EFFECTS OF DEFORMATION MODES ON THE SUSCEPTIBILITY OF DELAYED FRACTURE OF ADVANCED HIGH STRENGTH STEELS

by

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ABSTRACT

The effects of different deformation modes on the susceptibility to delayed fracture in advanced high strength steels (AHSSs) were evaluated using four types of commercially-produced sheet steels (DP780, DP980, TRIP780 and TRIP980). Specimens were strained by either uniaxial tension or bending prior to hydrogen charging and their delayed fracture response was compared with non-deformed samples. Micro hardness testing was performed to assess the effect of deformation on the variation of strength through the thickness of the sheet. Sheet edge effects were minimized by grinding and polishing of the edges and corners. Cleaning of the samples was performed before hydrogen charging. The samples were cathodically charged, using a DC power supply to provide consistent hydrogen charging, and were then transferred to a three-point test setup to evaluate delayed fracture. A simple three-point bend test was used to impose a tensile stress on one of the surfaces. The samples were held in the test setup until fracture occurred in order to assess their susceptibility to delayed fracture as a function of prior deformation mode. Following three point bend tests, scanning electron microscopy fractography and metallography were used to evaluate the fracture mechanisms and crack details.

The four steels responded differently to the deformation modes. In general, the steels in this study have an increasing susceptibility to delayed fracture with deformation. No deformation showed the least susceptibility, followed by uniaxial tension, and then bending. The surface region of the bent samples had a higher local effective strain that created a high dislocation density on the surface, making it more susceptible to delayed fracture. After the bending deformation the two surfaces were each tested. The samples tested from the concave down surface (surface in contact with the die) were more susceptible to delayed fracture than those samples tested from the concave up surface (surface not in contact with the die). The concave down surface was compressed on bending and stretched on unbending with additional tension occurring upon delayed fracture testing. The concave up surfaces were stretched during bending followed by compression on unbending then stretched during delayed fracture testing. Steels with an ultimate tensile strength of 980 MPa were more susceptible to delayed fracture, as compared to the 780 MPa strength steels. The TRIP980 steel was more susceptible to delayed fracture than the DP980 steel when strained with uniaxial tension or by bending. However, the DP780 steel was more susceptible to delayed fracture than the TRIP780 steel, for both uniaxial and bending deformation. The TRIP780 had some retained austenite after deformation, whereas the TRIP980 showed no evidence of retained austenite after deformation.
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CHAPTER 1 INTRODUCTION

The modern automotive industry is seeking ways to improve the performance of light weight vehicle parts, and governments in many countries are also imposing environmental, energy conservation and passenger safety standards [1]. Minimizing the size of automotive components and reducing the thickness of the sheet metal are two possible solutions to reduce the overall weight of parts. However, the strength and toughness of installed components might not meet appropriate safety standards after these changes. This potential risk can be addressed through the use of advanced high strength steels (AHSS) which can maintain the required high strength when used in thinner components [1]. Though AHSS are suitable to use in order to overcome such a loss of strength, they can be susceptible to delayed fracture. Although several studies have been conducted to investigate the various factors affecting delayed fracture, further understanding of the details of delayed fracture and especially the effect of prior-deformation on delayed fracture is warranted.

The prime focus of this research is on the effects of different deformation modes – no deformation, uniaxial tension and bending on the susceptibility of delayed fracture in DP780, DP980, TRIP780 and TRIP980 steels. Micro hardness tests were conducted to evaluate the effect of strain paths on the hardness and dislocation density distribution through the thickness of the deformed sheets. Delayed fracture is often caused by hydrogen. The role of diffusible hydrogen on both the hydrogen embrittlement and delayed fracture susceptibility of advanced high strength sheet steels were carefully considered in this project. Stress-strain curves of each type and grade of steel were an effective tool to control and estimate the stress level in three point bend testing, which was used to evaluate the susceptibility to delayed fracture. Scanning electron microscopy (SEM) images and fractography were used to assess hydrogen induced crack and fracture modes.

The primary content of this thesis provides background on the subject, as well as results and analysis from the experiments. A summary of prior studies on the delayed fracture phenomenon in advanced high strength steels (AHSS) obtained from recent articles focusing on the hydrogen and strain path effects is presented. AHSS are mainly used in automotive applications and they provide light weight, less fuel consumption and improved safety protection. To simulate the applications of these AHSS sheets, deformation, hydrogen charging, hydrogen analysis and a three point bend test are all included in this project. A methodology to evaluate delayed fracture susceptibility was designed and tested to simulate real world situations.
CHAPTER 2  BACKGROUND

There is one type of fracture—delayed fracture—which has recently become an important issue because of the greater use of high strength steels [2]. This type of fracture is often associated with hydrogen. Hydrogen in steels can be in two primary forms—diffusible and non-diffusible. Diffusible hydrogen is relatively mobile within the steel, whereas non-diffusible hydrogen is trapped at various sites within the steel. Diffusible hydrogen, which desorbs at low temperatures, has more influence on the delayed fracture of AHSS as compared to the higher temperature conditions [3]. In the automotive industry, most body parts are produced and processed at room temperature or temperatures slightly higher during the stamping process. As a result, the solubility, diffusivity, and trapping sites of hydrogen at room temperature need to be understood.

Most of the internal hydrogen is trapped at a variety of defects within the steel such as dislocations, grain boundaries, micro voids and inclusions [4]. These trapping sites are classified into two types, based on the binding energy value. A binding energy of 60 kJ/mol is the threshold value to determine the type of trapping site, the reversible traps have lower values while the irreversible trapping sites have greater values [5]. Other literature states that the transition binding energy between reversible and irreversible sites should be considered as 75 (weak) or 95 (strong) kJ/mol [6]. The binding energy is a type of activation energy and it reflects the difficulty of hydrogen to diffuse out of the various trapping sites. The activation energies for hydrogen to diffuse out of grain boundaries, dislocations, and micro-voids are reported as 17.2 kJ/mol, 26.8 kJ/mol, and 35.2kJ/mol, respectively [7]. Because of these low values of trapping activation energies, grain boundaries, dislocations, and micro-voids are regions where diffusible hydrogen usually resides and from where it is released. The next section reviews the literature to summarize several dominating mechanisms and models proposed to explain hydrogen induced fracture. The section also provides details about the role that hydrogen plays in affecting the various factors that influence the susceptibility of materials to hydrogen delayed fracture.

2.1 Dominating Mechanisms and Models of Delayed Fracture Caused by Hydrogen Embrittlement

Several mechanisms have been proposed to explain hydrogen induced delayed fracture. The two most highly cited mechanisms are 1) hydrogen-enhanced localized plasticity (HELP); and 2) hydrogen induced decohesion [8]. A third mechanism, the internal-pressure mechanism was previously considered to be classical, but it is not generally applicable for most AHSS, due to the precipitation of hydrogen in particles [9]. As a consequence, the two viable mechanisms will be introduced and explained in the
following subsections.

### 2.1.1 Hydrogen Enhanced Localized Plasticity (HELP)

The role hydrogen plays is to lower the critical stress value that is required to cause the movement of dislocations, and this fracture mechanism is generally considered to be HELP [8][10]. Hydrogen can also increase the dislocation density as well as dislocation mobility near areas with a high concentration of hydrogen [10]. This mechanism is considered to be applicable in the regions where hydrogen has been trapped and the accumulation is high. A large amount of hydrogen usually segregates to discontinuities in microstructure, such as dislocations, grain boundaries, and interfaces [11]. The hydrogen concentration is amplified by solute hydrogen at the crack tip—this location is where HELP usually occurs since hydrogen has a large influence [8]. After a micro crack initiates in the sample, the HELP mechanism accounts for further growth of the fracture. The type of fracture in this mechanism is usually transgranular along slip planes, but intergranular fracture can sometimes be observed [10]. If intergranular fracture occurs, it is usually detected in the regions near grain boundaries instead of within the grain boundaries [10].

Shear localization is another factor that is affected by HELP. This occurs when the localized flow stress is lower than that of adjacent regions in the material [8]. When there is a local decrease in flow stress with the sample, there will be a concentration of deformation in that region. Since this localized deformation occurs by shear, shear localization can sometimes be observed. The solute hydrogen is able to increase the localized flow by rising the mobility of dislocations [8].

Both temperature and strain rate play important roles in affecting the HELP mechanism. Under low strain rates and high temperatures where hydrogen is highly mobile, the effects of hydrogen are usually enhanced and may lead to fracture [8]. However, when the temperature is too high, it enhances hydrogen diffusivity to a point where hydrogen embrittlement will not occur. The hydrogen in the material deformed at a low strain rate and high temperature primarily reduces the elastic bonding between atoms allowing dislocations in the microstructure to move more easily [12].

### 2.1.2 Hydrogen-induced Decohesion

According to Troiano, the reduced lattice cohesive strength in high-stress areas may result in hydrogen embrittlement [13]. The high stress state raises the concentration of hydrogen and also reduces the maximum tensile strength of the material [14]. The concentration of hydrogen in conjunction with the stress state leads to the initiation of a crack, and the crack then propagates through the material [13][15]. A higher amount of hydrogen saturation will result in larger amounts of hydrogen around the crack tip. This effect will be greater in austenite than in ferrite or martensite, since hydrogen solubility is greater in austenite.
The decohesion model generally involves the nucleation and propagation of hydrogen-assisted microvoids by reducing the original tensile strength [14]. For this model, the hydrogen concentration is an essential parameter. When the hydrogen concentration exceeds a threshold value at stressed locations, it will play a role in diminishing the failure strain and leads to decohesion at lower local stresses as compared with the same conditions without hydrogen [14]. Another role of hydrogen in the decohesion model is to enlarge the mean size of microvoids, as seen by the dimple size on the fracture surface when compared to a steel without hydrogen at similar strains [14]. In general, larger microvoid size will make the fracture easier to occur. In summary, the hydrogen concentration needs to reach a certain critical value in order to activate this decohesion. This mechanism is more prevalent along grain boundaries, and at interfaces between different phases in the microstructure due to higher stress intensity factor in those regions [16].

2.1.3 Other Models Relevant to Delayed Fracture

Other models and mechanisms have also been proposed to explain hydrogen-induced delayed fracture. These mechanisms or ideas were proposed sometime ago or are for specific cases, but are worthy of a brief mention.

There is a proposed hydrogen gas model, where hydrogen atoms gather in cavities, become a gas phase during metal deformation, and cause fracture when the gas pressure reaches a critical value [13]. The stress corrosion cracking mechanism indicates that large amounts of hydrogen produced through corrosion are responsible for the hydrogen embrittlement, but this mechanism only applies under certain severe conditions [13]. These models have partially contributed to the understanding of the generally applicable mechanisms.

2.2 Factors Affecting Hydrogen Embrittlement

Hydrogen embrittlement is considered to be the main cause for delayed fracture in AHSSs. There are several main factors that can affect aspects of hydrogen embrittlement, including 1) plastic strain, 2) amount of diffusible hydrogen, 3) applied stress level, and 4) microstructure [1][2][17]. Dislocation mobility and micro-void density around the crack front are also generally considered to be factors associated with hydrogen embrittlement [8]. In the current section, the main factors that affect hydrogen embrittlement will be presented based on analyses used to explain the experimental results of several studies found in the literature. Principles to prevent or minimize the effects of delayed fracture are also discussed, as well as how they can be put into practice.
2.2.1 Plastic Strain

Experiments were conducted on DP1180 to explore the cause and effect relation of plastic strain on delayed fracture. Figure 2.1 [17] shows a schematic of the specimen used in these experiments. The distance between the two sides (d) was used to control the value of bending radius (r). In this test, a smaller radius (R) results in a higher degree of bending [17].

![Figure 2.1 Geometry of specimen and treatment applied in the experiment [17]](image)

Figure 2.1 Geometry of specimen and treatment applied in the experiment [17]

Figure 2.2 shows the relation between the time to fracture in DP1180 samples and the bending radius of the tests [17]. It was observed that there was no fracture on samples when the bending radius was larger than 4 mm, while fracture did occur on samples with bending radius of 3 mm or less [17]. In addition, the time to fracture remarkably decreased when the bending radius was decreased below 4 mm [17]. This decrease indicates that steels that undergo higher plastic strains (and also subjected to higher stress levels) are more susceptible to hydrogen embrittlement. Since the plastic deformation is a major aspect of the current study, a more detailed description of the plastic deformation examined will be provide in section 2.4.

![Figure 2.2 Plot of time to fracture versus bending radius for cold-rolled DP 1180 steel [17]](image)

Figure 2.2 Plot of time to fracture versus bending radius for cold-rolled DP 1180 steel [17]
2.2.2 Diffusible Hydrogen

The amount of diffusible hydrogen is another important factor that affects hydrogen embrittlement, since it was observed to aid crack propagation during low-strain–rate tensile tests [18]. A study was conducted on DP1180 samples to obtain the relationship between the amount of diffusible hydrogen uptake and pH level of the acid solution in which the sample was immersed. Figure 2.3 [17] shows the relationship among these parameters. It is also seen that as the time in the solution increases, the amount of diffusible hydrogen increases, until about 20 h where the amount levels off [17]. The influence of pH is not difficult to understand, since exposure to a higher concentration of hydrogen results in higher hydrogen absorption. The hydrogen absorption rate is higher when pH is equal to 1 during the initial time period in the solution.

![Diffusible hydrogen content versus dipping time in HCl for cold rolled DP 1180 steel](image)

Figure 2.3 Diffusible hydrogen content versus dipping time in HCl for cold rolled DP 1180 steel [17]

The test results shown in Figure 2.4 [17] explore a possible relationship between the dipping time in acid and the amount of diffusible hydrogen absorbed into the steel under different plastic strain levels. The diffusible hydrogen amount in samples is inversely proportional to the bending degree under the same bath conditions. At the same time, the increased stress associated with the larger bending degree also affects the amount of diffusible hydrogen. Bending deformation can have a larger degree of surface deformation than uniaxial tension, so the hydrogen amount inside a bent sample can be higher than that in a uniaxial deformed sample. The saturation means the amount of hydrogen in the steel is constant after the first 24 h of immersion into the acid solution. [17]. The possible significance of these results is that there might be a time window to control the amount of diffusible hydrogen inside the steel parts that will be in contact with acidic liquid solutions during production. Before reaching the saturation level, the smaller bending radius attains a higher amount of diffusible hydrogen [17]. The cause for the increase in hydrogen is due to the greater dislocation density and point defects within the samples with a smaller bend radius [17]. Dislocations, microvoids, and point defects are considered to be the preferential hydrogen trapping sites in several different types and grades of AHSS [17] [18].
Figure 2.4 Plot of diffusible hydrogen content versus dipping time for different bending radius under same level of pH = 3 for cold-rolled DP 1180 steel [17]

2.2.3 Stress Level and Fracture Modes

Stress is somewhat different from the two previous factors, since it influences the cracking mode and crack propagation mechanism in the steel [19]. Fractographic analysis shows that 1) quasi-cleavage (QC), 2) inter-granular (IG), and 3) micro-void coalescence (MVC) fracture surfaces are the representative modes commonly observed in the delayed fracture of AHSS [19].

