Determination of Damage Initiation Mechanisms in Aerospace Alloys Due to Stress Corrosion Cracking via In-Situ Microscale Characterization Techniques

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by

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ABSTRACT

Aluminum alloys are used on aerospace vehicles due to their high strength-to-weight ratio, formability and machinability. However, they become vulnerable to stress corrosion cracking (SCC) during their service life. SCC is primarily caused by the material’s stress condition, a suitable corrosive environment and material susceptibility. It is also influenced by a mixture of electrochemical, mechanical, and chemical factors. Due to the complexity of SCC, tools with better resolution and sensitivity are needed to better understand the impact and interaction of the contributing factors. A vast amount of research has been done to study SCC behavior, but the scale of characterization must be reduced to elucidate the key initiation mechanisms. In this work, it is shown that SCC initiation was detected early via micro-digital image correlation (micro-DIC) prior to the crack being discernible in microscopy images. The initial effort to monitor stress corrosion cracking in AA7075-T6 involved using a pixel resolution of 3.825 microns/pixel, frame rate of 10-15 min/image and an airbrush nozzle diameter of 0.3 mm for the speckle pattern, which led to the detection of crack initiation at 98% failure load. By using a pixel resolution that is 6 times smaller, a frame rate of up to 60 times less time per image, and an airbrush nozzle that is 2 times smaller, the first observation of strain concentration marking the eventual failure region of the AA7075-T6 sample was detected as early as 58% failure load. When the micro-DIC technique was applied to study SCC behavior in additively manufactured AlSi10Mg, the first observation
of localized strain marking the eventual failure region of the sample was detected at 78% failure load. X-ray synchrotron tomography was used to qualitatively assess the hydrogen bubble and precipitate formation and to quantitatively assess the post initiation crack growth in AA7075-T651. With improved micro-DIC parameters and correlation with experimental outcomes from x-ray synchrotron tomography, multiple factors contributing to SCC can be assessed to better understand the mechanisms of SCC initiation. Correlations of material exposure time and load with SCC initiation can provide data for developing corrosion control strategies and new and improved alloys or heat treatment, as well as understanding SCC behavior in alloys made through unconventional means, such as additive manufacturing. The impact of this work lies in the life extension of alloys and greater reusability and fatigue life extension of aerospace vehicles.
This dissertation is dedicated to my family for their love, support and encouragement.
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Table 5.2  Comparison of mechanical properties of SLM AlSi10Mg and its counterpart cast and wrought alloys. 

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<thead>
<tr>
<th>Property</th>
<th>SLM AlSi10Mg</th>
<th>Cast alloy</th>
<th>Wrought alloy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile strength</td>
<td>750 MPa</td>
<td>700 MPa</td>
<td>650 MPa</td>
</tr>
<tr>
<td>Yield strength</td>
<td>550 MPa</td>
<td>500 MPa</td>
<td>450 MPa</td>
</tr>
<tr>
<td>Elongation</td>
<td>15%</td>
<td>10%</td>
<td>5%</td>
</tr>
</tbody>
</table>

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CHAPTER 1
INTRODUCTION

This chapter introduces the significance and challenges of stress corrosion cracking (SCC) in aerospace structures made of aluminum alloys. The first section introduces the importance of aluminum alloys in aerospace structures, discusses the consequences of corrosion in such structures, and addresses how the understanding and mitigation solutions of corrosion are limited. The second section provides an overview of factors that contribute to SCC in high-strength aluminum alloys. The third section discusses the microstructure of 7075-T6 aluminum alloy and how it contributes to SCC. The fourth section discusses the current progress in investigating the mechanisms of SCC. The fifth section discusses the importance of understanding the pre-initiation and initiation stages of SCC. The sixth section introduces microscale techniques to be used in this study to address the need to investigate the initiation stage of SCC. The seventh section discusses the relation of the corrosion pit to SCC initiation. The eighth section concludes with a discussion of the motivation and technical objectives of this study.
1.1 Corrosion of aluminum alloys in aerospace structures

Aluminum alloys are used extensively in the aerospace industry due to their high strength-to-weight ratio, formability and machinability [6]. High strength alloys, particularly 2xxx and 7xxx series alloys, are commonly used for primary aircraft structures, including fuselage skins, stringers and frames, wing and empennage skins, spars and ribs, mechanical systems, forks, struts and fluid systems [7]. To maintain the structural integrity of the aircraft structure, mechanical strength is taken into consideration when designing aluminum alloys. However, improvements to manufacturing methods have yet to be made to protect aluminum alloys from corrosive environments. High strength alloys are especially vulnerable to corrosion due to frequent exposure to the marine environment and long usage during the aircraft’s service life [8].

Corrosion is the gradual degradation of a material due to chemical or electrochemical reaction to the environment. It is important to address corrosion-based damage as it is one the most frequently experienced and costliest type of damage that can cost the U.S. billions of dollars due to loss in flight hours from inspection and maintenance [9, 10, 11, 12, 13]. Typically, corrosion damage is only noticed and managed retroactively, after it has been identified by visual inspection through a timely process looking specifically for bubbling, blistering, and flaking [10, 14, 15]. Under stress conditions, high strength alloys become quickly and destructively corroded, often with little visual warning. Figure 1.1 shows that detection becomes increasingly challenging depending on the corrosion-based damage size. Corrosion reduces the lifespan of critical structural components and will lead to costly consequences if it is not addressed early enough. Although corrosion
of alloys is frequently studied, a proper understanding and solutions to mitigate stress-induced corrosion are still lacking.

Research to elucidate and mitigate corrosion has benefited from considerable advances in tracking the kinetics of corrosion in these alloys over the years. However, capturing the initiation of corrosion has proven elusive. A major hurdle in the past has been that the scale of characterization has been too large to probe the earliest stages of corrosion initiation [16]. Identifying local destabilization of surface oxides and the rates at which this happens requires high spatial resolution at
the nanoscale and highly sensitive in-situ techniques that captures the reactions and formation of products with rapid collection times.

1.2 Stress corrosion cracking in aerospace structures

High-strength aluminum alloys that contain Al, Cu, Zn and Mg tend to be highly affected by SCC mostly due to residual stresses caused by heat treatment and fabrication, and stresses from installation and service life. Different heat treatments, quenching rates and alloy chemistry were developed to make the alloys, especially 7xxx series, more resistant to SCC while maintaining strength [17]. However, due to their reliance on conductivity, they are limited to thin components, which still leaves a lack of solutions to reduce the alloys’ susceptibility to SCC [7]. Consequently, many aircraft that are still in service consist of older alloys with tempers that make them vulnerable to SCC. Although documentation of SCC in aircraft is vast, the mechanisms of SCC is complex, interdependent, and not yet well understood. There are other manufacturing methods currently being developed to create alloys that are more resistant to corrosion. Additive manufacturing (AM) is becoming more prevalent in industry since it allows for fabrication of parts with complex geometries and possibly enhanced resistance to corrosion. However, there is very limited information on how material processing plays a role in the corrosion performance of AM-prepared alloys.
1.3 The microstructure of 7075-T6 aluminum alloy and its effect on stress corrosion cracking

The aluminum alloy’s susceptibility to SCC is influenced by its microstructure, which is evident in the 7xxx series high strength aluminum alloys. Intergranular SCC is known to be a significant issue in these aluminum alloys, especially in the short-transverse crack orientations in plate and forgings [18, 19]. Out of the 7xxx series aluminum alloys, AA 7075-T6 is an important alloy to study because its susceptibility to SCC has led to service failure of aircraft [20, 21]. AA 7075-T6 has been studied extensively for its SCC behavior, but the initiation mechanisms is not well understood. Thus, it makes a good base material to study in this work to better understand the mechanisms that contribute to the initiation of SCC.

It has been reported that the chemical composition and ageing condition play a vital role in the material’s mechanical properties (Table 1.1) and SCC behavior [22, 23]. AA 7075-T6 consists mainly of Al, Zn, Mg and Cu, as well as other trace elements, as shown in Table 1.2.

Table 1.1: The mechanical properties of AA 7075-T6 [3].

<table>
<thead>
<tr>
<th>Material Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ultimate strength</td>
<td>572 MPa</td>
</tr>
<tr>
<td>Yield strength</td>
<td>503 MPa</td>
</tr>
<tr>
<td>Young’s modulus</td>
<td>71 GPa</td>
</tr>
<tr>
<td>Poisson’s ratio</td>
<td>0.33</td>
</tr>
</tbody>
</table>
Table 1.2: The chemical composition of AA 7075-T6 [4].

