INFLUENCE OF CRYSTALLOGRAPHIC TEXTURE IN X70 PIPELINE STEELS ON TOUGHNESS ANISOTROPY AND DELAMINATION

by

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ABSTRACT

The effects of microstructure and crystallographic texture in four commercially-produced API X70 pipeline steels and their relation to planar anisotropy of toughness and delamination were evaluated. The experimental steels were processed through either a hot strip mill, a Steckel mill, or a compact strip mill. Different processing routes were selected to obtain plates with potential variations in the microstructure and anisotropic characteristics. Tensile and Charpy impact testing were used to evaluate the mechanical properties in three orientations: longitudinal (L), transverse (T) and diagonal (D) with respect to the rolling direction to evaluate mechanical property anisotropy. The yield and tensile strengths were higher in the T orientation and toughness was lower in the D orientation for all plates. Delamination was observed in some of the ductile fracture surfaces of the impact samples. To further study the splitting behavior and effects on impact toughness, a modified impact test (MCVN) specimen with side grooves was designed to intensify induced stresses parallel to the notch root and thus facilitate evaluation of delamination. Scanning electron microscopy combined with electron backscattered diffraction (EBSD) were used to evaluate the grain size, microstructural constituents, and crystallographic texture to determine the factors leading to delamination and the anisotropy in toughness. The ferrite grain size is mainly responsible for the differences in DBTTs between the L and T orientations. The higher DBTT in the D orientation observed in pipeline steels is attributed to crystallographic texture. The higher DBTT in the D direction is due to the higher volume fraction of grains having their {100} planes parallel or close to the primary fracture plane for the D orientation. An equation based on a new “brittleness parameter,” based on an assessment of grain orientations based on EBSD data, was developed to predict the changes in DBTTs with respect to sample orientation based on grain size and texture. The calculated DBTTs correlated well with
the experimental values. The \{001\}<110> and \{113\}<110> components are the main preferred orientations that cause brittleness in the D direction, since their \{001\} planes make an angle less than 20° with the primary fracture plane of the samples oriented in the D direction. It was also concluded that delamination occurs due to banded bainite regions that were oriented such that their \{001\} planes make a small angle with the rolling plane leading to degradation in crack arrestability. The texture of the banded regions consisted of \{001\}<110>, \{113\}<110> or \{111\}<112> orientations. It was concluded that the \{001\}<110> and \{113\}<110> orientations promote splitting because their fracture strengths in the normal direction are low. The \{111\}<112> orientation has a calculated fracture strength more than twice the \{001\}<110> and \{113\}<110> orientations and therefore banded regions with the \{111\}<112> texture are more susceptible to cleavage fracture perpendicular to the normal direction.
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CHAPTER 1: Introduction

For helical welded large diameter pipe, the hoop direction of the pipe corresponds to an orientation which forms an angle to the strip rolling direction and the angle varies with pipe diameter for pipes produced from a specific coil width. Since anisotropy in mechanical properties has been observed in hot rolled strip and plate products in general, tensile and impact tests conducted transverse to the rolling direction as part of the qualification process at steel mills might not reflect the final mechanical properties in the pipe hoop direction, \( i.e. \) the direction of maximum applied stress in a pressurized pipe. Correspondingly, if strong anisotropy in mechanical properties exists in the steel, the potential may exist for failure due to the applied hoop stresses which were not predicted based on qualification test data. It is important for steelmakers to understand and control steel anisotropy to eliminate any unexpected mechanical response during service. In the absence of high inclusion and stringer content, crystallographic texture is a key metallurgical factor affecting mechanical property anisotropy, and the extent of texture is influenced by chemical composition and thermomechanical processing. Research has been performed to relate toughness anisotropy to texture, yet a fundamental understanding of the relation between microstructure, texture and toughness anisotropy has not been fully addressed. Another unresolved subject in pipeline steels is the splitting or delamination observed during toughness testing in the fracture surfaces of these steels. It is still not clear whether delamination is detrimental to toughness or not.

1.1 Thesis Objective

The primary objectives of this project were to understand the effects of microstructure and crystallographic texture on the mechanical property anisotropy, specifically toughness, and
delamination during fracture for X70 pipeline steels. The fundamental questions to be answered by this work are:

- What is the influence of crystallographic texture on toughness anisotropy in pipeline steels?
- How and why does delamination during fracture affect observed toughness? What is the relationship of the susceptibility to splitting with microstructure and texture?

1.2 Thesis Outline

Chapter 2 reviews the available literature, starting with the microstructures and mechanical properties of pipeline steels. Background information on the crystallographic texture development in hot rolled steels is discussed. Recent research in the literature on toughness anisotropy and delamination is presented.

Chapter 3 outlines the experimental design, highlighting the purpose of this project and the broad approach to achieve the project objectives. Chapter 4 gives the details of the materials, mechanical testing methods, and characterization procedures employed on the investigated steels.

Chapter 5 presents the experimental results obtained from characterization and mechanical testing including the microstructures, crystallographic textures, tensile properties, and impact properties for the investigated steels. Modified impact test data for selected alloys and heat treatment results are also discussed.

Chapter 6 discusses the main results and provides additional interpretation to determine the microstructure and texture effects on mechanical properties, anisotropy, and delamination.

Chapter 7 provides a summary of the main findings discussed in Chapters 5 and 6.

Chapter 8 outlines the potential areas for future work based on the finding from this project.
CHAPTER 2: Literature Review

This chapter provides a critical literature review on the microstructures and mechanical properties of pipeline steels, and fundamental background information on crystallographic texture development in rolled steels. Also, some of the previous research studies conducted on toughness anisotropy and delamination are presented here.

2.1 Pipeline Steels

Recently, due to increased energy consumption, there has been a large demand for pipeline steels used in crude oil and natural gas transportation at high operating pressures and potentially operating in severe environmental conditions. A main objective for the construction of a pipeline network is to use pipes with large diameters under high pressure to increase transportation efficiency and consequently reduce transportation cost [1-3]. Using higher strength steels, such as API grades X70 and X80, allows the use of larger diameter pipelines with a significant reduction in thickness both which result in economical advantages by decreasing the overall weight while increasing flow capacity. In order to meet API grade requirements, steels with a combination of both high strength and high toughness must be developed. In addition to strength and toughness, these steels should have excellent weldability, formability, resistance to hydrogen induced cracking (for sour service environments), resistance to stress corrosion cracking (in sour environments), and fatigue resistance to satisfy API requirements. Excellent mechanical properties are achieved by obtaining optimum microstructural characteristics of the steel as controlled by chemical composition and thermomechanical processing (TMP). Low carbon (C) contents are essential for alloy designs to ensure good weldability and formability. Alloy additions such as molybdenum (Mo) and microalloying elements including niobium (Nb),
vanadium (V), and titanium (Ti) are added to compensate for the decrease in strength due to the low carbon content [4-6].

Figure 2.1 summarizes the development of high strength pipeline steels from 1965 to 2000 [7]. In the 1970’s, to achieve the desired properties of X70 grade steels, hot rolling and normalizing practices used to produce X52 and X60 grade pipeline steels were substituted by thermomechanical rolling of steels with lower C contents and microalloyed with Nb and V. Accelerated cooling was introduced in the 1980’s resulting in the development of even higher strength material (X80 grade) with even lower C contents. Higher strength API grades up to X100 were developed by further alloying with Mo, nickel (Ni), and copper (Cu) [7].

![Diagram of API pipeline steels development](image)

Figure 2.1 Development of API pipeline steels [7].

### 2.1.1 Microstructures of Pipeline Steels

Different microstructure combinations have been observed in pipeline steels produced by thermomechanical controlled processing. Microstructures produced in pipeline steels include, depending on the thermomechanical processing history and alloying elements, a combination of ferrite-pearlite, polygonal ferrite, acicular ferrite, bainite and martensite-austenite (MA) constituents [5, 8]. It has been well accepted in the pipeline manufacturing industry that an
acicular ferrite dominated microstructure with uniform distribution of MA islands, which was first described in the early 1970’s by Smith et al. [9], gives the optimum mechanical properties [4, 5, 8, 10-13].

Collins et al. reviewed the microstructures of pipeline steels and their findings are correlated to the schematic continuous cooling transformation (CCT) diagram for pipeline steels shown in Figure 2.2 [14]. Upon slow cooling (path I in Figure 2.2), austenite transforms to polygonal ferrite at high temperatures near equilibrium conditions. Carbon partitions to the remaining austenite as ferrite grains nucleate and grow. Once the carbon enriched austenite reaches the eutectoid composition, the remaining austenite transforms to pearlite. The final microstructure is composed of equiaxed ferrite and pearlite colonies, as shown in Figure 2.3a. Reducing the carbon content and/or adding carbide forming alloys such as Nb, Ti, and V will shift the nose of the pearlite curve in Figure 2.2 to the right, thereby reducing the fraction of pearlite (Figure 2.3b). Increasing the cooling rate to path III in Figure 2.2, leads to the formation of acicular ferrite at lower temperatures (Figure 2.3c). Acicular ferrite forms by a mixed diffusion and shear mode at relatively low temperatures just above the upper bainite transformation temperature and is characterized as a fine grained non-equiaxed ferrite containing a high dislocation density substructure [10-13, 15]. In order to achieve an acicular ferrite microstructure in pipeline steels, the manganese content is typically increased and/or small amounts of Mo are added to the steel composition to slow the transformation rate and suppress the formation of polygonal ferrite [14]. Upper bainite, shown in Figure 2.3d, might form from the remaining austenite at lower temperatures and is usually a minor component of pipeline steels microstructure [14]. Upper bainite consists of parallel ferrite laths separated by low angle boundaries. Elongated cementite particles and sometimes retained austenite (RA) or MA islands
are distributed along the lath boundaries [14, 16]. However, in pipeline steels, sometimes the lath boundaries are absent of microconstituents due to the low carbon content in the steels compositions [14]. Lower bainite, which differs from upper bainite by the precipitation of cementite inside ferrite laths instead of on lath boundaries, is not generally present in pipeline steels. Rectangular shaped or blocky martensite-austenite constituents are formed finally in the remaining C-enriched austenite trapped between the ferrite and/or bainite grains [14].

Figure 2.2  Schematic continuous cooling transformation (CCT) diagram for pipeline steels [14].

2.1.2 Tensile Properties

The tensile property requirements for X70 linepipe steels as per the API specification [17] are shown in Table 2.1.

Kim et al. studied the effect of microstructure on the strength and toughness of linepipe steels [18]. Their study found that steels with bainitic microstructures have the highest strengths followed by acicular ferrite-polygonal ferrite steels. Steels with polygonal ferrite-pearlite microstructures showed the lowest strengths and could not meet the yield strength and yield to
Figure 2.3 Light optical micrographs showing the different microstructures of pipeline steels produced by the four cooling rates identified in Figure 2.2: (a) polygonal ferrite and pearlite (rate I), (b) polygonal ferrite with reduced pearlite (rate II), (c) acicular ferrite (rate III), and (d) upper bainite (rate IV) [14].

tensile strength ratio for X70 grade steels (485 MPa and 0.93). Also, the bainitic steels exhibited continuous yielding, while the yielding behavior of acicular ferrite-ferrite steels changed from quasi-continuous to continuous as the volume fraction of acicular ferrite increased. Ferrite-
pearlite steels exhibited discontinuous yielding behavior. Reducing the polygonal ferrite grain size increased the yield strength and decreased the work hardening rate, which increased the yield ratio. However, in an acicular ferrite dominated microstructure, the yield strength increased and also the work hardening rate at low strains increased due to the presence of a large density of mobile dislocations in acicular ferrite, therefore decreasing the yield ratio. Bainitic steels exhibited even higher work hardening rates than acicular ferrite, which led to lower yield ratios. Han et al. [19] reported that the increase in secondary hard phases such as MA, increased the yield strength, since MA constituents transformed at low temperatures. The study postulated that increasing the volume fraction of MA promotes mobile dislocations at boundaries between secondary phases and soft phases, leading to higher tensile strengths associated with continuous yielding.

Table 2.1 - Tensile Test Requirements for API-X70 Pipe [17]

<table>
<thead>
<tr>
<th>Yield Strength (MPa)</th>
<th>Tensile Strength (MPa)</th>
<th>Maximum Ratio (YS/TS)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Min</td>
<td>Max</td>
<td>Min</td>
</tr>
<tr>
<td>485</td>
<td>635</td>
<td>570</td>
</tr>
</tbody>
</table>

2.1.3 Impact Properties

For X70 linepipe steels, the average absorbed energy for the Charpy V-notch (CVN) impact test should be more than 40 J based upon full size test specimens at a test temperature of 0 °C, or a lower temperature, if requested by the customer [17]. The upper shelf energy (USE) is influenced by the microstructure type, volume fraction of phases, and grain size, while the transition temperature is mainly affected by the effective grain size [19-21], which acts as a barrier against cleavage crack propagation [20]. In linepipe steels, the transition temperature
should be low enough to avoid sudden brittle fracture and the absorption energy should be high enough to prevent unstable ductile fracture propagation [21]. Most research [18-24] agrees that high angle grain boundaries (HAGB), larger than 15°, deflect cleavage cracks resisting crack growth, while low angle grain boundaries (LAGB), less than 15°, do not effectively hinder crack growth. Therefore, grains separated by HAGBs are generally used to determine effective grain size. Several studies [18-22, 25] used EBSD analyses in order to define the effective grain size for the different microstructures present in higher strength linepipe steels, specifically X70 and X80. Polygonal ferrite grains have misorientation angles of 15° or higher and are treated as effective grain size. In contrast, the effective grain size for bainite or bainitic-ferrite microstructure is the prior austenite grain size, since the boundaries between parallel laths and packets are low angled. Although parallel acicular ferrite plates have LAGBs, most acicular ferrite plates are arranged differently and form HAGBs and the effective grain size for acicular ferrite is much lower than bainite [20, 22, 25]. Han et al. [19, 21] found that increasing the volume fraction of granular bainite over acicular ferrite lowers the USE and increases the transition temperature. Kim et al. [18] also found that acicular ferrite microstructure with small fractions of fine polygonal ferrite grains show low transition temperatures compared to acicular ferrite steels due to the small effective grain size of polygonal ferrite.

2.2 Texture

During steel processing the final crystallographic texture evolves by three means: deformation, recrystallization, and transformation. In cold rolling, where the material is processed in the ferrite phase, the final texture is achieved by deformation and recrystallization after annealing. In hot rolling, transformation of austenite to ferrite primarily controls the texture of hot rolled steel, which is the starting texture for cold rolling [26].
During hot rolling, austenite may transform to polygonal ferrite and pearlite by a diffusion mode, martensite by a shear mode, or acicular ferrite and bainite by mixed diffusion and shear modes. For each steel, the developed microstructure depends on the alloying elements added, the deformation amount, and the cooling rate. Also, the texture of the developed microstructure, i.e. ferrite, martensite, acicular ferrite, or bainite, can be related to the initial texture of the parent phase, austenite [26].

2.2.1 Crystallographic Texture of Hot Rolled Steel

In order to understand the texture results, it is important to know the main ideal orientations observed in rolling. Figure 2.4 illustrates the 2-dimension at $\varphi_2=45^\circ$ cross section of the Euler orientation distribution function (ODF) space showing the prominent texture fibers and relevant Miller indices in rolled cubic materials [27]. The rolling direction (RD) fiber components are typically formed by hot, warm, or cold rolling of steels [28, 29], while the textures along the transverse direction (TD) fiber are developed during hot rolling and in recrystallized steels [28, 29]. Microstructures with orientations along the normal direction (ND) fiber develop during ferritic rolling of low carbon steels and annealing [30]. The main orientations found in austenite are shown in Figure 2.5a [31]. Hot rolled austenite typically consists of the following texture components: copper (Cu) $\{112\}<111>$, S $\{123\}<634>$ (not shown in the $\varphi_2 = 45^\circ$ ODF section), brass (Br) $\{110\}<112>$, and Goss $\{110\}<001>$. If recrystallization takes place, most of these components are replaced by the cube component $\{001\}<100>$. Figure 2.5b shows the principal ideal orientations found in ferrite [31]. The cube component, if present in the austenite texture, transforms into the Goss, the rotated Goss $\{110\}<110>$, and the rotated cube $\{001\}<110>$. On the other hand, the Cu transforms to what is known as the transformed Cu $\{113\}<110>$ to $\{112\}<110>$ and the Br is replaced by the
following components: the transformed Br \{554\}<255> to \{332\}<113>, the rotated cube, and another component located at \{112\}<131>. Texture changes occurring during transformation of recrystallized austenite and deformed austenite are illustrated in Figures 2.5c and 2.5d, respectively [31].

Figure 2.4  Plot of $\varphi_2 = 45^\circ$ ODF section showing the position of the main texture components along with the RD, TD, and ND fibers [27].

2.2.2  Texture Development during Hot Rolling

During the rough rolling stage, steel is rolled in the austenite phase above the non-recrystallization temperature ($T_{NR}$). Increasing the reduction per pass and lowering the deformation temperature tend to decrease the recrystallized austenite grain size [32]. The austenite will fully recrystallize after each pass and the austenite texture consists mostly of the cube component \{001\}<100> [31, 33]. Steels produced without finish rolling show weak textures with peak intensities around the rotated cube component \{001\}<110> [26, 33, 34].

In the finish rolling stage, at temperatures below the austenite $T_{NR}$, plate or strip is typically reduced to its final thickness either in the single phase ($\gamma$ region) above the $A_{f3}$ temperature or in the two phase ($\alpha + \gamma$) region below the $A_{f3}$ temperature. This process is to flatten and accumulate rolling strain within the recrystallized austenite grains providing more ferrite nucleation sites for further transformation and grain refinement during accelerated
cooling. Finish rolling in the single phase temperature region, below \( T_{NR} \) produces flattened or elongated (pancaked) austenite grains containing deformation bands \([28, 35]\). These deformation bands act as nucleation sites for ferrite at the austenite grain boundaries. Increasing the amount of reduction in the finishing, increases the intensity of austenite texture, which leads to a stronger final texture in the ferrite phase after transformation, since no recrystallization takes place between passes \([26]\). The deformed austenite texture consisting mostly of the \{112\}<111> and \{110\}<112> components, transforms into the \{113\}<110> and \{332\}<113> components, respectively, during the austenite to ferrite transformation \([26, 33]\). Decreasing the finishing temperature above the \( A_{\gamma 3} \) is reported \([26, 34]\) to increase the intensity of the \{332\}<113> component in the TD fiber, while it has less influence on the \{113\}<110> component in the RD fiber. Deformation below \( A_{\gamma 3} \) is difficult to control due to the difference in deformation resistance between austenite and ferrite phases and causes anisotropy in strength and toughness in finished products \([36]\). Rolling in the \( \alpha + \gamma \) intercritical region will increase the strength of the final plate, however, it will reduce the toughness \([35]\). Therefore, finish rolling below the \( A_{\gamma 3} \) temperature is usually avoided \([35, 36]\). Rolling in the two phase region produces the \{554\}<225> and \{111\}<112> components in the TD fiber and the \{112\}<110> component in the RD fiber, which result from the rotation of the \{332\}<113> and \{113\}<110> components along their axes, due to the plane strain deformation of ferrite \([26]\). The \{111\}<112> component may further convert into the \{111\}<110> component by subsequent rolling \([26, 37]\).

In the cooling and coiling stage, the cooling rate and the cooling stop temperature are controlled. Bainitic microstructures, resulting from a mixed diffusion and shear transformation mode, are obtained by accelerated cooling instead of the ferrite-pearlite microstructures obtained after air cooling which normally only brings about diffusional transformations \([38]\). Further
Figure 2.5  Plots of $\varphi_2 = 45^\circ$ section of Euler space showing (a) the main orientations that play significant role during the processing of austenite, (b) the main orientations that play significant role during the processing of ferrite, (c) the ferrite texture components formed from the austenite cube component, and (d) the ferrite texture components formed from the austenite Cu and Br components [31].

An increase in the cooling rate will transform austenite into martensite by a shear transformation mode [26, 38]. The main texture components of the final microstructure after cooling are the \{332\}<113> and \{113\}<110> components along with the rotated cube \{001\}<110> component.
The overall texture is stronger at high cooling rates, where the final microstructure changes from ferrite to bainite to martensite [26, 38].

