EFFECT OF MICROSTRUCTURE ON THE FRACTURE RESPONSE OF ADVANCED HIGH STRENGTH STEELS

by

Mark David Taylor
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Golden, Colorado

Date _________________

Signed: ________________________
Mark D. Taylor

Signed: ________________________
Dr. David K. Matlock
Thesis Advisor

Golden, Colorado

Date _________________

Signed: ________________________
Dr. Ivar Reimanis
Professor and Head
Department of Metallurgical and Materials Engineering
ABSTRACT

The effect of constituent hardness on formability performance for higher-strength dual phase (DP) steels was evaluated. A commercially-produced DP steel with 1080 MPa ultimate tensile strength (UTS) was processed to create eight additional constituent hardness conditions by tempering and cold-rolling, processes that primarily affected constituent hardness properties. Using nanoindentation, ferrite and martensite hardness values for the nine conditions of the DP steel (as-received, four as-tempered, four temper cold-rolled) provided a range of hardness values to evaluate formability performance.

Formability performance for the nine steel conditions was evaluated using tensile and hole expansion testing. A decrease in martensite/ferrite hardness ratio corresponded to an increase in hole expansion ratio (HER), and an increase in yield strength (YS). A lower hardness ratio (increased similarity of ferrite and martensite hardness) was interpreted to increase strain-sharing between ferrite and martensite, which suppressed plastic strain localization to higher stresses for the case of YS, and to higher formability limits for the case of HER. A lower hardness ratio corresponded to a decrease in work-hardening, and was interpreted to be caused by the suppression of strain localization in ferrite. Multiple studies from literature correlated HER to tensile properties, and the nine steel conditions produced consistent trends with the data reported in each study, confirming the experimental HER and tensile properties obtained in the current study are consistent with literature.

The microstructural response to plastic deformation was evaluated using two DP steels with equivalent UTS and different hardness ratios. Nanoindentation analyses on tensile specimens deformed to the UTS revealed a greater increase in ferrite hardness for the higher hardness ratio steel, interpreted to be caused by the greater amount of work hardening. EBSD crystallographic orientation maps for the two DP steels showed that, whether by cold-rolling or tensile deformation, a DP microstructure heterogeneously accommodates strains imparted by plastic deformation. Strain maps generated using digital image correlation on deformed tensile specimens for both DP steels showed that strains heterogeneously develop in the microstructure at locations consistent with preferential fracture sites in DP steels, such as ferrite/martensite interfaces. The hardness ratio primarily affected the magnitude of the strain gradients, with a larger hardness ratio yielding a greater strain gradient. With further deformation, isolated regions of high strain linked to form bands of strain localization throughout the microstructure. A plane strain tensile analysis showed the DP steel with lower hardness ratio to have a lower void population, a finding consistent with results established in the M.Sc. thesis of M. D. Taylor. Using fractured tensile specimens, a lower void area pct at equivalent stress and strain was observed for the DP steel with lower hardness ratio, confirming a lower hardness ratio suppresses microstructural damage.
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CHAPTER 1
INTRODUCTION

Increased fuel efficiency regulations have encouraged the incorporation of advanced high strength steels (AHSS) by the automotive industry. Compared to traditional low-carbon steels, AHSS grades have superior strength, providing an avenue for automotive manufacturers to reduce vehicle mass by down-gauging sheet thickness and still satisfy design criteria. However, higher-strength AHSS grades (780 MPa ultimate tensile strength (UTS) or greater) have exhibited fractures during forming operations at limits below values expected using traditional prediction methods, such as forming limit diagrams.

Dual phase (DP) steels are a class of AHSS that are comprised primarily of ferrite and martensite, and are a common choice for automotive manufacturers. Relative to other AHSS, DP steels have a low alloying requirement, a wide range of producible strength levels, and multiple production strategies. The strength of ferrite and martensite are different, and the micro-scale interactions between ferrite and martensite in the presence of complex stress states are believed to be one of the causes for unpredictable behavior (excessive thinning and/or localized shear fracture), producing formability limits below what is required for successful incorporation into the automotive industry. Figure 1.1 shows two different commercially-produced DP steels with equivalent strength (980 MPa UTS) that have been formed into an automotive component, with one exhibiting fracture (right), and the other successfully formed (left). Though both steels in Figure 1.1 are comprised of a combination of microstructural properties that achieve a 980 MPa UTS, one set of microstructural properties is more favorable for formability.

Figure 1.1 Photograph of two commercially-produced DP980 steels that have been formed into an automotive component. The steel on the left was successfully formed, while the steel on the right exhibited fracture (indicated by arrows) [1].
Laboratory tests such as stretch bending and hole expansion have been used to characterize different formability aspects of higher-strength DP steels. For example, stretch bend tests on DP steels have established critical bending radius – UTS correlations for sheet formability [2], and hole expansion tests on DP steels have characterized morphological effects on stretching of sheared edges [3]. Such experiments provide valuable knowledge to the steel community, but often omit a quantitative analysis of the microstructure. Microstructural properties including grain size, martensite volume fraction, morphology, and constituent hardness all contribute to the resulting formability of DP steels. In determining the effects of the aforementioned microstructural properties on formability, changing one property usually results in others being changed as well, making a conclusive statement about the effects of any one property difficult. A study focused solely on the effect of constituent hardness on subsequent formability performance of DP steels has yet to be performed.

The current project focuses on the effects of constituent hardness on subsequent formability of DP steels. Multiple constituent hardness conditions using a single DP steel chemistry were created by tempering/cold-rolling, and quantified using nanoindentation. Tempering and cold-rolling are two processing treatments interpreted to primarily affect constituent hardness. Formability of the different hardness conditions was evaluated using tensile and hole expansion testing. The microstructural response to plastic deformation was also explored to observe the effects of different constituent hardness conditions on work hardening, grain-level strain partitioning, and microstructural damage.

Previous studies and evaluation techniques essential to the current project are discussed in CHAPTER 2. An outline of the project is discussed in CHAPTER 3, and CHAPTER 4 describes the experimental techniques used to modify, evaluate, and test the DP steels. CHAPTER 5 presents the results and discussion of the experimental data. A summary of the major conclusions is provided in CHAPTER 6, and suggestions to further the current study are presented in CHAPTER 7.
CHAPTER 2
BACKGROUND

Addressing the issue of shear fracture in higher-strength AHSS grades requires a fundamental understanding of a few key processing, measuring and testing techniques. First, the concept of DP steels, along with an overview of the fracture mechanisms associated with DP steels is presented. Next, the processing technique of tempering is presented, followed by a discussion on the fundamentals related to the processing technique of temper rolling. Nanoindentation, a measurement technique capable of quantifying the hardness of individual grains, is then discussed. Next, digital image correlation (DIC), a computational strain measurement technique, is discussed. The stretch bend test, a formability test accepted to represent industrial stamping operations, is then discussed. Next, a hole expansion test used to evaluate sheared edge stretchability is presented, citing specific work focusing on AHSS grades.

2.1 Introduction to DP Steels

Dual phase (DP) steels are generally composed of a soft, ferrite matrix with either hard martensite or bainite islands, depending on the cooling rate and alloying content. Figure 2.1 shows a scanning electron microscope (SEM) micrograph of a laboratory-produced, intercritically-annealed DP steel using a 0.15 wt pct carbon steel. The larger, darker regions are ferrite (“F”), and the smaller, lighter regions are martensite (“M”). Commercially-available DP steels have ultimate tensile strength (UTS) values ranging from 600 – 1200 MPa, and multiple DP strengths can be produced by controlling the alloy content, martensite volume fraction (MVF), and processing histories [4].

![Figure 2.1 SEM micrograph of a laboratory-produced, intercritically-annealed DP steel using a 0.15 wt pct carbon steel. The larger, darker regions are ferrite (indicated “F”), and the smaller, lighter regions are martensite (indicated “M”). Steel was etched with 2 pct nital for approximately 10 s [5].](image)
Dual phase steels are popular due to the relatively low alloying requirements compared to other AHSS grades, good combinations of strength and ductility, low yield ratio, and the capability of being produced by multiple processing strategies [6, 7]. Figure 2.2 shows temperature-time plots for two potential processing routes for DP steels [7]. Figure 2.2a represents a DP steel produced using an intercritical anneal, where a cold-rolled steel is initially heated to a temperature where both austenite and ferrite exist (labeled “1”). The steel is then cooled at a specific rate to transform the present austenite into martensite (labeled “2”). If the cooling rate is too slow, the cooling profile may intersect a constituent region (Ferrite, Pearlite, or Bainite), and transform partially or completely to one of the other microstructural constituents. The DP steel in Figure 2.1 was produced using an intercritical anneal. Figure 2.2b represents a DP steel produced from the hot-rolled (fully austenitic) condition. By controlling the cooling profile and alloy content, a specific fraction of the austenite can be transformed to ferrite (labeled “1”), with the remaining austenite transformed to martensite.

![Temperature-time plots for two different processing strategies to produce DP steels. The plot in (a) utilizes an intercritical anneal used to process cold-rolled sheet steel, and plot (b) utilizes controlled-cooling from the hot-rolled condition [7].](image)

In practice, low amounts (< 5 % volume fraction) of austenite have been observed in select DP steels [7]. Increased performance demands from industry have led to the development and production of DP steels with specific mechanical performance targets, such as increased yield ratio, improved bendability, etc [8]. The realization of DP steels with specific mechanical performance targets is due in large part to an increased fundamental understanding of how microstructural properties affect deformation in AHSS.

2.2 Microstructural Damage in DP Steels

Improving the formability of higher-strength DP steels (UTS above 780 MPa) requires a fundamental understanding of the microstructural properties and mechanisms that promote damage at the
micro-scale. Damage nucleation behavior of DP steels is first introduced, followed by characterization of damage evolution as a function of deformation.

2.2.1 Fracture of DP Steels

During plastic deformation of DP steels, ferrite primarily accommodates strain, and martensite primarily accommodates stress [9, 10], resulting in strain localization in the softer, ferrite phase. Because ferrite and martensite deform differently, strain partitioning between ferrite and martensite leads to a region of strain heterogeneity at the interface, causing the most commonly observed damage event in DP steels, decohesion (void nucleation) at the ferrite/martensite interface [11–21]. Figure 2.3a shows an SEM micrograph of a commercially-produced 0.15 wt pct carbon DP800 steel (DP steel with 800 MPa UTS) exhibiting ferrite/martensite decohesion (indicated with arrows) in response to tensile deformation. As the hardness difference between ferrite and martensite increases, strain partitioning to the ferrite increases, resulting in greater strain localization and increased void nucleation at ferrite/martensite interfaces [17, 18, 22–25]. Another commonly observed damage event in DP steels is martensite fracture [26], and is illustrated in Figure 2.3b with an SEM micrograph of the same DP steel in Figure 2.3a in response to a triaxial stress state that developed in the localized neck of a tensile specimen.

![SEM micrograph of DP800 steel](image1)

Figure 2.3 SEM micrograph of a 0.15 wt pct carbon commercially-produced DP800 steel exhibiting ferrite/martensite decohesion in (a), and martensite fracture in (b) in response to tensile deformation. For both micrographs, steels were etched with nital for approximately 10 s, and tensile axis is horizontal [17].

Martensite fracture occurs more frequently with a less equiaxed microstructure, as less equiaxed (elongated) martensite grains tend to develop “weak points” where higher stresses are likely to concentrate [27]. Two other observed damage events in DP steels are ferrite/ferrite interface decohesion and ferrite/inclusion decohesion. Ferrite/ferrite and ferrite/inclusion decohesion are observed less
frequently than ferrite/martensite decohesion and martensite fracture, and are believed to insignificantly contribute to fracture [17]. Cleaner steel production processes have decreased the inclusion populations present in AHSS grades, making ferrite/inclusion decohesion occurrences less frequent [28]. Martensite fracture occurs due to limited ductility, while the other three mentioned fracture mechanisms occur from strain incompatibility at the interface between constituents of different strength [17].

During industrial forming and in-service conditions, DP steels can experience multiple unique strain paths which can potentially induce different macroscopic modes of fracture. Studies have observed fracture modes of multi-phase steels in the presence of both uniaxial and triaxial stress states [10, 15, 17–19, 24, 27, 29–31], and have concluded that DP steels typically fail in a ductile manner by nucleation, growth, and coalescence of voids, independent of stress state and macroscopic fracture mode. To understand void nucleation, micro-scale strain partitioning between martensite and ferrite under different stress states has been evaluated using advanced techniques, such as small-angle X-ray scattering [32], in-situ neutron diffraction [33], finite element (FE) modeling [12], and electron backscatter diffraction (EBSD). Results have shown that, even during the linear-elastic portion of a stress-strain curve, micro-scale yielding at grain boundaries can occur, and that a high degree of strain partitioning can also manifest within individual grains [33]. Because a stress-strain curve represents the bulk response of an entire specimen, micro-scale yielding events are masked [12, 33]. Strain partitioning in DP steels persist until void nucleation occurs, and the critical interfacial strength between ferrite/martensite is believed to be a material constant equal to approximately 1050 – 2000 MPa [32], though some report the interfacial strength as high as 4000 MPa [34]. Neutron diffraction studies on DP steels have shown that each interfacial region in a microstructure is unique due to the particular combination of grain size, morphology, interface orientation with respect to the imposed stress state, and strength of adjacent constituents, and that the condition for void nucleation (critical interfacial strength) for each interface region will most likely be reached at different values of global strain [32]. Further, DIC studies on DP steels have shown greater strain heterogeneity to occur in larger ferrite grains, as shear bands can develop unrestricted over larger distances, and a higher density of dislocation pile-ups can occur at ferrite/martensite interfaces [12, 13]. Heterogeneous strain distributions within the microstructure facilitate void nucleation at earlier strains than would be expected if strains were uniformly distributed throughout the microstructure [33].

2.2.2 Damage Accumulation in DP Steels

The population, distribution, and growth of voids formed under uniaxial and triaxial stress states in DP steels have been characterized using X-ray tomography (XRT), a non-destructive technique designed to quantify voids in-situ [16, 31, 35–38]. Voids typically nucleate in DP steels by one of the
four damage events discussed in Sec. 2.2.1, and subsequent growth of the voids is dependent on multiple factors, including proximity to other voids, the mechanical properties of surrounding grains, and the imposed local stress state. Greater nucleation rates have been observed for non-equiaxed DP microstructures [13]. Figure 2.4 shows a 3-dimensional volumetric representation of a 0.08 wt pct carbon laboratory-produced DP600 tensile specimen prior to necking (Figure 2.4a) and after neck formation (Figure 2.4b). The 3-dimensional volumetric representations in Figure 2.4 were created by reconstructing multiple 2-dimensional image “slices” acquired using XRT. The gray color represents the steel specimen, and the black speckles represent voids. From the 3-D XRT scan in Figure 2.4b, the void size, distribution, morphology, and fraction as a function of strain can be determined. Figure 2.5 shows the average void diameter as a function of strain for the tensile specimen shown in Figure 2.4. The observed increase in void diameter with strain confirms void growth occurs.

![3-D XRT images](image)

**Figure 2.4** Images acquired using 3-D XRT of a DP600 steel tensile specimen before necking (a) and after necking (b). The gray color represents the steel, and the black spots represent the voids that have formed within the microstructure [16]. Tensile axis is vertical.

Some studies postulate that the loss of load-carrying capacity caused by an initial set of growing voids shed stress to the adjacent material, initiating a second population of smaller voids, and these smaller voids are what facilitate linking of the larger voids, leading to fracture [32, 35]. However, other studies show void nucleation to be a continuous event with increasing strain [16]. Figure 2.6 shows a plot of void fraction as a function of distance from the center of the specimen in Figure 2.4b at different deformation steps, where an increase in step number correlates with an increase in global strain. The void fraction varies with position across the specimen width for steps 5 and 6, with a higher void fraction in the
central region where the degree of stress triaxiality is the highest [17]. A triaxial stress state develops upon necking in a tensile test, and contributes to the heterogeneous distribution of void fraction across the specimen width in Figure 2.6 [32].

Figure 2.5 Plot of void diameter as a function of strain for the twenty largest voids present in the XRT analysis of the DP600 tensile specimen presented in Figure 2.4. The average void diameter appears to be increasing at an exponential rate with strain [16].

Figure 2.6 Plot of void fraction as a function of distance from the center of the laboratory-produced 0.08 wt pct carbon DP600 tensile specimen in Figure 2.4b. At later stages of deformation (i.e. step 6), the triaxial stress state that develops in the specimen center facilitates a greater density of voids [16].

The increase in void fraction with strain (Figure 2.6) reflects both the growth of existing voids (Figure 2.5), and the nucleation of new voids. Figure 2.7 shows a plot of void density as a function of local strain in a laboratory-produced 0.08 wt pct carbon DP600 steel for a round (smooth) tensile
specimen, and for a notched (R = 1 mm) round tensile specimen. The notch induces a triaxial stress state within the sample during loading, and the plot reveals that voids initiate at lower local strains and at a greater rate when compared to the smooth tensile sample. Figure 2.7 suggests that, in the presence of a triaxial stress state, the critical condition for void nucleation in DP steels is achieved at lower local strains compared to uniaxial tension [10, 31, 35, 36, 39]. Since formability tests, such as hole expansion and stretch bending generate triaxial stress states during testing, the accelerated nucleation and growth of voids may be a reason why the formability of DP steels is different depending on the particular test.

Figure 2.7 Evolution of void density in a smooth, round tensile specimen (smooth), and a notched tensile specimen (R=1mm) as a function of local strain for a laboratory-produced DP600 steel. The notched specimen appears to nucleate voids at lower local strains, and the void fraction increases at a faster rate compared to the smooth tensile specimen [36].

Figures 2.5 – 2.7 show the void growth, void area fraction, and void population as a function of both uniaxial and triaxial stress states, but evidence of void coalescence at the scale of the entire specimen has been difficult to observe. Void coalescence at the scale of the entire specimen in DP steels was determined to be an abrupt event occurring within a minimal increment of strain before fracture, with some studies reporting void coalescence to be absent in DP steel specimens strained to over 95 pct of the failure limit under a triaxial stress state [40].

2.3 Tempering

To modify the mechanical properties of a martensitic or a martensite/ferrite DP steel, a tempering process can be used. The technique of tempering will be introduced, including its primary purpose, microstructural mechanisms that occur, an equation used for characterization, and embrittlement phenomena. Next, temperatures commonly associated with tempering, along with microstructural
changes that occur at different temperature ranges are presented. Tempering phenomena observed in DP steels will then be discussed, including strain-aging, yield point elongation (YPE), and stress relaxation.

2.3.1 Introduction to Tempering

Fast cooling rates (quenching) employed by steel production facilities can leave certain AHSS grades, such as martensitic and martensite/ferrite DP steels in a brittle condition [41]. To improve ductility and impact energy absorption, a post-processing treatment known as tempering can be used [42]. Tempering is a heat treatment that softens the microstructure by relieving some portion of the carbon super-saturated martensitic phase [34]. Upon cooling, austenite undergoes a shear transformation to form martensite, and the martensite inherits the chemical identity of the parent austenite, potentially leading to a super-saturated carbon content. The solubility of carbon is higher in austenite than in martensite. Figure 2.8 shows an iron-carbon equilibrium phase diagram, and a maximum carbon solubility of 2.1 wt pct is observed for austenite (indicated by an arrow), while the maximum carbon solubility for martensite (α ferrite) is 0.022 wt pct. Carbon in excess of the solubility limit produces high-energy distortions in the lattice, increasing the tetragonality of the martensite crystal structure, making dislocation movement difficult, and is partly responsible for the brittle nature of martensite [43, 44]. Lattice distortion in martensite increases with increasing carbon content, and is one factor that gives higher-carbon martensite greater strength.

Tempering is a diffusion-controlled process that involves the re-arrangement of carbon atoms present in martensite [45]. During tempering, the carbon present in martensite will diffuse to dislocation cores or coalesce to form carbides, reducing the amount of carbon in solution, decreasing the tetragonality, and softening the martensite [46]. Softening through tempering is primarily used on martensitic and martensite/ferrite DP steels, as a crystal lattice super-saturated with carbon must be present. When tempering a DP steel, martensite primarily accounts for the softening of the microstructure, with ferrite only exhibiting a marginal reduction in hardness [47, 48]. Both time and temperature have an effect on the diffusion kinetics, and an equation combining the two variables was developed by Hollomon and Jaffe [49]. The parameter, known as the Hollomon-Jaffe Parameter (HJP) is presented as Equation (2.1, where T is temperature (Kelvin), t is time (hr), and C is a constant. A higher tempering temperature and/or a longer tempering time will result in a larger HJP. Time is characterized by a logarithmic function in Equation (2.1, so increasing the HJP is more effectively achieved by increasing temperature. Material hardness is usually plotted as a function of HJP [49], and an illustration for a 0.31 wt pct carbon martensitic steel is shown in Figure 2.9. Initially, a slight increase in hardness is observed around 8,000 HJP due to carbon atoms pinning mobile dislocations, but with increasing HJP, the hardness continually decreases. Higher-carbon martensite will have a higher initial hardness, but will
also soften at a faster rate compared to lower-carbon martensite since the high-energy lattice distortions provide greater driving force for carbon diffusion. It is important to remember that the HJP presented in Equation (2.1) only provides a functional relationship for the tempering parameters for which hardness can be plotted to. The hardness can differ for two different steels with the same HJP if the initial (as-quenched) hardness is different, and if the carbon-content in martensite is different.

\[
HJP = T(\log(t) + C)
\]  \hspace{1cm} (2.1)

Tempering treatments are usually performed at temperatures below 500 °C because of embrittlement phenomena that can occur at higher temperatures [42, 46, 48, 51, 52]. An embrittlement phenomena, referred to as temper embrittlement, is associated with the mobility of substitutional atoms that occurs at temperatures above 500 °C [53]. Some studies use temperatures above 500 °C, but the tempering times are usually short, giving insufficient time for temper embrittlement to occur [48, 51, 52].
Another embrittlement phenomena that has been observed at temperatures as low as 250 °C is referred to as tempered martensite embrittlement (TME) [55]. The condition promoting TME is usually associated with phosphorous and precipitation of cementite at prior austenite grain boundaries. The chart shown in Figure 2.10 details the different embrittlement phenomena as a function of temperature and carbon content. Most DP grades for automotive applications have carbon contents below 0.3 wt pct, but the martensite phase can locally exhibit carbon contents greater than 0.3 wt pct, depending on the MVF.

Figure 2.9 Plot of hardness (Rockwell C) as a function of HJP for a 0.31 wt pct martensitic steel [49].

Figure 2.10 Schematic showing the different embrittlement phenomena as a function of tempering temperature and carbon content [53].
2.3.2 Tempering Stages

The tempering response of martensitic and martensite/ferrite DP steels are dependent on multiple factors, including carbon content of the martensite, pre-strain, and alloying additions. For the same tempering treatment, a greater reduction in hardness has been observed in steels of higher-carbon martensite than for steels of lower-carbon martensite [56]. Pre-strain creates more dislocations within the structure, accelerating the diffusional process. Certain alloying additions are effective at retarding the softening response, and in some cases, have been purposely added to make certain steel grades resistant to softening [41, 45, 54, 56]. Alloying additions such as vanadium precipitate to form carbonitrides, and have been reported to increase both the YS and UTS during tempering [52, 55]. Though many factors affect the tempering response of martensitic and martensite/ferrite DP steels, three general stages of tempering are well-documented for the majority of tempering studies reported and performed in literature [46, 52, 57]:

Stage I: 100 – 250 °C, epsilon transition carbide and Cottrell atmosphere formation
Stage II: 200 – 300 °C, austenite decomposition, decreased tetragonality of martensite
Stage III: 250 – 350 °C, transition carbides transform to cementite

Above 400 °C, precipitates coarsen, and the onset of spheroidization of the microstructure begins [48, 52, 58–60]. Figures 2.11a – 2.11d show SEM micrographs of a laboratory-produced 0.21 wt pct carbon DP steel tempered for 1 hr at 200 °C, 400 °C, 500 °C, and 600 °C, respectively, and different microstructural characteristics can be observed. At 200 °C (Figure 2.11a), the microstructure appears similar to the untempered condition, an observation consistent with other studies performed on martensitic and martensite/ferrite DP steels at tempering temperatures below 200 °C [46, 48, 51, 52, 59]. At 400 °C (Figure 2.11b), cementite appears to have formed, which decreases the tetragonality of the martensite structure. At 500 °C (Figure 2.11c), the martensite structure appears to be replaced by ferrite and cementite, and at 600 °C (Figure 2.11d), spheroidization of the cementite occurred. Spheroidization is usually associated with tempering temperatures above 600 °C [61].

With an increase in tempering temperature, the microstructure becomes softer, leading to higher ductility and lower strength. The tensile properties as a function of tempering temperature for a 4340 martensitic steel are shown in Figure 2.12. For Figure 2.12, the tempering time was not reported, and is assumed to be 1 hr.
Figure 2.11  SEM micrographs of a laboratory-produced 0.21 wt pct carbon DP steel tempered for 1 hr at 200 °C in (a), 400 °C in (b), 500 °C in (c), and 600 °C in (d). At 200 °C temper, the microstructure appears similar to the untempered condition. At 400 °C temper, carbides have formed, and the martensite structure loses tetragonality. At 500 °C temper, martensite appears to have transformed into ferrite and carbides. At 600 °C temper, spheroidization appears to have occurred [48]. SEM micrographs a-d were acquired on a DP steel etched with nital.

Figure 2.12  Tensile properties of tensile strength (UTS), yield point (YS), elongation (TE) and reduction of area for a 4340 martensitic steel as a function of tempering temperature [55]. Tempering times were not reported, and are assumed to be 1 hr.
2.3.3 Tempering-Induced Behaviors Observed in DP Steels

The presence of ferrite in DP steels can produce unique mechanical behaviors after tempering. During the production of a DP steel, ferrite and austenite co-exist (Figure 2.2). A rapid quench then transforms the austenite to martensite (Sec. 2.1), and with an associated volume expansion. To accommodate the volume expansion associated with the austenite-to-martensite transformation, residual stresses and mobile dislocations are produced in the ferrite regions adjacent to the martensite. The density of the mobile dislocations and residual stresses increase with increasing MVF [58]. The residual stresses and mobile dislocations residing in ferrite adjacent to martensite are believed to contribute to the appearance of low YS and continuous yielding behavior in DP steels [54]. Upon tempering, the residual stresses relax, and carbon diffuses to dislocation cores (Cottrell atmospheres) and pins the newly created mobile dislocations, causing a phenomenon known as strain aging to occur [48, 54, 62]. During tensile loading, new mobile dislocations must be created to produce macroscopic yielding, so a higher stress is required to activate dislocation sources (upper yield point in Figure 2.13) [43]. As shown in Figure 2.13, the activation of dislocation sources causes a rapid decrease in the applied load to a level referred to as the lower yield point, a value which remains essentially constant in a region of discontinuous yielding known as YPE (yield elongation in Figure 2.13). During YPE, the gauge section yields discontinuously, as illustrated by the three bars inset in Figure 2.13. Discontinuous yielding persists until the entire gauge section has yielded and a sufficient population of mobile dislocations has been created. A higher YS and YPE caused by strain aging have been reported for tempering temperatures as high as 400 °C [34, 52, 54, 59].

Figure 2.13 Illustration of YPE (yield elongation) during a uniaxial tensile test [63]. After the upper yield point is achieved, mobile dislocations are heterogeneously created in the gauge section, leading to discontinuous yielding. Lüders bands continue to propagate over the entire gauge section, and are illustrated by the three bars inset on the graph [64].
Though minimal microstructural changes are observed in SEM micrographs at temperatures below 200 °C (Figure 2.11), mechanisms at the nano-scale activate at temperatures as low as 150 °C [46, 65]. Bake hardening, a common heat treatment used in the automotive industry, heats a steel to temperatures in the 120 – 190 °C range for less than an hour, and YPE has been observed, suggesting that carbon diffuses to mobile dislocations before carbide precipitation [41, 57].

2.4 Temper-Rolling

Temper-rolling, also known as skin passing, is a metalworking treatment performed at temperatures in the cold-working regime, where recovery and recrystallization are suppressed [63]. Temper-rolling treatments are normally small (< 1 pct) reductions in thickness [66], with a primary function of removing Lüder’s bands (YPE) in steels that have strain-aged through tempering or bake hardening. The plastic deformation associated with the reduction in thickness increases the mobile dislocation density, eliminating YPE and promoting continuous yielding. Lüders bands are regarded as aesthetically unpleasing in industrial forming applications.

