1.0 steel deformed at 250 °C, where the DIMT kinetics were rapid, the stress contribution from austenite decreased from 15 pct. during the onset of plastic yielding to 5 pct. at the UTS. This decrease suggested that as the DIMT progressed, the bulk mechanical properties in the steels were increasingly controlled by the properties of the BCC phases.

To elucidate the progression of stress partitioning as a function of ferrite content, the ratio of stresses in the austenite versus the BCC phases (\(\sigma_{\text{FCC}} / \sigma_{\text{BCC}}\)) was plotted for the QP980-1.0 and QP1180-1.0 steels in Figure 9.5. For all test conditions, once the sample deformed plastically, the ratio exceeded one, suggesting that stress was preferentially partitioned to the austenite. In general, the extent of stress partitioning to the austenite was higher in QP980-1.0 relative to QP1180-1.0. This difference was attributed to the presence of approximately 20 vol. pct. relatively soft intercritical ferrite in QP980-1.0, which likely held a disproportionately low fraction of the bulk stress. In QP980-1.0, the ratio tended to decrease as \(\varepsilon\) increased. Meanwhile, QP1180-1.0 showed a different behavior, with a ratio that tended to increase as \(\varepsilon\) increased. The root cause of this behavior was possibly due to changes in the work hardening rates of the constituent phases, which are discussed in greater detail in Section 9.4.4.
Figure 9.3 Austenite content as a function of true plastic strain for (a) QP980-1.0 and (b) QP1180-1.0. Burke-Matsumura (BM) model fits for (c and e) QP980-1.0 and (d and f) QP1180-1.0 separated by fit quality. A summary of the austenite stability parameter ($k$) determined from the BM model fitting is shown in (g). For all fits, an autocatalytic parameter ($p$) of one was selected, based on Samek et al. [45].
Figure 9.4 Individual-phase stresses for (a) QP980-1.0, (b) QP1180-1.0, and (c) DP980-1.4. The approximate yield points for each phase at 25 °C are labeled with vertical lines. The relative stress contribution of each phase was calculated for (d) QP980-1.0 and (e) QP1180-1.0 after Harjo et al. [245].

Figure 9.5 Ratio of stresses in the austenite to the BCC phases ($\sigma_{\text{FCC}}/\sigma_{\text{BCC}}$) for each deformation temperature. A horizontal line is plotted at a ratio of one. Ratios above one suggest preferential stress partitioning to the austenite, and likewise, ratios below one suggest stress partitioning to the BCC phases.
It was hypothesized that since stress was preferentially partitioned to austenite for each steel and test temperature, strain within the austenite could diverge from the bulk tensile strain. To test this hypothesis, it was prudent to estimate the phase-specific strains in austenite. Individual constituent properties, determined by nanoindentation after Cheng et al. [252], were used to estimate the strain in the austenite. Figure 9.6(a) describes the process; first, the austenite stresses measured with HEXRD were compared to a ε-σ curve generated by nanoindentation of an austenite grain within a Q&P steel of a similar composition and microstructure to QP1180-1.0. Then, the strain in the austenite was estimated based on a fit of the Hollomon equation against the stress-strain curve. The estimated strains in the austenite were plotted against the fraction of new martensite, and the curves were fit using the OC model (n = 2) for the QP980-1.0 and QP1180-1.0 steels in Figure 9.6(b,c). The fitting parameters that generated the best fit for each temperature and steel are provided in Table 9.4.

Based on the classic interpretation of the OC model [15], the OC fit parameters measured here did not show a clear correlation between the deformation temperature and the probability of a shear band intersection forming a martensite embryo. That is, β did not appear to be strongly temperature sensitive, as it ranged from 1.2 to 2.5 for the QP980-1.0 steel and 1.3 to 2.5 for the QP1180-1.0 steel. For reference, for a steel similar to QP1180-1.0 referred to as “QP3Mn”, Poling showed that β ranged from 0.7 to 1.4 over a temperature interval of 22 to 85 °C (n = 2) [7, 8]. Meanwhile, α appeared to be correlated to temperature, with a similar trend progressing as a function of temperature in both steels; α was high at 25 and 250 °C, while a local minimum was reached at 100 and 175 °C. The correlation of α against deformation temperature suggested that a lack of potential martensite nucleation sites (for example, shear bands) was responsible for the diminished DIMT kinetics at intermediate temperatures. A similar downward trend in α from 25 to 100 °C was reported by Poling [7, 8]. However, to our knowledge, an increase in α at higher temperatures has not been reported within the literature. Further analysis of the martensite nucleation site density within the austenite phase using microscopy could help to clarify the trend in α that was reported here.

Although QP980-1.0 and QP1180-1.0 exhibited qualitatively similar trends in α as a function of temperature, QP980-1.0 exhibited an overall higher α parameter. This difference may have been due to a different austenite composition for each steel. For instance, a higher local alloy content of the austenite in QP980-1.0 could have led to a lower stacking fault energy, a higher rate of shear band intersections, and thus a higher α parameter [15].

Using the austenite phase strain, rather than the bulk tensile strain, enhanced the fit quality of the OC model at elevated temperatures (175 and 250 °C). The improved fit was caused by the evolving stress partitioning to austenite as a function of strain and temperature, which led the bulk strain to poorly
represent the actual strain in the austenite phase. Despite the success of the model using the austenite phase strain, the OC model was still not a perfect representation of the DIMT behavior. For example, the fits for the QP980-1.0 steel at 175 °C and QP1180-1.0 at 100 °C exhibited relatively low $R^2$ values of 0.87 and 0.75. Systematic errors in the experimental method could exist; for instance, it was assumed that neither the austenite constituent phase properties nor the elastic moduli changed as a function of temperature. Follow on investigations to collect higher resolution constituent phase properties as a function of temperature would resolve those inaccuracies.

Table 9.4 Olson-Cohen (OC) Fitting Parameters ($n = 2$) for QP980-1.0 and QP1180-1.0

<table>
<thead>
<tr>
<th>Steel</th>
<th>Temperature (°C)</th>
<th>α</th>
<th>β</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>QP980-1.0</td>
<td>25</td>
<td>34.8</td>
<td>1.3</td>
<td>1.00</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>8.2</td>
<td>1.2</td>
<td>0.98</td>
</tr>
<tr>
<td></td>
<td>175</td>
<td>7.3</td>
<td>2.5</td>
<td>0.87</td>
</tr>
<tr>
<td></td>
<td>250</td>
<td>17.9</td>
<td>1.6</td>
<td>0.99</td>
</tr>
<tr>
<td>QP1180-1.0</td>
<td>25</td>
<td>5.1</td>
<td>2.5</td>
<td>0.97</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>1.6</td>
<td>2.5</td>
<td>0.75</td>
</tr>
<tr>
<td></td>
<td>175</td>
<td>3.1</td>
<td>1.3</td>
<td>0.98</td>
</tr>
<tr>
<td></td>
<td>250</td>
<td>5.5</td>
<td>1.7</td>
<td>0.99</td>
</tr>
</tbody>
</table>

9.4.4 Strengthening Contribution of TRIP

Given that the DIMT kinetics were characterized with exceptional resolution, it was prudent to correlate DIMT with the strain hardening behavior of each steel (Figure 9.7). After the onset of plastic deformation, the progression of DIMT was correlated to a linear increase in the bulk true stress, \( \sigma = \sigma_0 + K f^{\alpha'} \). Linear regression analysis was performed to evaluate the constant terms in this expression (\( \sigma_0 \) and \( K \)), and the results are summarize in Table 9.5. The stress at which the deformation-induced martensite began to form (\( \sigma_0 \)) appeared to be responsive to both microstructure and temperature, with the maximum \( \sigma_0 \) observed in the QP980-1.0 and QP1180-1.0 steel occurring at 175 °C. Meanwhile, the proportional constant (\( K \)) reached a maximum for both steels at 100 °C, and was relatively constant at 25, 175, and 250 °C. \( K \) was remarkably consistent between the steels at 25, 175, and 250 °C, suggesting that the the TRIP effect strengthened each steel to a similar extent as a function of the volume of deformation-induced martensite that formed. During the final stages of deformation before the onset of necking, the stress appeared to deviate slightly upward from the linear relation, which indicated that the potency of the TRIP-effect was enhanced at high stresses and strains.
Figure 9.6 Individual-phase properties of austenite grains in a Q&P steel after Cheng et al. [252] were implemented to estimate the FCC phase strain based on the phase stresses measured via high-energy X-ray diffraction (HEXRD). A schematic of the estimation process is shown in (a). Olson Cohen (OC) model fits for the FCC phase strain versus the deformation-induced martensite content for (b) QP980-1.0 and (c) QP1180-1.0.
Figure 9.7 Bulk true stress plotted against the amount of deformation-induced martensite, as measured in-situ with high-energy X-ray diffraction (HEXRD).

Table 9.5 Constants from the Linear Regression Analysis of DIMT versus Bulk Stress

<table>
<thead>
<tr>
<th>Steel</th>
<th>Temperature (°C)</th>
<th>K</th>
<th>σ₀</th>
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</tr>
</thead>
<tbody>
<tr>
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<td>47</td>
<td>830</td>
<td>0.99</td>
</tr>
<tr>
<td></td>
<td>100</td>
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<td>865</td>
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<td></td>
<td>175</td>
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<td></td>
<td>250</td>
<td>55</td>
<td>872</td>
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</tr>
<tr>
<td>QP1180-1.0</td>
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<td>53</td>
<td>1263</td>
<td>0.98</td>
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<td></td>
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<td>175</td>
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<td></td>
<td>250</td>
<td>49</td>
<td>1289</td>
<td>0.99</td>
</tr>
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</table>

Because the TRIP-effect strengthens materials by dislocation generation at austenite-martensite interfaces, as well as within the new deformation-induced martensite, the strengthening contribution of TRIP is directly connected to the dislocation density in the BCC phases. DF-WH analysis was performed on the HEXRD lineouts to calculate the BCC lattice microstrain, which is proportional to the dislocation density. BCC lattice microstrain is related to the bulk true plastic strain in Figure 9.8(a-c). Comparing the steels prior to deformation, the BCC lattice microstrain increased as the ratio of martensite to intercritical ferrite increased. (that is, DP980-1.4 < QP980-1.0-1.0 < QP1180-1.0). This relationship was sensible because martensite has a higher dislocation density than proeutectoid ferrite formed during intercritical annealing. In the early stages of deformation, the presence of intercritical ferrite facilitated rapid increases in lattice microstrain as a function of tensile strain, because the soft ferrite accommodated an outsized fraction of the bulk tensile strain. For instance, the steel with the greatest fraction of intercritical ferrite (DP980-1.4) exhibited the greatest increase in lattice microstrain as a function of true plastic strain. For all steels, increasing the deformation temperature appeared to increase the BCC lattice microstrain. In the austenite-containing QP980-1.0 and QP1180-1.0, this effect was easy to rationalize because DIMT was promoted at high temperatures, leading to the formation of deformation-induced martensite with a
high-dislocation density. In the austenite lacking DP980-1.4, the origin of the temperature sensitivity is less obvious. One explanation could be that dynamic strain aging mechanisms were increasingly active at elevated temperatures, which promoted the generation of new dislocations rather than the slip of existing dislocations, leading to an ultimately higher dislocation density and lattice microstrain. For all the steels, an additional contributing factor could be the increased activation of BCC slip systems at high temperatures. Further studies to resolve dislocation substructure and microstructure evolution as a function of temperature and strain may be useful for confirming the origin of this behavior.

Since dislocation interactions are the basis for strengthening in AHSS, it was imperative to consider the BCC phase stresses as a function of the BCC lattice microstrain. BCC phase stresses are plotted versus the BCC lattice microstrain in Figure 9.8(d-f). The yield stress of the BCC phases for each steel and temperature are plotted as horizontal dashed lines. The BCC phase yield stresses are a combination of the yield stress in ferrite, which was presumably low, and martensite, which was presumably high. These assumptions were confirmed in the data, as the BCC yield stresses were higher in steels with a greater ratio of martensite to ferrite. Interestingly, the BCC yield stress appeared to be more sensitive to temperature in steels with a smaller ratio of martensite to ferrite. This observation seems reasonable, because relative to martensite, ferrite is strengthened extensively by short-range mechanisms, which can be overcome by thermal activation at elevated temperatures [50].

Each steel exhibited unique stress-microstrain behaviors after yielding. For the Q&P steels, two stages of strain hardening were observed. Meanwhile, for the DP steel, only one stage of strain hardening was observed. For discussion, the stages of strain hardening are labeled I-III in Figure 9.8(d-f). In the early stages of plastic deformation in QP980-1.0 and DP980-1.4 (I), the lattice microstrain increased rapidly with minimal increases in the BCC phase stress. This rapid increase could be due to free dislocation generation and lack of dislocation interactions in intercritical ferrite, which had a long mean slip length. At the earliest stages of plastic deformation in QP1180-1.0 (II), the lattice microstrain appeared to briefly decrease with respect to the BCC phase stress. One possible explanation is that the application of stresses during the mechanical loading process caused the various BCC peaks (that is, martensite and ferrite) to overlap more closely, thereby reducing the overall peak breadth and the perceived lattice microstrain. As deformation progressed, the metastable austenite-containing steels exhibited an additional strain hardening stage (III) where the BCC phase stress increased steeply as the dislocation density increased. This steep increase suggested a potent strengthening effect of the increasing fraction of high-dislocation density in the new deformation-induced martensite. Interestingly, in stage III, the strengthening effect did not appear to be temperature sensitive. This makes sense because dislocation interactions contribute to strength as long-range barriers to dislocation motion, and so they have a relatively small thermal component; thus,
stress to overcome such interactions is not strongly thermally activated [50]. The use of spatially-resolved characterization techniques, such as transmission electron microscopy (TEM), are warranted to clarify the balance of dislocation generation via TRIP and/or other plasticity mechanisms (for example, Frank-Read sources).