Based on the fracture modes shown in Figure 2.5 [19], some similarities and differences can be observed. QC is generally seen at the start of the fracture process, and MVC appears at the end of crack propagation regardless of the yield strength of the steel [19]. The only difference between these two types of steels is in the transition region between QC and MVC, where IG cracking is observed only in the high strength steels but not in the low strength steels [20]. Intergranular type fractures should be one of the representative features observed on the fracture surface of high strength steels.

Figure 2.5 QC – IG – MVC mode of crack development for both high- and low-strength steels [19]
2.2.4 Microstructure

Mechanical properties are related to or resulting from the microstructure of the material. The martensitic phase is susceptible to hydrogen embrittlement, and the critical hydrogen concentration for hydrogen embrittlement in 1200 MPa-level martensitic steels is 4 ppm [21]. In general, the total hydrogen amount consists of both diffusible and non-diffusible hydrogen. As a result, the critical diffusible hydrogen is less than 4 ppm for martensite in dual phase (DP) and transformation induced plasticity (TRIP) steels. The martensitic microstructure is highly important to increase the strength level of steels. Tempered martensite is present in some AHSS, since it provides a relatively good combination of strength and toughness [22].

Another advantage of tempered microstructures is that they possess better hydrogen resistance than untempered microstructures for the same grades of steel [23][24]. Some of the martensite phase in TRIP steels is produced during deformation and hence is non-tempered. As such, the martensite in TRIP steels should be more susceptible to hydrogen embrittlement as compared to tempered DP steels. Microstructure and dislocation mobility play an important role in affecting hydrogen embrittlement susceptibility for steels with the same chemical composition [25].

2.3 Advanced High Strength Steels

Energy saving, environmental protection and passenger safety lead to the increasing adoption of high-strength steel sheets for auto-body members [1]. Dual-phase (DP) type steels and transformation induced plasticity (TRIP) type steels have been developed to satisfy not only the high strength, but also the high formability requirements [26]. In this section, these two types of AHSS are described, and results from previous studies are presented.

2.3.1 Dual Phase (DP) Steel

The microstructure of DP steel combines strong martensite islands dispersed in a large soft ferrite medium [27]. From a microstructure perspective, the ferrite phase generally deforms initially at a higher rate upon loading while the deformation of martensite phase is delayed [28]. When larger plastic strains are imposed on DP steels there will be deformation in both ferrite and martensite phases. For lower percent martensite in dual phase steels, deformation occurs essentially in the ferrite phase rather than in the martensite, while for higher amounts of martensite in dual phase steels, shearing between the ferrite and martensite interface can take place [28].

Ductile failure is observed to occur by the existence of micro-voids in the microstructure for single phase steels, and for DP steels with any volume fraction of martensite [28]. Cracks are observed to initiate in the martensitic phase and continue into interfaces but generally not penetrated into ferrite during
deformation [29]. There will be a larger probability of crack initiation and propagation in DP steels with larger volume fractions of martensite [29]. The grain size plays an important role in adjusting the area of interfaces where cracks can initiate, and these interfaces are also hydrogen trapping sites [30].

### 2.3.2 Transformation Induced Plasticity (TRIP) Steel

TRIP steel has retained austenite dispersed in a matrix of other phases [26]. TRIP steels have excellent formability and high strength as the retained austenite transforms into martensite under stress at room temperature as well as the increased resistance to necking during forming [31]. TRIP steels with some amount of aluminum have a higher delayed fracture strength, where the retained austenite is somewhat inhibited to transform by the addition of aluminum [32]. The retained austenite in microstructure plays a role in improving hydrogen embrittlement resistance.

The aluminum addition to TRIP steels also causes higher elongation and larger yield strength values because of a higher volume fraction of retained austenite in the microstructure [33]. The crack initiation is generally restrained by retained austenite during the strain induced transformation [34]. The martensite and austenite interface regions and martensite itself provide preferential trapping sites for hydrogen, rather than inside the austenite [30][35].

Among DP- and TRIP-type steels, TRIP steels have better elongation than DP steels, due to the original retained austenite phase present in the microstructure [26]. The transformation to martensite from austenite gradually increases the work hardening rate of TRIP steels [26]. The TRIP effect is not only the key mechanism of TRIP steels, but also provides excellent strength and formability. The ductile fracture of TRIP steels often initiates at the inhomogeneity in the microstructure between different phases rather than inside a single phase [36].

### 2.3.3 Relations between Hydrogen and AHSS

From a crystallographic perspective, BCC steels possess a more susceptibility to hydrogen degradation than FCC steels, which is due to the higher solubility of hydrogen in FCC microstructures [2]. In order to avoid hydrogen-delayed fracture (HDF), two conditions need to be met: 1) a higher solubility of hydrogen, and 2) a small amount of diffusible hydrogen, as compared to the total amount of hydrogen in steel [2]. Previous work has shown that intergranular ferrite is able to improve hydrogen-delayed fracture resistance, which has played a role as an obstacle to the crack propagation and in preventing the formation of carbides along grain boundaries of the prior austenite [37]. The morphology change of carbides from film-like to discrete leads to the decrease of the nucleation sites for cracks with a growing amount of intergranular ferrite [37].
Microstructural defect control is significant with respect to hydrogen embrittlement [38]. Some microstructural defects form reversible trapping sites with grain boundaries and dislocations as examples, while other defects are irreversible trapping sites with inclusions and voids as instances [38]. Dislocation density has a direct influence on hydrogen absorption, because diffusible hydrogen can become trapped in dislocations [38]. Dislocation movement greatly facilitates hydrogen diffusion by "taking hydrogen along with the dislocation motion", which increases a material's susceptibility to hydrogen embrittlement [38]. This phenomenon may be one reason why strain or stamping deformation can make the sample more prone to the hydrogen delayed fracture.

2.4 Sheet Metal Forming and Plastic Deformation

Sheet metal forming is an important technology in manufacturing. A significant amount of modern steels are primarily shaped as a cold-rolled sheet, and then are further manufactured for applications such as automobile and home appliance components at a lower cost [39]. A high yield strength is one of the primary advantages of advanced high strength sheet metal, which results in good stiffness and reduced weight for parts in many industries [39]. Steel not only provides sufficient strength, but also meets many of the weight requirements for final products. There are a large number of techniques to make sheet metal parts into different desired shapes and to produce different properties for multiple applications.

This section briefly reviews the primary deformation modes that are imposed during the current study. These deformation modes were designed to reflect typical but simplified deformation steps that are used in sheet metal forming processes.

2.4.1 Uniaxial Tension

Uniaxial tension is the stress state in a sample during a tensile test that deforms the metal in an uniaxial direction until the maximum load is reached [39]. Uniaxial tension has a relatively simple strain path, since there is only one direction that contains a principal stress component. In addition, there are no shear components along the primary deformation axis, and each face of the sample will stay perpendicular to one another, as in the initial conditions [39]. The uniaxial strain path is generally achieved by a tensile test on a mechanical or hydraulic test machine.

The increasing load on the sample induces deformation eventually leads to fracture. Elastic, homogeneous plastic and localized plastic are the three stages that occur before fracture during deformation [40]. Localized plastic deformation occurs after the ultimate strength until the point of fracture. The strain should be controlled at levels larger than the elastic limit to accomplish metal forming, but also lower than the forming limit to make sure the material does not break. The stress during deformation can increase the surface area but reduces the width and thickness, and this eventually leads to necking [41]. In an ordinary
tensile test, the stress can be calculated by using load divided by cross sectional area, and both engineering and true stress values can be determined. It is important to take the change in material properties during deformation into consideration when performing failure analysis in metals. Both the stress level and the strain path can have an effect on the failure [42].

The strain imposed during uniaxial tension is relatively uniform throughout the metal. The susceptibility to hydrogen will primarily be dependent on the amount of strain imposed.

2.4.2 Bending Under Tension Strain Path

Bending is a mode of sheet metal forming and is usually applied to form parts for the automotive industry, among others [43]. However, the large degree of deformation in the bending strain path causes necking and cracking issues in the material response behavior for AHSSs [44]. Based on the bending process, the effects of deformation on the material are more complex especially near the two surfaces.

During bending under tension, the upper surface of the sheet experiences tension during the initial bending onto the roller or die. Then the upper surface is compressed during the unbending step, when it comes off the roller or die. In contrast the lower surface sees compression during the initial bend onto the roller or die followed by tension during the unbending. When back tension is applied during bending, the neutral plane is located below the mid-thickness of the sheet, closer to the tooling. During the unbending the neutral plane is located above the mid-thickness closer to the top side of the sheet. The back tension present during the bending process strongly affects the offset of neutral plane away from the mid-thickness. Because of this neutral plane offset difference between bend and unbend, the strain in the center region of sheet is tensile for both the bend step and the unbend step. The upper region of the sheet has a strain path of tension followed by compression, and the lower part of the sheet has a strain path of compression followed by tension. The overall local effective strain will be higher in both surface regions of the sheet as compared to the center region.

The bending deformation creates higher effective strain near the surfaces of the sheet. These higher strains can allow absorption of larger amounts of hydrogen as compared to the center region of the sheet [45].

2.5 Effects of Strain Path on AHSS

Strain path has effects on the substructure and arrangement of dislocations, which can cause differences in mechanical behavior [46][47]. Apart from these influences, the dislocation density is also affected by different strain paths. The dislocation density will increase with straining, and this increase in density will make dislocations more difficult to move during the subsequent deformation steps [48][49]. The direction of the load or the imposed deformation direction during straining can make a difference in the
material response to further processing. A load applied in the opposite direction to the initial loading direction will lower the yield strength, due to the Bauschinger effect [47]. During uniaxial tension the strain is only in tension and uniform throughout the sample. During bending under tension, the upper and lower surfaces of the sheet experience different strain paths: compression after tension for the upper surface and tension after compression for the lower surface.

2.5.1 Dual Phase Steels

Both ferrite and martensite contribute to the overall mechanical behavior of dual-phase steels. In general, the overall mechanical properties of dual-phase steels are dependent on the fraction of the martensite phase, martensite morphology, the hardness of martensite with its specific carbon content, and ferrite grain size [50][51]. For DP780 and DP980 steels, the volume fraction of martensite plays a more important role in determining the strength of these samples than martensite hardness. Another aspect to consider is that martensite is a harder but more brittle phase. In general, the strain path is likely to affect the interfaces between martensite and ferrite. Decohesion between ferrite and martensite usually leads to crack initiation and the fracture propagates through martensite or even penetrates into adjacent ferrite [52]. Dislocation density is generally increased in the boundary region between the ferrite and martensite, and also in the ferrite adjacent to the martensite [47][53]. The increased volume fraction of martensite results in a higher dislocation density in surrounding ferrite since during the deformation the plastic strain is primarily accommodated in the ferrite [30]. Also, dislocation density increases when austenite transforms to martensite during thermal processing to produce DP steels.

2.5.2 Transformation Induced Plasticity Steels

The increased volume of martensite transformation from austenite usually leads to dislocation initiation and high residual stresses in phase boundary regions [46]. The stress partitioned to the martensite phase becomes higher and the stress partitioned to the austenite phase becomes lower during deformation, while no changes occur for stress partitioned to ferrite or bainite [54].

The austenite is easier to transform to martensite with more imposed plastic strain, a larger value of the triaxiality stress state and a higher strain rate [55][56] [57]. The transformation rate from austenite to martensite decreases when the value of strain imposed on TRIP steels becomes larger, and the rate during plane-strain deformation is faster than that with uniaxial tension [58]. TRIP steels possess better formability due to the balance between less hardening at the interface between ferrite and austenite and the improved stiffness of the martensite transformation [59]. The void initiation at interface regions between different phases is inhibited with an increasing volume fraction of retained austenite and higher amounts of bainite in the microstructure [59].
2.5.3 Strain Effects on Hydrogen Embrittlement in DP and TRIP Steels

The volume fraction of microstructural constituents plays an important role in resistance to hydrogen embrittlement. The ferrite grains exhibit larger elongation at high strain rates for TRIP steels but at low strain rates for DP steels under the same applied stress [57]. The 780 MPa strength grades of both DP- and TRIP-type steels have better formability and resistance to ductility loss, as compared to the 980 MPa grades. Dislocation density increases in the ferrite phase adjacent to retained austenite and fresh martensite that is formed from austenite in TRIP steels during straining [60]. The strain and dislocation density combine to make interface regions between ferrite, austenite, and martensite into key factors that affect hydrogen embrittlement in both DP and TRIP steels.

2.6 The Bauschinger Effect

The Bauschinger effect occurs during a stress reversal. It is a manifestation of kinematic hardening. Upon the reversal, the yield point is lower than would be expected. During the reversal, some of the dislocation pile ups at boundaries are relieved and there can also be some dislocation annihilation [53]. Both of these processes will cause the yield point to occur at a lower stress level than would be expected if the deformation were to have continued without the reversal.

The Bauschinger effect increases with increasing strain applied in medium carbon steels. The magnitude of the Bauschinger stress in compression first scenario is larger than that in tension first scenario [61]. However, the Bauschinger effect decreases in steels, and eventually saturates after a certain amount of strain [61]. The Bauschinger effect will reach a threshold value and not increase any more when the imposed strain is sufficiently large. For DP steels with the volume fraction of martensitic phase larger than 20%, a lower Bauschinger effect will be observed because of the shorter distance between martensite regions [53].

2.7 Hydrogen in Steel

Since the materials used in this study are DP and TRIP steels, the hydrogen solubility and diffusivity in different phases are noted here. The ratio of solubility of hydrogen in austenite over ferrite is around $2.5 \times 10^3$, but the diffusivity values in ferrite, austenite, and martensite at room temperature vary and are shown in Table 2.1 [30][62][63][64][65]. There are some other factors which can affect diffusivity, such as temperature. Dislocation density created during deformation is able to boost the diffusivity of hydrogen in steels, but the contradicting fact is this will also increase hydrogen trapping sites, which generally reduces diffusivity [30]. As a consequence, the effective hydrogen diffusivity in steel is determined by the balance of dislocation density and degree of deformation.
Table 2.1  Order of Magnitude Values for Hydrogen Diffusivity in Different Phases at Room Temperature

<table>
<thead>
<tr>
<th></th>
<th>Ferrite</th>
<th>Martensite</th>
<th>Austenite</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diffusivity (cm²)/s</td>
<td>10⁻⁴</td>
<td>10⁻⁸</td>
<td>10⁻¹²</td>
</tr>
</tbody>
</table>

Hydrogen induced cracks within martensite are normally found to originate along prior austenite grain boundaries where there is a high hydrogen concentration [66]. For smaller prior austenite grain sizes the decreased grain size leads to increasing grain boundary regions, which leads to higher crack initiation probability and intergranular fracture patterns that reflect the smaller grain size. Among boundaries between grains or phases, incoherent interface regions are considered as preferential hydrogen trapping sites due to the larger mismatch between the crystals [67]. The morphology of the phase plays a greater role in affecting hydrogen diffusion than phase size [68]. Coarse phases provide more hydrogen trapping sites than fine phases for longer times because of the presence of hydrogen concentration gradient in coarse phases [68]. Coarse phases generally lead to lower strength level and smaller diffusivity of hydrogen. Hydrogen can play an important role in increasing dislocations and slip plane reactions, which significantly increases vacancy density and development of voids [69].