During temper-rolling, certain parameters need to be considered to ensure uniform through-thickness deformation is achieved. Figure 2.14a shows a schematic of a cold-rolling process illustrating different dimensional attributes, where the rolls have a radius (R), and the initial sheet thickness (h₀) is reduced to h₁. To achieve uniform through-thickness deformation, a delta (Δ) parameter has been developed for different metalworking processes. For the cold-rolling setup shown in Figure 2.14a, the Δ–equation is shown as Equation 2.2. It is generally accepted that keeping Δ below 2 results in uniform through-thickness deformation. Under certain conditions, non-uniform strain distributions can develop (i.e. regions adjacent to the rolls can plastically deform while the center of the sheet steel remains undeformed), leading to residual stresses in the sheet [63]. An example of non-uniform through-thickness deformation during rolling is illustrated in Figure 2.14b, where only the shaded regions are plastically deformed.

$$\Delta = \sqrt{\frac{h_o}{4Rr}} [2 - r]$$  \hspace{1cm} (2.2)

Temper-rolling has also been used as a strengthening treatment, where experiments utilize thickness reductions exceeding 8 pct [67]. Boucek et al. temper-rolled a 0.005 wt pct carbon batch annealed steel up to 10 pct, and observed an increase in YS and UTS and a decrease in TE with increasing temper elongation, and the data for the longitudinal (L), diagonal (D), and transverse (T) orientations are shown in Figure 2.15a [67]. Another temper-rolling experiment performed on a DP600 steel showed a
similar behavior, with increasing YS (Rp 0.2) and UTS (TS), and decreasing elongation with increasing percent of temper rolling (up to 3 pct), and the data are shown in Figure 2.15b [68].

Figure 2.14  Schematic of cold-rolling in (a) with different dimensional parameters illustrated. Example of non-uniform plastic deformation during cold-rolling in (b), where only the shaded regions adjacent to the rolls plastically deform [63].

![Figure 2.14](image)

Figure 2.15  Plot of tensile properties as a function of percent temper elongation for a 0.005 wt pct steel in (a) [67], and plot of tensile properties as a function of pct temper rolling for a DP600 steel in (b) [68]. In both plots, an increase in YS and UTS, and decrease in TE is observed.

![Figure 2.15](image)

2.5 Nanoindentation

The technique of nanoindentation can be used to quantify the hardness of individual constituents in DP steels. First, an introduction to nanoindentation and to other hardness measurement techniques is presented. Next, essential components of nanoindenters, indenter tip geometries, data produced by the nanoindenter, hardness calculations, tip area function calculations, and a few experimental studies on steel using nanoindentation are discussed. The discussion focuses on the functions/equipment necessary to
obtain hardness measurements from metals. A more detailed discussion on the technique of
nanoindentation, associated phenomena, advanced capabilities, and test parameters has been previously
presented [40].

2.5.1 Introduction to Nanoindentation

Applications using nanoindentation have grown substantially in recent years as material behavior
at the sub-micron scale is increasingly being studied. Specific to metals, nanoindentation has become a
popular technique for quantifying the hardness of individual constituents within a microstructure [69–75].
Data from nanoindentation has extended beyond obtaining traditional hardness (H) and reduced modulus
(E) values to measuring internal friction, poisson’s ratio, and even aspects of fracture mechanics. Owing
to shallow indentation depths (10 – 70 nm), multiple indents can be performed within individual grains or
microstructural units of AHSS grades to quantitatively determine average phase hardness values [76, 77].
When considering the sub-micron grain size present in many AHSS grades, conventional hardness
measurement techniques such as Vicker’s, even when performed at the minimum load, can be too large to
accurately measure an individual grain [78, 79]. Constituent hardness values based on empirical
calculations involving chemical content have been used [11, 26], but inherently contain assumptions
based on cooling rate, chemical distribution, and post-processing (tempering, temper-rolling, case
hardening, galvanizing etc.) that could lead to potential error, especially when considering the multitude
of processing paths used to produce DP steels. Micropillar compression is a relatively new technique,
where a pillar of material created using a focused ion beam is subsequently deformed in uniaxial
compression, generating a stress-strain curve of an individual grain. Micropillar compression data are
valuable inputs for FE modelling of microstructures [80, 81]. However, specimen preparation time, cost,
and precision of the micropillar dimensions can potentially make the technique prohibitive [82]. Only a
few grains can be tested within a reasonable amount of time, leading to more uncertainty when attributing
the properties of only a few grains to an entire microstructure.

2.5.2 Nanoindenter Components

Each nanoindenter manufacturer has a unique configuration, but all contain similar essential
components. Figure 2.16 shows the most basic components on a nanoindenter. A load is applied onto a
sample surface through an indenter tip using high-precision transducers capable of applying loads up to
2 mN, with a 10 nN resolution. The two most common methods of load application are electromagnetic
and electrostatic actuation [83, 84]. Indenter displacement into the surface is measured using capacitors
with a resolution around 10 pm (1 X 10^{-11} m). In some nanoindentation configurations, the load-applying
and depth-sensing components are integrated into the same transducer [83, 85]. Because load and depth
are simultaneously recorded, most nanoindenters can operate in either load- or displacement-control. Specimens are placed on a motorized stage, where electric motors are used for coarse stage positioning (usually ~1 μm accuracy), and sub-micron positioning is achieved using piezoelectric devices [84].

![Schematic of the basic components in a nanoindentation system](image)

Figure 2.16 Schematic of the basic components in a nanoindentation system [83].

2.5.3 Nanoindenter Tip Geometry

Different commercially-available nanoindenter tip geometries have been used in steel research. A few considerations are required to select an optimal tip geometry for a particular application. Each tip geometry generates a unique stress-state in the material [86]. For accurate hardness measurements, a sufficient plastic zone must be generated. At sub-micron length scales, certain indenter geometries exhibit a phenomenon known as indentation size effect (ISE), a depth-dependence of material properties [75, 87–89]. Shown in Figure 2.17 are hardness values as a function of indentation depth for aluminum, copper, and different steels. The increase in hardness with decreasing indentation depth has been verified by different researchers [75, 77, 88–91].

![Plot of hardness (H) vs. indentation depth (h) for brass, aluminum, and different steels](image)

Figure 2.17 Plot of hardness (H) vs. indentation depth (h) for brass, aluminum, and different steels. A characteristic decrease in hardness with increasing contact depth is observed. Data was generated using a 3-sided Berkovich indenter tip [75].
Another consideration when selecting an indenter geometry is the potential for material pile-up during indenting. Depending on the material properties and indenter geometry, material displaced by the indenter can be ejected above the surface plane (pile-up), effectively increasing the contact area, and leading to an overestimation of hardness [92].

One common indenter geometry for metals research is the spherical tip, which yields accurate material properties independent of indentation depth (i.e. no ISE) [93], a desirable characteristic when varying indentation depth. However, spherical indenters require relatively large indentation depths in order to generate a sufficient plastic zone, meaning relatively large indents must be created in order to extract accurate hardness properties.

The cube-corner indenter is another common geometry, and is particularly useful when the indentation depth is limited, as the sharpness of the tip almost instantly generates a sufficient plastic zone upon contact. Data from cube-corner indenters can potentially exhibit ISE and material pile-up, both of which lead to an overestimation of hardness [94].

The most common indenter geometry in metals research is the Berkovich tip, a 3-sided geometry with the same contact area-to-depth ratio as a traditional Vicker’s (4-sided) indenter [84, 94]. An SEM image of a Berkovich indenter is shown in Figure 2.18. The Berkovich indenter can also exhibit ISE, but is more resistant to material pile-up. The hardness data in Figure 2.17 were generated using a Berkovich tip. Depending on the sharpness of the apex, Berkovich tips can also generate a sufficient plastic zone at shallow depths (15-40 nm). Berkovich, cube-corner, and Vickers indenters are all considered self-similar geometries; regardless of indentation depth, the stress/strain gradients around the indenter remain the same [95].

Figure 2.18  SEM image of a Berkovich indenter tip used for nanoindentation [94].
2.5.4 Nanoindentation Data Analysis

When an indent is created in a metal, both load and displacement are monitored, resulting in a plot similar to the schematic shown in Figure 2.19. While the maximum force can easily be obtained from the graph ($P_{\text{max}}$), the actual contact depth requires an analytical approach because intimate contact differs from the depth below the surface plane ($h_{\text{max}}$). From Figure 2.19, the indentation depth can be interpreted as the maximum depth coinciding with maximum load ($h_{\text{max}}$), the residual depth after the indenter is removed from the surface ($h_f$), or the depth obtained when the material stiffness, $S$, is extrapolated to zero load. The most widely accepted method to determine $H$ and $E$ from load-displacement curves is the Oliver-Pharr (OP) method [70, 75, 77, 96–98]. The OP method uses a portion of the slope of the unloading curve to determine $S$, because it is believed that the material behavior upon initial unloading is characterized by elastic recovery only [83].

![Figure 2.19](image)

**Figure 2.19** Representative load-displacement curve produced by nanoindentation. The final displacements can be interpreted as $h_{\text{max}}, h_f$, or $S$ [96].

The hardness calculated for nanoindentation data is shown in Equation (2.3, where $P$ is the load (in N), and $A$ is the projected contact area (in mm$^2$).

$$H = \frac{P}{A}$$  \hspace{1cm} (2.3)

To obtain an accurate projected contact area, tips such as the Berkovich indenter require an area function to be experimentally determined. Area functions are created by performing indents in a material with relatively homogeneous material properties over a range of ambient testing temperatures, such as fused silica. Indents on fused silica are performed at different loads, yielding different indentation depths. Because hardness and elastic modulus are intrinsic material properties, and because fused silica is
relatively free of ISE, an inverse regression can be performed to determine the contact area for the different indentation depths on fused silica. Assessing indenter area functions becomes increasingly important at shallower indentation depths. All tips are finitely sharp, and at some scale, a degree of curvature exists at the apex. In order to correct the deviation from an infinitely sharp Berkovich geometry, fitting coefficients are used. The Berkovich area function equation is given as Equation (2.4, where \( h_c \) is the indentation contact depth, and \( C_0 \) – \( C_5 \) are fitting coefficients [85].

\[
A = C_0 h_c^2 + C_1 h_c + C_2 h_c^{1/2} + C_3 h_c^{1/4} + C_4 h_c^{1/8} + C_5 h_c^{1/16}
\]  

(2.4)

For an ideal Berkovich tip, \( C_0 = 24.5 \) and \( C_1 - C_5 = 0 \). At large indentation depths, the \( C_0 \) term dominates and the tip converges to the ideal condition. Coefficients \( C_1 - C_5 \) are particularly useful in characterizing the tip dimensions at shallower contact depths (< 60 nm) [85]. The value of \( h_c \) is determined using Equation (2.5, where \( \varepsilon \) is a geometrical coefficient (0.75 for a Berkovich tip), and \( S \) is the stiffness obtained in Figure 2.19.

\[
h_c = h_{max} - h_s = h_{max} - \varepsilon \frac{P_{max}}{S}
\]  

(2.5)

The right-most quantity in Eq. (2.5 characterizes the surface deflection at the perimeter of contact. The surface deflection at the contact perimeter depends on indenter geometry [99]. A schematic showing different indentation depth readings (\( h, h_f, h_c, \) and \( h_s \)) for a conical indenter is shown in Figure 2.20. At maximum indentation depth (“h” in Figure 2.20), the actual contact depth is \( h_c \); \( h_s \) is the surface deflection below the initial surface.

![Schematic of the different contact depths during nanoindentation](image)

**Figure 2.20** Schematic of the different contact depths during nanoindentation. The contact depth used for calculating hardness using the O-P method is \( h_c \) [96].
Once the material stiffness is determined, the reduced modulus \( E_r \) can be calculated using Equation (2.6, where \( A \) is the contact area.

\[
S = \frac{dP}{dh} = \frac{2}{\sqrt{\pi}} E_r \sqrt{A} \tag{2.6}
\]

All indenters are finitely rigid, and compliance (deflection) of the indenter during testing occurs. Equation (2.7 shows the reduced modulus being composed of the material properties of the substrate, as well as the indenter, where \( \nu \) and \( E \) are the poisson’s ratio and elastic modulus of the substrate, respectively, and \( \nu_i \) and \( E_i \) are the poisson’s ratio and elastic modulus of the indenter. The material properties of the indenter are provided by the tip manufacturer.

\[
E_r = \frac{(1 - \nu^2)}{E} + \frac{(1 - \nu_i^2)}{E_i} \tag{2.7}
\]

2.5.5 Current Studies in Nanoindentation

A recent summary of current applications using nanoindentation spanning multiple material systems was written by Palacio et al., where most studies involved an integration of nanoindentation with another advanced technique such as SEM, transmission electron microscopy (TEM), or EBSD [100]. In one study, in-situ observation of nanoindentation using TEM allowed pop-ins from load-displacement curves to be uniquely attributed to either phase transformation or dislocation emission into adjacent grains [101]. Pop-ins refer to an increase in indentation depth at a constant load during the loading portion of a nanoindentation load-displacement curve (Figure 2.19), and a more detailed analysis of pop-ins is discussed elsewhere [40]. Another study used an SEM to monitor the deflection of a steel micro-beam being loaded by a nanoindenter [102].

Other nanoindentation experiments on steel have been performed to provide input for microstructure-based FE models. Attempts at replicating stress-strain curves from nanoindentation have been conducted using 3-D FE models [103, 104]. The material properties in the FE model were modified in an iterative process until the FE-simulated load-displacement curve matched that observed in nanoindentation experiments. The resulting stress-strain curve produced by the FE model was similar to that obtained experimentally. Some researchers believe spherical indenters are better-suited for obtaining stress-strain curves since they produce a relatively larger elastic segment [82, 93].

Recently, focus has been shifting from obtaining individual grain properties to characterizing hardness distributions within individual grains. Nanoindentation performed on individual ferrite grains in DP steels has revealed that hardness gradients exist, and that ferrite should be considered a heterogeneous
matrix [82, 105]. FE model improvements were achieved when hardness gradients within ferrite grains were used, and the models indicated that initial strain hardening occurs in the ferrite regions adjacent to martensite grains [106].

In transformation-induced plasticity (TRIP) steels, austenite transforms to martensite upon plastic deformation, and studies focused on the phase stability as a function of austenite grain size have been performed. By characteristics in the load-displacement curve, it was determined that smaller grain size and higher alloying content yielded a higher resistance to transformation [101]. Additional current studies in nanoindentation are reported elsewhere [40].

2.6 Digital Image Correlation

Digital image correlation (DIC) is a computer-aided strain-mapping technique capable of resolving heterogeneous strain gradients within a region of interest. For a particular region of interest, DIC uses images to correlate an initial pattern to a final pattern, and an illustration of the technique is shown in Figure 2.21. Strains are measured based on correlating the spatial location of a feature in the final image \((x_t, y_t)\) with its location from the initial image, \((x, y)\). The pixels comprising the image in Figure 2.21 are used to reference patterns between the conditions. A group of pixels, called a facet, are a unique fingerprint of the image, and each facet is analyzed to determine a local strain [107].

![Figure 2.21 Illustration of a facet showing a pattern before (a) and after (b) deformation. The movement of the point \((x, y)\) to \((x_t, y_t)\) is used to calculate local strain [108].](image)

The facet size and facet spacing set the resolution of the technique. Patterns at the nano- and micro-scale can be generated by depositing ink or silver nanodots [109–112], applying a digital grid [113, 114], or even using the contrast produced from a chemical etch [10, 27]. A more comprehensive explanation of the DIC technique is outlined elsewhere [115].

Since DIC calculations are based solely on images, the technique can be applied to any length scale as long as a discernable surface pattern is visible, and sufficient image resolution is achieved [107].
The DIC technique can generate a strain map for an entire tensile specimen using optical images, or for a single martensite/ferrite interface at 80,000X magnification using an SEM. When using an SEM for acquiring images, the potential for image drift must be considered. Drift can cause a shift in the image during acquisition, which can result in artificial strains when analyzed using DIC. However, DIC analyses using SEM images have been successfully performed [10, 107, 116]. Figure 2.22 shows a DIC analysis on a 0.14 wt pct carbon martensitic steel tensile specimen using a digital grid. The colors represent different strain levels and show the formation of a shear band.

![DIC analysis of a martensitic steel tensile specimen](Image)

Figure 2.22 Illustration of DIC results obtained from a martensitic steel tensile specimen. The color gradient, representing different levels of strain (color scale), show a shear band forming in the sample, leading to tensile failure. Tensile axis is horizontal, and specimen width (vertical direction) is 5 mm (Color image – see PDF) [113].

Unexpected shear fractures observed in industrial forming operations have motivated the evaluation of micro-scale deformation responses in AHSS grades, and DIC has been extensively applied to metals research [10, 109, 116]. For DP steels, strain partitioning has been observed at ferrite/martensite interfaces, with the severity of partitioning increasing with greater hardness difference between ferrite and martensite [116, 117]. Figure 2.23 shows a DIC analysis performed on a laboratory-produced 0.06 wt pct carbon DP600 steel in the as-received condition (a), and in the tempered condition (b). For a similar macroscopic tensile strain, the DP steel in the as-received condition exhibited higher localized strain values (0.7 pct vs 0.37 pct) at the martensite/ferrite interface. Both DIC analyses in Figure 2.23 were performed using the etched microstructure as the pattern grid. Different constituent hardness values for the different phases present in multi-phase AHSS grades causes the strain partitioning during plastic deformation (Figure 2.23), and suppression of these localized strain accumulations are believed to extend formability limits [17, 18, 117].

A DIC analysis performed on a nickel alloy has shown certain grain boundaries to contribute more to strain hardening than others, the contribution amount dependent on the crystallographic relationship of the two adjacent grains [118]. Sometimes, significant strain accumulation was observed on only one side of a grain boundary, indicating that slip transfer across the grain boundary was suppressed. DIC has also been used to observe twinning in shape-memory alloys, characterizing how strains produced by twinning can affect the crystallographic orientations of subsequent twinning [119].
Figure 2.23 DIC analysis on a laboratory-produced, 0.06 wt pct carbon DP600 steel in the as-received condition in (a), and in the tempered condition in (b). For a similar global strain during uniaxial tension, the tempered condition shows fewer regions of high localized strain [116]. Strain values correspond to the color scale in each figure. Color image – see PDF.

2.7 Stretch Bend Test

Material properties obtained from a uniaxial tensile test, such as yield strength (YS), UTS, uniform elongation (UE), TE, and r-value, have traditionally been used for selecting steel for different applications. However, some applications and forming operations experience a stress state more complex than uniaxial tension [3, 30], and properties such as TE are inaccurate indicators of performance [1, 3, 29, 120]. Tensile elongation involves global fracture, where mechanisms at the scale of the entire specimen are active [31], while complex forming operations can promote localized fracture, where mechanisms on the order of several grains dictate performance. In particular, DP steels are known to be susceptible to localized fracture [30], and the unpredictable formability performance is one challenge delaying extensive incorporation of new higher-strength AHSS grades by the automotive industry. One reason why localized shear fractures are difficult to predict is because limited necking is observed before fracture.

Specialized laboratory sheet metal forming tests have been developed to address localized fracture of higher-strength AHSS grades during industrial stamping operations [121]. One such test is the stretch bend test, and an illustration of the test setup is shown in Figure 2.24. Two hydraulic actuators situated 90° relative to each other subject a sheet steel specimen in tension over a die of specific radius (“Roller Assembly” in Figure 2.24) until fracture occurs, simulating material flow during forming. A triaxial stress state is achieved at the die/sheet interface, and is believed to represent the stress states experienced for some industrial stamping operations [30].
Figure 2.24  Schematic of the stretch bend test. The two hydraulic actuators subject the sheet steel specimen to tension over the die (Roller Assembly), causing fracture [2].

The parameter used to quantify formability for stretch bend tests is the die radius – sheet thickness (R/t) ratio. Figure 2.25a shows five commercially-produced DP780 stretch bend specimens that have fractured after being tested with different die radii. The stretch bend test is capable of producing global, tensile fracture away from the die at large R/t ratios (R = 12.7 mm in Figure 2.25a), and transitions (R = 6.4 mm in Figure 2.25a) to a localized, shear type fracture at the die/sheet interface at smaller R/t ratios (R = 3.1 mm in Figure 2.25a) [122–124].

(a)  
(b)

Figure 2.25  Image of five specimens tested to failure using the stretch bend test in (a) [125]. The specimens show a transition from a tensile failure (R = 12.7) to shear fracture (R = 3.1). Plot of failure stress as a function of R/t ratio in (b) [122]. The dashed vertical line represents R/t*, the industrial formability limit.

Localized failures in stretch bending are termed “shear fractures”, as fractures propagate through-thickness on a shear plane oriented 45° with respect to the sheet surface and tensile axis. Figure 2.25b
shows failure stress vs. R/t for a DP600 steel. Failure stresses at lower R/t values are associated with both transition fractures and shear fractures (R = 6.4 mm and 3.1 mm, respectively, in Figure 2.25a). The R/t at which the failure stress achieves the UTS is known as the critical R/t, or R/t*(indicated by a vertical dashed line in Figure 2.25b), and can be used as an industrial design limit. For R/t values greater than R/t*, the UTS is achieved, and only tensile failures away from the die occur. A low R/t* is desirable, as it increases design capability of auto-body components [122].

When testing three different steels of similar strength, Walp et al. [122] observed different R/t* for each steel, and proposed a strain-based rather than stress-based analysis to understand the role of microstructural properties in shear fracture susceptibility. Further advances in understanding of AHSS shear fractures using stretch bending were obtained by Kim et al. [124], showing that when deformation-induced heating was considered, fracture was accurately predicted in most cases. However, in select cases primarily involving the higher-strength AHSS grades, Kim et al. concluded that microstructure-based fracture mechanisms dominated formability limits, and alluded to the incorporation of damage mechanics into prediction models [124]. Microstructure-level damage mechanisms have traditionally played a secondary role in formability of steels, which is why conventional prediction methods, such as forming limit diagrams have been successfully used in the past. Stretch bend tests are valuable in determining industrial design limits, but the mechanisms that promote shear fracture over tensile fracture are of interest, especially as AHSS microstructures become more complex.

Conclusions based on stretch bend results produced by Hudgins [2] motivated the M.Sc. thesis of M. D. Taylor, where a plane strain tensile specimen was developed to replicate the shear fractures observed at low R/t ratios (small radii) [40]. The plane strain tensile geometry and plane strain tensile test are presented in Chapter 4. Taylor concluded that a higher martensite/ferrite hardness ratio corresponded to a higher void density at an equivalent deformation stage, and hypothesized that a decrease in hardness ratio for DP steels will suppress damage, thereby extending formability limits of higher-strength AHSS grades.

2.8 Hole Expansion Testing

In addition to the stretch bend test discussed in Sec. 2.7, the hole expansion test is another laboratory technique developed to evaluate the complex response during industrial forming operations of higher-strength AHSS grades [121]. An introduction to hole expansion testing, including test setup, testing hardware, specimen preparation effects, and potential stress states experienced during testing is presented. For AHSS grades, microstructural properties reported to have an effect on HER are discussed, followed by a discussion of previous experiments relating HER to tensile properties.
2.8.1 Hole Expansion Experimental Setup

Edge stretchability is the ability of a sheet steel to be stamped into a final part without failure by fracture or excessive thinning at a sheared edge or hole, and is an area of interest as the geometrical complexity of automobile components increases [78]. Formability of AHSS in complex forming operations departs from traditional measures of ductility, such as TE obtained from a uniaxial tensile test [22, 29, 30, 120]. Tensile tests produce global fracture, where fracture mechanisms present at the scale of the entire sample are considered [30], and hole expansion tests produce local fracture [36], where only a few grains in the vicinity of the crack can dictate performance. The hole expansion test is believed to correspond to in-die performance of certain forming applications, and a schematic of the test using a conical punch is shown in Figure 2.26 [121]. A 10 mm diameter hole is created in the center of a sheet steel specimen (usually 100 x 100 mm), and a concentric punch is pressed uni-axially upwards through the hole (red dotted line in Figure 2.26). The steel specimen is clamped to prevent the specimen from drawing upwards during testing. At the first observation of a 0.1 mm through-thickness crack, the test is stopped. The hole expansion ratio (HER) is defined in Equation (2.8, where \( d_o \) and \( d_f \) are the initial and final hole diameters, respectively.

![Figure 2.26 Schematic of the hole expansion test using a conical punch on a sheared hole. The upward flow of material around the hole edge represents the burr created during the hole shearing process. In the figure, the steel sheet is being tested in the burr-up orientation [121] (color image- see PDF).](image)

\[
HER = \left( \frac{d_f - d_o}{d_o} \right) \times 100
\]  

(2.8)

The \( d_f \) value in Equation (2.8 is measured after the hole expansion test is complete (i.e. a crack has formed). The method of crack detection can vary depending on test setup, from visual inspection [21,
to a computer-aided image inspection using cameras. A standardized test methodology for hole expansion testing, ISO 16630, was developed in an effort to increase the reproducibility of HER results between different laboratories. The current version of ISO 16630, however, affords many liberties as to how the test can be conducted.

The method used to create the hole in the specimen for hole expansion testing has been performed multiple ways, the most common being shearing, drilling, and electric discharge machining (EDM), each of which affect the resulting HER [78]. Sheared holes are most commonly encountered in industry, the most common condition considered in published studies, and are also the method outlined in ISO 16630. Shearing creates a region of heavily deformed grains adjacent to the hole edge, and is termed a shear-affected zone (SAZ). The SAZ created by shearing has been observed to decrease the HER compared to other hole preparation techniques, such as EDM [11, 76]. Figure 2.27 shows the microstructure for a laboratory-produced 0.07 wt pct carbon DP600 steel in the initial, undeformed condition (Figure 2.27a) and in the SAZ after shearing (Figure 2.27b) [26]. The SAZ contains heavily deformed grains adjacent to the hole edge, and the plastic deformation has strain-hardened the grains to hardness values greater than the bulk microstructure. Micro-voids have also been reported to form in the SAZ and are indicated in Figure 2.27b with arrows. The amount of plastic deformation in the SAZ is dependent on multiple factors, and researchers have estimated the equivalent strain to be on the order of 90 – 150 pct [11, 127]. Multiple studies have concluded that removal of the SAZ increased the HER [78, 126, 128], and methods to mitigate the SAZ after shearing include polishing, sanding and reaming [129, 130]. Holes created by drilling and EDM do not create a SAZ, and have exhibited superior HER values compared to sheared holes of the same material [26, 30, 79, 131, 132].

Figure 2.27 SEM Micrograph of a laboratory-produced 0.07 wt pct carbon DP600 steel microstructure in the initial condition in (a), and after having a hole sheared for hole expansion testing in (b). The microstructure in (b) is adjacent to the hole edge, and is representative of the majority of the SAZ [26]. Steel was etched with 2 pct nital.
The die/punch clearance used to produce sheared holes is another variable that needs to be considered [130], and an illustration of the parameter is shown in Figure 2.28. The die/punch clearance is known to affect subsequent HER because it affects the characteristics of the SAZ. For instance, a larger die/punch clearance will result in a larger burr after shearing [130]. The burr is created by material flow during the hole shearing process, and has been shown to affect HER values. Additionally, the material flow also affects the edge unevenness (roughness), which has been shown to negatively affect HER [131].

Most hole expansion experiments test the steel coupon with the burr facing upwards, and is represented in Figure 2.26 with the slightly inclined material adjacent to the hole edge. The ISO 16630 standard specifies the die/punch clearance as 12 +/- 2 pct of sheet thickness, and to test with the burr facing upwards. Keeping within tolerance for the die/punch clearance is important for reproducibility, and for comparison of multiple AHSS grades.

![Figure 2.28 Schematic showing the die/punch clearance. A larger clearance can produce a larger burr after shearing, and affects the characteristics of the SAZ.](image)

Figure 2.29 shows three common punch geometries used for hole expansion testing; conical, spherical, and flat-bottom [131]. Each punch geometry creates a unique strain-state within the sheet steel, and can cause failure at different HER values in the same steel specimen. Hole expansion testing using a flat-bottom punch was observed to produce the most material thinning before fracture, and the conical punch was observed to produce the least [131]. Of the three punch geometries, the conical punch (also shown in Figure 2.26) is most commonly used to evaluate formability [11, 129], and is the specified punch geometry for ISO 16630. The conical punch generates a stress state consisting of bending, tension, and frictional forces in the steel, and some researchers have shown the stress state of the sheared hole surface to be pure uniaxial tension [130, 132, 133]. A strain gradient exists in the steel during hole expansion testing, with material sufficiently far away from the sheared hole experiencing a stress state.
different than material in proximity to the sheared hole [133]. During hole expansion testing of a sheared hole using a conical punch, a SAZ containing a heavily deformed microstructure is subjected to a bending – tensile stress gradient until plastic deformation initiates a through-thickness crack [126, 134].

Figure 2.29 Photograph of three commonly-used punch geometries for hole expansion testing, including the spherical (left), conical (middle), and flat-bottom (right) punches [131].

