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

By
Mark D. Taylor
A thesis submitted to the Faculty and Board of Trustees of the Colorado School of Mines in partial fulfillment of the requirements for the degree of Master of Science (Materials and Metallurgical Engineering).

Golden, CO
Date 30 January 2013

Signed: __________________________
Mark D. Taylor

Signed: __________________________
Dr. David K. Matlock
Thesis Advisor

Golden, CO
Date 30 January 2013

Signed: __________________________
Dr. Michael J. Kaufman
Professor and Head
Department of Metallurgical and Materials Engineering
ABSTRACT

The materials selected to observe microstructural effects on formability included four 780 MPa strength, and four 980 MPa strength AHSS grades produced with varying processing conditions. The grades were an uncoated DP780, a high-yield DP780, a galvanized DP780, a TRIP780, a galvannealed DP980, a galvanized DP980, an uncoated DP980, and a fine-grained DP980. All AHSS grades were tensile tested to obtain values for ultimate tensile strength, yield strength, percent uniform and total elongation. An analysis was performed to quantify the average grain size of the primary and second-phase constituents, as well as the second-phase volume fraction present in each AHSS grade. Nanoindentation was performed for each AHSS grade to determine the average hardness of the primary and second-phase constituents present. Evolution of microstructural damage in response to deformation was analyzed using a plane strain tensile method developed to impose a localized through-thickness shear fracture. Samples of each AHSS grade were strained to progressively higher percentages of their failure displacement, and microstructural damage was observed using a scanning electron microscope on a metallographic section removed from the localized shear deformation region. Micrographs were analyzed using ImageJ®, and the resulting void percent and number of voids were determined for each test performed. A direct correlation was observed between the number of voids and hardness ratio. The strength of the microstructural constituents affected mechanical properties, suggesting that constituent strength values should be considered when predicting formability limits for higher-strength AHSS grades. Since all AHSS grades experienced some critical number of voids before fracture, it was concluded that suppression of void formation can extend the formability limits to higher strains. After observing a percent failure displacement value of 95%, it was determined that the final stage of fracture (void coalescence) is a rapid event that occurs at failure displacements greater than 95%. Hardness values obtained from nanoindentation were determined to accurately incorporate all strengthening effects present in the constituents. Due to the presence of 5% austenite, TRIP780 and DP980H exhibited different yielding behavior, different tensile stress-strain behavior than the other six DP steels, and had the highest total elongation in their respective strength groups. Austenite appeared to offer increased ductility without sacrificing any other material properties of interest.
TABLE OF CONTENTS

ABSTRACT ........................................................................................................................................... iii

LIST OF FIGURES .................................................................................................................................. vii

LIST OF TABLES .................................................................................................................................... xv

CHAPTER 1: INTRODUCTION .............................................................................................................. 1

CHAPTER 2: LITERATURE REVIEW .................................................................................................... 2

2.1 Introduction to AHSS ..................................................................................................................... 2

2.1.1 DP Steels ........................................................................................................................................ 2

2.1.2 TRIP Steels .................................................................................................................................... 4

2.1.3 Martensitic Steels .......................................................................................................................... 6

2.2 Microstructural Banding in AHSS ................................................................................................. 6

2.3 Limitations of AHSS ...................................................................................................................... 7

2.4 Fracture of AHSS ........................................................................................................................... 8

2.4.1 Physical Fracture Characteristics in AHSS .................................................................................. 9

2.4.2 Microstructural Factors Affecting Fracture .............................................................................. 10

2.4.3 Digital Image Correlation .......................................................................................................... 12

2.4.4 Void Nucleation, Growth, and Coalescence ............................................................................. 13

2.4.5 Void Growth Models .................................................................................................................. 16

2.5 Bending Under Tension Test: Formability Assessment ................................................................. 17

2.6 Plane Strain Tensile Samples ....................................................................................................... 19

2.7 Nanoindentation ............................................................................................................................. 21

2.7.1 Nanoindenter Machine Components ......................................................................................... 21

2.7.2 Data from Nanoindentation ....................................................................................................... 22

2.7.3 Nanoindenter Tip Geometries ................................................................................................. 23

2.7.4 Indenter Tip Calibration ............................................................................................................. 24
2.7.5  P vs. \( \delta \) Curve Analysis..................................................................................25
2.7.6  Indent Spacing.................................................................................................27
2.7.7  Sink-in and Pile-up in Materials.....................................................................29
2.7.8  Indentation Size Effect...................................................................................30
2.7.9  Continuous Stiffness Measurement.................................................................33
2.7.10 Material Pop-in Behavior..............................................................................33
2.7.11 Current Studies in Nanoindentation...............................................................34

CHAPTER 3: EXPERIMENTAL DESIGN........................................................................38
  3.1  Purpose of Project.............................................................................................38
  3.2  Design of Project.............................................................................................39

CHAPTER 4: EXPERIMENTAL METHODS....................................................................40
  4.1  Materials...........................................................................................................40
  4.2  Tensile Testing..................................................................................................41
  4.3  Metallography: Grain Size and MVF...............................................................42
  4.4  Fractography.....................................................................................................44
  4.5  Interrupted Plane Strain Tensile Test...............................................................44
      4.5.1 Plane Strain Tensile Geometry.................................................................44
      4.5.2 Plane Strain Tensile Testing Method..........................................................45
      4.5.3 Void Damage Analysis................................................................................46
  4.6  Nanoindentation...............................................................................................48
  4.7  X-Ray Diffraction.............................................................................................50

CHAPTER 5: RESULTS................................................................................................52
  5.1  Materials...........................................................................................................52
  5.2  XRD..................................................................................................................54
  5.3  Grain Size and MVF..........................................................................................56
  5.4  Nanoindentation...............................................................................................56
Figure 2.1  SEM image using SEI of a DP steel. The darker, larger appearing areas are ferrite, while the smaller, lighter colored areas are martensite [4].

Figure 2.2  Cooling schedule for the production of a cold-rolled DP steel using an intercritical annealing step (a), and cooling schedule for the production of a hot-rolled DP steel on the runout table (b) [1]. Both plots show that, depending on the cooling rate, phases other than martensite can form from the parent austenite.

Figure 2.3  SEM photo taken in SEI of a TRIP microstructure in (a). The large, smooth areas are ferrite (labeled “F”). The lighter-colored islands are austenite (labeled “A”), and the regions comprised of needle-like islands are bainite (labeled “B”). Stress-strain curve for a TRIP and DP steel is shown in (b). This comparison shows the increased ductility that TRIP steels possess.

Figure 2.4  Cooling schedule for a hot-rolled TRIP steel produced from hot rolling in (a). This schedule shows the amount of control on cooling rates and temperatures that is necessary for production. Cooling schedule for a cold-worked TRIP steel using an intercritical anneal in (b) [1].

Figure 2.5  LOM of microstructural banding due to chemical segregation in a DP steel. The dark phase is martensite, and the lighter phase is ferrite. Rolling direction is horizontal. Banded microstructures are very sensitive to certain mechanical properties, such as impact energy absorption [10].

Figure 2.6  Traditional FLD for different grades of AHSS. Formability of AHSS grades are better than predicted for uni-axial deformation, but decreases as the stress state becomes more bi-axial [13].

Figure 2.7  Fracture of a DP sheet specimen deformed in uni-axial tension (a), and fracture of a DP sheet specimen under a bi-axial strain state (b). Thickness is in the vertical direction. In uni-axial tension, there is substantial necking evident, whereas in the bi-axial strain state there is a negligible amount.

Figure 2.8  SEM images using SEI of a DP steel loaded in tension, etched with 2% Nital. Ferrite/martensite interface de-cohesion is shown in (a), and ferrite/ferrite interface de-cohesion in proximity to martensite grains is shown in (b). In both cases, the arrows point to the feature, and the tensile axis is horizontal.

Figure 2.9  SEM photo using SEI of a DP steel showing martensite fracture in (a) [16], and a DP steel with an irregular, fragmented inclusion in (b) [16]. Both steels etched with 2% Nital. In both images, arrows point to the feature, and tensile axis is horizontal.
Figure 2.10  SEM image of a DP steel of void formation at interfaces perpendicular to the loading axis in (a) [16], and void damage at a long, continuous band of martensite oriented parallel to the loading axis in (b). In both, loading axis is vertical [18]...

Figure 2.11  SEM photo of a DP1000 steel showing shear band formation in large ferrite grains, indicated by arrows in (a). Shear bands start and end on martensite grains [31]. SEM photo of strain concentrations in ferrite/pearlite steel highlighted using DIC technique, passing through ferrite in (b), indicated by the white arrow [9]. Bottom photo of (b) shows a shear band propagating through the thinnest section of pearlite. The legend on the far right indicates strain. Color photo, see PDF...

Figure 2.12  DIC analysis of a DP600 steel in the tempered (a) and untempered (b) condition. Etched with 2% Nital. Both photos were taken at a macro strain of 24.3%. In the tempered microstructure, the average region of strain concentration is around 40% and for the untempered microstructure, the average strain concentration is around 51% [28]. The colors on the map correspond to the strain legend shown to the right of each photo. Color, see PDF...

Figure 2.13  X-ray tomography scans of a DP600 steel before tensile testing (a), and immediately before fracture (b). Dimensions of the specimen were 1 x 1 x 1.5 mm. In (a), there are some initial impurities present in the material. In (b), the void density appears higher in the center than on the edges [15]. This technique illustrates the progression of void growth during tensile testing...

Figure 2.14  Graph of void diameter vs. strain in (a). The critical selection of voids grow at an exponential rate with increasing strain [15]. Graph of fraction of porosity vs. distance to center in (b), measured along the width of the sample[15]. Steps 4-6 refer to different amounts of strain, Step 6 being the highest. It can be seen that the porosity rapidly increases from Step 5 to Step 6, correlating well with the exponential increase of the largest voids in the sample shown in (a). Both graphs came from the analysis of the sample shown in Fig. 2.13b. The “RT” label in (a) is discussed in Sec. 2.4.5...

Figure 2.15  Graph of void nucleation as a function of strain in a DP steel for a smooth tensile geometry, and for a 1 mm radius notched sample. The notch induces a triaxial stress state, which is why the nucleation rate of voids is so much faster at equivalent strain to the smooth tensile sample. The two lines labeled “modeling” are mathematical fits to the data [17]...

Figure 2.16  Schematic of the bending under tension test used by A. Hudgins [34]. The two hydraulic cylinders are perpendicular to each other. The vertically oriented cylinder pulls down, putting the sheet metal strip in tension around the roller assembly. This test setup was determined to most accurately represent industrial stamping operations...

Figure 2.17  Photo showing all three modes of observed fractures for the bending under tension tests [12]. Numbers have been designated for each fracture type, shown at the bottom
of each metal strip. As the R/t ratio decreases, fracture types transition from type 1 to type 2, then ultimately to type 3.

Figure 2.18 Failure stress vs. R/t ratio for a 1.0 mm DP600 steel. The individual data points are fitted with a solid line. The location where the solid line transition from sloped to horizontal is the R/t* value, indicated by a dotted vertical line. Adapted from [35].

Figure 2.19 Schematic of a plane strain tensile sample in (a), and a strain evolution analysis of the plane strain tensile sample at different stages of loading in (b) [38]. The major strain is parallel to the loading axis, and the minor strain is across the width of the reduced section shown in (a), perpendicular to the tensile axis. All dimensions in mm in (a).

Figure 2.20 Schematic of GeoB in (a) for the graph of plane strain zone with respect to major strain for different geometries and materials in (b) [38]. (W_H/W_T) is the homogeneous plane strain width divided by the total specimen width. Tensile axis is vertical, and all dimensions in mm.

Figure 2.21 Schematic of the essential components of a nanoindenter [42]. There is a load-applying device, a displacement-measuring device, and a probe tip. Color, see PDF.

Figure 2.22 Traditional P vs. δ curve in (a) [43], and a P vs. δ curve produced using the CSM technique in (b) [46]. Material properties are obtained from the peaks of each serration in (b), giving material properties as a function of depth. The value of “S” in (a) is the stiffness of the material, and is the slope of the upper portion of the unloading curve [43].

Figure 2.23 SEM image of a Berkovich indenter tip. The angle of the tip is 70.3° on each side, making the angle 140.6° between each side [47].

Figure 2.24 A P vs. δ plot of 49 indents on a piece of fused silica to create a tip area function. Loads range from 100 μN to 10,000 μN.

Figure 2.25 Schematic of the different depths of interest using a Berkovich indenter. With a straight-edge indenter, h_c is the true contact depth, and not h, nor h_f.

Figure 2.26 Shear stress contours for a cone-shaped indenter in (a), and a spherical indenter in (b). Note for the spherical indenter, the location of maximum shear stress is not directly adjacent to the contact, but at an approximate z/a value of 0.7 [60].

Figure 2.27 Schematic depicting pile-up (a) and sink-in (b) behavior of materials. In both indents, the indentation depth is equal. The dotted triangle contact area is the area calculated using Eq. 2.4, and the actual contact area (solid line) is different for each indent, showing potential inaccuracies of contact area measurements that can arise if pile-up or sink-in are not accounted for [64].

Figure 2.28 AFM image of a conical indent on fused silica that exhibited pile-up around the edges. This additional contact area can lead to erroneous material property calculations [47].
Figure 2.29  Schematic of the hemispherical volume beneath the indenter that contains the loops of GNDs in (a). A nano-scale illustration of the indenter-material interface is shown in (b). At the nanoscale, there are many small surface steps, the vertical displacement of each step being equal to one burger’s vector, $b$ [51]. The rapid nucleation of the surface-step dislocations contributes to the increased hardness values at low indentation depths…………………………………………………………………………..31

Figure 2.30  Plot of $(H/H_0)^2$ vs. $1/h$ for a polycrystalline copper sample. From Eq. 2.7, it can be seen that the axis are formatted in a way that the data should follow a line with slope $h^*$ [51]. Linearizing the data shows the agreement between the model and experimental data…………………………………………………………………………………………………………………………32

Figure 2.31  Hardness vs. indentation depth plot showing eight separate tests using CSM (Test 001-008), and ten indentation tests in quasi-static (ISO Results). Agreement between the two methods is reasonably well [70]. The plot shows that CSM is capable of accurately extracting material properties using fewer indentation tests. Color photo, see PDF……33

Figure 2.32  A P vs. $\delta$ curve showing a large increase in depth with little to no additional force. The “$\Delta h$” values are termed a pop-in. This type of event is associated with the activation of dislocation sources, or the release of dislocations from a pinning source, such as a grain boundary [72]……………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………
refer to either f/f boundary intersection, f/m boundary intersection, or volume fraction counting. *Color photo, see PDF*.

**Figure 4.4** Schematic of plane strain tensile geometry for 1 mm thick sheet specimen in (a), and a 1.58 mm thick sheet specimen in (b). In both cases, the reduced section between semi-circular notches is 0.6 mm X 0.6 mm, and is held constant for all plane strain geometries. In (a) and (b), tensile axis is vertical.

**Figure 4.5** Photo illustrating the 0.5 inch device coupled with the 1-inch shepic extensometer. The reduction of 0.5 inches allows less elastic displacement to register on the extensometer.

**Figure 4.6** Schematic showing location of the plane of interest. The long, dotted line represents the cut in the direction of the arrows. The plane of interest is parallel to the cut, and the geometry is equivalent to that observed in Figure 4.4.

**Figure 4.7** Illustration describing the 9-photo documentation method. The region of interest can be captured with 9 photos (represented by the black boxes) at a magnification of 1600X in the FESEM.

**Figure 4.8** Illustration of background subtraction using ImageJ® software. A threshold is defined, and regions darker than the threshold remain. The resulting photo in (b) is able to have the voids characterized by their area fraction and the number of separate particles present.

**Figure 4.9** FESEM image using SEI of indents on a polished surface of steel DP980B in (a), and on an etched surface in (b). In (b), most indents were eaten away from the nital etch, so a grid with the indent positions is superimposed on the etched image, revealing the indent locations on the etched surface.

**Figure 4.10** Illustration acceptance/rejection criteria for indents with respect to the microstructure. Indents that land within 1.5 indent diameters of a grain boundary or second-phase constituent are filtered out, while the indents that are fully contained within a phase are categorized as a bulk property value.

**Figure 5.1** FESEM images using SEI of representative microstructures for DP780B in (a), DP780H in (b), DP780I in (c), and TRIP780 in (d). All grades were etched with Nital for approximately 10 seconds.

**Figure 5.2** FESEM images using SEI of representative microstructures for DP980A in (a), DP980B in (b), DP980H in (c), and DP980I in (d). All grades were etched with Nital for approximately 10 seconds.

**Figure 5.3** XRD scans of Intensity vs. 2theta for TRIP780 in (a), and DP780B in (b). In (a), small peaks along the vertical dashed lines are visible, indicating the presence of austenite. Solid vertical lines are BCC peaks, and dashed vertical lines are FCC peaks.
Figure 5.4  Hardness histograms for DP780B in (a), DP780H in (b), DP780I in (c), and TRIP780 in (d). The x-axis in each plot are equivalent………………………………………………57

Figure 5.5  Hardness histograms for DP980A in (a), DP980B in (b), DP980H in (c), and DP980I in (d). The x-axis in each plot are equivalent………………………………………………58

Figure 5.6  Tensile engineering stress-strain curves for the 780MPa and 980MPa steels. In (a), all four grades achieved a UTS above 780MPa. In (b), the 980MPa curves all failed with around 12% elongation, except DP980H, which was 16.3%. All tensile tests were performed with a strain rate of 2.5 *10^{-3} s^{-1} …………………………………………………60

Figure 5.7  Schematic showing the location of the values YS, UTS, pct el (tot), and pct el (uniform) on a representative stress-strain curve. The dashed lines 1, 2, and 3 all have a slope equal to the elastic portion of the stress-strain curve………………………………………………61

Figure 5.8  Schematic of the proposed plane strain tensile design in (a), with a dashed black arrow indicating the plane where fracture is predicted to occur. (b) LOM of one half of a failed DP780 sample using the geometry specified in Fig. 4.4a, etched with Nital. Color photo, see PDF……………………………………………………………………63

Figure 5.9  SEM micrographs using SEI of a bending under tension shear failure of a DP980 steel in (a) [14], and a plane strain tensile shear failure of a DP780 steel in (b). Both images contain ductile void coalescence, and have a slight directionality to the voids, indicating a shear failure. Exact location on fracture surface is unidentified………………………………63

Figure 5.10  Load vs. extensometer displacement (P vs. δ) for DP780B in (a), DP780H in (b), DP780I in (c), and TRIP780 in (d). The solid lines represent the displacement to failure, and the dashed lines represent subsequent displacement percentages………………………………64

Figure 5.11  Plot of P vs. δ curves for DP980A in (a), DP980B in (b), DP980H in (c), and DP980I in (d). In all plots, the solid line represents the displacement to failure, and the dashed lines represent subsequent displacement percentages………………………………65

Figure 5.12  Plot of void pct vs. pct failure displacement for 780 MPa strength materials in (a), and for 980 MPa strength materials in (b). Number of voids vs. pct failure displacement for 780 MPa strength materials in (c), and for 980 MPa strength materials in (d). For most grades, the trend appears to be an exponential increase after 80% failure displacement.67

Figure 5.13  SEM photo using SEI of DP780B tested to 80% failure displacement. Notice the light-colored band of martensite down the center of the sample, and how the band rotates counter-clockwise………………………………………………………………………68

Figure 5.14  SEM micrographs using BSE of initial and 80% failure displacement sample for DP780B in (a) and (b), and DP780H in (c) and (d). For (a)-(d), tensile axis is vertical, and images are representative of region of interest……………………………………..69
Figure 5.15  SEM micrographs using BSE of initial and 80% failure displacement sample for DP980B in (a) and (b), and DP980I in (c) and (d). For (a)-(d), tensile axis is vertical, and images are representative of region of interest ................................................................. 70

Figure 5.16  SEM micrograph using BSE of DP980H sample tested to 95% failure displacement. The image was taken in close proximity to one of the notches (partially seen in bottom right-hand corner. The alignment of voids right below the black line is believed to be the onset of void coalescence ................................................................. 71

Figure 6.1  Plot of # Voids vs. Hardness Ratio in (a). An increasing # Voids can be seen with an increasing Hardness Ratio. Plot of α' Hardness (GPa) vs. second-phase C-content (wt pct) in (b). The weak relationship suggests multiple strengthening mechanisms are present in the second-phase constituents ......................................................... 76

Figure 6.2  SEM image using BSE imaging of the area used for the 9-photo analysis on DP980A in (a), and on DP980I in (b). DP980A and DP980I were tested to 90% failure displacement. Many large voids are evident in (b), where (a) tends to contain only small voids .......... 78

Figure A.1  XRD scans for DP780B in (a), DP780H in (b), DP780I in (c), and TRIP780 in (d) .... 90

Figure A.2  XRD scans for DP980A in (a), DP980B in (b), DP980H in (c), and DP980I in (d) .... 91

Figure A.3  Histograms generated from ferrite hardness data in (a), and second-phase hardness data in (b) for DP780B ................................................................. 92

Figure A.4  Histograms generated from ferrite hardness data in (a), and second-phase hardness data in (b) for DP780H ................................................................. 92

Figure A.5  Histograms generated from ferrite hardness data in (a), and second-phase hardness data in (b) for DP780I ................................................................. 93

Figure A.6  Histograms generated from ferrite hardness data in (a), and second-phase hardness data in (b) for TRIP780 ................................................................. 93

Figure A.7  Histograms generated from ferrite hardness data in (a), and second-phase hardness data in (b) for DP980A ................................................................. 93

Figure A.8  Histograms generated from ferrite hardness data in (a), and second-phase hardness data in (b) for DP980B ................................................................. 94

Figure A.9  Histograms generated from ferrite hardness data in (a), and second-phase hardness data in (b) for DP980H ................................................................. 95

Figure A.10 Histograms generated from ferrite hardness data in (a), and second-phase hardness data in (b) for DP980I ................................................................. 95

Figure A.11 Histograms generated from ferrite hardness data in (a), and second-phase hardness data in (b) for TRIP780 ................................................................. 96

Figure A.11  Tensile curves for 780 MPa strength materials. Each plot has three RD tensile tests, and three TD tensile tests ........................................................................ 96
Figure A.12  Tensile curves for 980 MPa strength materials. Each plot has three RD tensile tests, and three TD tensile tests.................................................................................................................97

Figure A.13  Void Pct and # voids vs. Pct. Failure Displacement for DP780B in (a), DP780H in (b), DP780I in (c ), and TRIP780 in (d).................................................................................................................99

Figure A.14  Void Pct and # voids vs. Pct. Failure Displacement for DP980A in (a), DP980B in (b), DP980H in (c ), and DP980I in (d).................................................................................................................100
LIST OF TABLES

Table 4.1 Composition in wt pct of all As-Received Steel Grades. A Suffix of “A” Denotes a Galvannealed, “I” Denotes a Galvanized, “B” Denotes a Bare Cold-Rolled, and “H” Denotes a High-Yield Grade in the Case of DP780H. “H” for DP980H was an Adopted Designation from the Contributing Company…………………………………………………………40

Table 5.1 Values of α Grain Size, α’ Grain Size and MVF for the Eight AHSS Grades…………56

Table 5.2 Number of Counts Per Phase Using Nanoindentation. In Every Case, the Number of Martensite Counts is Lower Than Ferrite Counts…………………………………………………………59

Table 5.3 Values of Average Ferrite Hardness and Average Second-Phase Hardness for AHSS Grades…………………………………………………………………………………………………..59

Table 5.4 Values of YS, UTS, Pct el (tot), and Pct el (uniform) for the Eight AHSS Grades………61

Table 5.5 Values of Max Stress Reached in the Un-notched Region of Plane Strain Tensile Tests During 90% Failure Displacement. Compared to Yield Stress, Grades That Yielded Are Indicated With a “Y” in The Table…………………………………………………………66

Table 5.6 Values of Void Pct and # Voids for AHSS Grades. Values Were Taken at 90% Failure Displacement……………………………………………………………………………………………72

Table 6.1 Calculated Values of C-content (α’ ) and Hardness Ratio (α’/α) For All Eight AHSS Grades……………………………………………………………………………………………………74

Table 6.2 Comparison of DP780I and DP780B…………………………………………………………76

Table 6.3 Comparison of DP780H and TRIP780…………………………………………………………77

Table 6.4 Comparison of DP780I and TRIP780…………………………………………………………77

Table 6.5 Comparison of DP980A and DP980I…………………………………………………………78

Table 6.6 Comparison of DP980H and DP980I…………………………………………………………79

Table 6.7 Comparison of TRIP780 and DP980H…………………………………………………………79

Table 6.8 Comparison of DP780H and DP780I…………………………………………………………80

Table A.1 Material Properties With One Standard Deviation Quoted in Parenthesis Below Respective Value……………………………………………………………………………………………98
CHAPTER 1
INTRODUCTION

As industrial capability for sheet steel annealing becomes more advanced, new steel grades are being produced that possess both high strength and sufficient elongation. This combination of properties is of interest to the automobile industry, who continually strives for weight reduction and reduced material consumption without sacrificing passenger safety. The premise of an ever-growing collection of advanced high-strength steels (AHSS) is the utilization of metastable second-phase constituents that produce these high-strength properties. The majority of AHSS microstructures depart significantly from the equiaxed morphology observed in low-carbon and mild steels. Accompanying these complex microstructures are complex constituent interactions at the sub-micron range. The most pronounced interactions occur when a significant difference in mechanical/chemical properties between the constituents exists.