<table>
<thead>
<tr>
<th>Element</th>
<th>Zn</th>
<th>Mg</th>
<th>Cu</th>
<th>Si</th>
<th>Fe</th>
<th>Mn</th>
<th>Cr</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight fraction (%)</td>
<td>5.1 - 6.1</td>
<td>2.1 - 2.9</td>
<td>1.2 - 2.0</td>
<td>0.40</td>
<td>0.50</td>
<td>0.39</td>
<td>0.18 - 0.28</td>
<td>0.2</td>
<td>Balance</td>
</tr>
</tbody>
</table>

Peak-aged (T6) copper-containing AA 7075 (1.2 - 2.0 wt% Cu) is less resistant to SCC and experiences higher crack velocity than over-aged (T7) copper-containing 7xxx series alloys [20, 24]. The tensile behavior of the of AA 7075-T6 is affected by the segregation and precipitation of the alloying elements along the grain boundaries [23]. It consists of precipitates, including Al, Mg and Zn, that are anodic to the matrix and readily dissolve. The precipitates, Fe, Cu and Mn boost dissolution of the adjacent matrix since they cathodic to the matrix. When Zn and Mg combine, they form MgZn$_2$ precipitates in the grains. These precipitates strengthen the the alloy and act as an anode to the matrix phase. Certain temper conditions cause the MgZn$_2$ precipitates to continuously form along the grain boundaries. Dissolution of the MgZn$_2$ precipitates cause accelerated crack propagation in AA 7075-T6 under tensile stress, which leads to increased susceptibility to SCC [25, 23]. It has also been reported that the dissolution process supplies the hydrogen for hydrogen bubbles to form, and, consequently plays a role in the hydrogen embrittlement of AA 7075-T6, especially under tensile loading [26, 27].
1.4 Current progress for stress corrosion cracking and limitations

Novel imaging techniques are being used to explore SCC with detailed correlations while in-situ experiments are being performed. One technique is using vertical scanning interferometry (VSI) in conjunction with focused ion beam (FIB) measurements to observe corrosion initiation and inclusion degradation at the nanometer scale in 2D [28]. Correlated energy-dispersive X-ray spectroscopy (EDAX) measurements is another imaging technique that provides the rate of compositional change in the region to track the formation and eradication of inclusions and oxides via in-situ VSI [28]. A third imaging technique that has been used is synchrotron holotomography, which tracks the crack tip volume as it propagates and highlights precipitates and other intermetallic particles that are difficult to otherwise track [17]. Observing clusters of precipitates in real time allows for correlation with external data on precipitate-based failure.

There are also other in-situ tests that have been done to study SCC. Current mechanical testing of SCC frequently involves slow strain rate testing in NaCl solutions. In-situ measurements aim to analyze SCC while it occurs. One example is in-situ crack initiation detection via direct current potential drop (DCPD), which was used to study Alloy 600 to determine the mechanical factors that contributed to each stage leading to SCC initiation [29]. Digital image correlation (DIC) was used to perform in-situ characterization of corrosion fatigue of aluminum alloys that were exposed to a salt spray environment. The DIC measurements, combined with microscopy, revealed that corrosion products lead to hydrogen embrittlement competing with crack tip closure effect, which results in decreased fatigue life [30]. In-situ 3D synchrotron x-ray synchrotron tomography was
used to study the effects of microstructural features and the contribution of inclusion particles, precipitates and hydrogen bubbles on pitting corrosion and corrosion fatigue [17, 31, 32]. Raman spectroscopy, while scarcely used in testing involving metals, provides specific compositional chemistry data of oxides usually explored with indirect methods [33]. These measurements provide detailed insight into SCC, but they are not frequently linked together to understand the multi-scale and field interactions.

1.5 Importance of understanding pre-initiation and initiation stages

The understanding of pre-initiation and initiation of SCC is severely lacking, especially regarding the primary cause and driving factors of SCC. Characterization of SCC alone can only describe certain phenomena. Thus, improved and linked characterization processes (Table 1.3) are necessary in order to accurately define full resolution and time scale SCC processes. Modeling efforts to explore the complexities of SCC relies heavily on initial experimental inputs for developing model parameters. Characterization of additional properties, including the electrochemical profile, mechanical response, and microstructural evolution, are used to reinforce these models.
Table 1.3: A chart showing the link between measurement needs and what needs to be known to explore the complexities of SCC initiation [5].

<table>
<thead>
<tr>
<th>What needs to be measured</th>
<th>What needs to be known</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen concentration</td>
<td>Dominated mechanism of initiation</td>
</tr>
<tr>
<td></td>
<td>(cannot be measured)</td>
</tr>
<tr>
<td>Strain history</td>
<td>Initiation index and probability</td>
</tr>
<tr>
<td></td>
<td>(cannot be measured)</td>
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<tr>
<td>Plasticity</td>
<td>Probable location of initiation</td>
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<tr>
<td></td>
<td>(can be measured)</td>
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<tr>
<td>Alloy mechanical properties</td>
<td>Time and load of initiation</td>
</tr>
<tr>
<td></td>
<td>(can be measured)</td>
</tr>
<tr>
<td>Surface precipitates, inclusion, chemistry and size</td>
<td></td>
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<tr>
<td>Environmental conditions</td>
<td></td>
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<tr>
<td>Grain boundary chemistry and size</td>
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<tr>
<td>Grain boundary corrosion rate of precipitates with and without stress</td>
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</tbody>
</table>
1.6 Microscale techniques to investigate the initiation mechanisms of stress corrosion cracking

As mentioned in Section 1.1, the scale of initiation has been too large to detect the earliest stages of SCC, and detection becomes more challenging the smaller the corrosion damage is. There are multiple techniques that are being used to study SCC, as discussed in Section 1.4. Out of these techniques, there are two of them that have the potential to detection SCC initiation in situ. Micro-DIC is one technique that can possibly capture crack initiation based on local strain data. There have been multiple studies where researchers have used micro-DIC to measure cracks that cannot be visually seen as well as observe cracking due to SCC [34, 35, 36, 37, 38, 39, 40, 41]. This technique has yet to be used to explain the initiation mechanisms of SCC. X-ray synchrotron tomography is another technique with potential to capture SCC initiation at the micro-scale in 3D over time. It has been used to study SCC behavior, mechanical characterization of corrosion-induced damage, and fatigue crack initiation and growth from corrosion in AA 7075 [42, 43, 44, 32, 45, 46, 47]. With this technique, crack growth can be measured in 2D and 3D and capture various phenomena during SCC. These phenomena include hydrogen bubble formation, pit formation, and growth of particles, such as Mg$_2$Si and CrCl$_2$. X-ray tomography can provide insight on how SCC phenomena interact with stress to promote cracking at the microscale level.
1.7 Influence of corrosion pit on stress corrosion cracking initiation

The pit to crack transition during the SCC process contributes to crack initiation due to galvanic coupling between the intermetallic inclusions and aluminum matrix [17, 23]. Multiple studies have been done to observe the crack initiation during the pit to crack transition of hemispherical pits. It has been shown through numerical analysis of alloys subjected to corrosion that the formation of cracks is favored in sharper aspect ratio pits (oblong pits), which affects the stress concentration factor [48, 49]. From experimental studies, it was found that time to crack initiation decreases with increasing pit depths [50, 41]. Pit depths of 270 µm or greater exhibited high crack growth rates, especially for a pit depth of 440 µm [41]. Pit to crack transition of different pit shapes (corner-pit and through-pit) was also experimentally investigated in AA 7075-T6 [51, 52, 53]. It was found that the through-pit sample experienced less stress to initiate crack than the corner-pit sample, both in ambient laboratory and saltwater environments. Based on the multiple studies shown here, the pits on the samples tested in this work were chosen to be both hemispherical and oblong in shape, have a depth in the order of 400 µm, and have a through-pit configuration. With these factors considered, pits were electrochemically induced in the samples for this work, so that the crack initiation at the pit can be observed.
1.8 Motivation and technical objectives

There is a need for fundamental understanding of stress corrosion cracking initiation and propagation under stress, which will be addressed through microscale characterization techniques. AA 7075 samples will undergo SCC testing and analysis since they are used extensively in the aerospace industry for their high strength-to-density ratio [54] but are highly susceptible to SCC [17]. Being able to understanding the damage initiation mechanisms due to SCC will provide a pathway to address methods for maintaining the integrity of aluminum alloys and extending their lifespan.

Degradation of aircraft structural integrity can jeopardize safety and lead to high maintenance costs. Corrosion damage leads to high costs in equipment and maintenance. SCC is a commonly observed phenomenon in aircraft materials. It is exacerbated by residual tensile stresses from manufacturing and treatment processes. Operation loads and forces affect SCC in addition to buildup of corrosion by-products. SCC is especially relevant to high-strength aluminum alloys that contain aluminum, copper, zinc and magnesium. Understanding the pre-initiation, initiation and causes of SCC is lacking. Characterization of SCC must be done at all scales simultaneously for better understanding of its mechanisms.

This study is motivated by the need to elucidate the key initiation mechanisms of SCC. This includes detecting the earliest stage of crack initiation, determining the role of hydrogen bubbles and precipitates in SCC, and quantifying their effects via microscale characterization techniques. The objectives of this study are as follows:
• Capture and quantify the earliest stage of crack initiation via micro-DIC using the smallest pixel resolution, frame rate and speckle pattern resolution possible.

• Apply the micro-DIC technique to initiate the effort to assess SCC behavior in an additively manufactured aluminum alloy.

• Assess hydrogen bubble formation and presence of precipitates during SCC of AA 7075-T651 via x-ray synchrotron tomography.

• Assess and quantify the post initiation crack growth behavior in AA 7075-T651 samples of different geometries from x-ray synchrotron tomography measurements.