Because of the freedom in choosing test parameters, care must be exercised when HER data from different studies are compared, as the setup, punch geometry, hole preparation method, and crack detection method will affect the resulting HER. When performing hole expansion tests, keeping all test parameters constant is essential for accurate data analysis [132].

2.8.2 Microstructural Factors Influencing HER

Due to the limitations of traditional FLDs to predict formability of higher-strength AHSS, many studies have been performed to investigate the effects of microstructural properties on corresponding HER. In uniaxial tensile tests, DP steels can achieve good combinations of high strength and elongation from soft ferrite accommodating the majority of strain, and martensite accommodating the majority of stress [39, 126, 135]. However, hole expansion tests produce a complex, triaxial stress state within the material, and different phases are reported to exhibit different ductility limits depending on the stress state [128]. A limited dome height test performed on a martensitic steel produced an equivalent uniaxial strain of 150 pct, and supports the claim that stress state affects the ductility of a phase [22, 127]. Figure 2.30 shows an FE model simulation of the triaxial stress contours in a hole expansion specimen tested with a conical die. A triaxial stress state has been observed to accelerate strain localization into ferrite, making ferrite the “weakest link” in the microstructure by forming shear bands at earlier stages of deformation [22, 39]. The rapid strain localization in ferrite may be one reason why performance in HER appears
independent of TE obtained from uniaxial tensile tests. For AHSS, equivalent uniaxial strains experienced in hole expansion testing are usually higher than the TE [126].

Figure 2.30 Stress triaxiality contours for a FE model simulation of a hole expansion test using a conical die [128]. Units for the color scale is the ratio of mean stress to equivalent stress. *(Color image- see PDF).*

To minimize strain localization in ferrite in the presence of a triaxial stress state, a morphology consisting of a martensite “necklace” network surrounding ferrite grains has been proposed [3, 17, 22, 29, 39], though this view is challenged by some [132]. A martensite necklace would inhibit shear bands from propagating over long distances [21], as the shear bands would likely arrest at martensite islands [27]. Equiaxed and/or banded morphologies are less ideal since shear bands can potentially propagate over long distances before being arrested [78]. Another possible reason for the difficulty in correlating HER to tensile properties is because the optimal microstructure morphology for HER is an interconnected martensite network, while the optimum for uniaxial tension is fine-grained and equiaxed [20].

A smaller grain size has been reported to improve HER, as shear bands in ferrite would travel shorter distances before being arrested by martensite islands [127, 132]. Correlations between HER and MVF have also been established for DP steels, concluding that HER increases as a microstructure tends towards a single-phase composition [22, 26, 127, 132, 133, 136], even for a fully martensitic or bainitic microstructure [11, 25, 137]. Figure 2.31 shows an increase in HER and corresponding decrease in UE with increasing MVF for 0.15 wt pct carbon laboratory-produced DP steels. Figure 2.31 compares DP steels to a fully martensitic steel (0 pct ferrite), and suggests that for localized fracture, the absence of “weak links” demands a higher degree of strain accommodation by the bulk of the microstructure [11]. Some studies report a decreasing HER with increasing MVF [132], but in these select cases, the MVF was increasing towards 50 pct, which tends further away from a single-phase composition.
All microstructural properties (MVF, grain size, dislocation density, chemical composition, morphology) contribute synergistically to the formability performance, and attempting to change one property usually has an interdependent effect on others [22]. A challenge associated with most studies cited herein is that, when changing a microstructural property, such as MVF (Figure 2.31), other microstructural properties simultaneously change. Hence, it is difficult to conclusively state the effect of any single microstructural property on HER because of the inherent change of other microstructural properties.

One conclusion that has been relatively widespread among different studies pertaining to HER of DP steels is that a decrease in the relative hardness difference between ferrite and martensite will increase HER [3, 6, 11, 17, 22, 29, 30, 39, 46, 78, 130, 132, 138, 139]. A larger martensite/ferrite hardness ratio denotes greater strain partitioning in regions near the ferrite/martensite interface, leading to a higher degree of strain heterogeneity [22, 27, 29, 135], and ultimately interface incompatibility (decohesion) at lower global strain values [127, 138] (Figure 2.23). Figure 2.32 shows the HER increasing as a function of tempering temperature for a 0.13 wt pct carbon laboratory-produced DP steel. For DP steels, tempering primarily softens martensite (Sec. 2.3.1), thereby decreasing the martensite/ferrite hardness ratio.

A larger hardness ratio localizes strain in the softer ferrite phase at earlier stages of deformation, causing void nucleation [21, 140]. A lower hardness ratio denotes increased similitude between martensite and ferrite, and supports the previous claim that HER increases as a microstructure tends towards a single-phase composition. With a decreasing hardness ratio, the “weak link” becomes stronger and/or the martensite becomes softer, enabling the microstructure to accommodate more homogeneous plastic deformation and delay strain localization [29].
Figure 2.32 Plot of HER increasing with increasing tempering temperature for a 0.13 wt pct carbon laboratory-produced DP steel, indicating a decreased martensite/ferrite hardness ratio corresponds to improved edge stretchability [46].

2.8.3 HER and Tensile Correlations

Even with differing stress states, failure criteria, and initial condition of the microstructure, much interest has been directed to correlating HER values with tensile properties. Tensile testing is less time-intensive to perform, the necessary equipment is more readily-available, and the data analysis is more objective. Accurate predictions of HER based on tensile properties would be beneficial for industrial applications.

Advances have been made in correlating HER to tensile properties by considering material properties such as material thickness [126], r-value, TE of the transverse orientation, strain-hardening exponent [140], post-uniform elongation [132], and true fracture strain [120, 133]. Most studies developed an empirical equation that predicted HER based on different tensile properties, and the empirical equations are usually designed to be applicable for a wide range of steels. Comstock et al. developed two empirical equations, one using tensile properties of TE, r-value, and thickness, presented as Equation (2.9), and one using tensile properties of r-value and strain-hardening exponent, presented as Equation (2.10) [126]. For Equations (2.9) and (2.10, \( t \) is thickness (in), \( \varepsilon_t(pct) \) is the TE in the transverse orientation, \( r_m \) is the average anisotropy, and \( n_t \) is the strain-hardening exponent in the transverse orientation. Figures 2.33a and 2.33b show the experimentally-determined HER plotted against the calculated HER using Equation (2.9) and Equation (2.10), respectively. Tensile and hole expansion data were gathered from ferritic, ferritic stainless, and austenitic stainless steels on both machined holes (Figure 2.33a) and sheared holes (Figure 2.33b). For the data in Figure 2.33, hole expansion tests were performed with a lubricated 102 mm diameter spherical punch, and the solid lines represent a 1:1 correlation. The accuracy of Equations (2.9) and (2.10) in predicting HER values based on tensile properties is promising, but further improvements are needed before the equations can be applied to steel grades outside of those represented in Figure 2.33.
\[ HER \text{ (pct)} = 478t + 2.56\varepsilon_t \text{(pct)} + 35.3r_m - 58.2 \] (2.9)

\[ HER \text{ (pct)} = 85.7r_m - 31.4n_t - 23.6 \] (2.10)

Kumar et al. also performed a regression analysis for a large group of HER and tensile properties produced by different researchers, and the resulting equation is presented as Equation (2.11, where \( \sigma_{UTS} \) is the UTS, \( r_m \) is the average anisotropy, and \( \varepsilon_t \) is the TE [128]. Figure 2.34 shows the experimentally-determined HER values plotted against the HER values calculated using Equation (2.11, and a good correlation \( R^2 > 0.9 \) was achieved. Hole preparation method and punch geometry were not specified for the different steels. Some of the steel grades incorporated for the analysis by Kumar et al. are indicated in the legend of Figure 2.34. In Figure 2.34, the solid line represents a 1:1 correlation.

\[ HER \text{ (pct)} = -48 + 302e^{-0.0035\sigma_{UTS}} + 144(1 - e^{-0.6r_m}) + 0.1\varepsilon_t \] (2.11)

(a) 

(b)
Equation (2.11 developed by Kumar et al. is also promising for correlations between tensile properties and HER values. However, both the imposed stress state and the microstructural properties synergistically interact to produce the resulting HER, tensile properties, R/t*, or any other property derived from mechanical tests [22], and defining relationships between HER as a function of microstructural properties is believed to provide additional insight.
CHAPTER 3
EXPERIMENTAL DESIGN

The motivation for the current project is first introduced, citing specific conclusions from the M. Sc. Thesis of M. D. Taylor. The project focus is then introduced, followed by the experimental approach designed to address the research topic.

3.1 Purpose of Project

The M.Sc. Thesis of M. D. Taylor quantified the constituent hardness of commercially-produced DP steels, and concluded that a lower martensite/ferrite hardness ratio decreased the void population for an equivalent deformation using plane strain tensile specimens. Laboratory tests, such as stretch bend and hole expansion testing used to evaluate industrial forming operations, also generate complex, triaxial stress states upon deformation, and it is hypothesized that a lower martensite/ferrite hardness ratio will suppress microstructural damage, leading to higher forming limits. An increase in HER with decreasing martensite hardness was reported for eight commercially-produced DP980 steels [76], and it was hypothesized that the correlation would improve if microstructural variables of grain size, MVF, chemical content, and morphology remained constant. The current project focused on evaluating the isolated effects of constituent hardness on the subsequent formability performance of DP steels.

3.2 Design of Project

Four commercially-produced DP steels with UTS values above 980 MPa were provided for the present study. An initial material characterization including constituent grain size, MVF, retained austenite content, constituent hardness, tensile properties, and tempering response was conducted to determine a preferred DP steel for a subsequent, in-depth analysis relating constituent hardness effects to formability performance.

The primary focus of the current project was to evaluate the isolated effects of constituent hardness on the subsequent formability performance of DP steels. Tempering and cold-rolling treatments were used to methodically create different constituent hardness conditions from one single DP steel chemistry. Moderate tempering and cold-rolling treatments have minimal effect on the grain size, MVF, morphology, and chemical content, making constituent hardness the primary microstructural variable. Tempering was used to create four unique hardness conditions, each with a UTS below the as-received condition. To evaluate steel conditions of equivalent UTS, four additional hardness conditions were generated by cold-rolling the four tempered conditions to re-attain the UTS of the as-received condition, totaling nine conditions altogether (as-received, four tempered, four temper-cold rolled). Nanoindentation
was used to quantify the constituent hardness properties of the modified steel conditions. To evaluate formability, the different steel conditions were tested in both uniaxial tension and hole expansion. Correlations between constituent hardness properties and performance in uniaxial tensile and hole expansion testing were then evaluated.

The microstructural response after plastic deformation was of interest, and techniques including nanoindentation, EBSD, and DIC were used to evaluate select steel conditions. The plane strain tensile test developed in the M.Sc. Thesis of M. D. Taylor was performed for select steel conditions to determine whether consistent trends were observed, providing a link between the two studies.
CHAPTER 4
EXPERIMENTAL METHODOLOGY

The chemical contents and thicknesses for the four commercially-produced DP steels used in the current study are first introduced. Descriptions are provided for the experimental setup for tempering, along with the parameters used for a preliminary tempering study; the method used for cold-rolling, along with a study focusing on the increase in UTS as a function of percent cold-rolling (%CR); a grain size/MVF measuring technique designed for a two-phase microstructure; the experimental setup for X-ray diffraction (XRD) to assess retained austenite content; a nanoindentation method used to quantify individual constituent hardness values; the experimental setup used for the grain orientation measurement technique of EBSD; the method used for the computer-aided strain mapping technique of DIC; the experimental setups for uniaxial tensile testing, hole expansion testing, and plane strain tensile testing developed at the Colorado School of Mines; and a void analysis technique developed for fractured ASTM E8 tensile specimens. Appendix A is also included to quantify uncertainties associated with reported microstructural/mechanical properties in Chapter 5 calculated from multiple tests.

4.1 Materials

Four commercially-produced, un-coated DP steels with UTS greater than 980 MPa were provided by Arcelor-Mittal for the current study. The four steels were arbitrarily labeled A-D, and their chemical contents (in wt pct) along with their as-received thicknesses (in mm) are reported in Table 4.1. Steels A-D are considered low-carbon and have relatively low alloying additions, characteristic of DP steels. Characterization and testing techniques on Steels A-D were performed in the as-received condition, and modified conditions. The steel condition in which each technique was performed is reported.

<table>
<thead>
<tr>
<th>wt pct</th>
<th>Thickness (mm)</th>
<th>C</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Si</th>
<th>Al</th>
<th>Nb</th>
<th>Ti</th>
<th>N</th>
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<td>1.2</td>
<td>0.14</td>
<td>1.81</td>
<td>0.017</td>
<td>0.002</td>
<td>N/R</td>
<td>0.04</td>
<td>N/R</td>
<td>N/R</td>
<td>0.006</td>
</tr>
<tr>
<td>B</td>
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<td>0.14</td>
<td>2.04</td>
<td>0.009</td>
<td>0.001</td>
<td>N/R</td>
<td>N/R</td>
<td>N/R</td>
<td>N/R</td>
<td>0.005</td>
</tr>
<tr>
<td>C</td>
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<td>0.15</td>
<td>1.46</td>
<td>0.010</td>
<td>0.007</td>
<td>0.30</td>
<td>0.04</td>
<td>&lt;0.003</td>
<td>0.002</td>
<td>0.004</td>
</tr>
<tr>
<td>D</td>
<td>1.5</td>
<td>0.10</td>
<td>2.15</td>
<td>0.010</td>
<td>0.001</td>
<td>N/R</td>
<td>0.04</td>
<td>N/R</td>
<td>N/R</td>
<td>0.004</td>
</tr>
</tbody>
</table>

4.2 Tempering Study

To characterize the softening kinetics of steels A-D, a tempering experiment was performed. Tempering is a heat treatment that softens a steel microstructure, and is more extensively discussed in
Sec. 2.3. Coupons of each steel, approximately 13 X 13 mm, were polished to 1 μm diamond using standard metallographic techniques, placed inside a stainless steel bag flooded with argon gas, and creased closed. Argon gas provided an inert atmosphere to the steel coupons, reducing decarburization of the steel surface during tempering. Once the open-atmosphere Carbolite® furnace reached the target temperature, the stainless steel bags containing the specimens were placed inside the furnace, and 6 min were added to each tempering time to allow the steel specimens to reach temperature. Six individual tempering temperatures of 175, 200, 225, 250, 300, and 350 °C were used for the tempering experiment. Vicker’s hardness measurements were performed on the tempered steel coupons after 0.5, 1, 2, 4, 8, 16, 24, 48, 72, and 120 hrs for each tempering temperature to quantify the change in hardness as a function of both time and temperature. For each time step, the polished steel coupons were removed from the furnace, and allowed to cool to room temperature inside the stainless steel bag before hardness testing. Six Vicker’s indents performed using a 500 g load and a dwell time of 10 s on the in-plane orientation of the polished steel coupons were averaged for every reported hardness value. Each Vickers indent was spaced at least 3 indent diameters apart to avoid overlapping plastic zones [86]. For each steel, a total of 60 hardness values (6 temperatures, 10 times) were reported.

In addition to the steel coupons, one ASTM E8 standard size tensile specimen of each steel (A-D) was placed inside the stainless steel bag for each of the six individual tempering temperatures. The ASTM E8 standard size tensile specimens were tempered at each of the six temperatures for 120 hrs, resulting in six tempered tensile specimens for each steel. The tempered tensile specimens were tested according to Sec. 4.9 to evaluate the change in tensile properties as a function of tempering. An equation developed by Holloman and Jaffe combined the effects of both tempering time and temperature into one value, known as the Hollomon-Jaffe parameter (HJP), and the basis for this analysis was discussed in Sec. 2.3.1 [49]. Tensile properties generated using the tempered tensile specimens were evaluated as a function of HJP.

4.3 Cold-Rolling Experiment

The increase in UTS as a function of %CR was evaluated on tempered ASTM E8 standard size tensile specimens of steel C. Cold-rolling is a plastic deformation process capable of changing the mechanical properties of steels, and is more extensively discussed in Sec. 2.4. Cold-rolling for all tensile specimens was performed along the original rolling direction. One group of six tensile specimens in the as-received condition was tempered to 960 MPa UTS, and a second group of six tensile specimens in the as-received condition was tempered to 920 MPa UTS. For both groups, tensile specimens were placed inside a stainless steel bag flooded with argon gas, and creased closed. Once the open-atmosphere Carbolite® furnace reached the target temperature, the stainless steel bags containing the specimens were
placed inside the furnace, and 6 min. were added to each tempering time to allow the steel specimens to reach temperature. After the tempering treatment, specimens were removed from the furnace and allowed to cool to room temperature inside the stainless steel bag before analysis. The tempering time-temperature combinations used for the two groups were determined from the experimental results of Sec. 4.2. Machined tensile specimens were cold-rolled using a 92 mm (3-5/8 inch) diameter two-high Stanat® cold rolling mill at CSM, and a picture of the rolling mill is shown in Figure 4.1. To reduce friction, a heavy duty lubricant was generously applied to the tensile specimen and rolls during cold-rolling, and a guide fixture was used to help maintain straightness of the tensile specimen being inserted into the rolls. Multiple passes were used to achieve each %CR, and all passes were kept below the $\Delta = 2$ threshold for non-uniform deformation based on Equation 2.2 [63]. After the target %CR values were achieved, the tensile specimens were tested according to Sec. 4.9.

Figure 4.1 Photo of a 92 mm diameter two-high rolling mill at CSM. A bottle of lubricant used for the cold-rolling treatments is also shown in the picture.

One specimen from each group was tensile tested in the as-tempered condition to establish a reference UTS, and the other five tensile specimens from each group were cold-rolled to 5, 8, 10, 15, and 20 %CR to characterize the increase in UTS as a function of %CR. Equation 4.1 was used to calculate %CR, where $L_o$ and $L_f$ are the initial and final length, respectively, of the tensile specimen gauge length. In order to accurately measure the gauge length, a line perpendicular to the tensile axis was scribed across the width on each end of the gauge length, and these two scribed lines were measured using a caliper with 0.013 mm (0.0005 inch) resolution.

$$\%CR = \left( \frac{L_f - L_o}{L_o} \right) \times 100$$

(4.1)
4.4 Grain Size and Martensite Volume Fraction Analysis

A grain size/MVF measurement technique developed by Higginson & Sellars was adapted to evaluate steel microstructures in the as-received and modified conditions [141]. For each steel, the in-plane, longitudinal, and transverse orientations were mounted, polished to 1 μm diamond using standard metallographic techniques and etched with 2 pct nital for approximately 10 s to reveal the microstructure. Figure 4.2 shows a schematic of the in-plane, longitudinal, and transverse orientations in relation to the original rolling direction of a sheet steel.

![Rolling Direction](image)

Figure 4.2 Schematic illustrating the in-plane, longitudinal, and transverse orientations in relation to the original rolling direction.

Six SEM micrographs were acquired in random locations from both the in-plane orientation, and the longitudinal orientation, totaling twelve micrographs for each steel. Both the in-plane and longitudinal orientations were evaluated to account for potential deviations in grain size between different orientations. Micrographs were acquired using a JEOL 7000F FESEM at 5 kV accelerating voltage, 10 mm working distance, and a spot size of 3. For grain size/MVF analysis, an overlay consisting of three concentric circles with 60 proportionally-spaced tick marks was applied twice to each of the twelve micrographs, randomly locating the grid for each analysis. An illustration of the concentric circles on an SEM micrograph of the in-plane orientation of Steel B is shown in Figure 4.3.

For each measurement, the 60 tick marks were used to determine an average MVF using standard point counting methods, and the circumference intercepts with ferrite/ferrite and martensite/ferrite boundaries were separately counted to determine the average grain size for both martensite and ferrite. Equations 4.2 and 4.3 were used to determine average ferrite and martensite grain sizes, respectively, where \( L_\alpha \) is the average ferrite grain size, \( L_{\alpha'} \) is the average martensite grain size, \( L \) is the circumferential length of the three circles, \( V_{f\alpha} \) is the MVF, \( n_\alpha \) is the number of ferrite/ferrite boundary intercepts, and \( n_{\alpha'} \) is the number of ferrite/martensite boundary counts. For each steel, 24 separate measurements of MVF, ferrite grain size, and martensite grain size were obtained, and then averaged. Standard deviations were calculated for the 24 measurements of ferrite grain size, martensite grain size, and MVF to express the uncertainty associated with the reported averages, and are presented in Appendix A.
Figure 4.3 Concentric circle overlay with 60 proportionally-spaced tick marks used for the grain size/MVF analysis. Micrograph of the in-plane orientation of Steel B is shown. Steel B was etched with 2 pct nital for approximately 10 s.

\[
L_\alpha = \frac{(1 - V_{f-a'}) \cdot L}{(n_\alpha + 0.5n_{a'})} \tag{4.2}
\]

\[
L_{a'} = \frac{2 \cdot V_{f-a'} \cdot L}{n_{a'}} \tag{4.3}
\]

4.5 X-Ray Diffraction

The in-plane orientation of each steel in the as-received condition was evaluated for austenite content using XRD. Specimens for XRD analysis, approximately 25 X 18 mm, were mechanically polished to 6 μm diamond using standard metallographic techniques, then subsequently submerged for 5 min in a solution of 1 part hydrofluoric acid, 10 parts hydrogen peroxide, and 10 parts de-ionized water. The plastic beaker containing the acid solution was placed inside a larger, water-filled beaker at 21 °C for temperature control. After 5 min in solution, specimens were rinsed with ethanol, and air-dried. The acid treatment removed the surface-hardened layers induced by the 6 μm diamond mechanical polishing step. Surface-hardened layers created by mechanical polishing can potentially transform austenite at the surface to martensite, and give inaccurate measurements of the true amount of retained austenite present in the microstructure. The acid solution was prepared separately for each steel specimen.
A Phillips® X’Pert Diffractometer equipped with a copper target, 45 kV generator voltage, 40 mA tube current, and a step size of 0.0167 was used over a 35° - 105° scan range to analyze steel specimens. The resulting intensity versus 2θ plots were qualitatively observed for the presence of austenite.

4.6 Nanoindentation

Nanoindentation was used to quantify the bulk hardness, average ferrite hardness, and average martensite hardness on the in-plane and longitudinal orientations of steel specimens in the as-received and modified conditions. Specimens for nanoindentation analysis were mounted, polished to 1 μm diamond using standard metallographic techniques, and then vibratory polished with 0.05 μm colloidal silica for approximately 10 hrs. Vibratory polishing with colloidal silica is both a mechanical and chemical material removal method, and is believed to remove much of the surface-hardened layer induced by the 1 μm diamond polishing step [98]. Next, Vickers indents were placed on the specimen surface as fiducial markers to aid in locating the nanoindentation grid. The nanoindentation specimens were then attached to a metal plate using an adhesive (CrystalBond) to take advantage of the magnetic stage in the nanoindenter. A Hysitron® TI-950 Triboindenter equipped with a Berkovich indenter tip was used for all nanoindentation tests. A 15 X 15 array (225 total) of indents spaced 2 μm apart was performed to a depth of 40 nm using a 2 s load – 2 s hold – 2 s unload function. To collect the most accurate data, tip area functions were analyzed periodically to account for the blunting of the tip over time. At least three separate area functions were performed for the Berkovich tip over the course of the nanoindentation experiments, and a detailed explanation on performing a tip area calibration can be found elsewhere [85]. A load-displacement curve was produced by each indent, and the unloading portion of the curve was used to calculate a hardness value using the Oliver-Pharr method [96]. An example of a load-displacement curve for an indent performed to a depth of 40 nm on steel C is shown in Figure 4.4.

![Load-displacement curve](Figure 4.4 Load-displacement curve generated by an indent on steel C. Indent was performed to a depth of 40 nm, resulting in a required indentation force of 337 μN.)
Using the unloading portion of the curve (indicated by an arrow) and the tip area function, a hardness value was calculated. A more detailed explanation on calculating hardness from load-displacement curves is outlined in Sec. 2.5.4. The hardness values (225 total) generated for each steel condition were averaged to obtain a bulk hardness. In addition, a method outlined in the M.Sc. Thesis of M. D. Taylor [40] was applied to the 225 indents from each nanoindentation test to obtain constituent hardness values for both ferrite and martensite. Indents located within 1.5 indent diameters of an interface were discarded due to potential strengthening effects from the adjacent phase/boundary [86], and all other indents located entirely within either ferrite or martensite were used to determine the average ferrite and average martensite hardness, respectively.

To determine the location of each indent, an SEM micrograph of the 15 X 15 indent array was acquired on the as-polished surface, and on the surface after being etched with 2 pct nital for approximately 6 s. A JEOL 7000F FESEM with 5 kV accelerating voltage, 10 mm working distance, and a spot size of 3 was used for image acquisition of the nanoindentation arrays. When imaging the nanoindentation array in the etched condition, indents were difficult to resolve. A transparency containing the indent locations was created using the SEM micrograph of the as-polished surface, and the transparency was overlaid onto the SEM micrograph of the etched surface to reveal the indent locations within the microstructure. Figure 4.5 shows an SEM micrograph of steel C in the etched condition with the 15 X 15 indent array overlaid. A more detailed explanation of the nanoindentation technique can be found elsewhere [40].

![Figure 4.5](image)

**Figure 4.5** 15 X 15 indent array overlaid on a micrograph of Steel C in the etched condition, allowing the determination of each indent location with respect to the phases present.
Histograms for the ferrite hardness data and martensite hardness data were created for steels A-D in the as-received condition to determine whether an average was an appropriate parameter to characterize the constituent hardness, and are presented in Appendix A. Additionally, standard deviations for the average ferrite hardness and average martensite hardness were calculated for the modified conditions of steel C to express the uncertainty associated with the reported averages, and are presented in Appendix A.

4.7 Electron Backscatter Diffraction

Observations of crystallographic rotations within individual grains after plastic deformation were analyzed using EBSD on the in-plane orientation of the as-received condition of steel C, and one modified condition of steel C. Specimens for EBSD were mounted, polished to 1 μm diamond using standard metallographic techniques, and then vibratory polished using 0.05 μm colloidal silica for 4 hrs. The vibratory polish was used to remove surface-hardened layers caused by the 1 μm diamond polishing step, which can degrade the EBSD data. To avoid charging of the non-metallic mount inside the SEM during EBSD analysis, the entire mount, except for the steel surface to be analyzed, was masked with a conductive carbon agent. Charging of the surface can result in a drift of the electron beam during acquisition, producing an inaccurate rendition of the microstructure. A 50 X 50 μm region was analyzed using a 20 kV accelerating voltage, 13 spot size, 15 mm working distance, 90 nm step size, and 4 X 4 binning on a JEOL 7000F field emission SEM equipped with TSL OIM Data Collection software. The resulting inverse pole figure (IPF) and image quality (IQ) maps were qualitatively assessed.

4.8 Digital Image Correlation

To observe potential heterogeneous strain distributions at the microstructure-level caused by plastic deformation, the DIC technique was applied to the as-received condition of steel C, and a modified condition of steel C using ASTM E8 sub-size tensile specimens. For both conditions, the in-plane orientation of the gauge section was polished to 1 μm diamond using standard metallographic techniques, and then etched with 2 pct nital for approximately 8 s. Three areas within the gauge section were analyzed using DIC, and Vickers indents were used as fiducial markers to locate each area. Figure 4.6 shows an illustration of the Vickers indents and corresponding areas within the gauge length of an ASTM E8 sub-size tensile specimen.

Ten SEM images were acquired from each of the three areas in the initial (un-deformed) state. All images for the DIC study were acquired at a resolution of 1024 X 884 using the FEI® Helios 600 field emission SEM using an accelerating voltage of 5 kV, probe current of 0.17 nA, a working distance of 4.1 mm, and an image acquisition dwell time of 40 μs. After imaging, the ASTM E8 sub-size tensile specimen was removed from the SEM chamber and strained a pre-determined amount on the MTS®
Alliance screw-driven tensile frame using a strain rate of $2.5 \times 10^{-3} \text{s}^{-1}$. Strain was measured using a 25.4 mm (1 inch) extensometer. Once the desired deformation (strain) was achieved, the tensile specimen was unloaded at a cross-head speed of 2 mm/min. The deformed tensile specimen was then removed from the tensile frame, and placed back into the SEM chamber for imaging. Ten SEM images were acquired for each of the three areas previously imaged using the same microscope settings. Additional deformation steps followed the same tensile straining and image acquisition procedure outlined above.

![Figure 4.6](image_url)  
**Figure 4.6** Schematic illustrating the three areas (Area 1 – Area 3) on the in-plane orientation of the gauge section (dashed boxes) of an ASTM E8 sub-size tensile specimen. The Vickers indents, represented by the black diamonds, were used to aid in locating the three areas. Tensile axis is horizontal.