2.8  Fracture Due to Hydrogen

Decohesion and fracture in multiphase steels often result in void formation along ferrite and martensite interfaces, and continuous void formation may lead to necking or even fracture [70]. The degradation of cohesive strength between the phases in AHSSs is considered to be one of the most important causes of delayed fracture. Knowledge of the effect of hydrogen on the cohesive strength is important, especially for modeling and theoretical computations. After void and micro-crack formation growth, the coalescence and interaction of these defects will occur and lead to the final fracture.

The dislocation structure of the metal strongly influences the hydrogen effect and has been the focus of previous studies. Dislocations are considered as the preferential hydrogen trapping sites, especially the ones newly formed after deformation [30]. There is also an obvious transition of the fracture surfaces from void coalescence to cleavage, intergranular, and even transgranular, when contaminated with certain amounts of hydrogen [71] [72]. The fraction of intergranular or transgranular fracture features is proportional to the amount of diffusible hydrogen charged into the sample, and this phenomenon is also applicable to DP steels [71] [72].

In some cases, it is difficult to observe an obvious crack on the surface, while some void coalescence or even cracks may be present beneath the surface [52]. The fracture behavior will become more brittle with the increasing amount of martensite in the microstructure for both DP and TRIP steels [49].
The final fracture pattern is related to several factors such as the stress state, lateral constraints and overall triaxiality [73].
CHAPTER 3  EXPERIMENTAL DESIGN

This chapter outlines the objective and design ideas of this project. The basic methodology to effectively assess delayed fracture susceptibility and experimental approaches are also included.

3.1 Project Objective

A reproducible methodology to test and evaluate the susceptibility of delayed fracture for multiple grades of AHSSs (DP780, DP980, TRIP780 and TRIP980) after different modes of prior deformation was explored. Since hydrogen rapidly escapes from steel at room temperature, a consistent methodology that incorporates both hydrogen charging and delayed fracture assessment was developed. For the selected grades of AHSSs, an experimental analysis was performed that focused on the time to fracture for a given stress level at a constant hydrogen concentration. Micro-hardness and fractography of the samples was also performed.

3.2 Experimental Approach

Based on previous experiments performed on hydrogen embrittlement of AHSSs, the present analysis considers strain level, diffusible hydrogen amount, stress level, and fracture surface details. Among these factors, strain and stress have a defined relationship when deformed in uniaxial tension (engineering stress versus strain curve). Diffusible hydrogen amounts needed to be fixed at the same level, and as a result, the DP and TRIP samples were tested at different charging conditions to obtain the same level and amount of diffusible hydrogen. A goal of the present analysis was to isolate the effect deformation modes have on the delayed fracture susceptibility of AHSSs.

Samples were strained by either tensile or bending deformation, charged with hydrogen, and tested on a three-point bending test setup. Nickel coating was selected to retain diffusible hydrogen inside the sample, but a significant portion of the hydrogen escaped during the electroless coating process, due to the high temperatures needed for plating. As a result, the coating process was omitted from the methodology, and higher concentrations of hydrogen were charged into the sample in order to keep hydrogen above a certain level for a longer time. Additionally, specimen images were taken at different stages of the experimental testing to observe the evolution of cracking.

3.3 Deformation Processes

Samples were hydrogen-charged in the undeformed (as-received), uniaxial tensile deformed, and bending deformed conditions. Uniaxial tensile deformation was achieved using an electromechanical load
frame, and bending deformation was achieved using a modified servo-hydraulic frame, in which 2 actuators were situated 90° relative to one another. Following these deformation steps, grinding and ultrasonic cleaning were applied to the sample prior to hydrogen charging. There were two grinding steps with 240 grit sandpaper: one was to make the surface condition consistent for all samples, and the other was to eliminate the “shear effects” on the side edges of the samples. Ultrasonic cleaning was performed mainly to eliminate impurities from the sample surface prior to hydrogen charging.

### 3.4 Hydrogen Charging and Measuring Method

According to previous studies, high-pressure gaseous and electrolytic charging are the two standard methods to charge hydrogen into samples [30]. The electrolytic charging method was selected for the current project due to its simple setup, reproducibility, and minimal temperature effects (as it maintains the as-received microstructure more effectively [30]), as compared to the high-pressure gaseous charging method. Following these steps, the LECO® analyzer was used to measure the total amount of hydrogen in the steel samples. Based on literature and prior work, the amount of diffusible hydrogen has a greater influence on delayed fracture susceptibility than the total amount of hydrogen. As a result, two types of hydrogen measurements (water heating and air exposure) were made to obtain the amount of diffusible hydrogen inside the samples.

### 3.5 Selection of Stress and Strain Levels

Several techniques have been used to characterize delayed fractures, such as the U-bend test, continuous charging tensile test, and cup drawing test [2][22][74]. U-bend and cup drawing tests are far more severe than other approaches, and continuous charging does not accurately simulate many real world situations. To better simulate situations in the auto industry, the three-point bend test was used in the current project. The three-point bend test was used to evaluate delayed fracture, based on the relationship between the time to fracture and each deformation mode of four types of AHSS grades. 120% yield strength of each steel from the engineering stress versus strain curve was selected as the stress level for delayed fracture test, and the corresponding strain level was used to apply this stress during the three-point bend test.

### 3.6 Assessment of Delayed Fracture Susceptibility

Since delayed fracture is closely related to the factor “time,” the time to fracture is considered to be the most important parameter to evaluate a sample’s susceptibility to delayed fracture. The time to fracture consists of the time of crack initiation, propagation, and stabilization. Besides the obvious crack (for some cases) that may be observed on the surface, there is a certain amount of micro cracks branching from the primary crack. These secondary cracks were also examined through SEM and were used as a qualitative
assessment of the susceptibility to delayed fracture. At times, only micro cracking was observed and no primary cracks were present in the tested sample. Microstructural and fractographic images were also used to evaluate strain path effects and fracture surface details. Micro hardness profiles across both width and thickness directions were obtained as a quantitative assessment of the uniformity of the samples.
CHAPTER 4  EXPERIMENTAL METHODS

This chapter outlines the application of design ideas to actual experimental tests, and the improvement in methodology to effectively assess delayed fracture susceptibility. The AHSS materials used, as well as the equipment employed in the project, are explained, with a focus on the ultimate objective of the current study.

4.1 Materials

Four types of AHSS steels, a dual phase (DP) and a transformation-induced plasticity (TRIP) steel with a minimum ultimate tensile strength (UTS) of 780 MPa, and similar steels with a minimum UTS of 980 MPa, were selected for this study. Table 4.1 lists the compositions and Table 4.2 presents the dimensions and coating conditions of the four steels. Among these four steels, only DP780 was galvanized annealed (GA) and the other three were all uncoated.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Ti</th>
<th>Al</th>
<th>P</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td>DP780</td>
<td>0.137</td>
<td>1.95</td>
<td>0.02</td>
<td>0.01</td>
<td>0.243</td>
<td>-</td>
<td>-</td>
<td>0.002</td>
<td>0.068</td>
<td>0.023</td>
</tr>
<tr>
<td>DP980</td>
<td>0.158</td>
<td>1.69</td>
<td>1.086</td>
<td>0.011</td>
<td>0.022</td>
<td>-</td>
<td>-</td>
<td>0.023</td>
<td>0.052</td>
<td>0.014</td>
</tr>
<tr>
<td>TRIP780</td>
<td>0.19</td>
<td>1.59</td>
<td>1.63</td>
<td>-</td>
<td>0.03</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0.036</td>
<td>0.013</td>
</tr>
<tr>
<td>TRIP980</td>
<td>0.24</td>
<td>1.69</td>
<td>2.13</td>
<td>-</td>
<td>&lt;0.01</td>
<td>-</td>
<td>-</td>
<td>0.64</td>
<td>0.008</td>
<td>0.001</td>
</tr>
</tbody>
</table>

Table 4.2  Specimen Dimensions (in mm) and Coating Conditions of Different Deformation Modes

<table>
<thead>
<tr>
<th>Coat</th>
<th>Length</th>
<th>Width</th>
<th>Thickness</th>
<th>After Uniaxial Tension</th>
<th>Length</th>
<th>Width</th>
<th>Thickness</th>
<th>After Bending</th>
</tr>
</thead>
<tbody>
<tr>
<td>GA</td>
<td>30</td>
<td>13</td>
<td>1.0</td>
<td>30</td>
<td>12.58</td>
<td>0.98</td>
<td>30</td>
<td>12.62</td>
</tr>
<tr>
<td>N</td>
<td>30</td>
<td>13</td>
<td>1.6</td>
<td>30</td>
<td>12.55</td>
<td>1.56</td>
<td>30</td>
<td>12.57</td>
</tr>
<tr>
<td>TRIP780</td>
<td>30</td>
<td>13</td>
<td>1.2</td>
<td>30</td>
<td>12.50</td>
<td>1.18</td>
<td>30</td>
<td>12.55</td>
</tr>
<tr>
<td>TRIP980</td>
<td>30</td>
<td>13</td>
<td>1.2</td>
<td>30</td>
<td>12.60</td>
<td>1.19</td>
<td>30</td>
<td>12.60</td>
</tr>
</tbody>
</table>

All investigated steels reported in Table 4.1 were previously used by Alex Hudgins and Joe Ronevich [30] [75]. The steels have carbon contents ranging from 0.137 to 0.24 wt pct, and manganese (Mn) contents ranging from 1.59 to 1.95 wt pct.

Undeformed and uniaxial tensile strained samples were cut from a large sheet and sheared to smaller pieces with an area of 50.8 mm x 12.7 mm (2 in x 0.5 in). The bending strained samples were
sheared to a width of 12.7 mm (0.5 in) and to varying lengths, based on the geometry of samples before deformation. After deformation, testing samples with dimensions of 50.8 mm x 12.7 mm (2 in x 0.5 in) were extracted from the bending under tension sample and were hydrogen charged for delayed fracture testing. Due to the size requirements of the bending under tension test apparatus, the sample dimensions to perform the test are fairly long (minimum length is 35 cm). A small flat piece needs to be cut from the tested sample to match the sample size for other deformation modes, in order to be tested for delayed fracture in a comparable manner. Due to the deformation process applied to the samples, there are some changes in thickness and width. In general, the width and thickness were slightly reduced, as compared to the as-received condition. Figure 4.1 shows the top view of the large sheet steel sample and the orientation of sample extraction with the rolling direction. The uniaxial and bending samples were sheared out of the large sheet.

![Figure 4.1 Rolling direction of as-received sheet metal sample and experimental sample extraction orientation and method](image)

4.2 Pretreatment

The surface of each sample was ground with 240-grit sand paper in the rolling direction, in order to remove surface irregularities and to make all surfaces consistent enough to be comparable. Due to the results from preliminary tests, a method to eliminate “edge effects” was applied. This concern arises from the shearing of samples from a larger sheet and the preliminary results that show crack initiation always initiating at edges. Cross-sectional microstructure images of the edges of samples were analyzed to determine the size of the edge shear affected zone, and this region was removed. Figure 4.2 shows the hardness change with increasing distance from the edge to the center of the sample perpendicular to the thickness direction. According to Figure 4.2, 0.3 mm for 780 grade samples and 0.4 mm for 980 grade samples from the sample edge are considered to be shear affected zones, and this region needs to be eliminated by grinding with water (to avoid overheating the sample). Besides eliminating the edge effect zone, the sharp corners were also ground with 240-grit sand paper to reduce stress concentration in these regions. Ultrasonic cleaning with acetone was performed after the grinding process to clean the samples, prior to hydrogen charging.
4.3 Deformation

Uniaxial tensile deformation was achieved using an electromechanical frame, and bending deformation was achieved using a modified servo-hydraulic frame, in which 2 actuators situated 90° relative to one another subjected a sheet specimen in tension while being bent over different dies. A restraining force equal to 80% yield strength was applied to one actuator, while the other actuator simultaneously pulled the sheet steel over the die, “rolling over” a 50 to 75 mm section of the sheet. Figure 4.3 (a) shows the setup of the bending test equipment. Tests were performed for several samples in different conditions, in order to determine a suitable and consistent deformation. For the uniaxial strained samples, the percentage of strain was calculated based on the thickness strain value that was obtained from the bending tests.

Figure 4.3 (b) shows a schematic of where the sample was extracted after the strip was bent over the roller. Both upper and lower surfaces of these samples undergoing bending were evaluated by the delayed fracture test. The upper surface is referred to as the "concave up" condition and the lower surface as the "concave down" condition. The concave up surface was initially stretched during the bending onto the roller and compressed upon unbending. It was then stretched during the delayed fracture test. In contrast the concave down condition was initially compressed followed by stretching during the bending and unbending on the roller. It was subsequently stretched during the delayed fracture test. Because of these differences in strain paths, both surfaces were tested.

Due to the different thicknesses of the samples, different radii of bending dies were used for the bending test in order to maintain the same radius to thickness ratio (R/t ≈ 8). The bending radii (i.e. roller radii in the bending under tension test) are 7.9 mm (5/16 in), 9.4 mm (6/16 in) and 12.7 mm (8/16 in) for
sheet thicknesses of 1.0 mm, 1.2 mm, and 1.6 mm, respectively. The formula used to calculate effective strain of the bending samples is as following [39]:

\[ \varepsilon_{\text{eff}} = \frac{2}{\sqrt{3}} (\varepsilon_{\text{thickness}} - \varepsilon) \]  

Equation 4.1

The uniaxial deformation used in the prior deformation was consistent with the bending deformation by setting:

\[ \varepsilon_{\text{tension}} = \varepsilon_{\text{eff}} \]  

Equation 4.2

The extensometer used during the tensile deformation was 10 mm, with 15 pct maximum extension. The gauge length of the sample was 25.4 mm (1 in). The deformation was performed at a constant displacement rate of 0.0254 mm/s (0.001 in/s). Bending strain is calculated based on the change of thickness before and after the bending test, while the uniaxial strain is calculated based on the effective strain formula. Table 4.3 lists the strain applied on both uniaxial and bending cases in order to maintain the same effective strain. The strain is calculated based on the change in thickness with respect to original thickness, and uniaxial strain is based on the effective strain. The strain values in this table are used as the reference for the deformation step, and all detailed calculations are listed in Appendix I.

![Figure 4.3 Bending tests (a) front and side view of the set up and (b) schematic of sample after bending deformation and extracted region for further tests.](image)

**Table 4.3 Strain Applied on Both Uniaxial and Bending Cases in the Four Steels**

<table>
<thead>
<tr>
<th>(mm/mm)</th>
<th>DP780</th>
<th>DP980</th>
<th>TRIP780</th>
<th>TRIP980</th>
</tr>
</thead>
<tbody>
<tr>
<td>Uniaxial Strain</td>
<td>0.059</td>
<td>0.056</td>
<td>0.063</td>
<td>0.050</td>
</tr>
<tr>
<td>Bending Strain</td>
<td>-0.051</td>
<td>-0.049</td>
<td>-0.055</td>
<td>-0.043</td>
</tr>
<tr>
<td>Effective Strain</td>
<td>0.059</td>
<td>0.056</td>
<td>0.063</td>
<td>0.050</td>
</tr>
</tbody>
</table>
4.4 Hydrogen Charging

After deformation, hydrogen was electro-chemically charged into the sample with sulfuric acid solution, and the time to fracture and size of crack after a three-point test were measured to evaluate the susceptibility of the sample to delayed fracture. Detailed information about the crack was determined from photographic images taken using a high-resolution camera.