One current issue with AHSS is fracture occurring upon industrial stamping operations at strains below what is predicted by traditional Forming Limit Diagrams (FLDs). This observation is attributed to the complex interaction(s) between the constituents present in these AHSS grades. Many microstructural properties, including constituent grain size, grain morphology, martensite volume fraction (MVF), and the hardness of the individual constituents are known to have an effect on the formability of AHSS.

This work aims at replicating fracture comparable to that observed in industrial stamping operations using a uni-axial tensile frame, and relating the initiation of damage, as well as the accumulated damage to key microstructural properties. Of particular interest is the hardness values of the individual constituents, and how the hardness ratio between ferrite and second-phase constituents relates to the progression of microstructural damage. Since these shear fractures are most prevalent amongst higher strength AHSS grades, an in-depth microstructural analysis of four 780MPa Ultimate Tensile Strength (UTS) materials, and four 980MPa UTS materials was performed. In order to retain industrial relevance, all steel grades analyzed are commercially produced. A brief discussion to some techniques used, such as nanoindentation, is discussed in CHAPTER 2. CHAPTER 3 discusses the approach that will be taken to assess microstructural damage, followed by a detailed description of all test methods in CHAPTER 4. A presentation of obtained results is given in CHAPTER 5. A discussion of results is presented in CHAPTER 6, followed by conclusions in CHAPTER 7. Lastly, CHAPTER 8 outlines suggestions for future work to more conclusively determine which microstructural property, or combination of properties, are most detrimental to formability.
CHAPTER 2
LITERATURE REVIEW

In recent years, there have been many strides to successfully incorporate AHSS into automotive applications. The desire for lighter weight, reduced material consumption, less environmental impact, all without sacrificing passenger safety, has motivated sheet-steel companies to produce these superior, complex steel grades. As the knowledge base for these AHSS grades continues to expand, steel grades with specific mechanical properties are being produced that cater to unique applications. As technology in the field of analysis continues to progress, new tests are becoming economically feasible to further analyze the properties of these AHSS grades, adding significant contributions to the fundamental understandings of the complex interactions that arise at the sub-micron level. These complex interactions are attributed to the properties of the different constituents present, including grain size, grain morphology, hardness ratio between differing constituents, and the volume fraction of phases present. This chapter begins with a general introduction of AHSS, followed by the effect of the aforementioned microstructural properties on damage initiation and growth during deformation. Plane strain tensile geometries will then be discussed, and the chapter will conclude with an explanation of the technique and capabilities of nanoindentation.

2.1 Introduction to AHSS

In recent years, the popularity of AHSS has seen substantial growth, evidenced by the significant amount of current research being performed and by the multitude of companies that seek to take advantage of their superior mechanical properties compared to traditional low-carbon and mild steels. Processing techniques employed to produce current AHSS grades are a product of previous research aimed at understanding the fundamental kinetics of phase transformations, alloying effects, and non-equilibrium cooling effects. The most common commercial grades of AHSS currently available are the dual phase (DP), transformation-induced plasticity (TRIP), and martensitic grades. Each grade of steel requires unique combinations of processing and cooling strategies [1]. It is generally accepted that the strength and ductility of AHSS grades are primarily dependent on the volume fraction, distribution, and carbon content of martensite [1, 2].

2.1.1 DP Steels

The name, dual phase, assumes a steel with two distinct constituents. In reality, however, more than two constituents can be present. The term “dual phase” refers to one constituent as always ferrite, and the other constituent to be either bainite or martensite, depending on the cooling rate from the intercritical region [1, 3]. The intercritical region is a temperature range at which austenite and ferrite
coexist. Shown in Fig. 2.1 is a scanning electron microscope (SEM) image of a DP780 steel using secondary electron imaging (SEI). The ferrite grains are the larger, darker appearing grains, while the smaller, lighter grains are martensite. The numbers after “DP” refer to the minimal ultimate tensile strength (UTS) of a material. For instance, a DP980 would be a dual-phase steel with a minimum UTS of 980 MPa.

![SEM image using SEI of a DP steel. The darker, larger appearing areas are ferrite, while the smaller, lighter colored areas are martensite][4].

Figure 2.1

Multiple processing paths exist to produce DP steels. Shown in Fig. 2.2a is a temperature vs. time cooling schedule to produce a DP steel using an intercritical anneal. The initial structure of a DP steel processed in this manner is usually a cold-rolled steel containing ferrite and pearlite. Upon heating to the intercritical region, pearlite transforms to austenite. Austenite preferentially nucleates at ferrite/pearlite boundaries, but has also been observed to nucleate at ferrite/ferrite grain boundaries, as well as at spheroidized carbides [5, 6]. The amount of time the steel is held at the intercritical temperature (“1” in Fig. 2.2a), as well as the temperature itself, determines the fraction of austenite formed. The time dependence of austenite fraction is due mostly to diffusion of interstitial carbon, as well as substitutional atoms, such as manganese [2, 6]. The cooling rate (“2” in Fig. 2.2a) will determine the constituents present in the final microstructure. At a slower rate (i.e. line 2 had a shallower slope), the cooling path could cross into either the “Ferrite”, “Pearlite”, or “Bainite” region shown in Fig. 2.2a, and a portion of the austenitic microstructure would transform to non-martensitic constituents. In Fig. 2.2a, line 2 shows that cooling from the intercritical region crossed the martensite-start temperature (Ms) before intersecting any other region, indicating cooling was rapid enough, enabling austenite to fully transform into martensite.

Another method of producing a DP steel is hot rolling in the fully austenitic region, and subsequent controlled cooling to form the desired ratio of ferrite and martensite, and the cooling schedule for such is shown in Fig 2.2b. After hot rolling, the steel is comprised of severely deformed, fully
austenitic grains. Upon cooling, the path passes through the intercritical region, where phases of ferrite and austenite are stable (“1” in Fig. 2.2b). The resulting microstructure consists of ferrite and austenite (“2” in Fig. 2.2b). Once the temperature decreases below the Ms temperature, the remaining austenite undergoes a martensitic transformation. Figures 2.2a and 2.2b illustrate that cooling rates must be controlled to produce the correct phases in DP steels [6].

![Cooling schedule for the production of a cold-rolled DP steel using an intercritical annealing step (a), and cooling schedule for the production of a hot-rolled DP steel on the runout table (b) [1]. Both plots show that, depending on the cooling rate, phases other than martensite can form from the parent austenite.](image)

2.1.2 TRIP Steels

Traditional low-carbon TRIP steels have a microstructure composed of ferrite (50-55% vol. fraction), bainite (30-35% vol. fraction), and austenite (7-15% vol. fraction) [1]. Compared to DP steels of similar strength, the advantage of TRIP steels is increased ductility, due mainly to the presence of retained austenite. Upon straining, austenite deforms and can transform to martensite, hardening the steel and potentially retarding necking and crack growth. The key to a successful TRIP steel is to produce sufficiently stable austenite so it transforms upon deformation [1]. A SEM photo of a TRIP steel is shown in Fig. 2.3a. The large, smooth regions in Fig. 2.3a are ferrite (labeled “F”) while the lighter islands are austenite (labeled “A”), and the regions comprised of needle-like islands are bainite (labeled “B”). A comparison of TRIP780 and DP780 stress-strain curves in Figure 2.3b shows that the TRIP steel has a higher strain-to-failure than the DP steel. This additional elongation is desirable, as the formability potential increases.
Two different process/cooling schedules for TRIP steel production are shown in Figs. 2.4a and 2.4b. In Fig. 2.4a, a steel sheet is rolled in the fully austenitic region. After rolling, the steel is cooled into the intercritical region, where ferrite forms, rejecting carbon and enriching the remaining austenite. The steel is further cooled into the bainitic region, where the sheet is coiled. In this region, and during subsequent cooling to room temperature, the bainitic transformation from austenite proceeds, further enriching small islands of austenite with carbon. A fraction of the remaining austenite is sufficiently carbon-enriched that it remains untransformed, and is stable at room temperature. In Fig. 2.4b, TRIP steel is produced from an initial cold-rolled microstructure consisting of ferrite and pearlite. In much the same way as the DP steel processing in Fig. 2.2a, the steel is heated into the intercritical region, where ferrite and austenite coexist. Cooling is initially rapid (“1” line) to avoid formation of intercritical ferrite or pearlite. Once in the bainite region, the temperature is held to allow bainite to form from the austenite in the same way as in Fig. 2.4a. One advantage of starting with a cold-rolled, ferrite/pearlite structure is that upon annealing, the stored strain energy causes ferrite recrystallization, which decreases the grain size of the material [5, 6]. The resulting microstructure can potentially be more refined than the processing depicted in Fig 2.4a.

Figure 2.3  
(a) SEM photo taken in SEI of a TRIP microstructure in (a). The large, smooth areas are ferrite (labeled “F”). The lighter-colored islands are austenite (labeled “A”), and the regions comprised of needle-like islands are bainite (labeled “B”). Stress-strain curve for a TRIP and DP steel is shown in (b). This comparison shows the increased ductility that TRIP steels possess [55].
2.1.3 **Martensitic Steels**

Martensitic steel are well-suited for applications where a very high UTS is desired. A martensitic grade is achieved by quenching from the fully austenitic region, attaining a completely martensitic microstructure. In order to produce a martensitic steel, cooling rates must be fast enough to avoid the formation of other phases. The cooling schedule is identical to that seen for the DP steel in Fig. 2.2b, except on cooling, the path would not pass through the ferrite region.

2.2 **Microstructural Banding in AHSS**

Dual phase steels often exhibit microstructure banding, i.e. alternating layers of high- and low-martensite volume fractions, oriented on planes parallel to the rolling direction. Figure 2.5 illustrates microstructure banding using Light Optical Metallography (LOM) of a steel. Banding arises due to chemical segregation at high processing temperatures. Upon solidification, dendrite cores solidify as relatively pure metal while the inter-dendritic regions become enriched in solute, particularly manganese [7, 8]. Once solidified, the solute-lean regions transform to ferrite, while higher alloyed inter-dendritic regions transform to pearlite. Upon cold-rolling, these regions of high and low-solute content become elongated, leading to “bands” with different chemical compositions. With DP steels, after intercritical annealing (ICA), the solute-lean regions remain ferrite, and the solute-rich regions transform from austenite to martensite. Though the effects of chemical banding can be suppressed from a microstructural standpoint [7, 8], the chemical segregation still exists, and only a homogenization heat treatment can truly remedy banding. Homogenization is, in almost every case, uneconomical [7–10]. Certain mechanical
properties, such as yield strength (YS) and UTS, are not significantly affected by banding. Other properties, such as ductility and impact energy, are affected quite significantly [10].

Figure 2.5 LOM of microstructural banding due to chemical segregation in a DP steel. The dark phase is martensite, and the lighter phase is ferrite. Rolling direction is horizontal. Banded microstructures are very sensitive to certain mechanical properties, such as impact energy absorption [10].

2.3 Limitations of AHSS

Many mechanical properties of AHSS grades have been accurately predicted using traditional models and techniques. An example is accurate predictions of UTS using a simple rule-of-mixtures formula based on the volume fraction of phases present [1]. Prediction of other AHSS properties, however, requires methods beyond traditional techniques. Shear fractures have been observed in AHSS grades during industrial stamping operations that are not predicted by traditional forming limit diagrams (FLDs) [11–13]. An example of an FLD is shown in Fig. 2.6. The experimental fracture strains are shown as dashed lines, while the FLD predictions are solid lines. FLDs accurately predict fracture when the steel develops a localized neck before fracture. Under certain imposed stress states, specifically bending around tight curvatures, such as a die, failure was not preceded by a localized neck, and fracture was inaccurately predicted by the FLD [12, 13]. The challenge of accurately predicting shear fractures has currently inhibited the integration of these promising steels into the automotive industry. Examples of sheet steel that exhibit localized necking, and that exhibit unpredicted shear fracture are shown in Figs. 2.7a and 2.7b, respectively. Many explanations have been offered to address this unpredictable behavior, from deformation-induced heating altering strain rate sensitivity [12], to attributing a harder phase (i.e. martensite) to impose greater geometrical constraint on ferrite compared to a softer phase (pearlite) [13]. Experiments have been performed that suggests at small (<2.5 mm) bending radii, microstructural properties dominate failure criterion [12, 14].
Figure 2.6  Traditional FLD for different grades of AHSS. Formability of AHSS grades are better than predicted for uni-axial deformation, but decreases as the stress state becomes more bi-axial [13].

![Figure 2.6](image)

Figure 2.7  Fracture of a DP sheet specimen deformed in uni-axial tension (a), and fracture of a DP sheet specimen under a bi-axial strain state (b). Thickness is in the vertical direction. In uni-axial tension, there is substantial necking evident, whereas in the bi-axial strain state there is a negligible amount [13].

(a)  (b)

Figure 2.7  Fracture of a DP sheet specimen deformed in uni-axial tension (a), and fracture of a DP sheet specimen under a bi-axial strain state (b). Thickness is in the vertical direction. In uni-axial tension, there is substantial necking evident, whereas in the bi-axial strain state there is a negligible amount [13].

2.4  Fracture of AHSS

Many current studies on AHSS fracture have been conducted by numerous researchers [3, 7–10, 12, 13, 15–29]. The primary goal of current research is to develop a fundamental understanding of which microstructural properties play a more significant role in suppression of damage. Damage can be defined as any phenomena that adversely affect AHSS mechanical properties. The damage present in AHSS are
microstructurally-dependent void formation, and void formation at non-metallic inclusions in the matrix [19]. Void formation in AHSS grades is dependent on stress-state, due mostly to the complex microstructural interactions that develop between constituents.

2.4.1 Physical Fracture Characteristics in AHSS

The observed damage at the microstructural level in AHSS grades can be sub-divided into four major categories. The first category, and quite possibly the most observed [3, 15, 16, 18, 20, 23, 27], is de-cohesion at the ferrite/second-phase interface (grain boundary), where a void manifests. Figure 2.8a shows an SEM image of ferrite/martensite decohesion in a plane strain tensile sample of a DP steel. The second fracture category is de-cohesion at the ferrite/ferrite interface. This fracture mode is always associated with a second-phase constituent, such as martensite in close proximity [16, 22]. An example of ferrite/ferrite decohesion in a DP steel is shown in an SEM image in Fig. 2.8b. For Fig. 2.8, tensile axis is horizontal.

![SEM images using SEI of a DP steel loaded in tension, etched with 2% Nital. Ferrite/martensite interface decohesion is shown in (a), and ferrite/ferrite interface decohesion in proximity to martensite grains is shown in (b). In both cases, the arrows point to the feature, and the tensile axis is horizontal [16].](image)

Figure 2.8

The third fracture category is second-phase fracture, and mostly refers to martensite [18]. The entire constituent will fracture, remaining in two or more pieces, leaving a void behind, as shown for a DP steel in Fig. 2.9a. The last category is fracture due to inclusions. This damage mechanism is not often observed in modern “clean” AHSS grades, and is not seen to contribute appreciably to failure [10, 20]. An example of fracture due to inclusions in a DP steel is shown in an SEM image in Fig. 2.9b.
2.4.2 Microstructural Factors Affecting Fracture

Many experiments using an array of techniques have been employed to quantify and assess the microstructural features that affect failure in AHSS grades [3, 9, 12, 15–25, 30]. Some experiments focus on microstructural factors influencing void nucleation and/or shear band formation at the microstructural level. It is well accepted that ferrite/martensite de-cohesion occurs most prevalently when the grain boundary is oriented perpendicular to the loading axis [13, 18], and is illustrated with an SEM photo in Fig. 2.10a in a DP steel. Some portion of the grain boundary will always be perpendicular to the loading axis, and is the first place damage nucleation is expected. Another factor influencing fracture has already been discussed, and is microstructural banding. A DP steel with continuous bands of martensite is one of the most susceptible to damage nucleation [9, 18, 24]. A photo of void damage along a martensite band is shown in an SEM image of a DP steel in Fig 2.10b. De-cohesion first occurs on this long, continuous band of martensite. Continuity between adjacent bands requires deformation in the martensite band to equal that in the adjacent ferrite. Since martensite, especially untempered, is known to deform less than ferrite, significant stress is imparted to the martensite, and martensite will respond by either deforming due to constraints imposed by the ferrite interface, by void nucleation at the ferrite/martensite interface, or by fracture in the martensite band. With coarser-grain, banded microstructures, ferrite becomes less restricted, and thus is able to form localized shear bands that start and end on different martensite islands [15, 17]. Shear bands are illustrated with an SEM image of a DP1000 steel strained locally to 22% in Fig. 2.11a, and are indicated by arrows. When a shear band reaches a martensite band, a very large stress is induced into the martensite, and fracture will occur in the narrowest section of the martensite so the shear band can pass [22, 30]. With a discontinuous banded structure, shear bands in ferrite can develop.
and pass through the gaps without necessitating fracture of martensite [8], as illustrated in Fig. 2.11b. Figure 2.11b is a ferrite/pearlite steel with an overlaid Digital Image Correlation (DIC) strain map, showing strain concentrates in the dark regions (ferrite). The technique of DIC will be discussed in Sec. 2.4.3.

![Image 1](image1.png)

**Figure 2.10** SEM image of a DP steel of void formation at interfaces perpendicular to the loading axis in (a) [16], and void damage at a long, continuous band of martensite oriented parallel to the loading axis in (b). In both, loading axis is vertical. [18]

![Image 2](image2.png)

**Figure 2.11** SEM photo of a DP1000 steel showing shear band formation in large ferrite grains, indicated by arrows in (a). Shear bands start and end on martensite grains [31]. SEM photo of strain concentrations in ferrite/pearlite steel highlighted using DIC technique, passing through ferrite in (b), indicated by the white arrow [9]. Bottom photo of (b) shows a shear band propagating through the thinnest section of pearlite. The legend on the far right indicates strain. *Color photo, see PDF*
The volume fraction, morphology and distribution of second-phase also plays an important role in fracture of AHSS [23, 25, 32]. While some researchers indicate optimum strength and ductility are achieved with a martensite volume fraction (MVF) of 20% [3], others suggest having the highest amount of low-carbon martensite possible is best [24]. A high MVF can embrittle a material unless measures are taken to reduce the martensite strength. One well known method of recovering ductility from martensite is through tempering. Tempering is a heat treatment, usually below 450°C, that relieves some of the carbon super-saturation in the martensite, decreasing the tetragonality of its structure, and imparting better ductility than the untempered state [28]. Tempering decreases the martensite hardness, and correspondingly increases the extent of martensite deformation prior to fracture in AHSS [25]. Void nucleation was seen to initiate at lower imposed strain levels in untempered DP steels compared to tempered DP steels. The failure strain was also observed to be higher for the tempered DP steel, all other microstructural properties remaining constant [13]. In one study, martensite was seen to strain up to 50%, showing that martensite is capable of contributing appreciably to deformation [31].

An example of a microstructure resistant to void nucleation is an equiaxed, fine-grained (< 1μm grain size) DP steel free of inclusions with a moderate hardness ratio between constituents [24]. This combination of properties gives the optimum balance of ductility and strength, owing mostly to the increased plasticity of martensite [23]. With an equiaxed grain morphology, there are fewer geometrical “hot spots” where damage is likely to nucleate. Also the amount of stress imparted to martensite from ferrite is more evenly distributed, retarding high stress concentrations in the microstructure [23]. Further, smaller grains produce more grain boundary area between constituents, making localized events less likely [18]. Ferrite becomes more restricted by having more nearest-neighbors that are martensite, making shear band formation less likely. With a more uniform dispersion of martensite, the dislocation density in ferrite due to the austenite-to-martensite transformation becomes more uniform [8, 26]. When damage occurs in fine-grained materials, ferrite/martensite de-cohesion is the dominant mechanism, whereas with a coarse martensite structure, low-energy martensite cracking becomes more prevalent [3, 18, 27].