Images acquired for the three areas were evaluated using NCORR®, a 2-Dimensional DIC analysis software. Using NCORR® for DIC analysis of the microstructure, a specific region of the SEM image was selected, and an example is illustrated in Figure 4.7 by the shaded box overlaid on an SEM image of the in-plane orientation of steel C in the initial (un-deformed) state. Next, the subset size and subset spacing were chosen. Subset size determined the local precision of each strain measurement; a larger subset size yields an average of a larger region, and a smaller subset size yields an average of a smaller region. To increase local precision, a 15 pixel radius subset size was chosen for the present study. Subset spacing determined the number of pixels between each strain measurement. Smaller subset spacing yielded higher spatial resolution, but increased the processing time. A subset spacing of 2 pixels was chosen for the present study.

The final input for DIC analysis was to establish a reference point between the SEM images being analyzed. An illustration of a reference point is shown in Figure 4.7 with a black dot inside the selected region of the SEM image. A reference point was chosen on the un-deformed SEM image, and the software located the same point in the deformed SEM image. Figure 4.8a shows the 15 pixel radius subset of the reference point (black circle in Figure 4.7) from the un-deformed SEM image, and Figure 4.8b shows the 15 pixel radius subset for the reference point found in the deformed SEM image. Once an equivalent feature was established between the two SEM images, the DIC software initiated strain calculations. The computer-aided DIC technique is more extensively discussed in Sec. 2.6.
Figure 4.7  SEM image of the in-plane orientation of steel C. The selected region for DIC analysis is represented by the shaded box, and the black circle represents the reference point used to initiate strain correlations between the un-deformed and strained states. Steel C was etched with 2 pct nital for approximately 8 s to reveal the microstructure.

Figure 4.8  Subset of pixels (15 pixel radius) representing the reference point in the un-deformed SEM image (a), and in the deformed SEM image (b). The same feature was found in both states, establishing a spatial relationship between the two images.

Data analysis for DIC required multiple sequential steps. First, images from the same state (un-deformed, or strained) were analyzed with DIC to determine the background noise associated with the SEM image acquisition technique. Next, images from different states (un-deformed and deformed) were analyzed with DIC to characterize the distribution of strains within the microstructure.
4.9 Tensile Testing

Uniaxial tensile testing was conducted using an MTS® Alliance screw-driven tensile frame for steels A-D in the as-received and modified conditions. For all conditions, tensile specimens were tested with the tensile axis parallel to the rolling direction (RD), but for the as-received conditions, tensile specimens with the tensile axis perpendicular to the RD were also tested. Unless otherwise noted, ASTM E8 standard size tensile specimens with a gauge section of 57 mm x 12.7 mm x sheet thickness were used for tensile testing [142]. A strain rate of $2.5 \times 10^{-3} \text{ s}^{-1}$ was used, along with a 51 mm extensometer to measure strain in the gauge section. The data acquisition software, TestWorks4®, recorded the tensile test parameters in pounds (lbs), and inches. To obtain engineering stress (in MPa), and engineering strain, Equations 4.4 and 4.5 were used, respectively, where $\sigma_{\text{eng}}$ is engineering stress, $P$ is the force (in newtons), $A$ is the initial cross-sectional area (in mm$^2$), $e_{\text{eng}}$ is engineering strain, $\Delta l$ is the change in length (inches), and $l_o$ is the initial gauge length.

\[
\sigma_{\text{eng}} = \frac{P}{A} = \frac{\text{force (lb)} \cdot \frac{\text{4448 Newtons}}{\text{lb}}}{A} \tag{4.4}
\]

\[
e_{\text{eng}} = \frac{\Delta l}{l_o} \tag{4.5}
\]

For each tensile test, YS, UTS, UE, and TE were obtained, and a schematic showing the tensile properties in relation to an engineering stress-engineering strain tensile curve is presented in Figure 4.9. In the event of continuous yielding (shown in Figure 4.9), a 0.2 pct strain offset was used to quantify YS, and in the event of discontinuous yielding, the lower yield point (YPE region in Figure 2.12) was used.

![Figure 4.9](image)

**Figure 4.9** Representative engineering stress-strain curve illustrating different tensile property values.
When more than one test was performed for the same steel condition, standard deviations were calculated for the YS, UTS, UE and TE to express the uncertainty associated with the reported averages, and are presented in Appendix A.

4.10 Hole Expansion Testing

Hole expansion coupons approximately 100 x 100 mm were used for testing the as-received and modified conditions of steel C. A sheared, 10 mm diameter circular hole was created in the center of each test coupon with a die/sheet clearance between 10 – 12.5 pct, in accordance with the ISO 16630 standard. For modified conditions of steel C, the modifying process (tempering and/or cold-rolling) was performed prior to hole shearing. Hole shearing and hole expansion testing were performed at Arcelor-Mittal Global R&D in E. Chicago, IN. A cross-sectional illustration of the hole expansion test is shown in Figure 2.26. The shearing process used to create the 10 mm hole creates a burr around the hole edge, and the microstructure adjacent to the hole is heavily deformed [127]. The heavily deformed region adjacent to the sheared hole, referred to as the shear affected zone (SAZ), is presented in more detail in Sec. 2.8.1. All specimens were tested with the burr facing upwards, in accordance with ISO 16630.

An unlubricated, 60° conical punch traveling uniaxially at 20 mm/min was used for hole expansion tests. The conical punch and the 10 mm sheared hole were concentric throughout the test. A clamping force of 22 kN was used to prevent the specimens from drawing upwards during the test. A high-speed camera positioned such that the 10 mm sheared hole was centered in the image frame was used for image acquisition during testing, as well as for determining when to manually stop the conical punch travel. A through-thickness crack exceeding 0.1 mm in width was the criteria for stopping the conical punch travel. The HER was determined using Equation 4.6, where \( d_o \) and \( d_f \) are the initial and final hole diameters, respectively.

\[
HER = \left( \frac{d_f - d_o}{d_o} \right) \cdot 100
\]  

(4.6)

The initial and final hole diameters were measured using three different methods. The first method involved a unique software program developed by researchers at Arcelor-Mittal Global R&D in E. Chicago, IN. An initial image from the high-speed camera before punch travel initiated was used to obtain the initial hole diameter (\( d_o \)). During testing, the software monitored images of the hole edge, and recorded the diameter (\( d_f \)) in the image that first detected a 0.1 mm through-thickness crack. The second method employed was physical measurements of the hole diameter before and after testing. A caliper with 0.013 mm (0.0005 inch) resolution was used to make four measurements spaced 45° apart on the initial (\( d_o \)), and final (\( d_f \)) hole diameter. The third method used the same images as the first method, but
diameter measurements were made using ImageJ®. Four measurements spaced 45° apart were made on the initial \( (d_o) \), and final \( (d_f) \) diameters of the hole. To illustrate the measurement locations for the second and third methods, Figure 4.10 shows the four measurements \((M1 – M4)\) made on a sheared hole after expansion. Multiple hole expansion tests were performed for each condition of steel C and standard deviations were calculated for the HER values obtained by the three different HER measurement methods to express the uncertainty associated with the reported averages, and are presented in Appendix A.

Figure 4.10 Image of a hole expansion test specimen (after failure) of steel C illustrating the four measurement locations \((M1 – M4)\) for the second and third methods. The bright circle is the sheared hole surface, and a crack is located at the bottom of the sheared hole. The bright region in the center is the top of the 60° conical punch. The bright dots surrounding the sheared hole are from a LED light ring used for illumination during image acquisition.

4.11 Plane Strain Tensile Testing

A specimen geometry designed to induce a shear failure when tested on a uniaxial tensile frame was developed and presented in the M.Sc. Thesis of M. D. Taylor [40]. The specimen geometry, termed a plane strain tensile specimen, is shown schematically in Figure 4.11a for a steel sheet of 1.02 mm thickness. Two parallel semi-circular notches on opposite faces extend across the width (25.4 mm) of the specimen perpendicular to the tensile axis, generating a triaxial stress state in the region between the notches. The semi-circular notches were created using electric discharge machining (EDM). The furthest protrusions of each notch into the specimen are offset by 45° with respect to the tensile axis, promoting a shear failure. A detailed view of the notched region (circled in Figure 4.11a) is shown in Figure 4.11b. The produced shear failure from the tensile testing of the plane strain tensile specimen is interpreted to be similar to shear failures observed in stretch bend tests of higher-strength AHSS grades. One of the
benefits of uniaxial tensile frames compared to the stretch bending frame is that a test can be stopped at a predefined displacement before failure occurs.

Plane strain tensile specimens were machined for the as-received condition of steel C, and a modified condition of steel C. For each condition, the semi-circular notches were dimensioned such that a 0.6 mm x 0.6 mm reduced section was created between the notches, and the furthest protrusion of each notch into the material were 45° from each other relative to the tensile axis (Figure 4.11b).

One plane strain tensile specimen of each condition was tested to fracture on the MTS® Alliance screw-driven tensile frame to establish a reference failure displacement. A cross-head speed of 1 mm/min was used for all plane strain tensile tests. Load-displacement data for plane strain tensile tests were kept in units of kg-mm since defining stress and strain values for the complex, asymmetric reduced section was interpreted to provide limited additional insight. Failure displacement was measured using a 12.7 mm gauge length monitored with a 25.4 mm (1 inch) extensometer through use of a special fixturing unit available at CSM. Subsequent plane strain tensile specimens were tested to 90% of the failure displacement previously established, and then unloaded. A failure displacement of 90% was chosen because it was shown to result in significant localized void formation in plane strain tensile
specimens [40]. Load-displacement curves for a DP780 plane strain tensile specimen from a previous study [40] loaded to failure (solid line), and to 90% failure displacement (dashed line) are shown in Figure 4.12.

Figure 4.12    Load-displacement curves for a DP780 plane strain tensile specimen tested to failure (solid line), and to 90% of the failure displacement (dashed).

The specimens tested to 90% failure displacement were cross-sectioned at mid-width, and the longitudinal orientation was mounted, polished to 1 μm diamond using standard metallographic techniques, and then etched using 2 pct nital for approximately 8 s. Figure 4.13 shows a light optical micrograph (LOM) of a DP780 steel from a previous study [40] to illustrate the cross-sectioned plane.

The etched cross-section was then placed in the JEOL 7000F FESEM, and voids were observed using backscatter electron (BSE) imaging in composition mode with 20 kV accelerating voltage, 10 mm working distance, and a 14 spot size. The BSE imaging mode was chosen because of the high contrast produced between the steel surface and voids. Nine SEM micrographs were obtained from the region between the two semi-circular notches, and the approximate location of the micrograph areas are represented as black boxes in Figure 4.13. The nine SEM micrographs characterize the critical region where failure is known to occur. The seven innermost images (the two micrographs immediately adjacent to the semi-circular notches were omitted) were analyzed for their void content by using a grayscale threshold function in ImageJ®. A micrograph from the critical region (represented by one of the nine black boxes in Figure 4.13) of a DP780 steel [40] is shown in Figure 4.14a. The grayscale threshold function converted the SEM image in Figure 4.14a into the binary white/black image observed in Figure 4.14b. From Figure 4.14b, properties such as the void area percent, and number of voids at 90%
failure displacement were obtained. A more detailed description of the analysis technique is provided elsewhere [40].

![Image](image1.png)

**Figure 4.13** Illustration of the image acquisition method used to quantify the void population in the critical area of interest for the plane strain tensile tests. The nine rectangles represent the areas imaged, and are superimposed on a LOM micrograph of a DP780 steel from a previous study [40].

![Image](image2.png)

**Figure 4.14** SEM micrograph from the critical region (nine boxes in Figure 4.13) of the plane strain tensile specimen shown in Figure 4.13 in (a), and the same micrograph after processing with ImageJ® in (b).

### 4.12 Void Analysis of Fractured Tensile Specimens

The void area percentages as a function of thickness strain for the as-received condition of steel C, and for two modified conditions of steel C were characterized using fractured ASTM E8 standard size...
tensile specimens. ASTM E8 standard size tensile specimens were tested according to Sec. 4.9, and the fractured end was cross-sectioned through-thickness at mid-width, and the longitudinal orientation was mounted, polished to 1 μm diamond using standard metallographic techniques, then etched with 2 pct nital for approximately 8 s. SEM micrographs were acquired on the JEOL 7000F FESEM using the same microscope settings outlined in Sec. 4.11. Nine defined steps from the fracture end were established, and five SEM micrographs proportionally spaced along the thickness direction were acquired at each of the nine steps. Figure 4.15 shows an SEM micrograph of the longitudinal orientation for a modified condition of steel C, and illustrates the location of the five SEM micrographs (black boxes) for the first 8 steps. In Figure 4.15, the fracture end is on the left-hand size, the thickness direction is vertical, and the dashed white line indicates mid-thickness. The SEM micrographs acquired at each of the nine steps were analyzed for void area pct with ImageJ® using the same technique outlined in Sec. 4.11. Thickness strain for each step was calculated using Equation 4.7, where \( t_{local} \) is the local thickness (in mm), and \( t_i \) is the initial thickness of the tensile specimen (in mm).

\[
\text{Local Thickness Strain (pct)} = \frac{\left( t_i - t_{local} \right)}{t_i} \times 100
\]

(4.7)

![Figure 4.15](image)

Figure 4.15 SEM micrograph showing the longitudinal orientation of the fractured end of an ASTM E8 tensile specimen of a modified condition of steel C. Black boxes represent approximate locations where SEM micrographs were acquired for void analysis for the first 8 steps. Fracture surface is on the left-hand side of the image, and thickness direction is vertical. The white dashed line represents the mid-thickness of the tensile specimen.
CHAPTER 5
RESULTS & DISCUSSION

Results obtained using the experimental methods outlined in CHAPTER 4 are organized into three sections. First, results from the microstructural, tempering, and tensile characterizations of steels A-D are presented, and a preferred steel for subsequent analysis is identified. Using results from the first section, the second section presents nine different conditions of Steel C (i.e. the selected steel) created by tempering and cold-rolling. Hole expansion test data for the nine conditions are then presented, followed by constituent hardness values obtained using nanoindentation. Correlations between constituent hardness and both tensile and HER properties are presented, followed by a comparison of the generated data to selected studies from literature that focus on characterization of HER based on tensile properties. The third section presents the microstructural response to plastic deformation for two specific conditions of steel C using techniques of EBSD, DIC, and plane strain tensile tests.

5.1 Characterization of Candidate Steels

Microstructural properties for steels A-D in the as-received condition are first presented, followed by a tempering analysis performed to evaluate the softening response of each steel. Evolution of tensile properties as a function of tempering for steels A and C is then presented, followed by the results from a cold-rolling experiment on steel C.

5.1.1 Microstructural Properties of Steels A-D

Figures 5.1a – 5.1d show SEM micrographs of the in-plane orientation of steels A-D, respectively. In Figure 5.1, all steels were polished to 1 µm diamond using standard metallographic techniques, then etched with 2 pct nital for approximately 8 s. The darker, featureless regions are interpreted to be ferrite, and the brighter raised regions, sometimes accompanied by a white outline, are interpreted to be martensite. For visual reference, an “F” is placed on a ferrite grain, and an “M” is placed on a martensite grain in Figure 5.1c for steel C. Steels B, C, and D qualitatively appear more equiaxed than steel A, and steel D exhibits a bimodal distribution of martensite grain sizes. Some of the smaller martensite grains in steel D appear featureless and may potentially be austenite. The average grain size of steel C qualitatively appears to be larger than steels A, B, and D, and quantitative microstructural data is presented below.

Figure 5.2 shows representative engineering stress-engineering strain tensile curves for steels A-D in the as-received condition generated using ASTM E8 standard size tensile specimens with the RD parallel to the tensile axis. Tensile tests were conducted according to Sec. 4.9. Continuous yielding
behavior characteristic of DP steels is observed for all four steels. Steels A and B are classified as DP1180 (DP steel with minimum UTS greater than 1180 MPa), and steels C and D are classified as DP980.

Figure 5.1 SEM micrographs of the in-plane orientation for steels A – D. All steels were etched with 2 pct nital for approximately 8 s. Rolling direction is vertical for each micrograph.

Results from the tensile analysis, grain size/MVF analysis, and XRD analysis for steels A-D in the as-received condition are presented in Table 5.1. Each steel satisfied its respective minimum strength requirement, with steels A and B both achieving a UTS greater than 1180 MPa, and steels C and D both achieving a UTS greater than 980 MPa. Tensile properties of YS, UE, and TE also reported in Table 5.1 show steels C and D to exhibit larger UE and TE values compared to steels A and B. Tensile properties reported in Table 5.1 for steels A-D are the average of three tensile tests. Table A.1 reports the data in Table 5.1, along with a standard deviation for each reported tensile property. Steels A-D exhibited an average ferrite grain size (G.S. α) ranging from 1.4 to 2.9 μm, average martensite grain size (G.S. α’) ranging from 1.3 to 3.1 μm, and MVF ranging from 44 to 61 pct. Steels A and B exhibited a larger MVF
compared to steels C and D, and steel C exhibited the largest average ferrite and average martensite grain sizes. Grain sizes and MVF values reported in Table 5.1 were the average of 24 separate calculations, and Table A.1 in the appendix reports standard deviations for each reported value.

Table 5.1 – Microstructural and Tensile Properties for Steels A-D

<table>
<thead>
<tr>
<th>Steel</th>
<th>YS (MPa)</th>
<th>UTS (MPa)</th>
<th>UE (pct)</th>
<th>TE (pct)</th>
<th>G.S. $\alpha$ (μm)</th>
<th>G.S. $\alpha'$ (μm)</th>
<th>MVF (pct)</th>
<th>Austenite</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>946</td>
<td>1225</td>
<td>5.9</td>
<td>10.8</td>
<td>1.42</td>
<td>1.86</td>
<td>60.9</td>
<td>No</td>
</tr>
<tr>
<td>B</td>
<td>923</td>
<td>1221</td>
<td>5.9</td>
<td>10.1</td>
<td>1.43</td>
<td>1.60</td>
<td>56.0</td>
<td>No</td>
</tr>
<tr>
<td>C</td>
<td>627</td>
<td>1081</td>
<td>7.8</td>
<td>11.7</td>
<td>2.94</td>
<td>3.14</td>
<td>54.5</td>
<td>No</td>
</tr>
<tr>
<td>D</td>
<td>727</td>
<td>1024</td>
<td>7.2</td>
<td>13.2</td>
<td>1.60</td>
<td>1.30</td>
<td>44.0</td>
<td>No</td>
</tr>
</tbody>
</table>

The results of the XRD analyses are also reported in Table 5.1, with all steels exhibiting austenite contents below the detection limit of the technique (approximately 3 volume pct). Steels containing austenite contents below the detection limit were reported as having “No” detectable amount.

Results for the nanoindentation analysis on steels A-D in the as-received condition are reported in Table 5.2, with average ferrite hardness ranging from 2.57 to 3.21 GPa, average martensite hardness ranging from 6.67 to 7.29 GPa, average bulk hardness ranging from 4.50 to 5.72 GPa, and the average hardness ratio ranging from 2.4 to 3.2. Steels A and B (DP1180) exhibited harder ferrite and harder martensite values compared to steels C and D (DP980). Ferrite and martensite hardness values reported
in Table 5.2 were the average of all indents classified as ferrite and martensite, respectively, according to Sec. 4.6. To determine whether an average was an appropriate value to characterize constituent hardness, separate histograms for ferrite and martensite were created. Ferrite hardness and martensite hardness histograms for steel C are shown in Figures 5.3a and 5.3b, respectively, and both exhibit a relatively normal distribution. Hardness histograms for steels A, B, and D are shown in Figures A.1, A.2, and A.3, respectively. Based on the relatively normal distributions observed for ferrite and martensite hardness values, it was interpreted that the average constituent hardness values were appropriate to characterize the microstructure. In every case, martensite and ferrite hardness data were separated (i.e. the softest martensite hardness value was harder than the hardest ferrite hardness value).

<table>
<thead>
<tr>
<th>Steel</th>
<th>Hardness $\alpha$ (GPa)</th>
<th>Hardness $\alpha'$ (GPa)</th>
<th>$\alpha'/\alpha$</th>
<th>Avg Nano (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>2.97</td>
<td>7.04</td>
<td>2.37</td>
<td>5.61</td>
</tr>
<tr>
<td>B</td>
<td>3.21</td>
<td>7.29</td>
<td>2.27</td>
<td>5.51</td>
</tr>
<tr>
<td>C</td>
<td>2.15</td>
<td>6.84</td>
<td>3.18</td>
<td>5.02</td>
</tr>
<tr>
<td>D</td>
<td>2.57</td>
<td>6.67</td>
<td>2.60</td>
<td>4.50</td>
</tr>
</tbody>
</table>

Figure 5.3 Hardness histograms for ferrite in (a), and martensite in (b) for steel C in the as-received condition, obtained using nanoindentation. Both plots exhibit a relatively normal distribution.

During the project design phase, specific microstructural property ranges were desired to aid in achieving the project goals. An average grain size greater than 1.3 $\mu$m was desired so the nanoindentation technique would be within its resolution limit. A MVF between 30 and 65 pct was desired so the project focus would be applicable to higher-strength DP steels. Table 5.1 reports that steels A-D all meet the
grain size criteria and MVF criteria. Additionally, a high initial hardness ratio was desired so that a potentially larger range of hardness ratios could be generated upon tempering and cold-rolling. However, the degree of softening each steel exhibits upon tempering was required to be able to infer the range of hardness ratios produced by each steel.

5.1.2 Tempering Study of Steels A-D

Figure 5.4 shows the Vicker’s hardness (VHN) as a function of HJP for Steels A-D (preliminary study outlined in Sec. 4.2). An increase in time and/or temperature of the tempering treatment produces an increase in HJP, resulting in a softer microstructure (Equation 2.1). Steels A-D experienced slight hardening upon tempering at HJP values between 9,000 and 10,000, then continuously decreased in hardness with higher HJP values. The initial increase in constituent hardness values is consistent with previous studies involving tempering of martensite-containing steels [49]. Each data point shown in Figure 5.4 represents the average of six individual Vicker’s indents (Sec. 4.2).

The absolute change in hardness between the softest condition and the initial (as-received) condition of steels A – D is of interest to determine the softening response of each. Table 5.3 reports the Vicker’s hardness value for the as-received condition, and the maximum change in Vicker’s hardness due to tempering (ΔVHN) for steels A-D. Steels A and C produced the two largest differences in hardness upon tempering. Also reported in Table 5.3 is the change in hardness divided by the MVF to assess the
change in hardness per percent of MVF. The change in hardness per percent of MVF assumes that all softening occurred in martensite, and is interpreted to more accurately reflect the change in hardness of martensite, and subsequent hardness ratio. Table 5.3 also reports the average carbon content of martensite calculated using a basic rule of mixtures formula shown in Equation 5.1, where \( C_{\text{Bulk}} \) is the carbon content (wt pct) of each steel (Table 4.1), \( C_{\alpha'} \) is the carbon content (wt pct) of martensite, and a saturation of 0.022 wt pct carbon is assumed for ferrite.

\[
C_{\alpha'} = \frac{C_{\text{Bulk}} - 0.022(1 - MVF)}{MVF}
\]

(5.1)

The martensite carbon contents for steels A-D calculated using Equation 5.1 are similar, and suggest that other factors may be responsible for the observed change in hardness per percent of MVF for steels A-D. Steels A and C have the two largest changes in hardness per percent of MVF, making both good candidates for subsequent analysis. Evolution of tensile properties as a function of tempering was evaluated for steels A and C to determine the preferred steel for subsequent analysis.

Table 5.3 – Vickers Hardness Data Including the Initial Hardness, Change in Hardness Upon Tempering, and Average Martensite Carbon Content for Steels A-D

<table>
<thead>
<tr>
<th>Steel</th>
<th>VHN</th>
<th>( \Delta \text{VHN} )</th>
<th>( \Delta \text{VHN/MVF} )</th>
<th>Martensite C-Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>389</td>
<td>62</td>
<td>1.02</td>
<td>0.22</td>
</tr>
<tr>
<td>B</td>
<td>374</td>
<td>36</td>
<td>0.64</td>
<td>0.23</td>
</tr>
<tr>
<td>C</td>
<td>339</td>
<td>54</td>
<td>0.99</td>
<td>0.26</td>
</tr>
<tr>
<td>D</td>
<td>333</td>
<td>21</td>
<td>0.48</td>
<td>0.2</td>
</tr>
</tbody>
</table>

Tempering alters the constituent hardness of a microstructure, and the UTS obtained from uniaxial tensile testing was the parameter chosen to mechanically characterize the different constituent hardness conditions. A steel exhibiting a strong correlation between HJP and UTS will be best-suited for subsequent analysis.

5.1.3 Tensile Testing of Tempered Steels A and C

ASTM E8 standard size tensile specimens of steels A and C were tempered (discussed in Sec. 4.2) and tested according to Sec. 4.9. For all tests, the RD was parallel to the tensile axis, and the engineering stress-engineering strain tensile curves for the as-received condition, and tempered conditions of steels A and C are shown in Figures 5.5a and 5.5b, respectively. Other than the as-received condition, each tensile curve in Figure 5.5 represents a different tempering temperature. All tensile curves representing tempered conditions in Figure 5.5 exhibit YPE. Steel C exhibits a continual decrease in UTS with increasing tempering temperature. The UTS of steel A contains more variability, with different
tempering temperatures occasionally exhibiting equivalent UTS values. The HJP values, VHN values and tensile properties for the tensile curves shown in Figure 5.5 are reported in Table B.1, and the UTS and VHN values are plotted as a function of HJP for steels A and C in Figures 5.6a and 5.6b, respectively.

**Figure 5.5** Engineering stress-engineering strain tensile curves for the as-received condition and six tempered tensile specimens of steel A in (a). An equivalent UTS is observed for the 225 °C and 250 °C tempering temperatures. Engineering stress-engineering strain curves for the as-received condition, and six tempered tensile specimens of steel C in (b). With increasing tempering temperature at a constant time of 120 hrs, a continual decrease in UTS is observed.

**Figure 5.6** Plot of UTS and VHN as a function of HJP for steel A in (a), and steel C in (b). For both plots, the UTS-HJP data was fitted using a linear regression, and the VHN-HJP data was fitted using a 2nd degree polynomial function. In all cases, the correlation coefficient ($R^2$) is shown in the plot.
Each UTS value in Figure 5.6 represents one tensile test. In both plots, the UTS-HJP correlation was fit using a linear regression, and the VHN-HJP correlation was fit using a 2nd order polynomial regression. For each regression analysis, the correlation coefficient ($R^2$) is reported in the plots. The $R^2$ value for the UTS-HJP data are equivalent for steels A and C, and the $R^2$ value for the VHN-HJP relation is higher for steel C than for steel A. Based on a grain size greater than 1.3 μm, a MVF between 30 and 65 pct, a comparatively large change in hardness per percent of MVF upon tempering, and the highest $R^2$ values when correlating UTS and VHN data to HJP, steel C was chosen as the preferred steel for subsequent analysis. *For all subsequent experiments and discussions, only steel C is considered.*

5.1.4 Cold-Rolling Experiment: Steel C

After tempering, a DP steel typically experiences a decrease in UTS (Figure 5.5). To re-attain the as-received UTS, the process of cold-rolling was applied to strengthen the tempered steels. A preliminary study was performed to determine the amount of percent cold-roll (%CR) necessary to strengthen a steel back to the as-received UTS. To achieve a specific UTS based on tempering parameters (HJP), the equation for the linear fit to the UTS-HJP data in Figure 5.6b was used, and is presented as Equation 5.2.

$$UTS = -0.0399(HJP) + 1405$$ (5.2)

Two UTS values, 960 MPa and 920 MPa, were chosen to perform the cold-rolling experiment to examine whether the starting UTS value has an effect on the amount of strengthening imparted by cold-rolling. Using Equation 5.2, HJP values were determined for the two UTS values. After the HJP values were determined, a combination of time and temperature was chosen to satisfy Equation 2.1. Table 5.4 reports the target UTS, HJP, tempering time, tempering temperature, and resulting UTS for the two conditions.

<table>
<thead>
<tr>
<th>Target UTS (MPa)</th>
<th>HJP</th>
<th>Temp (°C)</th>
<th>Time (hr)</th>
<th>Resulting UTS (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>960</td>
<td>11158</td>
<td>250</td>
<td>21.6</td>
<td>970</td>
</tr>
<tr>
<td>920</td>
<td>12160</td>
<td>300</td>
<td>16.7</td>
<td>920</td>
</tr>
</tbody>
</table>

Six tensile specimens were tempered for each of the two target UTS conditions in Table 5.4. Specimens were tempered according to Sec. 4.3. One tensile specimen from each heat treatment was tested in the as-tempered condition (Resulting UTS reported in Table 5.4) to provide a basis on which to evaluate the increase in strength due to %CR. The five remaining tempered tensile specimens from each heat
treatment were cold-rolled to 5, 8, 10, 15 and 20 pct using the procedural outline in Sec. 4.3, and then tensile tested according to Sec. 4.9. Figure 5.7 shows the increase in UTS (ΔUTS) as a function of %CR, where the data generated using the 960 MPa target specimens are filled circles, and the data generated using the 920 MPa target specimens are open squares. Each data point in Figure 5.7 represents the results from one tensile test. During the processing of the different %CR values, a +/- 2 pct range was typical, and the actual %CR values are plotted (x-axis) in Figure 5.7. Table B.2 reports the tensile data, %CR values, and ΔUTS for the ten tensile specimens used to create the plot in Figure 5.7.