The schematic in Figure 1.2 shows the correlation between the micro-DIC and x-ray tomography experiments to understand the key initiation mechanisms of SCC. From the micro-DIC experiments the strain evolution and crack length can provide information on when crack initiation occurs. From the x-ray synchrotron tomography experiments, other SCC phenomena, such as anodic dissolution and hydrogen bubble formation, can be observed in the 2D and 3D perspective, and provide information on contributing factors to crack initiation. Figure 1.3 organizes the experiments to clarify the scope of this study. Incorporating micro-DIC and x-ray synchrotron tomography into the SCC experiments will provide answers to when and why SCC occurs in aluminum alloys.
Figure 1.2: A schematic showing the correlation of experiments and results of this study.

Figure 1.3: A schematic showing the organization of the scope for this study.
CHAPTER 2
INFLUENCES AND MECHANICS OF STRESS CORROSION CRACKING

This chapter first delves into the influences of SCC, with the three primary factors being mechanical stress condition, suitable corrosive environment and material susceptibility. The second section of this chapter discusses the driving mechanisms of SCC, which include anodic dissolution, hydrogen embrittlement, film rupture and microstructure. The third section describes the various failure modes that can occur during SCC. The last section of this chapter describes the standard testing methods for investigating SCC, which build the foundation for the experiments that were conducted in this study.

2.1 Influences of stress corrosion cracking

There are three primary factors that influence stress corrosion cracking: mechanical stress condition, suitable corrosive environment and material susceptibility. The connection between these factors is summarized in Figure 2.1. The stress condition can come from applied tensile stresses and residual stresses. The residual stresses are, generally, a result of manufacturing and can come from cold deformation, welding, heat treatment, machining and grinding [55]. The corrosive environ-
ment is generated from different sources. One source is the composition of aqueous solutions and the air that surrounds the alloy. This includes aqueous solutions with anions, especially Cl\(^-\), Br\(^-\) and I\(^-\) [20], and air with varying humidity or water condensate with impurities [7]. Temperature is another source that contributes to the corrosive environment since the alloy’s susceptibility to SCC increases with temperature [20]. Electrode potential is a third source that promotes anodic dissolution, which increases the generation of hydrogen ions [56]. Flow rate is the fourth source, which, as it increases, increases the corrosion potential but reduces crack growth rate [57]. The alloy’s susceptibility to SCC is a result of manufacturing factors, such as composition, heat treatment, microstructure and surface condition [2]. During the alloy fabrication process, the material undergoes rolling, forging and extrusion, which elongates the grains in the longitudinal direction and forms "pancake-like" microstructure. These elongated grain boundaries are retained during heat treatment and, thus, provide fracture paths for SCC to occur. SCC is favored in the short transverse direction, normal to the pancake microstructure, where sustained tensile stresses and residual stresses during heat treatment tend to occur [7, 58].

2.2 The driving mechanisms of stress corrosion cracking

There are several theories that explain the driving mechanisms of stress corrosion cracking: anodic dissolution, hydrogen embrittlement, and film rupture [59, 60]. Anodic dissolution is an electrochemical corrosive process in which ions transfer from a local anodic region to a cathodic region elsewhere [61]. This process occurs in many forms on corroded metal, including pitting corrosion
around inclusions, exposed metal near the crack tip, and precipitate reactions in the grain boundary [62]. When the metal is subjected to anodic dissolution, over time the grain boundary erodes and weakens, causing the metal to be more susceptible to SCC. The second theory, hydrogen embrittlement, occurs when hydrogen ions form during the corrosive process and permeate into the metal. These ions gather in defects, such as interstitial defects, crack tips, and grain boundaries [63]. Consequently, hydrogen bubbles form, which create and widen micro-cracks [17]. These micro-cracks cause the metal to become brittle and fail early on. Hydrogen embrittlement causes discontinuous cracks to form, which leads to a non-singular path of failure [60, 23, 64]. The third theory, film rupture, is related to anodic dissolution and occurs when stress-induced slip dislocations disrupt the metal’s protective oxide layer formation and exposes fresh metal [65]. The exposed metal quickly corrodes and replenishes thin protective oxide layer. During this process,
cracks propagate and suffer dislocations at the new crack tip. Film rupture can occur at the metallic surface in the weaker regions of the protective oxide or inside of the growing crack tips. The weak spots occur naturally around certain inclusions and grain boundaries [66].

The effect of the microstructure on the material’s susceptibility to SCC must be understood to apprehend the effects of slip mode in the formation and propagation of micro-cracks. In 7xxx aluminum alloys, the primary mode of SCC involves failure at the grain boundary due to the compositional structure of the precipitates in the grain [67]. Precipitate failure creates defects within the material, which allows hydrogen to infiltrate. Precipitates can slip and shear, creating ruptured areas for anodic dissolution to occur [68]. On a larger scale, inclusions create cathodic batteries for rapid dissolution that leads to pitting corrosion [69]. The microstructural features, such as size, coherence, volume fraction, distribution of strengthening precipitates, and intermetallic constituent particles, must be considered to understand the key initiation mechanisms of SCC [59]. Experiments to study SCC are typically run in the short transverse orientation, which is most susceptible to intergranular failure [7]. Other grain orientations modify the required stress and corrosion rates to propagate the full SCC attack [70, 71, 72].

2.3 Failure modes

The most common SCC failure mode is intergranular failure, where cracks propagate along weakened or dissolved grain boundaries and brittle failure occurs [68]. Occasionally, transgranular or mixed-mode failure can occur if the alloy experiences stresses that cause normal failure prior to
grain boundary failure as well as differences in corrosive fluid ion concentration or grain direction [68].

### 2.4 Standard testing for stress corrosion cracking

Recall that, in order for SCC to occur, the stress condition must be sufficient, the environment must be corrosive, and the material must be susceptible to corrosion. These three primary factors must be simulated in experiments to effectively study the mechanisms of SCC. To create the corrosive environment, the test samples are submerged into 3.5 wt% NaCl solution as per ASTM G44 [73]. The residual stresses remaining from the material heat treatment exacerbate SCC. Additionally, the elongated grain boundaries as a result of manufacturing and heat treatment allow for fracture paths to occur within the material. Slow strain rate testing, as per ASTM G129 [74], is the loading condition applied to the sample that extends for a long period of time to allow for corrosive propagation. This is to simulate operational loads and forces from buildup of corrosion by-products. There are multiple scales of interest when performing SCC testing: macroscale, microscale and nanoscale. Characterization of SCC must explore all of these scales for to properly understand the mechanisms of SCC.
CHAPTER 3
INSTRUMENTATION AND EXPERIMENTATION FOR IN-SITU STRESS CORROSION CRACKING DETECTION AT THE MICROSCALE LEVEL

This chapter describes the instruments, sample design and preparations, and experiment methods and setup that were used to investigate the initiation mechanisms of SCC at the microscale level. The first section provides details on an experiment that utilized a microscope to perform micro-digital image correlation (DIC) on an Al 7075 tensile specimen. During this experiment, this specimen was simultaneously subjected to slow strain rate testing and a corrosive environment (3.5 wt% NaCl solution). The second section provides details on an experiment that was performed at the 2-BM station at Argonne National Laboratory (ANL) using x-ray synchrotron tomography. This experiment was performed to capture crack initiation and propagation and SCC phenomena while Al 7075 tensile specimens were subjected to load and a corrosive environment. The chapter concludes with a discussion about how the experiments are correlated with each other.
3.1 Detecting stress corrosion cracking initiation and propagation on Al 7075 via in-situ micro-digital image correlation

DIC is a nondestructive evaluation technique that tracks changes in the local surface displacements. This technique involves taking multiple images of a randomized speckle pattern on a surface that are correlated with the reference image to obtain the displacements of the speckles and, thus, the local field strain data. Jumps in the displacement field are indicators of discontinuities within the surface and can facilitate the early detection of crack initiation [37]. Corrosion studies have demonstrated the usefulness of DIC to investigate intergranular crack growth in austenitic stainless steel in aqueous environments and measure displacements that cannot be seen by eye nor conventional optical microscopy [34, 35, 36]. DIC was also used to investigate SCC behavior in other metallic materials, such as Alloy 600 and API-5L X65 steel, and measured cracks at the microscale. It was found from literature that DIC detected crack lengths as small as 30 µm and crack openings as small as 0.45 µm [35, 37]. In those studies, the pixel resolutions used ranged from 1 µm/pixel to 3.45 µm/pixel [35, 38, 39, 37, 40, 41]. Although studies that involve the use of micro-DIC have been done to assess the strain progression and crack propagation in metals subjected to SCC, such results have yet to explain the initiation mechanisms of SCC. In this work, an experiment was done, using micro-DIC, to detect SCC initiation and propagation on Al 7075 at sub-pixel resolution. The purpose of the experiment was to capture the smallest crack size at the smallest pixel size possible and to track the crack propagation up to failure. The time in which crack initiation was captured was compared between the strain measurements from micro-DIC and microscopy images. These
results were then correlated with x-ray synchrotron tomography results to determine the relation between strain measurements and other SCC phenomena, such as hydrogen embrittlement.