Figure 9.8 BCC lattice microstrain plotted against true plastic strain (a-c) and BCC phase stress (d-f) for QP980-1.0, QP1180-1.0, and DP980-1.4. In (d-f), the nominal yield stresses of the BCC phases are labelled with horizontal lines for every test temperature. The stages of hardening in the BCC phases are labelled schematically, i.e. I-III.

9.4.5 Microstructure and Crystallographic Texture Evolution

To verify the trends observed in-situ, as well as to spatially-resolve the lattice strain and crystallographic texture evolution during deformation, EBSD maps for the QP980-1.0 steel were collected before testing and after fracture (the map was collected just outside the neck) at each temperature. In Figure 9.9(a), an image quality (IQ) plus phase map reveals the substructure and morphology of each constituent phase. Two morphologically distinct types of austenite were evident; austenite grains with an aspect ratio close to one, and much greater than one, were classified as “blocky” and “film” type, respectively. Meanwhile, BCC grains that were devoid of significant substructure prior to deformation were classified as ferrite, while BCC grains with a prevalent substructure were classified as martensite.

KAM maps, which show the magnitude of local lattice misorientation caused by the presence of geometrically necessary dislocations (GNDs), are plotted for the BCC and FCC phases in Figure 9.9(b,c). In the KAM map for the BCC phases, the ferrite appeared to exhibit a low initial KAM. Meanwhile, the
martensite exhibited a higher KAM due to its high GND density. The KAM in the austenite was greater than ferrite and less than martensite. The blocky austenite appeared to exhibit a lower KAM than film austenite. The difference in KAM between blocky and film austenite was possibly due to the higher interface-to-volume ratio of the film austenite, which accumulated a higher density of GNDs during Q&P processing and during DIMT.

To quantitatively describe the lattice strain evolution, KAM histograms for the BCC phases prior to deformation and after deformation at each temperature are shown in Figure 9.9(d). A bimodal distribution in the as-received condition clearly exemplified the low KAM of ferrite and the elevated KAM of martensite. After deformation at every temperature, the KAM increased, and the distributions appeared to become Gaussian, suggesting that the differences in KAM in ferrite and martensite were reduced after deformation. This evolution was consistent with the interpretation of the two-stage hardening behavior of QP980-1.0 discussed previously; after the intercritical ferrite was sufficiently strain hardened such that the GND density was similar in ferrite and martensite, slip progressed simultaneously in both phases. Thus, in the uniformly-elongated sections of each specimen, the GND densities of both phases were similar.

Considering temperature, the trend in the KAM generally matched the trend in BCC lattice microstrain (Figure 9.8(a)). For instance, the specimens deformed at 175 and 250 °C achieved the highest uniform elongation (Figure 9.2(a)), the highest BCC lattice microstrain (Figure 9.8(a)), and the highest average BCC KAM (Figure 9.9(d)). These results corroborated the merit of the DF-WH technique for estimating dislocation density.

To resolve longer-range lattice rotation during deformation, loading direction inverse pole figures (LD-IPF) were plotted for each EBSD scan in Figure 9.9(e). Texture change was observed in the BCC phases, as α<011> directions became aligned with the LD. Meanwhile, weak texture change occurred in the austenite, as γ<111> directions became aligned with the LD. The coordinated development of parallel α<011> and γ<111> textures is consistent with the Kurdjumov-Sachs relationship, which has been frequently reported in TRIP-assisted steels after deformation [62, 153, 268].

Note that in tests with extensive DIMT (i.e., QP980-1.0-1.0 deformed at 250 °C), the volume of austenite in the scan was relatively small, so there was less certainty in the FCC LD-IPFs. For instance, the FCC LD-IPF for the QP980-1.0 steel deformed at 250 °C appeared to exhibit a γ<011> component in addition to a γ<111> component. However, this was suspected to be due to the uncertainty of the measurement, rather than a tangible shift in texture development at 250 °C. Ultimately, the test temperature did not appreciably alter the crystallographic texture development, as each test temperature revealed a seemingly even evolution of texture. This behavior suggested that variations in crystallographic texture evolution were not responsible for the enhanced properties of QP980-1.0 at elevated temperatures.
Figure 9.9 Kernel average misorientation (KAM) and texture evolution was assessed using electron backscatter diffraction (EBSD) for the QP980-1.0 steel as-received and after fracture at 25, 100, 175, and 250 °C. EBSD maps were collected just outside the necked region of each fractured tensile specimen. (a) IQ + phase map of the as-received QP980-1.0, indicating the presence of ferrite, martensite, and austenite. KAM maps of (b) the BCC-indexed phases, i.e., ferrite and martensite, and (c) the FCC-indexed austenite. (d) KAM distributions of the BCC-indexed phases, to compare the local lattice rotation for each specimen. The mean KAM for each distribution is plotted with a black vertical line. (e) Loading direction-inverse pole figures (LD-IPF) for the BCC and FCC-indexed phases. Units for the LD-IPFs are multiples of a random distribution.
9.4.6 Fractographic Analysis

SEM fractography was performed to assess local fracture ductility. In Figure 9.10(a), an example SEM fractograph of a fractured specimen (QP1180-1.0 deformed at 250 °C) is shown. Evidence of diffuse necking, where the cross section at fracture was significantly smaller than that of the uniformly-elongated section, was evident on every fracture surface. The pct. reduction of area for each tensile specimen was measured, and the results are summarized as a function of test temperature in Figure 9.10(b). At low temperatures, the combination of austenite, martensite, and ferrite in QP980-1.0 achieved the highest reduction in area; this result agreed with the trends in bulk ductility shown in Figure 9.2(a-c). At 250 °C, the reduction in area was greatly enhanced for each steel relative to the lower test temperatures. It was interesting that each steel achieved similar reductions in area despite their vastly different uniform elongations. However, the direct comparison of reduction in area between the steels was not flawless, because the as-received thickness of the DP980-1.4 steel was greater than that of the QP980-1.0 and QP1180-1.0 steels. This difference could have led to the development of different stress states in the neck, and thus different apparent fracture ductility.

![Figure 9.10 SEM fractography over a range of length scales.](image)

(a) Representative SEM fractograph of QP1180-1.0 deformed at 250 °C, with the orange overlay representing the fracture surface. (b) Measurements of reduction in area for each steel and temperature. (c) Ductile dimples, commonly associated with ductile microvoid coalescence, on the surface of the same specimen. (d) Automated dimple segmentation via a watershed algorithm was used to collect (e) average dimple area for each steel and temperature.
To assess damage at the mesoscale, higher magnification images of the fracture surface were acquired (Figure 9.10(c)). Dimples covered the entirety of every specimen, which was indicative that final fracture occurred by ductile microvoid coalescence. A watershed algorithm (Figure 9.10(d)) was implemented to measure the areas of hundreds of dimples on every fracture surface. The average dimple area did not appear to significantly change as a function of test temperature. However, the average dimple area appeared to be slightly influenced by the scale of microstructure features; for instance, the highly refined DP980-1.4 exhibited the smallest average dimple area for most temperatures. Meanwhile, the QP980-1.0 exhibited the largest initial ferrite grain size and the largest average dimple area. These observations suggest that for high-strength steel microstructures, damage evolution via ductile microvoid coalescence is controlled by the scale of microstructure features and interfaces [165], and not necessarily by extrinsic deformation conditions (for example, strain rate or temperature).

9.5 Discussion

9.5.1 Measuring Constituent Phase Properties in AHSS using HEXRD

It has been shown that the bulk properties of AHSS are fundamentally coupled with the properties of their individual constituent phases [153, 202, 239, 252, 269]. For instance, Du et al. showed that the bulk toughness and micromechanical deformation capacity a Q&P steel could be enhanced by tuning the plastic compatibility of tempered martensite and austenite [202]. In many studies, constituent properties are determined with micromechanical testing techniques, such as micropillar compression or nanoindentation, then the constituent properties are used to interpret and predict the bulk properties [270]. There are some constraints on the practicality of using micromechanical testing to inform microstructure design. First, micromechanical testing is laborious, and so most studies only measure the properties of a handful of grains. Thus, it is difficult to capture the effects of stochastically derivative characteristics of the microstructure, like crystallographic texture, grain morphology, and local chemistry. Second, it is difficult to capture the plastic compatibility and interactions between the phases, so the choice of composite model (i.e., iso-stress versus iso-strain) can strongly affect the predicted bulk properties [269].

With bulk mechanical testing coupled with in-situ diffraction, the behavior of the bulk is measured, with the added advantages that it is possible to resolve phase volume fraction and individual-phase properties for a vast number of grains. For example, for several Q&P steels, Harjo et al. implemented neutron diffraction with bulk mechanical testing to show that tempered martensite contributed an outsized fraction of the bulk stress, while ferrite contributed relatively little [245]. Meanwhile, Hu et al. showed that in a QP980-1.0 steel, austenite was harder than ferrite, but less hard than tempered and new martensite [202]. The data collected in the present work contained less resolution of the stresses in the
BCC phases, which was due to a slightly different test configuration. In this experiment, the $2\theta$ range was maximized to capture a larger number of FCC and BCC peaks for phase volume fraction and Williamson Hall analysis. By increasing the $2\theta$ range, the $2\theta$ resolution was decreased, which made it difficult to deconvolve the overlapping peaks of ferrite and martensite formed during the Q&P process, and new martensite formed during deformation. Thus, all BCC-indexed phases were treated as a single phase, so the aggregate data did not represent either martensite or ferrite, but a combination of both. Here, it was observed that the BCC phases yielded prior to the austenite in both Q&P steels at each temperature. Furthermore, the measured stresses on the austenite were higher than on the BCC phases. Despite the lack of resolution over individual constituent properties relative to Harjo et al. and Hu et al., the wider $2\theta$ range was valuable for this work because it enhanced the reliability of the phase volume fraction and DF-WH techniques, which were instrumental for interpreting the microstructure-property relationships in each steel.

The HEXRD experiment performed here elucidated critical details about constituent properties that would have been impossible to obtain with conventional ex-situ experiments. However, the data were limited in several ways. First, the lack of known diffraction elastic moduli for each phase and temperature necessitated the use of approximated values from the literature ($E_\gamma = 200$ GPa and $E_\alpha = 210$ GPa) [245]. This assumption likely led to an overprediction of the phase stresses at elevated temperatures, because the elastic modulus is known to decrease with increasing temperature. Fortunately, the use of the stress contribution and stress ratio alleviated the need for accurate absolute stress measurements, as the stresses of each phase were comparatively assessed with respect to each other, and the temperature effects were assumed to be equivalent for a given condition. The second limitation was that residual stresses, existing prior to mechanical testing, were not resolved. All stresses were measured assuming that the $2\theta$ position of each peak prior to tensile deformation represented a stress of zero. However, some residual stresses likely existed in the steels because of Q&P processing, e.g., due to the volume expansion linked with the martensitic phase transformation on cooling. A third limitation of the test configuration was that bulk texture could not be resolved, because the HEXRD data represented only one slice of reciprocal space relative to the LD axis. If the samples were rotated around the LD during deformation, then bulk texture could have been resolved in-situ. This modification would have been interesting because the crystallographic texture of each constituent likely plays a role in dictating slip activation (i.e., by Schmid’s law [114]), dislocation interactions, and DIMT. Full-field bulk texture measurements will be extraordinarily important for further developing our understanding of plasticity in Q&P steels. This type of measurement is particularly important because Q&P steels exhibit low austenite fractions and small austenite grains, which makes EBSD-based crystallographic texture measurements challenging.
9.5.2 Effect of Stress Partitioning on DIMT Kinetics

The HEXRD data became exceedingly useful when it was coupled with constituent stress-strain relationships determined with micromechanical testing (Figure 9.6). By estimating the strain in austenite using a combination of HEXRD and nanoindentation data [252], the goodness of fit of the OC model was greatly improved relative to the same model calculated with the bulk tensile strain. This improvement suggested that stress partitioning plays a decisive role in determining the kinetics of DIMT. For instance, for the QP980-1.0 steel deformed at 175 and 250 °C, a strong deviation in the stress ratio ($\sigma_{\text{FCC}}/\sigma_{\text{BCC}}$) was observed as a function of bulk tensile strain, suggesting that the extent of the stress partitioning changed as the samples deformed (Figure 9.5). Likewise, the bulk tensile strain provided a poor fit against both the BM and OC models (Figure 9.3), while the estimated strain in austenite provided a better fit (Figure 9.6). It is theorized that constituent phase strain data measured with HEXRD would be useful for other multiphase materials that use martensitic phase transformations for strengthening. This result demonstrates the growing utility of bulk mechanical testing coupled with in-situ diffraction analysis.

Constituent properties measured with HEXRD elucidated the influence of the ratio of martensite to ferrite on strain partitioning and DIMT kinetics. The QP980-1.0 steel, which exhibited a smaller ratio of martensite to ferrite, underwent greater partitioning of stress to austenite (Figure 9.5). This stress partitioning resulted in more rapid DIMT kinetics relative to the bulk tensile strain in QP980-1.0 when compared to QP1180-1.0 (Figure 9.3). The causal relationship between stress partitioning and DIMT kinetics would have been impossible to measure ex-situ, because the elastic phase strains and stresses would have been recovered upon unloading.

