THE RELATIONSHIP BETWEEN MICROSTRUCTURE AND DAMAGE EVOLUTION IN HOT-ROLLED COMPLEX-PHASE STEEL SHEET

by

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Abstract

Complex-phase (CP) steels are employed in applications that require high-strength and good edge formability. These steels derive their strength from a fine-grained bainite-ferrite microstructure, and alloying to provide solid-solution and precipitation strengthening. CP steels are produced industrially through a process of controlled rolling and cooling to produce desirable microstructures.

Hole-expansion tests are typically used as a measure of edge formability for applications such as stretch-flanges. It has been shown that CP microstructures are susceptible to large fluctuations in hole-expansion performance with little change in processing or resulting tensile properties. The steel’s characteristics of damage evolution are critical to the hole-expansion performance.

This study investigates the role of microstructure in the development of damage in CP microstructural variants. Two variant pairs of different thicknesses were produced from the leading and trailing edge of industrially produced hot-rolled sheet. Each pair consisted of a variant with poor hole-expansion performance, and a variant with good hole-expansion performance. Each variant was tested via interrupted double-notched uniaxial tension testing to induce damage. Damage evolution in each variant was quantified by X-ray micro-computed tomography (XµCT), and supplementary optical micrography. The damage results were correlated with microstructural characteristics.

It was shown that poor hole-expansion variants failed by intergranular fracture. In these variants, void damage induced by hard martensite and retained austenite was not critical in producing failure. Purely void-damaged microstructures failed by ductile fracture, whereas cracked microstructures failed in a mixed brittle-ductile failure initiated by planar cracks. Microstructural banding of large elongated ferrite grains correlated with the existence of intergranular planar fractures.
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Contributors

The metallographic work of Jeff Laflamme proved to be both excellent in its quality and crucial to this thesis. Jeff conducted the polishing, etching, and imaging of optical micrographs contained in this study.
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Chapter 1

Introduction

This thesis investigates the evolution of internal damage as a function of strain in a series of hot-rolled steel variants. Metallographic characterization of microstructures is paired with measurements of internal damage by X-ray micro-computed tomography to obtain a better understanding of how microstructural features correlate with damage behaviour.

1.1 Motivation

Automobile manufacturers are facing strict regulations on their average fuel economy that require light-weighting of vehicles through strategic material choices. Regulations such as the corporate average fuel economy (CAFE) standards in the United States mandate that cars and light-duty trucks attain an average of 54.5 mpg by 2025, which is nearly double the average mandated for 2016 [1]. To meet these standards while maintaining vehicle safety it is necessary to develop high strength steels to replace conventional steels, with little increased material cost [2].

The industry’s response to increased demand for high strength steel with good formability has been the development of advanced high-strength steels (AHSSs) that can outperform conventional high-strength steels (Figure 1.1). Parts that require very high levels of energy absorption, while maintaining total elongation for formability, can be made from complex-phase (CP) steels [3]. Automotive parts are formed through a combination of modes: deep-drawing, stretching, stretch flanging, and bending, which are depicted in Figure 1.2.
Figure 1.1: Typical ranges for yield strength and tensile elongation showing the general decline in elongation at higher strengths for different classes of steel. [4]

Figure 1.2: Sheet metal parts are formed by a combination of the above 4 modes. [5]
Hole-expansion testing is used as a measure of edge formability during stretch/expansion forming operations. During these tests, a pierced or reamed hole in a sheet is expanded using a conical or hemispherical punch, with rigs like that of Figure 1.3, until edge cracking is visually detected [6–8]. The percentage change in hole diameter from start to finish is the reported metric for these tests, given by:

\[
\text{hole expansion (pct.)} = \frac{D_f - D_0}{D_0} \times 100
\]

where final hole diameter is denoted \(D_f\) and initial hole diameter \(D_0\). This measure of formability is of particular interest to automotive manufacturers as it is representative of the types of operations performed in automotive part production [5]. Hole-expansion performance has demonstrated direct links to microstructure [5,6,9], and particular industrial heats of CP steel have shown great variations in hole-expansion performance between materials with very similar tensile properties [10]. Gaining a better understanding of how CP microstructures develop internal damage can help to explain hole-expansion performance and allow for better microstructural design.

\[\text{Figure 1.3: Typical hole-expansion rig, showing a 60° conical punch. [11]}\]
1.2 Objectives

The focus of this thesis is to study differences in the evolution of internal damage in CP steels. Evolution of internal damage will be related to microstructure to help explain variations in hole-expansion performance despite similarities in uniaxial tensile properties. This study will utilize the 3-D imaging method of X-ray micro-computed tomography (XμCT) to measure void damage evolution, as well as conventional metallographic methods to document progressive internal damage. Studies of void damage in steel by XμCT have been conducted [12–18], but none of these studies examined CP steels such as the grade used in this study. Hole expansion in steels has been investigated with respect to microstructure [5–9], but not employing 3-D imaging methods.

This thesis therefore uniquely aims to use 3-D imaging data to help explain the hole-expansion damage mechanisms in CP steel microstructures. Two pairs of hot-rolled sheet were provided for this study. The sheet materials exhibited microstructural variation due to differences in thermomechanical processing. These variant pairs show large differences in hole-expansion performance despite having similar transverse tensile properties. Hence, further information about internal damage behaviour is necessary to explain their hole-expansion behaviour. Uniaxial tensile testing of double-notched samples will be used to concentrate internal damage to a small area of each sample. Evolution of internal damage at increasing strains will be investigated by interrupting these notched tensile tests and obtaining measures of internal damage in extracted regions of each sample. Measurements and observations of damage will be linked with microstructural features and hole-expansion performance.
1.3 Organization

The remainder of this thesis is divided into five chapters as outlined below:

- Chapter 2 provides a review of the literature to provide the necessary background for understanding the methods, results, and discussion contained in this thesis.

- Chapter 3 includes a description of the material variants received, as well as a description of the testing methods used to investigate those variants.

- Chapter 4 presents the results of the investigations outlined in Chapter 3.

- Chapter 5 comprises a discussion of the results and methods, considering their meaning with respect to the literature.

- Chapter 6 enumerates conclusions of the thesis and recommendations for future work.
Chapter 2

Literature Review

2.1 Complex-Phase (CP) Steel Microstructures

CP steel microstructures are defined as those with small amounts of martensite, retained austenite, and pearlite in a ferrite/bainite matrix. Polygonal or elongated ferrite nucleates at prior-austenite grain boundaries and acicular ferrite nucleates intragranularly [19]. Dislocation density is variable in all types of ferrite depending on prior austenite deformation [19–21]. Typically a granular bainite structure is produced, which consists of a bainitic ferrite matrix with evenly distributed martensite/retained austenite (MA) islands, but some conventional bainite is possible [6,22]. Precipitates take the form of rod-like (Fe,Mn)₃C at grain boundaries, randomly distributed cuboidal TiN, and (Ti,Nb)C located on sub-grain boundaries [6,22]. These steels typically have extreme grain refinement accomplished through suppression of recrystallization and through the grain boundary pinning effect of solid-solution and precipitated alloying elements [3].

2.1.1 Chemistry

Standard chemistries for AHSSs are outlined in ASTM Standard 1079-13 [23], which includes CP steels. Given the designation of CP steel, the maximum allowable alloying levels are outlined in Table 2.1. The amounts of each alloying element are varied based upon the desired microstructure and mechanical properties of the steel. Functions of the typical CP steel alloying elements are outlined in Table 2.2.
Table 2.1: Allowable maximum wt% of each alloying element for CP grade steels, adapted from [23].

<table>
<thead>
<tr>
<th>Designation/Grade</th>
<th>C</th>
<th>Mn+Al+Si</th>
<th>P</th>
<th>S</th>
<th>Cu</th>
<th>Ni</th>
<th>Cr+Mo</th>
<th>V+Nb+Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>600T/350Y</td>
<td>0.18</td>
<td>5.30</td>
<td>0.080</td>
<td>0.015</td>
<td>0.20</td>
<td>0.50</td>
<td>1.00</td>
<td>0.35</td>
</tr>
<tr>
<td>780T/500Y</td>
<td>0.18</td>
<td>5.50</td>
<td>0.080</td>
<td>0.015</td>
<td>0.20</td>
<td>0.50</td>
<td>1.00</td>
<td>0.35</td>
</tr>
<tr>
<td>980T/700Y</td>
<td>0.25</td>
<td>5.20</td>
<td>0.080</td>
<td>0.015</td>
<td>0.20</td>
<td>0.50</td>
<td>1.00</td>
<td>0.35</td>
</tr>
</tbody>
</table>

Table 2.2: Function of typical alloying elements in CP steels. Modified from [24].

<table>
<thead>
<tr>
<th>Element</th>
<th>Function</th>
</tr>
</thead>
<tbody>
<tr>
<td>C, Mn, Si, Ni, Cr, Mo</td>
<td>Increases solid-solution strength and hardness</td>
</tr>
<tr>
<td>V, Nb, Al, Ti</td>
<td>Increases strength and hardness through grain refinement</td>
</tr>
<tr>
<td>N</td>
<td>Increases amount of nitrides, required for strengthening or grain refinement</td>
</tr>
<tr>
<td>P, S</td>
<td>Generally considered impurities</td>
</tr>
</tbody>
</table>

Of particular importance to the microstructures of CP steels are Ti and Nb, which are responsible for the characteristic grain refinement strengthening of these steels. Suppression of recrystallization is accomplished either by solute drag effects of solid solution Ti and Nb, or by precipitation of carbonitrides [10], which also contribute to strengthening via precipitation hardening.

The promotion of bainite is also essential to the microstructures of CP steels. Niobium has known efficacy in preventing the formation of ferrite grains on austenite grain boundaries, and thus allowing for the formation of bainite at intermediate hold temperatures [25]. Additionally it has been shown that the presence of niobium carbides can promote the formation of bainite [26].

Carbon content can be reduced to promote ductility, without reducing strength in CP steels. Although carbon reduction does improve ductility, carbon is still essential for carbide formation.
Accordingly, carbon content should be maintained above 0.01wt% [27] to ensure that the benefits of carbide formation are retained.

Manganese is also of particular importance in these steels, because of its positive effect of solid solution strengthening, countered by its negative contribution of increased banding in rolled microstructures [28,29].

2.1.2 Processing

CP steels are produced as hot-rolled sheets, subjected to particular controlled rolling and controlled cooling through the bainite transformation region to promote the desired microstructure. Finish rolling temperature, rolling reduction, and cooling schedules all have an effect on the ferrite/bainite matrix of CP steels [30]. Grain refinement, microstructural banding, and the morphology of ferrite/bainite grains are influenced by these processing parameters.

2.1.2.1 Band Formation

Banding in steel microstructures is a well-documented phenomenon that occurs because of the macro- and micro-segregation of alloying elements in cast structures. Figure 2.1 shows that upon solidification, a cast structure cools from the outside in, forming small grains in the outer chill zone, columnar grains in the intermediate zone, and large equiaxed grains in the interior zone [29]. Columnar grains form by dendritic arm formation (Figure 2.2) during which solute is rejected from the solid into the liquid to maintain chemical equilibrium [29]. Hot rolling of these cast structures aligns dendritic arms in the rolling direction to form bands of solute-rich phases [29]. The interior zone is the last to solidify, and therefore has the highest solute concentration, which forms a sheet centerline band upon hot rolling [29]. Mn is more likely to concentrate in bands because of its low equilibrium partition ratio (i.e. the ratio of its equilibrium concentrations in solid versus liquid) and because it is present in high amounts [29]. Manganese stabilization of
austenite means that as manganese-free austenite transforms first to ferrite, it will reject carbon into the manganese-rich bands, such that these areas also become enriched in carbon [29].

**Figure 2.1:** Cast grain structure, showing three zones of crystal formation. [29]

**Figure 2.2:** Depiction of the dendrite formation during the solidification of a cast structure showing dark areas where solute is concentrated in the liquid. [29].
2.1.2.2 Prior Austenite Deformation and Controlled Cooling

Controlled rolling of austenite will influence grain structure by introducing pancaked deformation bands, and grain refinement, as illustrated by the schematic in Figure 2.3. Recrystallization during controlled rolling varies by temperature and reduction percentage as illustrated by Figure 2.4. Grain refinement is produced at each stage of deformation, deformation bands are introduced during deformation in the non-recrystallization region, and sub-grain structure is produced by deformation in the two-phase region [30]. Higher finish rolling temperature, lower percent reduction in finish rolling, and slower cooling rate will all promote a larger grain size [30].