3.1.1 Sample design and preparation

The geometry of the AA 7075-T6 and AM-prepared samples were made as per ASTM E8 [75] (Figure 3.1) and had electrochemically induced pits within the test section. The experimental effort for SCC pit to crack transition investigation requires unique sample development that enables the focus on this specific phase of SCC. Crack initiation from the corrosion pit is the first stage after the pit formation and this impacts fatigue life of the structure significantly. Thus, the transition from pit to crack initiation is an important area of study along with the crack growth. For this study, the pit was electrochemically created at the edge of each sample before subjecting it to uniaxial tension during SCC tests. This process involves exposing the sample to a corrosive fluid and using an electrochemical pitting circuit that makes use of wire connectors and a battery to accelerate corrosion.

Tape was used to create the shape of the corrosion pit on each sample and to protect the remaining areas of the sample from corrosion. As a result, the area not covered by the tape was electrochemically pitted. To ensure strong adhesion, all surfaces that would be in contact with the tape were cleaned with isopropyl alcohol. Electrochemical pitting involves connecting the aluminum sample at the positive terminal and a platinum electrode at negative terminal of a DC power supply to electrochemically induce a pit at a localized region. A circuit consisting of a 9V alkaline
Figure 3.1: (a) Image of the tensile sample. (b) Schematic of the sample geometry. (c) Close up view of the sample test section after electrochemically inducing a pit and applying a speckle pattern for micro-DIC measurements.
battery was used to induce a pit on the portion of the sample that was exposed to 3.5 wt% NaCl solution. A pit in the order of 400µm was created. There were slight variances in the shape of the pits due to the difficulty in controlling the accuracy of the shape during the electrochemical pitting process.

3.1.2 Instrumentation and experiment setup

High resolution for DIC is needed to capture the strains associated with crack formation and propagation. To achieve this, a Psylotech micro-tensile testing system was be coupled with a high-resolution microscope to increase the output spatial resolution for the DIC measurements, as shown in Figure 3.2. The magnification of the microscope was set to 300× to achieve a pixel resolution of 0.638 µm/pixel, which is less than the smallest pixel resolution reported in literature [41]. This pixel resolution ensures that cracks and deformation can be detected at a smaller scale. Additionally, the pixel resolution used for x-ray synchrotron tomography measurements was 0.67µm/pixel, so having the micro-DIC pixel resolution nearly consistent with the that of x-ray synchrotron tomography will allow for stronger correlation in the results.

The DIC speckle pattern was refined to enable higher magnification testing, using an Iwata Custom Micron B airbrush as it is capable of producing speckles for microscale deformation measurements [76]. Based on a study by Bertfield et al [76], the airbrushed micro-DIC speckle pattern is capable of producing a minimum measurement resolution of 1 µm. The image resolution can go as low as 10 µm/pixel, which means that a damage size of 10 µm can be detected within one pixel
of the micro-DIC image. There have also been other studies in which pixel sizes smaller than 1
µm/pixel were achieved when the Iwata Custom Micron B airbrush was used for micro-DIC mea-
surements [77, 78, 79, 80, 81, 82]. In case studies reported by Wanhill et al [7], cracks that were
4 mm or smaller were reported in aircraft with 7xxx series parts. Thus, the airbrushed micro-DIC
speckle pattern allowed for detection of such crack sizes.

A corrosion chamber was incorporated into the setup for sample exposure to 3.5 wt% NaCl
solution. The sample was exposed to this solution while being subjected to slow strain rate testing
as per ASTM G129 [74]. This in-situ micro-DIC experiment setup allowed for a more accurate
simulation of the SCC attack as the samples would be simultaneously subjected to SSRT and a
corrosove environment.

3.2 Detecting stress corrosion cracking initiation, propagation and phenomena on Al 7075
via in-situ x-ray synchrotron tomography

In-situ x-ray synchrotron tomography is a nondestructive technique that allows for tracking of
materials’ deformation behavior at the micro-scale in 3D over time. With this technique, various
phenomena associated with SCC can be observed. These phenomena include hydrogen bubble
formation, crack initiation and growth, pit formation, and growth of particles (e.g. Mg₂Si and
CrCl₂) [32, 44, 45, 83, 84, 50].
Figure 3.2: Micro-DIC setup for strain rate testing (left). Close-up view of the sample inserted in a corrosion chamber and clamped on the Psylotech micro-tensile testing system (right).
3.2.1 Sample design and preparation

An in-situ SCC x-ray synchrotron tomography experiment was done at the Advanced Photon Source (APS) beamline 2-BM to quantify stress corrosion crack formation and propagation rate and to correlate the results with anodic dissolution and hydrogen embrittlement. Samples with varying cross-section shapes and load concentrating artificial features were made (Figure 3.3). This choice of sample geometries were made to enable characterization of pit-to-crack transition where manufacturing pits with sizes between 50 and 400 $\mu$m is more accurate and reproducible on square cross-section samples. The cylindrical samples were preferred for tomography measurements because the volume probed in the sample is constant while rotating for 3D measurements while the square cross-section samples ensure more accurate control on pit manufacturing.

Prior to mounting the samples into the chamber, anti-corrosive paint was applied to both ends of each sample except for the test section. The samples were then placed into their respective chambers and submerged in 3.5 wt% NaCl solution (as per ASTM G44 [73]). Load was applied
Figure 3.4: Prepared samples for in-situ SCC measurements (left) and tomography setup and scan view for the notched sample (right).

to the samples using a tightening nut. Wire was taped to the inside wall of each chamber to allow for in-situ electro-pitting (accelerated corrosion). The prepared samples were placed on a rotation stage, where x-ray synchrotron tomography scans were taken from 0° to 180°. Figure 3.4 shows the experiment setup for x-ray synchrotron tomography measurements, and Figure 3.5 shows the parameters that were used in the experiment.

3.2.2 Instrumentation and experiment setup

When in-situ electro-pitting was performed, the samples were mechanically loaded up to 90% yield. Crack initiation was observed at the notch location, as demonstrated in Figure 3.6. The crack propagation was captured over 6 hours and was correlated with precipitates. Through image
Figure 3.5: Experiment parameters to be used for x-ray synchrotron tomography measurements.

During image processing, the number of black pixels correlating with the crack was plotted with respect to time, which showed that the slope was 24.9 pixels/min. Relating this slope with the pixel resolution, 0.67 μm, the crack growth rate was found to be 16.7 μm/min. Hydrogen bubbles formed during the crack propagation. After 48-96 hours, the corrosion was longer in the long-transverse direction than it was deep, and there had also been some growth in the short-transverse direction. After 168 hours, the intergranular corrosion had extended in the short-transverse direction and again became deeper and longer. Potentially, the cracking was only due to intergranular cracking. It was also noted that the sample cross-section shape and load concentrating artificial feature did not matter as noticeable evolution of SCC was observed in different sample configurations.
Figure 3.6: Crack formation in the notched sample.
CHAPTER 4
ASSESSING STRESS CORROSION CRACK INITIATION AND
PROPAGATION ON ALUMINUM ALLOY 7075 VIA IN-SITU
MICRO-DIGITAL IMAGE CORRELATION

This chapter discusses the micro-DIC efforts to investigate the initiation mechanisms of SCC in AA 7075-T6. The first section discusses the initial effort to monitor stress corrosion crack initiation and propagation using micro-DIC on AA 7075-T6 samples with electrochemically induced pits subjected to SSRT and 3.5 wt% NaCl solution. This involved observing crack initiation and propagation using pixel resolution of 3.825 μm/pixel, large speckle size in the range of 100 μm and high frame rate of 10-15 min/image. The second section discusses how using an improved corrosion chamber design to ensure microscope focus, a pixel resolution of 0.638 μm/pixel, a speckle size in the range of 10 μm and a frame rate ranging from 15 sec/image to 1 min/image improved the resolution in strain measurements and microscopy images. The third section discusses the strain progression captured during the incubation and transient stages of SCC and correlates the measurements with SCC initiation. The results reported in this section were obtained using a consistent frame rate of 15 sec/image and ensuring that microscope focus was maintained throughout the experiment. Post test microscopy images show signs of SCC, including discoloration, pit formation, cracks and hydrogen bubble formation. The last section of this chapter provides an in-depth dis-
The smallest crack length measured from this work was \( \sim 9 \mu m \), which is 21 \( \mu m \) less than the smallest crack length reported from studies that performed SCC experiments with micro-DIC.

It is difficult to detect the exact moment of crack initiation. However, studies have shown that DIC can capture early stages of crack growth where changes in crack size cannot be resolved visually. In this chapter, the results show that initiation was detected earlier in the micro-DIC strain maps than in the microscopy images. In order to observe the localized crack initiation, a pit was electrochemically induced onto each sample since pitting is a precursor to SCC. It was found that crack initiation was observed in pits as shallow as 40 \( \mu m \), but the number and growth rate of cracks increase with increasing pit depth [85, 41]. With smaller pixel resolution, speckle size and frame rate, which were used to obtain the results in Sections 4.2 and 4.3, crack initiation was detected at 274 minutes, which corresponds to 58\% failure load. The pixel resolution was selected to be less than 1 \( \mu m/pixel \) [41]. The Iwata Custom Micron B airbrush was used to produce a speckle pattern that can achieve a pixel resolution as small as 0.04 \( \mu m/pixel \) [82]. The frame rate for taking micro-DIC images was set to 15 sec/image, which was the lowest frame rate setting for the Keyence VH-ZST microscope that was used for the experiment.
4.1 Initial effort on monitoring stress corrosion crack initiation and propagation using low pixel resolution, large speckle size and high frame rate

The initial effort to monitor stress corrosion crack initiation and propagation involved performing micro-DIC on two ASTM E8 AA 7075-T6 samples with electrochemically induced pits that were subjected to SSRT [86]. One sample was submerged in 3.5 wt% NaCl solution, while the other one was exposed to the lab air. The micro-DIC images were taken at 50× magnification, which correlates with 3.825 µm/pixel resolution, and at varying time intervals. Images of the sample in 3.5 wt% NaCl solution were taken at 10 minute intervals, while images of the sample in air were taken at 15 minute intervals.