The electrolytic charging method was used for this project with a direct current DC power source, and a configuration of the test setup is shown in Figure 4.4. A DC power supply with a maximum voltage of 30V was used to provide varying currents up to 3A based on the different surface areas of the sample. A graphite bar was used as an anode, and the sample to be charged was the cathode. The current density and applied voltage were adjusted according to the size and shape of the sample, while the current is set constant. The samples were hydrogen-charged in a 1N sulfuric acid (H₂SO₄) solution with 10 mg arsenic trioxide (As₂O₃) per liter. According to Ronevich [30], As₂O₃ has the ability to inhibit the recombination of hydrogen ions to hydrogen gas. The amount of solution was determined by the surface area of the samples, approximating 6 ml/cm². A current density of 10 mA/cm² was used to charge the samples. In order to make all the samples have the same level of diffusible hydrogen, the duration of hydrogen charging was dependent on the measured amount of diffusible hydrogen. The details of the measurement and the corresponding data are presented in a later section.

![Figure 4.4](image_url)

Figure 4.4 Front view of the hydrogen charging set up with anode (graphite bar), cathode (sample) and electrolyte (sulfuric acid solution with arsenic trioxide)

4.5 Hydrogen Analysis

Sample coupons measuring 12.7 mm×5 mm×1.27 mm were used for hydrogen analysis. Before testing, the hydrogen analyzer was charged with argon gas for at least one hour and then calibrated with 5.7 ± 0.6 ppm calibration samples in order to ensure the accuracy of the results. Residual moisture on hydrogen-charged companion sample surfaces was removed using a tissue, prior to hydrogen content analysis. Next, companion samples with a 5 mm width were cut from samples to perform hydrogen analysis.
Test samples would yield the most accurate results when their weights were close to calibration samples (1g), so 5mm was the most suitable width to choose.

Measurements from the LECO® analyzer (Figure 4.5) for hydrogen were applied to obtain the diffusible hydrogen amount. As a result, several measurements of hydrogen were recorded, including the original value (immediately after hydrogen charging) and the final stable value (after a certain period, the amount does not change). The difference between these two values was considered to be the diffusible hydrogen amount in the sample. Hot water heating and air exposure at room temperature were the two ways to make hydrogen diffuse out of the specimen as much as possible. Based on the preliminary test results, the water heating method is faster, but can cause some "non-diffusible" hydrogen to become diffusible at this higher temperature condition. Similarly, the air exposure method may make some "diffusible" hydrogen become stable in permanent trapping sites. As a result, the average of both measurement methods is interpreted to represent the diffusible hydrogen content for this study. The amount of diffusible hydrogen inside each sample was controlled to be at a similar level, in order to make the results comparable. This analysis method also characterizes the relationship between the amount of diffusible hydrogen inside the samples and the charging time for the different types of steel.

Figure 4.5  Front view of the LECO hydrogen analyzer equipment with control panel (left) and melting furnace (right)

4.6  Three-Point Bend Test

After the sample was deformed, hydrogen charged, and analyzed (measurement takes place on a companion sample), it was tested on the three-point bend test. Figure 4.6 shows the setup and a schematic of the three-point bend test with dimensions. The setup is relatively simple and reproducible; the three-point test equipment is comprised of a bolt, a roller and a strut. A cylinder was put between the bolt and the sample, since the cylinder has a larger contact area with sample, and it makes the applied force more
uniform across the width. The sizes of the cylinders are selected dependent on the sample thicknesses in order to make different cases comparable, which were 7.9 mm (5/16 in), 9.4 mm (6/16 in) and 12.7 mm (8/16 in) of diameters for sheet thicknesses of 1.0 mm, 1.2 mm, and 1.6 mm, respectively. The radius to thickness ratio for this case is approximately 4.

![Figure 4.6 Schematic of the three-point test setup with dimensions of core parts.](image)

It was initially expected that the stress values could be determined based on the stress-strain curves of all four steels and the readings from a strain gauge attached to the sample surface. From preliminary tests it was determined that the elastic bending did not supply a large enough stress on the outer surface for a crack to develop within a reasonable period of time. Consequently, 120 pct of the YS was chosen for each steel in the three-point test, and the corresponding strain was controlled via the CEA-06-125UN-120 strain gauge to reach the desired value of stress. Table 4.4 shows the 120 pct YS and the corresponding strain for all the steels after charging.

Table 4.4 Strain Applied on Both Uniaxial and Bending Cases and Effective Strain in the Four Steels

<table>
<thead>
<tr>
<th></th>
<th>DP780</th>
<th>DP980</th>
<th>TRIP780</th>
<th>TRIP980</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield Strength (MPa)</td>
<td>536</td>
<td>680</td>
<td>500</td>
<td>574</td>
</tr>
<tr>
<td>120 pct YS (MPa)</td>
<td>643</td>
<td>816</td>
<td>600</td>
<td>689</td>
</tr>
<tr>
<td>Corresponding Strain (pct)</td>
<td>1.6</td>
<td>1.8</td>
<td>2.0</td>
<td>1.8</td>
</tr>
</tbody>
</table>

4.6.1 Stress Control

Before the “120 pct YS” standard was chosen, several other stress values were tried, such as 80 pct of YS and YS. However, the stresses at 80 pct YS and YS were insufficient to fracture the sample before hydrogen escaped. The 120 pct YS proved to work most effectively to observe surface cracks and to momentarily delay specimen fracture after the load was applied. For TRIP780 and TRIP980 steels, the stress-strain curves used came directly from Ronevich’s thesis [30], in Figure 4.7 [30], since exactly the
same materials were used. For DP780 and DP980 steels, stress strain curves were obtained by tensile tests with different hydrogen amounts inside, as shown in Figure 4.8. 120 pct YS values were calculated and found from these curves as standards for the three-point bend tests.

![Figure 4.7](image1)  
(a) Stress versus strain curve in TRIP780 (a) and TRIP980 (b) samples with various hydrogen levels [30].

![Figure 4.8](image2)  
(a) Stress versus strain curve in DP780 (a) and DP980 (b) samples without hydrogen and with 3.5 ppm hydrogen.

These curves are used as the reference of stress-strain behavior for all samples involved in the current project. These tests provide information about the delayed fracture in all four steels, under both uncharged and charged hydrogen conditions.

4.6.2 Strain Control

Based on the stress versus strain curve, the corresponding strain to 120 pct YS for the delayed fracture test was also fixed (as shown in Table 4.4). In order to make the stress on the surface at 120 pct YS, the strain on the surface should be equal or close to the strain that corresponds to 120% YS. The most
accurate strain control method was to use the strain gauge on the surface and obtain the desired value. The type of strain gauge used to check the method for uniformity and to obtain the appropriate strain level was CEA-06-125UN-120. To examine the uniformity of strain across the width of the test sample, three strain gauges were separately attached to a sample surface, and the samples were bent with the delayed fracture fixture. The strain on two edges and middle part of sample surface were all tested and plotted in Figure 4.9 (a). Since some cracks during preliminary testing were observed to be at a non-90° angle to the edge, the strain uniformity along the width is important to insure a uniformity of stress across the sample during the delayed fracture test. For each of these four specimens, the relationship between strain readings and deflection were obtained as reference. Figure 4.9 (b) presents the strain values on the surface versus deflection for all four steels. According to the results shown in Figure 4.9 (a) and (b), the uniformity is reasonable with the use of the cylinder between the bolt and the sample. Deflection was the only parameter adjusted to control the strain and stress level on the surface.

![Figure 4.9](image-url)

Figure 4.9 Strain on the surface versus deflection in DP780 at two edges and the middle part (a) and all four steels samples (b).

4.6.3 Assessment of Delayed Fracture Susceptibility: Micro-hardness

Micro hardness was measured and recorded for all four steels (DP780, DP980, TRIP780 and TRIP980) in both surface and thickness directions under no deformation, uniaxial and bending deformation treatment. Micro hardness is a good parameter to assess the effects of strain paths on materials, since it is an easy measurement, as compared to assessing directly microstructural changes. All the samples without deformation and with uniaxial and bending deformation were all sheared, mounted, ground, and polished to be ready for the micro hardness test. The variations between hardness values for the same material, same grade level, same type, and same deformation modes were compared.
4.6.4 Assessment of Delayed Fracture Susceptibility: Crack details

A standard assessment of delayed fracture susceptibility was conducted for samples that fractured during three-point bending. The most direct evaluation data were the time to fracture, which included the period from the initiation of the crack, to when the crack became stable. The time to fracture was determined using pictures from a camera taken at 1 min intervals for the first 10 min of the test, 5 min intervals for the next 20 min, and 15 min intervals until the fracture became stable. Typical timeframes ranged from 30 minutes to 180 minutes, but for most cases the crack stabilized after 60 minutes. The sample became curved after bending deformation rather than still remaining straight after uniaxial deformation. As a consequence of this result, there are two cases for bending deformed samples under three point bend test: the concave surface face up (concave up) and the convex surface face up (concave down). The second evaluation parameter was the density of the micro-cracks on the surface or secondary cracks along the primary crack. These details were obtained through high-resolution photography and SEM.

4.6.5 Assessment of Delayed Fracture Susceptibility: Fractography

Fractography was another factor used to evaluate the samples’ susceptibility to delayed fracture. In order to explore more detailed information on the cross-sectional area of the crack, scanning electron microscopy (SEM) was used for further analysis. After the time-to-fracture test, the samples were manually broken into two parts, and the fracture surfaces due to delayed cracking were analyzed by SEM, which is shown in Figure 4.10. The fracture surface profile, fracture type and any hints left to indicate crack or fracture properties were observed. Comparisons between fracture surfaces of different types of samples for the same type of steel and at different strength levels of the same type of steels were also conducted.

Figure 4.10  Schematic of the sample preparation for SEM fractographic analysis.

4.7 X-Ray Diffraction (XRD) Tests

XRD tests were performed to measure the retained austenite fraction in the as-received condition and after deformation for TRIP780 and TRIP980 steels. For the bending deformation case, both concave up and concave down conditions were tested and analyzed. The preparation of samples involved gentle
grinding on 240 sand paper to eliminate the oxide layer and chemical polishing for 20 minutes in a solution of 10 ml DI water, 10 ml hydrogen peroxide and 1ml hydrofluoric acid. The tests were performed with a Phillips X-pert diffractometer, using a copper x-ray tube. The range of scanning two theta angles was from 40° to 105°, and the step size was 0.0167 and 15.24 seconds per step. Eight peaks should be present for the best analysis (all peaks can be observed only when sufficient austenite present in the microstructure), and among these peaks are four austenite peaks ({111}, {200}, {220} and {311}). The plots were analyzed by the High Score software with profit program to obtain the areas under peaks, and these data were used to calculate the austenite fraction.
CHAPTER 5 RESULTS

This chapter presents the effects of deformation mode on the susceptibility to delayed fracture for four different AHSS. Data and calculated parameters are presented based on the experimental results. The focus is primarily on the time to fracture, crack observations and fractography.

5.1 As-received Microstructure

Figure 5.1(a) shows the three directions that are used for defining directions for the microstructural images. The metallographic images are in the plane of normal and rolling directions and are shown in Figure 5.1 (b), (i.e. perpendicular to the transverse direction) and the rolling direction is horizontal in the images. The images shown in Figure 5.2 are light optical micrographs (LOM) of the as-received steels for DP780 and DP 980. Moderate banding can also be observed in Figure 5.2. The ferrite matrix is the lighter region and martensite is the darker region. Table 5.1 shows the average ferrite grain size and volume fraction of martensite of the microstructures for DP780 and DP980. Grain size is calculated by the concentric circle methods on the middle part of microstructure images. Since there are only two phases present in the DP microstructure, the martensite fraction was calculated using ImageJ software and the ferrite fraction was the remaining fraction. The grain size in the DP980 sample is smaller than that in the DP780 samples. The higher volume fraction of martensite boosts the strength and hardness of the 980 grade samples but lowers the ductility. The images shown in Figure 5.3 are Light Optical Micrographs (LOM) of the as-received TRIP780 and TRIP 980. Moderate banding can be observed. The phases present are likely to be ferrite, martensite, austenite and/or bainite.

![Figure 5.1 Schematic of sample orientations (a) and microstructural image directions (b)](image-url)
Figure 5.2  Cross sectional microstructure images of (a) the as-received DP780 specimen and (b) the as-received DP980 specimen without strain (Etched with 2% nital).

Table 5.1  Grain Size and Martensite Phase Fraction in DP780 and 980 in the As-received Conditions

<table>
<thead>
<tr>
<th></th>
<th>DP780</th>
<th>DP980</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grain size (µm)</td>
<td>6.34</td>
<td>5.61</td>
</tr>
<tr>
<td>Martensitic fraction (pct)</td>
<td>35.6</td>
<td>48.5</td>
</tr>
</tbody>
</table>

Figure 5.3  Cross sectional microstructure images of (a) the as-received TRIP780 specimen and (b) the as-received TRIP980 specimen without strain (Etched with 2% nital).

5.2  Tensile Properties

Figure 5.4 shows the engineering stress-strain curves for DP780 and 980 in the as-received condition, as well as after charging with 3.5 ppm of hydrogen. DP980 shows a small amount of yield point elongation, while the DP780 presents continuous yielding. Figure 5.5 shows the engineering stress-strain curves obtained by Ronevich [30] for TRIP780 and TRIP980 in the as-received condition, as well as those
charged with different amounts of hydrogen. Both TRIP780 and TRIP980 samples show no yield elongation. Figures 5.4 and 5.5 illustrate the effects of hydrogen on the mechanical properties of both DP and TRIP steels. A significant reduction in elongation after H-charging is observed. Table 5.2 gives the uniform elongation (UE) of both uncharged and charged specimens, from which the reduction is clearly seen. Based on the required stress level (120 pct of YS) and stress versus strain curve, the corresponding strain value can be obtained. With the strain gauges on the sample surface, the corresponding deflection values are recorded and shown in Table 5.3. It presents the strain on the surface and the corresponding deflection needed for the delayed fracture tests; namely, stress at 120 pct of the yield strength. The tensile axis and bending direction are the rolling direction for all mechanical tests in this study.

![Figure 5.4](image1.png)

**Figure 5.4** Stress versus strain curve in the DP780 (a) and DP980 (b) samples, both without hydrogen and with 3.5 ppm hydrogen.

![Figure 5.5](image2.png)

**Figure 5.5** Stress versus strain curve in the TRIP780 (a) and TRIP980 (b) samples, both without hydrogen and with a certain amount of hydrogen [30].
Table 5.2 Uniform Elongation (in pct) for the Four Steels in Both Uncharged and 3.5 ppm Diffusible Hydrogen-charged Conditions [30].