![Figure 2.3: Schematic of changes in microstructure with deformation](image)

Figure 2.3: Schematic of changes in microstructure with deformation [31].
Figure 2.4: Recrystallization regions for 0.03% Nb steel obtained with samples reheated for 20 min at 1250°C followed by one rolling pass. [32]

Austenite transformation results in four different types of grain structure depending on the deformation introduced in controlled rolling, as outlined by Fukuda et al. [33]. Deformation in the high temperature recrystallization region will not refine austenite grains, and will therefore produce coarse ferrite nucleated on prior austenite grains along with acicular ferrite for the case of Nb steels. Deformation in the low temperature regions of the recrystallization zone will result in grain refinement of austenite, and a therefore a fine-grained ferrite. Deformation in the non-recrystallization regions will cause the nucleation of ferrite within austenite grains on deformation bands, producing an ultra-fine ferrite grain structure. With deformation in a combination of regions, a mixed microstructure can develop due to partial recrystallization, leading to a variety of ferrite grain sizes based upon local variations in recrystallization. Mixed microstructures will have some equiaxed grains formed in areas of recrystallized austenite and some elongated grains formed in pancaked unrecrystallized austenite.
Once a mixed microstructure is formed, large grains will persist throughout rolling [31]. Partially recrystallized microstructures will consist of fine grains that form on grain boundaries, with areas in between where larger grains will also be allowed to form. Because recrystallization of mix microstructures will preferentially occur in finer, previously recrystallized grains, the large grains of the microstructure will be more likely to remain unrecrystallized. It is particularly true of CP steels containing Nb that recrystallization will be retarded, leading to higher potential for mixed microstructures. Grain refinement in the recrystallization region will help reduce heterogeneity in grain size during partial or non-recrystallization deformation steps [31].

The transformation of austenite to bainite and ferrite can be effected by stress and texture in the parent austenite grains [34]. Therefore, prior deformation of austenite grains during finish rolling will affect the subsequent transformed phases and morphology, and transformation temperatures for forming the CP steel microstructure. The representative continuous cooling transformation (CCT) curves of Figure 2.5 help to explain this microstructural evolution.

![CCT curves of CP steel showing a) recrystallized austenite transformation, and b) pancaked austenite transformation. PF=polygonal ferrite, B=bainite, GB=granular bainite, M=martensite. [35]](image)

Deformation during finishing can change the characteristics of the relative bainite/ferrite/MA transformation from austenite. Polygonal ferrite will nucleate on prior austenite grain boundaries
due to high strain energy at the deformed boundaries [22]. Acicular ferrite will form when there is deformation in the non-recrystallization region, which provides sufficient nucleation potential intragranularly, and when the cooling rate is high enough [31,36,37]. Bainite and ferrite are in competition when either the potential for nucleation is low or the cooling rate is low [37]. With greater deformation there is an increase in the amount of MA, and a decrease in the bainite start/finish temperatures due to mechanical stabilization [20,37].

The development of texture in parent austenite grains during rolling is inherited by the final microstructure. During austenite deformation, the major textures developed are the brass and copper texture which are the \{110\}⟨112⟩ and \{112\}⟨111⟩ textures respectively, which transform to the \{332\}⟨113⟩ and \{113\}⟨110⟩ ferrite orientations respectively [38]. The benefit of the \{332\}⟨113⟩ texture is that it promotes greater strength, less anisotropy, and greater toughness [20].

The intensity of the \{332\}⟨113⟩ texture is promoted by lower finishing temperatures, with greater amounts of deformation [20]. Niobium will increase the \{332\}⟨113⟩ texture by retarding austenite recrystallization [20]. Additionally, greater cooling rates will also promote this texture.

\section*{2.2 Complex-phase Steel Mechanical Properties}

The extreme grain refinement, presence of carbides, solid solution alloying, and characteristics of the bainite-ferrite matrix contribute to the strength and formability of CP steels through a combination of mechanisms.

The strength of CP steels is derived from common steel strengthening mechanisms such as solid solution strengthening, grain refinement, and precipitation hardening, but also more unique mechanisms of upper bainite strengthening, and dislocation density [22].

Precipitation of four different sizes of precipitates occurs, which are formed at four different times during rolling and cooling. Coarse precipitates (>100nm) form in the austenite region, fine
precipitates (>20-50nm) form by strain-induced precipitation during cooling, fine precipitates (>10-20nm) are formed in the high-temperature ferrite region, and very fine precipitates (3-10nm) form during coiling [19]. Coarse and fine precipitates will be incoherent with surrounding grains, and will therefore be responsible for grain refinement, whereas very fine precipitates will be coherent and contribute to hardening [19].

Mechanical properties of bainite in CP steels are derived from cementite particles, MA, and precipitated carbides. Irregular/coarse ferrite/cementite/MA aggregations are produced due to low carbon content [39]. This type of granular bainite will likely resist dislocation motion on lath boundaries, which contributes to strength differently than the dispersion strengthening of fine-cementite lower bainite [19]. The previously noted precipitation strengthening effect of Nb and Ti carbides also contributes to bainite strengthening.

Dislocation density also plays a major role in CP steels. Dislocation density varies between grains as well as within grains [19]. This is a characteristic of high-strength micro alloyed steels that exists because of their CP microstructure. The irregularity and density of dislocations emerges as a result of the low-temperature bainite, and acicular ferrite transformation [19].

Toughness in CP steels is derived from their reliance on grain refinement, which does not reduce ductility as much as other strengthening mechanism [39]. Ductility is reduced with lower finishing temperatures, signaling that greater dislocation density strengthening comes at the cost of elongation [30].

2.3 Void Damage Evolution

Void damage is of interest in CP steels because the difference in strength between phases contributes to the nucleation of voids through strain incompatibility [40]. Nominal stress in the material is transferred through weaker microstructural constituents to stronger ones via a shear
transfer of stress along phase boundaries. Microscopic evidence of this mechanism of stress transfer can be seen in the development of shear bands at ferrite-martensite grain boundaries. Shear bands are seen during deformation of ferrite grains around martensite particles, as is shown in the micrographs of Figure 2.6 [40]. The strain incompatibility between phases leads to the potential for void damage due to particle or phase boundary failures. Void damage occurs progressively by void nucleation, growth, and coalescence [42].

![Figure 2.6: Development of slip lines in ferrite grains with strain increasing from a → c. [40]](image)

Void nucleation, growth, and coalescence are dependent on the stress state of the material both macroscopically and microstructurally. The tensile hydrostatic component of the stress state, i.e. the triaxiality of stress, will contribute to the evolution of void damage. Stress triaxiality can result from macroscopic flaws in the material such as notches and other stress concentrators, or from microstructural features like hard martensite grains or grain boundary triple points [43].

### 2.3.1 Void Nucleation

Void nucleation occurs when local stress in the microstructure exceeds a critical value corresponding to the grain boundary strength of local constituents [42,44]. Given the critical stress criteria, void nucleation can occur by cracking of hard phases, grain boundary decohesion
between hard and soft phases, or grain boundary decohesion between like phases [45]. The shear transfer of stress from soft to hard phases makes these grain boundaries particularly vulnerable, but hard phase precipitation on grain boundaries can weaken any grain boundary and provide a site for void nucleation [23]. As the critical stress is reached locally at different locations within a microstructure, voids will continually nucleate at increasing strains [46]. An example of void nucleation between ferrite and martensite grains can be seen in Figure 2.7.

![Figure 2.7: Nucleation of voids at martensite (light) – ferrite (dark) phase boundaries. [42]](image)

### 2.3.2 Void Growth

After nucleation, voids will continue to grow in the direction of stress application [45]. In the case of high stress triaxiality, voids will be encouraged to grow in three dimensions, as is the case for notched tensile samples [47].

### 2.3.3 Void Coalescence

Once voids reach a critical volume fraction, the load carrying capacity of a material is compromised, such that failure will occur by void coalescence in a ductile fracture event [44,48]. Void coalescence occurs by two mechanisms that have been observed. In one mechanism, shear
bands form between neighbouring voids, which results in the severing of the inter-void ligament [49]. This mechanism of void coalescence can result in a plane of voids known as void sheeting. The other mechanism of void coalescence is by the necking of inter-void ligaments to the point of void-void impingement [47,49].

2.4 Intergranular Fracture

Intergranular fractures in steels can be generally categorized as cracking failures that take place along grain boundaries due to the loss of cohesion [50]. Generally, impurities and alloying elements (Table 2.3) at grain boundaries lead to weakening and intergranular fracture [50,51]. In support of this mechanism, it has been shown that the degree of intergranular fracture in steel is proportional to the intergranular impurity concentration [52]. Prior austenite grain boundaries tend to provide locations for intergranular fracture due to segregation of impurities [31]. Diffusion of solutes to grain boundaries can occur independently, but solutes can also cooperatively diffuse due to mutual attraction [50]. Particular solutes such as Cr have been shown to promote the segregation of other solutes such as Ni/P or Ni/Sb, without itself participating in segregation to grain boundaries [50]. Additionally, formation of Fe₃C on prior austenite grain boundaries has been shown to cause embrittlement by rejecting solutes as it forms along the boundary [50]. In general, solute concentrations on grain boundaries will reduce cohesion of those boundaries, which can lead to embrittlement [53].
Table 2.3: Elements by periodic table group that contribute to grain boundary weakening in steel [50].

<table>
<thead>
<tr>
<th>IVB</th>
<th>VB</th>
<th>VIB</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>N</td>
<td>S</td>
</tr>
<tr>
<td>Ge</td>
<td>P</td>
<td>Se</td>
</tr>
<tr>
<td>Sn</td>
<td>As</td>
<td>Te</td>
</tr>
<tr>
<td></td>
<td>Sb</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Bi</td>
<td></td>
</tr>
</tbody>
</table>

2.5 X-ray Micro-computed Tomographic Damage Characterization

Typically, void damage characterization takes place via two dimensional analyses using SEM or optical micrography [44,49,54]. Attempting to determine three dimensional values from two dimensional sections requires serial sectioning of an entire volume. It is a much more attractive option to measure values such as void volume fraction with a single non-destructive measurement. Additionally, it is difficult to obtain spatial or morphological information about voids via two dimensional methods of investigation. It has been shown that X\textsubscript{µ}CT scanning can be used to image void damage in high strength steel, such that three dimensional information about damage can be ascertained [12,15,18,55,56].

2.5.1 X-ray Tomography Fundamentals

X-ray tomography relies on differences in absorption of X-rays between areas of different density within a volume. X-rays are transmitted from a source, towards a volume of interest, and incidents of X-ray transmission are measured by a one or two dimensional charged couple display (CCD) which sits opposite the X-ray source, on the other side of the sample. Figure 2.8 shows a typical X-ray tomography scanning setup.
The source of X-rays produced in lab-scale devices is of a cone-beam, polychromatic type [13]. Inside an X-ray tube, a large potential is generated between a cathode and anode, which causes electrons to travel from the cathode to the anode and impact the atoms of the anode material. Traveling electrons can dislodge electrons of the anode material’s atoms, causing an emission of a characteristic amount of X-ray energy (characteristic radiation) when a replacement electron drops down to fill the lower-energy position [58]. Another form of X-ray energy production is called Bremsstrahlung radiation, which is comprised of a spectrum of energy values emitted from the path-change interactions of traveling electrons with anode material atoms [58].
X-ray absorption/transmission is linked to the absorption coefficient of the material being illuminated by the beam. The local composition of a material determines the absorption coefficient for a given point within a volume because absorption coefficient, \( \mu \), is proportional to density, \( \rho \), and atomic number, \( Z \), according to:

\[
\mu = K \rho \frac{Z^4}{E^3}
\]  

where \( K \) is a constant and \( E \) is the energy of the incident photons [13].

X-ray tomography depends on the ratio of transmitted to incident X-rays along a given X-ray flight path, which is equal to the integral of the absorption coefficient along the path [14].

Tomography reconstructs 2-D slice-wise data based upon variations in X-ray flight path transmission ratio with respect to sample orientation [58]. X-ray transmission is recorded over a range of angular orientations, typically subdivided from an overall arc of at least 180 degrees. A filtered back-projection algorithm, which is a proprietary algorithm of Xradia Inc. for the reconstructions within this thesis, is used to map X-ray transmission variation to differences in material density at a given point in the reconstructed slice. Differences in density manifest as differences in greyscale intensity for each pixel within the reconstructed slices.