Figure 4.1 shows the average DIC strain in the x-direction of the region of crack propagation for the samples in 3.5 wt% NaCl solution and in air with respect to time. Both samples experienced a sudden increase in tensile strain after 400 minutes (94% failure load) while being subjected to tensile load. At 600 minutes (approaching failure), the sample in 3.5 wt% NaCl solution experienced 0.051 mm/mm more strain than the sample in air. This can be attributed to the extension of intergranular attack due to the sample’s exposure to a corrosive environment [37]. Incorrect strain measurements were observed between 0 minutes and 400 minutes. This may be due to the evaporation of the 3.5 wt% NaCl solution during testing, which caused the sample images to be distorted. To account for this, a trendline was obtained for data points between 90 minutes and 310 minutes. From this trendline, the y-intercept was obtained, which was used to interpolate the DIC
A downward nonlinear trend can be seen between 0 minutes and 90 minutes, which is also due to the image distortion.

Figure 4.1: (a) Comparison of strain over time for slow strain rate test in air and in 3.5 wt% NaCl solution. The data presented corresponds to average DIC strain measured in the crack propagation area. (b) Stress-strain curves from the slow strain rate test at $5 \times 10^{-7}$ s$^{-1}$ in air and in 3.5 wt% NaCl solution. The stress is based on the load readings from the miniature load frame, and the strain is based on the strain rate and time to failure.

Figure 4.1 shows the resulting stress-strain trend of the slow strain rate test for the samples in air and 3.5 wt% NaCl solution. In this figure, the load cell strain was based on the strain rate and time to failure. At the time of failure, the sample in 3.5 wt% NaCl solution failed in 50 seconds less time than the sample in air. Although both samples failed at the same frame strain, the sample in 3.5 wt% NaCl solution reached an ultimate tensile strength of 412 MPa, which is less than
the ultimate tensile strength of the sample in air (434 MPa). These results show that destructive instability is initiated earlier when the alloy is exposed to a corrosive environment. Although the sample in 3.5 wt% NaCl solution exhibited mostly ductile failure, minor embrittlement effects likely contributed to its lower ultimate tensile strength and early failure as such effects weaken the metal significantly [5].

Figure 4.2 presents DIC strain contour plots, obtained via Ncorr [87], and corresponding microscopic images that show crack progression for the samples in 3.5 wt% NaCl solution and in air. For both samples, the DIC strain contour plots show high tensile strain concentration at the edge of the pit. After approximately 460 minutes (98% failure load), microscopy images captured crack initiation from the sample exposed to 3.5 wt% NaCl solution. This crack initiation occurred earlier for this case than for the sample in air. At approximately 600 minutes (approaching failure), the DIC contour plots show that the area of high strain concentration is of higher magnitude for the sample exposed to 3.5 wt% NaCl solution than for the sample in air. This further highlights correlations between crack formation and propagation and corresponding strain.
Figure 4.2: Comparison of crack progression at the pit of the sample in 3.5wt% NaCl solution and air monitored via micro-DIC and microscopy. The DIC and microscopy images show that the crack was detected sooner and grew larger for the sample in 3.5wt% NaCl solution than for the sample in air.
Figure 4.3 shows that both samples experienced nonlinear growth in the crack length over time prior to failure. With microscopy, the smallest crack length that was visually seen was $\sim 41 \mu m$, while the largest crack length that was captured was $\sim 484 \mu m$ before failure. These sizes are within range of the crack lengths reported in literature [39, 37], but further improvements can be made to capture smaller crack lengths at an earlier time. Cook et al. report surface lengths of up to $250 \mu m$ and crack opening displacements of less than $5 \mu m$ [39]. Bolivar et al. measured crack lengths exceeding $55 \mu m$ and opening displacements of $0.45 \mu m$ [37]. For the sample in 3.5 wt% NaCl solution, the crack length grew earlier and more significantly than the crack length of the sample in air, which agrees with the results presented in Figure 4.2.

The crack length trend for the sample in 3.5 wt% NaCl is in agreement with the change in crack length reported in literature, where the initial crack length does not change for some time and eventually crack growth begins. During this incubation period, the damage process at the crack tip occurs, which is related to either to a time-dependent chemical reaction rate process near the crack tip or due to the spread of a damaging species, such as hydrogen, around the crack tip region [88]. Localized surface plasticity contributes to the crack growth during SCC and occurs in several steps that include film rupture by initial plastic deformation at the slip bands, anodic dissolution, attenuation of strain hardening, crack initiation and propagation. From these results, it is shown that the general location of the crack initiation is in the region of high strain concentration for both cases, which indicates that the effect of the mechanical load is dominant. However, damage acceleration is a direct result of exposure to a corrosive environment.
In summary, ASTM E8 AA7075-T6 samples with electrochemically induced pits were subjected to SSRT. During this test, one sample was exposed to a corrosive environment (3.5 wt% NaCl solution), while the other one was only exposed to air to observe the effects of SCC on pit-to-crack transition and crack initiation and propagation via micro-DIC and optical microscopy. The results of this work show that micro-DIC was able to capture early stages of cracking from

Figure 4.3: Crack length over time for slow strain rate test in 3.5wt% NaCl solution and air. The smallest crack length that can visually be seen was \( \sim 41 \, \mu m \), while the largest crack length that was captured was \( \sim 484 \, \mu m \).
the sample exposed to 3.5 wt% NaCl solution prior to the crack being visible in the microscopy images. It was also shown from the results that, when an alloy is exposed to a corrosive environment, it experiences greater area of high strain concentration and crack formation prior to failure, lower ultimate tensile stress, and earlier failure time. For the next experiment, which is discussed in Section 4.2, the pit-to-crack transition and crack initiation and propagation was observed at a smaller scale to gain a better understanding of the strain concentrations and damage mechanisms during SCC. Improvement to the micro-DIC experiment setup was also made to reduce the effect of fluid evaporation on image distortion for more accuracy in results.

4.2 Monitoring stress corrosion crack initiation and propagation using an improved corrosion chamber design and smaller pixel resolution, speckle size and frame rate

In Section 4.1, it was found that strain indicating crack initiation was detected at 460 minutes (95% failure load) into the SCC experiment. However, to ensure early crack initiation can be detected, the pixel resolution and frame rate were changed to obtain better resolution in strain measurements and microscopy images. Figure 4.4 shows the frame rates that were used to take images of the sample during the SCC experiment. Images were taken at 15 sec/image during the incubation stage, and the frame rate was changed to 1 min/image for the transient and steady stages. 15 sec/image was chosen as the lowest frame rate due to the limitation of the microscope system. Additionally, a glass window was added to the corrosion chamber design to reduce image distortion due to evaporation of the 3.5 wt% NaCl solution, as shown in Figure 4.5.
Figure 4.4: A schematic of the strain versus time plot showing time stamps of the three stages of the SCC test. During the incubation stage, a frame rate of 15 sec/image was used for micro-DIC. During the transient and steady stages, a frame rate of 1 min./image was used. The experiment lasted for 610.5 minutes prior to failure.

The magnifications and corresponding pixel sizes that can be achieved with the Keyence VH-ZST microscope are provided in Table 4.1. These were considered, so that images at pixel resolutions lower than 1 μm/pixel can be used. Images of the sample were taken at different magnifications to make a final selection as shown in Figure 4.5. It was observed that sight of the pitted
region is lost past $300\times$ magnification. $200\times$ is close in pixel resolution as $1 \mu m/pixel$, thus, $300\times$ was selected for the remaining SCC experiments with micro-DIC.

Table 4.1: Microscope magnifications that were considered for the SCC experiment with micro-DIC. The $300\times$ magnification was selected since it provided the clearest image of the pitted region of the sample while achieving the smallest pixel resolution possible.

<table>
<thead>
<tr>
<th>Magnification</th>
<th>Pixel Resolution</th>
</tr>
</thead>
<tbody>
<tr>
<td>$200\times$</td>
<td>$0.95 \mu m/pixel$</td>
</tr>
<tr>
<td>$300\times$</td>
<td>$0.638 \mu m/pixel$</td>
</tr>
<tr>
<td>$500\times$</td>
<td>$0.381 \mu m/pixel$</td>
</tr>
<tr>
<td>$1000\times$</td>
<td>$0.188 \mu m/pixel$</td>
</tr>
<tr>
<td>$1500\times$</td>
<td>$0.125 \mu m/pixel$</td>
</tr>
<tr>
<td>$2000\times$</td>
<td>$0.094 \mu m/pixel$</td>
</tr>
</tbody>
</table>
Figure 4.5: Four magnifications were used to view the pitted region of the sample. 300× was selected since it provided the lowest pixel resolution possible without compromising the view and clarity of the pitted region of the sample.