![Figure 5.7](image)

Figure 5.7  Plot of the increase in UTS (ΔUTS) as a function of %CR for tensile specimens of steel C tempered to 960 MPa (filled circles), and 920 MPa (open squares) UTS. A 2nd order polynomial regression was fitted to the data.

A 2nd order polynomial function was fit to the data in Figure 5.7, and the equation is reported as Equation 5.3.

\[ \text{ΔUTS(MPa) = } -0.173(%\text{CR})^2 + 10.7(%\text{CR}) \]  

(5.3)

The curve representing Equation 5.3 in Figure 5.7 has a correlation coefficient of 0.91, suggesting that the experimental data are characterized reasonably well. Equation 5.3 serves as an initial approximation for the amount of %CR required to achieve a particular UTS for tempered conditions of steel C.

5.2 Processing and Testing of Modified Steel C

Steel C was processed to create eight additional conditions by tempering and cold-rolling. The four conditions of steel C achieved by tempering are first presented, followed by the four conditions of steel C achieved by cold-rolling the previously tempered conditions. The eight modified conditions of
steel C were characterized/validated using tensile tests. All tensile tests were performed on ASTM E8 standard size tensile specimens according to Sec. 4.9.

For the nine conditions of steel C, hole expansion results are presented, followed by the constituent hardness results obtained using nanoindentation. Correlations between constituent hardness and the tensile/hole expansion properties are then presented. Select studies from literature that focus on characterizing HER based on tensile properties are then used to evaluate the nine conditions of steel C.

5.2.1 Four Tempered Conditions of Steel C

Steel C was tempered to produce four unique constituent hardness conditions. The as-received UTS for steel C was approximately 1080 MPa (Table 5.1), and four strength targets of 1050 MPa, 1020 MPa, 990 MPa, and 960 MPa were chosen. Each strength target was separated by 30 MPa to reduce the potential of overlapping UTS values when considering the inherent variability between similar tensile tests. For example, the 1081 MPa UTS reported for steel C in Table 5.1 was the average of three separate tensile tests whose individual UTS values were 1073 MPa, 1082 MPa, and 1089 MPa, resulting in a 16 MPa range. A UTS of 960 MPa was the lowest strength pursued so the project would remain relevant to higher-strength DP steels. Achieving the four strength targets was an iterative process of tempering, tensile testing, and potential modification of tempering parameters. For the first iteration, HJP values for the four strength targets were determined using Equation 5.2. Tempering time and tempering temperature combinations were chosen to satisfy the HJP, according to Equation 2.1. The target UTS, HJP, tempering time, tempering temperature, and resulting UTS for the four strength targets are reported in Table 5.5.

Table 5.5 –First Iteration of Target UTS, HJP, Tempering Temperature and Tempering Time for Four Tempered Conditions of Steel C

<table>
<thead>
<tr>
<th>Iteration 1 Target UTS (MPa)</th>
<th>HJP</th>
<th>Temp (°C)</th>
<th>time (hr)</th>
<th>Resulting UTS (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1050</td>
<td>8902</td>
<td>175</td>
<td>0.74</td>
<td>1067</td>
</tr>
<tr>
<td>1020</td>
<td>9654</td>
<td>200</td>
<td>2.57</td>
<td>n/a</td>
</tr>
<tr>
<td>990</td>
<td>10406</td>
<td>250</td>
<td>0.79</td>
<td>n/a</td>
</tr>
<tr>
<td>960</td>
<td>11158</td>
<td>275</td>
<td>2.3</td>
<td>n/a</td>
</tr>
</tbody>
</table>

The tempering parameters reported in Table 5.5 were used as first approximations to achieve the different strength targets. When the heat treatment for the 1050 MPa strength specimens was performed, the resulting UTS was 1067 MPa, significantly higher than the 1050 MPa target. The VHN vs. HJP plot in Figure 5.4 shows that an increase in hardness is initially observed, and it is believed that a specimen heat treated with an HJP of 8,902 might experience some of the strengthening effects caused by the increase in hardness, thereby complicating the results. To minimize the effects of hardening, the second
iteration involved lowering the target UTS value for all four conditions. In doing so, it was anticipated that the original strength targets (Table 5.5) could be achieved. For the second iteration, Table 5.6 reports the target UTS, HJP, tempering temperature, tempering time, resulting UTS, and difference between the target UTS and resulting UTS. Table 5.6 reports a decrease in the difference between the target UTS and resulting UTS with higher tempering temperatures. Each of the four resulting UTS values reported in Table 5.6 represents the average of three tensile tests. Table 5.7 reports the material designation, and the tensile properties of the four as-tempered (AT) steel conditions. The YS/UTS is a parameter that characterizes the relative increase in strength from plastic deformation, and is also reported in Table 5.7. Table A.3 presents the data shown in Table 5.7 with standard deviations for the UTS, YS, TE and UE.

Table 5.6 – Second Iteration of Target UTS, HJP, Tempering Parameters, Resulting UTS, and the Difference in UTS for Four Tempered Conditions of Steel C

<table>
<thead>
<tr>
<th>Iteration 2</th>
<th>Target UTS (MPa)</th>
<th>HJP</th>
<th>Temp (°C)</th>
<th>time (hr)</th>
<th>Resulting UTS (MPa)</th>
<th>Difference UTS (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1040</td>
<td>9150</td>
<td>175</td>
<td>2.7</td>
<td></td>
<td>1052</td>
<td>12</td>
</tr>
<tr>
<td>1020</td>
<td>9656</td>
<td>200</td>
<td>2.6</td>
<td></td>
<td>1029</td>
<td>9</td>
</tr>
<tr>
<td>981</td>
<td>10628</td>
<td>250</td>
<td>2.1</td>
<td></td>
<td>984</td>
<td>3</td>
</tr>
<tr>
<td>954</td>
<td>11313</td>
<td>275</td>
<td>4.4</td>
<td></td>
<td>954</td>
<td>0</td>
</tr>
</tbody>
</table>

Table 5.7 – Material Designation, and Tensile Properties (UTS, YS, TE, UE and YS/UTS) for Four Tempered Conditions of Steel C

<table>
<thead>
<tr>
<th>Condition</th>
<th>YS</th>
<th>UTS</th>
<th>UE</th>
<th>TE</th>
<th>YS/UTS</th>
</tr>
</thead>
<tbody>
<tr>
<td>1050AT</td>
<td>651</td>
<td>1052</td>
<td>7.7</td>
<td>11.7</td>
<td>0.62</td>
</tr>
<tr>
<td>1020AT</td>
<td>725</td>
<td>1029</td>
<td>6.9</td>
<td>11.0</td>
<td>0.70</td>
</tr>
<tr>
<td>990AT</td>
<td>780</td>
<td>984</td>
<td>6.4</td>
<td>11.1</td>
<td>0.79</td>
</tr>
<tr>
<td>960AT</td>
<td>809</td>
<td>954</td>
<td>5.6</td>
<td>10.1</td>
<td>0.85</td>
</tr>
</tbody>
</table>

The numerical portion of the material designation (Condition) in Table 5.7 differs slightly from the UTS, but the original strength target values were preserved as identifiers in order to maintain consistency and for simplicity. Figure 5.8a shows representative engineering stress-engineering strain tensile curves for the as-received condition and each of the four AT conditions reported in Table 5.7. Each tensile curve in Figure 5.8a achieved a different UTS, and all four tempered conditions exhibit YPE. A decrease in TE is observed with increasing tempering temperature, and similar trends have been observed by others when tempering DP steels below 400 °C [54, 59]. Figure 5.8b shows the UTS-HJP data for the four AT conditions reported in Table 5.6 (open circles) overlaid with the UTS-HJP data from the preliminary tempering study from Figure 5.6b (filled squares), and an excellent agreement is
observed. When fitting the data with a linear regression, the correlation coefficient improved from 0.98 (Figure 5.6b) to 0.99, suggesting that the prediction of the UTS using HJP was achieved for steel C.

![Stress-Strain Curves for Steel C](image1)

**Figure 5.8** Representative engineering stress-engineering strain tensile curves for steel C in the as-received condition, and the four tempered conditions in (a). All four tempered conditions exhibited some extent of YPE. Plot of UTS vs. HJP in (b) with the four tempered conditions of steel C reported in Table 5.7 (open circles) overlaid on the data from Figure 5.6b, improving the correlation coefficient from 0.98 to 0.99.

### 5.2.2 Cold-Rolling of Tempered Steel C

To evaluate different hardness conditions with equivalent UTS, four additional conditions of steel C were generated by cold-rolling the four tempered conditions (Table 5.7) back to the as-received UTS value of 1080 MPa. Achieving different constituent hardness values by tempering is effective, but parameters such as UTS also decrease upon tempering, and have been reported to correspond to an increase in HER [46]. Incorporating a group of steels all with 1080 MPa UTS, but different constituent hardness values can provide additional insight on the effect of constituent hardness on formability.

The percent cold-roll (%CR) values required to strengthen each of the four tempered steels to 1080 MPa UTS were obtained by an iterative process of cold-rolling, tensile testing, and potential changing of the %CR values. For the first iteration, %CR values were calculated using Equation 5.3, and are reported in Table 5.8. The starting UTS (Tempered) and the UTS after cold-rolling (Resulting) are also reported in Table 5.8. The Tempered UTS values reported in Table 5.8 are those of the four tempered conditions (1050AT, 1020AT, 990AT, and 960AT). The resulting UTS values reported in Table 5.8 are reasonably close to 1080 MPa, but improvements to the target %CR values were made, and Table 5.9
presents the second iteration of %CR values used for the four tempered steels, along with the final resulting UTS values.

Table 5.8 – First Iteration of Percent Cold-Roll Values Used to Achieve the Resulting UTS from the Tempered UTS.

<table>
<thead>
<tr>
<th>Iteration 1 Tempered UTS (MPa)</th>
<th>Iteration 1 %CR</th>
<th>Resulting UTS (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1052</td>
<td>2.2</td>
<td>1098</td>
</tr>
<tr>
<td>1029</td>
<td>4.3</td>
<td>1085</td>
</tr>
<tr>
<td>984</td>
<td>11.8</td>
<td>1069</td>
</tr>
<tr>
<td>954</td>
<td>19</td>
<td>1080</td>
</tr>
</tbody>
</table>

The Tempered UTS values reported in Table 5.9 are those of the four tempered conditions (Table 5.7). Also reported in Table 5.9 are the designations given to the four tempered, cold-rolled (TCR) conditions. The 1050TCR condition refers to the 1050AT condition that had been cold-rolled to achieve a 1080 MPa UTS, etc.

Table 5.9 – Material Designation, Second Iteration of %CR Values, and Final Resulting UTS Values For the Four Tempered, Cold-Rolled Conditions of Steel C

<table>
<thead>
<tr>
<th>Condition</th>
<th>Iteration 2 Tempered UTS (MPa)</th>
<th>Iteration 2 Final %CR</th>
<th>Final Resulting UTS (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1050TCR</td>
<td>1052</td>
<td>1.2</td>
<td>1083</td>
</tr>
<tr>
<td>1020TCR</td>
<td>1029</td>
<td>5.5</td>
<td>1090</td>
</tr>
<tr>
<td>990TCR</td>
<td>984</td>
<td>12.6</td>
<td>1083</td>
</tr>
<tr>
<td>960TCR</td>
<td>954</td>
<td>20.4</td>
<td>1080</td>
</tr>
</tbody>
</table>

The final resulting UTS values reported in Table 5.9 represent the average of three tensile tests, and all four conditions exhibit an average UTS around 1080 MPa. For the case of 1020TCR, the resulting UTS deviated further away from 1080 MPa compared to the %CR value reported in Table 5.8. The reason for an increased final %CR value (Table 5.9) is due to the control of the roll gap during cold-rolling. Table 5.10 reports the tensile properties for the four TCR conditions. Table A.4 shows the data in Table 5.10 with standard deviations for the UTS, YS, TE and UE.

Table 5.10 – Tensile Properties (UTS, YS, TE, UE, and YS/UTS) for Four TCR Conditions of Steel C

<table>
<thead>
<tr>
<th>Condition</th>
<th>YS</th>
<th>UTS</th>
<th>UE</th>
<th>TE</th>
<th>YS/UTS</th>
</tr>
</thead>
<tbody>
<tr>
<td>1050TCR</td>
<td>884</td>
<td>1083</td>
<td>5.6</td>
<td>9.8</td>
<td>0.82</td>
</tr>
<tr>
<td>1020TCR</td>
<td>979</td>
<td>1090</td>
<td>2.3</td>
<td>6.1</td>
<td>0.90</td>
</tr>
<tr>
<td>990TCR</td>
<td>1003</td>
<td>1083</td>
<td>1.3</td>
<td>3.9</td>
<td>0.93</td>
</tr>
<tr>
<td>960TCR</td>
<td>1007</td>
<td>1080</td>
<td>1.2</td>
<td>3.6</td>
<td>0.93</td>
</tr>
</tbody>
</table>
Figure 5.9a shows representative engineering stress-engineering strain tensile curves for the as-received condition and each of the four TCR conditions reported in Table 5.10, and all exhibit continuous yielding. An increase in %CR corresponded to a decrease in TE for the TCR conditions. Figure 5.9b shows a photograph of four tempered, cold-rolled tensile specimens, each representing one of the TCR conditions. Figure 5.9b shows that cold-rolling was performed on the as-machined tensile specimens, and that the different tempered conditions (Table 5.7) required different %CR values to re-attain 1080 MPa UTS.

Diffusion-controlled grain boundary mobility was inactive for the tempering treatments performed on steel C. However, cold-rolling is a plastic deformation process that can potentially alter the effective grain size. To account for the possibility of a change in grain size contributing to subsequent formability, a grain size/MVF analysis was performed on the 960TCR condition. The 960TCR condition experienced the largest %CR (20.4 %), and if an appreciable change in effective grain size existed in any of the TCR conditions, it was interpreted to be the condition that experienced the greatest %CR.

Table 5.11 reports the average ferrite grain size, average martensite grain size, and MVF for the as-received condition (from Table 5.1), and the 960TCR condition. Also reported in Table 5.11 are the absolute changes in grain size and MVF values from the as-received to the 960TCR condition.
Table 5.11 – Grain Size and MVF values for As-Received and 960TCR Conditions of Steel C

<table>
<thead>
<tr>
<th>Condition</th>
<th>G.S. α (μm)</th>
<th>G.S. α' (μm)</th>
<th>MVF (pct)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-Received</td>
<td>2.94</td>
<td>3.14</td>
<td>54.5</td>
</tr>
<tr>
<td>960TCR</td>
<td>3.25</td>
<td>3.90</td>
<td>56.6</td>
</tr>
<tr>
<td>Δ</td>
<td>+0.31</td>
<td>+0.76</td>
<td>+2.1</td>
</tr>
</tbody>
</table>

The average ferrite grain size, average martensite grain size, and MVF increased for the 960TCR condition. The apparent increase in MVF was unexpected since grain boundary mobility was suppressed. However, the apparent increase in MVF is marginal, and is believed to be within the uncertainty in MVF measurements. The percent increase in martensite grain size is greater than the percent increase in average ferrite grain size, and can partially be explained by the degraded etching response of the microstructure caused by tempering and cold-rolling. Figure 5.10 shows an SEM micrograph of the in-plane orientation of the 960TCR condition. The micrograph in Figure 5.10 was polished to 1 μm using standard metallographic techniques, then etched with 2 pct nital for approximately 8 s.

![SEM micrograph of the in-plane orientation of 960TCR condition. Steel was etched with 2 pct nital for approximately 8 s. The distinction between ferrite and martensite is decreased when compared to the as-received condition of steel C shown in Figure 5.1c.](image)

Figure 5.10 shows less distinction between ferrite and martensite after tempering and cold-rolling, with ferrite grains containing precipitated carbides, and the internal structure of martensite obscured. During the grain size analysis, martensite/martensite grain boundaries were counted (Equation 4.3), with more boundary counts resulting in a smaller grain size. The obscured boundaries between adjacent martensite...
grains in Figure 5.10 caused potential boundary counts to be overlooked. When compared to a representative micrograph on which the as-received grain size/MVF analysis was performed (Figure 5.1c), Figure 5.10 exhibits less distinction between ferrite and martensite, and the reduced distinction could also have contributed to the slightly higher MVF value reported in Table 5.11. The apparent increase in grain size for the 960TCR condition was interpreted to be within experimental uncertainty reported in Table A.1 for steel C, and was assumed to be equivalent to the as-received condition. Apparent differences in grain size for the 990TCR, 1020TCR and 1050TCR conditions were interpreted to be less than that observed for the 960TCR condition, and were also assumed to be equivalent to the as-received condition in the present analysis.

5.2.3 Hole Expansion Testing

The nine conditions of steel C (as-received, four AT, four TCR) were hole expansion tested to evaluate local formability performance. Hole expansion coupons were tempered using the same parameters for the four conditions (1050AT, 1020AT, 990AT, and 960AT) outlined in Table 5.6, and cold-rolled using the same parameters for the four conditions (1050TCR, 1020TCR, 990TCR, and 960TCR) outlined in Table 5.9. The plastic deformation across the width of the hole expansion coupons during cold-rolling was non-uniform, with the edges experiencing a larger amount of %CR than the center. To account for the non-uniform deformation across the width, measurements of %CR were made on the centerline of each coupon since the centerline is the location where the sheared hole was created. Sheared holes were created in the hole expansion coupons after tempering and cold-rolling treatments, and Figure 5.11 shows a photograph of eight hole expansion coupons prior to hole expansion testing for the eight modified conditions of steel C. The four AT conditions are shown on the bottom row, and all four have approximate dimensions of 100 x 100 mm. The four TCR conditions are shown on the top row, and the coupon dimensions in the rolling direction (vertical in Figure 5.11) were greater than 100 mm due to the cold-rolling process. The centerline is indicated by a vertical dashed line on the coupon surfaces in Figure 5.11, and the width direction is horizontal. All hole expansion coupon dimensions were greater than the minimum recommended by ISO 16630 (100 x 100 mm).

For the nine conditions of steel C, Table 5.12 reports the HER values for the three different measurement methods outlined in Sec. 4.10. Each reported HER value represents the average of 3 – 5 hole expansion tests, and standard deviations for the data in Table 5.12 are reported in Table A.5. During hole expansion testing, multiple cracks on the sheared hole surface were often observed before an individual crack satisfied the critical 0.1 mm through-thickness criteria to stop the test. In every case, the crack satisfying the 0.1 mm through-thickness criteria propagated along the rolling direction.
Table 5.12 –HER Measurements From Each of the Three Measurement Methods for the As-Received and Eight Modified Conditions of Steel C

<table>
<thead>
<tr>
<th>Condition</th>
<th>Computer HER</th>
<th>Caliper HER</th>
<th>ImageJ HER</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-R</td>
<td>29.3</td>
<td>26.6</td>
<td>25.7</td>
</tr>
<tr>
<td>1050AT</td>
<td>30.2</td>
<td>27.1</td>
<td>26.1</td>
</tr>
<tr>
<td>1020AT</td>
<td>31.8</td>
<td>28.1</td>
<td>26.8</td>
</tr>
<tr>
<td>990AT</td>
<td>38.4</td>
<td>35.5</td>
<td>33.9</td>
</tr>
<tr>
<td>960AT</td>
<td>43.2</td>
<td>40.6</td>
<td>38.9</td>
</tr>
<tr>
<td>1050TCR</td>
<td>31.8</td>
<td>28.0</td>
<td>26.8</td>
</tr>
<tr>
<td>1020TCR</td>
<td>37.2</td>
<td>33.5</td>
<td>32.0</td>
</tr>
<tr>
<td>990TCR</td>
<td>39.4</td>
<td>37.0</td>
<td>35.1</td>
</tr>
<tr>
<td>960TCR</td>
<td>39.3</td>
<td>36.5</td>
<td>34.1</td>
</tr>
</tbody>
</table>

Figure 5.12 shows a plot of the HER values obtained by the three measurement methods reported in Table 5.12 as a function of HJP for the four tempered (AT) conditions of steel C; an increase in HER with increasing HJP is observed. When the data from each measurement method are individually considered, all three measurement methods exhibit the same trend, and the trend is consistent with literature [46]. The Computer method consistently gave the highest HER, and the Caliper method consistently gave a median value between the Computer and ImageJ measurement methods. For each steel condition, the Caliper method produced HER values approximately 3 pct lower than the Computer method. For the same hole expansion test, the three measurement techniques each produced a different
HER, indicating that a single measurement technique should be used to avoid potential inconsistencies. It was determined that the Computer method was likely the most accurate measurement technique because operator bias was almost entirely removed from the analysis. Thus, the HER data in Table 5.12 generated with the Computer measurement method were utilized in subsequent correlations discussed below.

Figure 5.12  Plot of HER vs. HJP for the four tempered (AT) conditions of steel C, showing an increase in HER with increasing HJP. The Computer measurement method consistently gave the highest HER, and the ImageJ method consistently gave the lowest HER. All three measurement methods produced a similar trend.

5.2.4 Nanoindentation – Constituent Hardness for Eight Modified Conditions

Nanoindentation analyses for the nine conditions of steel C were performed on the in-plane and longitudinal orientations. Nanoindentation specimens were prepared and tested according to Sec. 4.6, and the results for the average ferrite hardness, average martensite hardness, martensite/ferrite hardness ratio, and bulk average hardness for the eight modified conditions are reported in Table 5.13 for the in-plane orientation. Nanoindentation data for the as-received condition reported in Table 5.2 are also reported in Table 5.13 for convenience. The range of hardness values reported for ferrite and martensite are consistent with results reported in literature [33, 70, 71, 74, 75, 98, 104, 143]. The hardness data in Table 5.13 quantitatively illustrate the effects of the different processing techniques on the constituent hardness values. With increasing HJP (lower UTS) for the four AT conditions, the martensite hardness decreased more appreciably (6.20 to 5.18 GPa) than the change in ferrite hardness (2.20 to 1.93 GPa), indicating that the tempering treatment primarily softened the martensite. The greater decrease in martensite hardness resulted in a decrease in the calculated martensite/ferrite hardness ratio. After cold-rolling, the ferrite hardness increased more appreciably relative to the change in martensite hardness, indicating that the cold-rolling treatment primarily strengthened ferrite. For example, cold-rolling
increased the ferrite hardness for the 960AT steel from 1.93 to 2.25 GPa without a significant change in martensite hardness. The greater increase in ferrite hardness also resulted in a decrease in the calculated martensite/ferrite hardness ratio. The hardness ratios produced from steel C ranged from 3.18 to 2.26, a range that was considered sufficient to evaluate influences on formability performance. Ferrite and martensite hardness histograms created for each of the conditions reported in Table 5.13 exhibited a similar behavior to that observed in Figure 5.3. Table A.6 reports ferrite and martensite hardness values from Table 5.13 with standard deviations.

Table 5.13 – Nanoindentation Data from In-Plane Orientation for the Nine Conditions of Steel C. Ferrite (α), Martensite (α’), Martensite/ferrite Hardness Ratio, and Bulk Average Hardness

<table>
<thead>
<tr>
<th>Condition</th>
<th>α (GPa)</th>
<th>α’ (GPa)</th>
<th>α'/α</th>
<th>Bulk Avg (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-R</td>
<td>2.15</td>
<td>6.84</td>
<td>3.18</td>
<td>5.02</td>
</tr>
<tr>
<td>1050AT</td>
<td>2.20</td>
<td>6.20</td>
<td>2.82</td>
<td>4.11</td>
</tr>
<tr>
<td>1020AT</td>
<td>2.25</td>
<td>6.05</td>
<td>2.69</td>
<td>4.44</td>
</tr>
<tr>
<td>990AT</td>
<td>2.05</td>
<td>5.27</td>
<td>2.57</td>
<td>3.67</td>
</tr>
<tr>
<td>960AT</td>
<td>1.93</td>
<td>5.18</td>
<td>2.68</td>
<td>3.59</td>
</tr>
<tr>
<td>1050TCR</td>
<td>2.45</td>
<td>6.21</td>
<td>2.54</td>
<td>4.00</td>
</tr>
<tr>
<td>1020TCR</td>
<td>2.18</td>
<td>5.53</td>
<td>2.54</td>
<td>4.01</td>
</tr>
<tr>
<td>990TCR</td>
<td>2.28</td>
<td>5.34</td>
<td>2.34</td>
<td>3.88</td>
</tr>
<tr>
<td>960TCR</td>
<td>2.25</td>
<td>5.08</td>
<td>2.26</td>
<td>3.71</td>
</tr>
</tbody>
</table>

Nanoindentation data were also obtained for the region adjacent to the sheared hole in untested hole expansion coupons at mid-thickness to characterize the change in constituent hardness properties that develop in response to the shearing process. Figure 5.13 shows an SEM micrograph of the longitudinal orientation of the region adjacent to the sheared hole for the 990TCR condition with the 15 X 15 indent array overlaid. The 990TCR condition of steel C in Figure 5.13 was polished to 0.05 μm using standard metallographic techniques, then etched with 2 pct nital for approximately 6 s. Figures C.1 – C.8 show SEM micrographs of the longitudinal orientation of the region adjacent to the sheared hole with the indent array overlaid for the other eight conditions of steel C. Compared to the as-received condition of steel C observed in Figures 4.5 and 5.1c, steel C in Figure 5.13 exhibits a microstructure with an appreciable amount of plastic deformation.

For the nine conditions of steel C, Table 5.14 reports the difference in average ferrite hardness, average martensite hardness, martensite/ferrite hardness ratio, and bulk average hardness between the region adjacent to the sheared hole and the initial state (Table 5.13). For example, the average ferrite hardness in the region adjacent to the sheared hole for the 990AT condition increased 1.1 GPa compared to the average ferrite hardness reported for the 990AT condition in Table 5.13. Table 5.14 reports an increase in the average ferrite hardness, average martensite hardness, and the bulk average hardness for
the region adjacent to the sheared hole compared to the unaffected material for every condition. In most cases, the increase in average ferrite hardness was greater than the increase in average martensite hardness, suggesting that ferrite work-hardened more readily in response to plastic deformation. The shearing process locally decreased the martensite/ferrite hardness ratio in every case, indicating that plastic deformation increased the strength similarity between ferrite and martensite in DP steels.

Figure 5.13  SEM micrograph from the longitudinal orientation of the region adjacent to the sheared hole for the 990TCR condition, with the nanoindentation array overlaid. The sheared surface is on the right-side of the micrograph. Location of SEM micrograph is at mid-thickness. Steel was etched with 2 pct nital for approximately 6 s.

Table 5.14 – Change in Ferrite (α), Martensite (α’), Martensite/ferrite Hardness Ratio, and Bulk Average Hardness for the Region Adjacent to Sheared Hole Compared to the Initial State for the Nine Conditions of Steel C.

<table>
<thead>
<tr>
<th>Condition</th>
<th>α (GPa)</th>
<th>α’ (GPa)</th>
<th>α’/α</th>
<th>Bulk Avg (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Δ As-R</td>
<td>1.2</td>
<td>0.3</td>
<td>-1.0</td>
<td>0.3</td>
</tr>
<tr>
<td>Δ 1050AT</td>
<td>1.1</td>
<td>0.6</td>
<td>-0.8</td>
<td>1.2</td>
</tr>
<tr>
<td>Δ 1020AT</td>
<td>0.8</td>
<td>0.3</td>
<td>-0.6</td>
<td>0.5</td>
</tr>
<tr>
<td>Δ 990AT</td>
<td>1.1</td>
<td>0.9</td>
<td>-0.6</td>
<td>1.3</td>
</tr>
<tr>
<td>Δ 960AT</td>
<td>1.1</td>
<td>0.7</td>
<td>-0.8</td>
<td>1.2</td>
</tr>
<tr>
<td>Δ 1050TCR</td>
<td>1.2</td>
<td>0.8</td>
<td>-0.6</td>
<td>1.8</td>
</tr>
<tr>
<td>Δ 1020TCR</td>
<td>1.2</td>
<td>1.4</td>
<td>-0.5</td>
<td>1.3</td>
</tr>
<tr>
<td>Δ 990TCR</td>
<td>0.7</td>
<td>0.9</td>
<td>-0.2</td>
<td>1.2</td>
</tr>
<tr>
<td>Δ 960TCR</td>
<td>0.9</td>
<td>0.7</td>
<td>-0.4</td>
<td>0.8</td>
</tr>
</tbody>
</table>
Table 5.14 also shows that, regardless of the initial constituent hardness properties, shearing induced a localized hardness gradient adjacent to the sheared hole with hardness greater than the constituent hardness properties reported in the unaffected material (Table 5.13), and the increase in hardness adjacent to the sheared hole is consistent with results reported by Gibbs [136]. A smaller reduction in martensite/ferrite hardness ratio evident for the TCR conditions was interpreted to reflect the effects of the prior cold-rolling. Ferrite and martensite hardness, along with standard deviations for the nine sheared hole conditions are reported in Figure A.6.