9.5.3 Influence of Dynamic Strain Aging

It was shown that the changes in the stress ratio ($\sigma_{\text{FCC}}/\sigma_{\text{BCC}}$) as a function of strain and temperature appeared to impede the ability of the strain-based kinetics models to correctly represent the kinetics of DIMT. For QP980-1.0, it is suspected that the decreasing stress ratio at elevated temperatures was due to activation of dynamic strain aging in the BCC phases, which had the effect of increasing the stresses in the BCC phases relative to the austenite.

The primary evidence for the activation of dynamic strain aging at elevated temperatures is that the amplitude of serrations in the instantaneous strain hardening exponent ($n_i$) appeared to increase in frequency and amplitude at higher temperatures for both QP980-1.0 and QP1180-1.0 (Figure 9.2). Due to the nature of the $n_i$-term, which was computed based on a derivative of data containing some intrinsic noise (i.e., from the load cell), some serrations in $n_i$ were expected, even in the absence of dynamic strain aging. Thus, it was prudent to confirm the presence of serrations in the true stress-strain data. Figure 9.11
shows true stress-strain curves for QP980-1.0, plotted over a range of true strains from 0.12 to 0.14. The data show the presence of serrations existing with greater amplitude and frequency at increasing temperatures. For instance, serrations were not obvious at 25 °C. Meanwhile, mild serrations were observed at 100 °C, and appeared to increase in quantity and amplitude at 175 and 250 °C.

The presence of serrations suggested that dynamic strain aging was increasingly active at elevated temperatures. This agreed with recent work by Mola et al., who observed the activation of serrated flow with increases in the deformation temperature over a range of 25 to 200 °C for a metastable austenitic steel [271]. Mola et al. proposed that dynamic strain aging in steels containing metastable austenite over the temperature range of 25 to 200 °C is caused by dislocation pinning at martensite/austenite boundaries, and accelerated carbon diffusion out of deformation-induced martensite to those boundaries [271]. It is noted that the carbon content in austenite was determined by HEXRD to be 1.16 weight percent (see Appendix C). Thus, the deformation-induced martensite nominally contained 1.16 weight percent carbon, while the carbon content of martensite predicted by the constrained carbon equilibrium model after Speer et al. is near zero [27, 272]. Thus, the argument by Mola et al. is sensible, because there was a clear driving force for carbon diffusion from the deformation-induced martensite to austenite, and carbon diffusion times in martensite are known to be comparatively short for the temperatures of interest here [271].

9.5.4 Competing Microstructure Development Processes at Elevated Temperatures

In addition to dynamic strain aging, numerous competing microstructure development processes were likely active during the elevated-temperature quasi-static tests, which could have influenced the chemical stability of austenite and the properties of the deformation-induced martensite. For instance, the duration of the plastic portion of the tensile test of QP980-1.0 at 250 °C was ∼35 minutes (2100 seconds). Quasi-static tensile tests at elevated temperatures can be considered akin to a tempering or “one-step” partitioning treatment during deformation; for instance, Yan et al. reported significant austenite retention and carbon enrichment (∼10 volume percent austenite having a carbon content of 0.88 weight percent) was reached during a one-step Q&P treatment of a 0.20C-1.58Si-1.55Mn steel after partitioning at 250 °C for 1000 seconds [273]. This would suggest that carbon partitioning from the deformation-induced martensite into the adjacent retained austenite could have increased the chemical stability of the remaining austenite in the elevated temperature tests of QP980-1.0 and QP1180-1.0. It is important to note that the carbon content of the austenite during mechanical loading could not be confirmed with the HEXRD data presented in this study, because the austenite lattice parameter shifted due to the imposed tensile stresses. The austenite carbon content during the static aging test of QP980-1.0 at 250 °C did not change.
significantly (see Appendix C), however, in the static aging condition, no deformation-induced martensite was formed during that test because no deformation was imposed. During the same static aging test, approximately one volume percent austenite was lost from the microstructure without an applied tensile strain. This suggested that some austenite may have decomposed to bainite during the test.

Figure 9.11 True stress-strain curves of QP980 at each temperature, plotted over a narrow strain range to show the increasing magnitude and number of serrations (marked with arrows) as a function of temperature.

The mechanical properties of the deformation-induced martensite may have dynamically changed via tempering during testing at 250 °C. For instance, dislocation recovery and extensive carbide precipitation have been reported during Q&P treatments similar to the times and temperatures investigated here [35], which could have the effect of changing the work hardening characteristics of the martensite [118]. Moving forward, understanding these competing microstructure development mechanisms during elevated-temperature deformation experiments will be critical for understanding the warm forming response of TRIP-assisted steels, by clarifying the origin of the temperature and strain-dependent stress partitioning that was reported here.
9.5.5 Adjusting Temperature During Sheet Forming to Tune the Stability of Austenite and the Mechanical Properties

The data presented here suggested that the deformation temperature affects the stability of austenite by a few mechanisms. First, the stress partitioning between the phases appeared to be temperature sensitive. For instance, at strains near the onset of strain localization, \( \sigma_{\text{FCC}}/\sigma_{\text{BCC}} \) decreased as a function of increasing temperature for QP980-1.0 (Figure 9.5). Hardening of the BCC phases by relative the austenite, perhaps by dynamic strain aging in the BCC phases, had the effect of partitioning strain to the austenite. For instance, at 250 °C, the estimated FCC phase strain at the UTS was \( \sim 0.25 \) for QP980-1.0 (Figure 9.6), while the bulk plastic tensile strain was \( \sim 0.20 \) (Figure 9.3(a)). This increased strain partitioning to austenite enhanced the kinetics of DIMT at high temperatures. Second, the number of martensite nucleation sites, as estimated by the OC model, appeared to exhibit a trough with respect to temperature (Table 9.4) that influenced the DIMT kinetics and the toughness of each steel.

These observations, coupled with previous work by the authors [167] and by Coryell et al. [46], suggest that temperature can be used in conjunction with strain rate to tune the stability of austenite during sheet forming operations. For example, the greatest DIMT and toughness was achieved for the QP980-1.0 and QP1180-1.0 steels at 250 °C, which would suggest that warm stamping could be used to maximize the formability of both steels. Likewise, if less formability was required to create a specific shape, then forming at 100 °C could be used to retard DIMT, thereby preserving the austenite so that the TRIP-effect could enhance the toughness of the formed part in service.

9.6 Conclusions

Austenite-containing AHSS, such as steels processed with the Q&P method, offer enhanced property combinations, due to the TRIP-effect and the controlled presence of several constituent phases (that is, martensite, ferrite, and austenite). The deformation responses of several AHSS with differing phase fractions were characterized by a combination of bulk HEXRD and spatially-resolved EBSD. This study constitutes the first concurrent measurements of stress partitioning and DIMT kinetics in Q&P steels for temperatures pertinent to industrial sheet forming processes. From this work, the following conclusions were made:

1. HEXRD resolved the DIMT kinetics for temperatures from 25 to 250 °C. Using constituent phase properties from the literature, along with individual-phase stresses resolved with HEXRD, we modified a deformation-induced martensitic transformation kinetics model to use the strain in austenite, rather than the bulk tensile strain. This modification enhanced the ability of the model to capture the transformation behavior of the Q&P steels.
2. The kinetics of DIMT were directly linked to the stress response of each steel (Figure 9.7). For instance, DIMT was inactive at 100 °C, leading to low toughness and strength, while the reactivation of DIMT at 250 °C greatly enhanced the toughness and strength. This observation may help to explain the ductility trough that has been reported in the literature. Tailoring the stress partitioning between the phases, for instance, by modifying the deformation temperature during sheet forming, could be used as a pathway to tune the bulk properties of austenite-containing AHSS. For instance, die temperatures could be increased to promote strain partitioning to austenite, leading to more complete DIMT, a more potent TRIP-effect, and thus greater formability.

3. Strain evolution in ferrite and martensite was tracked using a combination of HEXRD line profile analysis and EBSD-KAM analysis. The strain evolution was temperature-dependent, due to strain partitioning between the phases and the promotion of DIMT at certain temperatures. Strain evolution was used to rationalize the temperature-dependent work hardening that was observed in the bulk specimens.

9.7 Author Contributions

CBF: conceptualization, formal analysis, investigation, writing - original draft, and visualization. SRJL: data curation. BE, DS, CR, AS, and MMT: investigation. ALA, EJP, and JH: conceptualization, validation, and resources. JSP: investigation and software. AJC and KDC: conceptualization, funding acquisition, project administration, and supervision. All authors reviewed and edited the draft.

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auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344. Alec Saville was supported by the National Science Foundation Graduate Research Fellowship, USA, under Grant no. 2019260337.
The objective of this work was to elucidate the effects of temperature and strain rate on the deformation-induced martensitic transformation (DIMT) in Q&P steels. The motivation was that if DIMT could be controlled by tailoring the temperature and strain rate during sheet forming, then the mechanical properties during and after sheet forming could be enhanced.

10.1 Summary of Methods

To understand the effects of temperature and strain rate on DIMT and the mechanical properties, tensile tests were performed on several Q&P steels existing in two strength levels (980 and 1180 MPa). The kinetics of DIMT and microstructure evolution were tracked by a combination of techniques, including bulk X-ray diffraction (XRD) and electron microscopy. The tensile tests were performed over a range of strain rates from quasi-static ($10^{-4}$ s$^{-1}$) to dynamic ($10^{3}$ s$^{-1}$) and over a range of temperatures from 25 to 250 °C. In the preliminary stages of the work, represented by Experiments A and B, monotonic tensile tests were performed, and DIMT was tracked by ex-situ XRD in the as-received steels and after fracture. In the second phase of the work, represented by Experiments C, D, and E, the kinetics of DIMT were tracked with greater strain-resolution, using either interrupted tests with ex-situ XRD, or uninterrupted tests at synchrotron beamlines using in-situ high-energy X-ray diffraction (HEXRD). Where possible, line profile analysis was performed on the HEXRD data to gain additional insights about the deformation behavior of each constituent in the microstructure. For many test conditions, the bulk XRD data were complemented by spatially-resolved electron-backscatter diffraction kernel average misorientaiton (EBSD-KAM) analysis, which provided a semi-quantitative measurement of the density of geometrically necessary dislocations in each specimen.

10.2 Summary of Results

In Experiment A (Chapter 5), it was shown that DIMT was responsive to the orientation of tensile specimens relative to the rolling direction, for both strength levels of steel. Generally, the tensile performance was similar for each specimen orientation, but the DIMT kinetics were more rapid when the tensile axis was aligned with the rolling direction. Neither the amount of retained austenite existing prior to straining, nor the total amount of austenite transformed to martensite during deformation, were directly correlated to the strain hardening behavior of the steels. This suggested that the mechanical stability of
the austenite, rather than the volume fraction of austenite transformed to martensite, critically influenced
the deformation response.

In Experiment B (Chapter 6), DIMT appeared to be strain rate sensitive over the range of
$10^{-4}$ to $10^{-1}$ s$^{-1}$ for a QP980 steel. As the strain rate was increased, the extent of DIMT decreased. The
decreased DIMT kinetics at elevated strain rates had the effect of reducing the work hardening at high true
strains, which facilitated a more rapid onset of strain localization and fracture. Using a combination of
thermocouples and infrared thermography, it was determined that deformation-induced heating was
adiabatic for the strain rates of $10^{-2}$ and $10^{-1}$ s$^{-1}$. Based on this observation, it was hypothesized that
the deformation-induced heating could have been responsible for retarding the DIMT kinetics, by reducing
the thermodynamic driving force for DIMT. In Experiment B, as strain rate was increased from
$10^{-4}$ to $10^{-1}$ s$^{-1}$, deformation-induced heating caused the specimen temperature to rise by approximately
40 °C by the end of uniform elongation. Thus, a shortcoming of the experiment was that it was unclear
whether temperature, strain rate, or a combination of both independent variables, controlled the DIMT
kinetics. Experiment C (Chapter 7) clarified this ambiguity by adding an additional thermal-mechanical
condition; the additional specimens were tensile tested at a strain rate of $10^{-4}$ s$^{-1}$ with external resistance
heating, where the temperature profile was programmed to mimic the deformation-induced heat rise
measured at $10^{-1}$ s$^{-1}$. Interrupted tests of the specimens with and without external heating were
performed, with the conclusion that strain rate and temperature exhibited independent and dissonant
effects on the martensitic transformation. Increasing the strain rate from $10^{-4}$ to $10^{-1}$ s$^{-1}$ increased the
kinetics of DIMT. Meanwhile, increasing the temperature proportional to the rise associated with adiabatic
heating suppressed the martensitic transformation.