2.4.3 Digital Image Correlation

A vivid analysis of microstructural effects on strain partitioning can be performed with Digital Image Correlation (DIC). An example is shown in Fig. 2.11b. DIC is computer software that calculates relative displacement at the microstructural level between constituents (strain partitioning), and converts the displacement to local strain values. A detailed description of the DIC technique is outlined in [33]. DIC techniques on DP steel revealed that the tempered state exhibited a lesser strain gradient between constituents than the untempered state [28]. In both cases, the highest strains existed in the ferrite regions adjacent to martensite, which validates the prevalence of ferrite/martensite de-cohesion [31]. A DIC
analysis depicting a tempered and untempered DP600 steel is shown in Figs. 2.12a and 2.12b, respectively. The colors represent strain values corresponding to the strain legend to the right of each photo. The nature of AHSS with multiple constituents promotes inhomogeneous deformation, causing the observed strain partitioning between phases [16, 18]. The minimization of these strain gradients between different phases is of paramount interest [20].

![Figures 2.12](image)

**Figure 2.12** DIC analysis of a DP600 steel in the tempered (a) and untempered (b) condition. Etched with 2% Nital. Both photos were taken at a macro strain of 24.3%. In the tempered microstructure, the average region of strain concentration is around 40% and for the untempered microstructure, the average strain concentration is around 51% [28]. The colors on the map correspond to the strain legend shown to the right of each photo. *Color, see PDF.*

2.4.4 **Void Nucleation, Growth, and Coalescence**

The microstructural damage progression of void nucleation, growth and coalescence has been documented using the technique of X-Ray tomography [15, 17, 19, 21, 22]. X-Ray tomography uses high energy X-Rays that penetrate through the sample. When a void is present, the absence of material affects the X-Ray absorption, thus imaging a void. X-Ray tomography uses multiple 2-D scans that have been meshed and reconstructed to represent the bulk 3-D structure shown in Fig. 2.13a for an initial state, and just before fracture in Fig. 2.13b [19]. X-ray tomography is capable of completely reconstructing the size, distribution and morphology of voids in a material. The main limitation is the minimum detectable void size. X-Ray tomography has a maximum resolution of about 2 μm x 2 μm, meaning a void of 2 μm diameter is the smallest detectable [15]. This is quite large in terms of void size, suggesting that the voids must experience substantial growth before detection.
Figure 2.13 X-ray tomography scans of a DP600 steel before tensile testing (a), and immediately before fracture (b). Dimensions of the specimen were 1 x 1 x 1.5 mm. In (a), there are some initial impurities present in the material. In (b), the void density appears higher in the center than on the edges [15]. This technique illustrates the progression of void growth during tensile testing.

From the void reconstruction shown in Fig. 2.13, relationships such as the average void diameter can be plotted as a function of strain, as shown in Fig. 2.14a. The material analyzed in Fig. 2.14 is the DP600 steel shown in Fig. 2.13. It is interpreted that high local hydrostatic stresses develop in the nucleation stage, as most voids initiate spherically [16]. With increasing strain, voids tend to lose their spherical nature, and grow in response to the imposed strain gradient [19, 22]. Void growth has been shown to be dependent on the imposed strain gradient [3, 20, 22]. After voids lose their spherical nature, average void diameter is no longer an accurate method of data analysis. As void growth continues, interactions with neighboring voids occur, causing a coalescence of voids [22]. Accompanying Fig. 2.14a, Fig. 2.14b shows the fraction of voids as a function of distance from the specimen center. Steps 4 - 6 refer to different levels of strain at which X-Ray tomograms were taken. Step 6 is a strain greater than Step 5, and so forth. Figure 2.14b shows that void fraction significantly increases as the center is approached. However, non-uniform distribution of voids is disputed by some researchers [20]. The x-direction in Fig. 2.14b is along the width of the sample. Coupling with Fig 2.14a, it can be surmised that the largest voids are located close to the center region of the sample, where a more triaxial stress state exists. The 20 largest voids were tracked in Fig. 2.14a because the larger voids are believed to be more critical to fracture.
The critical selection of voids grow at an exponential rate with increasing strain [15]. Graph of fraction of porosity vs. distance to center in (b), measured along the width of the sample[15]. Steps 4-6 refer to different amounts of strain, Step 6 being the highest. It can be seen that the porosity rapidly increases from Step 5 to Step 6, correlating well with the exponential increase of the largest voids in the sample shown in (a). Both graphs came from the analysis of the sample shown in Fig. 2.13b. The “RT” label in (a) is discussed in Sec. 2.4.5.

The relationship in Fig 2.14a can be misleading since the graph depicts the growth rate for only the 20 largest voids in the material. The average void diameter does not follow this trend. Voids continually nucleate in a material during deformation, and an example of void density ($N$) as a function of strain in a DP steel is shown in Fig. 2.15 for a uni-axial (smooth) sample, and a notched ($R=1$ mm)
sample. Figure 2.15 shows that for an equivalent strain, the notched sample has a higher void density than the uni-axial [17, 20]. Recalling the uni-axial tensile sample in Fig. 2.13b, there was a higher density of voids in the center region compared to the outer region. This was explained by a more triaxial stress state that exists in the center region [3, 16, 31].

![Graph of void nucleation as a function of strain in a DP steel for a smooth tensile geometry, and for a 1 mm radius notched sample. The notch induces a triaxial stress state, which is why the nucleation rate of voids is so much faster at equivalent strain to the smooth tensile sample. The two lines labeled “modeling” are mathematical fits to the data [17].](image)

2.4.5 Void Growth Models

Recent advances have been made in computational modeling of AHSS to predict formability behaviors [22, 29]. The models continue to improve as the database of material properties continues to grow. In order to be able to predict certain behaviors of AHSS, complex computing is becoming increasingly essential. One of the most classical models for void growth is the Rice and Tracey model [22]. A fit to experimental data using the Rice and Tracey model is shown in Fig. 2.14a as the line labeled “RT”. The Rice and Tracey model, in initial form, was meant to characterize the growth of voids in a perfectly plastic matrix. Assuming a perfectly plastic matrix is a significant simplification of material behavior. Further, the model assumed that void nucleation was a one-time event, where Fig. 2.15 shows void nucleation to be a function of strain. Huang et al. modified the Rice and Tracey model to be able to incorporate mechanisms such as void-to-void interactions, as well as strain hardening effects, and has shown promising predictions for AHSS grades [29].
2.5 Bending Under Tension Test: Formability Assessment

This project was initiated in response to the results obtained by Hudgins [14] from bending under tension tests. An illustration of the test setup is shown in Fig 2.16. Two hydraulic cylinders exert force to put a steel sheet in tension over a die (“roller assembly” in Fig. 2.16). Depending on the radius of curvature of the roller assembly, different fracture classifications were observed.

![Diagram of bending under tension test](image)

Figure 2.16 Schematic of the bending under tension test used by A. Hudgins [34]. The two hydraulic cylinders are perpendicular to each other. The vertically oriented cylinder pulls down, putting the sheet metal strip in tension around the roller assembly. This test setup was determined to most accurately represent industrial stamping operations.

This type of test was chosen because it was observed to best represent the stress states experienced under industrial stamping operations [34]. Similar tests have been performed by other researchers to assess shear fracture susceptibility of AHSS grades when addressing forming operations [12, 35]. In all cases, equivalent fracture modes were observed between the different researchers. Figure 2.17 presents a photograph of three failed samples from a bending under tension test, and illustrates the three different modes of fracture observed. The critical parameter that dictated the observed fracture mode was the ratio of bending radius to sheet thickness \(R/t\). With sufficiently large \(R/t\) ratios, tensile failures in the strip were observed away from the die, indicated as type 1 in Fig 2.17 (furthest right). As \(R/t\) ratios decreased, fracture occurred in the material that had been rolled over the die, but still failed in a tensile manner. This type of fracture was defined as type 2 (middle sample). At even smaller bending radii, the sheet response transitioned to a complete shear failure directly adjacent to the die at stresses below what would have normally been expected from tensile data. This type of fracture is defined as type 3. Figure 2.18 illustrates, for a 1.0 mm thick DP600 steel, the effects of \(R/t\) on measured failure stresses for samples like those shown in Fig. 2.17. With an increase in \(R/t\), the stress at fracture increases from the low values associated with type 2 and 3 fracture to a plateau associated with type 1 fracture at locations
away from the die. The minimum R/t value associated with the type 1 plateau is defined as the “critical” R/t* and is used to compare the susceptibility of materials to exhibit the onset of shear fracture. Specifically, a decrease in R/t* corresponds to an increase in formability. The R/t* value is shown as a vertical dotted line in Fig. 2.18.

Figure 2.17 Photo showing all three modes of observed fractures for the bending under tension tests [12]. Numbers have been designated for each fracture type, shown at the bottom of each metal strip. As the R/t ratio decreases, fracture types transition from type 1 to type 2, then ultimately to type 3.

Figure 2.18 Failure stress vs. R/t ratio for a 1.0 mm DP600 steel. The individual data points are fitted with a solid line. The location where the solid line transition from sloped to horizontal is the R/t* value, indicated by a dotted vertical line. Adapted from [35].
In the region of Fig 2.18 to the left of R/t*, fracture types 2 and 3 were observed. Note also in Fig 2.17 that there appears to be very limited necking, or localization in the type 2 or 3 fracture cases. This limited appearance of necking makes these fractures unpredictable [12, 34, 35].

With data such as Fig. 2.18, constitutive models are becoming more accurate in predicting shear fractures in AHSS. An advancement in understanding of these shear fractures in AHSS was performed by J. H. Kim et al. [12], showing that incorporation of deformation-induced heating was able to accurately predict fracture in most cases. In select cases, specifically dealing with higher-strength AHSS, J.H. Kim et al. concluded that microstructural-based fracture mechanisms dominate formability limits, and alluded to the incorporation of damage mechanics into prediction models. Traditionally, microstructural damage mechanisms were seen to play a secondary role in fracture prediction, which is why FLDs are successfully used for lower-strength steels. In order to become more industrially applicable, Desai et al. [35] suggested shifting from a stress-based to a strain-based analysis.

2.6 Plane Strain Tensile Samples

Plane strain tensile tests which utilize special sample geometries tested on standard uni-axial tensile frames are often used to generate material response observed in more complex forming operations, such as the bending under tension tests mentioned in the previous section. Often, as observed in FLD diagrams, or with type 3 fracture in bending under tension tests, plane strain (or near plane strain) conditions identify the strain limits associated with forming and thus are viewed as critical to assess material properties [36–41].

In the case of plane strain tensile tests, a notched region is created to achieve the necessary stress-state, and an example of such geometry is shown in Fig. 2.19a. To achieve a plane strain state in bending under tension tests, a relatively large amount of material is required. With bending under tension frames, the test cannot be interrupted incrementally. With a uni-axial tensile frame, tests can be systematically interrupted to view damage at different deformation levels. Uni-axial tensile frames are more common, and sample preparation costs for specimens are less than for the bending under tension samples. Many researchers have successfully incorporated data obtained from plane strain tension tests into prediction models, such as yield loci prediction of AHSS grades [36, 39]. In plane strain tension tests, model predictions suggest the fracture mode can change with the imposed strain rate [40], so keeping consistent strain rates across all testing is of paramount importance.

The critical dimension for plane strain tensile samples is the effective width-to-height ratio [36]. In certain cases, an un-notched steel sheet specimen can be tested in plane strain if the width is sufficiently large and the height (distance between grips) is relatively small [39]. In most cases, a notch is used to avoid artifacts from the grips. A successful geometry used by P. Flores and M. -S. Aydin is shown in Fig. 2.19a. P. Flores et al. performed a DIC analysis to assess the evolution of strain in the
length- and width-directions of the sample, and concluded that the sample geometry in Fig. 2.19a achieves a plane strain state, shown by a graph of major and minor strain vs. distance across the sample in Fig 2.19b. Stages 1-7 refer to incrementally increasing values of strain.

![Figure 2.19](image)

**Figure 2.19**  Schematic of a plane strain tensile sample in (a), and a strain evolution analysis of the plane strain tensile sample at different stages of loading in (b) [38]. The major strain is parallel to the loading axis, and the minor strain is across the width of the reduced section shown in (a), perpendicular to the tensile axis. All dimensions in mm in (a).

The dramatic increase in strain in Fig. 2.19b towards the sample edges is due to the edge effect, where strain evolves at a much faster rate near the free surface than in the center. This is characteristic of any plane strain tensile sample, regardless if notched or not. The minor strain in the center evolves slightly with increasing strain, but is assumed to be insignificant, and can therefore be ignored [38]. The evolution of this plane strain zone is dependent on the material and geometry. Shown in Fig. 2.20b is a graph of the evolution of a plane strain zone with respect to major strain for geometries shown in Fig. 2.19a and 2.20a. For the geometry shown in Fig. 2.19a, different materials were used for the plot in Fig. 2.20b. In all cases, as the major strain is increased, the plane strain zone on the samples decrease. It can be seen in Fig. 2.20b that the larger width-to-thickness ratio of “Geo B” yields a plane strain zone over a longer range of major strain values. \( W_H \) is the homogeneous plane strain width, and \( W_T \) is the total specimen width. In all cases, the plane strain zone is always concentrated in the center of the width dimension.
Figure 2.20  Schematic of GeoB in (a) for the graph of plane strain zone with respect to major strain for different geometries and materials in (b) [38]. \((W_H/W_T)\) is the homogeneous plane strain width divided by the total specimen width. Tensile axis is vertical, and all dimensions in mm.

2.7  Nanoindentation

In recent years, the field of nanoindentation has expanded greatly due to its capabilities of obtaining properties at the sub-micron scale [41]. Nanoindentation remains a relatively new field; the accepted standard for analysis being published in 1992 [43]. The most commonly sought properties from nanoindentation are material hardness \((H)\) and elastic modulus \((E)\). This section reviews data obtained from a nanoindentation test, the method of analysis to obtain mechanical properties, common tip geometries available, as well as issues that must be considered when analyzing data. Lastly, a few of the more novel research techniques using nanoindentation on steel grades are highlighted.

2.7.1  Nanoindenter Machine Components

Multiple companies manufacture high-resolution nanoindenters, each using a unique configuration. Regardless of configuration, there are essential components that all nanoindenters share. Figure 2.21 illustrates the most essential components all manufactured nanoindenters contain. Loads as small as 1 \(\mu\)N are applied usually either by electromagnetic or electrostatic actuation [42, 44]. Displacement sensing is usually carried out using capacitors. A specific change in capacitance results in a known displacement of the indenter tip. In certain models, the load-application and displacement-measuring device are one in the same [45]. Since load application and displacement sensing are coupled readings, nanoindentation can be performed in either force- or displacement-control. The chosen method is dependent on the type of data being collected.
2.7.2 Data from Nanoindentation

When performing an indent, the load and vertical position of the indenter tip are recorded, creating a load vs. displacement (P vs. δ) curve, shown in Fig. 2.22a. From the unloading segment of this curve, hardness and elastic modulus are calculated. The units on the x-axis of Fig 2.22a are nanometers (nm), and the units on the y-axis are micro-Newton (μN). Another type of P vs. δ curve is created using a technique called Continuous Stiffness Measurement (CSM), discussed later. Using CSM, a seemingly equivalent curve to Fig 2.22a is created, but with CSM, there are multiple serrations in the loading portion of the curve, shown in the magnified circle in Fig. 2.22b. The serrations arise due to a sinusoidal loading path, and the importance of the serrations is to obtain material properties as a function of indentation depth. Depending on the material property of interest, the P vs. δ curve generated by a nanoindenter may exhibit different forms. However, detailing such tests is currently outside the scope of this project. The P vs. δ curves shown in Fig. 2.22 are primarily used for obtaining the material properties of hardness and elastic modulus.
2.7.3 Nanoindenter Tip Geometries

There exist multiple tip geometries for different applications. Three common tip geometries are the cube corner, Berkovich, and spherical indenter. Each tip interacts differently with the substrate and generates different stress-states in the material. Spherical indenters yield accurate material properties that are independent of indentation depth, a desirable characteristic when operating in load-control. With a spherical indenter, greater depths are required to generate a sufficient plastic zone required for accurate property extraction. A fully developed plastic zone exists when any increase in load results in a proportional increase in the contact radius; the mean contact pressure becomes constant [47].

Cube-corner tips are preferred when the material of interest is very thin, and the indentation depth is restricted. Accurate material properties can be obtained at very shallow depths because the tip almost instantly generates a sufficient plastic zone upon contact. Cube-corners can potentially exhibit an Indentation Size Effect (ISE), a depth-dependence on material properties, and can cause significant material pile-up, both leading to an overestimation of hardness. A cube corner indenter has 90° edges, and the apex is pressed into the material. The tip geometry most common to steel research is the Berkovich. A Berkovich is a 3-sided indenter, and is shown in Fig. 2.23. The angle between any two faces is 141°. Berkovich tips are more resistant to pile-up, and can generate a sufficient plastic zone almost instantly upon contact, depending on the sharpness of the tip. Berkovich tips still can experience...
an ISE. The angles of the Berkovich indenter were designed so it would have the same projected area-to-depth ratio as a Vickers indenter tip [47, 48]. Berkovich, cube-corner, and Vicker indenters are all self-similar geometries, meaning regardless of indentation depth, the stress/strain gradients around the indentation will not change [49].

![SEM image of a Berkovich indenter tip.](image)

**Figure 2.23** SEM image of a Berkovich indenter tip. The angle of the tip is 70.3° on each side, making the angle 140.6° between each side [47].

### 2.7.4 Indenter Tip Calibration

Prior to obtaining experimental data, each tip must be calibrated to determine a functional relationship between tip displacement and projected indentation area. For Berkovich indenters, calibrations are typically made on quartz using 49 indents with loads varying incrementally from 100 μN to 10,000 μN. By varying indentation loads on a material with known properties, such as fused quartz, the projected area of the indenter can be accurately determined. For an ideal Berkovich indenter the contact area, \( A \), as a function of contact depth, \( h_c \), is:

\[
A(h_c) = 24.5h_c^2
\]

**Eq. 2.1**

Equation 2.1 is only valid for an infinitely sharp Berkovich tip. In reality, Berkovich tips deviate slightly from Eq. 2.1 at very low indentation depths due to the curvature that exists at the tip apex. Even with the highest-precision manufacturing, tips are finitely sharp, and at some scale, a curvature exists. Tip area functions using multiple coefficients account for this curvature [50]. Tip calibration indents are almost always performed on fused silica because it is not seen to have an ISE [51], and its properties are constant over a range of ambient testing temperatures. An array of indents is performed at varying loads, and the
tip area function is fitted to the known properties of fused silica. Shown in Fig. 2.24 is a P vs. δ plot for 49 separate indents on fused silica with loads ranging from 100 μN to 10,000 μN.

Instead of an ideal Berkovich equation, such as equation 2.1, the data obtained from the P vs. δ curves shown in Fig. 2.24 are fitted to a function that contains 6 fitting parameters to capture the curvature of the tip apex at very low displacements, and is defined as:

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

Eq. 2.2

The constant $C_0$ equals 24.5 (Eq. 2.1), and constants $C_1$-$C_5$ vary to achieve the best fit to the data using a computer-aided analysis [45]. When the contact depth becomes relatively large, the constants $C_1$-$C_5$ become negligible, and $C_o$ dominates. With deeper indents, an ideal Berkovich geometry develops.

2.7.5 P vs. δ Curve Analysis

The most widely accepted method to determine hardness and elastic modulus from P vs. δ curves is the Oliver-Pharr (O-P) method [43, 52–56]. W.C. Oliver and G.M. Pharr pioneered the method used to analyze the majority of nanoindentation data. The fundamental basis for the O-P method included the work of Hertz in the 1880s who analyzed the effects of elastic contact between two spherical surfaces. The work of Sneddon, who derived general relationships among the load, displacement, and contact area
for an indenter whose geometry is a solid of revolution was also of paramount importance for the O-P method [43].

The O-P method measures the slope of the upper portion of the unloading data, termed $S$, or $dP/dh$, shown in Fig 2.22a. Usually, the top 5% of the unloading data is used to determine $S$ [45]. The initial unloading portion of the indent is used for property calculation because it is assumed, through elasticity based analyses, that the initial unloading behavior of the material is characterized by elastic recovery only [42]. Though it is known that a small amount of plastic recovery also occurs upon unloading [48], the magnitude is assumed negligible. This assumption is only valid for metals. Carrying these assumptions to other materials, such as viscoelastic polymers, could yield a Young’s Modulus overestimation up to 70% [57]. The relationship between $S$ and the reduced modulus, $E_r$, is defined as:

$$S = \frac{dP}{dh} = \frac{2}{\sqrt{\pi}} E_r \sqrt{A}$$

Eq. 2.3

where $A$ is the projected area of the indent. Since indenters are finitely rigid, there will always be some degree of compliance of the indenter when indenting into a material, so the value in Eq. 2.3 is termed the reduced modulus, defined as:

$$E_r = \frac{(1 - \nu^2)}{E} + \frac{(1 - \nu_i^2)}{E_i}$$

Eq. 2.4

where $E$ and $\nu$ are the are the Young’s Modulus and Poisson’s ratio for the specimen, and $E_i$ and $\nu_i$ are the same parameters for the indenter. The values for $E_i$ and $\nu_i$ are usually provided by the tip manufacturer.

The contact depth of the indent has been debated in literature [43, 58]. As seen in Fig 2.22a, the contact depth could be taken as $h_f$, the final depth when the load equals zero, $h_{max}$, the depth at maximum load, or the depth value when the $S$ line is extrapolated down to zero load (where dotted line intersects the x-axis). The extrapolation of $S$ was the method chosen by Doerner and Nix’s analysis [58], but did not prove satisfactory for all cases. Oliver and Pharr modified the original Doerner and Nix model by defining the contact depth equation as [43, 45]:

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

Eq. 2.5

where “$\varepsilon$” is a geometric constant, and the right-most quantity is the displacement of the surface at the perimeter of contact. The surface deflection at the contact perimeter depends on indenter geometry [59], and is defined in Fig. 2.25 which illustrates the different depths. Oliver and Pharr realized that the contact depth, $h_c$, most accurately represents the contact area the indenter experiences. The main modification
was to add the geometrical correction factor, \( \varepsilon \), to account for the amount of surface deflection created by each type of indenter. For instance, the value of \( \varepsilon \) for the indents in Fig. 2.24 using a Berkovich indenter was 0.75.