2.3 Anisotropy of Mechanical Properties

Anisotropy in strength and toughness has been observed in rolled steels, especially in bcc polycrystalline materials and is strongly related to texture [26]. A study by Inagaki et al. [29] related texture to mechanical properties in high strength steels and concluded that the \{113\}<110> component is undesirable in controlled rolled steels, since this texture component develops significant anisotropy in strength and toughness and causes brittleness to the steel in the 45° direction with respect to the rolling direction. On the other hand, the \{332\}<113> component produces less anisotropy in strength and toughness. Therefore, controlling the development of austenite textures to increase the density of the \{110\}<112> component, which leads to the formation of the desirable \{332\}<113>, \{554\}<225>, and \{111\}<112> components in the final texture, can improve strength and toughness [29]. Mourino et al. [30] found a relation between the increase in DBTT in the 45° direction and the increase in the \{001\}<110> component intensity in a study on several linepipe steels. Nafisi et al. [33] concluded that low anisotropy of mechanical properties can be linked to the presence of an adequate amount of \{332\}<113> texture component and relatively low intensities of the \{113\}<110> and \{001\}<110> components in a study on X100 steels. Baczynski et al. [27] studied the influence of preferred orientations on toughness for X80 pipeline steels with both polygonal and acicular ferrite microstructures. Charpy impact tests were conducted at room temperature, -60 °C, and -196 °C for specimens cut from the plate at 0°, 22.5°, 45°, 67.5°, and 90° with respect to the rolling direction. The study indicated that the \{112\}<110> component is responsible for the toughness anisotropy at high temperatures, where ductile fracture is observed. On the other hand,
{001}<110> and {110}<001> components cause toughness anisotropy associated with brittle fracture at low temperatures, though toughness anisotropy was not observed at low temperatures due to the low volume fraction of grains with these two texture components [27].

Several variations have been suggested to strengthen the {332}<113> component, which will lead to less anisotropy in mechanical properties. These include lowering the reheating temperature [34], lowering the finish rolling temperature [34], increasing the finish rolling reductions [39], increasing the cooling rates [40], and adding alloying elements such as Ni and Mn [29, 39].

2.4 Delamination

Splitting parallel to the rolling plane of fracture surfaces in impact specimens has been observed in ductile fracture of hot rolled steels [28, 38, 41]. Several factors have been suggested for the cause of splitting, including the presence of non-metallic inclusions such as MnS, the intensity of {001} textures, intergranular failure along prior austenite boundaries, phosphorus segregation to ferrite grain boundaries, microstructure anisotropy, pearlite and bainite banded structure elongated along the rolling direction, or a combination of these factors [28, 41].

The role of delamination reported in the literature on the DBTT is contradicting. Tanaka et al. [42] observed that both the absorbed energies and DBTTs decreased as the number of splits increased in controlled rolled steels. Rittmann et al. [43] and Shin et al. [41] found that the presence of separations in the fracture surfaces of impact samples, increases the transition temperatures of pipeline steels. Shin et al. found that the cause of splitting is due to elongated bainite regions and that the total length of separation increased as the volume fraction of bainite increased, in a study on several X80 pipeline steels [41].
2.5 Recent Studies on Delamination and Toughness Anisotropy in Pipeline Steels

Gervasyev et al. [44] studied several heavy-wall X80 pipeline steels and analyzed the microstructure and fracture behavior of these steels. Table 2.2 displays the chemical compositions in wt pct for the tested alloys [44]. Full scale burst tests were conducted on pipe sections to evaluate the crack arrestability. The results of the burst tests along with CVN impact energy values are shown in Table 2.3. All pipes exhibited acceptable crack propagation lengths except steel 2. Also, the CVN impact data did not correlate with the burst test results. Moreover, brittle splits parallel to the pipe wall were observed in some of the fracture surfaces, specifically in steel 2 [44].

Table 2.2 – Chemical Compositions for X80 Steels Investigated by Gervasyev et al. [44]

<table>
<thead>
<tr>
<th>wt pct</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Ti</th>
<th>Nb</th>
<th>V</th>
<th>Al</th>
<th>N</th>
<th>S</th>
<th>P</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steel 1</td>
<td>0.05</td>
<td>1.81</td>
<td>0.26</td>
<td>0.05</td>
<td>0.19</td>
<td>0.010</td>
<td>0.040</td>
<td>-</td>
<td>0.030</td>
<td>-</td>
<td>0.001</td>
<td>0.013</td>
<td>0.27</td>
<td></td>
</tr>
<tr>
<td>Steel 2</td>
<td>0.08</td>
<td>1.85</td>
<td>0.39</td>
<td>0.22</td>
<td>0.19</td>
<td>0.016</td>
<td>0.050</td>
<td>0.002</td>
<td>0.034</td>
<td>-</td>
<td>0.001</td>
<td>0.013</td>
<td>0.17</td>
<td></td>
</tr>
<tr>
<td>Steel 3</td>
<td>0.05</td>
<td>1.87</td>
<td>0.10</td>
<td>0.63</td>
<td>0.26</td>
<td>0.019</td>
<td>0.024</td>
<td>-</td>
<td>0.041</td>
<td>-</td>
<td>0.004</td>
<td>0.007</td>
<td>0.49</td>
<td></td>
</tr>
<tr>
<td>Steel 4</td>
<td>0.06</td>
<td>1.69</td>
<td>0.20</td>
<td>0.22</td>
<td>0.03</td>
<td>0.017</td>
<td>0.070</td>
<td>0.040</td>
<td>0.030</td>
<td>-</td>
<td>0.002</td>
<td>0.006</td>
<td>0.06</td>
<td></td>
</tr>
</tbody>
</table>

Table 2.3 – Full Scale Burst Test Parameters for Steels from Gervasyev et al. [44]

<table>
<thead>
<tr>
<th>Steel</th>
<th>Wall Thickness (mm)</th>
<th>Test Pressure (MPa)</th>
<th>Average Crack Propagation Distance (m)</th>
<th>Charpy Energy at -20 °C (J/cm²)</th>
</tr>
</thead>
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<tr>
<td>Steel 1</td>
<td>27.7</td>
<td>14.7</td>
<td>14.4</td>
<td>328</td>
</tr>
<tr>
<td>Steel 2</td>
<td>27.7</td>
<td>14.7</td>
<td>&gt;34.0</td>
<td>271</td>
</tr>
<tr>
<td>Steel 3</td>
<td>23.0</td>
<td>12.9</td>
<td>12.1</td>
<td>376</td>
</tr>
<tr>
<td>Steel 4</td>
<td>27.7</td>
<td>14.7</td>
<td>8.5</td>
<td>214</td>
</tr>
</tbody>
</table>

The microstructures of the investigated steels are presented in Figure 2.6. All steels had similar microstructures, which were characterized as a mixed microstructure of polygonal ferrite, quasi-polygonal ferrite, acicular ferrite, and bainitic ferrite with some MA islands [44]. Texture measurements were taken through the thickness of the plates by XRD and, reportedly, all alloys displayed a texture gradient except for steel 3, where the \{001\}<110> component (rotated cube component) was much sharper in the center of the plates [44]. Since the rotated cube component
is responsible for providing cleavage planes parallel to the plate normal [45], its high intensity can cause separations. However, only steel 2 showed high extent of splitting in the burst test even though steels 1 and 4 had comparable intensities of the rotated cube component (Figure 2.7). This led the authors to further characterize the steels through electron backscatter diffraction (EBSD). Figure 2.8 shows two EBSD maps for steels 2 and 4. The red and blue layers represent the distribution of grains with the rotated cube and the cube (\{001\}<100>) orientations respectively. The distribution of regions having the rotated cube orientations were more homogeneous for steel 4, while bands of grains with the rotated cube orientation were evident in steel 2 (Figure 2.8). This banded microstructure with such orientation was reported to be the main cause for failing the burst test [44].

Two non-standard mechanical tests were performed to evaluate the ductile crack propagation in order to link them with the burst test. The first method was the notched plate tensile test. Tensile test coupons with a chevron notch were tested for steels 1 and 2. Then, the specific fracture energy was calculated from the area under the load-displacement curve, which was defined as the ratio of total work to the initial cross-section [44]. The specific fracture energy for steels 1 and 2 were calculated to be 410 and 365 J/cm², respectively, and the data were consistent with the burst test results. However, it was reported that the specific fracture energy could not be used as a standard ductility measure because it depends on the sample geometry. The second test involved Charpy impact tests on samples machined from plates that were prestrained by rolling to different levels, and tested at -10 °C. The results show that the absorbed fracture energy decreased with consequent plastic deformation, as illustrated in Figure 2.9 and the effect of strain differed between the two alloys [44]. Specifically, the absorbed energy of steel 2 displayed a substantial drop after 5 percent prestrain, while steel 1 started to
Figure 2.6 Light optical micrographs showing microstructure of: (a) steel 1, (b) steel 2, (c) steel 3, and (d) steel 4 from Gervasyev et al. [44].

Figure 2.7 Distribution of \{001\}<110> intensity across the half-thickness of the pipe wall in studied steels by Gervasyev et al. [44].
show drastic decrease in the absorbed energy only after 25 percent prestrain [44]. Also, splits parallel to the rolling plane were observed in the fracture surfaces associated with the low toughness specimen. It was concluded that both the notched plate tensile and prestrained Charpy tests provide results that correlate well with the burst test data and can be used as small scale tests to assess the ductile crack arrestability of pipeline steels [44].

Figure 2.8 EBSD maps of the center layer for (a) steel 2 and (b) steel 4 from Gervasyev et al. [44]. The red and blue layers represent the distribution of grains with the \{001\}<110> and the \{001\}<100> orientations respectively.

Figure 2.9 Charpy impact absorbed energies in J/cm² vs. percent pre-strain for steels 1 and 2 investigated by Gervasyev et al. [44].
Joo et al. investigated the effects of delamination and crystallography on toughness anisotropy of X80 steels [46]. Figure 2.10 displays light optical microscopy (LOM) and scanning electron microscope (SEM) images for the studied steels. From the microstructural analysis, it was determined that the microstructure consisted of a matrix of mixed allotromorphic ferrite along with finer second phases such as pearlite and MA islands [46]. It was also reported that evidence of microstructural banding was observed. CVN impact testing was conducted in different orientations with notches perpendicular to the plate-normal to assess the toughness anisotropy. Specimen orientations and designations used are displayed in Figure 2.11. Anisotropy was present and the DBTT was higher in the D-D direction as shown in Figure 2.12. It was reported that longitudinal splits parallel to the rolling plane were observed in some of the broken surfaces of the impact specimen which exhibited ductile fracture behavior [46]. The delamination was believed to be caused by microstructural banding with differences in crystallography between adjacent bands [46]. It was concluded that the anisotropy was attributed to two factors, microstructural banding and crystallographic texture, where there are large densities of similarly-oriented cleavage planes in some orientations such as D-D orientation [46].

Joo et al. conducted follow up work to the previous study [46, 47] in order to further examine the effects of texture on toughness anisotropy. In this study [47], the same X80 steel was heat treated by austenitizing at 890 °C for 10 minutes so that banding in the microstructure was eliminated while the crystallographic texture was retained. It was reported that the banding was successfully removed and as a consequence delamination was eliminated in the Charpy broken specimens. However, strong anisotropy in the D-D direction was still observed as illustrated in Figure 2.13 [47]. It was concluded that texture plays a major role in the toughness anisotropy, specifically in the ductile to brittle transition region [47].
Figure 2.10 LOM and SEM images for showing microstructure of the X80 steel studied by Joo et al. using 2 pct nital etch [46]. (a) and (b) LOM, (c) and (d) SEM.

Figure 2.11 Orientation of Charpy specimen and the corresponding designations the steel investigated by Joo et al. [46].
Figure 2.12  Charpy impact test results: (a) as a function of temperature and (b) as a function of orientation for the X80 steel investigated by Joo et al. [46]. (See Figure 2.11 for symbol designations).

Figure 2.13  Charpy impact test results as a function of orientation for the heat treated X80 steel investigated by Joo et al. [47]. (See Figure 2.11 for symbol designations).

Mourino et al. studied the effect of texture on the anisotropy of mechanical properties of X80 pipeline steels [30]. The chemical compositions, and reheating and finish cooling temperatures for the investigated alloys are summarized in Table 2.4. Tensile and impact tests were carried out at 0, 22.5, 45, 67.5, and 90° with respect to the rolling direction. Figure 2.14a shows yield strength versus orientation with respect to the rolling direction for the studied steels. All steels showed an increase in strength of around 80 to 100 MPa in samples oriented at 67.5° and 90° to the rolling direction. The yield strength anisotropy was modeled using Taylor’s full
constraint theory [48] based on the crystallographic texture and the results are shown for the X80 plate in Figure 2.14b. The calculated results showed good agreement with the experimental yield strengths and the anisotropy in yield strength was mainly caused by the \{112\}<110> orientation [30]. Figures 2.15a and 2.15b show the absorbed energies of Charpy impact test specimens versus the sample orientation at selected temperatures for the X80 and B2 plates, respectively. Also shown in Figure 2.15 is the volume fraction of \{001\} planes parallel to the fracture planes versus sample orientation. All studied alloys showed similar anisotropic behavior, where the toughness was lowest, in terms of ductile to brittle transition temperatures, in the 45° direction with respect to the rolling direction. A corresponding increase in the volume fraction of \{001\} planes parallel to the fracture plane of the 45° orientation was also observed. It was concluded that the high intensity of the rotated cube component observed in the texture of the studied alloys was responsible for the increase in the \{001\} cleavage planes parallel to the fracture plane of the 45° direction, and thereby increasing the DBTT in the 45° orientation [30].

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Nb+Ti+V</th>
<th>Other</th>
<th>Reheating Temperature (°C)</th>
<th>Finish Rolling Temperature (°C)</th>
</tr>
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<tbody>
<tr>
<td>X80</td>
<td>0.070</td>
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<td></td>
<td>1290</td>
<td>350</td>
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<tr>
<td>A1</td>
<td>0.057</td>
<td>1.81</td>
<td>0.23</td>
<td></td>
<td>Ni, Cu, Mo</td>
<td>1190</td>
<td>580</td>
</tr>
<tr>
<td>A2</td>
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<td>1.86</td>
<td>0.30</td>
<td>&lt;0.15</td>
<td></td>
<td>1190</td>
<td>560</td>
</tr>
<tr>
<td>A3</td>
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<td>1.85</td>
<td>0.30</td>
<td></td>
<td></td>
<td>1160</td>
<td>540</td>
</tr>
<tr>
<td>B1</td>
<td>0.046</td>
<td>1.87</td>
<td>0.33</td>
<td></td>
<td></td>
<td>1190</td>
<td>500</td>
</tr>
<tr>
<td>B2</td>
<td>0.049</td>
<td>1.80</td>
<td>0.33</td>
<td></td>
<td></td>
<td>1140</td>
<td>550</td>
</tr>
<tr>
<td>B3</td>
<td>0.049</td>
<td>1.80</td>
<td>0.33</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 2.4 – Chemical Compositions and Processing Parameters for X80 Steels Investigated by Mourino et al. [30]
Figure 2.14  (a) Yield strength versus orientation with respect to the rolling direction, for the alloys studied by Mourino et al. [30]. (b) Predicted and experimental yield strengths versus test orientation for the X80 plate from [30].

Figure 2.15  Charpy impact energy and volume fraction of \{001\} planes parallel to the primary fracture planes versus sample orientation for: (a) X80 and (b) B2 plates studied by Mourino et al. [30].
CHAPTER 3: Experimental Design

This research project, a follow up to the Master of Science (MS) thesis entitled “Anisotropy of Mechanical Properties of API-X70 Spiral Welded Pipe Steels” of Haytham Al-Jabr [49], has a primary goal to understand the factors affecting toughness anisotropy in pipeline steels, crystallographic texture in particular, and to assess methods to control texture to increase toughness and reduce toughness anisotropy. Also, the causes of delamination during testing and the effects of delamination on the integrity of pipeline steels were investigated.

In the MS work [49], two X70 pipeline grades were investigated (identified as 0Ni and 0.3Ni). The study was aimed to investigate the microstructure and texture of both plates and relate the observations to the anisotropy in strength and toughness. For both steels, the DBTT was higher in the diagonal direction and the longitudinal and transverse curves were close to each other. The microstructure in terms of grain size and volume fraction of second phases was similar with respect to orientation for both alloys. A relation was found between the volume fraction of grains having their \{001\} plane parallel to the fracture plane and the DBTT, as shown in Figure 3.1. It was concluded that the anisotropy in toughness was mainly attributed to the crystallographic texture [49].

In summary, research has been performed to relate toughness anisotropy to texture, yet a fundamental understanding of the relation between microstructure, texture and toughness anisotropy has not been fully addressed. Another unresolved subject in linepipe steels is the splitting or delamination observed during toughness testing in the fracture surfaces of these steels. It is still not clear whether delamination is detrimental to toughness or not. Therefore, the fundamental questions to be answered by this work are:
• What is the influence of crystallographic texture on toughness anisotropy in pipeline steels?

• How and why does delamination during fracture affect observed toughness? What is the relationship of the susceptibility to splitting with microstructure and texture?

In order to answer the above questions, additional commercially produced X70 grade steels (Section 4.1), with processing histories different from the two alloys studied in the MS project, were acquired for the PhD study in order to have a wider matrix. Also, thermal processing cycles were applied to one plate to alter the texture for the purpose of further investigating the effects on toughness anisotropy and delamination (Section 4.2). In addition, a new impact toughness testing method was developed to intensify induced stresses parallel to notch root and thus facilitate evaluation of delamination (Section 4.5). The next chapter provides details on the experimental materials and procedures used in this project.

Figure 3.1 Fraction of grains with the \{001\} cleavage planes parallel to the fracture planes of Charpy impact specimen and the corresponding DBTT in the L (0°), T (90°), and D (45°) directions for the (a) 0Ni and (b) 0.3Ni plates [49].
CHAPTER 4: Experimental Procedure

This chapter presents the details of the materials, mechanical testing methods, and characterization procedures employed on the investigated steels.

4.1 Material

Two commercially produced X70 coil samples were received for this project in addition to the two alloys (0Ni and 0.3Ni) studied in the Master of Science project [49]. The first alloy was provided by SSAB, referred to as “Base” steel. The thickness of the plate produced was 13.74 mm (0.541 inch). The coil was produced in a Steckel rolling mill at SSAB Americas. The second steel was provided by SABIC with a thickness of 15.5 mm (0.611 inch), referred to as “15T” steel. The coil was produced in a hot strip mill (HSM) at HADEED, Saudi Arabia. The width and thickness of the two X70 steels provided by Essar Algoma for the MS project [49] were 1550 mm and 12.88 mm, respectively. The steels were produced at the Direct Strip Production Complex (DSPC) in Algoma, Canada. One steel contained no nickel (identified as “0Ni”) and the other with 0.3 wt pct nickel (identified as “0.3Ni”). The chemical compositions for the Base and 15T steels, along with the 0Ni and 0.3Ni steels studied in the MS project [49-51], are shown in Table 4.1.

4.2 Heat Treatment

Heat treatment schedules to alter the microstructure and texture were conducted on the 0Ni plate to assess the effects on toughness. The aim was to produce a ferritic/bainitic microstructure having strength within the specification of X70. The heat treatment was expected to eliminate any microstructural banding due to rolling and also weaken the texture. Trials were made for heat treatments to obtain a bainitic microstructure with matching hardness to the
as-received plate (200 HV) by austenitizing for a period of time then rapidly cooling to an intermediate temperature where the samples were held for sufficient time for isothermal transformation to bainite and then air cooled to room temperature. After several trials, the optimum schedule, where the hardness was 200 HV, was as follows: austenitize at 940 °C for 25 minutes in an air furnace, quench in a salt pot to an intermediate temperature of 300 °C for 10 minutes, and then air cool to room temperature. Rectangular samples were rough machined for standard and modified Charpy impact and tensile testing in the L, T, and D directions, as illustrated in Figure 4.1, and heat treated according to the selected schedule. After heat treatment, the tensile and impact samples were machined to their final dimensions as described in Sections 4.4-4.6.

<table>
<thead>
<tr>
<th>wt pct</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
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<td>15T</td>
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<td>1.520</td>
<td>0.20</td>
<td>0.009</td>
<td>0.130</td>
<td>0.172</td>
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<td>Low</td>
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</tr>
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</table>

### 4.3 Tensile Testing

Tensile tests were conducted at the Colorado School Mines, in the longitudinal (L, 0°), transverse (T, 90°) and diagonal (D, 45°) orientations with respect to the rolling direction for the investigated plate steels using an MTS Alliance RT/1100 machine equipped with an 89 KN (20,000 lb) load cell. Figure 4.2 shows the sample orientations with respect to the rolling direction. Standard flat tensile samples (12.5 mm (0.5 inch) wide), as shown in Figure 4.3, were
machined from the center of the plates with 5.6 mm (0.22 inches) thickness and tested at room temperature in air at 2.54 mm/min (0.1 in/min) according to ASTM standard A370 [52]. Yield strengths were calculated using 0.2% strain offset and the total strain to failure was measured in a 50.8 mm (2 inch) gauge length with a Shepic extensometer.