Since there was a strain rate effect on DIMT that was independent of temperature at intermediate
strain rates, it was prudent to explore the kinetics of DIMT at dynamic rates. Experiment D (Chapter 8)
aimed to resolve the DIMT behavior of the Q&P steels at dynamic strain rates using a Split-Hopkinson
Tension Bar (SHTB). In this study, specimens were deformed at a strain rate of $\sim 10^3$ s$^{-1}$, while a
high-speed synchrotron X-ray diffraction and radiography imaging system collected high-temporal
resolution data. The DIMT kinetics of both QP980 and QP1180 appeared to be similar during
deformation, to within the practical resolution of the HEXRD data. In both steels, the austenite fraction
after deformation was only slightly diminished in the uniformly-elongated gauge section, and was more
heavily diminished in a region inside the neck of the specimens after strain localization. Meanwhile, the
loading-direction stress on the $\alpha$(112) peak in each steel appeared to be similar, $\sim 1030$ MPa, to within the
practical resolution of the diffraction imaging system.
To provide a clearer understanding of the temperature-dependence of the DIMT kinetics, Experiment E (Chapter 9) described tensile tests that were performed at temperatures from 25 to 250 °C, while the DIMT behavior and the deformation response of the individual phases in QP980 and QP1180 were tracked using synchrotron HEXRD. The DIMT kinetics appeared to be linked to the deformation temperature, with a minimization of DIMT at 100 °C and a maximization of DIMT at 250 °C. When the DIMT data was compared against the bulk strains imposed during the tensile deformation, and against the strain in austenite estimated with HEXRD, the strain in austenite appeared to better describe the DIMT behavior using the Olson-Cohen (OC) model. The OC model suggested that the number of martensite nucleation sites, qualitatively assessed using the “α” term, were responsive to temperature. Meanwhile, the driving force for DIMT, qualitatively assessed using the “β” term, was not exceedingly responsive to temperature. The maximization of DIMT at 250 °C facilitated a profound increase in the work hardening rate at high true strains, which had the effect of improving the toughness. For instance, in QP980, the toughness increased from 168 to 260 MJ×m^{-3}, when the temperature was increased from 25 to 250 °C. This suggested that there exists a possibility to tailor sheet formability and as-formed properties by modifying sheet temperatures during forming.

10.3 Recommendations for Future Work

This work revealed several opportunities for potential areas of further research:

• Measuring the interactions of neighboring microstructure constituents, by spatially-resolved microscopy techniques such as transmission-electron microscopy (TEM), would be useful for interpreting the strain rate and temperature-dependence of DIMT. TEM could also be useful for measuring the density of shear band intersections, the primary sites for martensite nucleation, and also for measuring the stacking fault energy of austenite. Understanding the martensite nucleation site density as a function of effective strain, strain rate, and temperature would strengthen the interpretation of the OC model that was presented in this work.

• Measuring the properties of each phase/microconstituent within the Q&P steels as a function of temperature could be accomplished with hot-stage nanoindentation or micro-pillar compression testing. Elevated-temperature micro-mechanical testing would complement the bulk mechanical properties and HEXRD data that were presented here, and would allow for further improvement of the strain-based OC model for DIMT.

• To better understand the enhanced DIMT kinetics and toughness that were observed at elevated temperatures, experiments are needed to characterize the competing microstructure development
processes, such as austenite decomposition to bainite, dislocation recovery, carbon partitioning, and carbide precipitation, that occur during deformation at elevated temperatures. For instance, the influence of the strain/stress in austenite on the austenite decomposition kinetics would be relevant for interpreting the data presented in Chapter 9.

• In future high-resolution diffraction studies, such as those performed with synchrotron or neutron diffraction, it would be useful to distinguish between the ferrite, martensite, and bainite in the AHSS microstructures. These constituents could be distinguished using advanced XRD line profile analysis techniques like Rietveld refinement. Furthermore, resolving crystallographic texture with bulk diffraction techniques, like those available at the Los Alamos Neutron Science Center High-Pressure-Preferred Orientation powder diffractometer, would facilitate a greater understanding of the role of crystallographic texture on DIMT kinetics.

• Formability trials should be performed, where the forming temperature is tailored to modify the formability and as-formed properties of Q&P steels. Meanwhile, multi-step forming operations should be considered, because the DIMT kinetics could be tailored by modifying the temperature as a function of the effective strain. For instance, in the forming of a B-pillar component, two deep-drawing steps could be performed, separated by a several-minute hold time, so that the deformation-induced heat would be allowed time to dissipate. Allowing heat to dissipate could create a favorable austenite stability condition near the forming limit of the material, while allowing for supplemental lubrication of the sheet.

• The demand for sheet-type AHSS may be minimized by the adoption of battery electric vehicles, which are less reliant on frame lightweighting for enhanced energy efficiency. Thus, the temperature and strain rate dependence of DIMT should be explored in other emerging AHSS product segments, including in plate-type AHSS developed for impact and blast resistance.
REFERENCES


A.1 Yield Point Phenomena in QP980-1.4-B

To elucidate the contribution of TRIP to strain hardening, interrupted tensile tests were performed on QP980-1.4-B in Chapter 6. For a single specimen, austenite volume fraction was measured at various stages of deformation at each interruption. In this section, stress-strain behavior is compared for tensile specimens with different deformation pathways: (i) monotonically loaded, (ii) interrupted followed by immediate reloading, and (iii) interrupted followed by aging and then reloading. Sample (iii) was aged at room temperature for at least two hours while retained austenite was measured via X-ray diffraction (XRD).

A.1.1 Procedure

Sample (i) was monotonically loaded to failure. Sample (ii) was loaded in increments of two percent engineering strain, completely unloaded, and then reloaded; the process was repeated until the formation of a neck. Sample (iii) was loaded, unloaded, and then reloaded with the same procedure as (ii), except sample (iii) was fully-removed from the tensile frame after each incremental strain for XRD characterization of the constituent phases.

A.1.2 Effect of Deformation Pathway on Mechanical Behavior

In comparison to the monotonically loaded sample (i), interrupted testing of samples (ii) and (iii) did not drastically affect the yielding behavior. In all specimens, roundhouse yielding occurred at a high yield stress, due to the presence of tempered martensite in the microstructure [118]. Following yielding, the true strain hardening rate reached a minimum at an engineering strain of approximately 0.02, then increased with additional tensile strain until the onset of strain localization. Figure A.1 shows stress-strain curves for the entire interrupted tensile test of each sample.

Figure A.2 shows a more granular view of some distinct yield point effects that were observed in the interrupted tests. In sample (ii), a rounded yield point, followed by a gradual yield drop, was observed at all strain increments. In sample (iii), a rounded yield point of similar nature as (ii) was observed at true strains under five percent. After five percent strain, the yield points appeared to become less rounded and more serrated. The insets in Figure A.2 show a magnified view of each yield point.
The rounded yield points resembled those reported by Haasen and Kelly for pure single crystal FCC metals [194]; the proposed mechanism for a Haasen-Kelly yield point is dislocation rearrangements during elastic unloading. A more recent interpretation by Brown confirmed the Haasen-Kelly effect with emphasis of the role of dislocation dipoles as sources for slip [195]. Li et al. noticed similar yielding behavior during in-situ electron backscatter diffraction (EBSD) studies of martensitic transformations in a Q&P steel, but did not attribute the effect to a particular mechanism [193].

The serrated yield points observed in sample (iii) could be caused by static strain aging or pinning of mobile dislocations by interstitial carbon atmospheres [50]. Several researchers have observed dynamic strain aging in AHSS [155–157, 196], but the discovery of serrated yield point emergence during consecutive incremental strains is novel in the literature for Q&P steels. Because serrations consistent with strain aging appear only after tensile strains of several percent, it is hypothesized that strain aging occurs in the deformation-induced martensite. Deformation-induced martensite likely has a solute carbon content sufficient to cause strain aging because: (a) the partitioning process increases the carbon content of the parent austenite above the bulk alloy composition and (b) the new martensite has not been sufficiently tempered to form carbides.
Figure A.2 True stress-strain plots of QP980-1.4-B loaded (a) with interruption and immediate reloading and (b) with interruption and room temperature aging for several hours during XRD analysis. Unloading yield point effects are evident in both conditions, with (b) exhibiting more serrated behavior. The inset in (a) defines the stress increment ($\Delta \sigma_f$) and yield stress drop ($\Delta \sigma_y$).
Serrated yield points during reloading have also been attributed to strain incompatibility between phases with dissimilar properties (i.e., flow strength). For instance, Du et al. showed that partitioning temperature affected the character and amplitude of yield points in Q&P steels, with the most serrated and largest amplitude serrations occurring due to the presence of exceedingly hard martensite in the steel partitioned at the lowest temperature [239]. Using this interpretation of the behavior, perhaps tempering of the new martensite during aging led to a change in the mechanical properties of the martensite, and thus a change in the deformation inhomogeneity between the constituent phases. Furthermore, elastic stresses stored in the microstructure after unloading, e.g., due to plastic incompatibility, could have been alleviated during the aging period via dislocation creep.

A convenient method to quantify the stress increase and load drop caused by yield point phenomena is given by Geng and Nie for a magnesium alloy that exhibited similar yield point effects [274]. As shown in Figure A.2, the yield point can be quantified by the stress increment ($\Delta \sigma_f$) and yield stress drop ($\Delta \sigma_y$). Figure A.3 shows the evolution of the stress increment and the yield stress drop throughout tensile deformation. For both samples (ii) and (iii), the stress increment decreased linearly as tensile strain increased. Comparing samples (ii) and (iii), it appeared that aging increased the magnitude of the stress increment and modified the rate of change of the stress increment with respect to tensile elongation. Yield stress drop appeared to increase in both conditions with the application of tensile strain. However, there appeared to be a greater amount of scatter in the data, perhaps because serrations in the load cell readout were not fully resolved at the data acquisition rate of 10 Hz. Generally, serrations in sample (iii) at true strains of 5-15 percent exhibited the greatest yield stress drop, consistent with discontinuous yielding associated with strain aging.

### A.2 Yield Point Phenomena in a Metastable Austenitic Stainless Steel

In a related investigation at Colorado School of Mines by Thrun et al., the evolution of $\Delta \sigma_y$ was investigated as a function of aging time and stress history for a metastable austenitic stainless steel (AISI 304) [198]. In the early stages of deformation, while the steel contained only austenite, the yield points upon reloading were small and rounded. However, as martensite formed as a result of deformation, the yield points grew in magnitude.

Aging the specimens after unloading appeared to increase $\Delta \sigma_y$ (Figure A.4(a)). Thrun et al. suggested that this may have been due to strain aging of the steel, and thus a change in the deformation compatibility of the austenite and martensite phases. The extent of unloading, i.e., either partial or full unloading, also changed the progression of $\Delta \sigma_y$ as a function of applied strain (Figure A.4(b)). This observation is consistent with Du et al.’s interpretation of the the Bauschinger effect for multiphase alloys.
because partial unloading would impose smaller reverse plastic strains than full unloading \cite{239}, leading to a smaller $\Delta \sigma_y$ upon reloading.

Figure A.3 Strain dependence of (a) the stress increment ($\Delta \sigma_f$) and (b) the yield stress drop ($\Delta \sigma_y$) for QP980-1.4-B after being immediately reloaded (no age) and after being aged for 2 to 24 hours during XRD analysis.

Figure A.4 Yield stress drops ($\Delta \sigma_y$) for a fully austenitic stainless steel (AISI 304). (a) Shows the effect of room temperature aging hold time and (b) shows the effect of partial and complete unloading prior to immediate reloading. Reprinted with permission from Thrun et al. \cite{198} (see Appendix F for permissions).
APPENDIX B
AN EXAMPLE OF THE USE OF X-RAY DIFFRACTION LINE PROFILE ANALYSIS

A principal goal of this project was to quantitatively assess how strain is accommodated via a combination of dislocation motion and TRIP in third-generation AHSS, considering numerous deformation processing factors (i.e., effective strain and forming temperature) and microstructure factors (e.g., austenite stability, ferrite/martensite/austenite phase volume fraction, and phase deformability/hardness). However, it is challenging to decouple the contributions of dislocation motion and TRIP to bulk plastic strain accommodation. As a result, it is difficult to design AHSS microstructures for enhanced formability and strength. In this section, a pilot study is documented which aimed to test several X-ray diffraction (XRD) based analytical techniques that may help to measure: (i) microstrain accumulation in ferrite and austenite and (ii) DIMT, as a function of a given applied strain. To aid in the discussion and interpretation of these data, please recall the literature review presented in Chapter 2.6.

B.1 Interrupted Tensile Testing to Create Microstructures with Controlled Effective Strains

Standard sheet-type tensile specimens conforming to ASTM E8 [149] were removed from the rolling direction (RD) and transverse direction (TD) of a QP1180-1.6-A sheet. Interrupted tensile tests were performed using an MTS Alliance RT/100 load frame equipped with a two-inch extensometer. Tests were performed at an engineering strain rate of $8.1 \times 10^{-5} \text{ s}^{-1}$ and interrupted at 10 pct engineering strain. Load-displacement data generated from each tensile test were normalized by the dimensions of each tensile specimen to compute true stress as a function of true strain (Figure B.1(a)). Based on true stress and true strain, the instantaneous strain hardening exponent ($n_i$) was calculated after Equation 5.1.

Figure B.1(b) shows $n_i$ plotted as a function of true strain for the same tensile specimens as Figure B.1(a). For both specimens, $n_i$ reached a minimum at approximately 2 pct true strain, then increased with additional strain. This work hardening behavior suggested that the TRIP-effect contributed to strengthening. However, with the mechanical testing data alone, it is difficult to deconvolute the strengthening contributions of TRIP and dislocation interactions.
Figure B.1 True strain versus (a) true stress and (b) instantaneous strain hardening exponent ($n_i$) for QP1180-1.6-A tensile specimens deformed at a quasi-static rate along the RD and TD until 10 pct true strain.