![Diagram of surface profile and indenter](image)

**Figure 2.25** Schematic of the different depths of interest using a Berkovich indenter. With a straight-edge indenter, \( h_c \) is the true contact depth, and not \( h \) nor \( h_f \).

Once the contact depth is established, the projected area of the indent can be obtained using the tip area function described in Eq. 2.2. The hardness value obtained with the O-P method is simply [43]:

\[
H = \frac{P_{\text{max}}}{A} \quad \text{Eq. 2.6}
\]

### 2.7.6 Indent Spacing

When a tip creates an indent in a metallic surface, a plastic zone develops adjacent to the impression. This plastic zone could potentially affect subsequent nanoindentation tests if the generated plastic zones of two adjacent indents overlap. For this reason, it is important to space indents sufficiently far from each other. However, when the average constituent size in an AHSS grade is very small, spacing indents excessively far apart increase the probability of skipping over constituents. Completely skipping constituents becomes an issue when hardness or modulus mapping is desired.

Mathematical simulations have been developed to describe the shape and extent of the generated plastic zone underneath an indenter, and are available for many geometries [60]. As discussed in Ch. 5 by Fischer-Cripps [60], the initial conditions that defined the framework for the mathematical treatments of indentation plastic zones was Hertz’ 1882 work in contact mechanics. His assumptions about two spheres that come into contact, causing localized deformation, were:
I) the displacements and stresses must satisfy the differential equations of equilibrium for elastic bodies, and stresses must vanish at a great distance from the contact surface

II) both bodies are in frictionless contact

III) at the surface, the normal pressure is zero outside, and equal and opposite inside the area of contact.

IV) the distance between surfaces of the two bodies is zero inside, and greater than zero outside the area of contact, and

V) the integral of the pressure distribution within the area of contact with respect to the area of contact gives the force acting between the two bodies [61].

Hertz calculated the pressure distribution that would satisfy the five boundary conditions, but never calculated the stresses at points throughout the interior. Further, literature has established that friction exists between the indenter and specimen, and in certain applications, can have a significant effect [49].

The next advancement in stress field calculations came in 1885 from the work of Boussinesq, as also discussed in Ch. 5 of Fischer-Cripps [60], who realized any description of the indentation stress field associated with any particular indenter begins with the analysis of the condition of a point contact. This “Boussinesq” solution for a point contact allows the stress distribution to be determined for any distribution of pressure within a contact area by the principle of superposition; any contact configuration can be viewed as an appropriate distribution of point loads of varying intensity at the specimen surface. From the works of Hertz and Boussinesq, mathematical simulations have been performed using different indenter tip geometries on flat surfaces. Shown below is a mathematically-generated shear stress contour map for a cone-shaped indenter on a flat surface in Fig. 2.26a, and for a spherical indenter in Fig. 2.26b. In all cases, the mathematically calculated contours are smooth transitions due to compatibility equations at equilibrium [62]. From shear stress contour maps, the required indent spacing for negligible overlap of adjacent indent plastic zones can be determined. The shear stress contours shown in Fig. 2.26a suggests that indents must be spaced approximately three indent diameters apart to have a negligible overlap of plastic zone between indents. Mathematically-generated stress fields associated with other indenter geometries are available in Fischer-Cripps: Elastic Indentation Stress Fields [60]. Since the hydrostatic stress state generated beneath an indenter does not contribute to plastic deformation, the mean contact pressure is greater than that required to initiate yield compared to uniaxial compressive tests [63]. In Fig. 2.26, \( r \) equals the distance from the center of the indenter tip, and \( a \) equals the half-width contact distance.
2.7.7 Sink-in and Pile-up in Materials

Several factors can alter the calculated area in a nanoindentation test. During indentation, non-uniform plastic flow can lead to material being piled-up at indenter edges or can lead to an indentation smaller than calculated by a process referred to as “sink-in”. Figure 2.27 illustrates schematically “pile-up” in Fig. 2.27a, and sink-in in Fig. 2.27b. When pile-up occurs, the actual indention contact area, indicated by the solid line, is greater than the contact area calculated using Eq. 2.2, indicated by the dashed line triangle. Pile-up can produce an overestimation of hardness values up to 50% [47]. In contrast, if sink-in occurs, the actual indention is smaller than the calculated contact area. The extent of pile-up or sink-in is dependent on the tip geometry, and the mechanical properties of the material [64].

Susceptibility of pile-up or sink-in not only differs by material, but also by processing history. For instance, a well-annealed steel sample that exhibits a high strain hardening rate will tend to show far-field plasticity; strain hardening near the indenter tip will cause plastic deformation to occur further away from the contact, causing material to be displaced far away from the indentation [53]. If, however, the steel has been pre-strained (little to no remaining strain-hardening capability), the steel will deform more locally, creating a pile-up of material against the sides of the indenter to accommodate the displaced material [64, 49]. Materials that tend to be susceptible to pile-up have a high Young’s Modulus/Yield Strength (E/Y) ratio [52, 57].
Figure 2.27  Schematic depicting pile-up (a) and sink-in (b) behavior of materials. In both indents, the indention depth is equal. The dotted triangle contact area is the area calculated using Eq. 2.4, and the actual contact area (solid line) is different for each indent, showing potential inaccuracies of contact area measurements that can arise if pile-up or sink-in are not accounted for [64].

An illustration of pile-up that occurred during nanoindentation testing on fused silica with a conical indenter is shown in a photo taken with an atomic force microscope (AFM) in Fig. 2.28. The material around the edge of the indent (indicated by an arrow) that is raised above the specimen surface is the piled-up material.

Figure 2.28  AFM image of a conical indent on fused silica that exhibited pile-up around the edges. This “additional” contact area can lead to erroneous material property calculations [47].

2.7.8  Indentation Size Effect

As mentioned in Sec. 2.7.3, Indentation Size Effect (ISE) refers to a depth-dependence of material properties. The ISE is a function of indenter geometry; a spherical indenter tip, for example, does
not show an ISE [65]. Material properties such as hardness are intrinsic properties, meaning they are related to the material and should not vary with loading conditions [53, 66, 67]. The ISE has been attributed to Geometrically Necessary Dislocations (GNDs) that must be created upon initial contact in order for the material to accommodate the indenter shape [49]. Large strain gradients at initial contact cause this increase in GNDs, yielding a higher hardness value at low indentation depths [51, 54]. Ma and Clarke [66] described the generation of GNDs as Strain Gradient Plasticity (SGP), where the strain gradient is variable. SGP theory assumes the flow stress of metals depends on the density of statistically stored dislocations (SSDs) and GNDs [67]. The SSDs scale with the effective strain, are homogeneous throughout the material, and can increase through pre-strain, or cold-working. The GNDs are proportional to the strain gradient, which becomes significant when the indentation depth is shallow [53, 68]. The gradient increases at increasingly shallow depths because the distances over which the gradient persists are smaller. As indentation depths increase, the strain gradients decrease, and SSDs begin to be generated from the indenter, giving rise to the bulk hardness value. Upon initial contact, the ratio of GNDs to SSDs generated is very high, giving a high hardness value. Upon deeper indentation, the ratio of GNDs to SSDs is negligible, giving the bulk hardness value. The characteristic behavior of the ISE on a hardness vs. indentation depth plot was thoroughly studied by Nix and Gao [51], and is considered to be one of the classical papers when referring to ISE behavioral curves. The Nix and Gao model assumes loops of GNDs equally spaced apart in a hemispherical volume below the indenter, and is illustrated in Fig. 2.29a. The contact radius is $a$, the indentation depth, $h$, and the half-angle of the indenter, $\theta$. Figure 2.29b is a nano-scale illustration of the indenter-material surface contact. At the nano-scale, surface steps exist at the indenter-material surface interface. The vertical displacement of each of the surface steps is equal to one burger’s vector, $b$.

![Figure 2.29](image)

(a) Schematic of the hemispherical volume beneath the indenter that contains the loops of GNDs in (a). A nano-scale illustration of the indenter-material interface is shown in (b). At the nanoscale, there are many small surface steps, the vertical displacement of each step being equal to one burger’s vector, $b$ [51]. The rapid nucleation of the surface-step dislocations contributes to the increased hardness values at low indentation depths.
The radius of this volume is defined by the indenter contact radius. This “storage volume” is thought to be larger by other researchers [65, 69]. In Fe and Ni-Fe alloy systems, Durst et al. [69] concluded the storage volume was dependent on alloying content and Stacking Fault Energy (SFE). By defining the radius of the hemispherical volume and the spacing of the dislocation loops, the GND density can be determined. After the GND density is obtained, the shear stress as a function of dislocation density can be calculated using the Taylor equation. Finally, using the Von Mises flow rule and Tabor’s factor of 3, the equivalent flow stress can be converted to hardness. The final equation Nix and Gao obtained is shown in Equation 2.7

\[
\frac{H}{H_o} = \sqrt{1 + \frac{h^*}{h}}
\]

Eq. 2.7

where \(H_o\) is the intrinsic hardness that arises from predominantly SSDs, \(H\) is the hardness, \(h\) is the depth of penetration, and \(h^*\) is a characteristic length. A more in-depth treatment of the mathematical intricacies and geometrical assumptions is provided elsewhere [51]. The Nix and Gao model fitted to experimental nanoindentation data are shown in Fig. 2.30 for polycrystalline copper. The axes values are constructed in a way that the data should follow a line with slope \(h^*\), and a y-intercept equal to 1. Materials with a higher intrinsic hardness and/or high lattice friction generally have a smaller \(h^*\) and do not depend as strongly on depth [51, 68].

![Figure 2.30](image-url) Depth Dependence of Hardness cold worked polycrystalline Cu

\[
\frac{H}{H_o} = \sqrt{1 + \frac{h^*}{h}}
\]

\(H_o = 0.834 \text{ GPa}\)

\(h^* = 0.464 \mu\text{m}\)

Figure 2.30 Plot of \((H/H_o)^2\) vs. 1/h for a polycrystalline copper sample. From Eq. 2.7, it can be seen that the axis are formatted in a way that the data should follow a line with slope \(h^*\) [51]. Linearizing the data shows the agreement between the model and experimental data.
2.7.9 Continuous Stiffness Measurement

When characterizing material properties as a function of depth, such as the plot shown in Fig. 2.30, it becomes time-consuming to produce many quasi-static indents (Fig. 2.22a). One of the advantages of sub-angstrom depth-sensing resolution and sub-micro Newton resolution is that P vs. δ curves shown in Fig. 2.22b can be produced and analyzed using the constant stiffness measurement (CSM) technique, termed a “CSM curve”. A CSM curve is produced by imposing an oscillating force during indentation [46], which results in partial loading and unloading segments throughout the loading portion of the curve. The resulting displacement range from the oscillating force usually remains below 2 nm [70]. Hardness and elastic modulus values can be calculated at each unloading segment of the curve, giving mechanical properties as a function of indentation depth. Analyzed CSM curves depicting hardness vs. indentation depth are shown in Fig. 2.31 for a 200 nm gold coating on mica. The dark grey (Test 001 – 007) and black (Test 008) lines each represent a single CSM curve. The red dots (ISO Results) represent individual quasi-static indents, obtaining only a single value from the maximum unload segment. The CSM and quasi-static hardness values agree reasonably well, proving that CSM is capable of obtaining equivalent properties with fewer indents, and illustrates material behavior more clearly.

![Image of hardness vs. indentation depth plot](image_url)

*Figure 2.31* Hardness vs. indentation depth plot showing eight separate tests using CSM (Test 001-008), and ten indention tests in quasi-static (ISO Results). Agreement between the two methods is reasonably well [70]. The plot shows that CSM is capable of accurately extracting material properties using fewer indentation tests. *Color photo, see PDF*

2.7.10 Material Pop-in Behavior

An additional feature present in some P vs. δ curves is the appearance of a significant increase in displacement at a constant load, referred to as a “pop-in”. Figure 2.32 illustrates two examples of pop-in behavior, where two P vs. δ curves are superimposed, one with the indent on a grain boundary, and one with the indent at a grain interior. The pop-in segment for the grain boundary indent is labeled “Δh on
GB”, and the pop-in segment for the grain interior indent is labeled “Δh interior” in Fig. 2.32. The appearance of pop-in is attributed to the formation of an avalanche of dislocations being freed from an obstacle, such as a grain boundary, or by the activation of new dislocation sources. A pop-in suggests that “strain is accommodated by an abrupt avalanche of atomic activity, such as might be expected for activation of a dislocation source”, and marks the onset of irreversible flow [71]. In some metal systems, multiple pop-ins are observed, which suggests plastic zone growth promotes further dislocation motion by activation of new sources, or by crossing multiple grain boundaries. With an increasing plastic zone, more slip systems become activated, further adding to the dislocation multiplication [72]. Grain boundaries are effective barriers to slip transfer [72, 73]. When an indent is closer to a G.B., it takes less load to generate sufficient stress required to overcome the critical shear stress, and emit the dislocations into an adjacent grain [68]. Though pop-ins may appear an undesirable feature, they do not have an appreciable effect on the mechanical properties obtained [74, 75].

Figure 2.32  A P vs. δ curve showing a large increase in depth with little to no additional force. The “Δh” values are termed a pop-in. This type of event is associated with the activation of dislocation sources, or the release of dislocations from a pinning source, such as a grain boundary [72].

2.7.11  Current Studies in Nanoindentation

Nanoindentation can yield useful data for steel research, but when coupled with other established analysis techniques, potential information may become more insightful. A hardness vs. indentation depth plot is shown in Fig. 2.33 for different phases of steel. The characteristic shape of these curves is consistent with predictions based on the Nix and Gao model.
Figure 2.33  Plot of hardness vs. indentation depth for different phases of steel. In all cases, the hardness increases with decreasing depth, consistent with the Nix and Gao model [53].

Another property being investigated using nanoindentation by Kim et al. is the strain hardening exponent of different phases of steel [76]. Structural steel was pre-strained to different amounts, followed by subsequent nanoindentation testing. They found that a relationship exists between the strain hardening exponent, $n$, and the characteristic length, $h^*$. Using this method, a tensile test would no longer be necessary to obtain the strain hardening exponent; the value could be obtained from a single indent [76].

In order to determine the mechanisms that contribute to a hardness value, knowledge of the surrounding material must be known. From Sec. 2.7.6, a grain needs to be about 4 times the indent diameter in order for the plastic zone to be fully contained within a grain and yield a bulk property using a Berkovich tip. If a material has an average grain size of 1 μm, the nanoindent diameter must be 250 nm or smaller, meaning an indent depth of 40 nm is the largest that can be made. With indent depths increasing in a polycrystalline material, grain boundary strengthening, as well as second-phase constituents will start to contribute to the hardness value, provided both features exist. Shown in Fig. 2.34a is a schematic of four different indent size classifications. Individual grain properties are obtained at indent “#1”. Classification #2 includes grain boundary strengthening effect, and classification #3 contains both grain boundary and second phase strengthening. Finally, Classification #4 is the macroscopic hardness, averaging all strengthening effects, and is comparable to a Vicker’s test. Figure 2.34b shows the hardness vs. indentation depth for the 4 indent classifications in Fig. 2.34a for a coarse-grained (CG), fine-grained (FG), and very fine-grained (VFG) microstructure. At low depth, the first decrease in hardness is from ISE experienced by Classification “#1”. With progressively deeper indents, Classifications #2 and #3 are achieved, resulting in an increase in hardness [50, 54]. Finally, upon deeper indents, all strengthening effects are being averaged, yielding the macroscopic hardness, $H_o$ [77].
Schematic showing the four different indent size classifications in (a). Classification #1 can extract bulk grain properties, #2 incorporates grain boundary strengthening, #3 incorporates both grain boundary and second-phase constituents, and #4 is the macroscopic hardness. Hardness vs. indentation depth curve in (b) shows the effects of each indent classification on hardness. The initial decrease is due to the ISE, then the increase is due to strengthening effects from grain boundaries and second-phase constituents. The final plateau approaches the macroscopic hardness, $H_o$.

One of the main reasons nanoindentation has become popular in the steel research community is because mechanical properties of some of the smallest microstructural features can be determined. Certain AHSS grades have average grain sizes below 1 μm, making traditional Vicker’s microindentation incapable of obtaining individual phase properties. Though grain boundary strengthening is of interest to some researchers [50, 54], hardness values for individual phases are valuable for applications such as finite element modeling. Using a large-grained, single-phase material seems the most practical method of obtaining bulk properties. However, mechanical properties of an equivalent phase can differ between steel grades due to alloying, or different processing conditions [55].

Recently, experiments conducted by different researchers have been aimed at analyzing the effect of grain orientation on the hardness values [75, 78]. Hardness differences up to 30% have been reported due to crystallographic orientation. This finding was unexpected, as the generated stress-state below an indent is hydrostatic, which activates slip systems in all slip planes, regardless of orientation.

Other research groups aim to convert the P vs. δ data into yield strength. The phase properties obtained using nanoindentation were able to be correlated to the mechanical behavior of the individual constituents in a multi-phase steel [73, 79], and using a rule-of-mixtures formula, very promising stress strain curves were produced, shown in Fig. 2.35.
Figure 2.35  True stress-strain curve obtained by extracting properties from a nanoindentation test (dark line), compared to a true stress-strain curve obtained from a conventional tensile test (thin line). The two curves show good correlation [79].
CHAPTER 3  
EXPERIMENTAL DESIGN

3.1 Purpose of Project

The limitation of traditional FLDs to accurately predict formability limits raised many questions regarding the dominating microstructural effects that arise when considering higher strength AHSS grades. As mentioned in the previous section, technical literature has generated many plausible explanations for the unpredictable formability. Most experiments focused on bending under tension tests where a die radius was varied, and the attenuation of failure stress from the UTS was labeled the critical radius (R/t*). The transition to lower peak loads was also accompanied by a different fracture appearance, termed a shear failure. The researchers who determined these R/t* ratios gave valuable insight for industrial applications, but the fundamental microstructural factors that enable shear fractures are yet to be determined. Since shear fractures are characteristic of the lower peak loads in the bending under tension tests, it is of interest to investigate how their damage nucleates. Mentioned in the previous section, some of the dominating microstructural parameters that affect mechanical properties, particularly strength and ductility, in AHSS grades are the Martensite Volume Fraction (MVF), grain size, and carbon-content of the second-phase constituent. It is believed that, in addition to these microstructural properties, the strength of individual constituents are an important parameter to consider in predicting mechanical behavior. Carbon content of the second-phase constituent has a significant effect on the strength, but many other strengthening mechanisms, such as stacking fault energy, precipitation strengthening, solid-solution strengthening, and post-processing, such as temper-rolling, are also known to have an effect. The advantage of obtaining a hardness value for the individual constituents using nanoindentation is that it will incorporate all strengthening effects present.

In sheet forming tests, such as bending under tension, samples must be deformed until failure occurs, eliminating the possibility of viewing microstructural damage as a function of strain. By creating failure stress vs. R/t* plots such as Fig. 2.18, the failure stress can be interpreted as the culmination of all microstructural property interactions. In order to gain a fundamental understanding of these “interactions”, the effect of each contributing property must be established independent of the others. One method of observing the effect of microstructural properties on the fracture response of AHSS grades is to analyze the damage present at different stages during deformation. This project aims at developing a systematic method to observe microstructural damage in a plane strain state, and correlating the observed damage to grain size, MVF, carbon content of second-phase constituents, and hardness of constituents.
3.2 Design of Project

Eight commercially produced AHSS grades comprise the group of materials used for analysis. The work herein has a focus on retaining industrial relevance by using steel microstructures that are commercially produced. Four 780 MPa strength, and four 980 MPa strength AHSS grades were identified, and obtained from contributing partners: U.S. Steel Corp., Arcelor Mittal, and Pacific Northwest National Laboratory. AHSS grades ranged in thickness from 0.96 mm to 1.58 mm. The AHSS grades were received in the galvanized, galvannealed, or bare conditions. Eight grades were thought to provide a sufficient spectrum of microstructural properties that can be used for comparisons.

The analysis to observe damage nucleation at different stages during deformation was carried out using plane strain tension samples on a uni-axial tensile testing machine. Since shear failures observed in bending under tension tests were attributed to a tri-axial stress state that exists at small R/t values, tension samples were geometrically modified to achieve a similar stress-state. Uni-axial tensile frames are capable of loading to a predetermined amount of displacement, then unloading. Tested samples are then able to be viewed under a microscope to quantify the incurred damage.

Microstructural properties such as MVF, grain size, and carbon-content of second-phase constituents are known to affect the ductility and strength of AHSS. In addition, nanoindentation testing was used to quantify the strength of the different constituents present in the steel grades. The aforementioned microstructural properties affect formability, but the magnitude of influence for each individual property is yet to be determined.
CHAPTER 4
EXPERIMENTAL METHODS

This chapter outlines the techniques used to determine the different properties of interest. First, the AHSS grades for this study will be introduced, followed by a description of the setup used for tensile testing. Next, the method used to quantify grain size and MVF will be discussed. The method used for analyzing fracture surfaces will be introduced, followed by a detailed description of the plane strain tensile testing method used in this study. The technique used for nanoindentation will be detailed, and the text will end with an explanation on the X-ray diffraction (XRD) setup.

4.1 Materials

Eight commercially produced steels comprised the group of materials for analysis. There were four 780 MPa strength steels, and four 980 MPa strength steels. The 780 MPa steels included a galvannealed TRIP780, an uncoated DP780B, a galvanized DP780I, and a DP780H that possessed a high yield stress. The 980 MPa steels included an uncoated DP980B, a galvanized DP980I, a galvannealed DP980A, and an uncoated DP980H that possessed a very high strain-to-failure. Table 4.1 shows the chemical compositions of all 8 AHSS grades, along with their thickness dimension.