### 2.5.2 X-ray Tomography Artifacts

Undesirable image features are known to result as artifacts of phenomena associated with the X-ray beam, scintillator screen, and CCD [58,59]. Beam hardening is the brightening of pixels towards the outer regions of a sample in reconstructed slices, caused by the higher degree of X-ray transmission through thinner edge regions over the course of rotation [59]. Ring artifacts result from imperfections in the scintillator screen that converts X-rays to the visible spectrum. Because the scintillator is consistent with respect to rotation, these artifacts manifest as rings on
the reconstructed slices [59]. Streak artifacts are caused by high energy X-rays that occasionally pass through the scintillator screen and impact the CCD, resulting in dark linear streaks across the reconstructed slice. Figure 2.9 shows examples of X-ray micro-computed tomography artifacts.

![Figure 2.9: Examples of image artifacts as they appear in 2-D reconstructed slices, showing a) beam hardened slice with greyscale histogram values superimposed, b) slice showing ring artifacts, and c) slice showing streaking [59]](image-url)
2.5.3 Past X-ray Micro-computed Tomography (XµCT) Studies

Some of the void damage investigations noted in Section 2.3 involved the use of XµCT. Observation of continual void nucleation with increasing strain comes from the studies of Maire et al. [60], showing that both the number of voids increases dramatically at high strains and that the average void size stagnates due to the nucleation of many small voids (Figure 2.10). Another important observation of Maire et al. [56] is confirmation that stress triaxiality towards the central regions of tensile samples, and due to notch induced triaxiality, will induce greater amounts of void damage. 3-D renderings of voids at increasing strain from the study of Maire et al. [56] are shown in Figure 2.11.

![Figure 2.10](image)

**Figure 2.10:** Data showing a) the stagnation of average void diameter, despite model predictions, and b) the increased nucleation of voids with increasing strain. [60]
Other work has investigated void damage evolution in TWIP steels. Lorthios et al. [61] showed that voids in these steels tend to grow along the rolling direction of the material, as in studies of dual phase steels [16,18]. However, void volume fraction of failed materials was found to be much lower for TWIP steels. Whereas dual phase steels can reach void volume fractions of 2% at failure, failed TWIP samples were found to have void volume fractions around 0.02% [61].

Because steel is a high density material, it is uncommon for XµCT studies to be done using lab-scale X-ray sources that require very long scan times. A study by Gupta et al. [17] is one study that employed lab-scale XµCT to measure void damage. This study revealed void damage with void diameters ranging from 5-19 µm, and a void volume fraction of 0.03% at 17% nominal strain in the scanned volume.

It is most common to conduct tomography experiments on steel using a synchrotron X-ray source. Synchrotron X-ray sources are highly collimated, high flux energy sources [14]. High flux sources are able to provide sufficient X-ray transmission data in a much shorter time than lab-scale sources [14]. Collimation allows for significant increases in the signal-to-noise ratio, which

![Figure 2.11: 3-D renderings of voids in a volume of notched material in uniaxial tension [56].](image)
also reduces necessary scan times [14]. For these reasons, a higher resolution scan can be completed in significantly less time using synchrotron X-ray sources versus polychromatic cone sources such as the one used in this thesis.

2.6 Hole Expansion

Hole-expansion testing by Misra et al. [6] reported that results that do not correlate well with UTS, yield strength, or elongation, and vary significantly with only minor changes in chemistry and processing. Instead, microstructural features have been shown to be well correlated with hole-expansion performance. Specifically, the study of Misra et al. [6] demonstrated that hole expansion performance was negatively associated with the intensity of banding (including coarse ferrite), but positively associated with banding wavelength and existence of bainite. Other variations in these microstructures such as dislocation density, martensite volume fraction, and ferrite grain size correlated expectedly with uniaxial tensile properties, but exhibited anomalous correlations with hole-expansion.

Microstructures tested for variations in hole-expansion with variations in chemistry by Fang et al. [62] revealed similar results as above. Two main detriments to hole-expansion noted in this study were increased carbon weight percent, and greater difference in strength between microstructural constituents. The solid solution strengthening of Si in ferrite was shown to improve hole-expansion performance by reducing the strength difference between phases [9]. Fang et al. struggled to explain the anomalously poor hole-expansion behaviour in the microstructure of Figure 2.12 (d). Given the four microstructures of the study, it is evident that the microstructure in question shows significantly more banding and a higher volume fraction of non-ferritic phase than the other microstructures.
Figure 2.12: Four microstructures compared for hole-expansion behaviour in the study of Fang et al. [62]
Investigation of the relationship between hole edge quality and hole-expansion performance has revealed that cracking resistance is crucial to the prevention of hole-expansion failures [11]. Intergranular cracking was shown to be the dominant mode of cracking failure in hole-expansion. Microplasticity is therefore shown to be a more beneficial damage mechanism than intergranular cracking for promoting hole-expansion.

2.7 Contribution to the Literature

The preceding review of the literature has shown that studies of void damage evolution in sheet steels has taken place [12–18], as well as studies of hole-expansion of CP steels [5–9]. No studies have combined X\(\mu\)CT measurements with the study of damage evolution in CP steels.

The contribution of this thesis will be to measure the 3-D evolution of internal damage in CP steels. This thesis aims to correlate measures of damage evolution obtained by X\(\mu\)CT with microstructural characteristics of CP steel variants. The literature has shown that mechanical properties do not correlate well with hole-expansion performance [6], and that correlation of microstructural trends with hole-expansion can be anomalous [62]. This study will contribute to the literature by providing correlations between microstructure and hole-expansion performance via measurements of internal damage evolution.
Chapter 3

Materials and Experimental Methods

This chapter details the characteristics of the steel sheet materials received from ArcelorMittal Dofasco, as well as the methods used to perform mechanical tests and collect microstructural and damage data. Sheet thermal histories, compositions, and mechanical properties are provided. Descriptions of metallographic sample preparation are included, along with details of the double notched uniaxial tensile testing methods. Finally, the methods used to examine damage are outlined, including metallography, XµCT, and fracture analysis.

3.1 Received Materials Characteristics

Four hot-rolled steel variants of the grade designation ‘HR780SF’ were received, where HR designates ‘hot rolled’ and SF designates ‘stretch flangeable’. All four variants were from the same heat, and therefore had the same composition as presented in Table 3.1. Mechanical properties under transverse uniaxial tensile loading, coiling condition, and cooling classification of the four variants can be seen in Table 3.2, as measured by ArcelorMittal Dofasco [63].

<table>
<thead>
<tr>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Nb</th>
<th>Ti</th>
<th>Cr</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.05</td>
<td>1.5</td>
<td>0.55</td>
<td>0.03</td>
<td>0.15</td>
<td>0.6</td>
<td>Balance</td>
</tr>
</tbody>
</table>

Table 3.1: Elemental composition (wt%) of all steel variants received from ArcelorMittal Dofasco [63].
Table 3.2: Physical properties and transverse uniaxial tensile properties of received steel variants, with two pairings based on sheet thickness. Variants are designated numerically, e.g. V1 designates Variant 1 [63].

<table>
<thead>
<tr>
<th></th>
<th>V1</th>
<th>V2</th>
<th>V3</th>
<th>V4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gauge (mm)</td>
<td>2.9</td>
<td>2.9</td>
<td>3.2</td>
<td>3.2</td>
</tr>
<tr>
<td>Cooling</td>
<td>2 Step</td>
<td>1 Step</td>
<td>2 Step</td>
<td>2 Step</td>
</tr>
<tr>
<td>Coil</td>
<td>Far</td>
<td>Near</td>
<td>Far</td>
<td>Far</td>
</tr>
<tr>
<td>Edge of Coil</td>
<td>Leading</td>
<td>Trailing</td>
<td>Leading</td>
<td>Trailing</td>
</tr>
<tr>
<td>Yield Strength (MPa)</td>
<td>791</td>
<td>833</td>
<td>789</td>
<td>804</td>
</tr>
<tr>
<td>UTS (MPa)</td>
<td>866</td>
<td>900</td>
<td>868</td>
<td>881</td>
</tr>
<tr>
<td>Elongation (%)</td>
<td>15.3</td>
<td>16.1</td>
<td>16.5</td>
<td>14.3</td>
</tr>
<tr>
<td>Hole Expansion % (σ)</td>
<td>70.7 (6.2)</td>
<td>25.5 (2.2)</td>
<td>63.3 (12.7)</td>
<td>44.9 (8.7)</td>
</tr>
</tbody>
</table>

Variants were cooled from the finish rolling mill via one- or two-step water spray cooling before coiling. Cooling schematics constructed from ArcelorMittal Dofasco data [63] for each variant can be seen in Figure 3.1 and Figure 3.2. Coiled sheets were allowed to cool in an open-air environment. Complete roughing, finish rolling, and cooling path data is documented in Appendix B. The CCT curves of Section 2.1.2.2 act as a guide for explaining the evolution of microstructure in each variant.
Figure 3.1: Cooling schematics (part 1 of 2) showing thermal path between finish rolling and coiling.
Figure 3.2: Cooling schematics (part 2 of 2) showing thermal path between finish rolling and coiling for a) V3 and b) V4.
3.2 Metallography

Metallographic examinations were carried out to characterize the microstructures of received sheet variants. Investigations were completed through optical microscopy. The goal of metallography was to identify phases, phase morphology, phase distribution, relative grain size, and martensite/retained austenite volume fraction.

3.2.1 Sample Preparation

Samples were prepared for polishing by a two-stage extraction and mounting method:

1. Sections of material approximately 40 x 40mm were cut with a hacksaw from the received sheet material, with care being taken to note the rolling direction (RD) and transverse direction (TD) for future reference.

2. 40 x 40mm sections were cut to rectangles measuring approximately 15mm in the RD and 10mm in the TD using a Struers Accutom-5 Automatic precision cut-off machine equipped with an aluminum oxide-containing Bakelite cut-off wheel.

3. Rectangular samples were mounted in thermosetting pucks using a Buehler SimpliMet 1000 automatic mounting press. Diethyl phthalate was used as a hard surface material for the puck to promote edge retention of the sample during grinding and polishing, while Bakelite was the remainder of the puck.

Grinding and polishing of each sample were carried out in accordance with ASTM Standard E3-11 [64]. One additional water polish of approximately 45s on a rotational polishing wheel using a synthetic suede polishing cloth was conducted following the final colloidal silica polishing stage to removing remaining colloidal silica particles.
3.2.2 Etching Procedure

A variety of etchants were implemented to reveal microstructural features. In the case of all etchants, etching times were varied on a sample-to-sample basis to attain the optimal degree of etching and contrast.

Three different etchants were used to reveal different features of the as-received material microstructures through optical microscopy. Sodium metabisulfite, LePera, and Klemm’s etchants were prepared based upon the recommendations of ASTM Standard E407 – 07 [65], Hairer et al. [66], and Bramfitt et al. [24] respectively. To reveal general features of ferrite, bainite, and martensite in the multi-phase microstructure of the HR780SF materials, the LePera and sodium metabisulfite etchants were used. Klemm’s etchant was used to simultaneously highlight all MA, while darkening other phases.

3.2.3 Microstructural Characterization

All optical microscopy was carried out using a Zeiss Axioskop 2 at 100x and 500x magnification through air, and 1000x magnification through an oil immersion lens.

Qualitative and quantitative characterization of microstructure was carried out using the images obtained for each variant. Phases were identified by the known colouring effect of each etchant on a given phase in steel microstructures. The uniformity of phase distribution was noted, i.e. differences in banding of certain phases or distribution of features of different sizes. Changes between the sheet centerline and quarter point were used to determine microstructural changes through the thickness of the sheet. MA volume fraction was measured by thresholding out white-shaded MA particles in Klemm’s-etched microstructures. Multiple measurements were taken at the centerline and quarter point of each variant. ImageJ software was used for thresholding of MA particles and determining their centroids.
3.3 Double-notch Uniaxial Tensile Testing

Interrupted mechanical testing of the variants in this investigation was carried out by transverse tensile testing of uniaxial dog-bone samples containing a double-notch at the center point of the sample gauge region. Notching of samples was used to induce damage within a focused region. It was necessary to consistently induce damage in the same region of material so that a sub-volume of material could be extracted from the same region of every tensile sample for subsequent XμCT imaging.