5.2.5 Sheared Hole Characterization for Select Steel Conditions

Figure 5.13 shows an SEM micrograph adjacent to the sheared hole edge for the 990TCR condition, and an appreciable amount of plastic deformation within the microstructure is observed. The microstructural response to the creation of the sheared hole was an area of interest, as sheared holes are known to exhibit lower HER values when compared to holes created by methods such as drilling, or EDM [11, 76, 78]. The local shear strain imposed on the microstructure for the region adjacent to the sheared hole edge was evaluated by measuring grain rotations relative to the original rolling direction as a function of distance from the sheared hole edge for the AsR, 960AT, and 960TCR conditions to determine whether a difference in material flow could be observed. The AsR, 960AT, and 960TCR conditions were chosen because the samples exhibit different tensile behavior (Figures 5.8a and 5.9a), have the largest difference in UTS (AsR vs. 960AT in Table 5.7), and the largest difference in martensite/ferrite hardness ratio (AsR vs. 960TCR in Table 5.13). A more comprehensive discussion on the methodology (shown in Figures D.1 and D.2) used to obtain grain rotation measurements is provided in Appendix D. The grain rotation as a function of distance from the sheared hole edge (shown in Figure D.3), as well as the maximum values of grain rotation obtained for the three conditions (summarized in Table D.1) were consistent with the work of others [136, 144], and the SAZ depth and grain rotation values across the SAZ for the 960AT and 960TCR conditions were similar to each other (Table D.1). The SAZ depth was defined as the first non-zero angle measurement of grain rotation in Figure D.3. Compared to the AsR condition, the 960TCR condition exhibited a smaller SAZ (150 μm vs. 200 μm), less work-hardening, and a higher HER (Table 5.12). When comparing a DP590 and TRIP590 steel, Lee [144] reported that, for steels with the same UTS, the steel with lower work-hardening (DP590) exhibited a smaller SAZ and a higher HER, and the conclusion is consistent with the interpretation of the 960TCR and AsR conditions in the current study.

The microstructural damage, quantified using void area pct, was evaluated for the region adjacent to the sheared holes for the AsR, 960AT, and 960TCR conditions to determine whether the different martensite/ferrite hardness ratios affected void damage. A more comprehensive discussion detailing the
methods used to obtain the void area pct are provided in Appendix D. The depth of the SAZ behind the sheared edge was approximately 150 – 200 μm for the three conditions (Figure D.3), and the void area pct analysis evaluated the region adjacent to the sheared hole to a depth of 90 μm from the sheared hole edge. Figure D.7 identifies the area evaluated, and Figure D.8 shows an example SEM micrograph used for void analysis. The void area pct in the SAZ for the three conditions exhibited similar values (reported in Table D.2), and it was determined that, independent of the selected steel, the hole shearing process induced an insignificant amount of observable void damage (less than 1 pct in every case) to the SAZ. During the hole shearing process, a compressive stress is believed to be present [144], which acts to suppress void nucleation.

5.2.6 Data Correlations Between Nanoindentation Data, Tensile Properties and Hole Expansion Ratio

The martensite/ferrite hardness ratio reported in Table 5.13 was used to establish correlations with HER and tensile properties. The martensite/ferrite hardness ratio was chosen because it incorporates the hardness from both ferrite and martensite, and was interpreted to accurately represent the unique hardness condition of each steel.

Figure 5.14 correlates HER with martensite/ferrite hardness ratio, and shows that for both AT and TCR conditions, HER increases with a decrease in hardness ratio, and that both sets of conditions are best interpreted with a single function that also includes the as-received (AsR) condition. The trend observed in Figure 5.14 is also consistent with previously reported data [76]. Figure 5.14 provides quantitative validation to the claim that an increased similarity in hardness (i.e. lower hardness ratio) corresponds to improved performance in complex forming operations [11, 17, 22, 23, 29, 30, 39, 139, 145]. A lower hardness ratio increases the strain-sharing between ferrite and martensite during deformation, allowing the microstructure to delay strain localization in ferrite to higher formability limits. Figure 5.14 shows that HER increased when the ferrite strength and martensite strength converge, even for steels of equivalent UTS (solid shapes). A potential outlier in Figure 5.14 is the 960AT condition exhibiting the highest HER (43.2 %). Upon tempering, the ferrite softened an appreciable amount, which acted to increase the martensite/ferrite hardness ratio (Table 5.13) For subsequent plots in Sec. 5.2.5, the solid circle represents the AsR condition, the solid diamonds represent the tempered, cold-rolled (TCR) steel conditions, and the open squares represent the as-tempered (AT) steel conditions.

Figure 5.15 correlates YS with hardness ratio, and shows an increase in YS with decreasing hardness ratio. The AsR, AT, and TCR conditions can be characterized using a single function in Figure 5.15, and indicates that the correlation is independent of UTS. A lower hardness ratio improves martensite/ferrite interface compatibility, and is interpreted to increase strain-sharing between ferrite and martensite, thereby suppressing plastic strain to higher stresses.
Figure 5.14  Plot of HER as a function of martensite/ferrite hardness ratio for all nine conditions, showing an increase in HER with decreasing hardness ratio.

R² = 0.43

Figure 5.15  Plot of YS as function of hardness ratio showing an increase in YS with decreasing hardness ratio.

R² = 0.83

Figure 5.16 correlates the UE with hardness ratio, and shows an increasing UE with increasing hardness ratio. Figure 5.16 also shows that the AsR, AT, and TCR conditions can be characterized using a single function. Both UE and HER are measures of ductility, but the correlation between UE and hardness ratio in Figure 5.16 shows a trend opposite to that shown in Figure 5.14 relating HER to hardness ratio. Based on Figures 5.16 and 5.14, there is an apparent tradeoff between UE and HER in the current study.
Figure 5.17 correlates the YS/UTS with hardness ratio, and shows greater work-hardening (lower YS/UTS) with increasing hardness ratio. Figure 5.17 shows that the AsR, AT, and TCR conditions can be characterized using a single function. It is interpreted that a higher hardness ratio (greater strength disparity between martensite and ferrite) decreases strain-sharing between ferrite and martensite, causing heterogeneous plastic strain (i.e. yielding) to occur at lower stresses. In particular, when considering steels of equivalent UTS (filled shapes in Figure 5.17), a lower YS will necessitate a greater amount of work-hardening. With a lower hardness ratio, the improved strain-sharing suppresses yielding to higher stresses, and less strain-hardening is experienced [117, 127].

Figure 5.16  Plot of UE as a function of hardness ratio, showing an increase in UE with increasing hardness ratio.

Figure 5.17  Plot of YS/UTS as a function of hardness ratio, showing that an increase in strain-hardening is associated with an increase in hardness ratio.
From Figures 5.16 and 5.17, an increase in UE appears to correspond to an increase in work-hardening (lower YS/UTS), and Figure E.1 presents the correlation between UE with YS/UTS. For the nine conditions of steel C, less work-hardening (higher YS/UTS value) results in less plastic strain (UE) required to reach the UTS.

Figure 5.18 correlates HER with tensile properties to evaluate the associated response of two different mechanical tests as a function of constituent hardness. Figure 5.18a shows HER increasing with increasing YS/UTS, indicating that less work-hardening is associated with a higher HER. The AT and TCR data sets presented in Figure 5.18a exhibit similar trends, but at different magnitudes. Ferrite hardness is the primary difference between the AT and TCR conditions, and is interpreted to affect the difference in magnitude. The observed trends are consistent with the interpretation that a lower hardness ratio facilitates a higher HER (Figure 5.14), and that a lower hardness ratio corresponds to a decrease in work-hardening (Figure 5.17) [128]. Figure 5.18b shows that an increase in HER corresponds with a decrease in UE. Though HER and UE are both considered ductility/formability indexes, Figure 5.18b reports an inverse association between the two parameters. Figure 5.18b is consistent with the interpretation that an increase in UE corresponds with an increasing hardness ratio (Figure 5.16) and that an increasing hardness ratio corresponds to a decrease in HER (Figure 5.14). The AT and TCR data sets shown in Figure 5.18b follow the same trend, but at different magnitudes. A potential reason for the difference in magnitudes between the AT and TCR conditions in Figure 5.18b is that the AT conditions exhibited YPE, which could account for a portion of the increased UE. Additionally, the AT conditions have lower UTS, and the overall softer microstructure could have also contributed to the higher UE.

Figure 5.18 Plot showing HER increasing as a function of YS/UTS in (a), indicating lower strain hardening is associated with increased HER. Plot showing HER decreasing with increasing UE in (b), indicating that the microstructural mechanisms that promote a higher HER decrease the UE.
Tensile values and HER are macroscopic responses emanating from the underlying properties of the microstructure, and it would be erroneous to state that an increase in HER leads to a decrease in UE. Instead, a more accurate statement would be that a decrease in hardness ratio correlates with an increase HER and a decrease in UE.

5.2.7 HER Correlations with Average Martensite Hardness

A correlation between HER and the average martensite hardness is presented for the purpose of evaluating a hypothesis made based on the results obtained from a study involving eight commercially-produced DP980 steels [76]. A correlation was established between HER and average martensite hardness for eight commercially-produced DP980 steels, and it was hypothesized in Sec. 3.1 that the correlation was likely to improve if other microstructural properties, such as chemical content, grain size, grain morphology, and MVF were held constant. Figure 5.19a presents a correlation between HER and martensite hardness from the previous study involving eight commercially-produced DP980 steels [76], and Figure 5.19b shows the HER as a function of average martensite hardness for the nine conditions of steel C in the present study.

Figure 5.19 HER as a function of martensite hardness from a previous study involving eight different commercially-produced DP980 steels in (a) [76], and HER as a function of martensite hardness for the nine conditions of steel C reported in (b).
Both plots in Figure 5.19 exhibit an increase in HER with decreasing martensite hardness, but the correlation coefficient for the nine conditions of steel C in the present study was higher (0.91) than the correlation coefficient for the study involving eight commercially-produced DP steels (0.54). The improved correlation coefficient was interpreted to be due to the fact that the other aforementioned microstructural properties remained largely unchanged, and validated the hypothesis stated in Sec. 3.1. The HER and martensite hardness plotted in Figure 5.19b produced a stronger correlation (0.91) than the plot in Figure 5.14 between HER and the martensite/ferrite hardness ratio (0.43), and was interpreted to be an effect of the high MVF (55 pct) present in steel C.

5.2.8 HER Characterization Based on Tensile Properties

Tensile and hole expansion properties generated for the nine conditions of steel C were evaluated using different studies in literature that focused on correlating HER values to tensile properties. Recently, multiple studies aimed at predicting HER based on tensile properties have been performed [129, 140, 126] since mechanical tests such as tensile tests are more straight-forward and require equipment more readily available when compared to hole expansion. The studies [126, 129, 140] omit the effects of microstructural properties on HER and tensile properties, and instead focus solely on correlations between macroscopic properties.

Levy and Van Tyne [140] performed a study that correlated the circumferential HER (the engineering HER expressed as true strain) to the true strain hardening rate at uniform elongation observed in a tensile test. The circumferential HER was calculated using Equation 5.4, where \( HER \) is the HER value expressed in engineering strain (Equation 4.6). The HER values from Table 5.12 (engineering strain) were converted to circumferential HER (true strain) for the nine conditions of steel C, and are reported in Table 5.15.

\[
\text{Circumferential HER (pct)} = \ln \left( \frac{HER}{100} + 1 \right)
\]  

The true strain hardening rate at uniform elongation (TS) was interpreted to be a measure of the cohesive strength of the grain interfaces for steels [140], and was calculated using Equation 5.5 [140], where \( %\text{UE} \) is the UE in engineering strain (from Tables 5.7 and 5.10), and UTS is engineering UTS (from Tables 5.7 and 5.10). The TS value in Equation 5.5 is the true stress at instability, and is reported in Table 5.15 for the nine conditions of steel C.

\[
TS(MPa) = [1 + (%UE/100)] \times UTS
\]
Table 5.15 – Calculated Strain Hardening Rate at UE (TS) and Circumferential HER Values for Nine Conditions of Steel C

<table>
<thead>
<tr>
<th>Condition</th>
<th>TS (MPa)</th>
<th>Circum. HER</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-R</td>
<td>1165</td>
<td>0.26</td>
</tr>
<tr>
<td>1050AT</td>
<td>1133</td>
<td>0.26</td>
</tr>
<tr>
<td>1020AT</td>
<td>1100</td>
<td>0.28</td>
</tr>
<tr>
<td>990AT</td>
<td>1051</td>
<td>0.32</td>
</tr>
<tr>
<td>960AT</td>
<td>1007</td>
<td>0.36</td>
</tr>
<tr>
<td>1050TCR</td>
<td>1144</td>
<td>0.28</td>
</tr>
<tr>
<td>1020TCR</td>
<td>1115</td>
<td>0.32</td>
</tr>
<tr>
<td>990TCR</td>
<td>1097</td>
<td>0.33</td>
</tr>
<tr>
<td>960TCR</td>
<td>1093</td>
<td>0.33</td>
</tr>
</tbody>
</table>

Figure 5.20 shows the data from the study by Levy and Van Tyne [140] as filled circles, and shows that an increase in true strain hardening rate at uniform elongation corresponds to a lower circumferential HER. In Figure 5.20, limited data for TS values greater than 950 MPa are presented, and exhibit more scatter relative to the data for TS values below 950 MPa. The data for the nine conditions of steel C in Table 5.15 are presented as open squares in Figure 5.20, and exhibit a trend similar to the trend produced by the literature data [140] (i.e. decrease in circumferential HER with increase in TS).

The TS values for the nine conditions of steel C are all greater than 1000 MPa, and the circumferential HER values appear to be higher for the given TS values when compared to the literature data [140].
Higher circumferential HER for higher TS values were attributed to lower carbon martensite (i.e. lower hardness), increased ferrite strength, and in some cases, TRIP steels [140]. Processing treatments of tempering and cold-rolling used for steel C decreased martensite strength and increased ferrite strength, respectively (Table 5.13), and is consistent with the above explanation for a higher observed circumferential HER value for higher TS values.

An empirical equation which correlates HER values with selected tensile properties was developed by Kumar et al. using a regression analysis on a large dataset of steels with engineering UTS ranging from 265 to 956 MPa, and engineering TE ranging from 21 to 66 pct [128]. Multiple tensile parameters, including YS, UTS, r-value, TE, post-uniform elongation, strain hardening exponent, and UTS/YS ratio were evaluated, and the optimal fit to experimental data is presented as Equation 5.6, with \( \sigma_{UTS} \) the UTS (in MPa), \( r_m \) the average r-value, and \( \varepsilon_t \) total elongation (in pct) [128]. In Equation 5.6, the \( \varepsilon_t \) is assumed to be obtained using ASTM E8 standard size tensile specimens. The coefficients on the various terms in Equation 5.6 were determined solely by a best fit regression analysis, independent of any metallurgical interpretation.

\[
HER\ (pct) = -48 + 302e^{-0.035\sigma_{UTS}} + 144(1 - e^{-0.6r_m}) + 0.1\varepsilon_t
\]

(5.6)

Using the tensile properties for the nine conditions of steel C in Tables 5.7 and 5.10, HER values were calculated using Equation 5.6 and are reported in Table 5.16. From previous studies on DP steels, the \( r_m \) value was estimated to be 0.9 for all nine conditions [128, 136], as \( r_m \) values were not available in the current study. The \( r_m \) value likely changed upon cold-rolling, but with UE values below 10 pct for all cold-rolled tensile specimens (Table 5.10), it was unclear whether accurate r-values could be obtained from tensile data.

<table>
<thead>
<tr>
<th>Condition</th>
<th>HER (Table 5.12)</th>
<th>HER (Eq. 5.6)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-R</td>
<td>29.3</td>
<td>20.1</td>
</tr>
<tr>
<td>1050AT</td>
<td>30.2</td>
<td>20.9</td>
</tr>
<tr>
<td>1020AT</td>
<td>31.8</td>
<td>21.4</td>
</tr>
<tr>
<td>990AT</td>
<td>38.4</td>
<td>22.7</td>
</tr>
<tr>
<td>960AT</td>
<td>43.2</td>
<td>23.8</td>
</tr>
<tr>
<td>1050TCR</td>
<td>31.8</td>
<td>19.9</td>
</tr>
<tr>
<td>1020TCR</td>
<td>37.2</td>
<td>19.3</td>
</tr>
<tr>
<td>990TCR</td>
<td>39.4</td>
<td>19.3</td>
</tr>
<tr>
<td>960TCR</td>
<td>39.3</td>
<td>19.3</td>
</tr>
</tbody>
</table>
The experimentally determined HER values from Table 5.12 are also presented in Table 5.16 for convenience. For every condition, Equation 5.6 predicts an HER value lower than the experimental HER from Table 5.12. A potential reason for the lower HER values is because the nine conditions of steel C have tensile properties outside of the ranges used to create Equation 5.6.

Figure 5.21 shows the experimental HER plotted against the HER calculated using Equation 5.6 for multiple steels. The dotted line in Figure 5.21 represents a 1:1 correlation, the filled circles represent data from literature [128], and the open squares represent data for the nine conditions of steel C reported in Table 5.16.

![Figure 5.21](image)

Figure 5.21 Plot of experimental HER vs. predicted HER using Equation 5.6. The dotted line represents a 1:1 correlation, the literature data is presented as filled circles, and the nine conditions of steel C are presented as open squares [128].

The data corresponding to the nine conditions of steel C are located within a relatively narrow region of the plot near the 1:1 correlation line. Equation 5.6 indicates that HER increases with a decrease in UTS, an increase in total elongation, and a decrease in plastic anisotropy. An increase in HER with a decrease in UTS is consistent with the results for the AT conditions of steel C. However, an increase in HER with an increasing ε_t is contradictory to the nine conditions of steel C, but the effect of ε_t on HER in Equation 5.6 appears to be minimal.

The data from the nine conditions of steel C presented in Figures 5.20 and 5.21 (open squares) produced a relatively small range of HER values when compared to the range of HER values for the variety of steel grades used in literature [128, 140]. The range of HER values produced by the nine conditions of steel C was created with constituent hardness as the primary microstructural variable. Other
microstructural properties, such as grain size, morphology, and MVF, can also affect HER values [121]. Figure 5.20 showed that the HER and tensile properties obtained for the nine conditions of steel C produced similar data trends compared to results determined by other researchers [140], and Equation 5.6 is interpreted to be an acceptable first approximation in predicting HER values based on tensile properties. To further improve the accuracy of empirical HER equations, standardization of hole expansion testing would be beneficial. Most studies omit reference to the current ISO16630 standard for hole expansion testing. As shown in Figure 5.12, even for a single hole expansion test, the three different measurement techniques yielded three different HER values.

5.3 Microstructural Response to Plastic Deformation: As-Received and 960TCR Conditions

Correlations between constituent hardness and macroscopic formability parameters were established in Sec. 5.2, where the martensite/ferrite hardness ratio was shown to affect performance in tensile and hole expansion testing. The micro-scale response to plastic deformation, including work hardening, strain partitioning, strain distribution and void damage for different hardness ratios was of interest. The AsR and the 960TCR conditions were chosen for further analysis because of their equivalent UTS values, and their relatively large difference in total elongation (TE) values (11.7 and 4.0 pct, respectively), HER values (29 and 39 pct, respectively), and martensite/ferrite hardness ratio (3.2 and 2.3, respectively).

Four experiments were performed to evaluate the micro-scale response to plastic deformation for different martensite/ferrite hardness ratios. The first experiment involved nanoindentation and EBSD on ASTM E8 standard size tensile specimens in the initial state, and after deformation to the maximum uniform strain (i.e. at UTS) to evaluate work hardening and strain distribution. The second experiment used DIC to characterize strain partitioning between constituents in response to plastic deformation on ASTM E8 sub-size tensile specimens. The third experiment evaluated void density in deformed plane strain tensile specimens to quantify the damage accommodated by the microstructure at similar failure displacements. The plane strain tensile analysis also provided a link between the Ph.D. and the M.Sc. projects of M. D. Taylor. The fourth experiment characterized the void area fraction as a function of strain in the necked region adjacent to the fracture surface using ASTM E8 standard size tensile specimens.

5.3.1 Nanoindentation and EBSD Analysis on Deformed Tensile Specimens

To evaluate the constituent hardness change and potential grain orientation changes in response to tensile deformation, nanoindentation and EBSD analyses were performed on tensile specimens of the AsR and 960TCR conditions deformed to the UTS. For reference, Figure 5.22 shows engineering
stress-engineering strain tensile curves for the AsR and 960TCR conditions, and for each tensile curve, a star approximates the strain level (UTS) where each specimen was evaluated using nanoindentation and EBSD. All nanoindentation tests were performed according to Sec. 4.6, and the experimental procedure outlined in Sec. 4.9 was used to impose the tensile strain.

Table 5.17 reports the average ferrite hardness, average martensite hardness, martensite/ferrite hardness ratio, bulk average hardness, the UE, and the difference in stress between the UTS and the YS (UTS-YS) for the AsR and 960TCR conditions in the final (UE) state. Nanoindentation data for the AsR and 960TCR conditions prior to tensile deformation (i.e. the Initial state) were presented in Table 5.13, and are also reported in Table 5.17 for convenience. After deformation, the AsR condition exhibited an increase in ferrite strength, a decrease in martensite strength, a decrease in the martensite/ferrite hardness ratio, and a decrease in the bulk average hardness. An increase in ferrite hardness is interpreted to be caused by work hardening of the microstructure upon deformation (the stress increased 454 MPa from YS to UTS over 7.8 pct of plastic strain). A lower hardness ratio for the final state suggests that ferrite experienced a greater degree of strengthening relative to martensite, indicating that strain partitioning to ferrite grains likely occurred during tensile deformation. The decreases observed with the bulk average and martensite hardness values were unexpected. Depending on the specific region analyzed, a higher or lower local MVF could account for the decrease in bulk average hardness (i.e. a region with higher MVF will inherently produce a higher bulk average hardness since a greater number of the 225 indents are likely to be located within martensite). The decrease in martensite hardness could also account for the lower bulk average hardness. After deforming to the UTS, martensite was anticipated to either remain at the same hardness, or increase. The martensite hardness data for both the initial state and the final (UE) state had ranges between 4 GPa and 9 GPa, and the decreased average martensite hardness for the final state is interpreted to be a consequence of the particular region chosen for analysis, and would benefit from further study.

The 960TCR condition exhibited a marginal increase in martensite hardness (5.08 to 5.15 GPa), hardness ratio (2.26 to 2.34), and bulk average hardness (3.71 to 3.76 GPa), and a marginal decrease in ferrite hardness (2.25 to 2.20 GPa). The 960TCR condition work-hardened 73 MPa over 1.2 pct of plastic strain, and it was interpreted that the modest work hardening was insufficient for the current nanoindentation technique to accurately resolve a difference between the initial and final state. A greater amount of work hardening is interpreted to promote a measurable difference in constituent hardness using nanoindentation. For the 960TCR condition, the similarity in constituent hardness values and hardness ratios between the initial and final state reflect the minimal amount of work hardening that occurred in achieving the UTS.
Figure 5.22  Engineering stress-engineering strain curves for the As-R and 960TCR conditions. Both conditions were evaluated in the initial condition (0, 0), and also at the UTS using EBSD and nanoindentation. The UTS condition is indicated by a star on each curve.

Table 5.17 – Nanoindentation Data for 960TCR, 960TCR After Strained to UE, As-R, and As-R After Strained to the UE.

<table>
<thead>
<tr>
<th>Condition</th>
<th>$\alpha$ (GPa)</th>
<th>$\alpha'$ (GPa)</th>
<th>$\alpha'/\alpha$</th>
<th>Avg (GPa)</th>
<th>UE (pct)</th>
<th>UTS-YS (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>960TCR Initial</td>
<td>2.25</td>
<td>5.08</td>
<td>2.26</td>
<td>3.71</td>
<td>1.2</td>
<td>73</td>
</tr>
<tr>
<td>960TCR UE</td>
<td>2.20</td>
<td>5.15</td>
<td>2.34</td>
<td>3.76</td>
<td></td>
<td></td>
</tr>
<tr>
<td>As-R Initial</td>
<td>2.15</td>
<td>6.84</td>
<td>3.18</td>
<td>5.02</td>
<td>7.8</td>
<td>454</td>
</tr>
<tr>
<td>As-R UE</td>
<td>2.42</td>
<td>6.34</td>
<td>2.62</td>
<td>4.25</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

For the final (UE) state, Table 5.17 reports a lower ferrite hardness, martensite hardness, bulk average hardness, and hardness ratio for the 960TCR condition compared to the AsR condition. It is interpreted that the 960TCR condition achieved an equivalent UTS (1080 MPa) primarily because of the cold-rolling treatment that acted to strengthen ferrite, thereby increasing the similitude in deformation behavior between ferrite and martensite. The lower hardness ratio (increased hardness similarity) allowed the constituents to mutually accommodate higher stresses before strain partitioning occurred, leading to a higher YS and less work-hardening. The lower hardness ratio corresponding to a higher YS and less work-hardening (960TCR condition), and the higher hardness ratio corresponding to a lower YS and greater work-hardening (AsR condition) is consistent with the interpretations given for Figures 5.15 and 5.17.

Nanoindentation is an effective technique to evaluate the average increase in constituent strength due to work hardening, but is limited in describing the distribution (i.e. uniform or heterogeneous) of work-hardening within small individual ferrite grains. To determine whether strain is distributed...
uniformly or heterogeneously within individual ferrite grains, a different technique was required. Because strain partitioning to ferrite is likely to affect local variations of crystallographic orientations, EBSD was performed to evaluate the potential crystallographic texture for the AsR and 960TCR conditions in the initial state, and final (strained to the UTS) state. EBSD scans were performed according to Sec. 4.7.

Figures 5.23a and 5.23b show inverse pole figure (IPF) maps for the initial state and the final state, respectively, of the AsR condition. Different colors represent the different crystallographic orientations specified on the color triangle inset in Figure 5.23a. A change in orientation (color) within a grain is interpreted to be indicative of heterogeneous strain distribution [13, 135]. For the initial condition (Figure 5.23a), the relatively large regions of solid color represent ferrite grains, which are also indicated by white, featureless regions in the corresponding IQ maps shown in Figure 5.24. The IQ maps in Figures 5.24a and 5.24b represent the same areas shown for the IPF maps for the AsR condition in Figures 5.23a and 5.23b, respectively. The solid colors indicate that the crystallographic orientation of the ferrite grains in the initial state is relatively constant since minimal color changes are observed. Martensite grains are illustrated in Figure 5.23 by the relatively smaller regions containing different colors. Martensite grains are more easily distinguishable in the image quality (IQ) maps presented in Figure 5.24 and are indicated by the darker regions.

Figure 5.23 Inverse pole figure map of the As-R condition in the (a) initial state, and (b) after being strained (7.3 pct) to the UTS. Color gradients are more easily observed in ferrite grains in (b), suggesting the plastic deformation occurred in the form of heterogeneous strain localizations (Color image—see PDF).
Figure 5.23b shows the IPF map for the final state of the AsR condition (i.e. strained to 7.3 pct), where the relatively larger regions exhibiting moderate color gradients are ferrite grains. Color gradients within individual ferrite grains represent crystallographic rotations, and indicate that heterogeneous strain distributions were present within the microstructure. If an entire ferrite grain deformed uniformly, the grain would still exhibit a solid color. A higher density of color gradients was observed within individual ferrite grains in the final state (Figure 5.23b) compared to the initial state (Figure 5.23a), and suggests ferrite heterogeneously accommodated strain during tensile deformation.

Figure 5.24 Image Quality maps of the AsR condition in the (a) initial state, and (b) after being strained (7.3 pct) to the UTS. In both IQ maps, the darker regions are martensite, and the lighter-colored regions are ferrite. The areas represented by the IQ maps are the same areas represented by the IPF maps in Figure 5.23.