<table>
<thead>
<tr>
<th></th>
<th>wt pct</th>
<th>Thickness (mm)</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>DP980A</td>
<td>1.36</td>
<td>0.1</td>
<td>2.27</td>
<td>0.012</td>
<td>0.01</td>
<td>0.26</td>
<td>0.36</td>
<td>0.001</td>
<td></td>
</tr>
<tr>
<td>DP980B</td>
<td>1.00</td>
<td>0.09</td>
<td>2.05</td>
<td>0.62</td>
<td>N/R</td>
<td>0.02</td>
<td>N/R</td>
<td>0.01</td>
<td></td>
</tr>
<tr>
<td>DP980H</td>
<td>0.98</td>
<td>0.15</td>
<td>1.93</td>
<td>0.64</td>
<td>0.04</td>
<td>0.32</td>
<td>0.01</td>
<td>N/R</td>
<td></td>
</tr>
<tr>
<td>DP980I</td>
<td>1.19</td>
<td>0.1</td>
<td>2.1</td>
<td>0.06</td>
<td>0.01</td>
<td>0.2</td>
<td>0.19</td>
<td>0.028</td>
<td></td>
</tr>
<tr>
<td>DP780I</td>
<td>1.03</td>
<td>0.08</td>
<td>2.02</td>
<td>0.004</td>
<td>0.01</td>
<td>0.31</td>
<td>0.29</td>
<td>0.001</td>
<td></td>
</tr>
<tr>
<td>DP780H</td>
<td>1.01</td>
<td>0.09</td>
<td>2.12</td>
<td>0.033</td>
<td>0.01</td>
<td>0.24</td>
<td>0.29</td>
<td>0.001</td>
<td></td>
</tr>
<tr>
<td>DP780B</td>
<td>1.04</td>
<td>0.1</td>
<td>1.74</td>
<td>0.32</td>
<td>N/R</td>
<td>0.02</td>
<td>N/R</td>
<td>N/R</td>
<td></td>
</tr>
<tr>
<td>TRIP780</td>
<td>1.58</td>
<td>0.17</td>
<td>2.1</td>
<td>0.053</td>
<td>0.01</td>
<td>0.12</td>
<td>0.09</td>
<td>0.009</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>wt pct</th>
<th>Nb</th>
<th>V</th>
<th>Al</th>
<th>N</th>
<th>S</th>
<th>P</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>DP980A</td>
<td>0.002</td>
<td>0.001</td>
<td>0.043</td>
<td>0.005</td>
<td>0.006</td>
<td>0.012</td>
<td>0.03</td>
<td></td>
</tr>
<tr>
<td>DP980B</td>
<td>N/R</td>
<td>N/R</td>
<td>0.04</td>
<td>N/R</td>
<td>0.001</td>
<td>0.005</td>
<td>N/R</td>
<td></td>
</tr>
<tr>
<td>DP980H</td>
<td>N/R</td>
<td>N/R</td>
<td>0.03</td>
<td>N/R</td>
<td>0.001</td>
<td>0.01</td>
<td>0.04</td>
<td></td>
</tr>
<tr>
<td>DP980I</td>
<td>0.031</td>
<td>0.001</td>
<td>0.06</td>
<td>0.007</td>
<td>0.003</td>
<td>0.013</td>
<td>0.02</td>
<td></td>
</tr>
<tr>
<td>DP780I</td>
<td>0.003</td>
<td>0.001</td>
<td>0.045</td>
<td>0.004</td>
<td>0.004</td>
<td>0.015</td>
<td>0.02</td>
<td></td>
</tr>
<tr>
<td>DP780H</td>
<td>0.002</td>
<td>0.002</td>
<td>0.041</td>
<td>0.006</td>
<td>0.003</td>
<td>0.006</td>
<td>0.01</td>
<td></td>
</tr>
<tr>
<td>DP780B</td>
<td>N/R</td>
<td>N/R</td>
<td>0.06</td>
<td>N/R</td>
<td>0.005</td>
<td>0.01</td>
<td>N/R</td>
<td></td>
</tr>
<tr>
<td>TRIP780</td>
<td>0.005</td>
<td>0.003</td>
<td>0.73</td>
<td>0.004</td>
<td>0.001</td>
<td>0.014</td>
<td>0.02</td>
<td></td>
</tr>
</tbody>
</table>
4.2 Tensile Testing

Standard ASTM E8 tensile samples were used to quantify the material properties of yield strength (YS), UTS, percent total elongation (pct el tot), and percent uniform elongation (pct el uniform). Six tensile samples of each grade, three with the loading axis parallel to the rolling direction, and three samples with the loading axis perpendicular to the rolling direction (transverse) were tested. Samples were sheared from a sheet, and then the sheared edge was removed by milling. The tolerance held for all dimensions was +/- 0.0015 inches. A schematic of a tensile specimen with the dimensions used for all tests is shown in Fig. 4.1. Samples were tested in the as-received condition.

Tensile samples were tested at a strain rate of $2.5 \times 10^{-3} \text{s}^{-1}$ using a 2-inch gauge length, corresponding to a cross-head speed of 7.62 mm/min. An MTS Alliance RT/100 screw-driven tensile frame was used. A 2-inch shepic extensometer was used for displacement readings, and a 100 Hz data acquisition rate was used on the TestWorks4® software package.

![Figure 4.1](image) Dimensioned schematic of tensile geometry. All dimensions in mm. Samples were sheared from a sheet, then the sheared edges were removed by milling.

Data output from TestWorks4® is in the form of pounds (lbs.) for load and inches for extensometer displacement. In order to convert to stress units in MPa, the following equation was used:

$$\sigma_{\text{eng}} = \frac{P}{A} = \frac{\text{force}(\text{lb}) \times 4.448 \text{ Newtons}}{\text{lb} \times \text{Area}}$$

Eq. 4.1

In Eq. 4.1, $\sigma_{\text{eng}}$ is the engineering stress, $P$ is the load, and $A$ is the cross-sectional area of the gauge section of the tensile sample in mm$^2$. Engineering strain is calculated using the equation:

$$\varepsilon_{\text{eng}} = \frac{\Delta l}{l_o}$$

Eq. 4.2

In Eq. 4.2, $\Delta l$ is the extensometer reading in inches, and $l_o$ is the initial gauge length, in inches.
4.3 Metallography: Grain Size and MVF

In order to obtain grain size and MVF calculations for the eight AHSS grades, microstructural images of the eight AHSS grades were taken with a JEOL-7000F field-emission scanning electron microscope (FESEM). Images were taken in SEI mode using an accelerating voltage of 5 kV. Samples were polished to 1 μm roughness using a diamond solution, then etched with 2 pct. nitric acid (HNO₃), balance ethanol, for 10 seconds. The nitric acid solution will be hereinafter referred to as Nital.

Since different grain morphologies can exist in a steel grade depending on which orientation is observed, five photos in each of three orientations were obtained. The orientations were: 1) Face, 2) longitudinal (L), and 3) transverse (T), and are illustrated in Fig. 4.2. A total of 15 photos were used for grain size and MVF analysis for each AHSS grade. For consistency, photos taken for all orientations and all AHSS grades were at the same magnification, except DP780B. Using the same magnification as the other AHSS grades for DP780B would result in too few grains for analysis.

![Schematic of the three orientations in which SEM images were taken for grain size and MVF analysis. In the schematic, the rolling direction is horizontal.](image)

The method used for grain size analysis was for a two-phase microstructure, and a grain size value for both the primary and secondary constituents were calculated. The method came from Ch. 4.3 of Higginson and Sellars “Worked Examples in Quantitative Metallography” [80]. Three concentric circles with 60 proportionally spaced tick marks were overlaid onto a microstructure. A point count of primary and secondary phase was conducted using the 60 tick marks to provide a local phase volume fraction. In all AHSS grades considered, ferrite is the primary phase, and the second-phase constituent is bainite, martensite, austenite, or a mixture of the three, depending on the AHSS grade. Intersections of the three circles with ferrite/ferrite (f/f) and ferrite/martensite (f/m) boundaries were counted using a click-counter. A second-phase grain size and ferrite grain size were calculated using Equations 4.3 and 4.4, respectively:

\[
L_{α'} = \frac{2 * V_{f-α'} * L}{n_{α'}} \quad \text{Eq. 4.3}
\]

\[
L_{α} = \frac{(1 - V_{f-α'}) * L}{(n_{α} + 0.5n_{α'})} \quad \text{Eq. 4.4}
\]
where $L_{\alpha}$ is the average martensite grain size, $L_{\alpha}$ is the average ferrite grain size, $V_{f-a'}$ is the local MVF, $L$ is the total length of the circles used, $n_{\alpha}$ is the number of ferrite/ferrite counts per measurement field, and $n_{a\alpha}$ is the number of martensite/ferrite counts per measurement field. Twenty-five fields were taken for each AHSS grade, resulting in over 2000 boundary counts and 1500 MVF counts. The software used to create the circular overlays, as well as click-counting was ImageJ®. An illustration of an analyzed field for DP780B Face-orientation is shown in Fig. 4.3. The largest circle contains 30 tick marks, the middle circle, 20, and the smallest circle, 10. The numbers on the colored dots represent different categories of counts, and should be ignored.

Second-phase volume fraction calculation was also performed using ASTM standard E562 with a square grid of 100 points. Since this method was performed by sequential counting of points, potential bias was introduced. With the volume fraction count detailed for the grain size counting, a click-counter was used, where sequential counting was not involved and MVF values calculated in this method were seen to be less biased. The results obtained with both methods agreed reasonably well, and the values from the grain size analysis were chosen to be reported.

Figure 4.3 Illustration of the method in which primary and second-phase constituent grain sizes are calculated, as well as MVF. Photo is an SEM image using SEI of DP780B. Sample was etched with Nital for approximately 10 seconds. The colored dots refer to either $f/f$ boundary intersection, $f/m$ boundary intersection, or volume fraction counting. **Color photo, see PDF.**
4.4 Fractography

Fracture surfaces were analyzed using the FESEM in SEI mode. Imaging fracture surfaces was performed during the initial design of plane strain tensile samples to ensure a fracture surface comparable to the shear fractures in the bending under tension tests were being produced. Interpretation of fracture surfaces was performed with reference to the ASM Handbook Vol. 11, “Failure Analysis and Prevention”.

4.5 Interrupted Plane Strain Tensile Test

This section outlines the procedure for the plane strain tensile testing and analysis. First, the geometry of the plane strain tensile samples will be discussed, followed by the chosen method of testing. Lastly, the method of analysis will be discussed.

4.5.1 Plane Strain Tensile Geometry

A unique geometry was created to introduce a plane strain stress-state into a region of the sample during testing on the MTS Alliance RT/100 tensile frame. The geometry, shown for a 1 mm thick sample in Fig. 4.4a was designed to have a reduced section of 0.6 mm X 0.6 mm between the semi-circular notches. The offset geometry of the notches forced fracture to occur on a plane of maximum shear stress, the dominant fracture mode observed in bending under tension tests at small die radii. Recalling that sheet thicknesses for the eight AHSS grades vary from 0.98 mm to 1.58 mm, geometrical alterations to the semi-circular notches were necessary in order to hold the 0.6 mm X 0.6 mm dimension constant. Shown in Fig. 4.4b is the semi-circular notch geometry for the 1.58 mm thick TRIP780 steel.

Figure 4.4 Schematic of plane strain tensile geometry for 1 mm thick sheet specimen in (a), and a 1.58 mm thick sheet specimen in (b). In both cases, the reduced section between semi-circular notches is 0.6 mm X 0.6 mm, and is held constant for all plane strain geometries. In (a) and (b), tensile axis is vertical.
4.5.2 Plane Strain Tensile Testing Method

The test setup for the plane strain tensile samples involved loading one sample from each AHSS grade to failure on the MTS Alliance RT/100 tensile frame to determine the displacement in the gauge section. The gauge section for these tests was 0.5 inches, and a 1-inch shepic extensometer was used. A special device was used that pinned to the sample 0.5 inches apart, but had extensions on the grips that allowed a 1-inch extensometer to be used. A photographic illustration of the test set-up for plane strain tensile tests is shown in Fig. 4.5. From Fig. 4.4a, the actual gauge section is 0.6 mm, but such a device to measure a 0.6 mm gauge section was not available. The reason a 0.6 mm gauge length is desired is because upon loading, there is elastic displacement within the un-notched region of the tensile sample, contributing to the displacement reading of the extensometer. If the gauge section includes the un-notched region, the elastic deformation of the un-notched region will be superimposed on the plastic deformation of the notched region, potentially complicating interpretation. The elastic portion is able to be filtered out since elastic response is a linear relationship. Further complication arises when stresses in the un-notched region of the sample reach the yield strength, which cannot be accurately filtered out from the plastic deformation of the notched region. For all tests, a cross-head displacement rate of 1 mm/min was used.

Plane strain tension data was kept as load vs. displacement, and after the failure displacement was determined for each AHSS grade, subsequent samples were tested to a percentage of the failure displacement, then unloaded. The failure displacement percentage was determined independently for each steel grade, but the most common percent failure displacements were 60, 70, 80, 90, and 95%.

![Photo illustrating the 0.5 inch device coupled with the 1-inch shepic extensometer. The reduction of 0.5 inches allows less elastic displacement to register on the extensometer.](image)
4.5.3 Void Damage Analysis

The last portion of the interrupted plane strain tensile tests was the analysis of damage that occurred at different failure strain percentages for each AHSS grade. After testing, samples were cross-sectioned at mid-width, corresponding to the schematic in Fig. 4.6. The mid-width notched region was of interest because the middle of the sample is where a plane strain state is most likely prevalent.

![Figure 4.6 Schematic showing location of the plane of interest. The long, dotted line represents the cut in the direction of the arrows. The plane of interest is parallel to the cut, and the geometry is equivalent to that observed in Figure 4.4.](image)

After cross-sectioning, the samples were mounted in bakelite, and then polished down to a 1 μm diamond solution finish. After polishing, the samples were etched with Nital for approximately 10 seconds. Etched samples were put into the FESEM, and the region of interest was documented using a 9-photo method shown in Fig. 4.7. In order to increase contrast between the voids and the steel, BSE imaging mode was used at an accelerating voltage of 20 kV. The 9 photos, represented by the black boxes in Fig. 4.7, capture the region of interest for the plane strain tensile tests. The nine photos ordered along the region where fracture is known to occur gives insight into the nucleation of damage, and behavior of voids as failure is approached. This 9-photo documentation method was performed for each percent failure displacement test for each AHSS grade, and void properties such as the number of voids (# voids), and void percent (void pct.) were recorded. Void pct was calculated by dividing the area of voids in each photo by the total area of the image.

46
Figure 4.7 Illustration describing the 9-photo documentation method. The region of interest can be captured with 9 photos (represented by the black boxes) at a magnification of 1600X in the FESEM.

The photos were then analyzed using ImageJ® by subtracting out the background. This was performed by defining a threshold grayscale value. An image of the analysis method is shown in Fig. 4.8. In Fig. 4.8a, the FESEM photo taken using BSE imaging is shown with voids present (black regions). After defining a grayscale threshold value, pixels brighter and darker than the threshold were converted to a binary white/black, shown in Fig. 4.8b. Using the ImageJ® analysis tools, the area fraction of the voids, along with the number of separate voids were determined.

Figure 4.8 Illustration of background subtraction using ImageJ® software. A threshold is defined, and regions darker than the threshold remain. The resulting photo in (b) is able to have the voids characterized by their area fraction and the number of separate particles present.
4.6 Nanoindentation

In order to quantify the strength of the individual phases present in each AHSS grade, nanoindentation was performed using a Hysitron TI950 Triboindenter. For each AHSS grade, a 15 X 15 array of indents (totaling 225) spaced 2 μm apart was performed using a piezo-automated method. Indents were performed in displacement-control to a depth of 40 nm using a 2 sec. linear load, 2 sec. hold, 2 sec. linear unload loading function. Hardness values were generated by the machine software using the top 5% of the unloading portion of the P vs. δ curve (“S” in Fig. 2.22a).

Sample preparation for nanoindentation included polishing a sample from each AHSS grade to a 1 μm diamond solution finish, then subsequent vibratory polishing using 0.05 μm colloidal silica for approximately 14 hours. Long vibratory polishing is required for nanoindentation because the sample surface cannot have a roughness greater than 60 nm; i.e. the highest peak on the surface cannot be 60 nm higher than the lowest peak. This is especially important when only indenting to a 40 nm depth, as many problems may arise with roughness values in the 30-60 nm range. Vibratory polishing usually achieves a surface roughness around 4 - 7 nm, and slightly etches the surface, making microstructural features distinguishable in polished form. When using mechanical polishing methods, such as diamond solution, deformation is being introduced into the surface of the sample, which can lead to erroneous hardness values. By vibratory polishing for 14 hours, material is chemically removed from the surface, eliminating a significant amount of deformation from the diamond polishing.

After the indents are performed, the 15 X 15 array is imaged using the FESEM in SEI mode with an accelerating voltage of 5 kV, shown in Fig. 4.9a. The indents seen in Fig. 4.9a are on the 0.05 μm polished surface. The slight contrast seen on the surface is a response to the colloidal silica. It is difficult to determine the specific phase associated with each indent shown in Fig. 4.9a, or if an indent was placed too close to a grain boundary, requiring the data to be filtered out. In order to reveal the microstructure, etching the indented region with Nital for approximately 6 seconds was performed, and an example FESEM image using SEI is shown in Fig. 4.9b for the corresponding region shown in Fig. 4.9a. When etching with an acid, such as Nital, the amount of material removed from the surface is usually greater than the depth of the indents, and the indents disappear, making it impossible to identify where each of the 225 indents landed. In order to determine the exact positioning of the indents on the etched surface, both Fig. 4.9a and Fig. 4.9b must be taken at the same magnification in the FESEM. It was discovered that taking multiple images of the same region at high magnification (~45,000X) would deposit a carbon layer dense enough to passivate the local etching response of the surface. This high-magnification method was performed on three corners of the indent array, capturing one indent in each high-mag region. These rectangles can be seen in the etched image in Fig. 4.9b, indicated by arrows.
Figure 4.9 FESEM image using SEI of indents on a polished surface of steel DP980B in (a), and on an etched surface in (b). In (b), most indents were eaten away from the nital etch, so a grid with the indent positions is superimposed on the etched image, revealing the indent locations on the etched surface.

The boxes indicated by arrows in Fig. 4.9b provided a unique reference to determine where the indents are located. Using Fig. 4.9a, the indent positions are marked using a transparent sheet. Overlaying this transparent sheet with the indent positions onto the etched image in Fig. 4.9b taken at the same magnification, the corner indents that reside in the carbon pacified boxes are aligned with the transparent grid, and all indent locations are revealed.
Lastly, each indent must be categorized as a ferrite value, second-phase value, or filtered out due to its location on the etched microstructure. From the discussion on generated plastic zones of indenter tips in Sec. 2.7.6, indents using a Berkovich tip should be spaced at least 3 indent diameters apart since the generated plastic zone extends ~1.5 indent diameters past the edge of the indent. Using this criteria, any indent performed on the AHSS grades that is within 1.5 indent diameters of a grain boundary was filtered out to eliminate potential strengthening contributions of either the grain boundary or the adjacent constituent. Indents categorized as ferrite or second-phase represent indents that are completely contained (plastic zone included) within the constituent, and can be called a bulk value. An illustration of the acceptance/rejection criteria is shown in a zoomed-in view of 6 indents from Fig. 4.9b in Fig. 4.10.

![Illustration of acceptance/rejection criteria for indents](image)

Figure 4.10  Illustration acceptance/rejection criteria for indents with respect to the microstructure. Indents that land within 1.5 indent diameters of a grain boundary or second-phase constituent are filtered out, while the indents that are fully contained within a phase are categorized as a bulk property value.

4.7  X-Ray Diffraction

In order to quantify the amount of austenite present in the eight AHSS grades, X-Ray Diffraction (XRD) was performed. In certain AHSS grades, such as TRIP, austenite is known to exist, and must be considered when comparing mechanical properties of different AHSS grades. Preparation of XRD samples included grinding a 19 mm X 19 mm sample to 1200 grit sandpaper, then chemically removing layers of the surface using a solution of 10 parts water, 10 parts peroxide, and 1 part hydrofluoric acid. The solution was placed in a plastic beaker, which was placed into a larger, water-filled beaker for temperature control. The steel sample was placed in the chemical solution for approximately 5 minutes. Chemical thinning was very important because mechanical grinding methods could potentially transform the austenite at the surface to martensite through deformation, which would give a lower value for austenite than what truly exists. The XRD test was run using a copper target, varying 2-theta from 35° –
105°, a step size of 0.0167°, a tube current of 40 mA, and a generator voltage of 45 kV. Retained austenite volume fraction was calculated using the Rietveld analysis, which integrates the area beneath ferrite and austenite intensity peaks of similar inter-planar spacing.
CHAPTER 5
RESULTS

This chapter presents the results obtained from the experiments outlined in CHAPTER 4. The microstructures of the eight AHSS grades are first introduced. Next, XRD data are presented followed by grain size and MVF calculations. Nanoindentation data are presented along with an explanation of how average values were calculated. Next, tensile data are presented, followed by an explanation of how the reported values were obtained. Fractography results are presented to compare shear fractures from bending under tension tests to plane strain tensile failures. Lastly, data obtained from the interrupted plane strain tensile tests are presented.

5.1 Materials

The microstructures for the four 780 MPa strength materials are shown in Fig. 5.1. All photos are scaled equivalently, and were obtained from the “Face” orientation described in Sec. 4.3.

(a)- DP780B
(b)- DP780H
(c)- DP780I
(d)- TRIP780

Figure 5.1  FESEM images using SEI of representative microstructures for DP780B in (a), DP780H in (b), DP780I in (c), and TRIP780 in (d). All grades were etched with Nital for approximately 10 seconds.
The DP780B has a larger ferrite grain size than the DP780I, DP780H and TRIP780. The larger, smoother appearing grains in DP780B are interpreted as ferrite, and the smaller, rougher appearing grains that exhibit substructure are interpreted as martensite. The rougher regions are believed to be martensite because of the lath-like substructure present within the grains (long features that extend the entire distance of the grain). Both DP780I and DP780H appear to have very similar grain morphologies and their grain size is significantly smaller than DP780B with the appearance of intragranular features. The larger, smoother regions are interpreted as ferrite, and the smaller, rougher regions are believed to be either bainite or martensite, highlighted with black arrows. The DP780I grade is galvanized, meaning the cooling history had to include a hold around 450°C, which makes bainite formation possible. Since both DP780I and DP780H have similar chemical compositions, and their microstructures look very similar, both are interpreted to contain equivalent second phase constituents. The TRIP780 microstructure has rounded microstructural features compared to DP780B, DP780I and DP780H, and are highlighted by black arrows. These rounded features suggest its cooling history was most likely slow in the bainite region (Fig. 2.4). The TRIP780 is galvannealed, meaning the steel experienced a temperature of around 550°C for a period of time, which is supported by the rounded microstructural features. The large, smooth matrix is interpreted as ferrite, and the smaller grains are interpreted as either bainite or martensite. The small, smooth-appearing islands are interpreted as austenite, indicated by an “A” on one of the arrows.