Figure 4.1  The dimensions of the rectangular bars prepared for heat treatment prior to final machining for the impact and tensile test samples for the 0Ni plate. Samples were cut in the longitudinal, transverse, and diagonal orientations as shown in Figure 4.2.

Figure 4.2  Schematic drawing showing the different orientations of the samples machined from the rolled plate for the tensile and Charpy samples. L designates longitudinal direction, T designates transverse direction, and D designates diagonal direction. ND is the normal direction, TD is the transverse direction, and RD is the rolling direction.
Figure 4.3  Schematic showing sample dimensions of the tensile specimens. All dimensions are in mm. Adapted from [52].

4.4  **Standard Impact Testing**

Full size (55 mm x 10 mm x 10 mm) conventional Charpy V-Notch (CVN) impact samples, with the dimensions shown in Figure 4.4, were machined in each direction (L, T, and D) for the four plates and tested at different temperatures to determine the ductile to brittle transition temperature curves (DBTT), according to the ASTM E23 standard [53]. Figure 4.2 shows the sample orientations with respect to the rolling direction. Impact tests for the 0Ni and 0.3Ni plates were performed on a 358 J (264 ft-lb) capacity Tinius-Olsen machine. The upper shelf energies (USE) for the tested materials exceeded the capacity of the machine and additional samples were tested at room temperature at the National Institute of Standards and Technology (NIST) on an MPM Pendulum 9000 test machine, with a capacity of up to 950 J (700 ft-lb). Samples for the 15T, Base, and 0Ni heat treated plates were also tested at NIST. All testing below room temperature was done by submersing the samples in an isothermal bath of ethanol and liquid nitrogen for 10 minutes. Transition temperature curves were fitted mathematically using Equation 4.1 as illustrated in Figure 4.5 [54]:

```
5.6
```
In Equation 4.1, $Y$ is the energy absorbed for the energy absorbed vs temperature plots and the percent shear for the percent shear vs temperature plots, $T$ is the temperature (in °C) at a given $Y$ value, and $A$, $B$, $C$, and $T_o$ are defined in Figure 4.5. The 150 J transition temperature was determined for the energy absorbed vs temperature plots at the 150 J value on the transition curve. The 50 percent shear transition temperature was determined by the intersection of the 50 percent shear and the transition temperature curve from the percent shear vs temperature plots.

Figure 4.4   Schematic showing sample dimensions of the standard impact specimens.

4.5  Modified Impact Testing

Modified Charpy V-notch (MCVN) impact test specimens with side grooves or slits were designed and machined for the 15T and 0Ni (as-received and heat treated conditions) alloys in the L, T, and D directions, as shown in Figure 4.6. The difference from the conventional full size Charpy V-notch (CVN) specimen is that the specimen width is 12 mm with 1 mm deep sharp grooves on both sides so that the width and remaining geometry of the specimens, excluding the grooves, is identical to the standard 10 mm CVN sample, and thus the fracture surface area

\[
Y = A + B \cdot \tanh \left( \frac{T - T_0}{C} \right)
\]  

(4.1)
would be the same as the conventional CVN samples, as shown in Figure 4.6b. Modified Charpy V-notch samples were tested on a 358 J (264 ft-lb) capacity Tinius-Olsen machine at Colorado School of Mines. Transition temperature curves and values for the MCVN data were obtained using the same method for standard CVN data described in Section 4.4.

![Diagram](image.png)

**Figure 4.5** Schematic impact test plot describing the coefficients used in Equation 4.1 used to construct the ductile to brittle transition temperature curve [54].

### 4.6 Metallography

Metallographic samples on the L, T & D planes as shown in Figure 4.7 were mounted in Bakelite, ground with 240, 320, 400, and 600 grit SiC grinding paper, polished in three steps with 6 µm, 3 µm, and 1 µm diamond suspension, and etched with 2% nital solution. Water was avoided in all grinding and polishing steps because pitting was observed in samples polished with water-based suspensions. Therefore, alcohol-based solutions were used. Also, selected samples were etched with LePera’s etchant [55] (1:1 ratio of 1% aqueous sodium metabisulfite and 4% picric acid in ethanol). Microstructural characterization was conducted through light
optical microscopy (LOM) and scanning electron microscopy (SEM). Metallographic samples were examined with an Olympus PMG3 light optical microscope, environmental scanning electron microscope (ESEM), and a high resolution JEOL 7000F field emission scanning electron microscope (FESEM). The fracture surfaces of Charpy impact samples were evaluated with ESEM and FESEM.

Figure 4.6  
(a) Photo of the modified Charpy V notch (MCVN) sample with side slits for the 15T alloy, and (b) schematic drawing showing the geometry and dimensions of the modified Charpy sample for the 15T and 0Ni alloys.
Selected CVN samples in the L plane orientation that exhibited delamination were investigated near the splits of the broken specimen. The samples were initially nickel plated to preserve the fracture surfaces and prevent damage during sectioning and polishing. Nickel plating was conducted using a Caswell electroless nickel plating kit. The samples were sectioned after plating to expose the microstructure close to the splits in the fractured CVN specimen as shown in Figure 4.8. Then, the samples were mounted, ground, and polished as outlined in the procedure above.

Figure 4.7  Schematic drawing showing the different orientations of the metallographic samples with respect to the rolled plate: RD is the rolling direction, TD is the transverse direction, and ND is the direction normal to the rolling plane. For alternate reference the notations L, T, and D represent the longitudinal, transverse, and diagonal planes respectively.

4.7  Electron Backscatter Diffraction (EBSD)

Electron backscatter diffraction patterns were obtained using two different systems; a JEOL 7000F FESEM equipped with an EDAX DigiView camera, and a FEI Helios Nanolab DualBeam Focused Ion Beam-SEM (FIB-SEM) equipped with an EDAX Hikari camera. Both microscopes were operated at an accelerating voltage of 20 kV. Polished samples were mounted in the SEM at a 70° angle relative to the incident electron beam, enabling Bragg diffraction
conditions to be met without having to rock the electron source. When an electron beam strikes a surface inclined in this manner, the electrons penetrate the sample surface and are diffracted to produce distinct patterns based on the local lattice orientation. These patterns appear as multiple intersecting lines, termed Kikuchi bands. The arrangement of the bands can be used to index the pattern and obtain a lattice orientation if the patterns arise from a single crystal, or within one grain of a polycrystal [56]. The EBSD camera is used to collect the image of the Kikuchi patterns for indexing by the EDAX OIM™ Data Collection Software.

![Diagram showing sectioned CVN specimen](image)

**Figure 4.8** Schematic drawing showing the sectioned CVN specimen in the L plane orientation near the split.

The OIM Data Collection Software also computes two parameters of significance for the diffraction data: the confidence index (CI) and the image quality (IQ). The CI is a measure of the degree to which the software selected the correct orientation for each data set. CI values are based on the voting scheme used in ranking proposed solutions and is determined by the following equation [57]:

\[
CI = \frac{V_1 - V_2}{V_{ideal}}
\]

(4.2)

where, \(V_1\) is the number of votes for the first solution and \(V_2\) is the number of votes for the second solution. Finally, \(V_{ideal}\) is the total number of votes for all proposed solutions. Essentially, CI is a measure of the difference between the number of votes for the best solution and for the
second best solution. If the difference is large, the software has indicated that the best solution is highly likely and the assigned CI is therefore high. If the difference is small, there is uncertainty in the software solution and the CI will be low. The CI can range from zero to one and CI values greater than 0.1 correspond to a 95% probability that a Kikuchi pattern has been correctly indexed [56, 57].

Sample preparation for EBSD went through the same steps mentioned in Section 4.6 with an additional polishing step on a vibratory polisher using 0.05 μm colloidal silica. Various EBSD scans were collected for the investigated steels on the L, T, D, and normal planes (Figure 4.7). Scans to capture the bulk texture of the steels were performed on the normal plane and on an area of 800 x 1000 μm with a step size of 2 μm. For each alloy, two scans were taken at the surface, quarter thickness, and half thickness of the plate. For microstructural analysis, different areas were measured with step sizes ranging between 50 nm and 0.15 μm. Scans were performed at a working distance of 20 mm on the FESEM and 15 mm on the FIB. IQ maps, grain boundary misorientation angles, austenite volume fractions, average grain sizes, and crystallographic texture data were obtained using EDAX OIM™ Data Analysis Software.

4.8 Grain Size Measurements

Average grain size measurements were conducted using two techniques. The first method was by using the Abrams three circle procedure [58]. Grain size measurements were calculated on SEM images with a magnification of 2000X such that each field would yield around 100 counts, and counts were taken manually for each field. An average of 800 counts per specimen was collected over eight randomly selected images such that acceptable precision could be obtained. For each field, the number of intercepts was counted and divided by the total circumference of the three circles. Then the average grain size was calculated for each alloy in
the L, T, and D planes (Figure 4.7). Figure 4.9 shows a representative SEM image overlaid with concentric circles used to calculate the grain size using Abrams’ method.

The second method was Heyn’s line intercept method [58]. In this method, the average linear intercept lengths in the horizontal direction (TD for the L plane, RD for the T plane, and 45° with respect to the rolling direction for the D plane in Figure 4.10a) were calculated to determine the effective grain size. The average linear intercept length in the horizontal direction was used as another method to characterize the average grain size because the horizontal direction coincides with the direction of crack propagation in the Charpy impact testing for each test orientation. Additionally, the average linear intercept lengths in the vertical direction (ND in Figure 4.10b) were calculated to determine the grain aspect ratio. The grain aspect ratio was determined for each orientation by dividing the average intercept length in the horizontal direction by the average intercept length in the vertical direction. For both horizontal and vertical directions, calculations were performed on five randomly selected SEM images at 2000X so that approximately 100 counts were taken for each direction per field, as illustrated in Figure 4.10.

![Representative SEM image to demonstrate the method used to determine the average grain size using the concentric circles method.](image)
Figure 4.10  Schematic to demonstrate the method used to determine the (a) average linear intercept length in the TD for the L samples, RD for the T samples, and 45° direction for the D samples, and (b) average linear intercept length in the ND. The grain aspect ratio is then calculated by dividing the average linear intercept defined in (a) by the average linear intercept defined in (b).

Similar procedures for grain size measurement were performed on EBSD maps. For each alloy and orientation, two widely spaced scans were used to calculate the average grain size. All scans were performed at an accelerating voltage of 20 kV and a working distance of 15 mm. An area of 100 X 100 µm was selected for each scan with a step size of 0.1 µm. Color-coded grain maps were generated using EDAX OIM\textsuperscript{TM} Data Analysis Software for the grain size calculations. A grain tolerance angle of 15° was used as a threshold to calculate the grain size. Each image was divided into four equally sized images (50 X 50 µm) in order to perform the calculations with better precision. Therefore, a total of eight images per direction was used to perform the calculations. A representative color-coded image overlaid with concentric circles, horizontal lines, and vertical lines used for EBSD grain size calculations are shown in Figures 4.11a, 4.11b, and 4.11c, respectively.
Additionally, grain size distributions were calculated for all four steels in each direction on EBSD collected maps, according to the procedure for characterizing bimodal conditions outlined in the ASTM E-1181 standard [59].

The standard deviation, $s$, was calculated for both methods using the following equation:

$$s = \sqrt{\frac{\sum (x_i - x_m)^2}{n-1}}$$

(4.3)

where, $X_i$ is the average grain size for each field in first method and the mean lineal intercept in the second method, $X_m$ is mean value of $X_i$, and $n$ is the number of measured fields.

![Representative color-coded maps for the 15T alloy in the T plane to demonstrate the method used to determine the (a) average grain size using concentric circles method, (b) average linear intercept length in the horizontal direction, and (c) average linear intercept length in the vertical direction. Each colored grain shows a misorientation of 15° or higher with its neighbor.](image)
CHAPTER 5: Results

The experimental results obtained from characterization and mechanical testing are presented in this Chapter. The microstructures, crystallographic textures, tensile properties, and impact properties for the four studied alloys are presented here. Also, modified impact test data for selected alloys are discussed. Heat treatment results for the 0Ni plate with modified microstructure are also presented in this Chapter.

5.1 Microstructure

The microstructures of the four alloys viewed on the transverse plane are introduced here. Detailed microstructural characterization is presented in Section 5.3. Microstructural characterization was conducted using LOM, and SEM with metallographic samples prepared according to the procedure outlined in Section 4.6.

Figures 5.1 and 5.2 respectively display light optical micrographs and SEM images for the four steels viewed in the transverse direction with respect to the rolling direction. All microstructures are similar, irrespective of the processing method and chemistry, and are dominated by non equiaxed ferrite grains known as acicular ferrite in the pipeline steel terminology (sometimes referred to as bainitic ferrite or quasi-polygonal ferrite [2, 8, 46]), with second phases or microconstituents distributed in and between the ferrite grains. The fine ferritic-based microstructures provide the desirable strength and toughness combination necessary to produce X70/X80 API grades [4, 5, 8, 10-13]. The resulting microstructures exhibit no evidence of the prior austenite grain boundaries. The ferrite grain sizes are mixed ranging from approximately 2 µm to 10 µm with most of the grains being in the smaller range. Centerline segregation was evident in the 15T steel as pointed out by the arrow in Figure 5.1a; no such occurrence was observed in the other alloys. The Base steel microstructure contained limited
regions of banded structure where the grains were noticeably larger than the matrix. The 0Ni and 0.3Ni microstructures displayed discontinuous bands of relatively larger grains (pointed out by the arrow in Figures 5.1d and 5.2d) through the thickness of the plates, i.e. not confined to the mid thickness layer. The regions of elongated grains, observed in all for alloys, can reach up to 50 µm in length parallel to the rolling plane. In addition, the 0Ni and 0.3Ni microstructures contained bands of upper bainite as shown by the arrow in Figures 5.1c and 5.2c. The second phase constituents cannot be distinguished in the Figures 5.1 light optical micrographs and could include pearlite, martensite-austenite islands, retained austenite, or carbides. Figure 5.3 shows light optical micrographs for the four steels etched with LePera’s solution. White colored MA islands are clearly evident in addition to dark constituents interpreted as carbon enriched bainite and/or cementite. Also, it is interesting to note that, for the 15T steel (Figure 5.3a), the second phases are more prevalent in the segregated layer due to the carbon enriched residual austenite during transformation leading to a localized banded structure. Further microstructural characterization results by means of SEM and EBSD are presented in Section 5.3.

5.2 Mechanical Properties

Tensile testing and standard Charpy V-Notch (CVN) impact testing were conducted for the 15T, Base, 0Ni, and 0.3Ni alloys in the longitudinal (L, 0°), transverse (T, 90°) and diagonal (D, 45°) orientations with respect to the rolling direction as illustrated in Figure 4.2. The results are presented and discussed in this section.

5.2.1 Tensile Properties

Tensile properties for an average of two samples per direction are summarized in Table 5.1 and representative engineering stress-strain curves in the L, T, and D orientations for the 15T, Base, 0Ni, and 0.3Ni alloys are shown in Figures 5.4-5.7, respectively. Yield strengths
Figure 5.1 Light optical micrographs showing microstructures of the four steels taken in the mid thickness in the transverse direction (2 pct nital etch): (a) 15T, (b) Base, (c) 0Ni, and (d) 0.3Ni. The arrows point to centerline segregation in (a), upper bainite in (c), and large bainite region in (d).

were calculated using 0.2% offset and the total strain to failure was measured in a 50.8 mm (2 inch) gauge length with a Shepic extensometer. The 15T and 0.3Ni plates have relatively higher yield and tensile strengths than the Base and 0Ni plates. The orientation dependence is similar for all alloys where the yield and tensile strengths in the T direction are higher than that in the L and D directions, an observation which has also been reported in the literature [27-29, 60, 61]. The total elongation is between 19 and 26 percent for all orientations and the elongation is always lower in the T orientation for all four alloys. The tensile properties meet the API requirements for X70 (485 MPa minimum yield strength and 570 MPa minimum tensile strength.
in the pipe hoop direction) in the D direction, which is close to the hoop direction of the pipe except for the Base and 0Ni steels (the yield strengths were less than 485 MPa). However, it should be noted that the API requirement is to test the full thickness of the plate, while the tests were performed on 5.6 mm thick samples due to machine limitations.

Figure 5.2 SEM images showing microstructures of the four steels taken in the mid thickness in the transverse direction (2 pct nital etch): (a) 15T, (b) Base, (c) 0Ni, and (d) 0.3Ni. The arrows point to upper bainite in (b) and large bainite region in (c)
For all three orientations, Figure 5.4b shows that the 15T samples exhibited discontinuous yielding behavior, while the Base samples (Figure 5.5b) showed a quasi-continuous, yielding behavior. The 0Ni (Figure 5.6b) and 0.3Ni (Figure 5.7b) alloys displayed continuous yielding. Although the microstructure of the studied steels are similar, the continuous yielding of the 0Ni and 0.3Ni steels could be attributed to the presence of upper bainite and higher volume fraction of microconstituents (Section 5.3.2), leading to a higher initial density of mobile dislocations, and thus continuous yielding.

Figure 5.3 Light optical micrographs showing microstructures of the (a) 15T, (b) Base, (c) 0Ni, and (d) 0.3Ni steels, taken in the mid thickness in the transverse direction using LePera’s etchant.
Table 5.1 – Average Tensile Test Results for the Four Alloys in the L, T, and D directions, Based on Two Samples for each Direction

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<th>YS (0.2% offset) (MPa)</th>
<th>UTS (MPa)</th>
<th>Y/T Ratio</th>
<th>Percent Elongation (in 50.8 mm)</th>
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<td>627</td>
<td>0.86</td>
<td>23.8</td>
</tr>
<tr>
<td></td>
<td></td>
<td>D</td>
<td>527</td>
<td>605</td>
<td>0.87</td>
<td>26.3</td>
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<td></td>
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<td>609</td>
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<td>23.3</td>
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</table>

Figure 5.4 (a) Engineering stress-strain curves for the 15T steel tested in the three orientations. (b) Low strain values in (a) are amplified to illustrate yielding behavior. L designates longitudinal direction, T designates transverse direction, and D designates diagonal direction.
Figure 5.5  (a) Engineering stress-strain curves for the Base steel tested in the three orientations. (b) Low strain values in (a) are amplified to illustrate yielding behavior. L designates longitudinal direction, T designates transverse direction, and D designates diagonal direction.

Figure 5.6  (a) Engineering stress-strain curves for the 0Ni steel tested in the three orientations. (b) Low strain values in (a) are amplified to illustrate yielding behavior. L designates longitudinal direction, T designates transverse direction, and D designates diagonal direction.
5.2.2 Standard CVN Properties

Charpy V-notch impact testing was conducted to develop DBTT curves for the four alloys in the longitudinal, transverse, and diagonal directions. The results are shown in Figures 5.8-5.11 for energy absorbed versus temperature data and percent shear versus temperature (as determined following the ASTM E-23 “Guide for Estimating Fracture Appearance”). Each steel and sample orientation exhibits a well-defined transition from ductile to brittle behavior, with the transition more sharply defined by the percent shear curves. The dotted line in Figure 5.9b is an estimation of the curve behavior, since no specimens for the Base alloy were tested in the temperature range between -115 °C and liquid nitrogen temperature (-196 °C), and all specimen tested at and above -115 °C showed 100% shear for the L orientation. However, a steep transition curve similar to the T and D directions would be expected. Also, the absorbed energy values in the ellipses for the 0Ni and 0.3Ni plots at the upper shelf regions in Figure 5.10 and 5.11 were obtained at the maximum energy limit of the impact testing machine.
at CSM. Additional samples were tested on a higher capacity machine at room temperature to determine the USE values as stated in Chapter 4. Transition temperatures were determined from both the energy absorbed and percent shear data. The results for the 150 J transition temperatures and 50 percent shear transition temperatures from Figures 5.8-5.11 are summarized in Table 5.2. For all alloys, the DBTT values for the L and T orientations are similar to one another, while the DBTT is highest in the D direction. The severity of the anisotropic behavior of the transition temperature, as evident by the difference in DBTT for the D orientation from the L and T orientations, differed between the steels, being more pronounced in the 0Ni plate and weakest in the 15T alloy. The DBTT (in terms of 50 percent shear) for the L and T orientations is lower by around -20 °C for the 15T and Base alloys compared to the 0Ni and 0.3Ni alloys. The USE values for all four alloys are within close proximity to each other and there is only slight anisotropy in the USE between the different directions (Table 5.2).