<table>
<thead>
<tr>
<th>UE Strain</th>
<th>DP 780 Uncharged</th>
<th>DP 780 Charged</th>
<th>DP 980 Uncharged</th>
<th>DP 980 Charged</th>
<th>TRIP 780 Uncharged</th>
<th>TRIP 780 Charged</th>
<th>TRIP 980 Uncharged</th>
<th>TRIP 980 Charged</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10.0</td>
<td>2</td>
<td>9.0</td>
<td>2</td>
<td>30.0</td>
<td>7.0</td>
<td>13.0</td>
<td>7.0</td>
</tr>
</tbody>
</table>

Table 5.3 Strain and Deflection Values of Four Samples for Delayed Fracture Tests

<table>
<thead>
<tr>
<th></th>
<th>DP 780</th>
<th>DP 980</th>
<th>TRIP 780</th>
<th>TRIP 980</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surface Strain (pct)</td>
<td>1.6</td>
<td>1.8</td>
<td>2</td>
<td>1.8</td>
</tr>
<tr>
<td>Deflection (mm)</td>
<td>1.7</td>
<td>1.7</td>
<td>1.8</td>
<td>1.9</td>
</tr>
</tbody>
</table>

5.3 Micro Hardness

In order to assess the effects of deformation mode on the material, micro hardness testing along the surface and through the thickness was conducted for each material and for each deformation condition. Three sets of tests were performed for each sample under each condition, so the hardness obtained were the average values of three data set with standard deviation ranging from 10 to 21 HV. The results in this section are plots of hardness that are related to the strain distribution, especially through the thickness.

Figure 5.6 shows the micro hardness profile along the width of DP780 for all deformation modes—no deformation, uniaxial and bending. The hardness profiles are distinct for each of the deformation modes. The bending strain has the highest hardness, and uniaxial strain has lower hardness—but its hardness is still higher than that of the no-deformation condition.

![Micro hardness profile along the width of DP780 for all deformation modes](image)

Figure 5.6 Micro hardness profile along the width of DP780 for all deformation modes, including no deformation (ND), uniaxial (Uni), and bending (BCU-concave up and BCD-concave down) deformations
This increase in hardness is because of strain hardening due to deformation. When compared to uniaxial strain path, the bending strain path causes higher localized strain on the two surfaces which leads to higher hardness for the surface profile for DP780. Figure 5.7 shows a micro hardness profile through the thickness of DP780 for all deformation modes. Thickness direction is most relevant for the bending deformation. A “concave up shape” plot is seen for bending deformation through the thickness profile.

![Figure 5.7](image)

Figure 5.7  Micro hardness profile along the thickness of DP780 for all deformation modes, including no deformation (ND), uniaxial (Uni), and bending (Bend) deformations.

Figure 5.8 is a plot of the micro hardness profiles along the width of DP980 for all the deformation modes. The bending strain cases have the highest hardness, and the uniaxial strain case possesses a lower hardness that is still higher than that of the no-deformation condition.

![Figure 5.8](image)

Figure 5.8  Micro hardness profile along the width of DP980 with all deformation modes, including no deformation (ND), uniaxial (Uni), and bending (BCU-concave up and BCD-concave down conditions) deformations.
Figure 5.9 is a micro hardness profile along the thickness of DP980 for all deformation modes. Again, a “concave up shape” is seen for the bending deformation along the thickness profile.

Figure 5.9 Micro hardness profile along the thickness of DP980 with all deformation modes, including no deformation (ND), uniaxial (Uni), and bending (Bend) deformations

Figure 5.10 shows the micro hardness profile along the width of TRIP780 for all deformation modes. The hardness profiles are difficult to distinguish for each of the deformation modes. The bending strain and uniaxial strain conditions possess higher hardness than that of the no deformation condition. Figure 5.11 shows a micro hardness profile through the thickness of TRIP780 for all deformation modes. A “concave up shape” is seen in the bending deformation through the thickness profile.

Figure 5.10 Micro hardness profile along the width of TRIP780 with all deformation modes, including no deformation (ND), uniaxial (Uni), and bending (BCU-concave up and BCD-concave down) deformations
Figure 5.11  Micro hardness profile along the thickness of TRIP780 with all deformation modes, including no deformation (ND), uniaxial (Uni), and bending (Bend) deformations.

Figure 5.12 shows the micro hardness profile along the width of TRIP980 for all deformation modes. It is not easy to distinguish the hardness profile for the different deformation modes. The bending strain cases and uniaxial strain conditions possess higher hardness values than that of no deformation condition. Compared to the uniaxial strain path, the bending strain path creates more local strain on the two surfaces which leads to higher hardness along the surface profile. Figure 5.13 is a micro hardness profile through the thickness of TRIP980 for all deformation modes. Again, a “concave up shape” for the hardness profile corresponding to the bending deformation condition is observed.

Figure 5.12  Micro hardness profile along the width of TRIP980 with all deformation modes, including no deformation (ND), uniaxial (Uni), and bending (BCU-concave up and BCD-concave down) deformations.
Micro hardness profile along the thickness of TRIP980 with all deformation modes, including no deformation (ND), uniaxial (Uni), and bending (Bend) deformations. The hardness profiles along the surface direction were essentially flat for all four steels under all conditions. Table 5.4 shows a summary of the average hardness on the surface for each steel and each deformation mode. The hardness distribution through the thickness had an obvious concave shape distribution after the bending deformation. The hardness variations among the different deformation modes are larger for the DP steels, as compared to the TRIP steels, at both the 780 and 980 MPa strength levels.

Table 5.4  Hardness of All Samples for All Deformation Conditions

<table>
<thead>
<tr>
<th>Hardness (HV)</th>
<th>DP780</th>
<th>DP980</th>
<th>TRIP780</th>
<th>TRIP980</th>
</tr>
</thead>
<tbody>
<tr>
<td>No Deformation</td>
<td>227±17</td>
<td>259±10</td>
<td>223±12</td>
<td>262±14</td>
</tr>
<tr>
<td>Uniaxial Strain</td>
<td>249±12</td>
<td>275±14</td>
<td>256±14</td>
<td>313±17</td>
</tr>
<tr>
<td>Bending Concave Up</td>
<td>300±15</td>
<td>313±16</td>
<td>275±10</td>
<td>321±16</td>
</tr>
<tr>
<td>Bending Concave Down</td>
<td>285±14</td>
<td>323±19</td>
<td>263±21</td>
<td>332±13</td>
</tr>
</tbody>
</table>

5.4 X-ray Diffraction Tests

X-ray diffraction tests were performed on the TRIP780 and TRIP980 samples for different strain conditions in order to determine the volume fraction of retained austenite (Vol pct of RA). The as-received samples are expected to have the highest values of retained austenite for each material. The uniaxial and bending strain conditions were also tested to obtain amount of transformed austenite. Table 5.5 shows the retained austenite fraction of different strain conditions, based on XRD results. The TRIP780 samples contain a much larger amount of retained austenite fraction in the as-received condition, as compared to
TRIP980. However, all of the austenite in the TRIP980 transformed to martensite after deformation although the transformation amount is small. Larger amounts of austenite transformed to martensite with bending strain than uniaxial strain for the TRIP780 samples, but some amount of austenite still remains in the microstructure. Also, a gradient in retained austenite amount is likely due to the imposed strain gradient on the surfaces.

Table 5.5  Retained Austenite Volume Fraction of TRIP780 and TRIP980 for All Deformation Conditions

<table>
<thead>
<tr>
<th>V% of RA (pct)</th>
<th>TRIP780</th>
<th>TRIP980</th>
</tr>
</thead>
<tbody>
<tr>
<td>No Deformation</td>
<td>8.89</td>
<td>1.8</td>
</tr>
<tr>
<td>Uniaxial Strain</td>
<td>6.41</td>
<td>0</td>
</tr>
<tr>
<td>Bending Concave Up</td>
<td>3.33</td>
<td>0</td>
</tr>
<tr>
<td>Bending Concave Down</td>
<td>3.08</td>
<td>0</td>
</tr>
</tbody>
</table>

5.5  Amount of Diffusible Hydrogen

Measurements from the LECO analyzer consist of the initial amounts of hydrogen present immediately after hydrogen charging and the stable values after approximately 60 minutes. Based on the hydrogen amount values measured 60 minutes after charging, the hydrogen left in steel is considered to be trapped in sites with a high binding energy. These two values are considered to be the starting and ending points. There are two methods used to measure and calculate the amount of diffusible hydrogen: 1) water heating (80 °C), and 2) air exposure. The water heating method is faster, but may cause some "non-diffusible" hydrogen to become diffusible. In contrast, the air exposure method may cause some "diffusible" hydrogen to become stabilized in permanent trapping sites. As a result, the average value of the results from these two methods is interpreted as the diffusible hydrogen content. Three sets of tests have been performed for each condition of each type of alloy. The diffusible hydrogen content within each sample was controlled to similar levels in order to make the results comparable. The hydrogen analysis method was also used to characterize the relationship between diffusible hydrogen content and charging time. A diffusible hydrogen content of 3.5 ppm was selected as the standard for all delayed fracture. The hydrogen charging times for each steel to obtain 3.5 ppm are reported in Tables 5.6 to 5.9.

Table 5.6  Diffusible Hydrogen in DP780 Samples

<table>
<thead>
<tr>
<th>wt pct</th>
<th>No Deformation</th>
<th>Uniaxial Tension</th>
<th>Bending</th>
</tr>
</thead>
<tbody>
<tr>
<td>Charging Time</td>
<td>15 min</td>
<td>15 min</td>
<td>10 min</td>
</tr>
<tr>
<td>Water Heat Starting Point</td>
<td>5.50</td>
<td>6.31</td>
<td>6.08</td>
</tr>
<tr>
<td>Water Heat Ending Point</td>
<td>1.49</td>
<td>3.03</td>
<td>2.36</td>
</tr>
<tr>
<td>Diffusible H (water heating)</td>
<td>4.01</td>
<td>3.28</td>
<td>3.72</td>
</tr>
<tr>
<td>Air Exposure Starting Point</td>
<td>5.50</td>
<td>6.31</td>
<td>6.08</td>
</tr>
<tr>
<td>Air Exposure Ending Point</td>
<td>2.22</td>
<td>2.67</td>
<td>2.76</td>
</tr>
<tr>
<td>Diffusible H (air exposure)</td>
<td>3.28</td>
<td>3.64</td>
<td>3.32</td>
</tr>
<tr>
<td>Diffusible H Amount</td>
<td>3.65±0.21</td>
<td>3.46±0.18</td>
<td>3.52±0.18</td>
</tr>
</tbody>
</table>
Table 5.7  Diffusible Hydrogen in DP980 Samples

<table>
<thead>
<tr>
<th>wt pct</th>
<th>No Deformation</th>
<th>Uniaxial Tension</th>
<th>Bending</th>
</tr>
</thead>
<tbody>
<tr>
<td>Charging Time</td>
<td>60 min</td>
<td>30 min</td>
<td>15 min</td>
</tr>
<tr>
<td>Water Heat Starting Point</td>
<td>5.41</td>
<td>8.38</td>
<td>6.29</td>
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<tr>
<td>Water Heat Ending Point</td>
<td>1.57</td>
<td>4.89</td>
<td>2.45</td>
</tr>
<tr>
<td>Diffusible H (water heating)</td>
<td>3.84</td>
<td>3.49</td>
<td>3.84</td>
</tr>
<tr>
<td>Air Exposure Starting Point</td>
<td>5.41</td>
<td>8.38</td>
<td>6.29</td>
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<tr>
<td>Air Exposure Ending Point</td>
<td>2.33</td>
<td>4.61</td>
<td>3.20</td>
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<tr>
<td>Diffusible H (air exposure)</td>
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<td>3.77</td>
<td>3.10</td>
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<td>Diffusible H Amount</td>
<td>3.46±0.17</td>
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Table 5.8  Diffusible Hydrogen in TRIP780 Samples

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<th>Uniaxial Tension</th>
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</tr>
</thead>
<tbody>
<tr>
<td>Charging Time</td>
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<td>15 min</td>
<td>15 min</td>
</tr>
<tr>
<td>Water Heat Starting Point</td>
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<td>5.54</td>
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<td>Water Heat Ending Point</td>
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<td>4.06</td>
<td>4.38</td>
</tr>
<tr>
<td>Air Exposure Starting Point</td>
<td>8.69</td>
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<td>5.54</td>
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<tr>
<td>Air Exposure Ending Point</td>
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<td>Diffusible H (air exposure)</td>
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<td>2.55</td>
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<td>Diffusible H Amount</td>
<td>3.77±0.23</td>
<td>3.77±0.22</td>
<td>3.47±0.18</td>
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</table>

Table 5.9  Diffusible Hydrogen in TRIP980 Samples

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<th>wt pct</th>
<th>No Deformation</th>
<th>Uniaxial Tension</th>
<th>Bending</th>
</tr>
</thead>
<tbody>
<tr>
<td>Charging Time</td>
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<td>30 min</td>
<td>15 min</td>
</tr>
<tr>
<td>Water Heat Starting Point</td>
<td>7.35</td>
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<td>6.59</td>
</tr>
<tr>
<td>Water Heat Ending Point</td>
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<td>2.53</td>
</tr>
<tr>
<td>Diffusible H (water heating)</td>
<td>3.15</td>
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<td>4.06</td>
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<tr>
<td>Air Exposure Starting Point</td>
<td>7.35</td>
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<td>6.59</td>
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<td>Air Exposure Ending Point</td>
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</tr>
<tr>
<td>Diffusible H (air exposure)</td>
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<td>3.33</td>
<td>3.20</td>
</tr>
<tr>
<td>Diffusible H Amount</td>
<td>3.23±0.23</td>
<td>3.42±0.17</td>
<td>3.63±0.19</td>
</tr>
</tbody>
</table>

5.6  Microstructural Images

The middle section and the two outer surfaces of the specimen are the three regions analyzed for microstructure features. The LOM images in the following figures are arranged as the middle region, the top surface, and the bottom surface. The metallographic images shown in this section were all in the plane that contains the normal and rolling directions, and the longitudinal direction is in the horizontal direction.

Figure 5.14 shows an LOM micrograph of a DP780 sample that has been pre-deformed with
bending, hydrogen charged, and delayed fracture tested. Figure 5.14b and 5.14c show the microstructure adjacent to the outer surface and adjacent to the surface in contact with the die, respectively. The microstructural regions in Figure 5.14 appear to be free of cracks. The microstructure in Figures 5.14b and 5.14c are similar to each other and are similar to the microstructure in the middle region of the sample (Figure 5.14a). The top surface went through tension in bending deformation and then compression during a three-point delayed-fracture test, and the bottom surface saw compression and then tension. From the three microstructural images of three different locations, there was no obvious difference between each one.

Figure 5.14 Cross sectional microstructure images of hydrogen charged DP 780 specimen with bending strain adjacent to the (a) middle (b) top surface (c) bottom surface regions. (Etched with 2% nital)

Figure 5.15 shows an LOM micrograph of a DP980 sample that was deformed with bending, hydrogen charged, and delayed fracture tested. Figure 5.15b and 5.15c show the microstructure adjacent to the outer surface and adjacent to the surface in contact with the die, respectively.

Figure 5.15 Cross sectional microstructure images of the hydrogen charged DP 980 specimen with bending strain adjacent to the (a) middle part, (b) top surface, and (c) bottom surface. (Etched with 2% nital)
Figure 5.16 shows a LOM micrograph of a TRIP780 sample that has been pre-deformed with bending, hydrogen charged, and delayed fracture tested. Figure 5.16b and 5.16c show the microstructure adjacent to the outer surface and adjacent to the surface in contact with the die, respectively.