Strain concentration that potentially indicates crack initiation was first observed at 533.5 minutes as shown in Figure 4.6. Between 0 minutes and 533.5 minutes, data was lost due to clarity issues in the microscopy images, which rendered them to be unusable for DIC analysis. It can be seen in Figure 4.7 that the strain over time trend was affected by this clarity issue as well. Despite
this, these results show that strain correlating with crack propagation can be captured prior to the crack being visible in the microscopy images. Additionally, it can be seen from the DIC maps that strain resolution has improved and multiple sites of strain concentration can be seen around the pit.
Figure 4.6: DIC maps with corresponding microscopy images showing strain progression within the crack region at the bottom of the pit and multiple sites of potential initiation. Strain indicating crack initiation was detected at 533.5 min. Data was only able to be collected at 533.5 min. due to clarity issues in microscopy images before that time.
Figure 4.7: (a) Strain over time for slow strain rate test of the tensile sample in 3.5wt% NaCl solution. The outlier data point in the plot was due to image distortion caused by the microscope losing focus. The data presented corresponds to average DIC strain measured in the crack propagation area. (b) Stress-strain curve from the slow strain rate test at $5 \times 10^{-7}$ s$^{-1}$ in 3.5 wt% NaCl solution. The stress is based on the load readings from the miniature load frame, and the strain is based on the strain rate and time to failure.

Figure 4.8 focuses on the DIC map with the corresponding microscopy image prior to failure at 610.5 minutes of the SCC test. It was found that the region of failure occurred within the region of the DIC map that showed high strain concentration on the left side of the pit. These results show that DIC can capture the region of failure prior to the crack being visible under the microscope.
Figure 4.8: (a) DIC strain map and corresponding microscopy image of the tensile sample prior to failure at 610.5 minutes of the SCC test. The yellow box indicates the area in which failure of the tensile sample occurred. The red markers indicate distinctive characteristics of the sample that are within proximity of the failure region. (b) Microscopy image of the sample after failure, which occurred in the same region where high strain was detected from DIC.

In summary for this test, while improving the corrosion chamber design and using a smaller pixel resolution, speckle size and frame rate helped obtain strain data that showed the location of the crack propagation prior to being visible under the microscope, further improvements needs to be made to obtain more data points prior to the steady stage of SCC. To achieve this, another experiment was run in which the frame rate was kept consistently at the smallest frame rate possible,
and the microscope focus was closely monitored. The results of this experiment is discussed in Section 4.3.

4.3 Monitoring stress corrosion crack initiation and propagation using consistent frame rate and microscope focus correction

To ensure that no data is lost during the SCC test, the frame rate was kept consistent at 15 sec/image through all stages of SCC (Figure 4.9). Additionally, the microscope was closely monitored to refocus it as needed to ensure clarity of the images were maintained throughout the test. Figure 4.10 shows the average DIC strain in the x-direction of the region of crack propagation. Similar to Figure 4.1 in Section 4.1, this plot shows a sudden increase in tensile strain was detected after 400 minutes (84% failure load). This trend is further supported by the DIC maps (Figure 4.11), where SCC initiation was detected at 440 minutes (90% failure load).
Figure 4.9: A schematic of the strain versus time plot showing time stamps of the three stages of the SCC test. For all stages, a frame rate of 15 sec/image was used for micro-DIC. The experiment lasted for 778.25 minutes prior to failure.
Figure 4.10: (a) Strain over time for slow strain rate test of the tensile sample in 3.5wt% NaCl solution. The data presented corresponds to average DIC strain measured in the crack propagation area. (b) Stress-strain curves from the slow strain rate test at $5 \times 10^{-7} \text{s}^{-1}$ in 3.5 wt% NaCl solution. The stress is based on the load readings from the miniature load frame, and the strain is based on the strain rate and time to failure.
Figure 4.11: DIC maps with corresponding microscopy images of the AA 7075 T6 tensile sample showing strain progression within the crack region at the bottom of the pit during the incubation, transient and steady stages of SCC. Strain indicating crack initiation was detected at 440 min. (90% failure load). Note that the scale of the strain measurements is 0 – 0.2 mm/mm.
Figure 4.12 takes a closer look at the strain evolution in the DIC maps between 100 minutes and 460 minutes, which shows SCC initiation detected at 420 minutes (87% failure load). However, the DIC map at 300 minutes (64% failure load) shows signs of increasing strain localizing around the pit. Thus, DIC maps taken prior to 300 minutes (64% failure load), as shown in Figure 4.13, were observed. The DIC map at 274 minutes shows that high strain concentration correlating with the crack region (Figure 4.14) was beginning to localize on the left side of the pit. This correlates with 58% of the failure load and is taken as the first instance of crack initiation detection.
Figure 4.12: DIC maps with corresponding microscopy images of the AA 7075 T6 tensile sample between 100 minutes and 460 minutes of the SCC experiment, which is during the incubation and transient stages of SCC. The DIC maps show that increase in strain, indicating crack initiation, occurred at 420 minutes, even though the microscopy images do not show crack formation. This indicates that DIC strain measurements can capture signs of crack initiation before cracks become visible under a microscope. Note that the scale of the strain measurements is 0 – 0.05 mm/mm.
Figure 4.13: DIC maps with corresponding microscopy images of the AA 7075 T6 tensile sample between 0 minutes and 275 minutes of the SCC experiment, which is during the incubation stage of SCC. At 274 minutes (58% failure load), localized high strain concentration was detected and was within the region where the crack occurred. Note that the scale of the strain measurements is 0 – 0.02 mm/mm.
Figure 4.14: (a) DIC strain map and corresponding microscopy image of the tensile sample prior to failure at 778.25 minutes of the SCC test. The yellow box indicates the area in which failure of the tensile sample occurred. The red markers indicate distinctive characteristics of the sample that are within proximity of the failure region. (b) Microscopy image of the sample after failure, which occurred in the same region where high strain was detected from DIC.

Figure 4.15 shows the plot of crack length over time. A limited amount of data points were obtained due to the difficulty of detecting the crack growth from the microscopy images. This is likely due the the fineness of the speckle pattern obscuring the crack. However, it was observed that the growth of the crack length exhibited a similar trend as the crack length plot in Figure 4.3, where
the crack length does not change for some time and eventually growth in crack length begins. To better capture the growth in crack length over time, DIC analysis was done on the displacement maps using Matlab (The Mathworks, Inc.). Cracks can be detected from these maps by identifying discontinuities in the displacement field [37, 89]. This was done using Matlab's "edge" function, which outputs a binary image with ones at the crack edge positions and zeros every where else in the map [89]. This function finds the crack edges by looking for zero crossings after the image is filtered with a Laplacian of Gaussian filter [90]. Figure 4.16 shows representative strain and displacement maps with the corresponding binary image.
Figure 4.15: Growth of crack length over time for slow strain rate test in 3.5wt% NaCl solution starting at 737.5 minutes where change in length was detected. The smallest length that can visually be seen was $\sim 47 \mu m$, while the largest length that was captured was $\sim 119 \mu m$. 
Figure 4.16: (a) DIC strain map (in the x-direction) obtained at 778.25 minutes showing strain at the region where failure would eventually happen due to cracking. (b) Corresponding displacement map (in the x-direction) showing the displacement field. (c) Binarized image of the displacement map showing the crack location.

The binarized images were reduced to 400 × 300 in pixel dimensions, which is 4 times less than the pixel dimensions of the original microscopy images (1600 × 1200). This is due to the subset spacing that was set when running the DIC analysis on Ncorr [87]. The subset spacing was selected through an iterative process such that computational load was reduced but not large enough to cause noisy displacement data. The binarized images show displacement jumps indicating multiple cracks appearing within the region where the sample would eventually fail. One crack was measured using ImageJ to track its growth over time, which is plotted in Figure 4.17. To account for the reduction in pixel dimensions in the binarized displacement maps, the measured crack length in pixel units was multiplied by 4. This value was then multiplied by the pixel resolution of the microscopy images (0.638 µm/pixel) to determine the crack length in microns. Although the overall trend in Figure 4.17 showed increasing crack length, there was some variability in the data.
points, which could be attributed to noise or loss in data points due to the reduced pixel dimensions of the binarized image.

Figure 4.17: Growth of crack length over time for slow strain rate test in 3.5 wt% NaCl solution starting at 630 minutes where change in length was detected from the binarized DIC displacement maps. The smallest length that was measured was \( \sim 9 \, \mu m \), while the largest length that was measured was \( \sim 39 \, \mu m \).

When observing crack length from the optical microscopy images, the crack does appear visible until the sample is approaching failure at 737.5 minutes. By binarizing the DIC displacement maps, crack length can be measured earlier, which, in this case, was at 630 minutes of the SCC.
experiment (98% failure load). Additionally, the first observed crack from the binarized maps was measured to be 38 \( \mu m \) smaller than the first observed crack from the optical microscopy images. However, crack length could not be detected from the binarized displacement maps prior to 98% failure load. Thus, it is not a reliable method for identifying crack length at initiation. For future work, the method of using DIC for earlier detection of crack lengths can be further improved by linking principal tensile strains directly to cracks [91]. This approach will allow for crack detection independent of crack inclination and crack displacement direction.