Figure 5.12 compares the temperature dependence of the absorbed energy and percent shear data for the 15T steel in the L orientation. The behaviors in Figure 5.12 are characteristic of all the other steels in this study and show that even with a decrease in absorbed energy of over a factor of 2, primary fracture remains fully ductile down to approximately -115 °C, and the transition in fracture appearance for 100% shear to ~0% shear occurred over a very narrow temperature range. Also shown in Figure 5.12 are selected macrophotographs at the indicated temperatures. At the higher temperatures, the samples did not fracture completely and significant plastic deformation was observed as shown in Figure 5.12a for samples with absorbed energies above 400 J. At relatively lower temperatures the samples fully fractured and a typical 100 percent ductile fracture surface was observed (Figure 5.12b). This observation correlates with the differences in the curve shapes between the energy absorbed and percent shear data.
Figure 5.8 Standard Charpy V-Notch curves developed for the 15T samples in the longitudinal direction as (a) absorbed energy and (b) percent shear, in the transverse direction as (c) absorbed energy and (d) percent shear, and in the diagonal direction as (e) absorbed energy and (f) percent shear.
Figure 5.9 Standard Charpy V-Notch curves developed for the Base samples in the longitudinal direction as (a) absorbed energy and (b) percent shear, in the transverse direction as (c) absorbed energy and (d) percent shear, and in the diagonal direction as (e) absorbed energy and (f) percent shear.
Figure 5.10  Standard Charpy V-Notch curves developed for the 0Ni samples in the longitudinal direction as (a) absorbed energy and (b) percent shear, in the transverse direction as (c) absorbed energy and (d) percent shear, and in the diagonal direction as (e) absorbed energy and (f) percent shear.
Figure 5.11  Standard Charpy V-Notch curves developed for the 0.3Ni samples in the longitudinal direction as (a) absorbed energy and (b) percent shear, in the transverse direction as (c) absorbed energy and (d) percent shear, and in the diagonal direction as (e) absorbed energy and (f) percent shear.
Also, longitudinal splits were found in most of the fracture surfaces, particularly for the L orientation in the ductile to brittle transition region (Figure 5.12c) where samples exhibited 100% ductile fracture on primary fracture plane. The split samples were associated with lower energy than the samples that did not exhibit splitting. In addition, with a decrease in temperature (in the transition curves), the fracture surfaces, while exhibiting full ductility, became less smooth and significant portions of the surfaces were pulled out (Figure 5.12d), resulting in lower absorption energies, especially in the T and D orientations. Figure 5.13 shows an SEM image for the fracture surface taken near a longitudinal split for the Base sample in the L orientation tested at -90 °C similar to the split shown in Figure 5.12c. The splits are associated with planes of secondary cleavage fracture perpendicular to the primary fracture surface. Complete CVN fracture surface macrographs can be found in Appendix C.

<table>
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<tr>
<th>Steel</th>
<th>Direction</th>
<th>USE (J)</th>
<th>150 J (°C)</th>
<th>50% Shear (°C)</th>
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</tr>
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<td>T</td>
<td>450</td>
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<td>-112</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>405</td>
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<td>-85</td>
</tr>
<tr>
<td>Base</td>
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<td>440</td>
<td>-89</td>
<td>-93</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>460</td>
<td>-89</td>
<td>-90</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>440</td>
<td>-45</td>
<td>-50</td>
</tr>
<tr>
<td>0.3Ni</td>
<td>L</td>
<td>450</td>
<td>-90</td>
<td>-92</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>465</td>
<td>-88</td>
<td>-90</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>435</td>
<td>-65</td>
<td>-65</td>
</tr>
</tbody>
</table>
Figure 5.12  Charpy V-Notch energy absorbed and percent shear data for the 15T longitudinal samples. Also included are representative macrographs of the different 100 percent ductile fracture surface types observed: (a) bent specimen, (b) smooth fracture, and (c, d) delaminated fracture.

Figure 5.13  SEM image for the fracture surface taken near the longitudinal split for the Base sample tested in the L orientation tested at -90 °C: (a) low magnification and (b) high magnification.
5.3 Microstructural Characterization

The general microstructures of the studied steels were presented in Section 5.1. In this section, the morphologies, sizes, and distributions of the ferrite grains along with corresponding observations for the second phases or microconstituents are presented. Results are based on SEM and EBSD images and data, and used to interpret the effects of microstructure on the anisotropy of mechanical properties observed in Section 5.2.

EBSD scans were taken for the studied alloys in the L, T, and D planes as indicated in Figure 4.7. Representative image quality (IQ) maps in the transverse plane are shown in Figure 5.14 for the four plates. Images shown in Figure 5.14 were collected with a step size of 0.1 µm on a 100 x 100 µm area. No distinctive differences were found between the different orientations and complete IQ maps for all steels in the L, T, and D planes are summarized in Appendix A. From Figure 5.14, the microstructures are mixed showing pancaked grains with irregular shapes and sizes with microconstituents in and between boundaries. The majority of the microstructure consists of fine ferritic grains ranging from 1 to 10 µm in length parallel to the normal plane. Some larger grains are present with sizes around 20 and 30 µm. Also, regions of much larger elongated grains parallel to the normal direction, up to 50 µm in length, were observed in all four alloys (shown by the arrow in Figure 5.14a).

Figure 5.15 shows selected IQ maps with overlaid color-coded boundary segments in the transverse plane for the four alloys using a smaller step size (50 nm) on a smaller area (35 x 35 µm) to provide a more detailed examination of specific microstructural features. The red and green boundaries are low angle boundaries (LAB), between 2-5° and 5-15°, respectively, and the blue boundaries are high angle boundaries (HAB) larger than 15°. As stated earlier, most of the microstructure is composed of fine non equiaxed irregular shaped ferrite grains, i.e., acicular ferrite (AF) as indicated by labeled arrow in Figure 5.15b. Little or no substructure is
developed as evidenced by the relatively smooth appearance within the grains and the grains are
separated by HABs. MA or RA islands are located at irregular shaped grain boundaries. Some
more equiaxed grains are present with no substructure within the grains. These grains are
believed to be polygonal ferrite (PF), an example is highlighted in Figure 5.15a. PF grains can be
distinguished from AF in the image quality maps, as they exhibit high IQ values due to the
absence of dislocations and substructure, and correspondingly appear as very light grey [62]. An
eexample of the elongated regions is shown in Figure 5.15c. These regions or grains contain well
developed substructure and have sub-units or grains separated by LABs. These grains also
contain MA or RA islands inside or between boundaries. These larger grains are interpreted as
bainite (B).

Figures 5.16 and 5.17 display representative SEM images and EBSD IQ maps
superimposed with a phase map, respectively, of the differentmorphologies of microconstituents
observed in the studied steels. The austenite indexed areas are colored red and the remainder is
ferrite in Figure 5.17. The most common microconstituent existing in all four steels is shown in
Figures 5.16a and 5.17a. This complex incomplete transformation product consists of a mixture
of fragments of ferrite, cementite, martensite, and austenite. These secondary products are
formed as separate grains between irregular ferrite grains or inside larger ferrite or bainite grains.
Figures 5.16b and 5.17b show MA islands where sheaves of martensite are separated by thin
layers of retained austenite. Thin strips of austenite (colored in red) can be clearly observed
between the dark grey martensite particles in Figure 5.17b. Figures 5.16c and 5.17c, show
retained austenite, the smooth particle consists of retained austenite as evident in the IQ map in
Figure 5.17c, where the particle is completely indexed as austenite. MA and RA islands were
also observed either between grains (Figure 5.16b) or inside bainite grains (Figure 5.16c). The
fourth type of microconstituent observed is the elongated cementite precipitates along upper bainite lath boundaries as shown in Figures 5.16d and 5.17d. Upper bainite was only present in the 0Ni and 0.3Ni alloys, where packets of parallel ferrite grains were ordered with an angle
between 40 and 60°, and the ferrite grains were separated by low angle boundaries. Also, note
that no traces of austenite were found in the elongated particle (Figure 5.17d).

Figure 5.15  Selected IQ maps in the transverse plane for the (a) 15T, (b) Base, (c) 0Ni, and (d) 0.3Ni alloys. Red and green boundaries are low angle boundaries (2°-15°) and blue boundaries are high angle boundaries (greater than 15°).
Figure 5.16  Representative SEM images showing the microconstituents taken in the transverse direction using 2 pct nital etch: (a) Base steel showing incomplete transformation products, (b) 15T steel showing MA island, (c) 0.3Ni steel showing RA island, and (d) 0Ni steel showing cementite.

5.3.1 Grain Size Measurements

Grain size quantification was performed using different techniques on the L, T, and D planes defined in Figure 4.7 to provide data for correlation with toughness anisotropy. Average grain size measurements in each plane along with grain aspect ratios for all four alloys are displayed in Figure 5.18. The average grain sizes shown in Figure 5.18 are calculated on SEM images using the three concentric circles approach described in Section 4.8. For all steels, the grain sizes in each direction were comparable, indicating no anisotropy with respect to direction.
Figure 5.17 Representative IQ maps overlaid with phase maps showing the microconstituents taken in the transverse direction: (a) Base steel showing incomplete transformation products, (b) 0.3Ni steel showing MA island, (c) 0.3Ni steel showing RA island, and (d) 0.3Ni steel showing cementite. Ferrite is grey and austenite is red.

The average grain sizes were approximately 2.7, 2.8, 3.4, and 3.3 µm for the 15T, Base, 0Ni, 0.3Ni alloys, respectively. However, in order to assess grain size effects on toughness properties,
it is important to consider the distance between grains, as determined by grain aspect ratio, in the path of the crack propagation in the impact test specimen, which is parallel to the normal plane (ND). Therefore, the grain aspect ratio, defined in Figure 4.10, is a potentially more meaningful parameter to correlate with impact properties as shown in Figure 5.18. The grain aspect ratios for the 15T and Base steels were about 1.45 for all directions, with the exception of the T orientation for the Base steel where it was 1.65. For the 0Ni and 0.3Ni plates, the grain aspect ratios were similar for all orientations, around 1.28. The lower grain aspect ratios for the 0Ni and 0.3Ni steels are likely because the plates were processed from thin slabs and hence, subjected to lower total reduction during rolling compared to the other two alloys. Also, the increased aspect ratio in the T direction for the Base steel is possibly attributed to more severe rolling for the Base below T_{NR}, resulting in a more intense pancaked structure. It is worthwhile to note that even at 2000X some regions were difficult to resolve, as the microstructure was too fine. Also, the visibility of grain boundaries are subjected to the sample response to etching and the microstructural features could be more revealed in some samples than the others. Therefore, additional grain size measurements were made on EBSD scanned images. Images from EBSD enable setting specific values for grain boundary angles in measuring the grain size.

Average grain sizes (using concentric circles method) were measured on EBSD color-coded maps for different grain tolerance angles in order to observe the difference in grain size with increasing grain tolerance angle and are shown in Figure 5.19 for the 15T alloy in the T plane. The calculated average grain size increases by increasing the threshold tolerance angle from 5° to 15°, then plateaus after 15°. Therefore, average grain size measurements obtained from EBSD were calculated based on a minimum grain tolerance angle of 15°. Figure 5.20 displays average grain size measurements and grain aspect ratios for the four alloys in each plane.
Figure 5.18  Average grain sizes and grain aspect ratios measured from SEM images on the longitudinal (0°), diagonal (45°), and transverse (90°) planes identified in Figure 4.7 for the: (a) 15T, (b) Base, (c) 0Ni, and (d) 0.3Ni steels.

performed on EBSD images. For all steels, no considerable variation in the grain size as a function of orientation was observed, as was concluded from grain size measurements performed on SEM images. The average grain sizes from EBSD data were slightly higher than those taken on SEM images, around 3.2 µm for the 15T and Base alloys and around 3.5 µm for the 0Ni and
0.3Ni alloys. For all four alloys, the grain aspect ratio values were slightly higher in the T orientation, around 1.4, and the L and D orientations ranged between 1.2 and 1.35.

![Graph showing average grain size as a function of grain tolerance angle.](image)

**Figure 5.19** Average grain size as a function of grain tolerance angle.

As mentioned earlier, regions of relatively larger grains were observed in the microstructure, where the grains were much larger than the average grain size, as shown in Figure 5.21. Figure 5.21 shows a grain boundary map developed from EBSD data for the 15T steel in the T plane for grain boundary misorientation equal to or higher than 15°. The duplex or bimodal grain size is reported to be detrimental for impact toughness and causes significant scatter in the data [63, 64]. Therefore, it is important to consider the grain size distribution in order to correlate with the toughness properties. Grain size distribution calculations were performed on the four plates from EBSD data based on the procedure for characterizing bimodal conditions outlined in ASTM E-1181 [59]. Figures 5.22-5.25 display the percent of total intercept lengths versus
Figure 5.20  Average grain sizes and grain aspect ratios measured from EBSD data on the longitudinal (0°), diagonal (45°), and transverse (90°) planes identified in Figure 4.7 for the: (a) 15T, (b) Base, (c) 0Ni, and (d) 0.3Ni steels.

intercept length for each orientation for the 15T, Base, 0Ni, and 0.3Ni, respectively. For each alloy and direction, the intercept lengths in the direction parallel to the normal plane of the plate (direction of the crack propagation in the impact samples) were measured in small intervals (0.5 µm intervals). Then, the total intercept length for each interval was divided by the total
intercept length for all intervals and multiplied by 100. After that, the grains were divided into fine grains (smaller than 10 µm) and coarse grains (larger than 10 µm), and the average grain sizes for the fine and coarse grains were calculated and the percent of each type was determined, and the data are presented in Table 5.3. The average grain sizes in Table 5.3 are considered as the mean intercept length in the horizontal direction (Figure 4.11b) and calculations were performed on EBSD color-coded maps with a grain tolerance angle of 15°. From the data shown in Table 5.3, the fine grain size averaged between 3.3 and 3.6 µm for all alloys and orientations, while the average grain sizes for the coarse grains were between 12.6 and 17 µm. The percent occupied by the fine grains ranged between 68 and 93 percent with no specific trend. The D orientation, which showed the lowest toughness in terms of ductile to brittle transition temperature, did not distinctively show a lower percent of fine grains than the other orientations for all alloys. However, the Base steel displayed relatively higher percent of fine grains compared to the other alloys.

Figure 5.21 Representative grain boundary map developed from EBSD data for the 15T steel in the T direction for grain boundary misorientation equal to or higher than 15°.
Figure 5.22  Grain size distributions for the 15T alloy represented by the percent of total intercept lengths versus intercept length for the (a) longitudinal (0°), (b) transverse (90°), and (c) diagonal (45°) planes identified in Figure 4.7.

Figure 5.23  Grain size distributions for the Base alloy represented by the percent of total intercept lengths versus intercept length for the (a) longitudinal (0°), (b) transverse (90°), and (c) diagonal (45°) planes identified in Figure 4.7.

Figure 5.24  Grain size distributions for the 0Ni alloy represented by the percent of total intercept lengths versus intercept length for the (a) longitudinal (0°), (b) transverse (90°), and (c) diagonal (45°) planes identified in Figure 4.7.
Figure 5.25 Grain size distributions for the 0.3Ni alloy represented by the percent of total intercept lengths versus intercept length for the (a) longitudinal (0°), (b) transverse (90°), and (c) diagonal (45°) planes identified in Figure 4.7.

Table 5.3 – Average Grain Sizes, as the Mean Intercept Length Parallel to the Normal Plane, for the Fine and Coarse Grains Calculated for the Four Alloys in the Longitudinal, Transverse, and Diagonal Orientations

<table>
<thead>
<tr>
<th>Steel</th>
<th>Direction</th>
<th>Fine Grain Size (µm)</th>
<th>Percent of Fine Grains (%)</th>
<th>Coarse Grain Size (µm)</th>
<th>Percent of Coarse Grains (%)</th>
<th>Average Grain Size (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>15T</td>
<td>L</td>
<td>3.3</td>
<td>76.3</td>
<td>16.2</td>
<td>23.7</td>
<td>4.1</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>3.4</td>
<td>79.9</td>
<td>14.6</td>
<td>20.1</td>
<td>4.1</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>3.5</td>
<td>87.2</td>
<td>15.1</td>
<td>12.8</td>
<td>3.9</td>
</tr>
<tr>
<td>Base</td>
<td>L</td>
<td>3.3</td>
<td>92.5</td>
<td>12.6</td>
<td>7.5</td>
<td>3.5</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>3.6</td>
<td>80.4</td>
<td>15.1</td>
<td>19.6</td>
<td>4.2</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>3.5</td>
<td>86.1</td>
<td>14.4</td>
<td>13.9</td>
<td>3.9</td>
</tr>
<tr>
<td>0Ni</td>
<td>L</td>
<td>3.5</td>
<td>68</td>
<td>17</td>
<td>32</td>
<td>4.7</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>3.6</td>
<td>75.2</td>
<td>15.3</td>
<td>24.8</td>
<td>4.5</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>3.6</td>
<td>75.5</td>
<td>16</td>
<td>24.5</td>
<td>4.4</td>
</tr>
<tr>
<td>0.3Ni</td>
<td>L</td>
<td>3.6</td>
<td>78</td>
<td>16.8</td>
<td>22</td>
<td>4.3</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>3.5</td>
<td>74</td>
<td>14.9</td>
<td>26</td>
<td>4.3</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>3.4</td>
<td>85.6</td>
<td>13.4</td>
<td>14.4</td>
<td>3.8</td>
</tr>
</tbody>
</table>

5.3.2 Volume Fraction of Microconstituents and Grain Boundary Densities

The volume fractions of secondary microconstituents were measured from EBSD IQ maps for the four steels in the L, T, and D planes. Figure 5.26 displays representative EBSD images for the Base alloy in the transverse plane, showing the method used to quantify the
volume fraction of microconstituents. The microconstituents, except for austenite, appear as dark grey or black regions in the IQ map shown in Figure 5.26a due to their low IQ pattern. Austenite can be easily indexed and has a higher IQ value compared to martensite and cementite as shown by the red phase in Figure 5.26b. The difference in the quality of the indexed pattern was used to quantify the martensite and cementite volume fractions. The dark regions in Figure 5.26a were highlighted (blue) as shown in Figure 5.26c. Then, the volume fractions of secondary microconstituents were calculated by summing the volume fraction of austenite from Figure 5.26b with the other constituents highlighted in Figure 5.26c. The volume fractions of secondary microconstituents were calculated for each steel in each view direction from micrographs similar to that shown in Figure 5.26 and are summarized in Table 5.4. For each alloy, an average of two scans with an area of 0.1 x 0.1 μm was used for each view direction. The volume fractions of secondary microconstituents are slightly higher for the 0Ni and 0.3Ni than the 15T and Base steels and are similar with respect to orientation for all alloys.

The densities of low (2°-15°) and high (greater than 15°) angle boundaries were also calculated for each plate and orientation from EBSD data as the total boundary length per square area and are presented in Table 5.4. The high angle boundary density can be used as another measure (in addition to grain size) to assess crack propagation resistance. Increasing the density of high angle boundaries increases the resistance to crack propagation [65]. The low angle boundary density can qualitatively relate to the amount of substructure and dislocation density present in each alloy. Again, no significant difference in the boundary densities was found with respect to orientation for any of the alloys.
Figure 5.26  Example for the secondary microconstituents quantification. (a) IQ map for the Base alloy in the T plane, (b) IQ map in (a) overlaid with a phase map (austenite is red), and (c) IQ map in (a) highlighting the dark microconstituents (blue).
Table 5.4 – Volume Fractions of Secondary Microconstituents, and High and Low Angle Boundary Densities Calculated for the Four Alloys in the Longitudinal, Transverse, and Diagonal Orientations. The Uncertainty is the Range.