3.3.1 Sample Geometry and Preparation

A notched geometry was chosen to localize internal void damage to a consistent region of all tested samples. A standardized geometry was chosen for reproducibility, corresponding to ASTM E292 – 09 Specimen 4 [67], with the dimensions shown in Figure 3.3. Samples were cut from the as-received sheet, such that the samples thickness equaled the sheet thickness of the given variant.

![Diagram of the double-notch dog bone samples used in the tensile testing of sheet variants. Geometry comes from Specimen 4 of ASTM E292 – 09. All dimensions are in millimeters. [67]](image)

**Figure 3.3:** Diagram of the double-notch dog bone samples used in the tensile testing of sheet variants. Geometry comes from Specimen 4 of ASTM E292 – 09. All dimensions are in millimeters. [67]
Sample dimensions were chosen as a compromise between a large geometry with a uniform strain gradient over most of the notch region, and a small geometry with the ability to image the entire notch region within the field of view (FOV) of the XµCT objective lens. It is ideal for the volume of material being imaged to originate from a uniform strain region, which requires that the volume be away from the notch strain field. As well it is ideal for the volume of material imaged to be representative of as much of the sample as possible. The implication of the chosen geometry is that, given symmetry, one volume of material exists adjacent to the tensile centerline that is a sufficient distance from the notch strain field, while also representing nearly 50% of the linear space between the notches. The area between the notches selected for imaging can be seen in Figure 3.4.

![Diagram showing extracted area for XµCT imaging](image)

**Figure 3.4:** Magnified depiction of the area between the notches of double notched tensile samples, showing the area to be imaged during XµCT, showing hypothetical strain gradient in red.

Once all samples were machined, real dimensions were measured to maintain a record of notch variability. Distance between notches, notch depths, and notch root radii were recorded for both sides of all samples.
3.3.2 Tensile Testing Procedure

Interrupted tensile testing was carried out to induce void damage without allowing for the coalescence of voids into a failure event. Interrupted testing provided the greatest potential for retention of void data, as a coalescence event destroys the ability to measure all of the voids that contribute to the event. To induce different levels of void damage, tests were allowed to reach peak load, and then the test was allowed to continue until the load dropped by 2.5%, 5%, 7.5%, or 10% from the peak, as depicted in the Figure 3.5 example of a 10% load drop. At each load drop, three samples were tested per variant to allow for selection of the sample with the most uniform strain in the notch region for subsequent XμCT imaging. All tests were performed under cross-head extension control at a rate of 1.14mm/min, which was chosen based upon information from Zengtao Chen [68] regarding previous work of Chen and Datta [43].

![Figure 3.5](image)

**Figure 3.5:** Example plot of load versus extension during uniaxial tensile testing, showing 10% load drop from peak load. Test were carried out to four different levels of load drop.
3.3.3 Strain Measurement

3.3.3.1 Gridding

A grid of dots was applied to the notched region of each tensile sample, such that a measure of strain could be made after tensile testing. Dots were applied in an array of 0.5mm squares. Eleven dots were applied in the tensile direction and nine dots were applied in the transverse-tensile direction. A custom 2-axis micro-controlled stage with a spring-loaded marker grip was used to apply the grids. A photograph of the overall plotting assembly, and the application of the grid can be seen in Figure 3.6.

![Figure 3.6: Photographs showing a) the custom setup used to apply dot grids, and b) close-up of an example of a 9x11 grid with 0.5 mm spacing during grid application.](image-url)
3.3.3.2 Strain Measurements

Strain measurements in deformed tensile samples were made based upon the differences between the dot spacing of undeformed and deformed grids. Images were taken under a stereographic microscope at 1.2 X magnifications with image acquisition using an attached 1024 x 768 digital camera.

![Undeformed Grid](image1.png) ![Deformed Grid](image2.png)

**Figure 3.7**: Example of grid images taken with stereographic microscope and attached digital camera.

ImageJ software was used to threshold the black dots from the steel background and then determine the centroids of the dots. The process was used for both undeformed and deformed grids. Using MATLAB the dots were arranged in groups of four, corresponding to the corners of a strain “element”. Subsequently the undeformed and deformed element coordinates were fed into a strain calculation function that produced the major and minor engineering strain for each element. Using the strains from each element, basic two dimensional strain maps, as in Figure 3.8, were plotted for each tensile sample. Strain maps were used to identify the sample with the most uniform strain among all four central quadrants. From within the identified sample, the
central quadrant with the lowest standard deviation in major strain was selected for extraction and XµCT imaging.

![Figure 3.8 Example engineering strain map produced by the measurement of strain for each four-cornered element. Central quadrant with lowest standard deviation of strain is highlighted. Major strain corresponds to the tensile loading direction.]

**3.4 X-ray Micro-computed Tomography of Damage**

Internal measures of damage were made via XµCT scans using an Xradia MicroXCT-400 system. Void damage was collected for extracted sections of tensile samples, with known nominal strain. This allowed values such as void volume fraction, void number, and void size to be plotted for each variant as a function of nominal strain. Damage measurements obtained at increasing mean major strain levels are each taken from a different sample, due to the destructive nature of the XµCT sample extraction.
3.4.1 Specimen Preparation

Imaging samples were extracted from the notch region of tensile samples as per the steps outlined Figure 3.9. The 0.8mm square cross-section was chosen so that it would fit within the cylindrical FOV of the MicroXCT-400 scan volume. Half of the notch region was first cut out, with a 0.8mm thickness, and then the sheet thickness of the tensile sample was manually ground down to 0.8mm. Grinding was completed on a rotating wheel with 320 grit abrasive paper. The sample was secured in a grip to maintain a level surface while grinding each side. The sharp notch was ground off of one side to maintain a reference for sample orientation. A micrometer was used to monitor the amount of material being removed from each side to maintain symmetry about the sheet centerline.

Once extracted and resized, the imaging samples were mounted onto an extender rod. The extender rod was necessary to prevent the large sample holder from impeding the range of motion of the MicroXCT-400 X-ray source and detector structures. Samples were mounted onto a thin cylindrical rod with LePage Epoxy Steel. The final imaging sample, mounted and held in the pin-vise sample holder can be seen in Figure 3.10.
Figure 3.9: Imaging sample extraction method, showing a) cutting of 0.8mm strips, and b) manual grinding of both sides of the sheet to reduce thickness to 0.8mm.

Figure 3.10: Complete mounting setup showing a) extracted sample, b) extension rod, and c) pin vise sample holder.
3.4.2 Tomographic Acquisition

Acquiring tomographic scans involved placing samples in the XμCT cabinet, positioning them centrally, and determining the optimal beam parameters to maximize the signal-to-noise ratio in the resulting scans. Proprietary “XM Controller” software from Xradia was used to control the sample stage, X-ray source, and detector bank of the system. During tomographic image captures, the sample was rotated through 184°, during which several thousand projections/images were captured at incremental angles of rotation. The parameters used for image acquisition in the MicroXCT-400 in this investigation can be seen in Table 3.3.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Projections</td>
<td>3200</td>
</tr>
<tr>
<td>Exposure Time (s)</td>
<td>42</td>
</tr>
<tr>
<td>Source Voltage (kV)</td>
<td>100</td>
</tr>
<tr>
<td>Source Power (W)</td>
<td>10</td>
</tr>
<tr>
<td>Objective</td>
<td>20x</td>
</tr>
<tr>
<td>Source to Sample Distance (mm)</td>
<td>4.8</td>
</tr>
<tr>
<td>Detector to Sample Distance (mm)</td>
<td>28.8</td>
</tr>
<tr>
<td>Sample Rotation</td>
<td>184</td>
</tr>
<tr>
<td>Filter</td>
<td>LE#5</td>
</tr>
<tr>
<td>Binning</td>
<td>2</td>
</tr>
<tr>
<td>Multiple Reference Imaging</td>
<td>Average of 5 exposures every 800 projections</td>
</tr>
<tr>
<td>Dynamic Ring Removal</td>
<td>On</td>
</tr>
<tr>
<td>Camera Readout Time</td>
<td>Fast</td>
</tr>
</tbody>
</table>

3.4.2.1 Position

The sample was positioned in the cabinet of the XμCT, and in the FOV of the detector. The MicroXCT-400-compatible pin-vise sample holder was placed on the sample stage inside the XμCT cabinet. The source and detector were brought within a few centimeters of the sample at this time, but not yet as close as possible, as can be seen in Figure 3.11. At 0.5x, 4x, 10x, and
finally 20x magnification, the area of interest was centered in the X and Z axes in the FOV. The sample Y axis was adjusted so that the area of interest was centered vertically in the FOV. The detector bank was brought within one millimeter of touching the sample, and the source was brought to a distance that maintains the 6:1 source-to-detector distance-to-sample ratio. This ratio was found to be best for reducing edge blurring of the sample’s shadow on the detector, which is caused by partial (versus complete) light obstruction at the edges of the shadow.

Figure 3.11: Inside of the Xradia MicroXCT-400 cabinet showing X-ray source, sample stage, and detector positioning.

3.4.2.2 Filter

To produce tomographic images with the best signal-to-noise ratio, it was necessary to optimize the X-ray beam parameters. Samples were rotated to an angle that best represented an
average of the thickness, i.e. the square cross-section samples of this investigation were rotated to 22.5° from one of the square faces. At this angle a variety of long and short X-ray flight lengths through the sample are represented. A beam filter was first selected via the procedure outlined in Xradia Filter Selection Guide [69] to remove low energy X-rays that are more likely to be attenuated at the center of the sample than at the edges. Removal of these low energy X-rays reduces beam hardening, such that less centre to edge variation of the greyscale histogram will be evident.

3.4.2.3 Source Voltage

The X-ray source voltage was adjusted so that at 10W of power, there was a 25-35% transmission of X-rays through the area of interest during a given projection. This transmission ratio is suggested by Xradia for best image quality [69].

3.4.2.4 Exposure Time

The time for each projection was adjusted such that at least 2000 X-ray counts was received through each pixel on the CCD. The optimal number of X-ray counts is suggested by Xradia for best image quality [69].

3.4.2.5 Binning

Binning combines adjacent pixels of the CCD. The number quoted for binning represents the number of pixels combined in both directions of the CCD, e.g. a binning of 2 represents a combination of a 2 x 2 pixel array into one new virtual element on the CCD. With a binning of 1, the exposure time required to obtain 2000 counts through each pixel was about 7 minutes, which translates to a scan time of 18 days per sample. Because of time constraints a binning of 2 was
selected, which reduced the necessary exposure time to 45 seconds, resulting in a scan time of 2 days per sample.

3.4.2.6 Rotation

Using 180 degrees of rotation in XμCT scans with a cone-beam source can produce cone angle defects [58]. To remove the possibility of these defects, a rotation of 184 degrees was chosen.

3.4.2.7 Projections

The number of projections was selected based upon the minimum number required to satisfy the Nyquist criterion for angular sampling [58], which is defined by:

\[ N_{\text{min}} \geq \frac{md}{\Delta r} \]  

(3.1)

where \( d \) is the diameter of the FOV, and \( \Delta r \) is the linear sampling distance or the linear dimension of one pixel on the CCD. With binning of 2 and a 2048x2048 CCD, the minimum number of projections required was approximately 1560. Given that the above equation is for parallel beam geometry, oversampling of 3200 projections was used to ensure good image quality.

3.4.2.8 Dynamic Ring Removal

Dynamic ring removal, also known as dithering, was used to remove ring artifacts. The software randomly translates the sample stage in three axes between projections, and records these movements to be accounted for in subsequent image reconstruction. The ring artifacts, being a function of defects in the scintillating material of the detector, are smeared by the random movements of dynamical ring removal. The result is an image where ring artifacts are suppressed.
3.4.2.9 Multiple Reference Imaging

Multiple reference imaging, also known as flat-fielding, was used to normalize the projections with respect to the background noise of the beam through air. During flat-fielding, the sample is moved out of the field of view and reference images are taken using the same magnification and source conditions, and the projections through the sample are divided by these reference images. This normalization removes background noise as well as the “fish bowl” effect of the cone beam being brighter at the center than the edges. Reference images can be taken at many points during a tomographic scan and averaged, to account for potential changes in the scanning environment over the duration of the acquisition process.