Figures 4.18 and 4.19 show optical microscopy images of the damage that occurred on the sample after the SCC test. The dominant mechanisms of SCC are cracking and pitting corrosion [92], which are clearly seen in the microscopy images. Other signs that were detected include discoloration indicating local corrosion at the failed region and hydrogen bubble formation.
Figure 4.18: Post test microscopy images of the front and back sides of the left piece of the sample at different magnifications. The front side images reveal discoloration of the sample within the cross-section. The back side images show signs of corrosion, including discoloration, pit formation, cracks, and hydrogen bubble formation.
Figure 4.19: Post test microscopy images of the front and back sides of the right piece of the sample at different magnifications. Images of the front and back sides reveal discoloration of the sample at the test section. The back side images show signs of corrosion, including discoloration, pit formation, and cracks.
4.4 Results and Discussion

Several iterations of the SCC experiment with micro-DIC were done to ensure that SCC initiation, strain progression and crack propagation were captured. This involved modifying the corrosion chamber design, speckle pattern method, pixel resolution and frame rate, which has improved the quality of the results. The addition of the window in the corrosion chamber provided stability in the fluid level and reduced image distortion during the test. With this design, strain measurements that correlated with the sample undergoing tensile load were obtained, and no correction needed to be made when plotting the data. Using a speckle pattern that can achieve a resolution of 10 \( \mu \text{m/pixel} \) or less, along with a smaller pixel resolution for the microscope (0.638 \( \mu \text{m/pixel} \)) and frame rate of 15 sec/image, localized strain at the crack region becomes discernible at an earlier time. From the initial effort on monitoring SCC with micro-DIC, SCC initiation was detected at 460 minutes (98% failure load), where the DIC map showed a wide region of higher strain at the bottom of the pit (Figure 4.2) It was not until 570 minutes (99% failure load) that the high strain region began to localize at the crack location. Once the experiment design and parameters were modified, SCC initiation was detected at 274 minutes (Figure 4.13), which correlates with 58% failure load. During this time, strain data as low as 0.02 mm/mm began to localize at the crack location. Although the exact moment of crack initiation is not absolute, the results of this study show the possibility of detecting SCC initiation prior to the crack being discernible through strain data.
Post test microscopy images (Figures 4.18 and 4.19) have shown visible signs of SCC in the AA7075-T6 sample. Additionally, it was found that the ultimate tensile strength (UTS) was 532 MPa. Altenbach et al [17] reported that the UTS for AA7075-T6 was 574 MPa when in air and 557 MPa when in 3.5 wt% NaCl solution during a stress corrosion test. This reduction of UTS indicates degradation of the material due to SCC. In this work, the UTS was 532 MPa when the AA7075-T6 sample was submerged in 3.5 wt% NaCl solution during the SCC test (Figure 4.10). This UTS value obtained in this work was less than the UTS reported by Altenbach et al [17]. The implementation of a corrosion pit likely caused slight degradation to the sample prior to testing.

Although the SCC experiment with micro-DIC was refined to detect SCC initiation as early as possible, the results were compared to results reported in literature to assess the practicality of the test parameters. Table 4.2 compares the DIC test parameters and results of this work and literature. The smallest pixel resolution was used in this study. However, Duff and Marrow [35] used the same frame rate, which was 15 sec/image. Despite this, the smallest crack length was measured from this work, which was ~9 µm. Based on these comparisons, it can be inferred that using a combination of small pixel resolution and small frame rate for micro-DIC allows for early detection of SCC initiation based on strain measurements and measurement of crack sizes that are visually indiscernible.
Table 4.2: Comparison between this work and studies in which SCC was observed using DIC based on test parameters and crack length.

<table>
<thead>
<tr>
<th>Parameters and Results of This Study</th>
<th>Material Tested</th>
<th>Pixel Resolution</th>
<th>Frame Rate</th>
<th>Smallest Crack Length Measured</th>
<th>Method of Measuring Crack Length</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference [35]</td>
<td>AA7075-T6</td>
<td>0.638 µm/pixel</td>
<td>15 sec/image</td>
<td>9 µm</td>
<td>DIC analysis</td>
</tr>
<tr>
<td>Reference [38]</td>
<td>A type 304 H stainless steel</td>
<td>1.5 µm/pixel</td>
<td>15 sec/image</td>
<td>30 µm</td>
<td>DIC analysis</td>
</tr>
<tr>
<td>Reference [39]</td>
<td>AISI 304 stainless steel</td>
<td>Depends on density and character of visible features in the image</td>
<td>1 min/image</td>
<td>50 µm</td>
<td>Elongation measurements</td>
</tr>
<tr>
<td>Reference [37]</td>
<td>Austenitic stainless steel</td>
<td>1.85 µm/pixel</td>
<td>0.04 sec/image</td>
<td>250 µm</td>
<td>Optical microscopy and DIC analysis</td>
</tr>
<tr>
<td>Reference [37]</td>
<td>Alloy 600</td>
<td>3.45 µm/pixel</td>
<td>1 min/image</td>
<td>55 µm</td>
<td>DIC analysis</td>
</tr>
<tr>
<td>Reference [41]</td>
<td>API-5L X65 steel</td>
<td>1 µm/pixel</td>
<td>1000 cycles/image</td>
<td>60 µm</td>
<td>DIC analysis</td>
</tr>
<tr>
<td>Reference [40]</td>
<td>Pre-corroded R4 grade high-strength steel</td>
<td>3.45 µm/pixel</td>
<td>—</td>
<td>500 µm</td>
<td>DIC analysis</td>
</tr>
</tbody>
</table>

It is important to note that the studies listed in Table 4.2 involved testing different metal material since DIC was not commonly used to investigate SCC behavior on AA7075-T6. Table 4.3 compares the test parameters and results of this work and other studies in which AA7075 was subjected to SCC testing. Most studies that investigate SCC initiation in AA7075 were performed by Singh et al [44, 32] and Stannard et al [45]. They used x-ray synchrotron tomography to study...
the SCC phenomena, including hydrogen bubble formation, crack initiation and microstructural constituents. Stannard et al [45] used a pixel resolution (0.65 \( \mu \text{m/pixel} \)) that is closest to the pixel resolution used in this work (0.638 \( \mu \text{m/pixel} \)), but the smallest crack length that they measured was approximately 5 times larger (48 \( \mu \text{m} \)). Altenbach et al [17] and Bao et al [4] measured crack lengths that were as small as \( \sim 5 \) microns since they used equipment with sub-micron pixel resolution. Based on these comparisons, it is evident that more can be done to improve on the resolution of micro-DIC to establish sub-micron crack lengths. However, the advantage that micro-DIC has over the other techniques in Table 4.3 is that displacement and strain measurements can be obtained to predict the region of cracking prior to failure of the material.

To improve the resolution of micro-DIC, it can be combined with scanning electron microscopy (SEM). It has been shown in past studies that DIC and SEM were combined to enable high resolution displacement measurements at the sub-micron level [93, 94, 95, 96]. The biggest limitation is achieving focus with a sample submerged into a fluid for SCC. In addition, when SEM is used for kinematic measurements, several imaging artifacts can occur, which are primarily spatial distortion, drift distortion and scan line shifts [97, 98]. These imaging artifacts reduce the accuracy of DIC displacement and strain measurements [99, 100]. It was shown from this work that reducing the pixel resolution down to 0.638 \( \mu \text{m/pixel} \) resulted in image clarity issues due to the microscope’s sensitivity to slight movements from the loaded sample (Section 4.2). Thus, using SEM will be more of a challenge to maintain image quality during SSRT in a fluid at the sub-micron level. However, artifact induced errors can be reduced by measuring the artifact fields along with mechanical displacements in a separate manner [96].
Table 4.3: Comparison between this work and studies in which SCC behavior was investigated on AA7075 based on characterization method, pixel resolution, time of initiation and crack size.

<table>
<thead>
<tr>
<th>Parameters and Results of This Study</th>
<th>Material Tested</th>
<th>Characterization Method</th>
<th>Pixel Resolution</th>
<th>Smallest Crack Length Measured</th>
<th>Method of Measuring Crack Length</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference [101]</td>
<td>AA7075-T651</td>
<td>Microscopy</td>
<td>1 µm/pixel</td>
<td>50 µm</td>
<td>Microscopy</td>
</tr>
<tr>
<td>Reference [44]</td>
<td>AA7075-T651</td>
<td>X-ray synchrotron tomography</td>
<td>2.2 µm/pixel</td>
<td>100 µm</td>
<td>X-ray synchrotron tomography</td>
</tr>
<tr>
<td>Reference [32]</td>
<td>AA7075-T651</td>
<td>X-ray synchrotron tomography</td>
<td>2 µm/pixel</td>
<td>100 µm</td>
<td>X-ray synchrotron tomography</td>
</tr>
<tr>
<td>Reference [45]</td>
<td>AA7075-T651</td>
<td>X-ray synchrotron tomography</td>
<td>0.65 µm/pixel</td>
<td>48 µm</td>
<td>X-ray synchrotron tomography</td>
</tr>
<tr>
<td>Reference [17]</td>
<td>AA7075 T73, T4, T6</td>
<td>Synchrotron-based holotomography, x-ray fluorescence and scanning transmission electron microscopy</td>
<td>Sub-micron</td>
<td>~5 µm</td>
<td>Synchrotron-based holotomography</td>
</tr>
</tbody>
</table>
This chapter presented results obtained from several iterations of the SCC experiment with micro-DIC. The initial effort on monitoring SCC with micro-DIC involved using low pixel resolution, large speckle size and high frame rate. The first instance of increasing strain at the bottom of the pit was detected at 460 minutes (98% failure load) via DIC. Even though the DIC map showed a wide region of strain, which made the crack location not discernible, the experiment setup and parameters served as a basis to observe the pit-to-crack transition with micro-DIC and microscopy.