![Micrograph images](image1.png)

(a) Middle part  (b) Top surface  (c) Bottom surface

Figure 5.16  Cross-sectional microstructure images of the hydrogen charged TRIP 780 specimen with bending strain adjacent to the (a) middle part, (b) top surface, and (c) bottom surface. (Etched with 2% nital)

Figure 5.17 shows an LOM micrograph of a TRIP980 sample that has been pre-deformed with bending, hydrogen charged, and delayed fracture tested. Figure 5.17b and 5.17c show the microstructure adjacent to the outer surface (tensile) and adjacent to the surface in contact with the die (compression), respectively. Figure 5.17b and 5.17c are microstructural images of top and bottom regions where a high degree of tension and compression deformation occurred during bending deformation. Some retained austenite will transform to martensite during deformation based on the results from XRD in the previous section. The more deformation causes an increase in the amount of martensite, which is more susceptible to hydrogen [9,10]. As a result, samples with deformation have a higher susceptibility to hydrogen embrittlement and delayed fracture than the ones without deformation, which is consistent with the fact that no deformation samples did not fracture, even with the same level of hydrogen.

![Micrograph images](image2.png)

(a) Middle part  (b) Top surface  (c) Bottom surface

Figure 5.17  Cross sectional microstructure images of the hydrogen charged TRIP 980 specimen with bending strain adjacent to the (a) middle part (b) top surface and (c) bottom surface. (Etched with 2% nital)
5.7 Time to Fracture

A high-resolution camera was used to track and record the specimen surface conditions during three-point bend testing. The “0” time is defined as the instant that a load is fully applied to the specimen, and the ending time is defined by the time past "0" that is required to stabilize the crack. All pictures were extracted from the originals in order to enlarge the crack regions, which were subsequently analyzed.

Surface cracks after delayed-fracture testing were absent on hydrogen-charged DP780 samples without deformation and bending deformation in the concave up condition. Cracks were observed for DP780 samples with uniaxial and bending strain in concave down case. Figure 5.18 shows the enlarged photographs of the middle region of the sample with uniaxial deformation, from 0 min to 30 min. The developed cracks are circled. The crack may have initiated at the edge, but it takes time to propagate across the whole width. For DP780 with bending deformation in the concave down condition, Figure 5.19 shows the crack that propagated across the entire width of the specimen. For bending deformation, the crack initiated later than the uniaxial strain case, but the time to propagate across the entire width was shorter. After the crack length was extended for the full width, the crack started to open further.

![Image](a) 0 min  
(b) 1 min  
(c) 2 min  
(d) 5 min  
(e) 8 min  
(f) 28 min

Figure 5.18 Strained surface of hydrogen-charged DP 780 specimen with uniaxial strain (a) 0 min (b) 1 min (c) 2 min, (d) 5 min, (e) 8 min, and (f) 28 min after the load was applied. Developed cracks are circled in the photographs.
Figure 5.20 shows the crack length ratio as a function of time during the three-point bend testing of hydrogen-charged DP780 specimens with uniaxial and bending in the concave down conditions. The crack length ratio is calculated as the crack length in horizontal direction divided by the width of the sample. The crack ratio ranges from 0 to 1. The measurement of cracks was made by using the microbar shown in the photograph, since the crack length continued to grow during the test. Five tests were conducted for each of the DP780 sample conditions. There was only one out of five tests (20 pct) where cracking was observed with uniaxial strain. There were two strain paths for bending strains: the concave up condition and the concave down condition. No cracks were observed for the concave up condition (0 pct), while one out of five tests (20 pct) exhibited cracking in the concave down condition. The specimen with bending deformation in the concave down condition developed a longer crack in a shorter amount of time.

Figure 5.19 Strained surface and crack conditions of hydrogen-charged DP 780 specimen with bending strain in the concave down condition (a) 0 min, (b) 3 min, (c) 8 min, (d) 11 min, and (e) 30 min after the load was applied.
Surface cracks were absent for hydrogen-charged DP 980 samples with no deformation. For the case of hydrogen-charged samples with uniaxial deformation, Figure 5.21 shows a succession of photographs of crack development as a function of time.
A crack-evolution sequence similar to that observed in Figure 5.9 was also observed for hydrogen-charged samples with bending deformation, for both the concave down and the concave up conditions, as shown in Figures 5.22 and 5.23, respectively. All these cracks were initiated in the edge region of the sample. The initiation time of cracking in the concave down condition was the earliest, and the initiation time of cracking in the concave up condition was the latest. The stabilization time of crack propagation also followed the same sequence as the initiation time.

Figure 5.22  Strained surface and crack conditions of the hydrogen-charged DP 980 specimen with bending strain in the concave down condition (a) 0 min, (b) 3 min, (c) 8 min, (d) 11 min, and (e) 30 min after the load was applied.

Figure 5.24 shows the crack length ratio as a function of time during the three-point bend testing of hydrogen-charged DP980 specimens with uniaxial and bending strain treatments. For the bending deformation case, both the concave up and concave down conditions are shown in the same figure to be comparable. Ten tests were performed for each of the DP980 samples with uniaxial and bending strains. For bending strains, five tests were done for the concave up condition and five for the concave down condition. Five out of ten tests (50 pct) were observed to have cracks for the uniaxial strain. Four out of five tests (80
pct) were observed to have cracks in the concave down conditions, while only one out of five tests (20 pct) showed cracking in the concave up condition. The specimens with bending deformation in the concave down condition developed long cracks in a shorter amount of time and have the highest probability of delayed fracture occurrence, as compared to the specimen with uniaxial deformation.

![Figure 5.23](image)

**Figure 5.23** Strained surface and crack conditions of the hydrogen-charged DP 980 specimen with bending strain in the concave up condition (a) 0 min, (b) 10 min, (c) 12 min, (d) 13 min, and (e) 25 min after the load was applied.

![Figure 5.24](image)

**Figure 5.24** Crack length ratio development versus the time after loading of the hydrogen-charged DP980 specimen under uniaxial (a) and bending (b) strain treatment.
Surface cracks were absent from all hydrogen-charged TRIP780 samples with no deformation, uniaxial, and bending strains under both concave conditions. Ten tests were performed for each condition, and after all of these tests, no cracks were observed on the sample surfaces.

Surface cracks were absent on hydrogen-charged TRIP980 samples without deformation and bending deformation with the concave up condition. Cracks were observed for the TRIP 980 samples with uniaxial and bending strain in the concave down condition. Figure 5.25 shows the crack that propagated across the entire width of the specimen with uniaxial strain. A similar crack-evolution sequence was observed for the hydrogen-charged specimen with bending deformation in the concave down condition, and Figure 5.26 shows photographs of the specimen taken at different times. The TRIP980 steel with uniaxial strain developed a crack earlier than that of the bending strain condition, and also finished the propagation process earlier.

Figure 5.25 Strained surface and crack conditions of hydrogen-charged TRIP 980 specimen with uniaxial strain (a) 0 min, (b) 1 min, (c) 3 min, (d) 7 min, and (e) 15 min after the load was applied.

Figure 5.27 shows the crack length ratio as a function of time during the three-point bend testing of hydrogen-charged TRIP980 specimens with uniaxial and bending with concave down condition strain treatments. Five tests have been done for each of the TRIP980 samples with uniaxial and bending strains. There were four out of five tests (80%) with observed cracks for both uniaxial and bending strain with the concave down condition. The specimens in the bending deformation concave down condition developed a
shorter crack in a longer amount of time at the same probability of delayed fracture occurrence, as compared to the specimens with uniaxial deformation.

Figure 5.26 Strained surface and crack conditions of the hydrogen-charged TRIP 980 specimen with bending strain of concave down condition (a) 0 min, (b) 6 min, (c) 8 min, (d) 15 min, and (e) 30 min after the load was applied.

Figure 5.27 Crack length ratio development versus the time after the loading of the hydrogen-charged TRIP 980 specimens under uniaxial (a) and bending with the concave down condition (b) strain treatment.
Table 5.10 shows the crack occurrences for each specimen for all strain conditions, and the number of valid tests are also shown in bracket. The crack observations vary for the different steels and deformation modes. No cracks were observed for the no deformation condition for all of the steels, and no fracture was detected in the TRIP780 samples for all deformation modes. The bending deformation samples in the concave down condition had the highest crack occurrence, while bending deformation in the concave up condition had the lowest occurrences. Cracking was only observed once for the DP980 sample. The 980 MPa strength steels are more sensitive to the deformation modes and crack more easily, as compared to the 780 MPa steels. This trend is consistent with the observation that steels with a higher strength level are more prone to hydrogen embrittlement and delayed fracture [9.11].

<table>
<thead>
<tr>
<th>Deformation Modes</th>
<th>DP780 (pct)</th>
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<th>TRIP780 (pct)</th>
<th>TRIP980 (pct)</th>
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<td>No Deformation</td>
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<td>0 (5)</td>
<td>0 (5)</td>
<td>0 (5)</td>
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<tr>
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<td>0 (5)</td>
<td>80 (5)</td>
</tr>
<tr>
<td>Bending Concave UP</td>
<td>0 (5)</td>
<td>20 (5)</td>
<td>0 (5)</td>
<td>0 (5)</td>
</tr>
<tr>
<td>Bending Concave Down</td>
<td>20 (5)</td>
<td>80 (5)</td>
<td>0 (5)</td>
<td>80 (5)</td>
</tr>
</tbody>
</table>

Table 5.11 shows the average times to initiate and stabilize the fracture for each specimen for all strain conditions. Bending deformation makes the 980 MPa strength materials more prone to cracks, while uniaxial strain makes the DP780 samples easier to fracture. For the case of bending deformation in the concave up condition, only the DP980 samples initiated a crack with a relatively long initiation time. For bending deformation in the concave down condition, the cracking was observed sooner. Crack development and propagation across the width occurs sooner for DP980 in both uniaxial and bending deformation conditions than for DP780.

<table>
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<th>Time to Fracture (min)</th>
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<th>DP 980</th>
<th>TRIP780</th>
<th>TRIP980</th>
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<tr>
<td></td>
<td>Stabilization</td>
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<td>14</td>
<td>-</td>
</tr>
<tr>
<td>Bending Concave UP</td>
<td>Initiation</td>
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<td>10</td>
<td>-</td>
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<td>Bending Concave Down</td>
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<td>15</td>
<td>7</td>
<td>-</td>
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<tr>
<td></td>
<td>Stabilization</td>
<td>29</td>
<td>15</td>
<td>-</td>
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5.8 Crack Details

Based on observations of surface cracks, there were some micro-cracks along or near the primary crack. The micro-cracks are not quantified, but the images can still provide a secondary method for assessing susceptibility to delayed fracture.
The SEM images of surface cracks were recorded and analyzed for DP780 samples with both uniaxial and bending strain. Figure 5.28 shows three different locations on the sample surface with uniaxial deformation and the observable micro-cracks are circled. Edge 1 is the edge where the crack first initiated, and edge 2 is the end where that crack stops propagating. From the images in edge 1, there is a higher density of micro-cracks observed as compared to edge 2 regions. In the middle region shown in Figure 5.28 (b), some micro-cracks and an obvious gap between the main cracks can also be observed. In general, an obvious gap means two cracks initiated from two locations and sometimes coalesced into one main crack.

Figure 5.28  SEM images of surface cracks of hydrogen charged DP 780 specimen with uniaxial strain.

Figure 5.29 shows two different locations on the sample surface of DP780 with bending deformation under the concave down condition, and observable micro-cracks are circled. For edge 1, there is a higher density of micro-cracks observed near the edge region than in the same region of edge 2.

Figure 5.29  SEM images of surface cracks of the hydrogen-charged DP 780 specimen with bending strain under the concave down condition.

Figure 5.30 shows three different locations on the sample surface of DP980 with uniaxial deformation and the observable micro-cracks are circled. For edge 1, there is a slightly lower density of
micro-cracks observed than for edge 2 regions. The width of the main crack at edge 1 is larger than that in the middle and edge 2 regions. Based on the nature of this primary crack, there was no gap between the main cracks, since the crack was initiated from one end and propagated to the other end. There were more micro cracks observed in the DP980 samples than the DP780 samples under both the uniaxial and bending deformation conditions.

![SEM images of surface cracks of hydrogen charged DP 980 specimen with uniaxial strain.](image)

Figure 5.30 SEM images of surface cracks of hydrogen charged DP 980 specimen with uniaxial strain.

Figure 5.31 and 5.32 show the sample surface of DP980 with bending deformation under the concave down and concave up conditions, respectively. For edge 1, there was a higher density of micro-cracks observed than for edge 2. There was no gap between the main cracks, since the crack initiated from one end and propagated to the other end. Compared to the cracks in DP780, there is a higher slope for the primary crack; this may be due to the effects of high plastic deformation on the surface and the larger thickness of the sample. There is more crack path tortuosity in the concave down condition. However, the crack in the concave up condition is similar to that in the uniaxial strain condition.

![SEM images of surface cracks of the hydrogen-charged DP 980 specimen with bending strain under concave down condition.](image)

Figure 5.31 SEM images of surface cracks of the hydrogen-charged DP 980 specimen with bending strain under concave down condition.
Surface cracks were absent from hydrogen-charged TRIP780 samples with all deformation modes. As a result, no images are shown or analyzed for this sample. Figure 5.33 shows three different locations on the sample surface of TRIP980 with uniaxial deformation and the observable micro-cracks are circled. For edge 1, there was a higher density of micro-cracks observed than for edge 2. Based on the nature of the primary crack, there is a small gap between the main cracks, since the crack was initiated from both ends but at different times. The higher micro crack density and larger width of the crack indicates that cracks initiate earlier in edge 1 than in edge 2.

Figure 5.34 shows two different locations on the TRIP980 sample surface with bending deformation in the concave down condition, and observable micro-cracks are circled. For edge 1, there is a higher density of micro-cracks observed than for edge 2. Based on the nature of the primary crack, with no gap in the middle region, the crack initiated from one end and propagated to the other end. The slope of the
crack path in the TRIP980 condition is larger than that of the DP780 condition, but smaller than that in the DP980 condition. This difference may be due to the different thickness of these three materials. The thickness of the three specimens are DP980 (1.6 mm) > TRIP980 (1.2 mm) > DP780 (1.0 mm).

A lower micro crack density was observed in the middle regions of sample as compared to the edge regions, so higher micro-crack density is considered to be presented in crack initiation region. A slightly higher micro crack density was observed in the uniaxial strain conditions than both cases of bending conditions. More micro-cracks were observed in 980MPa samples than 780MPa samples under the same deformation conditions. There were more micro-cracks in DP steels under uniaxial strain than TRIP steels, while higher density of micro-cracks in TRIP samples under bending strain than DP samples. More micro cracks were detected around narrower cracks, as compared to wider cracks.

![Figure 5.34 SEM images of surface cracks of hydrogen charged TRIP 980 specimen with bending strain under concave down condition.](image)

(a) Edge 1  
(b) Edge 2

5.9 Fractography

The middle section and the two outer edges of the specimen fracture surfaces are the three regions that were analyzed for crack initiation, propagation, and stabilization. The SEM images in the following figures are arranged as crack edge 1, middle region, and crack edge 2. Edge 1 is the region where the crack was observed to initiate. All enlarged regions are marked with a box to show their location on the lower magnification optical images.