It is the recommendation of Xradia Inc. that reference images be taken at intervals of one quarter of the total number of projections and to take five reference images at each interval. Therefore, for the scans performed in this study, five reference images were taken at intervals of 800 frames.

3.4.3 Post-tomography Processing and Reconstruction of Images

Files collected during tomographic scans using the MicroXCT-400 are in a .txrm Xradia Inc. file format. Files need to be reconstructed using Xradia software, which implements a proprietary filtered back-projection algorithm to generate one voxel-thick slice-wise greyscale maps of the scan volume. Greyscale intensities on a voxel-by-voxel basis in reconstructed slices are meant to correspond to changes in X-ray attenuation. Therefore greyscale values are meant to map to differences in density.

Filtering was carried out, and two correction factors were identified to be input into the reconstruction recipe. These factors account for image artifacts inherent to tomographic image capture, and ensure the best image quality.
3.4.3.1 Despeckling

Despeckling was carried out on all .txrm tomography files to help remove streak artifacts prior to reconstruction. The despeckling filter of Xradia Inc.’s XMController software was used to perform Despeckling.

3.4.3.2 Center Shift Correction

During the reconstruction procedure, implemented in Xradia’s XMReconstructor software, a center shift factor was chosen. This factor corrects for the vertical axis of the sample not being perfectly centered on the CCD. To find the center shift correction, a single slice is reconstructed with a range of correction factors, and the center shift correction factor that results in the least amount of blurring of features is identified.

3.4.3.3 Beam Hardening Correction

X-rays will tend to be attenuated more frequently given a longer path through a material versus a shorter one. Therefore X-rays tend to be attenuated more frequently passing through the middle of samples than the edges. In reconstructed images, the result of global differences in X-ray attenuation is increasing greyscale intensity towards the edges of the sample, which is also called beam hardening.

To account for beam hardening, sample slices were reconstructed with a range of different beam hardening correction factors. Whichever factor was found to flatten the greyscale histogram across the whole sample was selected for input to the reconstruction recipe. A beam hardening factor in the range of 0.1-0.2 was required for all samples in this study.
3.4.4 Image Processing

Image processing was required to extract meaningful void damage data from the slices produced by the reconstructions. Noise inherent to the polychromatic X-ray source needed to be flattened before thresholding could be applied to capture a binary image representing void/non-void areas. Parameters used in denoising and thresholding can be found in Appendix C.

3.4.4.1 Denoising

Because the MicroXCT-400 employs a polychromatic X-ray source, changes in material attenuation of X-rays do not map directly to changes in greyscale intensity in reconstructed slices. Thus, a denoising of the image was required to smooth the data and allow for small features to be resolved. The work of Sloan [70], showed that a particular non-local means denoising algorithm of Baudes et al. [71] gave the best results for a study of dual-phase steel using the MicroXCT-400 with similar scan parameters to this investigation. Non-local denoising computes “true” greyscale values as a weighted average of all other pixels in the image, with larger weight given to pixels that have similar pixel neighbourhoods [71].

The denoising algorithm was downloaded in the form of a MATLAB script written by Jose Vicente Manjon-Herrera from the MATLAB Central File Exchange [72]. A custom script was written to run the denoising algorithm on stacks of reconstructed slices.

3.4.4.2 Thresholding

To convert the reconstructed and denoised slices from greyscale to binary, a locally adaptive thresholding technique was used. The work of Sloan [70] showed that the algorithm written by Guanglei Xiong [73], which was contributed to the MATLAB Central File Exchange, performed optimally for images such as those acquired in this study. This type of thresholding compares
pixels within a specified neighbourhood and counts the pixel as background if it is significantly darker than average or as foreground if it is significantly brighter. One artifact of this method of thresholding is that the edge of each sample was captured as one large region of background, corresponding to the entire outside surface of the sample. These artifacts were easily removed using a combination of volume-based and position-based filtering in the 3-D visualization software. As with denoising, a MATLAB script was written to threshold entire stacks of denoised slices.

### 3.4.5 Data Filtering and Quantification

Data filtering and quantification was conducted using the Avizo Fire 7.0 software package of Visualization Sciences Group. The thresholded void data was quantified to provide measures of 3-D position within the volume, and measures of individual void volumes. A minimum volume criterion of 43 μm³ was applied to the void data, based upon the 1.15 μm resolution of the XμCT scanner. This criterion was used so that, to be registered as a void, a set of voxels had to be statistically significant in three dimensions, i.e. a void had to have an average dimension three times the size of one voxel to be registered as a void. This voxel-based minimum volume criterion has been used by Toda et al [74] to filter noise from XμCT data.

### 3.5 Metallographic Damage Analysis

In addition to the tomographic damage analysis, damage was analyzed via metallographic methods. Optical microscopy of polished and etched samples was used to visually verify and characterize damage.
3.5.1 Sample Preparation

Two sections were cut from samples deformed at all stages of loading. One section was cut to expose the tensile centerline and one to expose the notch centerline, using the Struers Accutom-5 Automatic precision cut-off machine. Metallographic samples were prepared in both an etched and non-etched condition. Polishing and etching were done via the methods of Section 3.2.2.

3.5.2 Optical Microscopy of Damage

An exploratory optical microscopy procedure was undertaken to find damage in deformed samples. Damage was first investigated in polished but unetched samples so that voids and cracks would not blend in with microstructural features. Diamond indentation was used to mark reference locations on sample surfaces. Etched samples were reinvestigated using reference markers to locate damaged areas. Damage in etched samples could be correlated to microstructural features and compared between the steel sheet variants.

3.6 Fractography

Fractured samples from each variant were examined under SEM using a JEOL JSM-840 Scanning Electron Microscope at 20 kV and various magnifications. The fracture surface was cut from the failed sample and clamped in the SEM sample mount. Features of fracture surfaces were related to the expected mechanisms of failure in ductile versus brittle fracture. Characterization of the type of fracture was used to support the findings of metallographic and XμCT analysis.
Chapter 4

Results

This chapter contains the results of the experiments outlined in Chapter 3. The microstructures of the received materials are characterized. The strain data from the uniaxial testing of double-notch tensile tests and void damage data obtained from X\textsubscript{\mu}CT scanning are presented. A visual inspection of fracture surfaces of failed samples is also shown.

The V1/V2 pairing suggested a more promising opportunity to identify differences in void damage evolution due to its greater difference in hole-expansion properties compared to the V3/V4 pair. Large-scale microstructural variations found in V3 would also confound damage evolution measurements in small-scale samples. For this reason, limited testing was carried out on the V3/V4 pair. To validate the decision to limit testing, the V3/V4 pair microstructure was characterized, and a single point of void damage data was collected for this pair.

4.1 Microstructural Characterizations

Microstructural images presented here show phase distribution and morphology, relative grain size, MA volume fraction, and variations through the sheet thickness. Microstructural pairs are presented based on the variant pairs described in Table 3.2, which were grouped according to sheet thickness. Microstructural constituents are described using the following abbreviations: PF = polygonal ferrite, EF = elongated ferrite, GB = granular bainite, CV = conventional bainite, MA = martensite/retained austenite.

Comparing the V1/V2 to the V3/V4 pair shows similar inter-pair differences in grain morphology and distribution through the longitudinal cross-sections. For this reason, it is expected that correlations of microstructure with void damage in the V3/V4 pair would lead to
similar conclusions as the V1/V2 pair. This observation supports the exclusion of the V3/V4 pair from complete testing.

4.1.1 Qualities of Variant 1 and 2

The microstructures of V1 and V2 have many differences as a result of their 2-step and 1-step respective cooling paths. Figure 4.1 shows the micrographs taken from the sheet centerline of V1 and V2. The LePera etch used tints ferrite grains various shades of blue, as well as tan, depending on their orientation. Bainite grains are tinted a dark brown. MA is highlighted as white/yellow. Figure 4.3 shows the sheet centerline microstructures of V1 and V2 etched with sodium metabisulfite to delineate grain boundaries.

The microstructure of V1 shows a fine, uniformly distributed microstructure of ferrite, granular bainite, and MA. A sheet centerline band can be clearly seen in the transverse section image of Figure 4.4. This band contains a high concentration of bainite and MA relative to the rest of the microstructure. The symmetry of this microstructural feature about the sheet centerline and its small size suggests that it will not confound the correlation of void damage with microstructure.

A greater degree of alignment in the rolling direction can be seen in the microstructure of V2. Bands of large grains with their aspect ratio elongated in the rolling direction (“elongated ferrite”) are separated by bands of small grains, some of which are also elongated in the rolling direction. V2 shows a predominantly ferrite microstructure, with ferrite grains of varying size. Fewer MA grains are visible, and little-to-no dark brown bainite is visible.

For both variants, a trend of decreasing grain size is observed qualitatively with distance from the sheet centerline. The grain distribution and morphology trends appear to remain constant through the sheet thickness, despite the reduction in grain size as can be observed in Figure 4.2.
Figure 4.1: Microstructures of the sheet centerline. LePera etch showing ferrite in blue/tan, bainite in dark brown, and MA in white/yellow.

Figure 4.2: Microstructures of the sheet quarter point. LePera etch showing ferrite in blue/tan, bainite in dark brown, and MA in white/yellow.
Figure 4.3: Microstructures of the sheet centerline. Sodium metabisulfite etch showing ferrite/bainite uncolored and MA in brown/black.

Figure 4.4: Transverse section image of V1 showing sheet centerline band of martensite and bainite. LePera etchant showing ferrite in tan/blue, bainite in dark brown, and MA in white/yellow.
4.1.2 Qualities of Variant 3 and 4

The V3/V4 variant pair exhibits fewer differences in the types of phases present than the previously discussed V1/V2 pair, as they are both subjected to similar 2-step cooling paths. Figure 4.5 shows the sheet centerline micrographs for both variants, where the LePera etch has tinted ferrite and bainite various shades of blue, and MA as white/yellow. Note that the LePera tint colours presented differently in the V3/V4 pair.

V3 shows a wide range of ferrite grain sizes, distributed in patches throughout the microstructure. The grains appear equiaxed, with little preferential elongation in the rolling direction. There is some elongation of patches of similarly sized grains in the rolling direction. Few MA grains are evident. Regions of granular and conventional bainite are mixed in with the ferrite grains.

V4 shows a ferrite dominated grain structure as well, with a wide range of grain sizes. In V4 there is a high degree of preferential banding of grains in the rolling direction. Individual large grains also show aspect ratios that are elongated in the rolling direction. Layers of large and small grains alternate, with a greater amount of conventional bainite.

At the sheet quarter point for V3 and V4, both qualitatively show a slight reduction in grain size, as well as a reduction in the banding. Qualities of the sheet quarter point can be seen in Figure 4.6.

As well as trends through the sheet thickness, V4 also shows trends across the plane of the sheet. Large patches of similarly sized grains can be seen in Figure 4.7. This is in contrast to V3, which shows even distribution of phases and grain size through the plane. Such patches, being on the order of 100μm, could potentially cause concentrations of void damage that could interfere with the uniformity of void damage between the notches.
**Figure 4.5**: Microstructures of the sheet centerline. LePera etch showing ferrite and bainite in blue and MA in white/yellow.

**Figure 4.6**: Microstructures of the sheet quarter point. LePera etch showing ferrite and bainite in blue and MA in white/yellow.
4.1.3 Martensite/Retained-Austenite

Klemm’s etchant was used to reveal martensite and retained austenite in stark contrast to the background microstructure. Using Klemm’s etchant, MA shows up as bright white, while ferrite and bainite grains are tinted blue and grey depending on the grain orientation. MA measurements were taken at the sheet centerline as well as the sheet quarter point of 13 micrographs per sample. Examples of micrographs used to measure MA volume fraction can be seen in Figure 4.8. Measures of MA volume fraction, spacing, and particle size are presented in Table 4.1.
Figure 4.8: Sheet centerline micrographs showing highlighted MA. Klemm’s etchant highlights MA bright white.

Table 4.1: Martensite/retained austenite volume fraction, distribution, and particle size for all four variants at the sheet centerline (Mid) and the sheet quarterpoint (Quarter). Standard deviation is provided in parentheses.