Figures 5.25a and 5.25b show the IPF maps for the 960TCR condition in the initial state and the final state, respectively. Larger regions of a similar color exhibiting moderate color gradients are interpreted to be ferrite. Compared to the initial state of the AsR condition (Figure 5.23a), the initial state of the 960TCR condition in Figure 5.25a exhibits a considerable amount of color gradients within individual ferrite grains, a direct result of the plastic deformation experienced during cold-rolling. Whether by cold-rolling or tensile deformation, the EBSD technique illustrates that a DP microstructure responds heterogeneously to plastic deformation. The considerable amount of strain (as inferred by grain rotations) imparted to ferrite from the cold-rolling process is interpreted to be a primary cause for the increased ferrite strength observed in Table 5.13. The IPF map for the 960TCR condition in the final state (Figure 5.25b) also exhibits the presence of grain rotations within individual ferrite grains, and from
a qualitative perspective, it is difficult to differentiate between the two IPF maps in Figure 5.25. The IPF map in Figure 5.25b represents a state that has experienced 1.2 pct elongation, and an increase of 73 MPa compared to the IPF map shown in Figure 5.25a. Due to the modest change in mechanical properties between the IPF maps shown in Figure 5.25, it is difficult to separate grain rotations caused by cold-rolling from the grain rotations caused by tensile deformation. It is interpreted that the heterogeneous strain caused by cold-rolling is essentially indistinguishable from the additional heterogeneous strain caused by tensile deformation using EBSD.

![Figure 5.25](image)

Figure 5.25 Inverse pole figure map of the 960TCR condition in the (a) initial state, and (b) after being strained to the UTS. Color gradients are present in both IPF maps, and a qualitative observation of the two reveals similarity (Color image- see PDF).

The EBSD studies on the AsR and 960TCR conditions illustrate that strains are heterogeneously accommodated within individual ferrite grains upon plastic deformation. The EBSD technique is effective at illustrating grain orientation changes in response to deformation in DP steels provided that the initial state is comprised of grains with relatively constant crystallographic orientation. From a qualitative perspective, comparisons can become complicated when grain rotations in the initial state are present, such as the case of the 960TCR condition. Attempts at quantifying strain distributions based on EBSD crystallographic information would be non-trivial, especially when a separation of grain rotations caused by cold-rolling and tensile straining is necessary. The strain-mapping capability of DIC is one technique that may be able to separate the strains caused by cold-rolling, and reveal the strain distributions caused only by the tensile deformation.
5.3.2 Digital Image Correlation on Deformed Tensile Specimens

The nanoindentation experiment presented in Sec. 5.3.1 reported an increase in ferrite hardness after tensile deformation, suggesting that work-hardening primarily occurred in ferrite. The EBSD analysis presented in Sec. 5.3.1 illustrated that ferrite grains heterogeneously accommodated strain imparted by plastic deformation (cold-rolling and tensile), as inferred from crystallographic rotations. However, EBSD only provides an indirect illustration of the strains caused by cold-rolling and tensile deformation. In response to tensile deformation, the effect of constituent hardness on strain partitioning leading to microstructural damage (decohesion) is of interest.

To observe constituent hardness effects on the development of strain partitioning due to tensile deformation, a DIC analysis was performed on the AsR and 960TCR conditions. The DIC technique calculates strain based on surface features, separating strains caused by cold-rolling from those caused by tensile deformation. Using ASTM E8 sub-size tensile specimens, the 960TCR condition was produced using the tempering parameters for the 960AT condition in Table 5.6, and the %CR value for the 960TCR condition in Table 5.9. Table 5.18 reports the tensile properties for the AsR and 960TCR conditions produced using ASTM E8 sub-size tensile specimens. Unshaded values in Table 5.18 are the average of three tests. Table 5.18 reports the average UTS for the AsR condition to be approximately 25 MPa below the average UTS reported in Table 5.1. The tempering treatment for the 960TCR condition was consistent with previous experiments in that the UTS decreased by approximately 125 MPa to an average of 930 MPa before being cold-rolled to the tensile properties reported in Table 5.18.

<table>
<thead>
<tr>
<th>Condition</th>
<th>YS</th>
<th>UTS</th>
<th>UE</th>
<th>TE</th>
</tr>
</thead>
<tbody>
<tr>
<td>AsR</td>
<td>540</td>
<td>1054</td>
<td>8.9</td>
<td>14.3</td>
</tr>
<tr>
<td>AsR DIC</td>
<td>544</td>
<td>1058</td>
<td>11.7</td>
<td>n/a</td>
</tr>
<tr>
<td>960TCR</td>
<td>973</td>
<td>1058</td>
<td>1.5</td>
<td>5.3</td>
</tr>
<tr>
<td>960TCR DIC</td>
<td>1009</td>
<td>1087</td>
<td>0.8</td>
<td>n/a</td>
</tr>
</tbody>
</table>

Separate DIC analyses were performed for the AsR condition after achieving a stress of 1009 MPa and after being strained to the UTS, and for the 960TCR condition after being strained to the UTS. The approximate DIC analyses locations on the engineering stress-engineering strain tensile curves for the AsR and 960TCR conditions are indicated with stars in Figure 5.26 (3 total).
In Figure 5.26, the solid curve represents the AsR condition, and the dash-dot curve represents the 960TCR condition. The tensile properties for the two tensile curves shown in Figure 5.26 are reported in the shaded rows of Table 5.18 (condition + DIC), and values were obtained from one tensile test. In Table 5.18, the AsR DIC tensile properties were consistent with the AsR tensile properties. The 960TCR DIC specimen exhibited a YS of 1009 MPa, and a UTS of 1087 MPa, both approximately 30 MPa higher than expected based on the 960TCR tensile properties reported in Table 5.18. During the polishing of tensile specimen, the gauge section experienced mild bending, and could have contributed to the discrepancy in tensile properties.

Figure 5.27a and 5.27b show SEM micrographs of the in-plane orientation for the 960TCR condition and the AsR condition, respectively, used for DIC analysis. In Figure 5.27, specimens were polished to 1 µm using standard metallographic techniques, then subsequently etched with 2 pct nital for approximately 8 s. All DIC analyses were performed on the regions shown in the SEM micrographs in Figure 5.27, and the tensile axis was horizontal.

Strain maps created with DIC overlaid on the SEM micrographs for the 960TCR and AsR conditions strained to the UTS are shown in Figures 5.28a and 5.28b, respectively. The strain maps contain different colors, and the colors correspond to the different strains indicated on the color scale to the right of each image. The SEM micrographs are shown with the strain map overlaid to provide a spatial reference for where strain gradients developed within the microstructure. For each strain map presented, the average strain for the entire area calculated by the DIC software is reported in the bottom left corner.
Figure 5.27  SEM micrographs of the in-plane orientation for the 960TCR condition in (a), and for the AsR condition in (b) used for DIC analysis. Steel specimens were etched with 2 pct nital for approximately 8 s. Tensile axis was horizontal.

Figure 5.28  Strain maps generated using DIC for 960TCR condition in (a), and for AsR condition in (b). Both strain maps show higher concentrations of strain in ferrite regions adjacent to martensite, or in ferrite regions that are appreciably constrained by surrounding martensite. For an average strain of 0.7 %, the 960TCR conditions shows strains to range from 0.3 – 2.3 %. For an average strain of 13.9 %, the AsR condition shows strains to range from 6 – 52 %. A martensite grain is indicated by the “0.3%” arrow in (a), and by the “6%” arrow in (b). Color Image – see PDF

For the 960TCR condition in Figure 5.28a, an average strain of 0.7 pct was calculated from the DIC software, and is in good agreement with the UE of 0.8 pct reported in Table 5.18 for 960TCR DIC.
For a 0.7 pct average strain, local strain values ranged from 0.3 – 2.3 pct, indicating that strain partitioning occurred even at low plastic strains. Microstructural regions that experienced the highest local strains were ferrite in close proximity to martensite (indicated as “2.3%” in Figure 5.28a), or ferrite constrained by martensite, and regions experiencing the lowest strains were primarily martensite (indicated as “0.3%” in Figure 5.28a). While strains around 0.7 pct were observed in some regions of martensite, the majority of strains greater than 0.7 pct were located primarily in ferrite, with the highest strains located in ferrite adjacent to martensite. For the AsR condition in Figure 5.28b, the DIC software calculated an average strain of 13.9 pct, a value slightly higher than the 11.7 pct reported in Table 5.18 for AsR DIC. A possible reason for the discrepancy could be caused by the analysis area being too small to represent the bulk response of the microstructure. For an average strain of 13.9 pct, strains ranged from 6 – 52 pct, with lower strains residing primarily in martensite (indicated as “6%” in Figure 5.28b), and higher strains in ferrite within proximity to martensite (indicated as “52%” in Figure 5.28b). Locations of highest strains (martensite/ferrite interfaces) coincide with the microstructural locations where voids are known to preferentially form in DP steels [13].

Compared to the 960TCR condition in Figure 5.28a, the strain map for the AsR condition exhibits more developed bands of strain localization within ferrite grains, and a few bands are indicated in Figure 5.28b with white dashed lines. The higher strains present in ferrite after yielding are interpreted to be one of the contributors to work hardening [39, 117]. The strain gradients within the developed bands likely increased with further deformation, and are believed to be preferential locations for martensite/ferrite interface decohesion to occur.

The strain scales for the DIC maps in Figure 5.28 (color scale) were chosen to best illustrate the strain distribution for each steel condition. Figure 5.29 shows the strain maps from Figure 5.28 using equivalent strain scales to provide an equal comparison, and vividly illustrates the difference in strain magnitude between the 960TCR and AsR conditions. Compared to the AsR condition, the 960TCR condition appears to have negligible strain, and is consistent with the lower UE reported in Table 5.18. Figure 5.29 illustrates the effect of constituent hardness on deformation behavior during tensile testing. For the same load-carrying capacity (1054 MPa), the AsR condition (higher hardness ratio) experienced greater heterogeneous strain compared to the 960TCR condition (lower hardness ratio), and the greater heterogeneous strain observed for a higher hardness ratio supports the interpretation of Figure 5.14 that a lower hardness ratio allows the microstructure to delay strain localization [117]. One potential reason the strain bands in the 960TCR condition appear more diffuse, or less developed in Figure 5.28a compared to the AsR condition is because the overall strain is much lower. It is hypothesized that strain localization within ferrite begins within isolated regions in the microstructure (Figure 5.28a), and with progressive straining, the isolated regions link to form bands of strain localization.
Figure 5.29 Strain maps for the 960TCR condition in (a), and for the AsR condition in (b). Both conditions were strained to the UTS, and are illustrated using an equivalent strain scales. For an equivalent load-carrying capacity, the AsR condition exhibits a greater amount of heterogeneous strain distributions within the microstructure, and is believed to result from the higher constituent hardness ratio. Color image – see PDF

A second DIC analysis on the AsR condition was performed to further illustrate the effect of constituent hardness on strain partitioning, and to test the hypothesis of isolated regions of strain localization linking to form bands of strain localization. The approximate location of the second DIC analysis on the tensile curve for the AsR condition is represented by the star in Figure 5.26 designating a stress of 1009 MPa (3 pct strain). The DIC analysis for the AsR condition at a stress of 1009 MPa was chosen because 1009 MPa was the YS of the 960TCR condition (Table 5.18). It is of interest to observe strain partitioning in the AsR condition at 1009 MPa, a stress value where macroscopic yielding has yet to occur in the 960TCR condition. At stresses lower than 1009 MPa, ferrite and martensite in the 960TCR condition uniformly share strain since both constituents have the same elastic modulus.

Figures 5.30a and 5.30b show SEM micrographs with the strain maps on equivalent strain scales overlaid for the 960TCR and AsR conditions at 1009 MPa, respectively. At stresses below 1009 MPa, the ferrite and martensite present in the 960TCR condition are mutually deforming in the elastic regime, and is interpreted to be in response to the increased ferrite strength from cold-rolling (lower hardness ratio). For the AsR condition (higher hardness ratio), a stress of 1009 MPa is achieved only after the microstructure work hardens 465 MPa. For an average strain of 2.95 pct, local strains between 1.2 – 8 pct are observed. Before macroscopic yielding occurs in the 960TCR condition, the AsR condition has already experienced local plastic strains over 8 pct.
Strain maps for the 960TCR and AsR conditions in Figure 5.30 support the data trend in Figure 5.15, where a lower hardness ratio correlated with a higher YS. For the AsR condition in Figures 5.30b (2.95 pct strain) and 5.28b (13.9 pct strain), strain localizations are observed in similar areas, but strains appear more diffuse in the condition at 2.95 pct strain, supporting the hypothesis that strain partitioning first occurs in isolated regions within the microstructure, then link to form bands of shear localization.

Strain maps generated using DIC for different stress stages in the AsR and 960TCR conditions illustrate the strain partitioning that occurs at the microstructure-level in response to different constituent hardness ratios. In all cases, upon yielding, strain preferentially localizes inside ferrite grains, with highest strains occurring in the ferrite within proximity of martensite. Upon further deformation, the localized regions link to form bands of strain localization within the microstructure. Regardless of the martensite/ferrite hardness ratio, strains heterogeneously develop in the microstructure at locations consistent with preferential fracture sites in DP steels (martensite/ferrite interfaces). The hardness ratio primarily affected the magnitude of strain in the localized bands [116].

5.3.3 Void Density Analysis: Plane Strain Tensile Tests

A lower hardness ratio is interpreted to suppress microstructural damage in DP steels, enabling the accommodation of higher stresses before fracture [40]. For an equivalent imposed strain, a study involving six commercially-produced DP steels showed a lower martensite/ferrite hardness ratio to
correspond to a lower void population [40]. The study on the six commercially-produced DP steels utilized a special plane strain tensile specimen geometry which concentrated damage/fracture on a plane 45° with respect to the specimen surface and tensile direction by using offset semi-circular notches, and is shown in Figure 4.8. Plane strain tensile specimens of the AsR and 960TCR conditions were machined from the cold-rolled sheets used for hole expansion testing, and plane strain analysis was performed according to the method outlined in Sec. 4.11.

Figures 5.31a and 5.31b show the load-displacement curves for the AsR and 960TCR conditions, respectively. For each plot, the dotted curve represents a plane strain tensile specimen that was tested until fracture to determine a failure displacement. The solid curve represents the plane strain tensile specimen that was deformed to approximately 90 pct of the failure displacement, then unloaded for analysis. Based on previous results, loading to 90 pct of the failure displacement was shown to result in significant localized void formation in the plane strain tensile specimen [40].

![Load-displacement curves for the plane strain tensile specimen of the AsR condition in (a), and for the 960TCR condition in (b).](image)

The 960TCR condition exhibited a lower load, and lower displacement compared to the AsR condition. The displacement measurement method is one potential reason why the 960TCR condition exhibited a lower displacement. To measure displacement, a 12.7 mm gauge section was monitored, while the reduced section was approximately 0.6 mm. For the AsR condition, the entire gauge section plastically deformed at loads as low as 1400 kg., contributing to a larger displacement. The observed displacement for the AsR condition characterized the entire gauge section, with only a portion of the
displacement representing the deformation within 0.6 mm reduced section. For the 960TCR condition, the observed displacement was constrained to the 0.6 mm reduced section, as the 12.7 mm gauge section outside of the reduced section experienced a stress considerably lower than the YS.

Figure 5.32 shows the number of voids plotted as a function of martensite/ferrite hardness ratio for six commercially-produced DP steels (filled circles) from the M.Sc. thesis of M. D. Taylor [40]. An example SEM micrograph showing voids was presented in Sec. 4.11 as Figure 4.14. The linear regression and correlation coefficient presented in Figure 5.32 was created using the data from the six commercially-produced DP steels. Figure 5.32 also shows the number of voids plotted as a function of martensite/ferrite hardness ratio for the AsR (open square) and 960TCR (open diamond) conditions. A direct comparison of the data for the 960TCR and AsR conditions shows that an increase in hardness ratio corresponds to an increase in void population, an observation consistent with the interpretation that a decrease in hardness ratio suppresses void nucleation.

Figure 5.32 Number of voids as a function of martensite/ferrite hardness ratio, showing an increase in the number of voids with an increase in hardness ratio. Data representing a previous study on six commercially-produced DP steels are shown as filled circles.

The 960TCR and AsR conditions exhibit an increase in number of voids with increasing hardness ratio, a behavior consistent with the data from the six commercially-produced DP steels, though the slopes appear to differ. The void data for the 960TCR condition appears to be similar to the DP steels of comparable hardness ratio from the M.Sc. data, but the void data for the AsR condition are lower than expected. The six DP steels from the previous study were tested with the rolling direction (R.D.) parallel to the tensile axis, whereas the AsR and 960TCR conditions were tested with the R.D. transverse to the tensile axis. When the R.D. was parallel to the tensile axis, “stringer” type features were present in the analysis area, and voids were observed along these elongated “stringers”, increasing the void population.
for select DP steels from the previous study. When the R.D. was transverse to the tensile axis, “stringer”
type features were normal to the plane of analysis, decreasing the area fraction of “stringers” in the
analysis area, and could be one reason for the lower observed void population for the AsR and 960TCR
conditions compared to the six DP steels from the previous study.

5.3.4 Void Area Fraction Analysis on Fractured Tensile Specimens

Void properties adjacent to the fracture surface of ASTM E8 standard size tensile specimens were
evaluated for the AsR and 960TCR conditions. Strains greater than the TE develop in the necked region
of a tensile specimen, and the void properties for different martensite/ferrite hardness ratios were of
interest. Testing of ASTM E8 standard size tensile specimens was performed according to Sec. 4.9, and
void analysis was performed according to the procedure outlined in Sec. 4.12. An SEM micrograph of
the AsR and 960TCR conditions acquired using BSE imaging are shown in Figures 5.33a and 5.33b,
respectively. In Figure 5.33, thickness direction is vertical, and the fracture surface is on the left.

![SEM micrograph of the fractured end of an ASTM E8 tensile specimen for the AsR condition in (a), and the 960TCR condition in (b). Thickness direction is vertical, and fracture surface is on the left for each image. Specimens were etched with 2 pct nital for approximately 8 s.](image)

Figure 5.34 shows a plot of void area pct as a function of local thickness strain for the AsR,
960TCR, and 960AT conditions. The data trends in Figure 5.34 are similar to those obtained by
Steinbrunner, who also evaluated void area pct as a function of thickness strain for DP steels [19]. For
each dataset, an exponential fit was performed, and both AsR and 960TCR conditions exhibit correlation
coefficients of 0.93 and 0.95, respectively. For an equivalent local thickness strain, the 960TCR
condition exhibits a lower void area pct compared to the AsR condition, indicating that for equivalent

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strain and equivalent stress, the lower hardness ratio suppressed microstructural damage. The lower void area fraction caused by the lower hardness ratio necessitates a greater degree of stress triaxiality to generate the critical amount of voids to induce fracture. One index of stress triaxiality is the maximum thickness strain at tensile fracture, and Table 5.19 reports the maximum thickness strain experienced for the AsR, 960AT, and 960TCR conditions. The UE (Tables 5.1, 5.7, and 5.10) and the martensite/ferrite hardness ratios (Table 5.13) are also reported in Table 5.19 for convenience. In Table 5.19, an increasing maximum thickness strain is observed with decreasing martensite/ferrite hardness ratio, and steel conditions having the lowest UE in tensile tests exhibited the largest maximum thickness strain.

![Figure 5.34 Plot of void area pct as a function of local thickness strain (pct) for the AsR, 960AT, and 960TCR conditions. All conditions exhibit an exponential increase in voids with increasing local thickness strain, and the AsR condition had the greatest void area pct for a given strain value.](image)

<table>
<thead>
<tr>
<th>Condition</th>
<th>Max Strain (pct)</th>
<th>$\alpha'/\alpha$</th>
<th>UE</th>
</tr>
</thead>
<tbody>
<tr>
<td>AsR</td>
<td>42.5</td>
<td>3.2</td>
<td>7.8</td>
</tr>
<tr>
<td>960AT</td>
<td>58.9</td>
<td>2.7</td>
<td>5.6</td>
</tr>
<tr>
<td>960TCR</td>
<td>63.5</td>
<td>2.3</td>
<td>1.2</td>
</tr>
</tbody>
</table>

Table 5.19 – Max Thickness Strain (in pct) and Martensite/ferrite Hardness Ratio for AsR, 960AT, and 960TCR Conditions of Steel C.
The 960AT condition was also evaluated to provide further verification that a lower hardness ratio corresponded to a lower void area pct for an equivalent strain. The 960AT condition had a higher hardness ratio than the 960TCR condition and exhibited similar void area pct values at equivalent strains, but the correlation coefficient for the exponential fit was lower (0.73 vs. 0.95). Additionally, the 960AT condition experienced a stress 120 MPa below that of the 960TCR and AsR conditions, and it was interpreted that the higher stress imposed on the AsR and 960TCR conditions allowed the critical condition for void nucleation to occur more frequently.

The max thickness strain reported in Table 5.19 appears to increase with HER (Table 5.12), and a study that correlated engineering HER with true fracture strain using a variety of steels showed an increased engineering HER with increased true fracture strain [120]. Engineering HER and true fracture strain values for six steels from a study by Link [120], and for the AsR, 960AT, and 960TCR conditions are shown in Table 5.20. High strength low alloy (HSLA) steels containing ferrite/pearlite microstructures, and transformation-induced plasticity (TRIP) steels containing ferrite/austenite/bainite microstructures were present in the study by Link [120]. The IBF suffix on select DP steel grades designates steels with “improved bending and flangeability”. The max thickness strain reported in Table 5.19 for the AsR, 960AT, and 960TCR conditions was converted to true fracture strain by using Equation 5.7, where \( t_i \) and \( t_f \) are the initial and final thicknesses (in mm), \( w_i \) and \( w_f \) are the initial and final width (in mm), and \( A_i \) and \( A_f \) are the initial and final cross-sectional areas (mm\(^2\)) of the tensile gauge section. The change in tensile specimen width was assumed to be negligible, and the change in thickness was used to calculate the true fracture strain.

\[
\text{True Fracture Strain} \left( \varepsilon_f \right) = \ln \left( \frac{t_i \cdot w_i}{t_f \cdot w_f} \right) = \ln \left( \frac{A_i}{A_f} \right)
\]  

(5.7)

Figure 5.35 shows engineering HER plotted as a function of true fracture strain for the six steels from Link [120] as filled circles. The six steels were used to create the linear fit and the corresponding correlation coefficient of 0.98, indicating that the true fracture strain obtained from a uniaxial tensile test can be an accurate indicator of HER. Figure 5.35 also plots the three conditions of steel C as open squares, and the data appear to be consistent with the results of Link [120]. The 960AT and 960TCR conditions (higher UTS) exhibited larger HER values than the DP780 and TRIP780 steels (lower UTS) from [120], which suggests that the treatments of tempering and cold-rolling on steel C increased HER to values comparable to lower-strength AHSS grades.
Table 5.20 – Material Designation, HER, and True Fracture Strain for Six Steels from a Study by Link [120], and for the AsR, 960AT, and 960TCR Conditions.

<table>
<thead>
<tr>
<th>Steel</th>
<th>HER</th>
<th>True Fracture Strain (pct)</th>
</tr>
</thead>
<tbody>
<tr>
<td>HSLA 440</td>
<td>100</td>
<td>1.68</td>
</tr>
<tr>
<td>DP590</td>
<td>58</td>
<td>1.11</td>
</tr>
<tr>
<td>DP590IBF</td>
<td>66</td>
<td>1.28</td>
</tr>
<tr>
<td>DP780</td>
<td>24</td>
<td>0.57</td>
</tr>
<tr>
<td>DP780IBF</td>
<td>46</td>
<td>0.8</td>
</tr>
<tr>
<td>TRIP780</td>
<td>23</td>
<td>0.52</td>
</tr>
</tbody>
</table>

Ref. [120]

<table>
<thead>
<tr>
<th>Steel C</th>
<th>AsR</th>
<th>True Fracture Strain (pct)</th>
</tr>
</thead>
<tbody>
<tr>
<td>960AT</td>
<td>43.2</td>
<td>0.89</td>
</tr>
<tr>
<td>960TCR</td>
<td>39.3</td>
<td>1.01</td>
</tr>
</tbody>
</table>

Figure 5.35  Plot of engineering HER as a function of true fracture strain for six steels from [120] (filled circles), and for the AsR, 960AT, and 960TCR conditions (open squares) showing an increase in HER with increasing true fracture strain.
CHAPTER 6
SUMMARY

The M.Sc. thesis of M. D. Taylor concluded that a lower martensite/ferrite hardness ratio correlated with less microstructural damage in the presence of a triaxial stress state for a group of commercially-produced DP steels. It was hypothesized that increased similitude in ferrite and martensite strength (i.e. lower martensite/ferrite hardness ratio) will delay strain localization, thereby suppressing microstructural damage and achieve higher forming limits.

Steel C, a commercially-produced DP steel with 1081 MPa UTS, was processed to create eight additional constituent hardness conditions by tempering and cold-rolling, processes shown to primarily affect constituent hardness properties. Constituent hardness properties for the nine conditions of steel C (as-received, four as-tempered, four temper cold-rolled) were evaluated using nanoindentation, and exhibited ferrite hardness values from 1.9 to 2.5 GPa, martensite hardness values from 5.1 to 6.8 GPa, and martensite/ferrite hardness ratios from 2.3 to 3.2. The generated constituent hardness property ranges were determined to be sufficient to evaluate formability performance. The UTS of the nine conditions of steel C ranged from 954 to 1090 MPa.

The current project focused on evaluating the isolated effects of constituent hardness on subsequent formability performance for higher-strength DP steels using tensile and hole expansion testing. A hypothesis stating that correlations between martensite hardness and HER would improve if microstructural properties of grain size, MVF, morphology, and chemical content were constant was confirmed in the current study.

For the nine conditions of steel C, a lower martensite/ferrite hardness ratio corresponded to an increase in HER and an increase in YS. A lower martensite/ferrite hardness ratio (increased similarity in ferrite and martensite hardness) was interpreted to increase strain-sharing between ferrite and martensite. The increased strain-sharing facilitated similar deformation behavior of ferrite and martensite, and suppressed plastic strain localization to higher stresses for the case of YS, and to higher formability limits for the case of HER. A lower martensite/ferrite hardness ratio correlated with a decrease in work-hardening (increasing YS/UTS) and was interpreted to be caused by the suppression of plastic strain localization within ferrite.

Tensile and hole expansion properties for the nine conditions of steel C were evaluated using two different studies from literature that focused on characterizing HER based on tensile properties. The nine conditions of steel C produced consistent trends with the data reported in each study, confirming the experimental HER and tensile properties obtained in the current study are consistent with literature.
The microstructural response to plastic deformation for the AsR and 960TCR conditions (equivalent UTS, but different TE, HER, and constituent hardness) were evaluated using techniques of nanoindentation, EBSD, DIC, and plane strain tensile tests. After deforming the two DP steels to the UTS, the DP steel with a higher initial martensite/ferrite hardness ratio exhibited an increase in ferrite hardness, and is interpreted to be caused by work hardening of the microstructure upon deformation. A decrease in martensite/ferrite hardness ratio after plastic deformation was also observed, suggesting that ferrite experienced a greater degree of strengthening relative to martensite, and indicates that strain partitioning to ferrite grains likely occurred during tensile deformation. The DP steel with a lower initial martensite/ferrite hardness ratio exhibited minimal work hardening, and the constituent hardness properties after being deformed to the UTS were similar to the initial state.

Grain orientation maps created using EBSD for the initial state, and after being deformed to the UTS for the AsR and 960TCR conditions showed that, whether by cold-rolling or tensile deformation, a DP microstructure heterogeneously accommodates strains imparted by plastic deformation, as inferred from grain rotations within individual ferrite grains. The DP steel with higher initial martensite/ferrite hardness ratio exhibited a larger UE, and a greater increase in grain rotations within ferrite after being strained to the UTS compared to the DP steel of lower hardness ratio. For an equivalent stress, the DP steel with lower hardness ratio and lower UE suppressed strain localization to higher stresses.

Strain maps generated using DIC for the AsR and 960TCR conditions at different tensile deformation stages illustrate that, regardless of martensite/ferrite hardness ratio, strains heterogeneously develop in the microstructure at locations consistent with preferential fracture sites (ferrite/martensite interface) in DP steels. The hardness ratio primarily affected the magnitude of strain at a given stress, with higher martensite/ferrite hardness ratios exhibiting larger local strain gradients. Strain was observed to preferentially localize inside ferrite grains, and the highest strains occurred in the ferrite within proximity of martensite. Upon further deformation, isolated regions of strain link to form bands of strain localization within the microstructure, and preferentially form in large, connected regions of ferrite.