B.2 X-Ray Line Profile Fitting and Correction for Instrumental Broadening

Based on the literature review provided in Chapter 2.6, it was determined that XRD-LPA could be implemented to resolve dislocation content as a function of imposed strain, with the goal of understanding the strengthening contributions of dislocation motion and the TRIP-effect in QP1180-1.6-A. Because the goal of this study was to identify a workable method for future XRD-LPA analysis using a lab diffractometer for mechanical testing specimens, as well as for transmission-mode diffraction data collected in-situ at a synchrotron X-ray source. Several diffractometer configurations were selected. First, a Cu source ($\lambda = 1.54$ Å) was used with a Bragg-Brentano focusing mirror. Second, a Mo source ($\lambda = 0.71$ Å) was used with a programmable divergence slit in the “fixed slit” operating mode. Both configurations created an incident beam spot size of approximately 5 by 10 mm and used a PANalytical Galipix solid-state area detector for measuring the diffracted X-ray spectra. The Galipix detector is water-cooled, Cd/Te based, and is thus optimized for detecting higher-energy (shorter wavelength) radiation than traditional area detectors (such as Si-based detectors). This allows the Galipix detector to safely detect “hard” X-rays emitted from a Mo source, as well as “soft” X-rays emitted from a more common Cu source. The key benefit of implementing XRD-LPA analysis with a higher-energy X-ray beam is greater penetration into the sample surface. An additional consequence of using a higher-energy source is that the X-ray peaks are diffracted over a narrower $2\theta$ range, according to Bragg’s law. It was theorized that this could limit the angular ($2\theta$) resolution of the XRD-LPA.
Before any scans were performed on the QP1180-1.6-A tensile specimens, diffraction patterns were collected for a LaB$_6$ reference material (NIST SRM 660a). The purpose of collecting scans of LaB$_6$ was to quantify the peak broadening introduced by the optical configuration of the diffractometer (i.e., the instrumental broadening is the broadening which is not caused by defects in the QP1180-1.6-A crystal lattice). Figure B.2 shows the experimental scans of the LaB$_6$ standard material for both the Cu and Mo sources. Here, rather than plotting the XRD spectra as a function of $2\theta$, the intensity is plotted versus the momentum transfer of scattering ($Q$):

$$Q = \frac{4\pi sin\theta}{\lambda} \quad (B.1)$$

By plotting XRD spectra using $Q$, the diffraction data can be compared between sample conditions and X-ray sources independent of $\lambda$ and $\theta$. By comparing parts (a) and (b) in Figure B.2, it is apparent that the magnitude of the instrumental broadening for each configuration was similar. Meanwhile, the $K\alpha_1$ and $K\alpha_2$ peaks were separated by a greater extent with the Mo source compared to the Cu source. To analyze the shape and intensity of the diffracted peaks, each peak was fit with a “Pseudo-Voigt” function using PANalytical HighScore Plus. A Voigt function fitting procedure was used, because it is capable of determining both the Gaussian and Lorentzian components of an X-ray spectrum (the mathematical basis is discussed by de Keijser et al. [275]). For all of the diffraction spectra discussed here, a line is plotted to describe the Pseudo-Voigt fit, along with a plot of the difference between the experimental data and the Pseudo-Voigt fit; a low intensity (nearly flat) difference plot is desired because it indicates that the Pseudo-Voigt fit accurately represents the data.

Scans were performed on four QP1180-1.6-A specimens (RD, $\varepsilon = 0$ pct.; RD, $\varepsilon = 10$ pct.; TD, $\varepsilon = 0$ pct.; and TD, $\varepsilon = 10$ pct.) using both the Cu and Mo sources. For brevity, the raw scan data along with the Pseudo-Voigt fits are provided for two sample conditions only: RD, $\varepsilon = 0$ pct., Mo source; and RD, $\varepsilon = 10$ pct., Mo source. The scans are provided in Figure B.3, and are representative of the general appearance of the pre-strain and post-strain scans for both orientations of QP1180-1.6-A and for both X-ray sources. It is noted that the nominal locations of the four lowest-index austenite peaks are labeled with dotted vertical lines; for both orientations and X-ray sources, the intensity of the austenite peaks diminished greatly after straining, indicative of DIMIT. By cursory inspection of the XRD scans, the ferrite/martensite/bainite peaks appeared to slightly broaden as a consequence of the imposed tensile strain. After subtracting the instrumental broadening from the scans, the strain-induced peak broadening will be analyzed in greater detail below.
Figure B.2 Q versus diffracted intensity curves of a LaB$_6$ standard (NIST SRM 660a) for (a) Cu and (b) Mo X-ray sources. The K$\alpha_1$ and K$\alpha_2$ peaks were separated more using the Mo X-ray source. The Pseudo-Voigt fitting procedure exhibited little difference between the fit curves and the experimental data, as shown by the difference plot.
Figure B.3 Q versus diffracted intensity plots of QP1180-1.6-A for (a) 0 and (b) 10 pct true tensile strain. The plotted diffraction scans were collected using a Mo X-ray source. The intensity of austenite peaks appeared to diminish after the application of tensile strain, suggesting the occurrence of DIMT. The dotted lines shows the nominal locations of austenite peaks (from low to high Q: \{111\}, \{002\}, \{022\}, and \{113\}).
By performing the Pseudo-Voigt fit on the LaB$_6$ reference material, the Gaussian and Lorentzian components of the instrumental broadening, $\beta_G^{\text{inst}}$ and $\beta_L^{\text{inst}}$, respectively, were determined for every sufficiently-intense LaB$_6$ peak. Because of the high number of LaB$_6$ peaks over the range of $2.75 < Q < 7.75$, $\beta_G$ and $\beta_L$ could be approximated as a function of $Q$ using polynomial functions for both X-ray sources. Figure B.4(a),(b) show the instrumental broadening for the Cu and Mo sources, respectively. For both configurations, $\beta_G^{\text{inst}}$ tended to decrease slightly, while $\beta_L^{\text{inst}}$ increased as a function of $Q$. However, the increase of $\beta_L^{\text{inst}}$ as a function of $Q$ was much larger for the Cu source, perhaps because of the longer X-ray wavelength and therefore the larger $2\theta$ angle of the scan. After determining the instrumental broadening for each source, the experimental data was “Voigt-corrected” to account for the instrumental broadening. Based on de Keijser et al. [275], Voigt-correction is performed, based upon the individual Gaussian and Lorentzian components:

$$\beta_L^{\text{corr}} = \beta_L^{\text{exp}} - \beta_L^{\text{inst}}$$

$$\left(\beta_G^{\text{corr}}\right)^2 = \left(\beta_G^{\text{exp}}\right)^2 - \left(\beta_G^{\text{inst}}\right)^2$$

Where $\beta_L^{\text{exp}}$ and $\beta_G^{\text{exp}}$ are the experimentally measured Lorentzian and Gaussian components of the peak breadth for QP1180-1.6-A, and $\beta_L^{\text{corr}}$ and $\beta_G^{\text{corr}}$ are the Voigt-corrected components of the peak breadth. The total corrected peak broadening for QP1180-1.6-A was calculated using an empirical formula that relates $\beta_L^{\text{corr}}$ and $\beta_G^{\text{corr}}$ after de Keijser et al. (see Equation 6 in reference [275]).

To compare the Voigt-correction process for the two X-ray sources, total peak breadth was plotted as a function of $Q$ for the experimental and Voigt-corrected line profiles of a representative specimen (QP1180-1.6-A, RD, $\varepsilon = 0$ pct strain) in Figure B.4(c),(d). For both the experimental and Voigt-corrected data, the Cu source generally measured greater peak breadths than the Mo source. This could be caused by several factors. First, the X-ray penetration depth and interaction volume is much larger for the higher-energy Mo source, so perhaps the scans represented a region of the microstructure with less metallographic polishing-induced residual strain. It is anticipated that the maximum depth of any polishing-induced strain is approximately the particle size of the polishing media used during the final polishing step, i.e. 1 $\mu$m. Second, the Cu source instrumental broadening exhibited a sharp increase in $\beta_L^{\text{inst}}$ at high $2\theta$ values; perhaps the Voigt-correction approach could not fully account for this sharp increase. For both sources, the measured peak breadth appeared to be only slightly larger than the instrumental broadening. The use of narrower Soller slits and receiving slits could reduce the instrumental broadening, thus enhancing the size-strain resolution at the consequence of reducing the number of X-ray
counts per second. For experiments performed at a synchrotron beamline designed for XRD-LPA, e.g., at the APS 1-ID beamline, the instrumental broadening is much smaller than the corrected peak breadth. This is because the X-ray beam is highly collimated, the spot size is small, and the area detector has a high density of pixels and a large area. For this reason, the XRD-LPA presented in this appendix has a lower resolution than the XRD-LPA presented in Chapter 9; thus, this appendix represents a "lower bound" resolution of the XRD-LPA technique.

B.3 Correction for Elastic Anisotropy

For all the sample conditions (for both X-ray sources before and after Voigt correction), the peak breadth appeared to be \{hkl\}-dependent and increased non-linearly as a function of Q (for example, see Figure B.4(c),(d)). The presence of non-linear \{hkl\}-dependence is commonly associated with strain anisotropy, and suggested that either mWH or DF-WH analysis could enhance the reliability of the size-strain analysis.

Two correction methods (mWH and DF-WH) were applied to account for the elastic anisotropy in ferrite. In both methods, broadening analysis was based on the Voigt-corrected integral breadth. With the mWH analysis, significant nonlinearity was observed in the \((\Delta K^2 - \alpha)/K^2\) versus \(H^2\) plots, which are necessary prerequisites for determination of \(q\) (the parameter which describes the screw/edge character of dislocations in the specimen). Thus, the mWH approach did not correctly adjust for the strain anisotropy of the specimens. Bakshi et al. recently showed that the use of integral breadth to describe peak broadening, rather than FWHM, could cause this issue for martensitic steels [92].

Compared to the mWH method, the DF-WH method generated more reasonable results, particularly for the Mo source. Figure B.5(a),(b) plots the fitting index \(R^2\) for iterative solutions of Equation 2.10, where \(\omega_{h00}\) was varied from 0.5 to 1. For most sample conditions, the best fit was achieved for \(\omega_{h00}\) values between 0.6 and 0.75; this result appeared to agree with the Kröner model, which predicted a \(\omega_{h00}\) of 0.734 [99]. In general, the fitting index was higher for scans created with the Mo source; this is likely due to the lower instrumental broadening correction error and higher interaction volume of the Mo source.

After the optimal \(\omega_{h00}\) was determined for each sample condition, the direct-fitted Williamson-Hall (DF-WH) plots were constructed (Figure B.5(c),(d)). The magnitude of the slopes (equal to the true microstrain, \(\varepsilon\)) of each plot created with the Cu source are relatively indistinguishable due to the low fitting index. However, the high fitting index of the DF-WH plots created with the Mo source allows for comparison of the microstrain in each specimen. For the pre-strain specimens, the measured true microstrain was 10.4 and 11.3 pct for the RD and TD specimens, respectively. These values are comparatively larger than some reported values of true microstrain that are reported for Q&P steels [213].
Figure B.4 (a),(b) Q versus the Gaussian component, the Lorentzian component, and the total peak breadth associated with the instrumental broadening of the Cu and Mo X-ray sources, respectively. (c),(d) Instrumental broadening was subtracted from the breadth of ferrite peaks (QP1180-1.6-A, RD, ε = 0 pct strain) using the Voigt-correction method after de Keijser et al. [275] for both the Cu and Mo X-ray sources. Note that the Voigt-corrected breadth of ferrite peaks in QP1180-1.6-A exhibited some significant differences for the two X-ray sources, perhaps due to uncertainty in the correction technique, differences in the depth of X-ray penetration and interaction volume, and differences in the diffracted intensities.
This difference could be caused by the use of $\beta$ rather than FWHM for the DF-WH analysis. Additionally, this difference could be caused by dislocation density modifications caused by processing; two possible examples are: (i) the potential formation of fresh (untempered) martensite during the final quench or (ii) the implementation of temper rolling (a process used to increase flatness, tailor the yield strength, and suppress potential yield point phenomena). For both the pre-strain and post-strain specimens, the microstrain was slightly higher along the TD versus the RD. However, it is unclear what influence crystallographic texture may exhibit in the interpretation of these data. After the application of 10 percent strain, the true microstrain in ferrite for each specimen increased by 2.3 and 2.0 pct for the RD and TD specimens, respectively.

B.4 Strain Accommodation by Multiple Mechanisms: Dislocation Motion and DIMT

In the previous section, it was concluded that microstrain accumulation as a result of tensile deformation was resolvable using the Mo source. To summarize, the true microstrain determined from the DF-WH method is plotted for each sample condition in Figure B.6(a).

Generally, XRD-based strain analysis requires scans with high angular resolution and signal-to-noise ratio, along with a wide Q range to resolve at least four to six peaks for each phase of interest. As a consequence of this requirement, the scans collected in this study for DF-WH analysis were suitable for measurement of the retained austenite volume fraction by the direct comparison method after Cullity [76]. The results of the retained austenite measurement are shown in Figure B.6(b). For every specimen, the Mo source measured a higher volume percent of retained austenite; perhaps the deeper X-ray penetration reduced the potential impact of polishing-induced DIMT. Both X-ray sources suggested that a nominally similar amount of DIMT occurred for specimens from the RD and TD.
Figure B.5 The direct fitting Williamson-Hall (DF-WH) method was applied to ferrite peaks from XRD scans of QP1180-1.6-A, for tensile specimens before and after straining along the RD and TD. (a),(b) show the $\omega_{h00}$ versus fitting index ($R^2$) plots for the Cu and Mo X-ray source, respectively. Generally, a higher fitting index was reached using the Mo source. (c),(d) show the DF-WH plots for the Cu and Mo sources, respectively, created using the $\omega_{h00}$ that generated the highest fitting index in (a),(b). The slope of the DF-WH plot is equal to the microstrain in the ferrite contained within the X-ray interaction volume. Note that the legends in (a),(b) also apply to the data in (c),(d).
Figure B.6 (a) Microstrain measured via the DF-WH method for each sample condition and X-ray source. Microstrain was determined as the slope of the DF-WH plot for each sample type as shown in Figure B.5. (b) Retained austenite content for each sample condition and X-ray source determined by the direct comparison method after Cullity [76] with temperature factors from ASTM E975 [82]. In (a), the number above each bar is the $R^2$ value of the linear fit; note that for every specimen, the Mo source generated a higher $R^2$ value. In all cases, the Mo source measured a slightly greater volume fraction of retained austenite, perhaps due to the larger interaction volume and thus a diminished effect of polishing-induced DIMT.
To determine whether any austenite decomposition mechanisms were active at the highest test temperature, a QP980-1.0 specimen was held at 250 °C for 30 minutes without a tensile load, while HEXRD data was collected. This condition was referred to as “static aging”. To compare the static aging condition against a sample deformed to failure, a plot of austenite fraction versus time for each test is given in Figure C.1. In the “deformation to fracture” specimen, the onset of plastic deformation occurred after approximately five minutes.