<table>
<thead>
<tr>
<th></th>
<th>V1</th>
<th>V2</th>
<th>V3</th>
<th>V4</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Volume Fraction %</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mid</td>
<td>2.5(0.1)</td>
<td>0.06(0.01)</td>
<td>1.5(0.1)</td>
<td>2.6(0.04)</td>
</tr>
<tr>
<td>Quarter</td>
<td>2.1(0.1)</td>
<td>0.03(0.02)</td>
<td>1.0(0)</td>
<td>2.00(0.24)</td>
</tr>
<tr>
<td><strong>Mean Particle Spacing (μm)</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mid</td>
<td>3.28(0.07)</td>
<td>7.47(2.46)</td>
<td>3.70(0.07)</td>
<td>3.03(0.06)</td>
</tr>
<tr>
<td>Quarter</td>
<td>3.47(0.11)</td>
<td>15.22(9.25)</td>
<td>3.70(0.22)</td>
<td>3.76(0.27)</td>
</tr>
<tr>
<td><strong>Mean Particle Size (μm)</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mid</td>
<td>1.18(0.01)</td>
<td>0.25(0.06)</td>
<td>1.04(0.06)</td>
<td>1.20(0.04)</td>
</tr>
<tr>
<td>Quarter</td>
<td>1.17(0.003)</td>
<td>0.31(0.09)</td>
<td>1.14(0.04)</td>
<td>1.20(0.03)</td>
</tr>
</tbody>
</table>
MA particle size and particle spacing trends were also measured. Figure 4.9 shows the trends exhibited by the MA particles for each microstructure. The MA volume fraction shows the opposite trend when comparing the V1/V2 and V3/V4 pair. In all three measures V2 is an outlier, in that MA volume fraction and mean particle equivalent diameter are lower, and mean particle spacing is much higher. This corresponds to the relative absence of MA particles observed in Figure 4.8 (b) and the relatively small size of those that are visible.
Figure 4.9: Martensite/retained austenite data per variant showing a) volume fraction, b) mean particle size, and c) mean particle spacing.
4.2 Double-notch Uniaxial Tensile Testing

The V1/V2 pair was tested via the procedure described in Section 3.3.2. Testing for the V3/V4 pair was only done at a 10% load drop to provide a comparison of near-failure void damage states.

In both V1 and V2 there was a much greater increase in strain from 7.5% to 10% load drop than between the other stages of load drop. Subsequent testing to 9% load drop was performed for V1 to fill in the data gap. It was not deemed essential to acquire additional data for V2 because of internal cracking already present at lower load drops; little void data would be collected from additional tensile testing in V2 because crack development represents a destruction of void data and distracts from void analysis.

Sub-volumes with the most uniform strain were selected from each sample, as outlined in Section 3.3. The nominal major strain in the selected sub-volumes are summarized in Table 4.2. Appendix A provides values of mean major strain in the most uniform quadrant of all three tests performed at each load drop for each variant. The trend of strain development can be seen in Figure 4.10. Strain tends to quickly develop in the central notch region only immediately prior to failure.

<table>
<thead>
<tr>
<th>Load Drop (%)</th>
<th>Nominal Major Strain in Sub-volume (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>V1</td>
</tr>
<tr>
<td>10.0</td>
<td>17.5</td>
</tr>
<tr>
<td>9.0</td>
<td>11.1</td>
</tr>
<tr>
<td>7.5</td>
<td>8.8</td>
</tr>
<tr>
<td>5.0</td>
<td>6.0</td>
</tr>
<tr>
<td>2.5</td>
<td>5.4</td>
</tr>
</tbody>
</table>
Figure 4.10: Nominal strain in selected sub-volumes for double-notch tensile samples of the V1/V2 pair.

4.3 Damage Evolution

A measure of void volume fraction was the target metric for determining how damage evolved with increasing strain in the notch region of tensile samples. Additionally, the number of voids and the size of those voids in each sub-volume were used to characterize damage evolution. Void data is presented as determined from the analysis of XμCT data using 3-D imaging software.

4.3.1 Variants 1 and 2

A summary of void damage measurements for the V1/V2 pair is provided in Table 4.3. Trends in both the void volume fraction and void size are evident.
4.3.1.1 Void Volume Fraction

Void damage measurements from the V1/V2 pair in Figure 4.11 show that void damage, along with strain, develops quickly towards failure. The large amounts of void damage at the highest level of strain represent two different phenomena in the cases of V1 and V2. Figure 4.11 (a) represents a development of void volume fraction both in terms of number of voids, as well as size of voids in V1. This mechanism of void damage evolution is corroborated by the 3-D void rendering of voids from the XµCT data. In V2 a sizeable crack was present at all levels of strain, but this large crack was removed from the void analysis via a void volume criterion. Around the large crack there were also many micro-cracks that could not be removed without also removing actual void data. Consequently the highest void volume fraction data point for V2 includes a significant degree of micro-crack data, such that it does not purely represent void evolution.

Table 4.3: Void data obtained for V1/V2 pair obtained from XµCT data.

<table>
<thead>
<tr>
<th>Variant</th>
<th>1</th>
<th>2</th>
<th>1</th>
<th>2</th>
<th>1</th>
<th>2</th>
<th>1</th>
<th>2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean Major Strain (%)</td>
<td>5.4</td>
<td>4.5</td>
<td>6.0</td>
<td>7.1</td>
<td>8.8</td>
<td>8.8</td>
<td>11.1</td>
<td>17.5</td>
</tr>
<tr>
<td>Void Volume Fraction (10⁻⁶)</td>
<td>57</td>
<td>7</td>
<td>28</td>
<td>40</td>
<td>64</td>
<td>5</td>
<td>112</td>
<td>462</td>
</tr>
<tr>
<td>Number of Voids</td>
<td>191</td>
<td>11</td>
<td>77</td>
<td>21</td>
<td>118</td>
<td>23</td>
<td>92</td>
<td>2983</td>
</tr>
<tr>
<td>MaxVoid Volume (μm³)</td>
<td>2039</td>
<td>2001</td>
<td>2252</td>
<td>842</td>
<td>6545</td>
<td>1241</td>
<td>2947</td>
<td>10167</td>
</tr>
<tr>
<td>Mean Void Volume (μm³)</td>
<td>230</td>
<td>458</td>
<td>305</td>
<td>260</td>
<td>462</td>
<td>162</td>
<td>399</td>
<td>148</td>
</tr>
<tr>
<td>Max Void Equivalent Diameter (μm)</td>
<td>16</td>
<td>16</td>
<td>16</td>
<td>12</td>
<td>23</td>
<td>13</td>
<td>18</td>
<td>27</td>
</tr>
<tr>
<td>Mean Void Equivalent Diameter (μm)</td>
<td>7</td>
<td>8</td>
<td>7</td>
<td>7</td>
<td>8</td>
<td>6</td>
<td>8</td>
<td>6</td>
</tr>
</tbody>
</table>

4.3.1.1 Void Volume Fraction

Void damage measurements from the V1/V2 pair in Figure 4.11 show that void damage, along with strain, develops quickly towards failure. The large amounts of void damage at the highest level of strain represent two different phenomena in the cases of V1 and V2. Figure 4.11 (a) represents a development of void volume fraction both in terms of number of voids, as well as size of voids in V1. This mechanism of void damage evolution is corroborated by the 3-D void rendering of voids from the XµCT data. In V2 a sizeable crack was present at all levels of strain, but this large crack was removed from the void analysis via a void volume criterion. Around the large crack there were also many micro-cracks that could not be removed without also removing actual void data. Consequently the highest void volume fraction data point for V2 includes a significant degree of micro-crack data, such that it does not purely represent void evolution.
The difference in the type of void evolution can be seen in Figure 4.12. The low-strain samples show that void volume fraction develops at lower strains in V1 than in V2.

**Figure 4.11:** Void volume fraction at nominal strain in selected sub-volumes for double-notch tensile samples of the V1/V2 pair.

**Figure 4.12:** Cluster of void volume fraction data for low nominal major strains in selected sub-volumes for double-notch tensile samples of the V1/V2 pair.
4.3.1.2 Void Size and Count

The void damage in the two variants develops differently because of the early and substantial crack development in V2. The development of voids in V1 is characterized by the nucleation/growth of a greater number of voids than V2. The highest strain sample of V1 shows a drastic increase in number of voids as well as a decrease in mean void size (Table 4.3), which indicates that the nucleation and growth of a large number of small voids overshadows the growth of existing voids. The trend in V2 shows that the number of voids increases, but not nearly as much as V1, as a result of the nucleation of the micro-cracks previously described in Section 4.3.1.1. The relative increase in void count is evident in Figure 4.13.

![Graph showing the number of voids measured in V1/V2 sub-volumes.](image)

**Figure 4.13**: Graph showing the number of voids measured in V1/V2 sub-volumes.

The distribution of void equivalent diameters shows that while voids grow, there is a primary tendency for the appearance of small voids with increasing strain. The distribution of void sizes with developing strain can be seen in Figure 4.14 and Figure 4.15.
Figure 4.14: Distribution of void diameters for V1 samples at each value of mean major strain. Line in d) representing a count of 60 is included for easy comparison with the maximum value on charts a) through c).
Figure 4.15: Distributions of void diameters for V2 samples at each value of mean major strain. Line in d) representing a count of 60 is included for easy comparison with the maximum value on charts a) through c).
4.3.1.3 3-D Void Renderings

The observations of Sections 4.3.1.1 and 4.3.1.2 are corroborated by the 3-D volume renderings of voids, as captured by the XµCT scans. Figure 4.16 - 4.20 show the comparison of void damage at each strain level in the selected sub-volumes of V1 and V2. The tensile axis in the 3-D images is oriented vertically, which corresponds to the transverse direction as per Section 3.3.2. The axis perpendicular to the tensile axis is the rolling direction. The greater number of voids, with smaller mean equivalent diameters, is evident in the 3-D void renderings of V1 when compared with V2. Whereas there was a crack captured in the XµCT scans of all V2 samples, this crack was removed from the rendered images to facilitate visualization of qualitative void damage trends.
Figure 4.16: 3-D void renderings (red) of the sub-volumes after a 2.5% drop from peak load: a) V1 and b) V2
Figure 4.17: 3-D void renderings (red) of the sub-volumes after a 5% drop from peak load: a) V1 and b) V2
Figure 4.18: 3-D void renderings (red) of the sub-volumes after a 7.5% load drop from peak load: a)V1 and b)V2
Figure 4.19: 3-D void renderings (red) of the sub-volumes after a 9% load drop from peak load.
Figure 4.20: 3-D void renderings (red) of the sub-volumes after a 10% load drop from peak load: a) V1 and b) V2.
4.3.2 Variants 3 and 4

Void damage data in V3 versus V4 showed a similar relationship as the V1/V2 pair. Data was captured for the V3/V4 pair at only one strain level, and is summarized in Table 4.4. As in V2, V4 also exhibited cracking, but less micro-cracking around the large crack allowed for easier removal of the macro- and micro-crack data from the void volume analysis. Figure 4.21 shows that V3, which has superior hole-expansion performance to V4, contained a much larger number of voids, and that those voids had a lower mean equivalent diameter. The tensile axis in the 3-D images is oriented vertically, which corresponds to the transvers direction as per Section 3.3.2. The axis perpendicular to the tensile axis is the rolling direction.