The microstructures of the four 980 MPa strength steels are shown in Fig. 5.2. All images are scaled the same and equivalent to the images shown in Fig. 5.1, and were obtained from the “Face” orientation described in Sec. 4.3. Both DP980I and DP980A contain dispersed second-phase constituents within ferrite grains, decreasing their average grain size. In DP980I and DP980A, the smooth, larger grains are ferrite, and the smaller grains that appear less equiaxed are interpreted as either bainite or martensite. The DP980A was galvannealed, and the DP980I was galvanized, making bainite formation possible for the same reason stated above for DP780I. DP980B has a larger grain size, and a higher volume fraction of second phase present compared to DP980I, DP980A, and DP980H. The smooth areas in DP980B are ferrite, and the rougher areas appear to be martensite, evidenced by the lath-like structure that persists over the majority of the grains, indicated by a black arrow. When etched with Nital, the long white features in the grains are characteristic of the martensitic transformation. Of the 980 MPa grades, DP980H appears to have the most equiaxed microstructure, and has a smaller average grain size. The larger grains are ferrite, and the smaller grains are either bainite, martensite, or potentially austenite. From a qualitative analysis, a range of MVF values and grain size values exist amongst the 980 MPa strength materials. The chemical compositions of the AHSS grades in Figs. 5.1 and 5.2 were presented in Table 4.1.
Figure 5.2  FESEM images using SEI of representative microstructures for DP980A in (a), DP980B in (b), DP980H in (c), and DP980I in (d). All grades were etched with Nital for approximately 10 seconds.

5.2  XRD

XRD scans were performed to determine whether austenite was present in the AHSS grades. Analysis of XRD data yields phase volume fractions to a resolution of approximately +/- 3%. Austenite present in quantities below 3% introduces difficulties in detection, and is reported as zero. Tests were performed using the method outlined in Sec. 4.7. With XRD, the intensity of the peaks shown in Fig. 5.3 were used to calculate an approximate phase volume fraction. The solid vertical lines represent angles where body-centered cubic (BCC) ferrite exists, and the dashed vertical lines represent angles where face-centered cubic (FCC) austenite exists. Analyzing the AHSS grades, DP780B, DP780I, DP780H, DP980A, DP980B, and DP980I contained 0% retained austenite, and all their XRD scans appeared
similar to the DP780B Intensity vs. 2theta plot in Fig. 5.3b. The TRIP780 and DP980H both had approximately 5.0% retained austenite, and their XRD scans appear similar to the TRIP780 Intensity vs. 2theta plot in Fig. 5.3a. At the dashed vertical lines in Fig. 5.3a, small peaks are visible, indicating the presence of austenite. Subtracting out the background signal, the Rietveld analysis, which integrates the curve to determine the area beneath intensity peaks of similar interplanar spacing was used to determine phase volume fraction. Plots of all eight XRD scans are provided in Fig.A.1 for 780 MPa strength materials, and in Fig. A.2 for 980 MPa strength materials.

Figure 5.3  XRD scans of Intensity vs. 2theta for TRIP780 in (a), and DP780B in (b). In (a), small peaks along the vertical dashed lines are visible, indicating the presence of austenite. Solid vertical lines are BCC peaks, and dashed vertical lines are FCC peaks.
The presence of austenite in steels usually imparts better ductility than if austenite were absent. Upon straining, the austenite can undergo a transformation to martensite, increasing the strength of the material, as well as potentially retarding necking. Due to the presence of austenite, DP980H and TRIP780 are expected to have the highest total elongation values amongst the 980 MPa strength materials and 780 MPa strength materials, respectively.

5.3 Grain Size and MVF

Values for ferrite (α) grain size, second-phase (α’) grain size, and MVF were obtained for each AHSS grade using the method outlined in Sec. 4.3, and are shown in Table 5.1. For each grade, 25 determinations of the indicated parameter were made, and the value in Table 5.1 represents the average of the 25 values. The empty columns in Table 5.1 will be filled in as the properties are discussed.

Table 5.1 Values of α Grain Size, α’ Grain Size and MVF for the Eight AHSS Grades.

<table>
<thead>
<tr>
<th>Material</th>
<th>Y.S. (MPa)</th>
<th>UTS (MPa)</th>
<th>pct el (total)</th>
<th>pct el (uniform)</th>
<th>Grain Size α</th>
<th>Grain Size α’</th>
<th>MVF</th>
<th>α’ (GPa)</th>
<th>α (GPa)</th>
<th>Void Pct (90%)</th>
<th># Voids (90%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DP980A</td>
<td>1.25</td>
<td>0.62</td>
<td>24.1</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DP980I</td>
<td>1.56</td>
<td>0.98</td>
<td>26.2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DP980B</td>
<td>1.6</td>
<td>1.71</td>
<td>50</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DP980H</td>
<td>1.18</td>
<td>0.6</td>
<td>26.3</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>TRIP780</td>
<td>1.71</td>
<td>0.81</td>
<td>27.5</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DP780I</td>
<td>1.71</td>
<td>0.87</td>
<td>28.4</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DP780B</td>
<td>3.35</td>
<td>2.15</td>
<td>36.5</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DP780H</td>
<td>1.57</td>
<td>0.87</td>
<td>27.5</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

A smaller grain size and a higher MVF both increase the strength and decrease the ductility of a material. For AHSS grades of comparable strength, the weakening effect of a larger grain size can be offset with a higher MVF, such a comparison being DP980B and DP980A. An interesting observation was that DP780B and DP980B had the highest MVF, and also had the largest grain size in their respective strength classes. From the literature review in CHAPTER 2, decreasing the grain size to increase strength appears to be more desirable than increasing the MVF since the presence of second-phase constituents are what gives rise to strain partitioning and subsequent void nucleation during deformation.

5.4 Nanoindentation

Hardness values for primary and second-phase constituents were obtained for each AHSS grade in the study. There were 225 indents performed on each AHSS grade, and after removing data points influenced by interfaces, discussed in Sec. 4.6, a histogram of the hardness value for each constituent was created. Figure 5.4 shows the obtained hardness histograms for the 780 MPa strength materials, while Fig. 5.5 shows hardness histograms for the 980 MPa strength materials. For ease of comparison, the
x-axis in every histogram is equivalent. One consequence of keeping the x-axis equivalent in all AHSS grades is that data can appear to be skewed, even if the data are inherently normally distributed. Separate hardness histograms are shown in Figs. A.3 – A.10 using an x-axis specific for each constituent in each AHSS grade. For all AHSS grades, histograms divide into two distinct groups; the low hardness values for ferrite and the high hardness values for second-phase. Note that the distribution in measurements for the second-phase data is greater than for ferrite data. The spread in second-phase data is due partly to the potential presence of more than one second-phase constituent, such as the presence of austenite in DP980H and TRIP780.

![HardnessHistograms](image)

(a) - DP780B  
(b) - DP780H  
(c) - DP780I  
(d) - TRIP780

Figure 5.4 Hardness histograms for DP780B in (a), DP780H in (b), DP780I in (c), and TRIP780 in (d). The x-axis in each plot are equivalent.
Finer-grained materials had fewer phase property measurements because more data points were removed due to proximity to interfaces. For every AHSS grade, the number of ferrite data points was always greater than the number of second-phase data points. One reason for this is because the MVF seen in Table 5.1 is 50 or less for every AHSS grade. Second, the morphologies of the second-phase constituents, as qualitatively observed in Figs. 5.1 and 5.2, included significant amounts of non-equiaxed features, resulting in second-phase constituents with high grain boundary-to-area ratios. With a higher MVF, such as DP980B, the number of second-phase data points was higher, as shown in Table 5.2, which summarizes the total number of usable data points out of the 225 performed for each steel grade.

![Hardness histograms for DP980A in (a), DP980B in (b), DP980H in (c), and DP980I in (d). The x-axis in each plot are equivalent.](image)

Figure 5.5
From each intensity peak shown in Figs. 5.4 and 5.5, an average value was obtained and reported in Table 5.3. If the two hardness intensity peaks overlapped, the analysis to obtain average values would have required fitting the data to a double-Gaussian function. No overlap existed in the ferrite and second-phase hardness values in the histograms.

Table 5.2 Number of Counts Per Phase Using Nanoindentation. In Every Case, the Number of Martensite Counts is Lower Than Ferrite Counts.

<table>
<thead>
<tr>
<th>Material</th>
<th>Ferrite Counts</th>
<th>Second-Phase Counts</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>DP980A</td>
<td>55</td>
<td>6</td>
<td>61</td>
</tr>
<tr>
<td>DP980I</td>
<td>83</td>
<td>17</td>
<td>100</td>
</tr>
<tr>
<td>DP980B</td>
<td>66</td>
<td>33</td>
<td>99</td>
</tr>
<tr>
<td>DP980H</td>
<td>76</td>
<td>10</td>
<td>86</td>
</tr>
<tr>
<td>TRIP780</td>
<td>82</td>
<td>20</td>
<td>102</td>
</tr>
<tr>
<td>DP780I</td>
<td>67</td>
<td>11</td>
<td>78</td>
</tr>
<tr>
<td>DP780B</td>
<td>99</td>
<td>22</td>
<td>121</td>
</tr>
<tr>
<td>DP780H</td>
<td>61</td>
<td>12</td>
<td>73</td>
</tr>
</tbody>
</table>

Table 5.3 Values of Average Ferrite Hardness and Average Second-Phase Hardness for AHSS Grades.

<table>
<thead>
<tr>
<th>Material</th>
<th>Y.S. (MPa)</th>
<th>UTS (MPa)</th>
<th>pct el (total)</th>
<th>pct el (uniform)</th>
<th>Grain Size $a$</th>
<th>Grain Size $a'$</th>
<th>MVF</th>
<th>$a'$ (GPa)</th>
<th>$a$ (GPa)</th>
<th>Void Pct (90%)</th>
<th># Voids (90%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DP980A</td>
<td>1.25</td>
<td>0.62</td>
<td>26.1</td>
<td>9.7</td>
<td>3.6</td>
<td>3.6</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DP980I</td>
<td>1.56</td>
<td>0.98</td>
<td>26.2</td>
<td>8.3</td>
<td>3.7</td>
<td>3.7</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DP980B</td>
<td>1.6</td>
<td>1.71</td>
<td>50</td>
<td>7.9</td>
<td>3.8</td>
<td>3.8</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DP980H</td>
<td>1.18</td>
<td>0.6</td>
<td>26.3</td>
<td>8.2</td>
<td>3.3</td>
<td>3.3</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>TRIP780</td>
<td>1.71</td>
<td>0.81</td>
<td>27.5</td>
<td>9.1</td>
<td>3.4</td>
<td>3.4</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DP780I</td>
<td>1.71</td>
<td>0.87</td>
<td>28.4</td>
<td>8.3</td>
<td>3</td>
<td>3</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DP780B</td>
<td>3.35</td>
<td>2.15</td>
<td>36.5</td>
<td>7.2</td>
<td>3.1</td>
<td>3.1</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DP780H</td>
<td>1.57</td>
<td>0.87</td>
<td>27.5</td>
<td>9.1</td>
<td>3.3</td>
<td>3.3</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The results from Table 5.3 show that the average ferrite hardness values for the 780 MPa strength materials are slightly lower (3.2 GPa) than the average ferrite hardness values for the 980 MPa materials (3.6 GPa). Since ferrite is the matrix phase, and is known to accommodate the majority of strain during loading, it is believed that increasing the strength of the “ductile” matrix would increase the overall strength of the AHSS grade more effectively than continually increasing the strength of the second-phase constituent. It is interesting to note that some 780 MPa strength materials have a higher second-phase (9.1 GPa) and ferrite hardness (3.4 GPa) value than some 980 MPa strength materials, such as comparing TRIP780 to DP980H (8.2 and 3.3 GPa, respectively). The ratio between second-phase hardness and ferrite hardness is believed to correlate to the degree of strain partitioning in a microstructure.
5.5 Tensile Testing

Figure 5.6 shows select engineering stress vs. strain tensile curves for each AHSS grade with the tensile axis parallel to the rolling direction (RD). For clarity, the four 780 MPa strength grades are plotted in Fig. 5.6a, and the four 980 MPa strength grades are plotted in Fig. 5.6b using equivalent axes. Three tensile tests with the tensile axis parallel to the RD, and three with the tensile axis parallel to the transverse direction (TD) were performed for each AHSS grade. For all AHSS grades tested, the three RD tensile curves were very similar, and the three TD tensile curves were very similar. The six engineering stress vs. strain tensile curves for each AHSS grade are shown in Fig. A.11 for 780 MPa strength materials, and in Fig. A.12 for the 980 MPa strength materials. The selected tensile curves shown in Fig. 5.6 are indicated by solid curves in Figs. A.11 and A.12 for all AHSS grades. The properties reported in Table 5.4 for YS, UTS, pct el (tot), and pct el (uniform) represent averages of the data obtained from the three tensile tests performed in the RD for each AHSS grade. Only in certain AHSS grades did a difference in mechanical properties exist between average RD and TD tensile tests, and when present, the difference was less than 2%.

The YS values were obtained using a 0.2 pct offset, as illustrated by the schematic stress-strain curve in Fig. 5.7. The stress value where the dashed line labeled “1” in Fig. 5.7 intersects the stress-strain curve is the YS. Line “1” intersects the x-axis at an engineering strain value of 0.002. The UTS is the highest stress attained during the tensile test, and is labeled in Fig 5.7. The pct el (uniform) was obtained by the intersection of the dashed line labeled “2” with the x-axis. Line “2” intersects the UTS value.
pct el (tot) value was obtained by the intersection of the dashed line labeled “3” with the x-axis. Line “3” intersects the last data point before fracture. All three dashed lines have a slope equal to the elastic portion of the stress-strain curve in Fig. 5.7.

Table 5.4  Values of YS, UTS, Pct el (tot), and Pct el (uniform) for the Eight AHSS Grades.

<table>
<thead>
<tr>
<th>Material</th>
<th>Y.S. (MPa)</th>
<th>UTS (MPa)</th>
<th>pct el (total)</th>
<th>pct el (uniform)</th>
<th>Grain Size α</th>
<th>Grain Size α'</th>
<th>MVF</th>
<th>α (GPa)</th>
<th>α' (GPa)</th>
<th>Void Pct (90%)</th>
<th># Voids (90%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DP980A</td>
<td>680</td>
<td>965</td>
<td>12.1</td>
<td>7</td>
<td>1.25</td>
<td>0.62</td>
<td>24.1</td>
<td>9.7</td>
<td>3.6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DP980I</td>
<td>726</td>
<td>971</td>
<td>11.7</td>
<td>5.8</td>
<td>1.56</td>
<td>0.98</td>
<td>26.2</td>
<td>8.3</td>
<td>3.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DP980B</td>
<td>655</td>
<td>1013</td>
<td>12.5</td>
<td>7.4</td>
<td>1.6</td>
<td>1.71</td>
<td>50</td>
<td>7.9</td>
<td>3.8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DP980H</td>
<td>620</td>
<td>969</td>
<td>16.3</td>
<td>12.2</td>
<td>1.18</td>
<td>0.6</td>
<td>26.3</td>
<td>8.2</td>
<td>3.3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>TRIP780</td>
<td>519</td>
<td>853</td>
<td>18.2</td>
<td>13</td>
<td>1.71</td>
<td>0.81</td>
<td>27.5</td>
<td>9.1</td>
<td>3.4</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DP780I</td>
<td>491</td>
<td>816</td>
<td>17.8</td>
<td>11.3</td>
<td>1.71</td>
<td>0.87</td>
<td>28.4</td>
<td>8.3</td>
<td>3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DP780B</td>
<td>491</td>
<td>826</td>
<td>17.6</td>
<td>11.2</td>
<td>3.35</td>
<td>2.15</td>
<td>36.5</td>
<td>7.2</td>
<td>3.1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DP780H</td>
<td>608</td>
<td>862</td>
<td>13.4</td>
<td>8.2</td>
<td>1.57</td>
<td>0.87</td>
<td>27.5</td>
<td>9.1</td>
<td>3.3</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 5.7  Schematic showing the location of the values YS, UTS, pct el (tot), and pct el (uniform) on a representative stress-strain curve. The dashed lines 1, 2, and 3 all have a slope equal to the elastic portion of the stress-strain curve.

Many observations and trends can be made from the tensile curves shown in Fig. 5.6. All 780 MPa strength materials achieved a UTS above 780 MPa, and had YS values that ranged from 490 – 610 MPa. The TRIP780 had the highest pct el (tot) and the highest pct el (uniform), thought to be due to austenite transformation to martensite once the YS is exceeded. DP780H had the lowest pct total elongation, but exhibited the highest UTS and YS. One explanation for the higher strength and lower
ductility of DP780H is the presence of the highest second-phase and ferrite hardness of the 780 MPa grades. Further, DP780H experienced temper-rolling, meaning a light post-processing deformation pass was performed to increase dislocation density in the microstructure. Steel grades DP780I and DP780B had very different MVF, grain size, and constituent hardness values, but achieved almost identical tensile properties, suggesting that equivalent tensile properties are able to be produced with different combinations of MVF, grain size, and constituent hardness values.

The tensile curves shown for the 980 MPa materials in Fig. 5.6 all have higher UTS values than the 780 MPa strength materials, but only DP980B achieved a UTS greater than 980 MPa. The DP980B grade also contained the highest MVF. The DP980H had the highest pct el (tot). The work hardening of the DP980H was also different than for DP980B, DP980I, and DP980A; a distinct inflection upon yielding is observed. This inflection is termed incipient yielding, and is most likely due to either the response of metastable austenite to deformation, or to aging. Upon straining past the YS, the ductile austenite transformed to martensite, which strengthened the material upon further straining. The DP980B, DP980A, and DP980I grades all exhibited continuous yielding, and combinations of similar UTS and pct el (tot), but exhibited different grain sizes, MVFs, and phase hardness values, further suggesting that similar tensile properties can be achieved through different combinations of MVF, grain size, and phase hardness values. The magnitude of influence MVF, grain size, and phase hardness values have on strength and ductility is of paramount interest.

An uncertainty analysis was performed for the values reported in Table 5.4 in Table A.1. A number in parentheses representing one standard deviation is shown below each reported value. There were no large standard deviations for any of the data sets, indicating the methods chosen for obtaining material properties are relatively reproducible. The number of data points used for each property calculation is detailed in CHAPTER 4.

5.6 Fractography

In order to verify the proposed plane strain tensile design, the notched geometry shown in Fig. 4.4 was tested to failure, and the failure is shown in a LOM of the sample cross-sectioned at fracture in Fig. 5.8b for a DP780 steel. Failure is observed to occur on a through-thickness plane oriented 45° with respect to the tensile axis without observable localized necking. The failure geometry of the sample in Fig. 5.8b, and the predicted failure geometry in Fig. 5.8a (plane indicated by dashed arrow) are equivalent, confirming the success of the proposed plane strain tensile design to induce shear fracture. Shear stresses are at a maximum on a plane 45° from the tensile axis during uni-axial loading, and is the location that a shear fracture is expected to manifest.
To assess whether the plane strain tensile fracture surface was comparable to shear failures that occurred in bending under tension tests, the fracture surface of a plane strain tensile geometry in Fig. 5.8b was compared to a shear fracture surface of a DP980 steel generated during a bending under tension test. Comparison of the bending under tension shear fracture [34] to the shear fracture produced using the plane strain tensile test is shown in SEM photographs using SEI in Figs. 5.9a and 5.9b, respectively. Both fracture surfaces exhibit ductile void coalescence, and the voids have a slight directionality associated with them, indicating a shear failure. The features in Fig. 5.9b are representative of the entire fracture surface, and the photo in Fig. 5.9a was taken at an unknown location on the shear fracture surface.

Figure 5.8 Schematic of the proposed plane strain tensile design in (a), with a dashed black arrow indicating the plane where fracture is predicted to occur on. (b) LOM of one half of a failed DP780 sample using the geometry specified in Fig. 4.4a, etched with Nital. *Color photo, see PDF.*

Figure 5.9 SEM micrographs using SEI of a bending under tension shear failure of a DP980 steel in (a) [14], and a plane strain tensile shear failure of a DP780 steel in (b). Both images contain ductile void coalescence, and have a slight directionality to the voids, indicating a shear failure. Exact location on fracture surface is unidentified.
5.7 Interrupted Plane Strain Tensile Test

For each AHSS grade, one plane strain tensile sample was tested to determine the measured displacement at failure using a 12.7 mm (0.5-inch) gauge length with an extensometer. Then, subsequent tests were performed to a percentage of the failure displacement. Figures 5.10 and 5.11 show the P vs. δ curves for the 780 MPa grades and 980 MPa grades, respectively. In each plot, the solid line is the sample that was tested to failure, and the dashed lines are subsequent tests performed to the indicated percentages of the failure displacement. In some cases, the “failure” curve appears step-wise, a consequence of selecting a low data acquisition rate on the computer control system. In all cases, the curves overlay on each other reasonably well. In order to have curves perfectly overlay, the specimen dimensions must be equivalent with micron-precision, which is an impractical expectation. The slight deviations in the P vs. δ curves exist because of the machining variation between samples, as well as the inherent microstructural variability that exists between samples.

![Graphs showing P vs. δ curves for different grades](image)

Figure 5.10 Load vs. extensometer displacement (P vs. δ) for DP780B in (a), DP780H in (b), DP780I in (c), and TRIP780 in (d). The solid lines represent the displacement to failure, and the dashed lines represent subsequent displacement percentages.
Figure 5.11  Plot of P vs. δ curves for DP980A in (a), DP980B in (b), DP980H in (c), and DP980I in (d). In all plots, the solid line represents the displacement to failure, and the dashed lines represent subsequent displacement percentages.

For analysis and testing, the P vs. δ data for each AHSS grade were used to evaluate test conditions as this procedure provided a self-consistent comparison without the need to compensate for differences in sheet thickness or notch depths. For multiple AHSS grades, the un-notched region of the 12.7 mm gauge length experienced yielding. A description of which AHSS grades experienced yielding in the un-notched region is summarized in Table 5.5. This calculation was based on the maximum load reached during the 90% failure displacement tests divided by the cross-sectional area of the un-notched region. If this calculated stress was higher than the reported YS for the AHSS grade in Table 5.4, then yielding of the un-notched region occurred. The TRIP780 material that exhibited the highest pct total elongation had a failure displacement of around 0.006 inches, whereas the DP780I had a failure displacement of 0.012 inches. The reason for the increased failure displacement for DP780I is that the un-notched material in the 12.7 mm gauge section yielded, indicated in Table 5.5.
Table 5.5  Values of Max Stress Reached in the Un-notched Region of Plane Strain Tensile Tests During 90% Failure Displacement. Compared to Yield Stress, Grades That Yielded Are Indicated With a “Y” in The Table.