In the static aging condition, the austenite content in QP980-1.0 decreased from 13 to 12 vol. pct. after 30 minutes at 250 °C. This decrease suggested that some austenite decomposition mechanisms were active for the times and temperatures of interest in this study. However, this amount was small, and was likely not detectable with common measurement techniques, like ex-situ EBSD or X-ray diffraction. Meanwhile, in the specimen deformed to fracture, the austenite content decreased from 13 to 3 vol. pct. Thus, by comparing the two behaviors, it was inferred that during the tensile tests, most of the austenite transformed because of DIMT (∼9 vol. pct.), while a small fraction transformed because of austenite decomposition (∼1 vol. pct.).

The lattice parameter ($a_0$) of each austenite peak, $\gamma\{111\}$, $\gamma\{200\}$, $\gamma\{220\}$, $\gamma\{311\}$, $\gamma\{222\}$, and $\gamma\{331\}$ was calculated based on the d-spacing ($d_{hkl}$) determined from HEXRD after Equation C.1:

$$a_0 = d_{hkl} \times \sqrt{h^2 + k^2 + l^2} \quad (C.1)$$
The mean lattice parameter ($\bar{a}_0$) was calculated based on $a_0$ from the six peaks. Prior to any heating, $\bar{a}_0$ was determined to be 3.6060 and 3.6047 Å for QP980-1.0 and QP1180-1.0, respectively. The carbon content of the austenite ($C_\gamma$) was estimated based on Equation C.2 after Cullity [76]:

$$\bar{a}_0 = 3.555 + 0.044C_\gamma$$  (C.2)

Solving Equation C.2 suggested that $C_\gamma$ was equal to 1.16 and 1.13 weight percent for QP980-1.0 and QP1180-1.0. During the 30 minute static aging treatment at 250 °C, $\bar{a}_0$ increased by 0.00072 Å for QP980-1.0. This suggested that carbon partitioning to austenite was minimal during static aging, on the order of $\sim$ 0.016 weight percent. It is noted that because HEXRD is a bulk measurement technique, $\bar{a}_0$ and $C_\gamma$ describe the average of all the austenite grains within the interaction volume of the X-ray beam. Thus, some variations in $\bar{a}_0$ and $C_\gamma$ could have existed due to variations in local residual stresses and carbon content, but those variations could not be resolved without supplemental techniques (e.g., atom probe tomography).
This appendix exists to share some useful codes which were developed over the course of this dissertation.

D.1 Load Frame Compliance Adjustment Script

During mechanical testing of real materials with a load frame that is not infinitely rigid, both the test specimen and the load frame deform elastically. When displacement or strain is directly measured on the surface of the specimen, for instance, with either digital image correlation or with an extensometer, the elastic compliance of the load frame is not critical. However, when crosshead displacement is used to indirectly infer the engineering or true strain, this load frame deflection must be removed from the total displacement to determine the displacement of the sample.

Based on the assumption that the load frame always deforms elastically, and that the sample initially deforms elastically, the springs-in-series reciprocal addition relationship can be used to correct for frame compliance (Equation D.1):

\[
k_{\text{frame}} = \frac{1}{k_{\text{effective}}} - \frac{1}{k_{\text{sample}}} \quad (D.1)
\]

Where \( k_{\text{frame}} \), \( k_{\text{effective}} \), and \( k_{\text{sample}} \) are the spring constants of the load frame, total system, and mechanical test specimen, respectively, with units of load per length (e.g., kN/mm). \( k_{\text{effective}} \) can be measured empirically from the slope of the elastic portion of the load-displacement data. \( k_{\text{sample}} \) can be calculated based on the theoretical Young’s modulus of the steel \( E_{\text{theoretical}} = 200 \text{ GPa} \), the initial area of the sample \( (A_0) \), and the initial length of the sample \( (l_0) \), using Equation D.2:

\[
k_{\text{sample}} = E_{\text{theoretical}} \times \frac{A_0}{l_0} \quad (D.2)
\]

A MATLAB script was created to perform this analysis in an automated fashion. First, the script imports a set of sample data for a QP980-1.4-A specimen deformed at quasi-static strain rate at room temperature. The sample data, along with a copy of the MATLAB script is available at https://data.mendeley.com/datasets/4p6w99kxg5/1 [276]. After importing the data, the script asks the user to draw a box around the elastic portion of the load-displacement curve. Finally, the script will calculate the corrected load-displacement data. The script is included below. An example of the
functionality of the script is shown in Figure D.1.

%%% correctcomplaince.m
% C.B. Finfrock, Colorado School of Mines, rev. March 2022
% this script contains code that can adjust for load frame compliance in tension.
% it must be paired with the 'samplestressstraindata.mat' dataset, which is
% available at: data.mendeley.com

%% be mindful of units. this script is built for SI:
% force is in kN
% displacement is in mm

%%% initialize workspace and load data
clear; clc; close all
load('sample_load_disp_data.mat')
% this is a sample dataset for QP980.
% you can use your own data here.

%%% user inputs
theoretical_elastic_modulus_GPa = 200; %for steel
width=1.41;
thickness=1.069;
initial_area_mm2 = width*thickness;
initial_length_mm = 6;

%%% plot load-disp data
figure(1);
plot(Disp_mm, Force_kN, 'k')
hold on; xlabel('Disp (mm)'); ylabel('Force (kN)')

%%% draw a rectangle in a linear-elastic portion of the load-displacement curve
disp('Draw a rectangle in the elastic portion of the load-disp curve.')
disp('After you draw the rectangle, press any key to continue.')
roi = drawrectangle;
pause % this is a wait for user step that allows the user to draw
% and reposition the rectangle in the appropriate region.
% Press any key to continue.

%% fit the effective compliance of the system (e.g. the sample and load frame)

tf = inROI(roi,Disp_mm,Force_kN);
Disp_mm_elastic=Disp_mm(tf==1);
Force_kN_elastic=Force_kN(tf==1);

figure(1); hold on; plot(Disp_mm_elastic, Force_kN_elastic, 'r')

k_effective=polyfit(Disp_mm_elastic, Force_kN_elastic, 1);

%% check the linear fit

Force_kN_elastic_fit=polyval(k_effective, Disp_mm_elastic);
k_effective(:,2)=[]

% ***uncomment this plot to check***
% the fit should closely match the experimental data.
% figure(2);
% plot(Disp_mm_elastic, Force_kN_elastic_fit, 'k')
% hold on
% scatter(Disp_mm_elastic, Force_kN_elastic, 'r.')
% hold off

%% use springs-in-series reciprocal addition relationship to correct for compliance
% e.g., 1/k_effective=1/k_sample+1/k_loadframe
% the units for each compliance are kN/mm

k_sample=(theoretical_elastic_modulus_GPa*initial_area_mm2)/initial_length_mm %kN/mm
k_loadframe=1/((1/k_effective)-(1/k_sample))
Disp_mm_corr = Disp_mm-(Force_kN./k_loadframe);
Disp_mm_corr = Disp_mm_corr-Disp_mm_corr(1,1);

%% center displacement start at zero for the corrected data
startload=Force_kN(1,1);
startdisp=startload/k_sample;
Disp_mm_corr=Disp_mm_corr+startdisp;
Disp_mm_corr(1,1)=0;
Force_kN(1,1)=0;

%% plot the corrected data on the raw data
figure(1); plot(Disp_mm_corr, Force_kN, 'b')
legend('Raw Data', 'Elastic Region', 'Corrected Data', 'Location','southeast');
legend('boxoff')
xlim([-0.1, max(Disp_mm)+0.2])
ylim([0, max(Force_kN)*1.05])
hold off

---

Figure D.1 Plot generated by the compliance correction MATLAB script, using data for a quasi-static room temperature test of QP980-1.4-A performed using the DSI Gleeble 3500. The blue box in the elastic portion of the raw data curve was created by the user to measure the effective compliance of the system ($k_{effective}$).
D.2 Austenite Volume Fraction Computation Function

The Cullity method [76] was used at many stages of this dissertation to calculate the phase volume fraction of austenite \( (V_\gamma) \) from bulk X-ray diffraction scans. To automate the calculation, the following MATLAB function was developed as a robust tool for estimating \( V_\gamma \) from the integrated intensities of three ferrite peaks (\( \{002\}, \{112\}, \) and \( \{022\} \)) and three austenite peaks (\( \{002\}, \{022\}, \) and \( \{113\} \)). The code, along with two sample datasets, are available at https://data.mendeley.com/datasets/p5r92rmjng/1 [277].

```matlab
function [RA_sixpeak_pct] = RA_calc(exp_File, X_ray_source)

% this function imports a list of peaks measured using XRD.
% exp_File is an excel spreadsheet with a specific format created using line
% profile analysis in Malvern Panalytical’s HighScore software.

% this function uses three peaks of each phase (austenite and ferrite) to
% calculate the austenite volume fraction based on the Cullity integrated
% intensity method. If the function cannot find a peak, it assumes the peak
% intensity is zero. This can lead to anomolous volume fraction (the script
% will print a note if this occurs).

% two example data files are available on data.mendeley.com.

% right now, the function is set up to compute austenite volume percent
% measured with either a Copper Source, 'Cu', or a Molybdenum source, 'Mo'.

% Example to call this function using the example dataset:

% As-received material:
% [RA_sixpeak_pct] = RA_calc('QP980_zerostrain_peaks.xlsx', 'Cu')

% After some strain:
% [RA_sixpeak_pct] = RA_calc('QP980_12C_peaks.xlsx', 'Cu')
```

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%% Import the data
[No, Pos2, dspacing, Areacts2] = importpeaks(exp_File);
all = [No, Pos2, dspacing, Areacts2];

%% Search for Three Ferrite Peaks in List: {002}, {112}, {022}
nominal_dspacing_of_ferrite = [1.4307, 1.1688, 1.0126]';
rangelower = nominal_dspacing_of_ferrite - 0.01;
rangeupper = nominal_dspacing_of_ferrite + 0.01;
Interval = [rangelower rangeupper];
ii = 0;
for i = 1:length(Pos2)
    idx = find(dspacing(i,1) > Interval(:,1) & dspacing(i,1) < Interval(:,2));
    if isempty(idx) == 0
        ii = ii + 1;
        ferrite(ii,:) = all(i,:);
    end
end
numpeaksferrite = size(ferrite);
if numpeaksferrite(1,1)~= 3
    fprintf('could not find three ferrite peaks')
end

%% Search for Three Austenite Peaks in List: {002}, {022}, {113}
nominal_dspacing_of_austenite = [1.8012, 1.27393, 1.08658]';
rangelower = nominal_dspacing_of_austenite - 0.03;
rangeupper = nominal_dspacing_of_austenite + 0.03;
Interval = [rangelower rangeupper];
i = 0;
for i = 1:length(Pos2)
    idx = find(dspacing(i,1) > Interval(:,1) & dspacing(i,1) < Interval(:,2));
    if isempty(idx) == 0
        ii = ii + 1;
    end
end
austenite(ii,:) = all(i,:);
end
end

numpeaksaustenite = size(austenite);
if numpeaksaustenite(1,1)~= 3
    fprintf('could not find three austenite peaks')
end

%%% Determine X-ray wavelength based on X-ray source input
if X_ray_source == 'Cu'
    wavelength_ang = 1.5406;
end
end
if X_ray_source == 'Mo'
    wavelength_ang = 0.7107;
end

%%% Determine intensity ratios based on theoretical intensities
% see cullity or optical principles of the diffractions of X-rays.
% in this section, _a refers to austenite and _f refers to ferrite.