<table>
<thead>
<tr>
<th></th>
<th>V3</th>
<th>V4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean Major Strain (%)</td>
<td>18</td>
<td>14</td>
</tr>
<tr>
<td>Void Volume Fraction (10^-6)</td>
<td>1490</td>
<td>1031</td>
</tr>
<tr>
<td>Number of Voids</td>
<td>9052</td>
<td>802</td>
</tr>
<tr>
<td>Max Void Volume (μm^3)</td>
<td>6824</td>
<td>21875</td>
</tr>
<tr>
<td>Mean Void Volume (μm^3)</td>
<td>141</td>
<td>729</td>
</tr>
<tr>
<td>Max Void Equivalent Diameter (μm)</td>
<td>24</td>
<td>35</td>
</tr>
<tr>
<td>Mean Void Equivalent Diameter (μm)</td>
<td>6</td>
<td>10</td>
</tr>
</tbody>
</table>
Figure 4.21: 3-D void renderings (red) of the sub-volumes after a 10% drop from peak load: a) V3 and b)V4.
4.4 Metallographic Damage Analysis

4.4.1 Titanium Nitride Damage

Square or rectangular titanium nitride particles with a longest dimension of approximately 2-10μm were present in all variants, and their contribution to damage was examined for the V1/V2 pair. Optical micrographs were taken at each level of strain for each variant, and the state of titanium nitrides was captured. The damage associated with titanium nitride particles in the V1/V2 pair is illustrated in Figure 4.22. The undamaged particles can be seen in the micrographs of undeformed samples. In samples strained in the intermediate range, cracking of the particle has occurred, which nucleates a void. Some titanium nitrides did not crack, even after failure, but this was not the typically observed behaviour. There was not an observed correlation between magnitude of strain and the sizes of the voids nucleated at titanium nitride particles at intermediate stages of deformation. Upon failure, both variants show growth of titanium nitride-nucleated voids along with the deformation of surrounding microstructure. In both the ductile and brittle failure cases of V1 and V2 respectively, failure was not observed to be preferentially associated with the relatively large titanium nitride voids, as was evident from the location of voids adjacent to, but isolated from, failure surfaces.
Figure 4.22: Micrographs showing typical damage associated with titanium nitride particles for a-d) V1 with increasing strain to failure, and e-h) V2 with increasing strain to failure. LePera etch.
4.4.2 Crack Formation and Growth

V2 exhibited failure by the formation and growth of internal planar cracks. The cracks were observed forming soon after necking, and were evident at all levels of strain thereafter. Crack growth was positively associated with increased strain. Cracks were observed nucleating at multiple locations through the thickness of the V2 samples, although certain cracks tended to preferentially grow towards failure. By examination of the cracks on both the longitudinal and the transverse planes of deformed samples, the cracks were identified as planar. It can be seen in the micrographs of Figure 4.23 that the dominant mechanism of cracking is intergranular. Intergranular cracking follows grain boundaries to produce the type of jagged cracks seen in V2. Most crack propagation occurs along the large rolling-direction elongated ferrite grains, as per Figure 4.23 (c – f), but some cracking can be seen along bands of smaller grains as is evident in Figure 4.23 (a) and (b). Some instances of intergranular cracking such as those of Figure 4.23 (a) and (d) show some sacrificial grains or pieces of grains that have been broken, but most crack growth occurs by exclusively intergranular propagation.
Figure 4.23: Micrographs showing the development of internal, planar intergranular cracks in variant 2 at a) and b) 5.0% mean major strain, c) and d) 7.6% mean major strain, and e) and f) failed. No etch, some relief produced by polishing solution.
4.5 Fractography

Fracture for V1 and V2 samples were examined using optical and scanning electron microscopy. Optical microscopy was used to examine the profile of failed samples, while SEM was used to examine the three-dimensional failure surfaces. Ductile fracture was evident in V1, while brittle fracture was exhibited by V2.

4.5.1 Optical Micrographs

Fracture samples were cut to expose the profile of the through-thickness fracture surface. The micrographs for V1 and V2 fracture profiles can be seen in Figure 4.24. V1 demonstrates a relatively ductile behaviour, with very little sub-surface cracking, and a smooth fracture profile compared to V2. One main area of damage nucleated in V1 at the sheet centerline, via ductile splitting of the sample to form a heavily deformed macro-crack around the sheet centerline band of bainite and MA. Greater amounts of grain deformation around the fracture surface are visible in the V1 micrograph of Figure 4.24 (b) relative to the deformation in the V2 micrograph of Figure 4.24 (e), which also demonstrates ductile versus brittle fracture in V1 versus V2 respectively. The micrographs of V2 show that cracking initiates at multiple points as planar cracks (i.e. splits) in the rolling plane, although two deep cracks appear to dominate damage development.
Figure 4.24: Micrographs showing the profile of fracture surfaces for a-c) V1, and d-f) V2. LePera etch.
4.5.2 SEM Fractography

Fracture surfaces were examined using SEM to visualize the topography of the fracture surface of V1 versus V2. The images acquired, showing the nature of fracture in each variant, are depicted in Figure 4.25 and Figure 4.26. The fracture surface of V1 exhibits typical ductile fracture dimples. Although V1 contains one central void/crack, the fracture surface surrounding this area demonstrates purely ductile fracture. Figure 4.26 (a) shows that the fracture in V2 occurs by evolution of the observed intergranular cracks into large rolling plane splits. V2 shows evidence of large voids, but little dimpled fracture. Smooth fracture surfaces dominate the failure of V2, where veins can be seen running along the smooth fracture plane indicating the propagation of cracks. These characteristics of the V2 fracture surface are evidence that brittle fracture is occurring.
Figure 4.25: SEM images captured showing the fracture surface of V1, with a) half-section of fracture surface, b) close-up of central macro-void, c) and d) dimpled fracture surface.
Figure 4.26: SEM images captured showing the fracture surface of V2, with a) half-section of fracture surface, b) close-up of central crack, c) and d) smooth fracture surface.
Chapter 5

Discussion

5.1 Microstructural Evolution

Microstructural variants were provided for this study from industrial steel coils. Differences in processing were artifacts of either industrial practices or controlled variation of processing parameters. Based upon observations of microstructure, and prior knowledge of microstructural evolution in similar steels, an explanation of microstructural features can be made.

5.1.1 Variant 1 and 2

There are significant differences in the processing of the V1/V2 pair, all of which may have contributed to their microstructural differences. The relative location of each variant on the coil (i.e. leading versus trailing edge), and the characteristics of their controlled cooling are the biggest differences between the two variants. Additionally, small changes in roughing and finish rolling temperature, times, and deformation could account for some microstructural differences.

V2 experiences a greater amount of time in the austenite region between roughing and finish rolling. Because the V2 is the trailing edge of the sheet, it waits in-coil at elevated temperature while the leading edge proceeds through finish rolling. The final passes of roughing occur in the partial recrystallization region, which would form a mixed recrystallized and non-recrystallized microstructure [32]. The time spent at elevated temperature would allow for austenite grain growth of unrecrystallized grains, and subsequent pancaking of these large austenite grains into large elongated austenite grains. The austenite grain growth experienced by the trailing edge of the coil could partially explain the greater degree of banding and elongated grains associated with V2 and V4, especially giving the ability of Ti and Nb to retard recrystallization during finish
rolling [19], and by the persistence of mixed microstructures through preferentially iterative recrystallization of fine-grained regions but not large-grain regions [31].

V1 and V2 also experience differences in controlled cooling, namely 2-step and 1-step cooling, respectively. V1 and V2 are held in the ferrite region after the first step of cooling for 6s and 3s respectively, but V1 is also held in the bainite region for 4s after a second cooling step. The two-step cooling of V1 explains the fine ferrite and fine bainite microstructure produced. Fine polygonal ferrite grains and granular bainite are produced during the first hold temperature. The V1 bainite hold produces the conventional bainite grains between areas of existing ferrite. Some MA is also a result of high-carbon areas cooling during the accelerated second cooling step. V2 develops even finer-grained ferrite during the cooling from austenite with little to no bainite, but is then allowed to cool in the coil. In-coil cooling at high temperature would allow for the growth of large ferrite grains between existing bands of finer ferrite.

Carbon segregation in the microstructures of the V1/V2 pair is a result of a difference in their cooling schedules. Whereas carbon in V1 is contained within bainitic cementite, martensite, and retained austenite, there are no such phases to act as sinks for carbon in the microstructures of V2. Grain boundaries act as a possible sink for carbon in V2. Carbon may also be located in solution in large ferrite grains, providing strengthening. It is possible that coiling at high temperature has precipitated a much larger volume fraction of nano-scale carbides than in V1. The micrographs of this study could not resolve such small features, which would explain their apparent absence in the images of Section 4.1. Both microstructures exhibit small carbides located uniformly throughout the microstructure that account for some of the carbon.

A secondary influence on microstructural development is the differences in finish rolling conditions between the variants. In the final two passes of finish rolling for V2, the strain is about 14% higher than for V1. The pancaking of austenite grains immediately before cooling,
particularly of the mixed-microstructure austenite grains presumed to have developed in V2, could promote polygonal ferrite, according to the CCT curves in Figure 2.5.

5.1.2 Variant 3 and 4

The differences in the V3/V4 pair are less distinct than the V1/V2 pair. These microstructures likely derive their differences mostly from the slight differences in their controlled cooling, and the greater amount of time V4 sits at high temperature between roughing and finishing. The phases present in both microstructures are similar. The degree of banding in V4 is greater and more continuous than V3.

As with V2, it is thought that a main difference and potential driver for the formation of banding is the austenite hold between roughing and finishing. Particularly in the case of the V3/V4 pair, there is no significant difference in finishing, and both variants are subject to a two-step controlled cooling. Therefore the main difference thought to be responsible for the severity of banding and elongated ferrite grains in V4, is the long austenite hold.

Both of the hold temperatures during cooling (and by extension, the coiling temperature) are lower for the V4 microstructure, which changes the morphology of the transformed bainite and MA products. While both microstructures contain polygonal ferrite at similar grain size, the bainite morphology differs. V3 has mostly granular bainite, whereas V4 contains some granular bainite but a much greater amount of conventional bainite than V3. The conventional bainite was arranged into long bands as indicated in Section 4.1.2. The lower temperature hold would cause the nucleation of lath-type conventional bainite over the granular type seen at high temperatures. The lower temperature bainite hold also produces a greater amount of MA in the V4 microstructure.
There is potential for a secondary effect of a higher degree of austenite deformation in the low-temperature finishing stages. The same mechanisms proposed for V2 are thought to be at work here, i.e. the promotion of polygonal ferrite via the pancaking of the prior mixed-microstructure austenite.

5.2 Double-notch Uniaxial Tensile Testing

The purpose of double-notch uniaxial testing was to produce damage in a very localized region, but also to promote uniformly distributed strains in the central part of this region. The dimensions chosen for these samples proved to be adequate at achieving this goal.

5.2.1 Strain Distribution

In the region of interest, at the center of the notch region, the standard deviation of the major strain was kept very low, below 1%, with only one exception where the deviation reached 2.3%, and strains within sub-regions was kept below 3%. This meant that damage data for extracted regions was representative of a narrow range of major strain, such that damage data could be reasonably correlated with singular strain levels.

The highest strains were located away from the notch centerline in the major axis, such that relatively low major strains were located in the region of interest. This represents a constraining effect of the notch region with respect to major strain. It is usually reported that individual voids will grow in the direction of tensile stress [45], but little elongation of voids was exhibited in the voids of this investigation. The macro stress triaxiality imposed by the notch is thought to promote pure dilation of voids in three dimensions as opposed to elongation [75].
5.2.2 Geometric Considerations

Although the strain distribution in the notch region was deemed satisfactory, and damage appeared to occur symmetrically, there were some geometry inconsistencies in the notched samples. Because of machining difficulties, there was an average asymmetry of 8% in the notch depth, and 21% in root radius. With greater symmetry in notches, the strain distribution could have been even more uniform.

5.3 Damage Characteristics

5.3.1 Void Damage

Void damage trends were consistent with hole-expansion behaviour in this study. Variants with good hole-expansion (V1 and V3) developed a large number of very small voids. The development of many small voids tended to be uniform within the imaged volume as per Figure 4.16 - Figure 4.21. This type of void damage behaviour represents little risk for failure. Small voids are evidence of void nucleation and growth; however, coalescence is not promoted in these microstructures. Additionally, large amounts of small void damage are indicative of plasticity and energy absorption, contributing to the elongation of the necked material.

In variants with poor hole-expansion (V2 and V4), the number of voids remained relatively low until high strains. A large internal crack formed in both of these variants, which deterred the formation of voids. Energy absorption by the propagating crack results in lower stress in the surrounding microstructure, such that void nucleation and growth is suppressed. Some evidence of void nucleation, such as that at TiN particles, was seen at very low strains. These voids are allowed to nucleate because the large internal crack has not yet formed. For these reasons, the variants with poor hole-expansion exhibit relatively fewer voids, but the voids that do nucleate
tend to be large voids that nucleate and grow at locations of relatively large stress concentrations such as TiN particles.

At high strains, there was significant appearance of small voids (i.e. those that meet the minimum volume criterion) in both the of V1 and V2 microstructures. Exponential void nucleation is expected because when the stress is high enough, there will be decohesion and particle cracking ubiquitously throughout the microstructure [46,60]. This behaviour is seen in V2 despite the presence of intergranular cracking, because immediately before fracture the stress in ligaments of material between the planar cracks increases dramatically.