Plane strain tensile tests for the AsR and 960TCR conditions showed that a decrease in hardness ratio corresponded to a lower void population, a trend consistent with results established in the M.Sc. thesis of M. D. Taylor. Void area pct as a function of distance from the fracture surface of ASTM E8 standard size tensile specimens showed a lower void area pct at equivalent stress and strain for the DP steel with lower hardness ratio, confirming that lower hardness ratios suppress microstructural damage. A larger maximum thickness strain was observed for the DP steel with lower hardness ratio, indicating a higher triaxial stress state was required to produce the critical void condition for fracture. A larger maximum true thickness strain corresponded to larger HER values, and the trend was consistent with results reported in literature.
CHAPTER 7
FUTURE WORK

The current project has established constituent hardness effects on the deformation and formability of DP steels while holding grain size and MVF constant. An extension of the current work would be to vary grain size, and hold MVF constant to determine the effects of grain size on deformation and formability of DP steels. To isolate morphological effects, an equiaxed microstructure should be considered.

Characterizing the critical interfacial strains for void nucleation as a function of martensite/ferrite hardness ratio for DP steels under tensile deformation would provide insight on whether the critical strain is a function of hardness ratio, or if the critical strain is an absolute value. Evaluation of strain partitioning at equivalent strains for different martensite/ferrite hardness ratios could clarify whether the magnitude of observed strain gradients are a function of hardness ratio, or purely a function of global strain. Reproduction of DIC strain maps using a microstructure-based FEM would provide a computational tool on which deformation behavior for different constituent hardness property combinations could be evaluated. Incorporating bi-axial tensile tests would illustrate the strain behavior of DP steels for a stress state more representative of certain forming operations.

A more comprehensive analysis on the constituent hardness evolution upon tensile deformation of DP steels could aid in determining whether only ferrite work hardens, or if martensite will also work harden when the martensite/ferrite hardness ratio is sufficiently low. Such an experiment would begin to characterize the specific role of martensite in DP steels. Incorporating bi-axial tensile tests would illustrate constituent hardness changes for a stress state more representative of certain forming operations.
REFERENCES CITED


A. C. Fischer-Cripps, “Critical Review of Analysis and Interpretation of Nanoindentation Test


[127] B. S. Levy, M. Gibbs, and C. J. Van Tyne, “Failure During Sheared Edge Stretching of Dual-


APPENDIX A

COMPILATION OF EXPERIMENTAL DATA WITH UNCERTAINTY ANALYSES

Figure A.1 Hardness histogram for ferrite in (a) and martensite in (b) for the as-received condition of steel A. Both histograms approximate a normal distribution, with an average ferrite hardness of 2.97 GPa, and an average martensite hardness of 7.04 GPa.

Figure A.2 Hardness histogram for ferrite in (a) and martensite in (b) for the as-received condition of steel B. Both histograms approximate a normal distribution, with an average ferrite hardness of 3.21 GPa, and an average martensite hardness of 7.29 GPa.
Figure A.3  Hardness histogram for ferrite in (a) and martensite in (b) for the as-received condition of steel D. Both histograms approximate a normal distribution, with an average ferrite hardness of 2.57 GPa, and an average martensite hardness of 6.67 GPa.

Table A.1 – Average Microstructural and Tensile Properties in the As-Received Condition for Steels A-D. One Standard Deviation is Reported Below (In Parentheses) Each Reported Microstructural and Tensile Property. Standard Deviations for Tensile Properties Calculated from Three Separate Tests. Standard Deviations for Grain Size and MVF Calculated from Twenty-Four Separate Measurements.

<table>
<thead>
<tr>
<th>Steel</th>
<th>YS (MPa)</th>
<th>UTS (MPa)</th>
<th>UE (pct)</th>
<th>TE (pct)</th>
<th>G.S. α (μm)</th>
<th>G.S. α’ (μm)</th>
<th>MVF (pct)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>946</td>
<td>1225</td>
<td>5.9</td>
<td>10.8</td>
<td>1.42</td>
<td>1.86</td>
<td>60.9</td>
</tr>
<tr>
<td></td>
<td>(15.9)</td>
<td>(6.2)</td>
<td>(0.3)</td>
<td>(0.1)</td>
<td>(0.2)</td>
<td>(0.4)</td>
<td>(6.1)</td>
</tr>
<tr>
<td>B</td>
<td>923</td>
<td>1221</td>
<td>5.9</td>
<td>10.1</td>
<td>1.43</td>
<td>1.60</td>
<td>56.0</td>
</tr>
<tr>
<td></td>
<td>(5.8)</td>
<td>(2.7)</td>
<td>(0.1)</td>
<td>(0.4)</td>
<td>(0.2)</td>
<td>(0.2)</td>
<td>(4.8)</td>
</tr>
<tr>
<td>C</td>
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<td>1081</td>
<td>7.8</td>
<td>11.7</td>
<td>2.94</td>
<td>3.14</td>
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<td>(7.9)</td>
<td>(0.4)</td>
<td>(0.2)</td>
<td>(0.7)</td>
<td>(0.5)</td>
<td>(6.7)</td>
</tr>
<tr>
<td>D</td>
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<td>1.30</td>
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<td>(14.1)</td>
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<td>(0.2)</td>
<td>(0.2)</td>
<td>(0.2)</td>
<td>(5.6)</td>
</tr>
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<table>
<thead>
<tr>
<th>Condition</th>
<th>UTS</th>
<th>YS</th>
<th>TE</th>
<th>UE</th>
</tr>
</thead>
<tbody>
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<td>1050AT</td>
<td>1052</td>
<td>651</td>
<td>11.7</td>
<td>7.7</td>
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<tr>
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<td>(18.6)</td>
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<td>(0.49)</td>
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<tr>
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<td>780</td>
<td>11.1</td>
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<tr>
<td></td>
<td>(11.3)</td>
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<td>(0.38)</td>
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<td>960AT</td>
<td>954</td>
<td>809</td>
<td>10.1</td>
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<tr>
<td></td>
<td>(6.8)</td>
<td>(8.1)</td>
<td>(0.28)</td>
<td>(0.12)</td>
</tr>
</tbody>
</table>

Table A.3 – Tensile Properties (UTS, YS, TE, and UE) for Four Temper, Cold-Rolled Conditions of Steel C. One Standard Deviation is Reported Below (In Parentheses) Each Reported Tensile Property. Standard Deviations for Tensile Properties Calculated for Three Separate Tests.

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<th>Condition</th>
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<th>YS</th>
<th>TE</th>
<th>UE</th>
</tr>
</thead>
<tbody>
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<td>1050TCR</td>
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<td>884</td>
<td>9.8</td>
<td>5.6</td>
</tr>
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<td></td>
<td>(19)</td>
<td>(34)</td>
<td>(1.1)</td>
<td>(0.4)</td>
</tr>
<tr>
<td>1020TCR</td>
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<td>979</td>
<td>6.1</td>
<td>2.3</td>
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<tr>
<td></td>
<td>(5)</td>
<td>(14)</td>
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<td>(0.2)</td>
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<td>1003</td>
<td>3.9</td>
<td>1.3</td>
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<tr>
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<td>(16)</td>
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<td>960TCR</td>
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<td>1.2</td>
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<td></td>
<td>(5)</td>
<td>(14)</td>
<td>(0.1)</td>
<td>(0.1)</td>
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Table A.4 – Average HER Values For Each of the Three Measurement Methods for the As-Received and Eight Modified Conditions of Steel C. One Standard Deviation is Reported Below (In Parentheses) Each Reported HER Measurement. Standard Deviations for HER Values Calculated from Three to Five Separate Tests.

<table>
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<th>Condition</th>
<th>Computer HER</th>
<th>Caliper HER</th>
<th>ImageJ HER</th>
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<td>As-R</td>
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<td>(1.6)</td>
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<td>26.1</td>
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<td>(0.6)</td>
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<td>(5.0)</td>
<td>(4.7)</td>
<td>(3.5)</td>
</tr>
<tr>
<td>960TCR</td>
<td>39.3</td>
<td>36.5</td>
<td>34.1</td>
</tr>
<tr>
<td></td>
<td>(3.3)</td>
<td>(3.1)</td>
<td>(2.7)</td>
</tr>
</tbody>
</table>
Table A.5 – Average Ferrite (α) and Martensite (α’) Hardness Values for the Nine Conditions of Steel C Obtained Using Nanoindentation. One Standard Deviation is Reported Below (In Parentheses) Each Reported Constituent Hardness Value. Standard Deviations for Average Ferrite and Average Martensite Hardness Calculated from Over Fifty Separate Measurements.

<table>
<thead>
<tr>
<th>Condition</th>
<th>α</th>
<th>α’</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-R</td>
<td>2.15</td>
<td>6.84</td>
</tr>
<tr>
<td></td>
<td>(0.42)</td>
<td>(0.96)</td>
</tr>
<tr>
<td>1050AT</td>
<td>2.20</td>
<td>6.20</td>
</tr>
<tr>
<td></td>
<td>(0.33)</td>
<td>(0.68)</td>
</tr>
<tr>
<td>1020AT</td>
<td>2.25</td>
<td>6.05</td>
</tr>
<tr>
<td></td>
<td>(0.50)</td>
<td>(0.81)</td>
</tr>
<tr>
<td>990AT</td>
<td>2.05</td>
<td>5.27</td>
</tr>
<tr>
<td></td>
<td>(0.28)</td>
<td>(0.86)</td>
</tr>
<tr>
<td>960AT</td>
<td>1.93</td>
<td>5.18</td>
</tr>
<tr>
<td></td>
<td>(0.28)</td>
<td>(0.85)</td>
</tr>
<tr>
<td>1050TCR</td>
<td>2.45</td>
<td>6.21</td>
</tr>
<tr>
<td></td>
<td>(0.33)</td>
<td>(0.74)</td>
</tr>
<tr>
<td>1020TCR</td>
<td>2.18</td>
<td>5.53</td>
</tr>
<tr>
<td></td>
<td>(0.27)</td>
<td>(0.69)</td>
</tr>
<tr>
<td>990TCR</td>
<td>2.28</td>
<td>5.34</td>
</tr>
<tr>
<td></td>
<td>(0.38)</td>
<td>(0.74)</td>
</tr>
<tr>
<td>960TCR</td>
<td>2.25</td>
<td>5.08</td>
</tr>
<tr>
<td></td>
<td>(0.38)</td>
<td>(0.78)</td>
</tr>
</tbody>
</table>
Table A.6 – Average Ferrite ($\alpha$) and Martensite ($\alpha'$) Hardness Values for the Region Adjacent to the Sheared Hole for the Nine Conditions of Steel C. One Standard Deviation is Reported Below (In Parentheses) Each Reported Constituent Hardness Value. Standard Deviations for Average Ferrite and Average Martensite Hardness Calculated from Over Fifty Separate Measurements.

<table>
<thead>
<tr>
<th>Condition</th>
<th>$\alpha$</th>
<th>$\alpha'$</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-R Sheared Hole</td>
<td>3.32</td>
<td>7.09</td>
</tr>
<tr>
<td></td>
<td>(0.47)</td>
<td>(0.74)</td>
</tr>
<tr>
<td>1050AT Sheared Hole</td>
<td>3.31</td>
<td>6.78</td>
</tr>
<tr>
<td></td>
<td>(0.37)</td>
<td>(0.76)</td>
</tr>
<tr>
<td>1020AT Sheared Hole</td>
<td>3.07</td>
<td>6.39</td>
</tr>
<tr>
<td></td>
<td>(0.41)</td>
<td>(0.68)</td>
</tr>
<tr>
<td>990AT Sheared Hole</td>
<td>3.13</td>
<td>6.21</td>
</tr>
<tr>
<td></td>
<td>(0.56)</td>
<td>(0.85)</td>
</tr>
<tr>
<td>960AT Sheared Hole</td>
<td>3.07</td>
<td>5.84</td>
</tr>
<tr>
<td></td>
<td>(0.41)</td>
<td>(0.80)</td>
</tr>
<tr>
<td>1050TCR Sheared Hole</td>
<td>3.64</td>
<td>6.99</td>
</tr>
<tr>
<td></td>
<td>(0.55)</td>
<td>(0.79)</td>
</tr>
<tr>
<td>1020TCR Sheared Hole</td>
<td>3.38</td>
<td>6.94</td>
</tr>
<tr>
<td></td>
<td>(0.37)</td>
<td>(0.92)</td>
</tr>
<tr>
<td>990TCR Sheared Hole</td>
<td>2.97</td>
<td>6.23</td>
</tr>
<tr>
<td></td>
<td>(0.47)</td>
<td>(0.91)</td>
</tr>
<tr>
<td>960TCR Sheared Hole</td>
<td>3.10</td>
<td>5.78</td>
</tr>
<tr>
<td></td>
<td>(0.37)</td>
<td>(0.87)</td>
</tr>
</tbody>
</table>
## APPENDIX B

### TENSILE DATA FOR TEMPERED AND COLD-ROLLED STEEL C

Table B.1 – Tempering Temperature, UTS, TE, HJP, and VHN Values for Tensile Specimens of Steels A and C Used for Preliminary Tempering Experiment.

<table>
<thead>
<tr>
<th>Tempering Temp (°C)</th>
<th>UTS (MPa)</th>
<th>TE (pct)</th>
<th>HJP</th>
<th>VHN</th>
</tr>
</thead>
<tbody>
<tr>
<td>AsR</td>
<td>1225</td>
<td>10.8</td>
<td>0</td>
<td>389</td>
</tr>
<tr>
<td>175</td>
<td>1187</td>
<td>9.2</td>
<td>9891</td>
<td>381</td>
</tr>
<tr>
<td>200</td>
<td>1170</td>
<td>9.25</td>
<td>10443</td>
<td>381</td>
</tr>
<tr>
<td>Steel A</td>
<td>225</td>
<td>1128</td>
<td>8.85</td>
<td>10995</td>
</tr>
<tr>
<td>250</td>
<td>1128</td>
<td>8.35</td>
<td>11547</td>
<td>354</td>
</tr>
<tr>
<td>300</td>
<td>1067</td>
<td>8.85</td>
<td>12651</td>
<td>358</td>
</tr>
<tr>
<td>350</td>
<td>1013</td>
<td>8.75</td>
<td>13755</td>
<td>326</td>
</tr>
<tr>
<td>As-R</td>
<td>1082</td>
<td>11.6</td>
<td>0</td>
<td>339</td>
</tr>
<tr>
<td>175</td>
<td>1006</td>
<td>10.4</td>
<td>9891</td>
<td>331</td>
</tr>
<tr>
<td>200</td>
<td>991</td>
<td>11.1</td>
<td>10443</td>
<td>331</td>
</tr>
<tr>
<td>Steel C</td>
<td>225</td>
<td>966</td>
<td>10</td>
<td>10995</td>
</tr>
<tr>
<td>250</td>
<td>946</td>
<td>9.5</td>
<td>11547</td>
<td>323</td>
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<tr>
<td>300</td>
<td>913</td>
<td>9.65</td>
<td>12651</td>
<td>308</td>
</tr>
<tr>
<td>350</td>
<td>848</td>
<td>11.7</td>
<td>13755</td>
<td>285</td>
</tr>
</tbody>
</table>

Table B.2 – Tensile Properties of YS, UTS, UE, and TE, Along With %CR and Change in UTS for Specimens of Steel C Used for Preliminary Cold-Rolling Experiment. For Both Strength Conditions, Tensile Properties of As-Tempered Specimen is Shaded Gray. TE Values Reporting N/A Exhibited Fracture Outside the Extensometer.

<table>
<thead>
<tr>
<th>%CR</th>
<th>YS</th>
<th>UTS</th>
<th>UE</th>
<th>TE</th>
<th>ΔUTS</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>758</td>
<td>920</td>
<td>5.9</td>
<td>10.2</td>
<td>0</td>
</tr>
<tr>
<td>4.9</td>
<td>899</td>
<td>981</td>
<td>1.7</td>
<td>6.3</td>
<td>61</td>
</tr>
<tr>
<td>9.6</td>
<td>928</td>
<td>1021</td>
<td>1.2</td>
<td>4</td>
<td>100</td>
</tr>
<tr>
<td>10.2</td>
<td>948</td>
<td>1016</td>
<td>1.3</td>
<td>4.2</td>
<td>96</td>
</tr>
<tr>
<td>14.9</td>
<td>942</td>
<td>1037</td>
<td>1.3</td>
<td>3.8</td>
<td>117</td>
</tr>
<tr>
<td>20.6</td>
<td>933</td>
<td>1045</td>
<td>1.2</td>
<td>3.7</td>
<td>124</td>
</tr>
<tr>
<td>0</td>
<td>788</td>
<td>969</td>
<td>5.3</td>
<td>9.9</td>
<td>0</td>
</tr>
<tr>
<td>5.4</td>
<td>919</td>
<td>1030</td>
<td>1.9</td>
<td>5.7</td>
<td>61</td>
</tr>
<tr>
<td>8.8</td>
<td>927</td>
<td>1045</td>
<td>1.2</td>
<td>n/a</td>
<td>76</td>
</tr>
<tr>
<td>10.2</td>
<td>941</td>
<td>1042</td>
<td>1.5</td>
<td>4.3</td>
<td>73</td>
</tr>
<tr>
<td>15</td>
<td>984</td>
<td>1088</td>
<td>1.2</td>
<td>4</td>
<td>119</td>
</tr>
<tr>
<td>25.7</td>
<td>1041</td>
<td>1133</td>
<td>1.1</td>
<td>n/a</td>
<td>164</td>
</tr>
</tbody>
</table>
APPENDIX C
SEM MICROGRAPHS OF SHEARED HOLE MICROSTRUCTURE

Figure C.1  SEM Micrograph showing the longitudinal orientation of the region adjacent to the sheared edge in the as-received condition of steel C with the nanoindentation array overlaid. Sheared edge is on the left in figure. Steel was etched with 2 pct nital for 6 s.

Figure C.2  SEM Micrograph showing the longitudinal orientation of the region adjacent to the sheared edge in the 1050TCR condition of steel C with the nanoindentation array overlaid. Sheared edge is on the right in figure. Steel was etched with 2 pct nital for 6 s.
Figure C.3  SEM Micrograph showing the longitudinal orientation of the region adjacent to the sheared edge in the 1050AT condition of steel C with the nanoindentation array overlaid. Sheared edge is on the left in figure. Steel was etched with 2 pct nital for 6 s.

Figure C.4  SEM Micrograph showing the longitudinal orientation of the region adjacent to the sheared edge in the 1020TCR condition of steel C with the nanoindentation array overlaid. Sheared edge is on the right in figure. Steel was etched with 2 pct nital for 6 s.
Figure C.5  SEM Micrograph showing the longitudinal orientation of the region adjacent to the sheared edge in the 1020AT condition of steel C with the nanoindentation array overlaid. Sheared edge is on the left in figure. Steel was etched with 2 pct nital for 6 s.

Figure C.6  SEM Micrograph showing the longitudinal orientation of the region adjacent to the sheared edge in the 990AT condition of steel C with the nanoindentation array overlaid. Sheared edge is on the left in figure. Steel was etched with 2 pct nital for 6 s.
Figure C.7  SEM Micrograph showing the longitudinal orientation of the region adjacent to the sheared edge in the 960TCR condition of steel C with the nanoindentation array overlaid. Sheared edge is on the right in figure. Steel was etched with 2 pct nital for 6 s.

Figure C.8  SEM Micrograph showing the longitudinal orientation of the region adjacent to the sheared edge in the 960AT condition of steel C with the nanoindentation array overlaid. Sheared edge is on the left in figure. Steel was etched with 2 pct nital for 6 s.
APPENDIX D
SHEARED HOLE ANALYSIS

For the grain rotation analysis, SEM micrographs adjacent to the sheared hole were acquired from the longitudinal orientation of the AsR, 960AT, and 960TCR conditions, and an SEM micrograph for the 960AT condition is shown in Figure D.1. In Figure D.1, the 960AT condition was polished to 1 μm diamond using standard metallographic techniques, then etched with nital for approximately 8 s. Nine defined distances, corresponding to 250, 200, 150, 100, 70, 50, 30, 20, and 10 μm from the sheared hole edge, were used to make grain rotation measurements. An enlarged view of the dashed box in Figure D.1 is shown in Figure D.2. The dashed vertical lines in Figure D.2 correspond to the nine defined distances, and the solid lines represent the average grain angle at each of the distances.

![SEM micrograph of the longitudinal orientation (sheet thickness is vertical) adjacent to the sheared hole edge for the 960AT condition. SEM micrograph of 960AT condition was etched with 2 pct nital.](image)

Grain rotation as a function of distance from the sheared hole edge for the three conditions is shown in Figure D.3, and the data are plotted so the data trend emulates the material flow behavior observed in Figure D.2. Table D.1 reports the maximum grain rotation for the three conditions, with both 960AT and 960TCR conditions exhibiting a higher maximum grain rotation compared to the AsR condition. For all three conditions, the maximum grain rotation measurement was located closest to the sheared hole edge. The 960AT and 960TCR conditions exhibited similar grain rotation values, and the grain rotation data for the 960AT condition were omitted from Figure D.3. Compared to the AsR
condition, the 960TCR condition exhibits a smaller SAZ width (defined by the first non-zero angle measurement in Figure D.3), and a larger maximum grain rotation angle (85.1 ° vs. 64.7 °).

Figure D.2 Enlarged view of the region near the sheared hole edge for the 960AT condition (dashed box in Figure D.1). Grain rotation with respect to the original rolling direction (horizontal) at nine defined distances (indicated by dashed vertical lines) from the sheared hole edge were obtained. Measurements were obtained at mid-thickness, and the red solid lines represent the average grain rotation of the microstructure at the different distances. *Color Image – see PDF.*

Figure D.3 Grain rotation (in degrees) with respect to the original rolling direction for the AsR and 960TCR conditions. The 960AT condition exhibited a trend similar to the 960TCR condition, and for sake of clarity, was omitted from this figure.
Table D.1 Maximum Grain Rotation Angle, and Strain for AsR, 960AT, and 960TCR Conditions.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Max. Grain Rotation (degrees)</th>
<th>Shear Strain</th>
</tr>
</thead>
<tbody>
<tr>
<td>AsR</td>
<td>64.7</td>
<td>2.1</td>
</tr>
<tr>
<td>960AT</td>
<td>85.5</td>
<td>12.6</td>
</tr>
<tr>
<td>960TCR</td>
<td>85.1</td>
<td>11.7</td>
</tr>
</tbody>
</table>

Data trends produced by the AsR and 960TCR conditions in Figure D.3 are consistent with the data trends in Figure D.4 from Gibbs [136] when grain rotation as a function of distance from the sheared hole edge was characterized for five steels with UTS values ranging from 580 – 770 MPa. In Figure D.4, the steel with the lowest UTS (L15) corresponded to the largest SAZ depth, and in all cases, an increase in grain rotation was observed as the sheared edge was approached. The direction of increasing values for the axes in Figure D.3 are reversed compared to Figure D.4, but both plots show the same data trend.

![Graph](image)

Figure D.4 Grain rotation as a function of distance from the sheared hole edge for five different steels with UTS ranging from 580 – 770 MPa [136]. The data trends are consistent with the data reported for the AsR, 960AT, and 960TCR conditions in the current study. Figure adapted from [136]. For reference, UTS values for the five steel conditions are reported in the table to the right of the plot.

<table>
<thead>
<tr>
<th>Condition</th>
<th>UTS (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>L15</td>
<td>581</td>
</tr>
<tr>
<td>C19</td>
<td>624</td>
</tr>
<tr>
<td>L24</td>
<td>712</td>
</tr>
<tr>
<td>L31</td>
<td>769</td>
</tr>
<tr>
<td>L38</td>
<td>756</td>
</tr>
</tbody>
</table>

Differences in SAZ depth in Figures D.3 and D.4 were attributed to the difference in work-hardening behavior, and punch/die clearance [144]. Since the AsR, 960AT, and 960TCR conditions kept a narrow tolerance of the punch/die clearance (10 – 12.5 %), the differences in SAZ depth can be attributed to the work-hardening behavior of the materials. Figure D.3 shows the steel with lower work-
hardening behavior (960TCR) to correspond to a smaller SAZ, an observation that was consistent with the work of S. B. Lee [144].

Figure D.5 shows the maximum grain rotation angle for five different steels analyzed by S. B. Lee, and the range of rotation angles was consistent with those observed for the AsR, 960AT, and 960TCR conditions reported in Table D.1. S.B. Lee converted grain rotations to strain values, and determined that strains much larger than the TE were achieved, and attributed the increased ductility of the sheared hole edge to the suppression of damage formation from the stress state generated during shearing, and also stated a compressive stress was present during shearing [144]. Strain values were calculated based on grain rotations for the AsR, 960AT, and 960TCR conditions using the method outlined in [144], and are reported in Table D.1. For all three conditions, strains much higher than the TE were achieved in the SAZ.

![Figure D.5](image1.png)

Figure D.5 Light optical micrographs corresponding to the Low-C condition (top micrograph) and the TRIP780 condition (bottom micrograph), and a plot of the maximum rotation angle for five different steels analyzed by Lee. The maximum rotation angle for both the Low-C (86.1 °) and the TRIP780 (58.2 °) conditions are reported on the plot. Figure adapted from Lee [144].

The DP590 and TRIP590 steels in Figure D.5 were further studied by Lee, and Figure D.6 shows light optical micrographs for the region adjacent to the sheared hole edge for DP590 (upper left), and for TRIP590 (lower left). On both SEM micrographs, a line is superimposed to illustrate the rotation of the grains as the sheared hole edge is approached. The illustrative lines corresponding to grain rotations were further analyzed in the plot (upper right of Figure D.6), where it can be seen that the steel with lower
work-hardening (DP590) exhibited a smaller SAZ, and was interpreted to be one factor in achieving a higher HER [144]. Lee stated a smaller SAZ (less work-hardening) can minimize the amount of damage volume, and result in improved HER.

![Light optical micrographs showing the region adjacent to the sheared hole edge for the DP590 condition (top micrograph), and for the TRIP590 condition (bottom micrograph). In the two micrographs, a black line is superimposed on the microstructures to illustrate the flow of material induced by the shearing process. The black lines used for the DP590 and TRIP590 conditions are also shown in the plot at the top of the figure. The DP590 steel had a higher HER than the TRIP590 steel, and is believed to be due to a smaller SAZ depth, and the decreased work-hardening of the microstructure. Figure adapted from Lee [144].](image)

The void damage in the region adjacent to the sheared hole edge was also quantified for the AsR, 960AT, and 960TCR conditions, and void area pct was chosen as the parameter to characterize the amount of void damage incurred from the shearing process. The entire sheared hole edge (to a depth of 90μm behind the sheared hole edge) was characterized using 13 – 16 SEM micrographs in BSE imaging. An SEM micrograph from the longitudinal orientation of the region adjacent to the sheared hole edge is shown in Figure D.7 for the AsR condition. Fifteen boxes are shown in Figure D.7, and represent the approximate locations where SEM micrographs were acquired for void analysis. Images were acquired using the JEOL 7000F FESEM using the same microscope parameters outlined in Sec. 4.11, and the resulting micrographs were analyzed for void content using the method also outlined in Sec. 4.11 (ImageJ). Figure D.8a shows an example SEM micrograph acquired from the region adjacent to the sheared hole (darkened box in Figure D.7), and Figure D.8b represents the SEM micrograph after being processed with ImageJ. The resulting void area pct values for the three conditions are reported in
Table D.2. A low area pct of voids were observed for the three conditions (less than 1 pct), indicating that high strains can be achieved with little associated void damage. The presence of very few voids is consistent with the interpretation of Lee that the stress state during the hole shearing process has a compressive component, as very little damage is observed [144].

Figure D.7 SEM micrograph of the AsR condition showing the sheared hole edge (right). The 15 boxes represent locations where SEM micrographs were acquired for void analysis. The longitudinal orientation is shown (thickness direction is vertical), and the specimen was etched with 2 pct nital.

Figure D.8 SEM micrograph using BSE imaging of the region adjacent to the sheared hole edge (indicated by the darkened box in Figure D.7) in (a), and the corresponding image produced when the micrograph in (a) was processed with ImageJ in (b). From the image in (b), the void area pct can be determined.
Table D.2  Void Area Pct for AsR, 960AT, and 960TCR Conditions. All Three Conditions Exhibit Void Area Pct Values Below 1 Pct, Indicating Microstructural Damage is Largely Suppressed During Hole Shearing.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Void Area Pct</th>
</tr>
</thead>
<tbody>
<tr>
<td>AsR</td>
<td>0.144</td>
</tr>
<tr>
<td>960AT</td>
<td>0.145</td>
</tr>
<tr>
<td>960TCR</td>
<td>0.091</td>
</tr>
</tbody>
</table>
Figure E.1  Plot of UE as a function of YS/UTS, showing a decrease in UE with less strain-hardening. The solid circle represents the As-R condition, the solid diamonds represent the TCR conditions (equivalent UTS), and the outlined squares represent the AT conditions.