% determine sin(theta)/lambda, used for determining scattering factor in
% next step
sin_theta_over_lambda_f = sind(ferrite(:,2)./2)./wavelength_ang;
sin_theta_over_lambda_a = sind(austenite(:,2)./2)./wavelength_ang;

% determine atomic scattering factor (f), based on Cullity appendix 10
sinthetaoverlambdaforiron=[0:0.1:1.2];
f=[26,23.1,18.9,15.6,13.3,11.6,10.2,8.9,7.9,7,6.3,5.7,5.2];
appendixtenfit = polyfit(sinthetaoverlambdaforiron, f, 5);
f_f = polyval(appendixtenfit, sin_theta_over_lambda_f);
f_a = polyval(appendixtenfit, sin_theta_over_lambda_a);
% determine structure factor squared (|F|^2), based on Cullity 4-6
% for ferrite, this if |F|^2 = 4*(f^2)
% for austenite, this is |F|^2 = 16*(f^2)
Fsquared_f = 4.*(f_f.^2);
Fsquared_a = 16.*(f_a.^2);

% Input multiplicities
p_f = [6, 24, 12]';
p_a = [6, 12, 24]';

% Determine lattice parameter based on lowest index peak used for analysis
a_f_angstrom = ferrite(1,3).*(2);
a_a_angstrom = austenite(1,3).*(2);

% Calculate unit cell volume
v_f_angstromcubed = a_f_angstrom.^3;
v_a_angstromcubed = a_a_angstrom.^3;

% Input temperature factor, T = e^(-2M), based on ASTM E975, 2013, Table 2
T_f = [0.9172, 0.8784, 0.8413]';
T_a = [0.9467, 0.8962, 0.8601]';

% Calculate Lorentz Polarization Factor, (1 + cos^2(2)/(sin^2()*cos())
LP_f(:,1) = (1+(cosd(ferrite(:,2)).^2))./((sind(ferrite(:,2)./2).^2).*... cosd(ferrite(:,2)./2));
LP_a(:,1) = (1+(cosd(austenite(:,2)).^2))./((sind(austenite(:,2)./2).^2).*... cosd(austenite(:,2)./2));

% Calculate R-ratio, based on Cullity quantitative phase analysis, 12-5
R_f(:,1) = (1./(v_f_angstromcubed.^2)).*(Fsquared_f(:,1).*p_f(:,1).*... LP_f(:,1).*T_f(:,1));
R_a(:,1) = (1./(v_a_angstromcubed.^2)).*(Fsquared_a(:,1).*p_a(:,1).*...
\[ \text{LP}_a(:,1) \times \text{T}_a(:,1) \]

\%\% Compute RA Fraction from Six Peaks, based on intensity ratios
\[ \text{IoverR}_f(:,1) = \text{ferrite(:,4)}.\text{/R}_f(:,1); \]
\[ \text{IoverR}_a(:,1) = \text{austenite(:,4)}.\text{/R}_a(:,1); \]

\[ \text{RA\_sixpeak\_pct} = \text{mean(IoverR}_a)/(\text{mean(IoverR}_a)+\text{mean(IoverR}_f))*100; \]
end

function [No, Pos2, dspacing, Areacts2] = importpeaks(workbookFile, sheetName, dataLines)
\%\% Input handling

\% If no sheet is specified, read first sheet
if nargin == 1 || isempty(sheetName)
    sheetName = 1;
end

\% If row start and end points are not specified, define defaults
if nargin <= 2
    dataLines = [2, 12];
end

\%\% Set up the Import Options and import the data
opts = spreadsheetImportOptions("NumVariables", 16);

% Specify sheet and range
opts.Sheet = sheetName;
opts.DataRange = "A" + dataLines(1, 1) + ":P" + dataLines(1, 2);

% Specify column names and types
opts.VariableNames = ["No", "Pos2", "dspacing", "Var4", "Var5", "Var6", "Var7", ...
    "Var8", "Var9", "Var10", "Var11", "Var12", "Var13", "Var14", "Var15", "Areacts2"];
 opts.SelectedVariableNames = ["No", "Pos2", "dspacing", "Areacts2"];

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% Specify variable properties
opts = setvaropts(opts, ["Var4", "Var5", "Var6", "Var7", "Var8", "Var9", "Var10", "Var11", "Var12", "Var13", "Var14", "Var15"], "WhitespaceRule", "preserve");
opts = setvaropts(opts, ["Var4", "Var5", "Var6", "Var7", "Var8", "Var9", "Var10", "Var11", "Var12", "Var13", "Var14", "Var15"], "EmptyFieldRule", "auto");

% Import the data
tbl = readtable(workbookFile, opts, "UseExcel", false);

for idx = 2:size(dataLines, 1)
    opts.DataRange = "A" + dataLines(idx, 1) + ":P" + dataLines(idx, 2);
    tb = readtable(workbookFile, opts, "UseExcel", false);
    tbl = [tbl; tb]; %#ok<AGROW>
end

%% Convert to output type
No = tbl.No;
Pos2 = tbl.Pos2;
dspacing = tbl.dspacing;
Areacts2 = tbl.Areacts2;
end

D.3 Digital Image Correlation Hardware Trigger

The hardware components of the digital image correlation system developed for the ITC SP400 are discussed in Appendix E. The code for the Arduino microcontroller is provided here. Adjustments to the code can be made to change the acquisition rate and number of recorded images. The code can be uploaded to an Arduino board using the open-source Arduino Software integrated development environment, which is available for download at: https://www.arduino.cc/en/software.

// C++ code written by C.B. Finfrock
//
int i = 0;
int time = 0;
int triggerburstpulse = 0;
int aquisitionmode = 0;
int triggersinglepulse = 0;
int pulselength_millisecond = 0;
int FPS = 0;
int dwelltime_millisecond = 0;
int burstcount = 0;
int arm_analog_signal = 0;
int analog_arm_time_seconds = 0;
int loopindex = 0;

void setup()
{
  pinMode(2, INPUT);
  pinMode(4, INPUT);
  pinMode(7, INPUT);
  pinMode(5, OUTPUT);
  Serial.begin(9600);

  pinMode(A1, INPUT);
  pinMode(8, OUTPUT);
  pinMode(13, OUTPUT);
}

void loop()
{
i = 0;
FPS = 1;
burstcount = 400;
pulselength_millisecond = 500;
dwelltime_millisecond = (1000 / FPS - pulselength_millisecond);

triggersinglepulse = LOW;
triggerburstpulse = LOW;
arm_analog_signal = 0;
triggersinglepulse = digitalRead(2);
triggerburstpulse = digitalRead(4);
arm_analog_signal = digitalRead(7);
loopindex = 0;
if (arm_analog_signal == HIGH) {
    digitalWrite(5, HIGH);
    delay(500); // Wait for 500 millisecond(s)
    digitalWrite(5, LOW);
    delay(500); // Wait for 500 millisecond(s)
    digitalWrite(5, HIGH);
    delay(500); // Wait for 500 millisecond(s)
    digitalWrite(5, LOW);
    delay(500); // Wait for 500 millisecond(s)
    digitalWrite(5, HIGH);
    Serial.println("Analog trigger armed!");
    Serial.println(analogRead(A1));
    arm_analog_signal = digitalRead(7);
    while (loopindex == 0 && arm_analog_signal == LOW) {
        arm_analog_signal = digitalRead(7);
        if (arm_analog_signal == HIGH) {
            delay(1500); // Wait for 1500 millisecond(s)
            Serial.println("Analog trigger disarmed!");
            digitalWrite(5, LOW);
        }
    }
    if (analogRead(A1) >= 5 && loopindex == 0) {
        Serial.println("Analog trigger initiated!");
        for (i = 1; i <= burstcount; i += 1) {
            Serial.println(i);
            digitalWrite(i, HIGH);
        }
    }
digitalWrite(13, HIGH);
delay(pulselength_millisecond); // Wait for pulselength_millisecond millisecond(s)
digitalWrite(8, LOW);
digitalWrite(13, LOW);
delay(dwelltime_millisecond); // Wait for dwelltime_millisecond millisecond(s)
}
delay(50); // Wait for 50 millisecond(s)
if (i >= burstcount) {
    Serial.print(burstcount);
    Serial.println(" pulses sent!");
    Serial.println("Waiting for trigger initiation...");
    loopindex = 1;
    arm_analog_signal = HIGH;
    digitalWrite(5, LOW);
}
}
}
}
if (triggerburstpulse == HIGH || trigger_singlepulse == HIGH) {
    if (triggerburstpulse == HIGH) {
        Serial.print("Sending burst pulse in 3...");
        delay(1000); // Wait for 1000 millisecond(s)
        Serial.print(" 2...");
        delay(1000); // Wait for 1000 millisecond(s)
        Serial.println(" 1...");
        delay(1000); // Wait for 1000 millisecond(s)
        for (i = 1; i <= burstcount; i += 1) {
            Serial.println(i);
            digitalWrite(8, HIGH);
            digitalWrite(13, HIGH);
            delay(pulselength_millisecond); // Wait for pulselength_millisecond millisecond(s)
            digitalWrite(8, LOW);
            digitalWrite(13, LOW);
        }
    }
    if (triggersinglepulse == HIGH) {
        Serial.print("Sending single pulse in 3...");
        delay(1000); // Wait for 1000 millisecond(s)
        Serial.print(" 2...");
        delay(1000); // Wait for 1000 millisecond(s)
        Serial.println(" 1...");
        delay(1000); // Wait for 1000 millisecond(s)
        digitalWrite(8, HIGH);
        digitalWrite(13, HIGH);
        delay(pulselength_millisecond); // Wait for pulselength_millisecond millisecond(s)
        digitalWrite(8, LOW);
        digitalWrite(13, LOW);
    }
}
delay(dwelltime_millisecond); // Wait for dwelltime_millisecond millisecond(s)
}
delay(50); // Wait for 50 millisecond(s)
if (i >= burstcount) {
  Serial.print(burstcount);
  Serial.println(" pulses sent!");
  Serial.println("Waiting for trigger initiation...");
}
triggerburstpulse = LOW;
} else {
  Serial.print("Sending single pulse in 3...");
  delay(1000); // Wait for 1000 millisecond(s)
  Serial.print(" 2...");
  delay(1000); // Wait for 1000 millisecond(s)
  Serial.println(" 1...");
  delay(1000); // Wait for 1000 millisecond(s)
  Serial.println("1");
  digitalWrite(8, HIGH);
  digitalWrite(13, HIGH);
  delay(pulselength_millisecond); // Wait for pulselength_millisecond millisecond(s)
  digitalWrite(8, LOW);
  digitalWrite(13, LOW);
  delay(50); // Wait for 50 millisecond(s)
  Serial.println("One pulse sent!");
  Serial.println("Waiting for trigger initiation...");
  triggersinglepulse = LOW;
} else {
  delay(50); // Wait for 50 millisecond(s)
}
}
This appendix describes the design and preliminary testing of a custom stereo-DIC system. The system was optimized to resolve strain evolution with high spatial and temporal resolution on the ITC SP400 forming press. The ITC SP400 forming press was acquired by K. Clarke to support metal formability research at Mines. Thus, it was optimized to exist in a relatively compact footprint, while providing the control modes, instrumentation, and significant clamp and punch forces relevant for formability testing of high strength and thickness structural materials, such as third-generation AHSS. To complement the unique capability of the ITC SP400, a stereo-DIC system was desired to augment other instrumentation built into the system. For instance, although the frame is equipped with hydraulic and mechanical sensors to measure punch and clamp force and displacement, stereo-DIC was desired to characterize the strain distribution, effective strain, and strain state in three-dimensional formability test specimens.

E.1 Justification for Designing a Custom System

Numerous commercially available, “turn-key” solutions exist that greatly simplify the setup process for a stereo-DIC system. However, these systems are often costly and difficult to integrate into test configurations with complex geometries. For instance, due to the horizontal orientation of the test specimens in the ITC SP400, several lights, cameras, and structures must be oriented between the top of the ITC SP400 and the ceiling of the laboratory. Meanwhile, machine vision cameras and lenses are commercially available that can be implemented along with custom framing support structures to create very rigid, compact, precise, and economical stereo DIC systems.

One key motivation for designing a custom system was the potential forward-compatibility with future experimental configurations. For instance, because high-rate stereo-DIC analysis may be desired for experiments performed using the high-punch velocity MTS MetalForm 866 forming press and the high-displacement rate MTS 810 tensile frame, the system discussed here was designed to achieve a relatively high frame rate. Due to the experimental geometry, each of these systems would require a level of custom fabrication that could not be easily achieved with a “turn-key” system. An additional benefit of a custom system is that future alterations, such as the installation of an infrared thermal camera or a strobe-based lighting system could be simply integrated into the setup due to the configurable hardware and open-source software of the trigger system.
E.2 Basic System Requirements

Adherence to numerous DIC “best practices” is vital to maximize the resolution and repeatability of a stereo-DIC system. “A Good Practices Guide for Digital Image Correlation” by the International Digital Image Correlation Society greatly assisted in the design of the stereo-DIC system discussed here [278]. Based on this resource, some requirements for the system were:

1. A highly rigid mounting structure for the cameras and lenses, with three lockable translation axes (x, y, and z) and one lockable rotation axis for each camera.

2. Fiber optic-based lighting to illuminate the sample surface without exposing the cameras, lenses, and sample region of interest (ROI) to excess heat.

3. Two small form-factor cameras with global shutter technology (for reduced motion blur), high sensor efficiency, low noise, high dynamic range, acquisition rates up to 75 frames per second (FPS), and acceptable resolution.

4. Low distortion lenses with a minimum 50 mm depth of field, acceptable magnification, and low stand off distance (SOD). Here, it is noted that depth of field is intrinsically linked to the selected aperture size and SOD, and thus also to the nature of the lighting and camera settings used during the measurement.

5. Triggering capability for synchronous image acquisition within several milliseconds.

As explained in the previous section, an additional requirement was that the proposed system could fit in the two foot “position envelope” between the top of the ITC SP400 forming press and the ceiling of the laboratory. Thus, the dimensions of the camera, lighting, trigger, and mounting structure were to be minimized where possible.