Figure 5.35 shows SEM images at different magnifications of the three regions of the fracture surface, for the DP780 specimen with uniaxial deformation. Edge 1 in Figure 5.35 shows a mixed mode of cleavage type fracture and micro-void coalescence: several small flat regions connected with small areas of ductile dimples. This observation is consistent with the fact that the initiation of a crack usually has quasi-cleavage or intergranular fractures due to hydrogen embrittlement [9,12]. Micro cracks are present on the fracture
surface, possibly due to inclusions, chemical segregations, or hydrogen, as well as the overload conditions. The middle region shows micro-void coalescence features in Figure 5.35e.

Figure 5.35 SEM images of fracture surface of the DP 780 samples with uniaxial strain.

Figure 5.36 shows SEM images at different magnifications of the three regions on the fracture surface for the DP780 specimen with bending deformation under the concave down condition. Compared with the unaxial strain case, the bending strain condition exhibited less flat regions of cleavage-type fractures. This may be one reason why the cracks initiated later in the bending deformation condition.
Figures 5.37 and 5.38 show the SEM images at different magnifications in the three regions of the fracture surface for the DP980 specimens with both uniaxial and bending deformation in the concave up condition, respectively. For the uniaxial deformation, edge 1 and middle region shows larger areas of flat fracture compared to edge 2. Both the initiation edge and middle section show large regions of quasi-cleavage type fracture although more ductile tearing is observed in the middle. This cleavage pattern is likely due to the high hydrogen sensitivity and microstructure susceptibility. However, for edge 2, the fracture type is likely a mixed mode with quasi-cleavage and micro-void coalescence.
For bending deformation in Figure 5.38 in the concave up condition, there are more ductile fracture features observed on the fracture surface, as compared to the uniaxial deformation condition. This may be one of the facts that support later crack initiation and propagation. The ductile fracture of this condition is also consistent with the fact that concave up condition has much lower probability of delayed fracture.
For bending deformation in the concave down condition, edge 1 in Figure 5.39 shows obvious large areas of flat regions, and the middle region has regions of a quasi-cleavage connected with small areas of ductile dimples. However, the other edge region shows some ductile features with a large crack. The fracture in this sample produced higher amounts of flat regions and cleavage-type fractures than in the concave up condition, but a less brittle type of fracture was detected (smaller cleavage region), as compared to the uniaxial strain condition.

Surface cracks were absent from hydrogen-charged TRIP780 samples with all deformation modes. As a result, no images were shown or analyzed for this sample.
Figures 5.40 and 5.41 show SEM images for the three fracture surface regions of the TRIP980 specimens with uniaxial, and bending deformations, respectively. For uniaxial deformation, edge 1 shows a large crack surrounded by some flat regions, and the middle region has one large crack connected to some areas of flat fracture. However, edge 2 shows a ductile fracture features. The brittle to ductile transition feature from the crack initiation edge to the other end of this condition is clear. According to the observed morphology, the fracture type that occurs around edge 1 is likely to be quasi-cleavage. In the middle region, there was a large crack on the surface, but some of the area near the crack exhibited a ductile type of fracture while some other area showed a brittle type of fracture. The general trend of the fracture surface varies from brittle to partially ductile and brittle to ductile, moving from the crack initiation point up to the crack stabilization regions.
For the bending deformation, Figure 5.41 shows the two fracture surface regions with large flat areas, as well as a large crack and a smaller crack in the edge 1 region. However, the crack propagation region shows micro-void coalescence features. These three images also match the general trend of change from brittle to ductile along the fracture surface.

The initiation region of cracks generally involves regions of higher fraction of flat and cleavage patterns than other regions. A slightly higher fraction of cleavage regions were observed in the uniaxial strain conditions than both cases of bending conditions. More brittle type of fracture was observed in 980 MPa samples than 780 MPa samples under the same deformation conditions. There were more brittle like cleavage regions present in DP steels under uniaxial strain than TRIP steels, while there was a greater amount of flat regions observed in TRIP samples under bending strain than DP samples.
5.10 Summary

In general the hardness of 980 MPa steels is higher than that of 780 MPa steels. The hardness after deformation is larger than that of no-deformation cases. Among the deformation modes, bending strain samples acquire a higher hardness than that with uniaxial strain, and uniaxial strain obtains a larger hardness than the no-deformation condition for all four materials. For the DP microstructure, both DP780 and DP980 steels exhibit an obvious difference in micro hardness between different deformation modes. However, the hardness differences between bending deformation and uniaxial deformation for TRIP780 and TRIP980 specimens are similar. The DP780 is slightly harder than TRIP780 in both the uniaxial and bending deformation treatments. TRIP980 is slightly harder than DP980 in uniaxial deformation treatments.
Bending deformation specimens in the concave down condition are more susceptible to delayed fracture than uniaxial deformation specimens, and are much more susceptible to delayed fracture than a specimen without deformation for all four steels. In addition, specifically for the 980 MPa strengths, bending deformation makes the steel more susceptible to delayed fracture than uniaxial tension for both DP and TRIP steels. For 780 MPa strengths there is no significant difference between the uniaxial and bending deformations for both DP and TRIP steels. For the bending deformation condition, the concave down condition is more susceptible to delayed fracture, as compared to the concave up condition of DP780, DP980, and TRIP980 steels.

DP980 steel is more susceptible to delayed fracture than DP780, in both uniaxial and bending deformation treatments. However, the susceptibility difference for bending deformation is more significant than that for the uniaxial deformation for both steels. TRIP980 steel is more susceptible to delayed fracture, as compared to TRIP780, in both uniaxial and bending deformation treatments. However, the susceptibility difference between the bending deformation and the uniaxial deformation are similar for both steels.

DP780 is more susceptible to delayed fracture than TRIP780 after either uniaxial or bending deformation. In contrast TRIP980 is slightly more susceptible to delayed fracture as compared to DP980 after either uniaxial or bending deformation. This large difference is considered possible due to the retained austenite in microstructure. There is some austenite remaining in TRIP780 samples while there is none left in TRIP980 samples.

The middle section and the two outer edges of the specimen fracture surfaces are the three regions that were analyzed for crack initiation, propagation, and stabilization. From both the micro-crack and the fractographic perspective, edge initiation region is generally more prone to hydrogen embrittlement and fracture. There is more micro-crack density and larger regions of cleavage in the crack initiation regions than the other edge and middle section. The initiation region is considered as the location where hydrogen makes the most contribution to impair the mechanical properties of samples.
CHAPTER 6  DISCUSSION

This chapter presents the interpretations of the experimental results. Using theories from the literature, with the hardness profiles and microscopic and fractographic images, explanations of the observed behaviors are presented.

6.1  Microstructure

Cracks are observed to initiate in the martensitic phase and continue into interfaces but generally do not penetrated into ferrite during deformation [29]. There is a larger effect of hydrogen on mechanical property changes in the DP980 steel due to higher amount of martensite (48.5 pct > 35.6 pct in Table 5.1) in DP980 as compared to DP780.

For TRIP steels, hydrogen concentration and behavior is more complex due to the austenite-to-martensite phase transformation. The hydrogen diffusivity in TRIP steels increases when the transformation occurs compared to DP steels due to the increasing diffusivity of hydrogen in martensite, as compared to austenite [30]. This increased diffusivity is considered to be one factor that contributes to the embrittlement of TRIP steels. If there is no austenite remaining in the microstructure after deformation (e.g. TRIP980 in the current study), the hydrogen diffusivity will increase since it diffuses out of austenite to martensite or adjacent ferrite. If some austenite still remains in microstructure (e.g. TRIP780 in the current study), the hydrogen diffusivity will only slightly increase since austenite that remains in the microstructure will contain significant amounts of hydrogen. The phase transformation produces an increase in dislocation density due to the associated volume change, which usually happens near the interfaces [30].

Among boundaries between grains or phases, incoherent interface regions are considered as preferential hydrogen trapping sites due to the larger mismatch between the crystals [67]. The grain boundary area is inversely proportional to the average grain size, and higher strength steels possess microstructures consisting of smaller grain. This relationship partially shows why the 980 MPa strength steels are more prone to hydrogen effects than the 780 MPa steels, which are shown in Table 5.10 and Table 5.11.

Although both DP and TRIP steels contain martensite after deformation, the mechanism of interactions with hydrogen is different. For DP steels, martensite is created from the original processing, but for TRIP steels, some “fresh” martensite is produced during the austenite-to-martensite phase transformation. Due to the results from the XRD tests, the amount of newly formed martensite is low in TRIP980, but high in TRIP780. All of the austenite transformed to martensite in the TRIP980 steel, but there was some austenite remaining in the TRIP780 steel after the pre-strain deformation step. The
remaining austenite may be one of the reasons why TRIP780 exhibited the best delayed fracture resistance during the tests, since austenite has a higher solubility of hydrogen. This presence of austenite in the TRIP780 steel after deformation is the main difference between the DP and TRIP type microstructures, which possibly explains the different outcomes of the TRIP780 and TRIP980 samples during delayed fracture tests.

### 6.2 Hydrogen Effects on Delayed Fracture

Diffusivity and solubility of hydrogen in different phases and locations have a large effect on delayed fracture susceptibility [30][62][63][64][65]. The diffusivity of hydrogen in ferrite, martensite, and austenite are shown in Table 2.1. The different times to fracture are analyzed as the result of different diffusivity in different microstructure phases. In addition to these different values, different phase fractions also influence hydrogen behavior and outcome.

In the current study delayed fracture susceptibility was evaluated by the probability of fracture, time to fracture, and micro cracks on the surfaces of a specimen. Both microstructure and deformation mode have an influence on the cracking. Consequently, microstructure and deformation mode were analyzed and compared to explain the results. Hydrogen can play an important role in increasing dislocations and slip plane reactions, which significantly increases vacancy density and development of voids [69]. This effect of hydrogen is consistent with what was observed for lower hydrogen content samples that did not break during delayed fracture tests during preliminary testing. During the preliminary testing, no cracks were observed in any of the four steels even after uniaxial and bending deformation when small amounts of hydrogen were charged into samples. Table 6.1 shows the total hydrogen in the different steels after the first 30 minutes of the delayed fracture test.

<table>
<thead>
<tr>
<th>(min)</th>
<th>DP 780 Uniaxial</th>
<th>Bending</th>
<th>DP 980 Uniaxial</th>
<th>Bending</th>
<th>TRIP 780 Uniaxial</th>
<th>Bending</th>
<th>TRIP 980 Uniaxial</th>
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<td>3.14</td>
<td>3.52</td>
<td>3.98</td>
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</tbody>
</table>

Since the cracks observed during the delayed fracture tests were essentially stabilized at 30 minutes or earlier after the load was applied, the hydrogen amount in the specimen for the first 30 minutes after applying the load was recorded in 5-minute intervals. Hydrogen is trapped at dislocations and grain...
boundaries [30], so the higher initial amounts reflect the higher density of dislocations and larger total area of grain boundaries in microstructure. After uniaxial deformation DP980 and TRIP780 contain highest amount of hydrogen. This increase may result from the higher dislocation density in DP980 and the higher amount of retained austenite in TRIP780. According to Table 6.1, most of the diffusible hydrogen has diffused out of samples during the first 30 minutes, and that is why cracks gradually become stable after this period of time. Also, the load required to break samples decreased after there is a crack present on the sample surface. There is a large change in the hydrogen amount during the initial 10 to 20 minutes. This period is when crack initiation and propagation generally occurs. After the crack formed it was easier for hydrogen to diffuse out of the steel.

6.3 Effects of Deformation on Microstructure

The overall mechanical properties of dual-phase steels are dependent on the fraction of the martensite phase, martensite morphology, the hardness of martensite with its specific carbon content and ferrite grain size [50][51]. Based on the results, the martensite phase fraction of DP980 is higher than that of the DP780 samples. Martensite also acts as a preferential trapping site for hydrogen due to large dislocation density within martensite. The DP980 steel is more susceptible to hydrogen induced delayed fracture than DP780 samples for all deformation processes. In addition, the strain path is likely to have an effect on the interfaces between martensite and ferrite. Decohesion between ferrite and martensite usually leads to crack initiation and fracture propagates through martensite or even penetrates into adjacent ferrite [52]. Since no phase transformation occurs in DP steels during deformation, the effects of the strain path mainly influence the dislocation density and residual stress. The increase in dislocation density due to the prior deformation is consistent with the higher susceptibility to delayed fracture of DP steels after deformation. Furthermore, there was no large difference between uniaxial and bending deformation cases since the strain path has no effect on phase changes in DP steels.

Different from DP microstructure, microstructural constituents change occurs during deformation for the TRIP steels. The increased volume of martensite formed from austenite usually leads to dislocation initiation and high residual stress in phase boundary areas [46]. This change is consistent with the fact that high-strength TRIP steels (e.g. TRIP980) are slightly more susceptible to delayed fracture after deformation in Table 5.10 and Table 5.11. As a result, the 780 MPa strength DP and TRIP type steels have better formability and resistance to ductility loss than the 980 MPa strength steels. In addition, TRIP780 shows great resistance to hydrogen-induced delayed fracture during tests for different conditions. In the TRIP780 the austenite remaining in the microstructure after deformation prevented hydrogen diffusing due to higher hydrogen solubility in austenite.
Dislocation density increases in the ferrite phase adjacent to retained austenite and fresh martensite transformed from austenite in TRIP steels after straining [60]. Both the prior strain and dislocation density work together to make the interface regions between ferrite, austenite, and martensite the key features contributing to hydrogen embrittlement. The void initiation at interface regions between different phases is inhibited with an increasing volume fraction of retained austenite and higher amounts of bainite in the microstructure [59]. The difference in amount of retained austenite after deformation is consistent with the fact that the TRIP980 steel (all austenite transformed to martensite) is more susceptible to hydrogen and fracture, while no cracks were observed for the TRIP780 steel where a certain amount of austenite remained after all deformation conditions.

6.4 Effect of Bending on Strength Profiles

One of the interesting results from the current study was the difference in delayed fracture after bending deformation for the concave up versus the concave down side of the sheet. To better understand the cause of this difference this section and the next two sections try to provide some insight.

Figure 6.1 shows the strain and stress distributions through the sheet thickness (vertical in Figure 6.1) during bending under tension. In Figure 6.1, the top, horizontal line corresponds to the outer surface, and the bottom, horizontal line corresponds to the surface that is in contact with the die during bending under tension. Due to the tensile forces (approximately 80% YS) imposed by the hydraulic actuators, the neutral axis (plane in the sheet where stress and strain are zero) moves from the “free bending” configuration in the middle of thickness profile. Both areas of strain and stress are larger on the top surface than the bottom surface, due to the extra tensile force. There was no dynamic friction between the roller and the sample during the bending test, since there was no relative motion between sample and roller.

Figure 6.1b shows the strain and stress distribution during the unbending process (i.e. when the strip is leaving the roller). In this case the neutral plane is closer to the top surface of the sheet. The actual bending deformation imposed during the present study had both the bending and the unbending steps. The net result will be the sum of the two steps. The strains are added together and the effect on the strength profile through the sheet is also due to the two steps.