<table>
<thead>
<tr>
<th>Steel</th>
<th>Direction</th>
<th>VF of Secondary Microconstituents (%)</th>
<th>HAB Density (µm⁻¹)</th>
<th>LAB Density (µm⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>15T</td>
<td>L</td>
<td>1.65 ± 0.25</td>
<td>0.85 ± 0.01</td>
<td>0.75 ± 0.13</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>2.15 ± 0.10</td>
<td>0.71 ± 0.01</td>
<td>0.90 ± 0.05</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>1.65 ± 0.65</td>
<td>0.84 ± 0.01</td>
<td>0.60 ± 0.06</td>
</tr>
<tr>
<td>Base</td>
<td>L</td>
<td>2.75 ± 0.25</td>
<td>0.94 ± 0.02</td>
<td>0.86 ± 0.01</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>1.95 ± 0.35</td>
<td>0.84 ± 0.01</td>
<td>0.59 ± 0.01</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>2.05 ± 1.05</td>
<td>0.82 ± 0.01</td>
<td>0.67 ± 0.04</td>
</tr>
<tr>
<td>0Ni</td>
<td>L</td>
<td>3.65 ± 0.35</td>
<td>0.58 ± 0.02</td>
<td>0.78 ± 0.04</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>3.45 ± 0.55</td>
<td>0.75 ± 0.01</td>
<td>0.61 ± 0.02</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>3.70 ± 0.50</td>
<td>0.72 ± 0.04</td>
<td>0.91 ± 0.04</td>
</tr>
<tr>
<td>0.3Ni</td>
<td>L</td>
<td>4.70 ± 0.40</td>
<td>0.85 ± 0.07</td>
<td>0.80 ± 0.01</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>8.90 ± 1.85</td>
<td>0.80 ± 0.03</td>
<td>1.15 ± 0.08</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>3.25 ± 0.50</td>
<td>0.85 ± 0.15</td>
<td>0.79 ± 0.02</td>
</tr>
</tbody>
</table>

5.4 Crystallographic Texture

The crystallographic textures were determined using EDAX OIM™ Data Analysis software from EBSD collected data. For each alloy, two scans with an area of 1 x 0.8 mm and 2 µm step size were performed on the normal plane of the sample to capture the macrotexture of each plate. Orientation distribution function (ODF) sections at φ₂ = 45°, where the main ideal orientations of rolled materials are found, were developed for the four alloys at half thickness of the plate and the results are shown in Figure 5.27. Full ODF section for φ₂ from 0° to 90° at 5° intervals are presented in Appendix B for the four steels. The texture is similar for all reported alloys, which extends from the transformed copper in the area around the {113}<110> and {112}<110> to the transformed brass in the vicinity of the {554}<225> and {332}<113>. The intermediate components are products of the transformed Goss and transformed S component [31]. Some intensities are also found around the rotated cube {001}<110>. The developed texture resembles the transformation texture resulting from deformed austenite, described by Jonas [31]. Note there was no evidence of cube {001}<010> and rotated Goss {110}<110>
components, reported to be transformation products of recrystallized austenite [31]. The maximum intensity is around 4.5 times random for the studied steels. As stated earlier, the developed textures and sharpness of the textures displayed in Figure 5.27 are comparable despite the different processing routes. Figure 5.28 shows ODF sections at $\varphi_2 = 45^\circ$ for the 15T alloy in the half thickness, quarter thickness, and near the surface of the plate. A texture gradient is clearly observed with a maximum intensity of 4.5 at the half thickness, 3.2 at quarter thickness, and 1.3 near the surface. Similar observations were found for the other three alloys and are presented in Appendix B.

The intensities measured along the RD (at $\varphi_1 = 0^\circ$ in Figure 5.27) and TD (at $\varphi_1 = 90^\circ$ in Figure 5.27) fibers for the four steels are plotted in Figure 5.29. The main texture components are displayed in Figure 5.29 in Miller indices, where the \{hkl\} plane of the crystal is parallel to the normal plane of the sample and the <uvw> direction of the crystal is parallel to the rolling direction of the sample. The intensities of the RD and TD fibers are almost similar for the four alloys. For the 15T alloy, the intensities around the \{112\}<110> and \{113\}<110> components in the RD fibers are relatively lower compared to the other alloys, while the intensity in the region between the \{111\}<112> and \{332\}<113> components in the TD fibers are higher than the rest. The 0.3Ni steel has a lower intensity at the rotated cube \{001\}<110> component.

It is generally accepted that brittle fracture in bcc materials occurs on \{001\} planes [66, 67]. Therefore, for each steel and test orientation, the fraction of grains with \{001\} planes parallel to the cleavage fracture plane for each direction was calculated from the EBSD data using a tolerance of +/- 5°. For each alloy, the fraction of \{001\} planes was calculated from a scan area of 1 x 0.8 mm in the normal plane (same scans used to obtain texture measurements) and an average of two scans were used. These data were correlated with transition temperatures.
Figure 5.27  Plots of $\varphi_2 = 45^\circ$ ODF sections representing crystallographic texture: (a) 15T, (b) Base, (c) 0Ni, and (d) 0.3Ni steels. (e) Key for the ODF’s: the numbers in the legend correspond to orientation intensities. (f) Key showing the position of the main texture components along with the RD, TD, and ND fibers.
Figure 5.28  Plots of $\varphi_2 = 45^\circ$ ODF sections representing crystallographic texture for the 15T steel at: (a) half thickness, (b) quarter thickness, and (c) near the surface. (d) Key for the ODF’s: the numbers in the legend correspond to orientation intensities.
and the results are summarized in Figure 5.30. Figure 5.30a summarizes the fraction of \{001\} planes parallel to the primary CVN fracture planes for all steels and orientations, measured on polished EBSD specimen. For all plates, the fraction of grains having their \{001\} plane parallel to the fracture plane is higher in the D direction. Figure 5.30b correlates DBTT (50% shear transition temperature from Table 5.3) with test orientation for each alloy and shows similar systematic variations in DBTT with orientation, all of which show the highest transition temperature for the D direction. Although the anisotropy in the DBTT (Figure 5.30b) is higher in the Base and 0Ni steels than the 15T and 0.3Ni steels, the fraction of cleavage planes in the D (°45) orientation is almost the same for all alloys. Also, the fraction of \{001\} planes and DBTT are slightly lower in the L direction than the T direction.

![Intensity plots for RD (a) and TD (b) fibers for the four alloys.](image-url)
Figure 5.30 (a) Fraction of grains with the \{001\} cleavage planes parallel to the fracture planes of CVN specimen and (b) corresponding ductile to brittle transition temperatures (DBTT) based on 50 pct shear, for the four plates in the L (0°), T (90°), and D (45°) directions for CVN tests.

5.5 Modified CVN Impact Results

To further study the splitting behavior and effects on impact toughness, a modified impact test (MCVN) specimen with side grooves or slits was designed and machined for the 15T alloy and 0Ni alloy in the longitudinal, transverse, and diagonal direction, as described in Section 4.5. The side slits were designed to increase lateral constraint and thus increase the tensile stress in the direction parallel to the rolling plane normal. With the increase in induced stress, splitting was expected to increase, providing a further aspect to study delamination. The modified impact samples were tested at several temperatures to develop the ductile to brittle transition temperatures. The resulting impact data in the form of energy absorbed and percent shear are shown in Figure 5.31 for the 15T alloy and in Figure 5.32 for the 0Ni. In each figure the corresponding conventional CVN impact data are also shown. Consistent with modifications to the local stress state at the notch tip, the absorbed energies are much lower for the MCVN samples for both alloys at any specific temperature (above the lower shelf) and the DBTTs are
also higher for the modified geometries. Increases of 30 to 40 °C for the L and T orientation and 15 to 25 °C in the D orientation were observed in the DBTT, and the upper shelf energies decreased by half, as displayed in Table 5.5.

Figure 5.33 shows representative fracture surfaces of the MCVN impact specimen for the 0Ni plate which exhibited 100 percent ductile fracture. At room temperature (Figure 5.33a), the samples fractured completely as opposed to the bent fracture observed in the conventional impact samples reported earlier (Figure 5.12a). In addition, splits are present in all samples exhibiting ductile fracture and the severity and number of splits increases with decreasing temperature (Figures 5.33a-c). It was also noted that the absorbed energies decreased as the number and severity of splits increased. Complete CVN and MCVN fracture surface macrographs are presented in Appendices C and D, respectively.

5.6 Heat Treatment

The 0Ni plate was subjected to heat treatment to further investigate the sources of toughness anisotropy and delamination. Rectangular samples were rough machined for Charpy impact and tensile testing in the L, T, and D directions and heat treated according to the schedule outlined in Section 4.2. Microstructure and texture characterization, and tensile and Charpy-V impact testing results are presented here for the 0Ni heat treated condition and compared to the 0Ni as-received plate results presented above.

5.6.1 Microstructure

Figures 5.34a and 5.34b display light optical and SEM micrographs, respectively, for the microstructure of the heat treated 0Ni plate taken at the mid-thickness in the transverse orientation. The heat treated plate microstructure was mostly ferritic and the grains were more equiaxed, because during heat treatment, ferrite was formed from equiaxed austenite grains.
Figure 5.31  Charpy V-notch curves for the 15T steel for the conventional CVN specimens and MCVN specimens in the longitudinal direction as (a) absorbed energy and (b) percent shear, in the transverse direction as (c) absorbed energy and (d) percent shear, and in the diagonal direction as (e) absorbed energy and (f) percent shear.
Figure 5.32  Charpy V-notch curves for the 0Ni steel for the conventional CVN specimens and MCVN specimens in the longitudinal direction as (a) absorbed energy and (b) percent shear, in the transverse direction as (c) absorbed energy and (d) percent shear, and in the diagonal direction as (e) absorbed energy and (f) percent shear.
Table 5.5 – Summary of the Upper Shelf Energy and Transition Temperature Determined as the 50 percent Shear Transition Temperature for the Standard and Modified Charpy V-notch tests for the 15T and 0Ni Steels in the L, T, and D directions

<table>
<thead>
<tr>
<th>Steel</th>
<th>Direction</th>
<th>CVN USE (J)</th>
<th>CVN 50% Shear (°C)</th>
<th>MCVN USE (J)</th>
<th>MCVN 50% Shear (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>15T</td>
<td>L</td>
<td>450</td>
<td>-115</td>
<td>250</td>
<td>-75</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>450</td>
<td>-112</td>
<td>220</td>
<td>-70</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>405</td>
<td>-85</td>
<td>230</td>
<td>-60</td>
</tr>
<tr>
<td>0Ni</td>
<td>L</td>
<td>440</td>
<td>-93</td>
<td>225</td>
<td>-65</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>450</td>
<td>-90</td>
<td>230</td>
<td>-55</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>440</td>
<td>-50</td>
<td>220</td>
<td>-35</td>
</tr>
</tbody>
</table>

Figure 5.33 Macrographs of fracture surfaces of the modified impact samples for the 0Ni alloy in the L direction tested at: (a) 20 °C, (b) 0 °C, and (c) -20 °C.

Instead of pancaked grains as in the case for the as-received plate (Figures 5.1c and 5.2c). However, the ferrite grains appear larger in the heat treated steel. Figure 5.35 shows EBSD image quality maps for the 0Ni heat treated plate, taken in the mid-thickness of the plates in the T orientation. The blue lines shown in Figure 5.35b are HABs with misorientation greater than 15°. The red and green lines are LABs with misorientations between 2-5° and 5-15°, respectively. The banded structure observed in the microstructure of the four alloys was not found in the heat treated steel. Although the microstructure is equiaxed compared to the flattened microstructure for the as-received condition (Figure 5.14c), the grains still have irregular boundaries. Also, as evident in Figure 5.35b, the amount of low angle boundaries is much less
than in the as-received condition, which indicates that the heat treated plate has less developed substructure. Additionally, MA and RA islands are present along ferrite grain boundaries. The densities of low and high angle boundaries in the L, T, and D planes are summarized in Table 5.6 along with the volume fraction of secondary microconstituents using the method described in Section 5.3.2. Complete IQ maps in the L, T, and D planes are presented in Appendix A.

![Figure 5.34 Microstructure of the 0Ni heat treated steel taken in the mid thickness in the transverse direction (2 pct nital etch): (a) light optical micrograph and (b) SEM image.](image)

Average grain size measurements, grain aspect ratios and grain size distributions were calculated from EBSD collected data for the heat treated 0Ni alloy. Average grain size measurements in each plane (Figure 4.7) along with grain aspect ratios for the 0Ni heat treated condition are displayed in Figures 5.36. Similar to the as-received plate (Figure 5.18c), the grain sizes for the heat treated plate were comparable in each direction, indicating no anisotropy with respect to direction. The average grain sizes for the heat treated condition were slightly higher, around 4.0 µm compared to 3.5 µm for the as-received condition. The grain aspect ratio values for the heat treated condition, shown in Figure 5.36, were lower than those for the as-received
condition (Figure 5.18c) and close to each other for all orientations, ranging between 1.0 and 1.1, and indicating that the grains for the heat treated plate are more equiaxed compared to the pancaked structure of the as-received condition.

Figure 5.35  Image quality maps for the 0Ni heat treated steel in the transverse direction: (a) low magnification and (b) high magnification overlaid with grain boundary segments. Red and green boundaries are low angle boundaries (2°-15°) and blue boundaries are high angle boundaries (greater than 15°).

Table 5.6 – Volume Fractions of Secondary Microconstituents, and High and Low Angle Boundaries Densities Calculated for the As-Received and Heat Treated 0Ni Steel in the Longitudinal, Transverse, and Diagonal Orientations. The Uncertainty is the Range.

<table>
<thead>
<tr>
<th>Steel</th>
<th>Direction</th>
<th>VF of Secondary Microconstituents (%)</th>
<th>HAB Density (µm⁻¹)</th>
<th>LAB Density (µm⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-Received</td>
<td>L</td>
<td>3.65 ± 0.35</td>
<td>0.58 ± 0.02</td>
<td>0.78 ± 0.04</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>3.45 ± 0.55</td>
<td>0.75 ± 0.01</td>
<td>0.61 ± 0.02</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>3.70 ± 0.50</td>
<td>0.72 ± 0.04</td>
<td>0.91 ± 0.04</td>
</tr>
<tr>
<td>Heat Treated</td>
<td>L</td>
<td>5.75 ± 0.55</td>
<td>0.70 ± 0.04</td>
<td>0.16 ± 0.03</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>4.05 ± 0.15</td>
<td>0.63 ± 0.01</td>
<td>0.23 ± 0.04</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>4.05 ± 0.50</td>
<td>0.55 ± 0.07</td>
<td>0.22 ± 0.02</td>
</tr>
</tbody>
</table>
Figure 5.36  Average grain sizes and grain aspect ratios measured from EBSD data on the longitudinal (0°), diagonal (45°), and transverse (90°) planes identified in Figure 4.7 for the 0Ni heat treated alloy.

Grain size distribution calculations were also performed on the heat treated plate based on the procedure described in Section 5.3.1. Figure 5.37 displays the percent of total intercept lengths versus intercept length for each orientation for the heat treated 0Ni alloy. The average grain size, in terms of lineal intercept lengths, for the fine and coarse grains and the percent of each type are presented in Table 5.7. From the data shown in Tables 5.3 and 5.7, the fine grain size averaged around 3.6 µm and 4.2 µm for the as-received and heat treated conditions, respectively, while the average grain sizes for the coarse grains were around 16 µm and 14 µm for the as-received and heat treated conditions, respectively. The percent occupied by the fine grains ranged between 68 and 80 percent with no specific trend. Also, regions of large elongated grains, which sometimes reached up to 50 µm in length, were not present after heat treatment as most of the coarse grains were between 10 µm and 25 µm for the heat treated plate (Figures 5.24 and 5.37).
Figure 5.37  Grain size distributions for the heat treated 0Ni alloy represented by the percent of total intercept lengths versus intercept length for the (a) longitudinal (0°), (b) transverse (90°), and (c) diagonal (45°) planes identified in Figure 4.7.

Table 5.7 – Average Grain Sizes, as the Mean Intercept Length Parallel to the Normal Plane, for the Fine and Coarse Grains Calculated for the As-Received and Heat Treated 0Ni Alloy in the Longitudinal, Transverse, and Diagonal Orientations

<table>
<thead>
<tr>
<th>Steel</th>
<th>Direction</th>
<th>Fine Grain Size (µm)</th>
<th>Percent of Fine Grains (%)</th>
<th>Coarse Grain Size (µm)</th>
<th>Percent of Fine Grains (%)</th>
<th>Average Grain Size (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-Received</td>
<td>L</td>
<td>3.5</td>
<td>68</td>
<td>17</td>
<td>32</td>
<td>4.7</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>3.6</td>
<td>75.2</td>
<td>15.3</td>
<td>24.8</td>
<td>4.5</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>3.6</td>
<td>75.5</td>
<td>16</td>
<td>24.5</td>
<td>4.4</td>
</tr>
<tr>
<td>Heat Treated</td>
<td>L</td>
<td>4.2</td>
<td>80.9</td>
<td>13.9</td>
<td>19.1</td>
<td>4.88</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>4.3</td>
<td>79.6</td>
<td>13.0</td>
<td>20.4</td>
<td>5.19</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>4.2</td>
<td>72.9</td>
<td>14.2</td>
<td>27.1</td>
<td>5.26</td>
</tr>
</tbody>
</table>

5.6.2  Tensile Properties

Representative engineering stress-strain curves in the L, T, and D orientations for the heat treated 0Ni steel are shown in Figure 5.38. Yield strengths were calculated using 0.2 pct offset and the total strain to failure was measured in a 50.8 mm (2 inch) gauge length with a Shepic extensometer. The yield and tensile strengths were similar for all orientations for the heat treated plate as shown in Figure 5.38, indicating that the anisotropy in strength was eliminated, whereas the YS and TS were higher in the T direction for the as-received steel (Figure 5.6). However, a significant drop in strength, specifically in the yield strength, was associated with the applied
heat treatment schedule, even though hardness values were close to the as-received condition (200 HV). Also, the total elongation increased by around 5% after heat treatment. Tensile properties for an average of two samples per direction are summarized for both the as-received and heat treated 0Ni plates in Table 5.8.

![Engineering stress-strain curves for the 0Ni heat treated steel tested in the three orientations. L designates longitudinal direction, T designates transverse direction, and D designates diagonal direction.](image)

### Figure 5.38

Engineering stress-strain curves for the 0Ni heat treated steel tested in the three orientations. L designates longitudinal direction, T designates transverse direction, and D designates diagonal direction.

### Table 5.8 – Average Tensile Test Results for the As-Received and Heat Treated 0Ni Alloy in the L, T, and D Directions, Based on Two Samples for each Direction

<table>
<thead>
<tr>
<th>Steel</th>
<th>Direction</th>
<th>YS (0.2 pct offset) (MPa)</th>
<th>TS (MPa)</th>
<th>YS/TS Ratio</th>
<th>pct Elongation (in 50.8 mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-Received</td>
<td>L</td>
<td>476</td>
<td>582</td>
<td>0.82</td>
<td>23.8</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>490</td>
<td>604</td>
<td>0.81</td>
<td>21.9</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>469</td>
<td>569</td>
<td>0.82</td>
<td>25.4</td>
</tr>
<tr>
<td>Heat Treated</td>
<td>L</td>
<td>365</td>
<td>536</td>
<td>0.68</td>
<td>28.5</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>383</td>
<td>545</td>
<td>0.70</td>
<td>28.0</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>386</td>
<td>541</td>
<td>0.71</td>
<td>29.3</td>
</tr>
</tbody>
</table>
5.6.3 Impact Properties

Conventional and modified Charpy V-notch impact testing were conducted to develop ductile to brittle transition temperature (DBTT) curves for the heat treated 0Ni steel in the longitudinal, transverse, and diagonal directions to compare the data to the as-received condition. The results for the conventional CVN testing are shown in Figure 5.39 for energy absorbed versus temperature data and percent shear versus temperature. The tests were performed on an MPM Pendulum 9000 test machine, with a capacity of up to 950 J (700 ft-lb) at NIST. Each steel and sample orientation exhibited a well-defined transition from ductile to brittle behavior. The DBTT in the D direction is closer to the L and T curves compared to the other four commercially produced alloys (Figures 5.8-5.11). Also, the absorbed energy and percent shear curves showed a similar behavior, specifically at the upper shelf region. However, for the other four investigated alloys, the absorbed energies decreased as the temperature decreased in the upper shelf region while the samples still exhibited 100 percent ductility. The difference in behavior for the commercially produced alloys between the energy absorbed and percent shear curves is because splitting was observed in the fracture surfaces of samples tested at lower temperatures in the upper shelf region. However, none of the broken samples for the heat treated plate showed splitting.

Transition temperatures were determined from both the energy absorbed and percent shear data. The results for the heat treated alloy for the 150 J transition temperatures and 50 percent shear transition temperatures from Figure 5.39 are summarized in Table 5.9 along with the results for the as-received plate presented previously (Section 5.3). There is no significant anisotropy in the upper shelf energy between the different directions or before and after heat treatment. By comparing the DBTTs between the as-received and heat treated conditions, the
DBTTs are lowered by around 10 °C after heat treatment in the L and T orientations and around 38 °C in the D orientation, reducing the anisotropy significantly.

Although the fracture surfaces of the conventional CVN samples for the heat treated plate did not exhibit splitting, almost all of the tested samples at the upper shelf region did not break and a definitive conclusion regarding the occurrence of delaminated fracture, cannot be drawn. Therefore, modified CVN impact samples were prepared for the heat treated steel and tested to assess delamination. These results are compared to the as-received MCVN data presented in Section 5.5. The MCVN impact curves are shown in Figure 5.40 for the heat treated alloy along with the conventional CVN impact data. Similar to the as-received 0Ni plate (Figure 5.32), the absorbed energies are much lower for the MCVN samples at any specific temperature and the DBTTs are also higher for the modified geometries. Table 5.10 displays the upper shelf energy and DBTTs determined as the 50 percent shear transition temperature for the modified CVN tests for the as-received and heat treated 0Ni Steels in the L, T, and D directions. From the data in Tables 5.9 and 5.10, an increase of 30 to 40 °C is observed in the DBTT for both conditions in all orientations except for the as-received condition in the D orientation, where the DBTT increased by only 15 °C. The upper shelf energies decreased by one half and one third for the as-received and heat treated conditions, respectively.