E.3 Mounting Structure

A mounting structure was designed and built with extruded T-slot aluminum framing rails. The design intent was to provide the necessary degrees of freedom for each camera, while existing in a compact and rigid form. Because T-slot framing rails were used, the operator can slide the cameras along the x, y, and z axes then lock them into place using mechanical brakes. Meanwhile, the distance between the two cameras and the stereo angle (the angle between the imaging planes of both cameras) can be adjusted and locked using the two rotation stages. Schematics of the mounting structure attached to the top of the ITC SP400 forming press are shown in Figure E.1. The two selected cameras (discussed in detail in the next section) are shown in the schematics to describe the orientation of the cameras with respect to the forming press;
the cameras will point downward through the conical hole in the top of the forming press, such that the field of view (FOV) of the cameras will be focused on the horizontally-oriented formability specimen. Besides fixing the cameras at a selected position, the mounting structure also includes additional features for fastening other equipment, including fiber optic light tubes. If desired, a small thermal camera could be attached to the mounting structure between the two DIC cameras.

![Figure E.1](image)

Figure E.1 (a) Three-dimensional and (b) top view of the extruded t-slot framing structure designed to sit atop the top of the ITC SP400 forming press (shown in dark gray). Note the conical viewing hole in the center of the upper plate of the forming press.

E.4 Camera and Lens Selection

Two Basler acA2440-75um cameras along with two Schneider Kreuznach Mk-APO-CPN f2.8 40 mm lenses were selected for the stereo-DIC setup. Because the cameras are optimized for five megapixel (MP) imaging at 75 FPS, the cameras should effectively resolve strain evolution for strain rates up to approximately $1 \, \text{s}^{-1}$. The cameras feature modern machine vision technology, including: a Sony IMX250 complementary metal oxide semiconductor (CMOS) sensor with very small 3.45 µm pixels, high dynamic range, low noise, a global shutter to minimize motion aberrations, multiple opto-coupled digital input/output ports, and USB 3.0 data transfer. The Sony IMX250 sensor allows high rate and spatial resolution imaging with a comparatively small lens and camera form factor; the entire lens-camera combination is approximately 100 mm in length. The fixed focal length lenses were chosen to minimize image distortions commonly associated with zoom lenses. In the future, extension tubes could be acquired to modify the magnification and SOD of the camera/lens combination for other DIC configurations.

E.5 Lights and Polarizing Filters

A Dollan Jenner Fiber Lite® Mi-LED fiber optic light along with cross-polarizing filters will be used to illuminate the specimens. A fiber optic system was selected to locate the hot light source away from the cameras, lenses, and ROI. This was desired because heat sources can sometimes induce flickering.
aberrations in DIC images, which can increase the uncertainty in strain data [278]. The use of
cross-polarizing filters should greatly reduce glare from the surface of test specimens. However, the use of
cross-polarizing filters will reduce the amount of light that is received by the cameras, necessitating longer
exposure times. Some experiments will be performed to assess whether the use of cross-polarizing filters is
necessary, especially for samples with lower reflectivity (e.g., specimens painted with a DIC speckle
pattern). Some additional investigations are warranted to study the efficacy of the lighting setup. For
example, at present, it is unclear if the dimmer on the LED light source could cause flickering during
high-rate image acquisition; at acquisition rates higher than 50 to 60 Hz, it is possible that a flickering
effect due to the duty cycle of the LED light source could cause brightness variations between sequential
DIC images.

E.6 Speckle Pattern Optimization

A Correlated Solutions VIC Speckle Pattern Application Kit was purchased to enhance the
repeatability and quality of the DIC speckle pattern. The kit will facilitate the application of black
speckles onto test specimens using stamp rollers with various speckle sizes. According to the speckle size
suggestion table provided by Correlated Solutions [279], a nominal dot size of 0.18 mm (0.007 in) has been
selected for preliminary testing of the DIC system, based upon the resolution of the cameras (5 MP) and
the nominal FOV (5 to 10 cm). As a general rule, a speckle should be approximately 3-5 pixels in
diameter [278]. During preliminary testing, speckles created with black spray paint on a white substrate
generated speckles larger than the ideal size. Thus, the Speckle Pattern Application Kit should help to
improve strain resolution versus the spray paint based speckle application approach.

E.7 Trigger System for Synchronous Image Acquisition

To reduce measurement error, high resolution stereo image acquisition of moving objects requires that
both cameras acquire images at as close to the same time as possible. For instance, for two cameras
acquiring images at 1 Hz without any synchronization system in place, the delay duration between photo
acquisition in Camera 1 and Camera 2 may be anywhere from 0 to 1 s. If the subject of the image is
moving across the ROI at a displacement rate of 1 mm/s, then for a nominal delay of 0.5 s, the location of
the object would be shifted by 0.5 mm between the images acquired by each camera. Due to this
translational shift in the absence of any synchronization system, it is difficult to correlate stereo images of
moving objects with one another; the concept of the lack of agreement between stereo images is often
referred to as “epipolar error”. Epipolar error can be caused by numerous factors, including system
vibrations, image distortion due to heat waves, and lack of synchronization.
Synchronizing the image acquisition is particularly important and increasingly difficult for high frame rates and/or when object motion is rapid. For instance, to synchronize the two Basler cameras operating at an acquisition rate of 75 FPS and an exposure time of 10 ms, the DIC system must send 75 “trigger” signals per second to each camera. Furthermore, if it is desirable that both cameras are triggered to acquire an image within a time duration of less than 10 pct of the exposure time to reduce epipolar error, then the delay between photo acquisition in Camera 1 and Camera 2 must be less than 1 ms.

Two general types of trigger mechanisms are recognized for triggering multiple cameras: software based and hardware based. In a software based trigger system, Boolean logic is used to request the acquisition of an image for each camera using a unique USB 3.0 data cable. Although the system is robust and simple, it is not necessarily possible to reliably send signals with unique USB 3.0 ports at exactly the same time. Thus, it is difficult to assure sub millisecond synchrony. In a hardware based trigger system, rather than wiring each camera individually to a computer, the cameras are wired on the same circuit to guarantee that they receive an “acquire” signal at the same moment. Three specific trigger configurations were evaluated to measure the image acquisition delay between cameras. The methods are listed in order of increasing synchrony. Generally, it is understood that an increase in synchrony is accompanied by an increase in hardware/wiring complexity. However, once the trigger system has been installed, both methods are relatively simple to use while performing deformation processing experiments.

E.7.1 Software Trigger with USB 3.0

With the software trigger technique, software signals were sent by the Basler Pylon software to each camera to start acquiring images at a given acquisition rate. Although the Basler Pylon standard development kit includes some tools to adjust characteristics of the images, the software does not support synchronization for cameras that connect to the controlling computer via USB 3.0. Instead, the images were synchronized manually using a “clapper-board” technique, where an LED was illuminated in the FOV shared by each camera. Subsequently, images procured prior to the illumination of the LED were removed from the analysis. This resulted in a set of images that were synchronized to within a delay up to the inverse of the frame rate, or $1/(75 \text{ FPS}) = 13.3 \text{ ms}$. This delay would perhaps be acceptable for quasi static testing, but not for higher rate testing. An additional problem with this method was that to minimize epipolar error, the full available acquisition rate was required, even in cases where using 75 FPS would create prohibitively large sets of images. For instance, a five minute formability test using both cameras at 75 FPS would create 45,000 unique images. Given a file size of 5 MB per image, the test would require 225 GB of data storage capacity. Of course, the image acquisition rate could be reduced to decrease the data storage burden, but this would lead to a proportional increase in epipolar error.
**E.7.2 GPIO-Based Software/Hardware Hybrid Trigger in Controller Responder Configuration**

A software/hardware hybrid trigger was developed to decrease the delay between images. In this approach, one camera was designated as the “controller” and the second camera was designated as a “responder”. The design intent was that as the “controller” received an “acquire” signal from the Basler Pylon software, it would send a second “acquire” signal to the “responder” with a general-purpose input/output (GPIO) connector cable. Although the system reliably triggered both cameras, the delay between cameras was neither reduced nor repeatable. The average delay between image acquisition by the “controller” and “responder” was $2.87 \pm 0.57$ s. This suggested that the hybrid trigger offered no distinct advantage over the software trigger for stereo imaging.

**E.7.3 Arduino Based Hardware Trigger in Responder Responder Configuration**

Based on the significant delays of the software based and software/hardware based triggering systems, it was determined that a completely hardware-based trigger was preferable. In a hardware-based triggering system, the key advantage is that the two cameras share the trigger signal circuit. Therefore, both cameras act as synchronous “responders” while an external device acts as a “controller”. An open-source microcontroller board known as an Arduino UNO was used to send the digital “acquire” signal based upon numerous user inputs. The Arduino-based hardware trigger was initially designed to operate in three distinct modes (each mode can be activated by pressing a unique button on a breadboard). The three modes behave as discussed below:

1. **Manual single trigger mode:** Three seconds after a button is pressed, this mode sends one “acquire” signal to capture an image from both cameras synchronously. The mode is useful for acquiring one pre-deformation image or for collecting calibration images of a standardized calibration grid.

2. **Manual burst trigger mode:** Three seconds after a button is pressed, this mode sends a burst of “acquire” signals at a predetermined rate (in FPS) for a predetermined amount of time. The rate and time can be controlled by connecting the Arduino board to a computer using USB, then modifying a code that contains the correct settings. Because of the enhanced functionality of Mode 3 (below), it is assumed that Mode 2 will be used only in a few special cases such as system debugging.

3. **Forming press analog-output burst trigger mode:** This mode behaves the same as Mode 2, except the image acquisition begins only when an analog signal is received from the ITC SP400 forming press computer. The analog signal from the forming press is programmable in the forming press control software; for preliminary experiments, the software has been configured to begin image acquisition.
when the punch is moving upward toward the formability testing blank and reaches a distance of 3 mm between the top of the punch and the bottom of the specimen. It is expected that this mode will allow synchronization of the image acquisition signals with the forming press sensor readouts (i.e. clamp force, clamp displacement, punch force, and punch displacement). This mode is only operational when it is armed by pressing the “arm forming press trigger” button on the breadboard. It was determined that operating in this mode should require the press of a button for “arming”; in the absence of an arming function, the cameras could be triggered in undesirable circumstances, such as during the forming press warm-up cycle or during tooling changes.

Although the wiring of the Arduino-based trigger system is not documented in depth here, a brief discussion of the design is warranted. First, a high level schematic of the system wiring is provided in Figure E.2.

Figure E.2 Schematic of the Arduino based hardware trigger. When the punch moves within a certain distance of the formability testing specimen, a programmable signal is sent from the ITC SP400 forming press to an analog input on the Arduino microcontroller. Then, a synchronous trigger is sent to both cameras to acquire images at the specified settings (e.g. FPS, exposure time, and detector gain). The acquired images are stored on the internal buffer of each camera, then transferred to the forming press computer for analysis.

It is noted that to eliminate the chance of damaging the cameras by applying too much current or voltage to the input lines, the digital trigger from the Arduino microcontroller was attached to opto-coupled input lines on the cameras. Furthermore, resistors were added where necessary to reduce the current and voltage. The Arduino-based trigger system was conceptualized, programmed (C++), modeled, and debugged using the cloud-based computer-aided design software Autodesk TINKERCAD® Circuits.
An image of the interactive model is shown in Figure E.3. The code for the Arduino-based hardware trigger is provided in Appendix D.

Figure E.3 Wiring schematic of the Arduino-based hardware trigger system. Initial circuit design, microcontroller coding (C++), and debugging were performed using the Autodesk TINKERCAD® Circuits cloud-based design platform. The analog signal sent from the ITC SP400 was simulated using the oscilloscope shown at the bottom of the animation. The single or burst pulses sent to the two cameras were measured using the yellow voltage-time plots at the top of the animation.

Initial performance of the Arduino-based hardware trigger was promising. Using the device to initiate image acquisition was straightforward and images appeared to be well synchronized within practical measurement limits. When acquiring images (75 FPS) of a moving object displayed on a digital screen with a refresh rate of 60 Hz, it was evident that the images were delayed from one another by a time interval less than the inverse of the refresh rate: \( \frac{1}{60 \text{ FPS}} = 16.7 \text{ ms} \). With a high refresh rate screen (or with another technique such as flashing an strobe light at a known frequency, the delay could be quantified with greater temporal resolution. However, for the purpose of the proposed (low strain rate) formability experiments, quantifying the delay with a higher level of rigor is not necessary.
E.8 Example of a Formability Test of QP980-1.4-A using the DIC System

A preliminary sheet formability experiment is documented here, to demonstrate the capability of the DIC system. DIC-Engine (DICE), an open-source software developed by Sandia National Laboratories, was selected for processing the DIC images. A Matlab script was developed to visualize the strain in the plane of the sheets. An example of a Marciniak formability test of a 3-inch wide sample of QP980-1.4-A, monitored with the DIC system, is provided in Figure E.4. At a frame rate of one frame per second and a punch displacement rate of 0.33 millimeters per second, approximately fifty pairs of images were acquired until failure. The strain maps in Figure E.4(a) resolve the strain in the major and minor axes, respectively, immediately after fracture. The strain path diagram in Figure E.4(b) shows the evolution of strain on the sheet surface, which was calculated based on the \( \sim 100 \) subsets in the correlation which existed within the black circle in the strain maps shown in Figure E.4(a). Each point plotted on the strain path diagram was “safe”. “Failure” was defined as the point at which a localized trough formed on the sample surface (see Hosford and Caddell [65]).

![DIC analysis of a three inch wide hourglass sample of QP980-1.4-A. (a) Two-dimensional strain maps for the in-plane principal strains, \( \varepsilon_1 \) and \( \varepsilon_2 \). (b) Strain path diagram of “safe” forming strains for QP980-1.4-A, calculated based on the DIC analysis in (a).](image)
## APPENDIX F

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