<table>
<thead>
<tr>
<th>Material</th>
<th>Max Stress (MPa)</th>
<th>Yield Stress (MPa)</th>
<th>Yielded? (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DP980A</td>
<td>567</td>
<td>680</td>
<td>N</td>
</tr>
<tr>
<td>DP980I</td>
<td>614</td>
<td>726</td>
<td>N</td>
</tr>
<tr>
<td>DP980B</td>
<td>736</td>
<td>655</td>
<td>Y</td>
</tr>
<tr>
<td>DP980H</td>
<td>710</td>
<td>620</td>
<td>Y</td>
</tr>
<tr>
<td>TRIP780</td>
<td>421</td>
<td>519</td>
<td>N</td>
</tr>
<tr>
<td>DP780I</td>
<td>622</td>
<td>491</td>
<td>Y</td>
</tr>
<tr>
<td>DP780B</td>
<td>637</td>
<td>491</td>
<td>Y</td>
</tr>
<tr>
<td>DP780H</td>
<td>643</td>
<td>608</td>
<td>Y</td>
</tr>
</tbody>
</table>

Void development data on samples deformed to 90% of the failure displacement were used as a basis for selection of subsequent tests (i.e. percent failure displacement levels). From each of the P vs. δ curves in Figs. 5.10 and 5.11, the 9-photo analysis, detailed in Sec. 4.5.3, was performed. Of the nine photos, the seven center photos were used for analysis. The radius of the notches used for each AHSS grade varied depending on sheet thickness, as shown for DP780B and TRIP780 in Figs. 4.4a and 4.4b, respectively. It was believed that any potential differences in stress gradient between notch radii would be mitigated by the omission of the two outermost images. Different stress gradients present in each notch radius could potentially lead to unique behavior that deviates from the behavior observed in the bulk of the cross-section. Figures 5.12a and 5.12b show the void pct vs. pct failure displacement for 780 MPa strength materials and 980 MPa materials, respectively. The axes for Figs. 5.12a and 5.12b are equivalent. In Figs. 5.12c and 5.12d, the # voids vs. pct failure displacement for the 780 MPa strength materials and 980 MPa strength materials are respectively summarized. The axes for Figs. 5.12c and 5.12d are equivalent. The lines connecting data points in Fig. 5.12 are used to distinguish each steel data set from each other, and are not interpretations of material behavioral trends with strain-to-failure.

With increasing percent failure displacement, void pct for all AHSS grades increased. Certain AHSS grades exhibit an exponential increase in void pct between 80 and 90% failure displacement, indicating that damage nucleates at a faster rate at higher percent failure displacements. An observation in multiple AHSS grades was that up to 70% failure displacement, void pct, as well as # voids, remained unchanged from the corresponding values in the unstrained microstructure, suggesting that microstructural strain accommodation for percent failure displacements up to 70% was in the form of grain deformation. One example of grain deformation in response to the imposed plane strain state is shown in Fig. 5.13 for DP780B tested to 80% failure displacement. Highly deformed regions can be
qualitatively observed in Fig 5.13 in close proximity to the notches. Also, DP780B has a vertical martensite band down the center of the sample (lighter colored, indicated by four white arrows), and this vertical band has been deformed from its initially straight nature in response to the imposed strain gradient. Another qualitative microstructural analysis of grain deformation in response to the imposed strain gradient is illustrated in the initial, unstrained state, and after testing to 80% failure displacement in Fig. 5.14 for DP780B and DP780H, and in Fig. 5.15 for DP980B and DP980I. All photos in Figs. 5.14 and 5.15 are representative of the initial state in the case of the “Initial”, and of the middle seven photos of the 9-photo analysis in the case of the “80%” failure displacement, and tensile axis is vertical. The directionality of the grains in Figs. 5.14 and 5.15 in the 80% failure displacement photo illustrate the amount of deformation grains can accommodate before fracture occurs.

![Graphs showing void pct vs. pct failure displacement and number of voids vs. pct failure displacement for different grades of steel](image_url)

Figure 5.12 Plot of void pct vs. pct failure displacement for 780 MPa strength materials in (a), and for 980 MPa strength materials in (b). Number of voids vs. pct failure displacement for 780 MPa strength materials in (c), and for 980 MPa strength materials in (d). For most grades, the trend appears to be an exponential increase after 80% failure displacement.
Figure 5.12 shows that the # voids vs. percent failure displacement data for all AHSS grades follow a similar trend to the void pct data. The similar behavior of # voids and void pct with increasing pct failure displacement suggests that the increase in # voids controls the increase in void pct as opposed to growth of a few voids. However, for the case of DP980I, Fig. 5.12 shows that the # voids minimally increases with increasing percent failure displacement, but the void pct increases exponentially, suggesting that void growth is the dominant failure mechanism. In other grades, such as DP980H, Fig. 5.12 shows that void pct does not increase when going from 90% to 95% failure displacement, but the # voids increases exponentially. This suggests that nucleation of small voids occurs at the onset of fracture, as evidenced by the 95% failure displacement data point for DP980H in Fig. 5.12d. In every test, the most dominant location where voids nucleated was at the interface between ferrite and second-phase constituents. At 90% failure displacement, all AHSS grades are close to fracturing and the void percent can vary by almost an order of magnitude when DP980B and DP980I are compared. The final stage of failure in all AHSS grades tested is a rapid event. Even with the DP980H sample that was tested to 95% failure displacement, void coalescence was not observed, suggesting that the final nucleation of voids and coalescence leading to fracture must occur at failure displacements above 95%. At 95% failure displacement, the onset of coalescence in the form of aligning voids was believed to have been observed, and is shown in Fig. 5.16 for DP980H tested to 95% failure displacement.

Figure 5.13 SEM photo using SEI of DP780B tested to 80% failure displacement. Notice the light-colored band of martensite down the center of the sample, and how the band rotates counter-clockwise.
Figure 5.14  SEM micrographs using BSE of initial and 80% failure displacement sample for DP780B in (a) and (b), and DP780H in (c) and (d). For (a)-(d), tensile axis is vertical, and images are representative of region of interest.
Figure 5.15  SEM micrographs using BSE of initial and 80% failure displacement sample for DP980B in (a) and (b), and DP980I in (c) and (d). For (a)-(d), tensile axis is vertical, and images are representative of region of interest.
One consideration when considering small void pct values (i.e. < 0.02 pct) in Figs. 5.12a and 5.12b is the potential effect of one large void on the calculated percent. For instance, one large void more than doubled the void pct value reported for TRIP780 at 60% failure displacement, and one void also doubled the unstrained state for DP980H. Though these abnormally large voids can be classified as outliers, it is believed that all representative voids in the images should be analyzed because all the voids contribute to damage in a material. In all AHSS grades, the # voids and/or void pct values exponentially increase around 80-90%. The 90% failure displacement data point for all AHSS grades was determined to contain significant damage in response to the imposed stress-state, and values for void pct and # voids are recorded in Table 5.6 below. To gain a better view of the relationship between # voids and void pct as a function of percent failure displacement for each individual AHSS grade, plots for each AHSS grade are located in Fig. A.6.
Table 5.6  Values of Void Pct and # Voids for AHSS Grades. Values Were Taken at 90% Failure Displacement.

<table>
<thead>
<tr>
<th>Material</th>
<th>Y.S. (MPa)</th>
<th>UTS (MPa)</th>
<th>pct el (total)</th>
<th>pct el (uniform)</th>
<th>Grain Size α</th>
<th>Grain Size α'</th>
<th>MVF</th>
<th>α' (GPa)</th>
<th>α (GPa)</th>
<th>Void Pct (90%)</th>
<th># Voids (90%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DP980A</td>
<td>680</td>
<td>965</td>
<td>12.1</td>
<td>7</td>
<td>1.25</td>
<td>0.62</td>
<td>24.1</td>
<td>9.7</td>
<td>3.6</td>
<td>0.082</td>
<td>158</td>
</tr>
<tr>
<td>DP980I</td>
<td>726</td>
<td>971</td>
<td>11.7</td>
<td>5.8</td>
<td>1.56</td>
<td>0.98</td>
<td>26.2</td>
<td>8.3</td>
<td>3.7</td>
<td>0.109</td>
<td>41</td>
</tr>
<tr>
<td>DP980B</td>
<td>655</td>
<td>1013</td>
<td>12.5</td>
<td>7.4</td>
<td>1.6</td>
<td>1.71</td>
<td>50</td>
<td>7.9</td>
<td>3.8</td>
<td>0.014</td>
<td>73</td>
</tr>
<tr>
<td>DP980H</td>
<td>620</td>
<td>969</td>
<td>16.3</td>
<td>12.2</td>
<td>1.18</td>
<td>0.6</td>
<td>26.3</td>
<td>8.2</td>
<td>3.3</td>
<td>0.044</td>
<td>50</td>
</tr>
<tr>
<td>TRIP780</td>
<td>519</td>
<td>853</td>
<td>18.2</td>
<td>13</td>
<td>1.71</td>
<td>0.81</td>
<td>27.5</td>
<td>9.1</td>
<td>3.4</td>
<td>0.058</td>
<td>116</td>
</tr>
<tr>
<td>DP780I</td>
<td>491</td>
<td>816</td>
<td>17.8</td>
<td>11.3</td>
<td>1.71</td>
<td>0.87</td>
<td>28.4</td>
<td>8.3</td>
<td>3</td>
<td>0.043</td>
<td>178</td>
</tr>
<tr>
<td>DP780B</td>
<td>491</td>
<td>826</td>
<td>17.6</td>
<td>11.2</td>
<td>3.35</td>
<td>2.15</td>
<td>36.5</td>
<td>7.2</td>
<td>3.1</td>
<td>0.083</td>
<td>54</td>
</tr>
<tr>
<td>DP780H</td>
<td>608</td>
<td>862</td>
<td>13.4</td>
<td>8.2</td>
<td>1.57</td>
<td>0.87</td>
<td>27.5</td>
<td>9.1</td>
<td>3.3</td>
<td>0.069</td>
<td>178</td>
</tr>
</tbody>
</table>
CHAPTER 6
DISCUSSION

This chapter begins with an interpretation of some features seen in the tensile tests. Next, the hardness values obtained from nanoindentation data are discussed. Next, an evaluation of the plane strain tensile testing method is discussed, followed by an explanation of the data obtained from the plane strain tensile analysis. Lastly, different AHSS grades are compared to one another to assess the effects and relationships of their material properties.

6.1 Tensile Testing

Figure 5.6 shows that the behaviors of the eight tensile curves fall into three distinct categories. Grades DP780B, DP780I, DP980A, DP980B, and DP980I exhibit continuous yielding and have stress-strain behaviors characteristic of DP steels. Grade DP780H exhibits continuous yielding with an abnormally high YS for 780 MPa strength steels. DP780H was temper-rolled, which explains the higher YS and lower pct el (tot). Grades DP980H and TRIP780 both exhibit an inflection on yielding, and also exhibit the highest pct el (tot) for their respective strength groups. The yield inflection and the superior ductility are most likely due either to the presence of retained austenite in their microstructure, or aging.

6.2 Nanoindentation

The data reported from nanoindentation tests are an average ferrite hardness value, and an average second-phase hardness value for each AHSS grade. Since most histograms in Figs. A.3-A.10 appear normally distributed, an average value was determined to be a good representation of the data. In some cases, such as the second-phase hardness histograms for DP980H and DP980A, too few data points were obtained to confidently claim a normal distribution. With additional nanoindentation experiments performed on DP980A and DP980H, the second-phase data will potentially converge to a normal distribution.

6.3 Plane Strain Tensile Testing: Method Analysis

The plane strain tensile testing method used for this project was created in response to the limitation of the bending under tension frame at ASPPRC to be systematically interrupted during deformation. The similar behavior between the # voids as a function of deformation shown in Figs. 5.12c and 5.12d and the void density data shown in Fig. 2.15 suggests that the plane strain tensile method produced results and behavioral curves consistent with steels analyzed using 3-D X-Ray tomography.

6.4 Plane Strain Tensile Testing: Data Analysis

Figure 5.12 shows a general increase in void pct and # voids with increasing pct failure displacement for all AHSS grades. At relatively high percent failure displacements (60-70%), the # voids and void pct values increased. The void pct value at 90% failure displacement in Fig. 5.12b varies by
almost an order of magnitude when comparing DP980I (0.109%) and DP980B (0.014%). The unique values for # voids and void pct for each AHSS grade indicate a microstructurally dependent response to the imposed plane strain state.

Selected material properties in Table 5.6 were plotted against the # voids and void pct values at 90% failure displacement for all eight AHSS grades to assess whether relationships existed. To evaluate relationships between microstructural parameters more comprehensively, two additional parameters were required. The first calculated parameter involved the hardness values obtained from the nanoindentation analysis. The second-phase hardness value divided by the ferrite hardness value for each AHSS was termed the hardness ratio, and is reported in Table 6.1 under the column heading “α'/α”. The second calculated property was an estimate of the carbon content present in the second-phase constituents based on a rule-of-mixtures formula. It was assumed that ferrite, in all eight AHSS grades, contained a carbon content of 0.022 wt pct. Second-phase volume fraction (MVF in Table 6.1) was assumed to comprise all constituents not identified as ferrite in the eight AHSS microstructures. Further, the density of ferrite and second-phase constituents were assumed equivalent. Using carbon contents from Table 4.1, second-phase constituent carbon contents were calculated for each AHSS grade using Eq. 6.1 below.

\[ C_{\text{bulk wt pct}} = 0.022 \times (1 - \text{MVF}) + C_{\alpha'} \times (\text{MVF}) \]  
Eq. 6.1

where \( C_{\text{bulk wt pct}} \) is the carbon content in wt pct from Table 4.1, \( \text{MVF} \) is the second-phase volume fraction from Table 6.1, and \( C_{\alpha'} \) is the second-phase carbon content. Second-phase carbon content is reported in Table 6.1 under the column header “C-content (α’)”.

<table>
<thead>
<tr>
<th>Material</th>
<th>UTS (MPa)</th>
<th>pct el (total)</th>
<th>Grain Size α</th>
<th>Grain Size α'</th>
<th>MVF</th>
<th>C-content (α)</th>
<th>a' (GPa)</th>
<th>a (GPa)</th>
<th>α'/α</th>
<th>Void Pct (90%)</th>
<th># Voids (90%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DP980A</td>
<td>965</td>
<td>12.1</td>
<td>1.25</td>
<td>0.62</td>
<td>24.1</td>
<td>0.35</td>
<td>9.7</td>
<td>3.6</td>
<td>2.69</td>
<td>0.082</td>
<td>158</td>
</tr>
<tr>
<td>DP980I</td>
<td>971</td>
<td>11.7</td>
<td>1.56</td>
<td>0.98</td>
<td>26.2</td>
<td>0.32</td>
<td>8.3</td>
<td>3.7</td>
<td>2.24</td>
<td>0.109</td>
<td>41</td>
</tr>
<tr>
<td>DP980B</td>
<td>1013</td>
<td>12.5</td>
<td>1.6</td>
<td>1.71</td>
<td>50</td>
<td>0.16</td>
<td>7.9</td>
<td>3.8</td>
<td>2.08</td>
<td>0.014</td>
<td>73</td>
</tr>
<tr>
<td>DP980H</td>
<td>969</td>
<td>16.3</td>
<td>1.18</td>
<td>0.6</td>
<td>26.3</td>
<td>0.51</td>
<td>8.2</td>
<td>3.3</td>
<td>2.48</td>
<td>0.044</td>
<td>50</td>
</tr>
<tr>
<td>TRIP780</td>
<td>853</td>
<td>18.2</td>
<td>1.71</td>
<td>0.81</td>
<td>27.5</td>
<td>0.56</td>
<td>9.1</td>
<td>3.4</td>
<td>2.68</td>
<td>0.058</td>
<td>116</td>
</tr>
<tr>
<td>DP780I</td>
<td>816</td>
<td>17.8</td>
<td>1.71</td>
<td>0.87</td>
<td>28.4</td>
<td>0.23</td>
<td>8.3</td>
<td>3</td>
<td>2.77</td>
<td>0.043</td>
<td>178</td>
</tr>
<tr>
<td>DP780B</td>
<td>826</td>
<td>17.6</td>
<td>3.35</td>
<td>2.15</td>
<td>36.5</td>
<td>0.24</td>
<td>7.2</td>
<td>3.1</td>
<td>2.32</td>
<td>0.083</td>
<td>54</td>
</tr>
<tr>
<td>DP780H</td>
<td>862</td>
<td>13.4</td>
<td>1.57</td>
<td>0.87</td>
<td>27.5</td>
<td>0.27</td>
<td>9.1</td>
<td>3.3</td>
<td>2.76</td>
<td>0.069</td>
<td>178</td>
</tr>
</tbody>
</table>
Many material properties in Table 6.1 were plotted against # voids, void pct and/or hardness ratio to observe whether any relationship exists. The potential relationships considered included:

1) Hardness Ratio vs. pct el (tot)
2) # Voids vs. Grain Size ($\alpha'$)
3) Void Pct vs. Grain Size ($\alpha'$)
4) Void Pct vs. pct el (tot)
5) # Voids vs. MVF
6) Void Pct vs. MVF
7) # Voids vs. Void Pct
8) # Voids vs. pct el (tot)
9) Hardness ($\alpha'$) vs. pct el (tot)
10) Void Pct vs. Hardness Ratio
11) MVF vs. UTS
12) Hardness ($\alpha'$) vs. UTS
13) C-content vs. Hardness ($\alpha'$)
14) # Voids vs. Hardness Ratio

For plots 1-13, no observable correlations between the indicated parameters existed, indicating that most material properties are dependent on more than one material property. The data in plots 1-13 was mostly scatter with no observable relationship. The only plot that exhibited a relationship was # voids vs. hardness ratio, shown below in Fig. 6.1a. Figure 6.1a shows an increase in # voids with an increase in hardness ratio. A higher hardness ratio denotes a greater strength disparity between constituents in a microstructure which yields a higher degree of strain partitioning upon deformation, promoting void formation. The relationship shown in Fig. 6.1a suggests that the hardness ratio has a dominant effect on the # voids nucleated in a microstructure. In Fig. 6.1a, eight data points are present; there are two data points that partially overlap in the upper right-hand corner of the graph. Figure 6.1b shows a plot of Hardness ($\alpha'$) vs. C-content ($\alpha'$). As mentioned earlier, multiple strengthening mechanisms contribute to a constituent hardness value in addition to C-content. The weak relationship shown in Fig. 6.1b suggests multiple strengthening mechanisms are present in the second-phase constituents of the AHSS grades studied. There seems to be only a slight increase in second-phase hardness with increasing C-content. Shown also in Fig. 6.1 is the degree of correlation of the data to a linear fit ($R^2$). An $R^2$ value closer to 1 indicates a stronger linear relationship.
Figure 6.1   Plot of # Voids vs. Hardness Ratio in (a). An increasing # Voids can be seen with an increasing Hardness Ratio. Plot of α’ Hardness (GPa) vs. second-phase C-content (wt pct) in (b). The weak relationship suggests multiple strengthening mechanisms are present in the second-phase constituents.

6.5 Comparisons Between AHSS Grades

Table 6.1 shows many AHSS grades exhibiting similar material properties in certain categories. When two AHSS grades exhibit similar material properties for given categories, it allows comparisons to be made between categories of differing material properties. The benefit of having eight AHSS grades is that multiple comparisons were able to be made, isolating the effects of certain material properties on mechanical response.

1) The first comparison of AHSS grades is between DP780I and DP780B, shown below in Table 6.2. Grades DP780I and DP780B have similar UTS, pct el (tot), C-content (α’), and ferrite hardness, and have different grain sizes (α’ and α), MVF, second-phase hardness, hardness ratio, void pct (90%), and # voids (90%).

<table>
<thead>
<tr>
<th>Material</th>
<th>UTS (MPa)</th>
<th>pct el (total)</th>
<th>Grain Size α</th>
<th>Grain Size α’</th>
<th>MVF</th>
<th>C-content (α’)</th>
<th>α (GPa)</th>
<th>α’/α</th>
<th>Void Pct (90%)</th>
<th># Voids (90%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DP780I</td>
<td>816</td>
<td>17.8</td>
<td>1.71</td>
<td>0.87</td>
<td>28.4</td>
<td>0.23</td>
<td>8.3</td>
<td>3</td>
<td>2.77</td>
<td>0.043</td>
</tr>
<tr>
<td>DP780B</td>
<td>826</td>
<td>17.6</td>
<td>3.35</td>
<td>2.15</td>
<td>36.5</td>
<td>0.24</td>
<td>7.2</td>
<td>3</td>
<td>2.32</td>
<td>0.083</td>
</tr>
</tbody>
</table>

The strength of the DP780I was due in part to a smaller grain size and higher second-phase hardness, while the ductility came from a lower MVF. The strength of DP780B was due in part to the
higher MVF, and the ductility arose from the larger grain size and lower second-phase hardness. The increased second-phase hardness of DP780I for a similar second-phase carbon could be due to a different tempering process than experienced by DP780B. This comparison shows that similar UTS and pct el (tot) can be achieved using different combinations of MVF, grain size, and constituent hardness values.

2) A comparison can be made between TRIP780 and DP780H, shown below in Table 6.3. Grades DP780H and TRIP780 have similar UTS, grain size (α’ and α), MVF, second-phase hardness, ferrite hardness, hardness ratio, void pct (90%), and # voids (90%), and different pct el (tot) and second-phase carbon content.

Table 6.3  Comparison of DP780H and TRIP780.

<table>
<thead>
<tr>
<th>Material</th>
<th>UTS (MPa)</th>
<th>pct el (total)</th>
<th>Grain Size α</th>
<th>Grain Size α'</th>
<th>MVF</th>
<th>C-content (α')</th>
<th>α' (GPa)</th>
<th>α (GPa)</th>
<th>α'/α</th>
<th>Void Pct (90%)</th>
<th># Voids (90%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TRIP780</td>
<td>853</td>
<td>18.2</td>
<td>1.71</td>
<td>0.81</td>
<td>27.5</td>
<td>0.56</td>
<td>9.1</td>
<td>3.4</td>
<td>2.68</td>
<td>0.058</td>
<td>116</td>
</tr>
<tr>
<td>DP780H</td>
<td>862</td>
<td>13.4</td>
<td>1.57</td>
<td>0.87</td>
<td>27.5</td>
<td>0.27</td>
<td>9.1</td>
<td>3.3</td>
<td>2.76</td>
<td>0.069</td>
<td>178</td>
</tr>
</tbody>
</table>

The increase in pct el (tot) of TRIP780 is interpreted to be due to the presence of 5% retained austenite. This comparison shows that the presence of austenite increases ductility without appreciably affecting other material properties. The similar second-phase hardness values despite a lower carbon-content for DP780H was attributed to the temper-roll treatment experienced by DP780H.