Figure 6.2 shows the addition of the strains and the net strength profile expected after the bending deformation. The strains add to a constant total strain as shown in the left of the figure. It should be noted that although the net strain is constant the effective strain near the top surface and near the bottom surface of the sheet will be larger than in the center. In the center there is just the addition of tensile strains from both steps in the bending process. On the top and bottom there has been some strain reversal – tension followed by compression on the top and compression followed by tension on the bottom. The effective strain for both locations will be greater than what is shown in the figure as net strain. With the effective strain being
larger in the surface regions the overall effect on strength is seen. The regions near the two surfaces will be stronger due to strain hardening as compared to the region in the center of the strip where the strain is in tension and nearly constant. The higher effective strain near the surfaces causes those regions of the strip to have a higher strength.

![Diagram of bending and unbending forces](image)

**Figure 6.1** Illustration of the effect of back force on the position of the neutral plane, stress and strain distribution diagram of (a) bending and (b) unbending for a unit of sheet metal, with bending under tension deformation.

![Result diagram](image)

**Figure 6.2** Illustration of the effect of back force on the position of the neutral plane and resulting stress distribution of a unit of sheet metal with both bending and unbending deformation.

Based on strain hardening, the strain imposed on the sample would have different local hardening effects. Figure 6.3 shows the micro hardness profiles through the thickness of all four samples, with bending deformation. The local effective strains on the top and bottom surfaces are larger than the strain in the middle. The 0 in horizontal axis denotes the top surface edge. The hardness values increased with an increasing strain imposed on the specimen, with higher hardness on the top and bottom surfaces than that in the middle region. The bending deformation makes the sample acquire higher hardness on both surfaces, and cracks are also easier to initiate in regions of higher hardness. Among 980 MPa strength steels, TRIP has minor higher hardness values than DP due to the transformation of martensite upon deformation. The time to initiate and propagate cracks in the TRIP steel was earlier than that in the DP steels. For the 780
MPa strength samples, the DP steels attain a larger hardness than in the TRIP steels, which is consistent with higher strength and strain hardening rate.

![Micro hardness profile along the thickness of DP steels](image)

![Micro hardness profile along the thickness of TRIP steels](image)

Figure 6.3 Micro hardness profile along the thickness of (a) DP780, (b) DP980, (c) TRIP780 and (d) TRIP980 with bending deformation.

The increasing hardness of DP and TRIP steels occurs by different mechanisms. The imposed strain of deformation produces a large amount of dislocations, and this increased dislocation density makes a contribution to increasing strength and hardness in steels. TRIP steels become harder through the same strain hardening mechanism, but in addition, the increase in the amount of martensite phase in the microstructure via austenite transformation due to deformation adds additional strength. The increased dislocation density and phase transformation in microstructure are discussed in the following sections.

For the concave up condition, the surface region sees tension, then compression during the bending and unbending, followed by tension during the delayed fracture test. In contrast, for the concave down condition, the surface sees compression, then tension, followed by tension during the delayed fracture test.
As a result, for the concave up condition, there is a Bauschinger effect during the bending deformation and then another Bauschinger effect during the three-point test. In contrast, for the concave down condition, there is a continuation of the tensile strain after tension from unbending. The concave down condition will have an increase in dislocation density for the delayed fracture test following tension during unbending. This sequence of tensile strains results in a higher susceptibility to hydrogen embrittlement of concave down case compared to concave up case, which is consistent with the observations made in the current study.

Compared to the difference among no deformation, uniaxial, and bending deformation, the different outcomes from the concave up and concave down conditions were “unexpected”. According to the similar magnitude of strain imposed on the sample and the micro hardness along the surface, other factors must be considered that affect the results. Cracks in the concave up condition were only observed in the DP980 steel, and one of the largest differences between DP980 and other steels is the thickness. Figure 6.4 presents the strain changes on the surface in the concave up and concave down conditions from bending deformation up to delayed fracture test. Actually, the concave up and concave down scenarios are just two surface sides of the same sample. As a result, the side view of sample after bending and unbending process is in the same shape but reversed direction. However, the degree of curvature becomes different after delayed fracture test since the sample is not flat any longer after unbending test due to spring back effect.

Figure 6.4  Geometry and strain change during three-point delayed fracture tests of (a) concave up and (b) concave down conditions. The surface tested is the lower surface shown in these illustrations for both cases.

Although the total change of strain is similar, the strain change route is very different. For the concave up condition, the strain changes from positive during bending to negative during unbending to positive during delayed fracture testing. For the concave down condition, the strain changes from negative during bending to positive during unbending and continues as positive during the delayed fracture test.
Compared to the concave up condition, the concave down condition has seen a different strain path during the prior deformation and the delayed fracture test.

Although the total strain imparted to the steels were the same for uniaxial and bending deformation steps, the differences in the strain paths during the deformation coupled with the additional strain imposed during the delayed fracture test causes the material response to be different. For the DP980 steel with the same deformation conditions (i.e. R/t values), the high strength causes a greater susceptibility to delayed fracture. This result is supported by the fact that only the DP980 samples exhibited cracks on the surface for the concave up condition, although at a lower probability.

6.5 Effects of Prior Strain on Dislocation Density

According to the literature review, strain path has effects on the substructure and arrangement of dislocations, which can cause differences in mechanical behavior [46][47]. During the bending under tension tests, the top and bottom surfaces go through different strain paths: compression after tension for the top surface and tension after compression for the bottom surface. The dislocation density increases during both uniaxial and bending deformation. However, the dislocation and hardness distributions are different with different deformation modes. The uniaxial strain causes the increase in hardness to remain essentially consistent through the entire thickness, unlike the hardness profiles for the bending under tension deformation. Figure 6.5 shows a schematic of the strain and dislocation density profile through the thickness orientation of samples with uniaxial and bending strain. The dashed line denotes the original strain and dislocation density for the as-received condition, and the solid line represents the strain distribution and dislocation density after deformation. From the two diagrams, a higher dislocation density is found in the surface regions of samples after bending deformation. Based on the background material presented in Chapter 2, dislocations are preferential low energy trapping sites for hydrogen. These dislocations are partially responsible for the higher rate of hydrogen absorption for a given time.

![Figure 6.5 Thickness orientation of strain and dislocation density profile of samples with (a) uniaxial and (b) bending under tension deformation](image-url)
As a consequence, prior strain makes the sample more prone to hydrogen embrittlement, due to higher dislocation density and more hydrogen trapping sites. A higher dislocation density provides more hydrogen trapping sites and also decreases the ductility of the material. This reasoning is consistent with the results that a higher number of fractures and shorter times to fracture are observed for the DP780, DP980, and TRIP980 samples after bending deformation.

### 6.6 Fractography and Fracture

An important aspect of hydrogen in metals, which has been the focus of many studies, is the dislocation structure of the metal. Dislocation is one of the preferential hydrogen trapping sites, especially those newly formed after deformation [30]. Dislocations and the hydrogen it traps are able to create plastic regions inside the specimen. With the increased plastic regions, there is an increasing probability of cracking and even fracture occurring. For the DP980 steels, a higher fraction of cleavage planes and cracks were observed for both uniaxial and bending strain conditions, as compared to the DP780 specimens. This result is consistent with the correlation between dislocation density and cleavage fractures observed. There is an obvious transition of the fracture surfaces from void coalescence to cleavage when contaminated with a certain amount of hydrogen [71][72]. For the 780 MPa strength steels, DP780 shows higher susceptibility to hydrogen-induced delayed fracture than TRIP780, which may partially result from the presence of a higher amount of BCC phases (namely, ferrite and martensite). FCC phase (such as austenite), which is only present in TRIP780 steel after deformation, diminishes the hydrogen effects on mechanical property degradation. This result is consistent with the fact that for TRIP980 steel, a higher fraction of cleavage planes and cracks were also observed for both uniaxial and bending deformation conditions than in the TRIP780 steel. It has been observed that the major crack was generally initiated along interface regions, and propagates through martensite or even penetrate into adjacent ferrite [52]. A crack has a larger probability to initiate and propagate along interface regions between martensite with other phases. The 980 MPa level steels possess higher amount of martensite phase, so these samples are more prone to delayed fractures as compared to the 780 MPa level steels for both DP and TRIP.

In addition to the large primary crack, there were also some micro-cracks emanating from the major crack especially in the edge regions of the samples. Among the different steels in the current study, the more brittle grades seem to have greater coalescence of the micro-cracks on the surface. This result is shown by the distribution of micro-cracks in DP steels being closer to one another than for the TRIP steels. When there are cracks present in the microstructure or on the surface, the load required to deform samples becomes lower due to the stress concentration effect. The amount of micro-cracks is also larger in 980 MPa level and DP steels than 780 MPa level and TRIP steels.
CHAPTER 7 CONCLUSIONS

(1) The total hydrogen amount is proportional to the charging time and current density. For the same specimen geometry, different prior deformation modes cause different amounts of hydrogen to be absorbed. The total amount of hydrogen inside the charged samples with the same current density and charging time is: Bending > Uniaxial Tension > No Deformation. The hydrogen amount changes during the three-point bend test, especially in the period of 10–20 minutes after the load is applied.

(2) Four advanced high strength steels (AHSS) (DP780, DP980, TRIP780 and TRIP 980) were analyzed to evaluate their susceptibility to delayed fracture after deformation. Both uniaxial and bending pre-deformation makes the steel more susceptible to delayed fracture as compared to no deformation. Specimens with prior bending deformation with the concave down surface tested in the three point bend test are more susceptible to delayed fracture as compared to uniaxial deformation. For 980 MPa strength steels, bending deformation results in greater susceptibility to delayed fracture than uniaxial tension for both DP and TRIP steels. For 780 MPa strength steels, there is no significant difference between the uniaxial and bending deformations for both DP and TRIP steels. Specifically for the bending deformation condition, the concave down condition is much more susceptible to delayed fracture, as compared to the concave up condition for DP780, DP980, and TRIP980 steels.

(3) In general, the hardness of 980 MPa steels is higher than that of 780 MPa steels. The hardness after deformation is larger than that of non-deformation cases. Among deformation modes, bending strain samples exhibit a higher hardness than that with uniaxial strain for all four materials. For the DP microstructure, both 780 and 980 MPa steels show an obvious difference in micro hardness between different deformation modes. However, the hardness for bending deformation and uniaxial deformation for the TRIP780 and TRIP980 specimens show similar trends. For 780 MPa steels, DP steels are slightly harder than TRIP steels after both the uniaxial and bending deformation. For 980 MPa steels, TRIP steels are slightly harder than DP steels after uniaxial deformation, and are similar to the hardness of DP specimens treated after bending deformation.

(4) For the DP steels, the 980 MPa grade steel is more susceptible to delayed fracture than the 780 MPa grade steel, in both uniaxial and bending deformation treatment. However, the susceptibility difference between the bending deformation specimens is more significant than that of the uniaxial deformation specimen within each grade. For the TRIP steels, the 980 MPa grade steel is more susceptible, as compared to the 780 MPa grade steel, after both uniaxial and bending deformation with concave down
surface being tested. However, the susceptibility difference between the bending deformation specimen with concave down case and the uniaxial deformation specimen are similar within each grade.

(5) For 780 MPa steels, DP steels are more susceptible to delayed fracture than TRIP steels after both uniaxial and bending deformation. However, the susceptibility difference between the bending deformation and the uniaxial deformation specimen is similar. For 980 MPa steels, TRIP steels are slightly more susceptible to DP steels in both uniaxial and bending deformation for the concave down case. The susceptibility difference between bending deformation with concave down and uniaxial deformation is similar.

(6) In general, the delayed fracture crack initiation region appears more brittle than the other edge and middle regions for all the steels in this study that exhibited delayed fracture. For 780 MPa steels, DP steels are more prone to hydrogen with more cleavage fracture appearance than the TRIP steels after both uniaxial and bending deformation for the concave down case. Specifically for the DP780 steel, the fracture surface after uniaxial deformation condition appears more brittle than after bending for the concave down case. For 980 MPa steels, the TRIP steel is slightly more susceptible to delayed fracture after both uniaxial and bending deformation for the concave down case as compared to the DP steel.
REFERENCES


APPENDIX I   HYDROGEN CONTENT IN THIS STUDY

For collection of uniaxial tensile data for the thesis work two geometries were utilized; ASTM E-8 sub-sized samples and modified ASTM A514 samples for the Lujan Center.

Table I.1  Diffusible Hydrogen in DP780 Samples

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Table I.2  Diffusible Hydrogen in DP980 Samples

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Table I.3  Diffusible Hydrogen in TRIP780 Samples

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<td>7.25</td>
<td>8.69</td>
<td>10.10</td>
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<td>Air Exposure Ending Point</td>
<td>3.98</td>
<td>4.95</td>
<td>3.27</td>
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<td>3.74</td>
<td>6.83</td>
</tr>
<tr>
<td>Diffusible H amount</td>
<td>3.05</td>
<td>3.77</td>
<td>6.07</td>
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<td>wt pct</td>
<td>No Deformation</td>
<td>Uniaxial Tension</td>
</tr>
<tr>
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<td>-------------</td>
<td>----------------</td>
<td>------------------</td>
</tr>
<tr>
<td></td>
<td>30 min</td>
<td>60 min</td>
<td>90 min</td>
</tr>
<tr>
<td>Water Heat Starting Point</td>
<td>7.35</td>
<td>11.20</td>
<td>13.60</td>
</tr>
<tr>
<td>Water Heat Ending Point</td>
<td>4.20</td>
<td>5.52</td>
<td>5.55</td>
</tr>
<tr>
<td>Diffusible H amount 1</td>
<td>3.15</td>
<td>5.68</td>
<td>8.05</td>
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<tr>
<td>Air Exposure Starting Point</td>
<td>7.35</td>
<td>11.20</td>
<td>13.60</td>
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<tr>
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<td>4.04</td>
<td>5.48</td>
<td>5.60</td>
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<tr>
<td>Diffusible H amount 2</td>
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<td>5.72</td>
<td>8.00</td>
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<td>Diffusible H amount 3</td>
<td>3.23</td>
<td>5.70</td>
<td>8.03</td>
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Table II.1  Thickness before and after Bending Deformation

<table>
<thead>
<tr>
<th></th>
<th>DP 780</th>
<th>DP 980</th>
<th>TRIP780</th>
<th>TRIP980</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness Before Deformation</td>
<td>0.040</td>
<td>0.063</td>
<td>0.047</td>
<td>0.047</td>
</tr>
<tr>
<td>Thickness After Deformation</td>
<td>0.038</td>
<td>0.060</td>
<td>0.0445</td>
<td>0.045</td>
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</tbody>
</table>

Effective strains are calculated by the formula as following:

DP780: $\varepsilon = \ln \left( \frac{0.038}{0.040} \right) \times \frac{2}{\sqrt{3}} = -0.059$

DP980: $\varepsilon = \ln \left( \frac{0.060}{0.063} \right) \times \frac{2}{\sqrt{3}} = -0.056$

TRIP780: $\varepsilon = \ln \left( \frac{0.0445}{0.047} \right) \times \frac{2}{\sqrt{3}} = -0.063$

DP780: $\varepsilon = \ln \left( \frac{0.045}{0.047} \right) \times \frac{2}{\sqrt{3}} = -0.050$