Figure 5.41 shows representative fracture surfaces of the MCVN impact specimen in the L direction which exhibited 100 percent ductile fracture for heat treated 0Ni plate. For the as-received samples, splits are present in all samples exhibiting ductile fracture and the severity and number of splits increased with decreasing temperature (Figures 5.33a-5.33c). It was also noted that the absorbed energies decreased as the number and severity of splits increased. However, splits were not observed in any of the fracture surfaces of the heat treated samples.
Figure 5.39 Standard Charpy V-Notch curves developed for the 0Ni heat treated (HT) samples in the longitudinal direction as (a) absorbed energy and (b) percent shear, in the transverse direction as (c) absorbed energy and (d) percent shear, and in the diagonal direction as (e) absorbed energy and (f) percent shear.
Figure 5.40 Impact curves for the 0Ni heat treated (HT) steel for the standard CVN specimens and MCVN specimens in the longitudinal direction as (a) absorbed energy and (b) percent shear, in the transverse direction as (c) absorbed energy and (d) percent shear, and in the diagonal direction as (e) absorbed energy and (f) percent shear.
The difference in the upper shelf energies between the two conditions (Table 5.10) is attributed to the presence of splitting in the as-received samples, reducing the USE by ~80 J. Complete CVN and MCVN fracture surface macrographs can be found in Appendices C and D, respectively.

Table 5.9 – Summary of the Upper Shelf Energy and Transition Temperatures Determined as 150 J Transition Temperatures and the 50 percent Shear Transition Temperature for the As-Received and Heat Treated 0Ni Steel in the L, T, and D Directions

<table>
<thead>
<tr>
<th>Steel</th>
<th>Direction</th>
<th>USE (J)</th>
<th>150 J (°C)</th>
<th>50% Shear (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-Received</td>
<td>L</td>
<td>440</td>
<td>-89</td>
<td>-93</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>450</td>
<td>-89</td>
<td>-90</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>440</td>
<td>-45</td>
<td>-50</td>
</tr>
<tr>
<td>Heat Treated</td>
<td>L</td>
<td>450</td>
<td>-96</td>
<td>-98</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>450</td>
<td>-108</td>
<td>-104</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>450</td>
<td>-88</td>
<td>-88</td>
</tr>
</tbody>
</table>

Table 5.10 – Summary of the Upper Shelf Energy and Transition Temperature Determined as the 50 percent Shear Transition Temperature for the Modified Charpy V-notch Test for the As-Received and Heat Treated 0Ni Steels in the L, T, and D Directions

<table>
<thead>
<tr>
<th>Steel</th>
<th>Direction</th>
<th>USE (J)</th>
<th>50% Shear (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-Received</td>
<td>L</td>
<td>225</td>
<td>-65</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>230</td>
<td>-55</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>220</td>
<td>-35</td>
</tr>
<tr>
<td>Heat Treated</td>
<td>L</td>
<td>310</td>
<td>-65</td>
</tr>
<tr>
<td></td>
<td>T</td>
<td>300</td>
<td>-65</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>300</td>
<td>-55</td>
</tr>
</tbody>
</table>

5.6.4 Texture

The crystallographic texture was measured by EDAX OIM™ Data Analysis Software from EBSD collected data. Texture measurements were taken for the as-received and heat treated 0Ni alloy at the half thickness of the plates. Figure 5.42a shows an orientation distribution function (ODF) section at \( \phi_2 = 45^\circ \) of the heat treated plate. The texture is to some extent a
weakened form of the as-received plate discussed previously (Section 5.4) and shown in Figure 5.42b, with a maximum of 3.2 times random compared to 4.8 times random for the as-received plate. Figure 5.43a shows the intensities measured along the RD fiber (the left hand side of the ODF section at $\varphi_2 = 45^\circ$) and Figure 5.43b shows the TD fiber (the right hand side of the ODF section $\varphi_2 = 45^\circ$) for the as-received and heat treated 0Ni steel. There is a slight decrease in the intensity of $\{001\}<110>$ component. However, the intensities around the $\{112\}<110>$ and $\{113\}<110>$ components in the RD fiber decreased by half after heat treatment (Figure 5.43a). Also, the TD fiber component intensities are weaker for the heat treated condition (Figure 5.43b).

![Macrographs of fracture surfaces of the modified impact samples for the heat treated 0Ni alloy in the L direction tested at: (a) 20 °C, (b) 0 °C, and (c) -20 °C.](image)

Figure 5.41 Macrographs of fracture surfaces of the modified impact samples for the heat treated 0Ni alloy in the L direction tested at: (a) 20 °C, (b) 0 °C, and (c) -20 °C.

Figure 5.44a shows the fraction of $\{001\}$ planes parallel to the primary CVN fracture planes for the as-received and heat treated 0Ni alloy in the three orientations, measured on polished EBSD specimens at half thickness. For both conditions, the fraction of $\{001\}$ planes is higher near the D direction. Figure 5.44b correlates DBTT (50 percent shear) with test orientation for the 0Ni alloy for both conditions. Although the anisotropy in the DBTT has weakened after heat treatment (Figure 5.44b), the fraction of cleavage planes in the D ($^\circ45$) orientation still remains higher. The observed high density of $\{001\}$ planes in the D orientation after heat treatment, suggests that other orientations and factors should be considered in order to
understand the cause of toughness anisotropy. A model to relate the toughness anisotropy is proposed and discussed in Chapter 6.

Figure 5.42 Plots of $\varphi_2 = 45^\circ$ ODF sections representing crystallographic texture for the 0Ni steel for the: (a) heat treated and (b) as-received plates. (c) Key for the ODF’s: the numbers in the legend correspond to orientation intensities.

Figure 5.43 Intensities along the (a) RD ($<110>$//RD) and (b) TD ($<110>$//TD) fibers for the as-received and heat treated 0Ni steels.
Figure 5.44  (a) Fraction of grains with the \{001\} cleavage planes parallel to the fracture planes of Charpy impact specimen and (b) corresponding ductile to brittle transition temperatures (DBTT) based on 50 percent shear measured on standard CVN samples, for the as-received and heat treated 0Ni plates in the L (0°), T (90°), and D (45°) directions for Charpy impact tests.
CHAPTER 6: Discussion

This chapter reviews key results presented in Chapter 5 and provides additional interpretation of these results. The main objective of this project is to understand the influence of microstructure, texture, and fracture behavior on toughness anisotropy and delamination observed in pipeline steels. Four X70 grade commercially produced pipeline steels, with different chemistries (Table 4.1) and processing routes were investigated in this project. Additionally, one alloy (0Ni steel) was thermally processed to change the microstructure and texture for further assessment. The causes of toughness anisotropy and splitting during fracture observed in Charpy V-notch broken specimen are discussed here.

6.1 Review of Mechanical Properties

Tensile test properties are summarized in Table 5.1 for the four as-received alloys and in Table 5.8 for the 0Ni heat treated alloy. In order to quantify the average strength of the investigated plates $\sigma_{avg}$ is defined, which is the average of the yield strengths measured along the different directions:

$$\sigma_{avg} = \frac{2}{\pi} \int_{0}^{\pi/2} \sigma(\theta) d\theta = \frac{1}{4}(\sigma_0 + 2\sigma_{45} + \sigma_{90})$$

(6.1)

where $\theta$ is the angle to the rolling direction. The average yield strengths for the 15T, Base, as-received 0Ni, 0.3Ni, and heat treated 0Ni steels are 529, 487, 476, 508, and 382 MPa, respectively. An extended Hall-Petch relation for predicting the yield strength ($\sigma_y$) can be written as [68]:

$$\sigma_y = \sigma_i + \sigma_s + \sigma_p + \sigma_d + \sigma_{ss} + \sigma_t + k_y d^{-1/2}$$

(6.2)
where $\sigma_i$ is the lattice friction stress, $\sigma_s$ is solid solution strengthening, $\sigma_p$ is precipitation strengthening, $\sigma_d$ is dislocation strengthening, $\sigma_{ss}$ is sub-grain size strengthening, $\sigma_t$ is crystallographic texture strengthening, $k_y$ is the locking parameter, and $d$ is the grain size. The differences in grain sizes alone do not account for the differences observed in yield strengths. The differences in yield strength between the four as-received alloys are due to a combination of the strengthening parameters, since they have different chemical compositions and processing histories. The lower strength for the 0Ni heat treated plate is mainly attributed to the loss of precipitation strengthening and decrease in dislocation density and substructure after heat treatment since the microstructure was mainly composed of polygonal ferrite.

Standard impact test results for the studied plates were presented in Sections 5.2.2 and 5.6.3. The upper shelf energies were similar for all investigated alloys in the three orientations, around 450 J. For all four alloys in the as-received conditions, the ductile to brittle transition temperatures (DBTT) were higher in the D direction and close to each other in the L and T directions. The anisotropy in the DBTT was significantly reduced in the 0Ni steel after heat treatment. The anisotropy in toughness is considered further in Section 6.4. By comparing the average 50 percent shear DBTTs of the L and T orientations, the DBTTs for the 15T and Base alloys are lower than the 0Ni and 0.3Ni alloys by around 20 °C. Also, the DBTTs decreased in the L and T directions by approximately 10 °C after heat treatment for the 0Ni plate. The DBTT can be estimated by an equation similar to Equation 6.2 as the following [68]:

\[
T = T_o - kd^{1/2}
\]  
(6.3)

where $T$ is the transition temperature, $T_o$ is a function of $\sigma_s$, $\sigma_p$, $\sigma_d$, and $\sigma_i$, and $k$ is a constant. Typical values of $k$ used for low carbon ferritic or bainitic steels are between 11.5 and 15 °C/mm$^{-1/2}$ [68]. Based on the grain sizes summarized in Figure 5.20 for the four alloys in the
as-received conditions, calculated transition temperatures for the 15T and Base alloys would be lower by around 9 °C than for the other two alloys. Therefore, since the DBTTs for the 15T and Base alloys are lower by an amount (~20 °C) greater than predicted based only on grain size, the observed differences in DBTTs must also reflect other microstructural differences. An expanded version of Equation 6.3 to account for crystallographic texture is developed and discussed in Section 6.4.2.

6.2 Review of Microstructure

The microstructures of the four alloys in the as-received conditions were mixed, consisting mainly of fine acicular ferrite grains with some polygonal ferrite grains and coarse bainite regions. Also, microconstituents were found dispersed in the matrix and between grains. The secondary microconstituents contained martensite, austenite, cementite, or a mixture. Although the four steels have different chemical compositions and were produced in different rolling mills, the microstructures, crystallographic textures, and mechanical properties were similar. The C, Mn, Si, and Al contents were within the same range for the four alloys. The alloys chemical compositions differed in the amounts of Ni, Cr, Mo, Ti, Nb, V, and Cu additions. The most significant difference in the processing between the four plates is that the 0Ni and 0.3Ni alloys were rolled from thin slabs (85 mm in thickness), while the 15T and Base plates had a starting slab thickness of 200 mm. Therefore, a higher total reduction was imposed to the 15T and Base plates. Details on the processing history of the four alloys (rough rolling, finish rolling, cooling rate, coiling temperatures, etc.) were proprietary and thus a complete understanding of the effects of processing parameters cannot be achieved. Nonetheless, based on the final microstructures of the four alloys, microstructural development during rolling is interpreted as the following. Recrystallized austenite grains were refined during rough rolling above the
recrystallization stop temperature by repeated reduction and recrystallization. Then, the austenite grains were flattened or pancaked during finish rolling below the recrystallization stop temperature, and deformation bands were formed within the pancaked grains which provided nucleation sites for ferrite in addition to austenite grain boundaries. Upon cooling, equiaxed ferrite nucleated first at austenite grain boundaries and the carbon partitioned to the untransformed austenite. Then, acicular ferrite formed on grain boundaries and possibly deformation bands by a mixed diffusion and shear mechanism. At lower temperatures, bainite was formed in the manganese-enriched layers, where the highly substructured, larger grains developed. Finally, the remaining carbon-enriched austenite transformed to MA, RA, cementite, or a mixture of microconstituents.

For the 0Ni heat treated plate, the microstructure transformed to austenite during reheating at 1040 °C. Then, austenite transformed back to equiaxed ferrite upon cooling and holding at the intermediate hold temperature (300 °C). Finally, the remaining carbon-enriched austenite decomposed to MA islands after quenching to room temperature.

6.3 Anisotropy in Strength

A comparison of the planar anisotropy in yield strength for the investigated steels is shown in Figure 6.1 by plotting $\sigma_\theta/\sigma_{avg}$, where $\sigma_\theta$ is the yield strength measured at a specific orientation, $\theta$, (i.e. 0°, 45°, or 90°) to the rolling direction and $\sigma_{avg}$ is the average yield strength obtained from Equation 6.1. The orientation dependence is similar for the four as-received alloys, where the yield strength is higher in the T direction. The anisotropy in yield strength is highest for the 0.3Ni steel and lowest in the 15T steel. Comparing the yield strength profiles for the as-received and heat treated 0Ni plates in Figure 6.1c, the increasing tendency in yield strength in the T direction was not observed after heat treatment and the yield strength in the T
direction is close to the average yield strength. Similar yield strength anisotropy profiles have been observed in the literature for pipeline steel [29, 38, 61]. Much work has been done on relating the anisotropy in yield strength to texture [29, 38, 61, 69, 70]. Investigating the yield strength anisotropy is not a primary objective of this study and detailed analysis has not been carried out. However, several models have been developed to predict the anisotropy in yield strength based on crystal plasticity and texture and the predicted strengths showed good agreement with the experimental values [29, 38, 61, 69, 70]. Inagaki et al. [29] showed that the relative yield strength of the RD fiber components, specifically the \{113\}<110>, are highly anisotropic. Compared to a random orientation, the relative yield strength for a \{113\}<110> orientation was higher in the T direction, lower in the D direction, and close to the average in the L direction, while the \{001\}<110> orientation had a relative yield strength lower than a random orientation in all three orientations. The results of the current project are consistent with Inagaki’s assessment as shown by the consideration of texture presented in Table 6.1. Table 6.1 summarizes the intensities of the main texture components of the RD and TD fibers (Figures 5.29 and 5.43) for the studied alloys. Examining Figure 6.1 and Table 6.1, the yield strength anisotropy is lowest in the 15T alloy and has a lower intensity of the \{113\}<110> texture compared to the other as-received alloys. Also, yield strength in the T direction is close to the average yield strength for the heat treated 0Ni plate and the intensity of the \{113\}<110> component was reduced from around 4.3 to 2.1 times random. However, all texture components should be taken into account in order to accurately quantify the yield strength anisotropy.
Figure 6.1  Normalized yield strength for the (a) 15T, (b) Base, (c) as-received and heat treated 0Ni, and (d) 0.3Ni alloys in the L (0°), T (90°), and D (45°) directions, in the form of $\sigma_\theta/\sigma_{avg}$.

Table 6.1 – The Intensities of the Main Texture Components Measured for the Studied Alloys

<table>
<thead>
<tr>
<th>Steel</th>
<th>Texture Component (times random)</th>
</tr>
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<td>{001}</td>
</tr>
<tr>
<td></td>
<td>&lt;110&gt;</td>
</tr>
<tr>
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</tr>
<tr>
<td>Base</td>
<td>2.8</td>
</tr>
<tr>
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<td>0.3Ni</td>
<td>1.6</td>
</tr>
<tr>
<td>Heat Treated</td>
<td>2.1</td>
</tr>
</tbody>
</table>

6.4  Anisotropy in Toughness

The DBTTs and USEs in the L, T, and D directions for the standard Charpy impact testing for the investigated alloys are summarized in Tables 5.2 and 5.9. For all alloys, the DBTT is highest in the D direction, and the L and T DBTTs are close to each other. After heat treatment
of the 0Ni plate, the DBTTs decreased by around 10 °C in the L and T directions and around 40 °C in the D direction, along with a reduction in the DBTT anisotropy (Table 5.9). There is no significant anisotropy in the USE between the different directions for all the investigated alloys. The microstructural and texture effects on toughness are discussed in this section.

Factors that affect anisotropy in mechanical properties in plate steels include the following: grain size and shape; size, morphology, and orientation of inclusions; volume fraction and distribution of second phases; and crystallographic texture [27, 61]. There was no evidence of the presence of elongated sulfide inclusions in the experimental plates as the sulfur content was low, below 30 ppm in all alloys. The absence of sulfide inclusions contributes to the fact that there was no anisotropy between the L and T directions in the absorbed energy in Charpy impact specimen in the upper shelf region as shown in Table 5.2. If present, elongated inclusions in the rolling direction would lead to lower upper shelf energies in the T direction [71].

6.4.1 Microstructural Effects on Toughness Anisotropy

Grain size measurements in the L, T, and D planes (primary fracture planes of the three impact test orientations) were evaluated using different techniques in Chapter 5. The grain sizes, grain aspect ratios, high angle boundary densities, and volume fractions of microconstituents were close to each other in the L, T, and D orientations for the four as-received steels (Figures 5.18 and 5.20, and Tables 5.3 and 5.4) and the 0Ni heat treated steel (Figure 5.36 and Tables 5.6 and 5.7). Observed variations in the results between the L, T, and D orientations were attributed to location sensitivity since the microstructures are mixed and the measured areas are relatively small, especially for the EBSD data. However, the grain aspect ratios were slightly higher in the T orientation than the L and D orientations for the four alloys in the as-received conditions for results obtained from EBSD (Figure 5.20) and for the 15T and Base alloys for
results calculated on SEM images (Figure 5.18). An increase in the grain aspect ratios in the T plane was expected since the austenite grains would be more elongated in the T plane due to rolling. Since the slab thicknesses were around 200 mm for the 15T and Base alloys and 85 mm for the 0Ni and 0.3Ni alloys and the final thicknesses were 15.5, 13.7, 12.7, 12.7 mm for the 15T, Base, 0Ni, and 0.3Ni plates respectively, the total reductions were higher for the 15T and Base alloys compared to the 0Ni and 0.3Ni alloys. Therefore, the grains would be more elongated in the T plane for the 15T and Base alloys and hence more susceptible to cleavage crack propagation than in the L and D planes. This would explain the slight increase in the DBTT in the T orientation compared to the L orientation, specifically for the 15T and Base alloys, since the texture of the four alloys does not have a strong influence on the DBTT between the L and T orientations, as will be explained in the following section. However, the increase in the DBTT in the D orientation could not be linked to the microstructure.

6.4.2 Texture Effects on Toughness Anisotropy

The higher DBTT values for the D orientations in the impact test results observed in the studied alloys were mainly due to the crystallographic texture. An increase in the fraction of \{001\} planes parallel to the primary fracture plane for the D orientation compared to the L and T orientations was observed in the four alloys in the as-received conditions (Section 5.4). The density of \{001\} planes was similar for the four alloys in the three orientations. However, the severity of the anisotropy in the DBTTs was different between the four alloys. Also, the density of \{001\} planes parallel to the D orientation for the 0Ni heat treated plate was still high compared to the L and T orientations even though the anisotropy in the transition temperatures was significantly reduced (Section 5.6.4). This suggests that other contributions in addition to the density of \{001\} planes should be considered in order to understand the cause of toughness
anisotropy. To assess these additional contributions, a fracture analysis based on Griffith fracture criteria is discussed below.

An equation based on Griffith theory for brittle fracture for the fracture stress ($\sigma_f$) can be written as [29, 72]:

$$\sigma_f \cos^2 \theta = \left[ \frac{2E\gamma_p}{\pi(1-\nu^2)c} \right]^{\frac{1}{2}}$$

or

$$\sigma_f = \left[ \frac{2E\gamma_p}{\pi(1-\nu^2)c} \right]^{\frac{1}{2}} \frac{1}{\cos^2 \theta}$$

(6.4)

(6.5)

where $E$, $\gamma_p$, $\nu$, and $c$ are Young’s modulus, effective surface energy, Poisson’s ratio, and half crack length, respectively. $\theta$ is the angle between the most favorably oriented cleavage plane normal (closest \{001\} plane) and the tensile axis (test direction for Charpy impact test).