3) A comparison can be made between DP780I and TRIP780, shown in Table 6.4. Grades DP780I and TRIP780 have similar pct el (tot), grain size (α’ and α), MVF, hardness ratio, void pct (90%), and # voids (90%), and different UTS, second-phase carbon content, second-phase hardness, and ferrite hardness values.

Table 6.4  Comparison of DP780I and TRIP780.

<table>
<thead>
<tr>
<th>Material</th>
<th>UTS (MPa)</th>
<th>pct el (total)</th>
<th>Grain Size α</th>
<th>Grain Size α'</th>
<th>MVF</th>
<th>C-content (α')</th>
<th>α' (GPa)</th>
<th>α (GPa)</th>
<th>α'/α</th>
<th>Void Pct (90%)</th>
<th># Voids (90%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TRIP780</td>
<td>853</td>
<td>18.2</td>
<td>1.71</td>
<td>0.81</td>
<td>27.5</td>
<td>0.56</td>
<td>9.1</td>
<td>3.4</td>
<td>2.68</td>
<td>0.058</td>
<td>116</td>
</tr>
<tr>
<td>DP780I</td>
<td>816</td>
<td>17.8</td>
<td>1.71</td>
<td>0.87</td>
<td>28.4</td>
<td>0.23</td>
<td>8.3</td>
<td>3</td>
<td>2.77</td>
<td>0.043</td>
<td>178</td>
</tr>
</tbody>
</table>

The higher UTS of TRIP780 is due in part to the higher ferrite and second-phase hardness values. TRIP780 exhibited a similar elongation despite the harder phases due to the presence of 5% retained austenite. DP780I exhibited a lower ferrite hardness most likely because of the lower amount of Silicon present. The high second-phase hardness of DP780I suggests other strengthening mechanisms are present.
A comparison can be made between DP980A and DP980I, shown in Table 6.5. Grades DP980A and DP980I have similar UTS, pct el (tot), MVF, second-phase carbon content, ferrite hardness, and void pct (90%), and have different grain size (α’ and α), second-phase hardness, hardness ratio, and # voids (90%) values.

<table>
<thead>
<tr>
<th>Material</th>
<th>UTS (MPa)</th>
<th>pct el (total)</th>
<th>Grain Size α</th>
<th>Grain Size α’</th>
<th>MVF</th>
<th>C-content (α’)</th>
<th>α’ (GPa)</th>
<th>α (GPa)</th>
<th>α’/α</th>
<th>Void Pct (90%)</th>
<th># Voids (90%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DP980A</td>
<td>965</td>
<td>12.1</td>
<td>1.25</td>
<td>0.62</td>
<td>24.1</td>
<td>0.35</td>
<td>3.6</td>
<td>2.69</td>
<td>0.082</td>
<td>158</td>
<td></td>
</tr>
<tr>
<td>DP980I</td>
<td>971</td>
<td>11.7</td>
<td>1.56</td>
<td>0.98</td>
<td>26.2</td>
<td>0.32</td>
<td>8.3</td>
<td>2.24</td>
<td>0.109</td>
<td>41</td>
<td></td>
</tr>
</tbody>
</table>

The strength of DP980A came from the combined effects of a smaller grain size (α’ and α) and a higher second-phase hardness value. From the comparison of properties in Table 6.5, DP980I was expected to have a higher pct el (tot) value. In Sec. 5.7, the unique behavior of the void pct and # voids at 90% failure displacement for DP980I was discussed. For DP980I, as higher percent failure displacement values were reached, # voids did not increase, but the void pct increased exponentially, indicating void growth. The areas used for the 9-photo analysis of DP980A and DP980I tested to 90% failure displacement are shown in Figs. 6.2a and 6.2b, respectively. Many large voids (black dots in gray region) are observed in DP980I compared to DP980A. The existence of many large voids could be one reason why DP980I possesses a lower pct el (tot) than predicted. Large voids cause larger stress gradients in the material, which potentially reduces ductility and can lead to premature fracture.

Figure 6.2 SEM image using BSE imaging of the area used for the 9-photo analysis on DP980A in (a), and on DP980I in (b). DP980A and DP980I were tested to 90% failure displacement. Many large voids are evident in (b), where (a) tends to contain only small voids.

78
5) A comparison can be made between DP980H and DP980I, shown in Table 6.6. Grades DP980I and DP980H have similar UTS, MVF, second-phase hardness, and # voids (90%), and have different pct el (tot), grain size (α’ and α), second-phase carbon content, ferrite hardness, hardness ratio, and void pct (90%) values.

Table 6.6 Comparison of DP980H and DP980I.

<table>
<thead>
<tr>
<th>Material</th>
<th>UTS (MPa)</th>
<th>pct el (total)</th>
<th>Grain Size α</th>
<th>Grain Size α’</th>
<th>MVF</th>
<th>C-content (α’/α)</th>
<th>α’ (GPa)</th>
<th>α (GPa)</th>
<th>α’/α Void Pct (90%)</th>
<th># Voids (90%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DP980I</td>
<td>971</td>
<td>11.7</td>
<td>1.56</td>
<td>0.98</td>
<td>26.2</td>
<td>0.32</td>
<td>8.3</td>
<td>3.7</td>
<td>2.24</td>
<td>0.109</td>
</tr>
<tr>
<td>DP980H</td>
<td>969</td>
<td>16.3</td>
<td>1.18</td>
<td>0.6</td>
<td>26.3</td>
<td>0.51</td>
<td>8.2</td>
<td>3.3</td>
<td>2.48</td>
<td>0.044</td>
</tr>
</tbody>
</table>

The strength of D980H was due in part to a decreased grain size (α’ and α), while the strength of DP980I was due in part to a higher ferrite hardness value. One explanation for the similar second-phase hardness values with differing second-phase carbon contents is that DP980H was tempered to a higher degree than DP980I. DP980H has a higher pct el (tot) due to the presence of 5% retained austenite. For a similar # voids (90%) value, the void pct (90%) of DP980I is over two times larger, indicating the presence of larger voids than in DP980H. The presence of larger voids could be limiting the ductility of DP980I. This comparison highlights the effect austenite can have on AHSS grades during deformation. The additional elongation while not appreciably affecting any other microstructural properties makes austenite a desirable phase to incorporate into future AHSS grades.

6) A comparison can be made between TRIP780 and DP980H, shown in Table 6.7. Grades DP980H and TRIP780 have similar MVF, second-phase carbon content, ferrite hardness, and void pct (90%), and different UTS, pct el (tot), grain size (α’ and α), second-phase hardness, hardness ratio, and # voids (90%) values.

Table 6.7 Comparison of TRIP780 and DP980H.

<table>
<thead>
<tr>
<th>Material</th>
<th>UTS (MPa)</th>
<th>pct el (total)</th>
<th>Grain Size α</th>
<th>Grain Size α’</th>
<th>MVF</th>
<th>C-content (α’/α)</th>
<th>α’ (GPa)</th>
<th>α (GPa)</th>
<th>α’/α Void Pct (90%)</th>
<th># Voids (90%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DP980H</td>
<td>969</td>
<td>16.3</td>
<td>1.18</td>
<td>0.6</td>
<td>26.3</td>
<td>0.51</td>
<td>8.2</td>
<td>3.3</td>
<td>2.48</td>
<td>0.044</td>
</tr>
<tr>
<td>TRIP780</td>
<td>853</td>
<td>18.2</td>
<td>1.71</td>
<td>0.81</td>
<td>27.5</td>
<td>0.56</td>
<td>9.1</td>
<td>3.4</td>
<td>2.68</td>
<td>0.058</td>
</tr>
</tbody>
</table>

The strength for DP980H came in part from the smaller grain size (α’ and α) at the expense of pct el (tot). The increase in second-phase hardness for TRIP780 could be due to the slightly higher second-phase carbon content. The lower second-phase hardness imparts a higher ductility to DP980H than would be expected if the second-phase hardness values were similar.
A comparison can be made between DP780H and DP780I, shown below in Table 6.8. Grades DP780H and DP780I have similar grain size ($\alpha'$ and $\alpha$), MVF, second-phase carbon content, hardness ratio, void pct (90%), and # voids (90%), and different UTS, pct el (tot), second-phase hardness, and ferrite hardness values.

Table 6.8: Comparison of DP780H and DP780I.

<table>
<thead>
<tr>
<th>Material</th>
<th>UTS (MPa)</th>
<th>pct el (total)</th>
<th>Grain Size $\alpha$</th>
<th>Grain Size $\alpha'$</th>
<th>MVF</th>
<th>C-content ($\alpha'$)</th>
<th>$\alpha'$ (GPa)</th>
<th>$\alpha$ (GPa)</th>
<th>$\alpha'/\alpha$</th>
<th>Void Pct (90%)</th>
<th># Voids (90%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DP780I</td>
<td>816</td>
<td>17.8</td>
<td>1.71</td>
<td>0.87</td>
<td>28.4</td>
<td>0.23</td>
<td>8.3</td>
<td>3</td>
<td>2.77</td>
<td>0.043</td>
<td>178</td>
</tr>
<tr>
<td>DP780H</td>
<td>862</td>
<td>13.4</td>
<td>1.57</td>
<td>0.87</td>
<td>27.5</td>
<td>0.27</td>
<td>9.1</td>
<td>3.3</td>
<td>2.76</td>
<td>0.069</td>
<td>178</td>
</tr>
</tbody>
</table>

The higher UTS and lower pct el (tot) of DP780H is due to the higher second-phase and ferrite hardness values. The comparison shown in Table 6.8 shows that the hardness values of the individual constituents can influence formability, a factor alluded to by previous experiments using bending under tension [12], [14], [35].

The comparisons of AHSS grades shown in Tables 6.2-6.8 indicate that similar mechanical properties can be achieved using different combinations of MVF, grain size, and constituent hardness values. An optimal combination of MVF, grain size, and constituent hardness values is believed to exist that would maximize the strength and ductility of AHSS grades. For a similar UTS value, the presence of austenite increased the ductility in every comparison, indicating the desire to incorporate higher quantities of austenite in future AHSS production.
CHAPTER 7
CONCLUSIONS

The purpose of this project was to identify the microstructural factors that influence fracture in AHSS grades. When considering multiple phases with differing strength levels, particularly in higher-strength AHSS grades, complex microstructural interactions develop. Understanding the role of the different microstructural properties that affect fracture response was of interest. There were eight main conclusions from this project that contribute to the understanding of formability of higher-strength AHSS grades.

1) The plane strain tensile method used to observe microstructural damage was successful in producing void behavior similar to steels analyzed using 3-D X-Ray tomography. The offset notch successfully imposed a localized through-thickness shear fracture, and testing samples to higher percent failure displacements produced damage evolution in response to the plane strain state.

2) The method using nanoindentation was successful in obtaining hardness values for ferrite and second-phase constituents. After filtering out the data containing interfacial effects, the remaining hardness values separated into two distinct peaks; the lower hardness peak being ferrite, and the higher hardness peak being second-phase. The technique of nanoindentation quantitatively measures all strengthening effects present in primary and second-phase constituents.

3) The number of voids nucleated in the AHSS grades during the plane strain tensile testing showed a dependence on hardness ratio. Since all grades contained a critical number of voids before failure, it is plausible that the suppression of voids will extend formability limits for AHSS grades by reaching this critical number of voids at higher strains.

4) When performing the analysis portion of the plane strain tensile method, almost all voids formed at the interface between primary and second-phase constituents, confirming that hardness ratio plays a significant role in void nucleation.

5) The strength (i.e. hardness) of the individual constituents that comprise an AHSS grade has an effect on mechanical properties, and must be considered when predicting formability. For example, from the comparison of DP780I and DP780H, an increase in constituent hardness values led to decreased total
elongation and higher UTS. AHSS grades comprised of constituents with higher hardness values tend to elongate less, decreasing formability limits.

6) The final stage of failure in the AHSS grades tested is a rapid event and occurred over a very small displacement increment. The number of voids and void percent in the AHSS grades at 70% failure displacement began to increase over the unstrained state, suggesting the majority of damage accumulated in the microstructure was grain deformation.

7) From observations of a plane strain tensile sample tested to 95% failure displacement (DP980H), void coalescence was determined to occur at percent failure displacements greater than 95%. Since void coalescence was absent from all AHSS grades tested to 90% failure displacement, it is assumed void coalescence occurs at failure displacements greater than 95% for all AHSS grades tested.

8) The presence of retained austenite in the AHSS grades TRIP780 and DP980H greatly increased elongation properties despite their similar properties to the other AHSS grades in the study. When TRIP780 was compared to DP780H, all properties except C-content and pct el (tot) were similar, and TRIP780 had a higher elongation. The in-situ phase transformation of austenite to martensite is seen as a desirable phenomenon, as it increases ductility without compromise of other properties of interest.
CHAPTER 8
FUTURE WORK

This project has established relationships between constituent hardness values and formability limits. The next step in this project would be to quantify the effects of constituent hardness values on formability. One method to accomplish this would be to systematically change one of the parameters of interest, specifically constituent hardness. This can be performed by obtaining a commercially produced DP steel, then temper samples to different amounts to soften the second-phase constituent, decreasing absolute hardness, as well as the hardness ratio. A similar analysis to what was performed in this work could be used to quantify the effects of constituent strength on formability limits. To further analyze critical strains on void nucleation, another project would be quantifying the critical strain accommodated by a primary/second-phase interface before void nucleation using DIC. Testing a DP steel with varying hardness ratios would give insight into the critical void nucleation strain for a given hardness ratio.
REFERENCES


Figure A.1  XRD scans for DP780B in (a), DP780H in (b), DP780I in (c), and TRIP780 in (d).
Figure A.2 XRD scans for DP980A in (a), DP980B in (b), DP980H in (c), and DP980I in (d).
Figure A.3  Histograms generated from ferrite hardness data in (a), and second-phase hardness data in (b) for DP780B.

Figure A.4  Histograms generated from ferrite hardness data in (a), and second-phase hardness data in (b) for DP780H.
Figure A.5  Histograms generated from ferrite hardness data in (a), and second-phase hardness data in (b) for DP780I.

Figure A.6  Histograms generated from ferrite hardness data in (a), and second-phase hardness data in (b) for TRIP780.
Figure A.7  Histograms generated from ferrite hardness data in (a), and second-phase hardness data in (b) for DP980A.

Figure A.8  Histograms generated from ferrite hardness data in (a), and second-phase hardness data in (b) for DP980B.
Figure A.9  Histograms generated from ferrite hardness data in (a), and second-phase hardness data in (b) for DP980H.

Figure A.10  Histograms generated from ferrite hardness data in (a), and second-phase hardness data in (b) for DP980I.
Figure A.11  Tensile curves for 780 MPa strength materials. Each plot has three RD tensile tests, and three TD tensile tests.
Figure A.12  Tensile curves for 980 MPa strength materials. Each plot has three RD tensile tests, and three TD tensile tests.
Table A.1: Material Properties With One Standard Deviation Quoted in Parentheses Below Respective Value.

<table>
<thead>
<tr>
<th>Material</th>
<th>Y.S. (MPa)</th>
<th>UTS (MPa)</th>
<th>% el (total)</th>
<th>% el (uniform)</th>
<th>Grain Size α</th>
<th>Grain Size α'</th>
<th>MVF</th>
<th>α' (GPa)</th>
<th>α (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RD</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DP980A</td>
<td>679.83 (1.4)</td>
<td>965.25 (5.5)</td>
<td>12.13 (0.12)</td>
<td>7 (0.10)</td>
<td>1.25 (0.06)</td>
<td>0.62 (0.07)</td>
<td>24.1 (2.8)</td>
<td>9.7 (1.24)</td>
<td>3.6 (0.08)</td>
</tr>
<tr>
<td>TD</td>
<td>667.85 (11.9)</td>
<td>979.84 (1.2)</td>
<td>9.88 (0.03)</td>
<td>6.22 (0.03)</td>
<td>1.56 (0.08)</td>
<td>0.98 (0.09)</td>
<td>26.2 (2.2)</td>
<td>8.3 (0.71)</td>
<td>3.7 (0.08)</td>
</tr>
<tr>
<td>RD</td>
<td>725.88 (8.5)</td>
<td>971.4 (0.2)</td>
<td>11.7 (0.09)</td>
<td>5.83 (0.14)</td>
<td>1.56 (0.08)</td>
<td>0.98 (0.09)</td>
<td>26.2 (2.2)</td>
<td>8.3 (0.71)</td>
<td>3.7 (0.08)</td>
</tr>
<tr>
<td>TD</td>
<td>683.99 (26.7)</td>
<td>961.91 (1.4)</td>
<td>11.82 (0.08)</td>
<td>6.38 (0.16)</td>
<td>1.6 (0.15)</td>
<td>1.71 (0.14)</td>
<td>50.0 (3.9)</td>
<td>7.9 (0.36)</td>
<td>3.8 (0.09)</td>
</tr>
<tr>
<td>RD</td>
<td>655.32 (7.9)</td>
<td>1012.86 (1.26)</td>
<td>12.53 (0.5)</td>
<td>7.42 (0.14)</td>
<td>1.18 (0.07)</td>
<td>0.6 (0.07)</td>
<td>26.3 (2.9)</td>
<td>8.7 (1.13)</td>
<td>3.3 (0.06)</td>
</tr>
<tr>
<td>TD</td>
<td>654.38 (21.1)</td>
<td>1029.26 (4.53)</td>
<td>10.75 (0.44)</td>
<td>6.83 (0.38)</td>
<td>1.6 (0.15)</td>
<td>1.71 (0.14)</td>
<td>50.0 (3.9)</td>
<td>7.9 (0.36)</td>
<td>3.8 (0.09)</td>
</tr>
<tr>
<td>RD</td>
<td>619.59 (5.0)</td>
<td>969.39 (3.0)</td>
<td>16.33 (0.23)</td>
<td>12.2 (0.17)</td>
<td>1.18 (0.07)</td>
<td>0.6 (0.07)</td>
<td>26.3 (2.9)</td>
<td>8.7 (1.13)</td>
<td>3.3 (0.06)</td>
</tr>
<tr>
<td>TD</td>
<td>534.46 (6.2)</td>
<td>933.22 (7.3)</td>
<td>17.03 (0.46)</td>
<td>12.53 (0.06)</td>
<td>1.6 (0.15)</td>
<td>1.71 (0.14)</td>
<td>50.0 (3.9)</td>
<td>7.9 (0.36)</td>
<td>3.8 (0.09)</td>
</tr>
<tr>
<td>RD</td>
<td>518.68 (4.0)</td>
<td>852.54 (0.7)</td>
<td>18.2 (0.26)</td>
<td>12.96 (0.15)</td>
<td>1.71 (0.14)</td>
<td>0.81 (0.06)</td>
<td>27.5 (2.0)</td>
<td>9.1 (0.60)</td>
<td>3.4 (0.05)</td>
</tr>
<tr>
<td>TD</td>
<td>485.98 (5.1)</td>
<td>843.22 (1.6)</td>
<td>19.63 (0.25)</td>
<td>13.83 (0.35)</td>
<td>1.71 (0.14)</td>
<td>0.81 (0.06)</td>
<td>27.5 (2.0)</td>
<td>9.1 (0.60)</td>
<td>3.4 (0.05)</td>
</tr>
<tr>
<td>RD</td>
<td>490.63 (1.9)</td>
<td>815.77 (6.3)</td>
<td>17.83 (0.15)</td>
<td>11.25 (0.55)</td>
<td>1.71 (0.11)</td>
<td>0.87 (0.07)</td>
<td>28.4 (2.8)</td>
<td>8.3 (0.49)</td>
<td>3 (0.08)</td>
</tr>
<tr>
<td>TD</td>
<td>487.12 (1.5)</td>
<td>824.9 (3.2)</td>
<td>17.1 (0.10)</td>
<td>11.5 (0.25)</td>
<td>1.71 (0.11)</td>
<td>0.87 (0.07)</td>
<td>28.4 (2.8)</td>
<td>8.3 (0.49)</td>
<td>3 (0.08)</td>
</tr>
<tr>
<td>RD</td>
<td>491.18 (0.7)</td>
<td>825.63 (0.8)</td>
<td>17.55 (0.56)</td>
<td>11.2 (0.2)</td>
<td>3.35 (0.20)</td>
<td>2.15 (0.187)</td>
<td>36.5 (2.8)</td>
<td>7.2 (0.32)</td>
<td>3.1 (0.05)</td>
</tr>
<tr>
<td>TD</td>
<td>501.14 (0.8)</td>
<td>834.39 (1.9)</td>
<td>16.63 (0.29)</td>
<td>10.62 (0.28)</td>
<td>3.35 (0.20)</td>
<td>2.15 (0.187)</td>
<td>36.5 (2.8)</td>
<td>7.2 (0.32)</td>
<td>3.1 (0.05)</td>
</tr>
<tr>
<td>RD</td>
<td>607.52 (2.3)</td>
<td>861.69 (0.7)</td>
<td>13.37 (0.12)</td>
<td>8.23 (0.25)</td>
<td>1.57 (0.07)</td>
<td>0.87 (0.05)</td>
<td>27.5 (1.8)</td>
<td>9.1 (0.56)</td>
<td>3.3 (0.10)</td>
</tr>
<tr>
<td>TD</td>
<td>601.66 (2.6)</td>
<td>856.51 (1.9)</td>
<td>14.77 (0.15)</td>
<td>8.5 (0.10)</td>
<td>1.57 (0.07)</td>
<td>0.87 (0.05)</td>
<td>27.5 (1.8)</td>
<td>9.1 (0.56)</td>
<td>3.3 (0.10)</td>
</tr>
</tbody>
</table>
Figure A.13  Void Pct and # voids vs. Pct. Failure Displacement for DP780B in (a), DP780H in (b), DP780I in (c), and TRIP780 in (d).
Figure A.14 Void Pct and # voids vs. Pct. Failure Displacement for DP980A in (a), DP980B in (b), DP980H in (c), and DP980I in (d).