There was little evidence of void sheeting or coalescence into continuous macro-voids in the materials studied. This behaviour is likely because there was a uniform nucleation of voids in these materials. Voids were not associated with banded or elongated features of these microstructures, so there was less potential for linking of voids in coalescence events prior to failure.

5.3.2 Intergranular Cracking

Cracks formed as internal decohesions in the plane of the sheet during tensile testing. The planar nature of cracking (i.e. splitting) suggests that the microstructural banding contributes to damage in both the transverse and rolling directions. Cracks began by splitting at the centerpoint of the notch and the sheet centerline, and grew towards the notches and also grew in the tensile direction. Because the failure of the crack presented as a planar split, it seems a through-thickness constraint of the crack that provides stability until the point of failure.

The intergranular nature of the crack is confirmed by three observations. It is seen that cracks runs along grain boundaries. The crack follows a jagged path as it does so, propagating around
impeding grains. Additionally, the crack produces little debris, indicating that grain boundary decohesion has occurred, as opposed to transgranular fracture and break-up of grains.

Although the crack was mostly intergranular, there was some cracking of individual grains, as observed in Section 4.4.2. As the crack propagated, it likely encountered grains that were in the direct path of the propagation. As the crack bridges grains there is potential for cracking of weak grains, which is the phenomenon observed in this case.

The crack was mostly observed to propagate along bands of large elongated ferrite grains, but there were some cracks that propagated through regions of fine ferrite grains as well. The crack will propagate through the grain boundaries that are weakest, and because of local variations in grain boundary strength, bands of fine ferrite grains will sometimes represent the lowest energy path.

5.3.3 Fracture Surface

The fracture surface of V1 shows typical ductile fracture characteristics. The surface shows many dimples between which ligaments of material necked and failed. The uniform dimples are representative of void growth to the point of void-void impingement. At the point of void-void impingement, the remaining material necks and factures, leaving behind the dimple as an artifact of a half inner void surface.

V2 has a mixed failure surface comprised of areas of ductility and areas of brittle fracture. The regions of brittle fracture correspond to the internal planar cracks noted previously. On the surface of these cracks, there is evidence of void damage, but these voids are independent of the failure event and are too sparse to develop into a ductile fracture surface. In areas between the brittle fracture surfaces, ductile damage is evident. Sections of material that exist between planar
cracks carry the entire tensile load. Hence, these areas end up failing in a ductile manner at the moment of fracture, as indicated by their dimpled surface.

5.4 Microstructural Contribution to Damage

The first voids to nucleate are large and very sparsely distributed. These voids are associated with TiN particle cracking or decohesion of the TiN from the surrounding microstructure. Because of the size of these particles, they cause relatively large stress concentrations, which are the first to nucleate voids. These particles and their associated voids have been observed at every stage of applied deformation, and do not preferentially attract or promote other void damage or cracking. Likely because these particles are so sparsely located, they appear to be insignificant contributors to damage, and provide little energy absorption.

The MA distribution in V1 can be used to explain its void damage characteristics. There are a large number of evenly distributed voids in V1, and there are also a large number of small MA particles. Void nucleation at these particles represents the classic mechanism of void nucleation in dual-phase steels. The growth of voids initiated by hard phases is proportional to the size of the hard phase, because the size is proportional to the stress triaxiality imposed by the particle at its interface with the surrounding matrix. Since the MA particles in V1 are small, they tend to nucleate and grow small voids. The large interparticle spacing of MA relative to their particle size prevents void-void interactions (i.e. coalescence) which would lead to premature failure. The void damage behavior of V3 is caused by the same microstructural features.

There was no cracking in the case of V1 failures, because the types of microstructural features necessary to promote cracking were not present. The grains of V1 are equiaxed and evenly distributed comprising both ferrite and bainite grains. There is no continuous path for cracks to
develop, and the grain boundaries in this microstructure likely do not possess the same weakening mechanisms at play that embrittle the grain boundaries of V2.

V2 cracking failures are likely produced by characteristics of its banded structure and elongated grains. Since hole-expansion tests are terminated upon the observation of an edge crack, the splitting cracks characteristic of banded microstructures will be particularly detrimental. Bands of elongated grains provided direct fracture paths for uninhibited cracking to occur. Additionally there is likely some grain boundary weakening characteristic of the elongated ferrite grains in V2. Precipitation of cementite on grain boundaries has been documented for Nb-Ti strengthened steels, which includes the rejection of solute into the affected boundaries [19]. Given the high amount of carbon unaccounted for in optical micrographs of Section 4.1.1, cementite formation on ferrite grain boundaries would help to account for some carbon, and would explain grain boundary weakening via the mechanisms proposed by McMahon [50].

5.5 Relation to Hole-expansion

Previous work has identified banding as the dominant microstructural detriment to hole-expansion performance [6,62], and has shown that intergranular cracks promote the types of failures detected during hole-expansion [11]. This thesis has shown the same relationship between banding, intergranular cracking, and hole-expansion performance. The mechanism proposed herein is grain boundary weakening along bands of large elongated ferrite grains. An additional mechanism associated with poor hole-expansion performance is strength disparity between phases as noted by Cai et al. [9]. A strength disparity is expected between large-grain and fine-grain ferrite bands because of the local strength increase caused by grain refinement in fine-grain ferrite bands. Hence, this disparity could therefore also be contributing to poor hole-expansion via intergranular cracks.
There is also an existing suggestion that bainite promotes good hole expansion [6,62]. Based on the proposed intergranular fracture mechanisms, it is thought that hole-expansion performance increases with bainite in some microstructures because bainite acts as a sink for carbon. Carbon contained in bainitic cementite would not be available to promote weakening of grain boundaries.

The intergranular cracks present in V2 occur by planar splitting, as do types of failures described in other work [11]. Because of the circumferential strains placed on the edge of hole-expansion samples, splitting failures could represent a specific susceptibility to failure. For this reason, banding and intergranular fracture are of particular relevance in hole-expansion testing.
Chapter 6

Conclusions and Recommendations

6.1 Conclusions

- Double-notched uniaxial tensile samples provided a means for studying damage evolution. Uniform major strain was produced in the central notch region, providing a volume from which a sample of nominal major strain could be extracted. Samples tested to increasing levels of load drop from peak load resulted in comparable strains for the microstructural variants involved.

- \(X\mu CT\) images provided a good measure of void damage data, from which void volume fractions, void size, and void count could be extracted. The quantities extracted were compared with other measures of damage and microstructure to fully characterize the evolution of damage.

- In all variants, large TiN particles were shown to nucleate voids at very early stages of deformation. The voids nucleated at these particles tended to be relatively large, but because of their sparse distribution they did not critically contribute to damage or failure.

- The microstructural variants exhibiting good hole-expansion performance developed a large number of very small voids. These voids correlated with MA hard particles. These small voids did not develop into networks of coalesced voids. Failure in these microstructures was by ductile fracture.

- The microstructural variants exhibiting poor hole-expansion developed intergranular planar cracks upon necking. Cracking distracted from the potential for void nucleation in
these microstructures, such that relatively few voids were produced at low levels of strain. Voids that developed did so independent of cracking. Intergranular cracks were associated with large ferrite grains arranged in bands, separated by areas of finer-grained structure. These microstructures failed primarily by brittle fracture with some ductile failure of the ligaments of material between cracks.

6.2 Recommendations

- Further investigation should image a greater number of XμCT samples. More data points could be collected at each level of strain to corroborate void volume fraction data. Samples for a complete set of strain levels for the V3/V4 pair could be imaged to provide more complete evidence that this pair follows the same void evolution as the V1/V2 pair.

- Imaging and investigation of steels tested in typical hole-expansion rigs could provide a more certain understanding of microstructural performance. The exact stress-state and geometry of hole-expansion testing is not replicated with notched tensile testing. Situation-specific modes of failure could be confirmed and correlated with microstructure via hole-expansion test samples.

- More detailed and higher resolution investigation of microstructures would increase the certainty of the grain boundary embrittlement mechanisms proposed in this study. TEM investigation into the structure of each microstructural constituent, as well as the structure and composition of grain boundaries would contribute to a more complete understanding of the fracture events that have been documented.

- Microstructural variants should be produced with greater consideration given to experimental variables. The microstructural variants provided have multiple significant differences in their controlled rolling and cooling schedules, which complicated the
correlation of damage with particular processing parameters. It would be beneficial to design processing schedules that alter one variable at a time to see if it changes hole-expansion behaviour significantly.
References


Appendix A

Double-Notch Uniaxial Tensile Testing Strain Data

At each load drop, three tests were performed for each variant. Around the center-point of the sample, strain grids were divided into four quadrants situated symmetrically about tensile axis and notch axis, as was depicted in Figure 3.8. The sample with the most uniform strain across all quadrants was selected for XμCT imaging. From within the selected sample, the quadrant with the lowest mean major strain was chosen for extraction and imaging. The quadrant with the most uniform strain from each test is reported in Table A.1.

**Table A.1:** Mean major strain measurements from the quadrant with the most uniform strain, for all uniaxial tensile tests performed. Standard deviation is reported in parentheses, and the sample from each set that was selected for XμCT imaging is highlighted.

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<th>V2</th>
<th>V3</th>
<th>V4</th>
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<td>Test 1</td>
<td>Test 2</td>
<td>Test 3</td>
<td>Test 1</td>
</tr>
<tr>
<td>2.5</td>
<td>3.9(2.0)</td>
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<td>5.4(1.3)</td>
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<td>5.0</td>
<td>6.0(1.1)</td>
<td>6.2(1.4)</td>
<td>8.5(1.3)</td>
<td>-</td>
</tr>
<tr>
<td>7.5</td>
<td>8.8(1.8)</td>
<td>9.6(1.6)</td>
<td>8.1(2.1)</td>
<td>-</td>
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<tr>
<td>9.0</td>
<td>13.5(2.4)</td>
<td>11.1(1.2)</td>
<td>12.8(2.3)</td>
<td>-</td>
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<td>10.0</td>
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<td>17.5(2.7)</td>
<td>16.3(2.5)</td>
<td>Sample Broke</td>
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Appendix B

Received Material Thermomechanical Processing History

Tables B.1-B.6 contain detailed information regarding the rolling and cooling history for all four sheet variants from ArcelorMittal Dofasco [63]. Temperature measurements were taken by pyrometer at the locations noted. Other temperatures and cooling rates are interpolated based on modeled results.

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<th>Total time (sec)</th>
<th>Temperature (°C)</th>
<th>Cooling Rate (°C/sec)</th>
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<td></td>
<td>Stand 7</td>
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Table B.2: Controlled rolling parameters for V2. Controlled rolling is followed by controlled cooling [63].

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<th>Cooling Rate (°C/sec)</th>
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<th>Effective Strain</th>
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**Table B.3:** Controlled cooling data for a) V1 and b) V2. Controlled cooling was followed by controlled rolling [63].

(a)

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<th>Cooling Rate (°C/sec)</th>
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(b)

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**Table B.4:** Controlled rolling parameters for V3. Controlled rolling is followed by controlled cooling [63].

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Table B.5: Controlled rolling parameters for V4. Controlled rolling is followed by controlled cooling [63].

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<th>Thickness (mm)</th>
<th>Effective Strain</th>
<th>Effective Strain Rate (sec⁻¹)</th>
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Table B.6: Controlled cooling data for a) V3 and b) V4. Controlled cooling was followed by coiling [63].

(a)

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(b)

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Appendix C

Image Processing Parameters

C.1 Denoising Parameters

The work of Sloan [70] includes a parametric study of variable inputs to the non-local means denoising algorithm of Baudes et al. [71] to determine the best values to use for the denoising of steel image slices containing void damage. The algorithm requires the input of a window size, patch size, and filtering factor, $h$. The window size, $t$, is the side length of the square that defines a window within the image that will be compared to other windows. The patch size, $f$, is the size of a patch within the window that will be sampled and compared to other the same patch locations in other windows. It was determined that a window size of 7 pixels, and a patch size of 1 pixel produces the most accurate results in thresholding of voids. The recommendation of Baudes et al. that the standard deviation of greyscale values in an image be used as the filtering factor was confirmed.

C.2 Thresholding Parameters

Thresholding using the locally adaptive methods of Guanglei Xiong requires the input of a window size in which pixels will be compared, and a constant $C$ [76]. It was determined that a window size of 31 and a $C$ value of 0.065 were optimal to preserve the morphology of voids in thresholded slices.