Therefore, for a given material and crack length:

$$\sigma_f \propto \frac{1}{\cos^2 \theta}$$

(6.6)

Based on Equation 6.6, an anisotropy factor is defined as $\cos^2 \theta$. The anisotropy factor can be evaluated for all crystallographic orientations in the three test directions from the texture data. Figure 6.2 shows the anisotropy factor ($\cos^2 \theta$) for brittle fracture at the three orientations 0° (L), 45° (D), and 90° (T) with respect to the rolling direction for the main ideal texture components. The anisotropy factor is determined at each orientation for the main RD fiber components and TD fiber components, i.e. the angle between the nearest \{001\} plane for each texture component and primary fracture plane are calculated for each impact test direction and are plotted for the RD fiber components in Figure 6.2a and the TD fiber components in Figure 6.2b. The equations used to calculate the anisotropy factor from ODF data are presented in Appendix E. From
Figure 6.2a, it is clear that most of the RD fiber components contribute to observed anisotropy at 45° to the rolling direction, especially the rotated cube component {001}<110>, since its {001} plane is parallel to the plane of the D orientation (θ=0). Yet, the other preferred orientations should not be neglected. For instance, the {113}<110> orientation makes a small angle (θ~18°) between the {001} plane and the D direction fracture plane. From Figure 6.2b, the TD components display less anisotropy (excluding the rotated cube) or rather less brittleness in the 45° direction, especially the {332}<113> component. Examining Table 6.1, it is evident that the intensity of the rotated cube component is only slightly decreased after heat treatment, which would give an indication to the close fraction of {001} planes observed parallel to the D fracture plane between the as-received and heat treated conditions (Figure 5.44a). However, the intensities of the {113}<110> and {112}<110> components decreased after heat treatment, and their contribution to the brittleness in the 45° direction was not considered in the calculations in Figure 5.44a. Also, the 15T alloy, which showed less anisotropy in the DBTT, had a lower intensity around the {113}<110> component compared to the other alloys in the as-received conditions.

Figure 6.3 displays a schematic of a broken Charpy impact sample showing grains with different orientations to further facilitate the perception described above. Grains A and C are oriented such that they have a {001} plane parallel to the primary fracture plane (perpendicular to the test direction), which is favored for cleavage fracture. Grains B and D are not preferentially oriented for fracture in the primary fracture plane. However, grains B and D are oriented such that their {001} planes makes an angle, θ, with the primary fracture plane. The closer the angle to the primary fracture plane, the more the grain will be susceptible to brittle fracture. Also, grains B and C have {001} planes parallel to the normal plane. Therefore, a
model was developed to evaluate the brittleness in each test orientation based on the intensity of every texture component and θ from the crystallographic texture data. A brittleness parameter (B) is identified as the following:

\[
B = B_o \sum_{i=1}^{n} \sum_{\theta=0}^{\theta_c} (I_{i,\theta} \cdot \cos^2 \theta)
\]  

(6.7)

where \( B_o \) is a normalizing factor, \( I \) is the intensity of an orientation in the ODF, and \( \theta_c \) is a threshold value for the maximum angle between closest \{001\} plane and the primary fracture plane that would induce cleavage fracture instead of ductile fracture in a critical temperature range. All orientations in the ODF space (not only the RD and TD fiber components) which have an angle of \( \theta_c \) or less between a \{001\} plane and the primary fracture plane were considered in the calculations for each test direction. The following discussion, shows the basis on which \( \theta_c \) was determined.

Figure 6.2 Anisotropy factor calculated for the main ideal texture components found in the (a) RD fiber and (b) TD fiber. The anisotropy factor is calculated for the L (0°), T (90°), and D (45°) directions for Charpy impact tests.
Allen et al. [73] tested single crystal iron in tension at various orientations and temperatures. Tensile tests at -196 °C with the tensile axis parallel or near the [001] exhibited brittle fracture, while crystals with orientations close to the [110] and [111] showed ductile fracture. This observation would be expected based on the relation in Equation 6.6, since the fracture strength would be lower if the sample is oriented closer to the [001]. With a decrease in testing temperature (eventually, to -196 °C), there would be a critical temperature below which the fracture strength would be lower than the yield strength for samples tested at or close to the [001], while still the yield strength is lower for samples with orientations tested further away. However, samples that showed ductile fracture at -196 °C, had lower yield strengths than the fracture strengths observed in brittle samples oriented within 20 to 25° from the [001]. The authors [73] concluded that the resolved shear stress required for slip is higher when samples tested at low temperatures and oriented with an angle smaller than 20 to 25° from the [001].
Therefore, for the assessment of Equation 6.7, a value of 20° was selected for θ_c based on the findings of Allen et al. [73]. As discussed below, calculations based on the 20° limit exhibited good agreement with the DBTT anisotropy for all alloys.

To account for the effect of texture on the DBTT, the brittleness parameter, B, is introduced into Equation 6.3 which leads to Equation 6.8:

\[
DBTT = aB - kd^{-\frac{1}{2}} + c
\]  

(6.8)

where a is a constant which depends on the strength of the texture and c is a function of \(\sigma_n\), \(\sigma_p\), and \(\sigma_d\). In the analysis here, DBTT is the 50 percent shear transition temperature. Assuming that the grain size is constant with respect to orientation, Equation 6.8 can be simplified as:

\[
DBTT = aB - c_1
\]  

(6.9)

where

\[
c_1 = kd^{-\frac{1}{2}} - c
\]  

(6.10)

Figure 6.4 displays the DBTT versus the brittleness parameter from Equation 6.7 for the four alloys in the as-received condition and the 0Ni heat treated alloy. The data for each alloy are fitted to Equation 6.9. The slopes for the four alloys in the as-received condition are similar, 20 °C per unit of brittleness parameter. The slope for the 0Ni heat treated alloy is lower, 12 °C per unit of brittleness parameter. The difference in the slopes, a, is because the texture is weaker in the heat treated plate by approximately 2/3 compared to the as-received plates. The maximum intensity of texture is 3.2 for the heat treated plate and around 4.6 for the as-received alloys. Therefore, the crystallographic texture effect on toughness would be anticipated to be less by the same amount. Table 6.2 summaries the experimental and calculated DBTTs for the studied alloys, and a and \(c_1\) values used based on Equation 6.9. The calculated DBTTs show good agreement with the experimental values. However, the small difference between the L and T
transition temperatures, specifically in the Base and heat treated alloy, is not reflected in the calculated DBTTs from Equation 6.9, since the brittleness parameters calculated for all the alloys were similar for the L and T orientations. The anisotropy between the L and T directions is due to grain size differences as explained in Section 6.4.1, which was assumed as a constant in Equation 6.9. Therefore, Equation 6.8, which incorporates grain size, was used to calculate the DBTTs for the studied alloys and the data are shown in Table 6.3. The grain size values calculated by the concentric circles method multiplied by the grain aspect ratios (Figures 5.20 and 5.36) were considered as the effective grain size, \( d \), in Equation 6.8 and \( k \) was estimated to be \( 11.5 \, ^\circ\text{C/mm}^{-1/2} \). The calculated DBTTs from Equation 6.8 also correlate well with the experimental values and account for the differences between the L and T orientations observed in the 15T, Base, and 0Ni heat treated alloy. Note that the -115 \(^\circ\text{C}\) experimental DBTT for the Base alloy in the L direction is only an estimated value which could be lower, since no samples were tested between -115 \(^\circ\text{C}\) and -196 \(^\circ\text{C}\) (Section 5.2.2). The constant \( c \) in Equation 6.8, which accounts for the increase in transition temperature due to solid solution, precipitation, and dislocation strengthening, is lower for the 15T and Base alloys than the 0Ni and 0.3Ni alloys by around 20 \(^\circ\text{C}\). Therefore, the higher DBTTs for the 0Ni and 0.3Ni alloys are likely due to the negative effect of these strengthening mechanisms.

The \{001\}<110> and \{113\}<110> components are the only two main texture components (\textit{i.e.} components with high intensities) that make a \( \theta_c \) less than 20\(^\circ\) with the D orientation (0 and \( \sim 18\(^\circ\) \), respectively). Therefore, in order to decrease toughness anisotropy, the intensities of the \{001\}<110> and \{113\}<110> components should be reduced. Ray and Jonas [26] summarized the effects of composition changes and processing variables, reported in the literature, on the development of the \{113\}<110> and \{332\}<113> components. Increasing
the Mn, Ni, Mo, and Cr contents, sharpens the \{332\}<113> component at the expense of the \{113\}<110> component. Also, similar observations were found by decreasing the prior austenite grain size and increasing the cooling rate. Several interpretations were discussed for these observations, though a definitive explanation of these effects was not provided [26].

Figure 6.4 The DBTT versus the brittleness parameter defined in Equation 3.7 for the four alloys in the as-received condition and the heat treated 0Ni alloy (HT).
Table 6.2 – Experimental and Calculated Values of the DBTTs for the Studied Alloys Based on Equation 6.9

<table>
<thead>
<tr>
<th>Steel</th>
<th>Direction</th>
<th>Experimental DBTT (°C)</th>
<th>Calculated DBTT (°C)</th>
<th>a (°C)</th>
<th>c₁ (°C)</th>
</tr>
</thead>
<tbody>
<tr>
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<td>-115</td>
<td>20</td>
<td>135</td>
</tr>
<tr>
<td></td>
<td>T</td>
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<td></td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>-85</td>
<td>-81</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Base</td>
<td>L</td>
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<td>-115</td>
<td>20</td>
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</tr>
<tr>
<td></td>
<td>T</td>
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</tr>
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Table 6.3 – Experimental and Calculated Values of the DBTTs for the Studied Alloys Based on Equation 6.8

<table>
<thead>
<tr>
<th>Steel</th>
<th>Direction</th>
<th>Experimental DBTT (°C)</th>
<th>Calculated DBTT (°C)</th>
<th>A (°C)</th>
<th>K (°C/mm⁻¹/²)</th>
<th>c (°C)</th>
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<td>-86</td>
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</table>
6.5 Delamination

Splitting in ductile fracture surfaces in the temperature range between -60 and -100 °C has been observed in some standard impact test specimen for the four as-received alloys. The splits were parallel to the rolling plane and were observed mainly in the L and T orientations. Also, splitting was associated with a decrease in the absorbed energy. Several factors have been reported for the cause of splitting and are mentioned in Section 2.4. For the current study, splitting is mainly caused by the banded structure favorably oriented for cleavage fracture parallel to the rolling plane, as will be discussed in this section.

Figure 6.5 displays EBSD image quality maps overlaid with inverse pole figure maps showing the microstructure of the 0Ni plate before (Figure 6.5a) and after (Figure 6.5b) heat treatment. Figure 6.5a shows banded regions with similarly oriented grains and very fine grains in between for the as-received 0Ni plate. While for the heat treated 0Ni plate in Figure 6.5b, the grains are randomly oriented and elongated regions are eliminated; however, the grains are larger than the fine grains in the as-received condition. Figures 6.6a and 6.6b show the microtexture of the upper and lower banded regions in Figure 6.5a. The texture of the upper band in Figure 6.5a has a \{111\}<112> orientation, while the lower region has a \{113\}<110> orientation. Most of the banded regions that were observed for the four commercially produced alloys had either one of these two orientations and few of the banded regions observed had a \{001\}<110> orientation. If \(\theta\) is redefined as the angle between closest \{001\} plane and the normal plane, which is the plane parallel to the splits in the Charpy fractured specimen, \(\theta\) would be 0° for the \{001\}<110> orientation, 25° for the \{113\}<110> orientation, and 54° for the \{111\}<112> orientation. Using the relation in Equation 6.6 for a given grain size, the relative fracture strengths for different orientation combinations can be determined as shown in Equations 6.11 and 6.12:
\[
\frac{(\sigma_f)_{\{111\}<112>}}{(\sigma_f)_{\{113\}<110>}} = \frac{(\cos^2 \theta)_{\{113\}<110>}}{(\cos^2 \theta)_{\{111\}<112>}} = 2.4
\] (6.11)

and

\[
\frac{(\sigma_f)_{\{111\}<112>}}{(\sigma_f)_{\{001\}<110>}} = \frac{(\cos^2 \theta)_{\{001\}<110>}}{(\cos^2 \theta)_{\{111\}<112>}} = 2.9
\] (6.12)

The fracture strength of the \{111\}<112> oriented region in the \(z\) direction is larger than the fracture strengths of the \{113\}<110> and \{001\}<110> oriented regions by factors of 2.4 and 2.9, respectively. So the \{113\}<110> and \{001\}<110> orientations are more susceptible to splitting since they have a small angle between the \{001\} plane and the rolling plane and thus smaller fracture strengths in the normal direction.

![Figure 6.5](image_url)

(a) Image quality maps overlaid with inverse pole figure maps for the 0Ni alloy in the transverse plane: (a) as-received and (b) heat treated. The arrows in (a) point to the upper and lower banded regions considered in Figure 6.6.
Figure 6.6  ODF sections at $\varphi_2 = 45^\circ$ showing the microtextures of the (a) upper and (b) lower banded regions of the as-received 0Ni plate in Figure 6.5a.

To further investigate the effect of grain orientations on delamination, the fracture surface for the Charpy specimen for the as-received 0Ni steel tested in the L direction at -80 °C was nickel plated and sectioned as shown in the schematic in Figure 4.8 to expose the microstructure near the split. Figures 6.7a and 6.7b show EBSD image quality maps overlaid with a color coded map and an inverse pole figure map respectively, of a region near the end of a split shown in Figure 4.8. Figure 6.7a shows that the split propagates through the large green grain (pointed out by the arrow) and is arrested by the small brown grain (pointed out by the arrow). The microtextures of the green and brown grains in Figure 6.7a are presented in Figures 6.7c and 6.7d, respectively. The large green grain has a $\{113\}<110>$ orientation, while the small brown grain has a $\{110\}<110>$ orientation. The $\{113\}<110>$ orientation has a $\theta$ of 25°, while the $\{110\}<110>$ orientation has a $\theta$ of 45° with respect to the normal plane. This observation supports the assumption that cleavage fracture is strongly influenced by the angle between the
planes and the fracture plane, \textit{i.e.} the rolling plane. So a small $\theta$ would increase the susceptibility to cleavage fracture propagation and, in this case, the occurrence of delamination.

Figure 6.8 displays a schematic of a half impact test specimen showing the stresses acting on the notch under a bending force. In Figure 6.8, $\sigma_y$ is the applied stress, and $\sigma_x$ and $\sigma_z$ are induced as a consequence of plain strain conditions. Under plane strain condition, the induced stress is greater in the $y$ direction than in the $x$ and $z$ directions. Splitting occurs when:

$$\sigma_{f,y} > \sigma_y \text{ and } \sigma_{f,z} \leq \sigma_z$$  \hfill (6.13)

where $\sigma_{f,y}$ and $\sigma_{f,z}$ are the cleavage fracture strengths in the $y$ and $z$ directions, and $\sigma_y$ and $\sigma_z$ are the stresses acting in the $y$ and $z$ directions defined in Figure 6.8, respectively. However, if $\sigma_y > \sigma_{f,y}$, cleavage fracture would occur on the primary fracture plane. Also, it would be reasonable to assume that grains B and C shown schematically in Figure 6.3 are more susceptible to cleavage fracture (splitting), since their \{001\} plane normal is parallel to the $z$ direction. For bainitic steels, the fracture strength can be related to the packet boundary or grain size ($d$) by the following equation [74]:

$$\sigma_f = \left[ \frac{4E_y\mu}{(1-v^2)d} \right]^{\frac{1}{2}}$$  \hfill (6.14)

and therefore,

$$\sigma_f \propto d^{-\frac{1}{2}}$$  \hfill (6.15)

The mechanism for delamination is interpreted as the following. When stress is induced in the $z$ direction, cracks form at the weakest locations, \textit{i.e.} at inclusions or microconstituents [28, 74]. Once the crack is initiated, it will propagate if sufficient stress is applied to produce cleavage fracture. Based on the relations in Equations 6.6 and 6.15, the large elongated grains (around 50 $\mu$m in length) that are favorably oriented for cleavage fracture parallel to the ND plane (the
{001}<110> and {113}<110> oriented grains) have significantly lower fracture strengths and thereby, are not strong enough to arrest cracks when initiated, leading to splitting. Since splitting changes the stress state from plane strain to plane stress making it difficult for cleavage fracture to occur, it would be expected that the DBTTs would decrease due to the presence of delamination. The reason that such behavior was not observed in the present study and in the work by Gervasyev et al. [44] and Shin et al. [41], is that splitting is associated with the presence of large bainite grains. Therefore, the toughening effect from delamination, if present, is countered by the large grain size of bainite, leading to an increase in the DBTT. Joo [46, 75] concluded that delamination improved the toughness of X80 pipeline steel because splitting would reduce constraint in the normal direction and therefore impede brittle fracture, decreasing the transition temperature. The conclusion was supported by the fact that brittle fracture was not observed in impact samples tested at low temperatures in the 45° to the rolling direction orientation while the longitudinal and transverse samples showed delaminated ductile fracture at the same temperatures. In the current study, delamination was also rarely observed in the standard impact samples tested in the D orientation (see Appendix C for the fracture surface macrographs). However, the reason is because splitting in the L and T orientations occurred at relatively low temperatures where the D orientation samples exhibited brittle fracture due to texture as explained in Section 6.4. In addition, the modified impact samples tested in the D orientation for the 15T and 0Ni alloys in the as-received conditions that exhibited ductile fracture showed splitting (Appendix D), while the anisotropic behavior in the DBTTs was still observed.
Figure 6.7 Image quality maps overlaid with (a) a color coded map and (b) an inverse pole figure map showing the end of a split in a standard Charpy impact sample for the as-received 0Ni steel tested in the L direction at -80 °C. (c) ODF section at $\phi_2 = 45^\circ$ showing the texture of the green grain pointed out by the arrow in (a). (d) ODF section at $\phi_2 = 45^\circ$ showing the texture of the brown grain pointed out by the arrow in (a).
Figure 6.8  Schematic of a fracture surface of a Charpy impact test specimen showing the principal stresses acting on the sample during plane strain conditions. $\sigma_y > \sigma_z$ and $\sigma_z = \nu(\sigma_x + \sigma_y)$. 
CHAPTER 7: Summary

The purpose of this project was to investigate the effects of microstructure and texture on toughness anisotropy and delamination during fracture in pipeline steels. Four commercially produced X70 pipeline steels with different chemistries and processing methods (hot strip mill, Steckel mill, and compact strip mill) were used in this investigation. Additionally, one steel was thermally processed to change the microstructure and texture. The main conclusions from this project are summarized below.

The microstructures of the four commercially produced alloys were mixed, consisting of polygonal ferrite, acicular ferrite, and bainite, with cementite, martensite-austenite, and retained austenite microconstituents in and between boundaries. Average grain sizes were similar with respect to orientation for the four alloys. The grain aspect ratios were slightly higher in the transverse plane compared to the longitudinal and diagonal planes, specifically for the 15T and Base alloys.

The crystallographic textures for the four plates exhibited typical bcc transformation textures originating from deformed austenite, where the higher intensities are located in the neighborhood of the \{113\}<110> and \{112\}<110> components, the \{554\}<225> and\{332\}<113> components, and the rotated cube \{001\}<110>.

All studied alloys exhibited similar orientation dependence of mechanical properties. The yield and tensile strengths were higher in the transverse direction than in the longitudinal and diagonal directions. The ductile to brittle transition temperatures (DBTT) for the standard impact testing were highest in the D direction, while the L and T orientations are close to one another for the 0Ni and 0.3Ni plates and slightly lower in the L orientation for the 15T and Base plates. The severity of the anisotropic behavior of the transition temperature differed between the steels,
being more pronounced in the 0Ni and Base alloys and weaker in the 0.3Ni and 15T alloys. The upper shelf energy values for all four alloys are within close proximity to each other and between the different directions. Splitting parallel to the rolling plane in the ductile fracture surfaces of the high end of transition temperature has been observed in some impact test specimen with a decrease in the absorbed energy associated with splitting.

Modified impact specimens were designed and tested to assess delamination. The modified specimens showed lower absorbed energies at the upper shelf regions and higher transition temperatures compared to the standard impact specimen. Also, as expected, splitting increased in these modified specimens due to the increased triaxial stress state at the notch root.

The 0Ni alloy was heat treated to minimize microstructural banding due to rolling and also to weaken the texture. The microstructure of the heat treated plate consisted of equiaxed ferrite grains with fine microconstituents between the grain boundaries. The anisotropy in tensile properties was eliminated after heat treatment, however, the yield and tensile strengths also decreased after heat treatment. The anisotropic behavior of the DBTT was significantly reduced after heat treatment. Also, delamination was eliminated after heat treatment as was evidently observed in the modified impact samples fracture surfaces, which resulted in higher upper shelf energies compared to the as-received alloy.

The effects of microstructure and texture on toughness anisotropy were analyzed. The grain size is mainly responsible for the differences in DBTTs between the L and T orientations, if present. The higher DBTT in the D orientation observed in pipeline steels is attributed to crystallographic texture. The higher DBTT in the D direction is due to the higher volume fraction of grains having their {100} planes parallel or close to the primary fracture plane for the D orientation. An equation based on a new “brittleness parameter” was developed to predict the
changes in DBTTs with respect to sample orientation based on grain size and texture. The calculated DBTTs correlated well with the experimental values. The \{001\}<110> and \{113\}<110> components are the main preferred orientations that cause brittleness in the D direction, since their \{001\} planes make an angle less than 20° with the primary fracture plane of the samples oriented in the D direction.

The causes of delamination in Charpy impact test fracture surfaces were assessed. Splitting parallel to the rolling plane occurs at elongated bainite regions that were oriented such that the angle between the \{001\} planes and the rolling plane was small, thus reducing the fracture strength of that grain in the normal direction. The texture of the banded regions consisted of \{001\}<110>, \{113\}<110> or \{111\}<112> orientations. It was concluded that the \{001\}<110> and \{113\}<110> orientations promote splitting because their fracture strengths in the normal direction are low. The \{111\}<112> orientation has a calculated fracture strength more than twice as the \{001\}<110> and \{113\}<110> orientations and therefore banded regions with the \{111\}<112> texture are more susceptible to cleavage fracture perpendicular to the normal direction.
CHAPTER 8: Future Work

The results showed that of the main texture components found in the investigated steels, the \{001\}<110> and \{113\}<110> components were mainly responsible for both the lower toughness in the diagonal direction and delamination. The \{001\}<110> component can be inherited from the \{001\}<010> recrystallized austenite and \{110\}<112> deformed austenite components. The \{113\}<110> component is transformed from the \{112\}<111> deformed austenite component. The effects of processing parameters and alloying elements on the sharpness of these components are not clearly understood. Therefore, it would be worthwhile to explore rolling processing parameters and/or specific alloys on the development of these components.

Modified Charpy V-notch (MCVN) impact test specimens with side grooves or slits were designed to intensify induced stresses parallel to notch root and thus facilitate evaluation of delamination. The modified impact specimens showed lower absorbed energies at high temperatures. Conventional impact specimens for high toughness X70 steels exhibited absorbed energies above 400 J at the upper shelf region, exceeding the capacity of most impact machines. The MCVN samples can be evaluated on standard impact machines. Also, the modified specimen exhibited higher transition temperatures, making it easier to define the DBTT without the necessity of going to extremely low temperatures which is difficult to control. In addition, MCVN samples fully break, even at room temperature, making the recorded absorbed energies more meaningful and also causing less wear and damage to the anvils and strikers of the machines. Therefore, the modified samples could be evaluated to replace the standard Charpy samples for high toughness pipeline steels testing requirements. Drop weight tear testing
(DWTT) was not considered in this project. Attempts can be made to correlate the modified impact results to DWTT data.

A decrease in the texture intensity, obtained after heat treatment, has eliminated delamination, and produced a reduction of the anisotropy profile and a shift of the DBTT to lower values, without affecting the absorbed energy on the USE. However, the strength of the plate was reduced after heat treatment. Different heat treatment schedules could be explored, perhaps using a lower isothermal hold temperature to increase strength of the plate in order to meet the X70 strength requirement.

A model was proposed to predict the anisotropy in DBTTs that incorporates both microstructure and crystallographic texture. The proposed model could be further validated on a wider range of steels.
REFERENCES


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APPENDIX A: Image Quality (IQ) Maps

Figure A.1  (a) and (b) IQ maps of the 15T steel in the longitudinal plane taken near the center of the plate. Green and blue boundaries are low angle (2°-15°) and high angle (greater than 15°) boundaries, respectively.

Figure A.2  (a) and (b) IQ maps of the 15T steel in the transverse plane taken near the center of the plate. Green and blue boundaries are low angle (2°-15°) and high angle (greater than 15°) boundaries, respectively.
Figure A.3  (a) and (b) IQ maps of the 15T steel in the diagonal plane taken near the center of the plate. Green and blue boundaries are low angle (2°-15°) and high angle (greater than 15°) boundaries, respectively.

Figure A.4  (a) and (b) IQ maps of the Base steel in the longitudinal plane taken near the center of the plate. Green and blue boundaries are low angle (2°-15°) and high angle (greater than 15°) boundaries, respectively.
Figure A.5  (a) and (b) IQ maps of the Base steel in the transverse plane taken near the center of the plate. Green and blue boundaries are low angle (2°-15°) and high angle (greater than 15°) boundaries, respectively.

Figure A.6  (a) and (b) IQ maps of the Base steel in the diagonal plane taken near the center of the plate. Green and blue boundaries are low angle (2°-15°) and high angle (greater than 15°) boundaries, respectively.
Figure A.7 (a) and (b) IQ maps of the 0Ni steel in the longitudinal plane taken near the center of the plate. Green and blue boundaries are low angle (2°-15°) and high angle (greater than 15°) boundaries, respectively.

Figure A.8 (a) and (b) IQ maps of the 0Ni steel in the transverse plane taken near the center of the plate. Green and blue boundaries are low angle (2°-15°) and high angle (greater than 15°) boundaries, respectively.
Figure A.9  (a) and (b) IQ maps of the 0Ni steel in the diagonal plane taken near the center of the plate. Green and blue boundaries are low angle (2°-15°) and high angle (greater than 15°) boundaries, respectively.

Figure A.10  (a) and (b) IQ maps of the 0.3Ni steel in the longitudinal plane taken near the center of the plate. Green and blue boundaries are low angle (2°-15°) and high angle (greater than 15°) boundaries, respectively.
Figure A.11 (a) and (b) IQ maps of the 0.3Ni steel in the transverse plane taken near the center of the plate. Green and blue boundaries are low angle (2°-15°) and high angle (greater than 15°) boundaries, respectively.

Figure A.12 (a) and (b) IQ maps of the 0.3Ni steel in the diagonal plane taken near the center of the plate. Green and blue boundaries are low angle (2°-15°) and high angle (greater than 15°) boundaries, respectively.
Figure A.13  (a) and (b) IQ maps of the heat treated 0Ni steel in the longitudinal plane taken near the center of the plate. Green and blue boundaries are low angle (2°-15°) and high angle (greater than 15°) boundaries, respectively.

Figure A.14  (a) and (b) IQ maps of the heat treated 0Ni steel in the transverse plane taken near the center of the plate. Green and blue boundaries are low angle (2°-15°) and high angle (greater than 15°) boundaries, respectively.
Figure A.15 (a) and (b) IQ maps of the heat treated 0Ni steel in the diagonal plane taken near the center of the plate. Green and blue boundaries are low angle (2°-15°) and high angle (greater than 15°) boundaries, respectively.
APPENDIX B: Orientation Distribution Functions (ODF)

Figure B.1  Texture of the 15T steel. (a) Full ODF sections for φ₂ from 0° to 90° at 5° intervals at half thickness. Plots of φ₂ = 45° ODF sections at: (b) half thickness, (c) quarter thickness, and (d) near the surface.
Figure B.2  Texture of the Base steel. (a) Full ODF sections for $\varphi_2$ from $0^\circ$ to $90^\circ$ at $5^\circ$ intervals at half thickness. Plots of $\varphi_2 = 45^\circ$ ODF sections at: (b) half thickness, (c) quarter thickness, and (d) near the surface.
Figure B.3 Texture of the 0Ni steel. (a) Full ODF sections for $\varphi_2$ from 0° to 90° at 5° intervals at half thickness. Plots of $\varphi_2 = 45°$ ODF sections at: (b) half thickness, (c) quarter thickness, and (d) near the surface.
Figure B.4  Texture of the 0.3Ni steel. (a) Full ODF sections for $\varphi_2$ from $0^\circ$ to $90^\circ$ at $5^\circ$ intervals at half thickness. Plots of $\varphi_2 = 45^\circ$ ODF sections at: (b) half thickness, (c) quarter thickness, and (d) near the surface.
Figure B.5 Texture of the heat treated 0Ni steel. (a) Full ODF sections for $\varphi_2$ from 0° to 90° at 5° intervals at half thickness. Plots of $\varphi_2 = 45^\circ$ ODF sections at: (b) half thickness and (c) quarter thickness.
APPENDIX C: Standard Charpy Macrographs

Figure C.1 Macrographs of the fracture surfaces for the 15T plate in the longitudinal direction tested at (a) 20 °C, (b) 0 °C, (c) -10 °C, (d) -20 °C, (e) -30 °C, (f) -40 °C, (g) -50 °C, (h) -60 °C, (i) -70 °C, (j) -80 °C, (k) -90 °C, and (l) -100 °C.
Figure C.2 Macrographs of the fracture surfaces for the 15T plate in the longitudinal direction tested at (a) -110 °C, (b) -120 °C, (c) -130 °C, (d) -140 °C, and (e) -196 °C; in the transverse direction tested at (f) 20 °C, (g) 0 °C, (h) -10 °C, (i) -20 °C, (j) -30 °C, (k) -40 °C, and (l) -50 °C.
Figure C.3 Macrographs of the fracture surfaces for the 15T plate in the transverse direction tested at (a) -60 °C, (b) -70 °C, (c) -80 °C, (d) -90 °C, (e) -100 °C, (f) -110 °C, (g) -120 °C, (h) -130 °C, (i) -140 °C, and (j) -196 °C; in the diagonal direction tested at (k) 20 °C, and (l) 0 °C.
Figure C.4 Macrographs of the fracture surfaces for the 15T plate in the diagonal direction tested at (a) -10 °C, (b) -20 °C, (c) -30 °C, (d) -40 °C, (e) -50 °C, (f) -60 °C, (g) -70 °C, (h) -80 °C, (i) -90 °C, (j) -100 °C, (k) -110 °C, (l) -120 °C, (m) -130 °C, (n) -140 °C, and (o) -196 °C.
Figure C.5  Macrographs of the fracture surfaces for the Base plate in the longitudinal direction tested at (a) 20 °C, (b) -20 °C, (c) -30 °C, (d) -40 °C, (e) -50 °C, (f) -55 °C, (g) -60 °C, (h) -65 °C, (i) -70 °C, (j) -75 °C, (k) -80 °C, (l) -85 °C, (m) -90 °C, (n) -95 °C, and (o) -100 °C.
Figure C.6  Macrographs of the fracture surfaces for the Base plate in the longitudinal direction tested at (a) -105 °C, (b) -110 °C, (c) -115 °C, and (d) -196 °C; in the transverse direction tested at (e) 20 °C, (f) -20 °C, (g) -30 °C, (h) -40 °C, (i) -50 °C, (j) -55 °C, (k) -60 °C, (l) -65 °C, (m) -70 °C, (n) -75 °C, and (o) -80 °C.
Figure C.7 Macrographs of the fracture surfaces for the Base plate in the transverse direction tested at (a) -85 °C, (b) -90 °C, (c) -95 °C, (d) -100 °C, (e) -105 °C, (f) -110 °C, (g) -115 °C, and (h) -196 °C; in the diagonal direction tested at (i) 20 °C, (j) -20 °C, (k) -30 °C, (l) -40 °C, (m) -50 °C, (n) -55 °C, and (o) -60 °C.
Figure C.8 Macrographs of the fracture surfaces for the Base plate in the diagonal direction tested at (a) -65 °C, (b) -70 °C, (c) -75 °C, (d) -80 °C, (e) -85 °C, (f) -90 °C, (g) -95 °C, (h) -100 °C, (i) -105 °C, (j) -110 °C, (k) -115 °C, and (l) -196 °C.
Figure C.9 Macrographs of the fracture surfaces for the 0Ni plate in the longitudinal direction tested at (a) 20 °C, (b) 0 °C, (c) -40 °C, (d) -60 °C, (e) -80 °C, (f) -85 °C, (g) -90 °C, (h) -95 °C, (i) 105 °C, (j) -115 °C, and (k) -196 °C; in the transverse direction tested at (l) 20 °C.
Figure C.10 Macrographs of the fracture surfaces for the 0Ni plate in the transverse direction tested at (a) 0 °C, (b) -40 °C, (c) -60 °C, (d) -80 °C, (e) -90 °C, (f) -95 °C, (g) -105 °C, (h) -110 °C, (i) 115 °C, and (j) -196 °C; in the diagonal direction tested at (k) 20 °C, and (l) 0 °C.
Figure C.11  Macrographs of the fracture surfaces for the 0Ni plate in the diagonal direction tested at (a) -35 °C, (b) -40 °C, (c) -45 °C, (d) -50 °C, (e) -60 °C, (f) -80 °C, (g) -105 °C, (h) -115 °C, and (i) -196 °C.
Figure C.12 Macrographs of the fracture surfaces for the 0.3Ni plate in the longitudinal direction tested at (a) 20 °C, (b) 0 °C, (c) -40 °C, (d) -60 °C, (e) -75 °C, (f) -85 °C, (g) -90 °C, (h) -95 °C, (i) 100 °C, (j) -115 °C, and (k) -196 °C; in the transverse direction tested at (l) 20 °C.
Figure C.13 Macrographs of the fracture surfaces for the 0.3Ni plate in the transverse direction tested at (a) 0 °C, (b) -40 °C, (c) -60 °C, (d) -75 °C, (e) -90 °C, (f) -95 °C, (g) -100 °C, (h) -110 °C, (i) 115 °C, and (j) -196 °C; in the diagonal direction tested at (k) 20 °C, and (l) 0 °C.
Figure C.14 Macrographs of the fracture surfaces for the 0.3Ni plate in the diagonal direction tested at (a) -40 °C, (b) -45 °C, (c) -50 °C, (d) -60 °C, (e) -70 °C, (f) -75 °C, (g) -100 °C, (h) -115 °C, and (i) -196 °C.
Figure C.15  Macrographs of the fracture surfaces for the heat treated 0Ni plate in the longitudinal direction tested at (a) 20 °C, (b) 0 °C, (c) -20 °C, (d) -30 °C, (e) -40 °C, (f) -50 °C, (g) -60 °C, (h) -70 °C, (i) -90 °C, (j) -95 °C, (k) -100 °C, and (l) -196 °C.
Figure C.16 Macrographs of the fracture surfaces for the heat treated 0Ni plate in the transverse direction tested at (a) 20 °C, (b) 0 °C, (c) -20 °C, (d) -30 °C, (e) -40 °C, (f) -50 °C, (g) -60 °C, (h) -70 °C, (i) -100 °C, (j) -115 °C, (k) -125 °C, and (l) -196 °C.
Figure C.17  Macrographs of the fracture surfaces for the heat treated 0Ni plate in the diagonal direction tested at (a) 20 °C, (b) 0 °C, (c) -20 °C, (d) -30 °C, (e) -40 °C, (f) -50 °C, (g) -60 °C, (h) -70 °C, (i) -80 °C, (j) -90 °C, (k) -100 °C, and (l) -196 °C.
Figure D.1 Macrographs of the fracture surfaces for the 15T plate in the longitudinal direction tested at (a) 20 °C, (b) 0 °C, (c) -20 °C, (d) -40 °C, (e) -50 °C, (f) -60 °C, (g) -70 °C, (h) -80 °C, (i) -90 °C, (j) -100 °C, (k) -110 °C, and (l) -196 °C.
Figure D.2 Macrographs of the fracture surfaces for the 15T plate in the transverse direction tested at (a) 20 °C, (b) 0 °C, (c) -20 °C, (d) -40 °C, (e) -50 °C, (f) -60 °C, (g) -70 °C, (h) -80 °C, (i) -90 °C, (j) -100 °C, (k) -110 °C, and (l) -196 °C.
Figure D.3 Macrographs of the fracture surfaces for the 15T plate in the diagonal direction tested at (a) 20 °C, (b) 0 °C, (c) -20 °C, (d) -40 °C, (e) -50 °C, (f) -60 °C, (g) -70 °C, (h) -80 °C, (i) -90 °C, (j) -100 °C, (k) -110 °C, and (l) -196 °C.
Figure D.4 Macrographs of the fracture surfaces for the 0Ni plate in the longitudinal direction tested at (a) 20 °C, (b) 0 °C, (c) -20 °C, (d) -40 °C, (e) -50 °C, (f) -60 °C, (g) -70 °C, (h) -80 °C, (i) -90 °C, (j) -100 °C, (k) -110 °C, and (l) -196 °C.
Figure D.5  Macrographs of the fracture surfaces for the 0Ni plate in the transverse direction tested at (a) 20 °C, (b) 0 °C, (c) -20 °C, (d) -40 °C, (e) -50 °C, (f) -60 °C, (g) -70 °C, (h) -80 °C, (i) -90 °C, (j) -100 °C, (k) -110 °C, and (l) -196 °C.
Figure D.6 Macrographs of the fracture surfaces for the 0Ni plate in the diagonal direction tested at (a) 20 °C, (b) 0 °C, (c) -20 °C, (d) -40 °C, (e) -50 °C, (f) -60 °C, (g) -70 °C, (h) -80 °C, (i) -90 °C, (j) -100 °C, (k) -110 °C, and (l) -196 °C.
Figure D.7 Macrographs of the fracture surfaces for the heat treated 0Ni plate in the longitudinal direction tested at (a) 20 °C, (b) 0 °C, (c) -20 °C, (d) -40 °C, (e) -50 °C, (f) -60 °C, (g) -70 °C, (h) -80 °C, (i) -90 °C, (j) -100 °C, (k) -110 °C, and (l) -196 °C.
Figure D.8 Macrographs of the fracture surfaces for the heat treated 0Ni plate in the transverse direction tested at (a) 20 °C, (b) 0 °C, (c) -20 °C, (d) -40 °C, (e) -50 °C, (f) -60 °C, (g) -70 °C, (h) -80 °C, (i) -90 °C, (j) -100 °C, (k) -110 °C, and (l) -196 °C.
Figure D.9 Macrographs of the fracture surfaces for the heat treated 0Ni plate in the diagonal direction tested at (a) 20 °C, (b) 0 °C, (c) -20 °C, (d) -40 °C, (e) -50 °C, (f) -60 °C, (g) -70 °C, (h) -80 °C, (i) -90 °C, (j) -100 °C, (k) -110 °C, and (l) -196 °C.
APPENDIX E: Calculating the anisotropy factor \( (\cos^2 \theta) \) from ODFs

The orientation matrix can be represented in Miller indices by the following:

\[
[g] = \begin{bmatrix}
g_{11} & g_{12} & g_{13} \\
g_{21} & g_{22} & g_{23} \\
g_{31} & g_{32} & g_{33}
\end{bmatrix}
\]

where the columns are the direction cosines for the sample axes RD ([uvw]), TD, and ND ((hkl)) expressed in the crystal coordinate system. Miller indices can be obtained from Euler angles (in Bunge notation) by the following:

\[
[g] = \begin{bmatrix}
\cos \varphi_1 \cos \varphi_2 - \sin \varphi_1 \sin \varphi_2 \cos \Phi & \sin \varphi_1 \cos \varphi_2 + \cos \varphi_1 \sin \varphi_2 \cos \Phi & \sin \varphi_2 \sin \Phi \\
- \cos \varphi_1 \sin \varphi_2 - \sin \varphi_1 \cos \varphi_2 \cos \Phi & - \sin \varphi_1 \sin \varphi_2 + \cos \varphi_1 \cos \varphi_2 \cos \Phi & \cos \varphi_2 \sin \Phi \\
\sin \varphi_1 \sin \Phi & - \cos \varphi_1 \sin \Phi & \cos \Phi
\end{bmatrix}
\]

To determine the direction cosine for the DD (45° to the rolling direction) sample axis, the orientation matrix is multiplied by the DD sample axis [011] as the following:

\[
\begin{bmatrix}
g_{11} & g_{12} & g_{13} \\
g_{21} & g_{22} & g_{23} \\
g_{31} & g_{32} & g_{33}
\end{bmatrix}
\begin{bmatrix}
0 \\
1 \\
1
\end{bmatrix}
= \begin{bmatrix}
g'_1 \\
g'_2 \\
g'_3
\end{bmatrix}
\]

Then the angle, \( \theta \), between the crystal <001> plane normal and sample direction can be calculated for each sample direction by the following:

\[
\cos \theta = \frac{h_1 h_2 + k_1 k_2 + l_1 l_2}{\sqrt{h_1^2 + k_1^2 + l_1^2} \sqrt{h_2^2 + k_2^2 + l_2^2}}
\]

where \([h_1 \, k_1 \, l_1]\) are the samples direction cosines and \([h_2 \, k_2 \, l_2]\) is either [001], [010], or [100], whichever makes the smallest angle.