For the second iteration of the experiment, SCC initiation was monitored using an improved corrosion chamber design and smaller pixel resolution, speckle size and frame rate. These changes have improved the quality of the results. The inclusion of a window on the corrosion chamber helped reduce image distortion, and using a smaller pixel resolution, speckle size and frame rate produced strain maps that show high strain regions that correspond to the crack locations. However, clarity issues in the microscopy images prior to 533.5 minutes (99% failure load) led to skewed strain data, which made it difficult to discern SCC initiation prior to 533.5 minutes (99% failure load). The third iteration of the experiment involved monitoring SCC initiation and propagation using consistent frame rate and microscope focus correction. With these changes, high strain localizing at the crack region was detected at 274 minutes (58% failure load) with micro-DIC. When the results were compared with literature, it was found that, by using a combination of small pixel resolution, small frame rate, and pre-initiation of the pit, the ability to detect SCC initiation at an earlier time and measure small crack lengths has been improved. However, from literature, SCC tests per-
formed using synchrotron-based holotomography and corrosion characterization using SEM have shown to detect crack lengths as small as 5 μm at the sub-micron scale. Although micro-DIC is capable of predicting the region of eventual cracking based on strain and displacement measurements, the resolution can be improved if it is combined with SEM, which is to be considered for future work. Now that the test parameters for SCC experiments with micro-DIC have been determined, these were implemented in another experiment that involved monitoring SCC in additively manufactured (AM) aluminum (AlSi10Mg), which is discussed in the next chapter.
CHAPTER 5
ASSESSING STRESS CORROSION CRACK INITIATION AND PROPAGATION ON ADDITIVELY MANUFACTURED ALUMINUM VIA IN-SITU MICRO-DIGITAL IMAGE CORRELATION

This chapter discusses the application of micro-DIC to investigate SCC initiation in additively manufactured (AM) AlSi10Mg, which was made using selective laser melting (SLM). The first section discusses the trend in the strain measurements during the SCC test. During the incubation and transient stages of SCC, the DIC maps detected multiple areas of high strain within the AlSi10Mg sample, which may be an indicator of formation of pits. Pits within the cross-section of the sample’s failure region were also observed in the post test optical microscopy images. The first observation of strain corresponding to the region of eventual failure was detected prior to the sample reaching the steady stage of SCC. The second section of this chapter compares the stress corrosion behavior of AM AlSi10Mg and published literature on standard manufactured aluminum alloys. The comparisons focus on mechanical performance, additive manufacturing processes, material composition and SCC phenomena.
5.1 Application of micro-digital image correlation approach to the study of stress corrosion cracking in additively manufactured AlSi10Mg

The use of additive manufacturing (AM) for fabricating parts is becoming prevalent in industry since it allows for complex geometries to be manufactured. Various AM techniques have been developed depending the material that is being fabricated and the applications of the 3D printed parts. Additionally, AM can potentially promote cost-effectiveness in producing customized products with tailorable properties and improve design-to-manufacture time [102, 103]. While AM has its advantages, reliability and consistency in material processing is still a challenge until this day because of inherent defects in the printed parts. This is especially an issue for AM metallic parts because defects, such as porosity, can affect the parts’ corrosion performance [104, 105]. AlSi10Mg is one metallic material that is commonly made using an AM process called selective laser manufacturing [106]. It has its applications in the automotive and aerospace industry, and it is also studied extensively for its mechanical behavior and characteristics [107, 108, 109]. There were also studies done to understand the corrosion behavior of AlSi10Mg, but there have been contradictory conclusions on how the manufacturing process affects the AM metal’s corrosion performance.

Several studies have been done to investigate the effect of surface finish and microstructure on corrosion performance of AM-prepared alloys [110]. Leon et al. found that polished AM-prepared samples have better corrosion resistance than the unpolished ones [111]. In another study by Fathi et al., corrosion potential values indicated that SLM AlSi10Mg had better corrosion resistance
than their cast counterparts [112], but these results contradict with those obtained by Revilla et al. [106]. While Leon et al. [111] hypothesized that the presence of cavities and irregular surface morphology in the unpolished samples contributed to higher corrosion rates, Fathi et al. [112] suggested that polishing the surface of AM-prepared alloys are detrimental to the continuous oxide layer formation on them. Thus, it is unclear how surface finish plays a role in the corrosion of SLM AlSi10Mg. The microstructure of AM-prepared alloys has shown to be beneficial when it comes to corrosion protection [110]. Revilla et al. [106] found that SLM AlSi10Mg was more resistant to localized corrosion than its cast counterpart due to having less Si-Mg segregation. Similarly, Leon et al. [113] found that the microstructure of cast AlSi10Mg was more susceptible to localized corrosion, which was hypothesized to be due to the segregation of Fe- and Mn- containing phases, irregular porosity and dendritic microstructure. However statistical analysis was not done on these results to support this hypothesis. Based on these studies, it is unclear what factors specifically enhance SLM AlSi10Mg’s resistance to corrosion. Thus, understanding the corrosion performance of SLM AlSi10Mg is still a challenge.

In terms of SLM AlSi10Mg’s SCC behavior, there is limited information on this. Studies on this topic mainly focus on finding solutions to AM parameters that will help improve the stress corrosion resistance of AlSi10Mg [111, 114, 115]. However, understanding how SCC starts in AM-prepared alloys and its mechanisms has yet to be explored. In light of the advances in microDIC to establish early SCC initiation capture demonstrated in Chapter 4, an application to SLM AlSi10Mg was pursued here. The study on this material was motivated by its importance as a lightweight metal alloy with good potential in automotive and aerospace applications. More impor-
tantly, the studies suggesting its potential to demonstrate better corrosion properties than standard equivalent cast alloys makes it one of the most promising candidates for improved SCC properties in this class of materials. In this work, an SCC experiment on a SLM AlSi10Mg tensile sample was performed. A pit was electrochemically induced onto the sample’s test section to allow for pit-to-crack transition during the test. Micro-DIC was used to take strain measurements and microscopy images in situ. This experiment was done to detect early signs of SCC initiation in SLM AlSi10Mg and to compare the SCC behavior of SLM AlSi10Mg to that of standard manufactured aluminum alloy in the literature with similar alloy composition. The significance of the results from this work is to gain a better understanding of SCC behavior in AM-prepared alloys, which will provide valuable information for the use of these alloys as well as for improving AM processes.

5.2 Monitoring stress corrosion crack initiation and propagation in additively manufactured AlSi10Mg sample

An SCC experiment was performed on an SLM AlSi10Mg tensile sample. The dimensions of the sample and experiment setup were kept consistent with the dimensions and setup described in Chapter 4. Similar to the Al 7075-T6 samples, a pit was electrochemically induced onto the test section of the SLM AlSi10Mg sample to observe the pit-to-crack transition during the SCC test. The frame rate for taking microscopy images of the sample was kept consistent at 15 sec/image, and the microscope was closely monitored to ensure that it maintained focus throughout the experiment. The pixel resolution for the microscopy images was 0.638 µm/pixel. Figure 5.1 shows
a schematic that shows the trend in strain over time. The sample experiences the incubation stage for a short time period of 100 minutes before reaching the transient stage. This trend was observed from the experimental strain data, as shown in Figure 5.2.

Figure 5.1: A schematic of the strain versus time plot showing time stamps of the three stages of the SCC test on the SLM AlSi10Mg sample. For all stages, a frame rate of 15 sec/image was used for micro-DIC. The experiment lasted for 637.5 minutes prior to failure.
Figure 5.2: (a) Strain over time plot for slow strain rate test of the SLM AlSi10Mg sample in 3.5wt% NaCl solution. The data presented corresponds to average DIC strain in the x-direction measured in the crack propagation area. (b) Stress-strain curves from the slow strain rate test at $5 \times 10^{-7}$ s$^{-1}$ in 3.5 wt% NaCl solution. The stress is based on the load readings from the miniature load frame, and the strain is based on the strain rate and time to failure.

Figure 5.3 shows the DIC maps and their corresponding microscopy images for all three stages of the SCC experiment. At 300 minutes, which corresponds to 78% failure load, the first observation of strain concentration indicating SCC initiation detection was found in the DIC map. The DIC maps and microscopy images were observed more closely in the incubation and transient stages of the SCC experiment, as shown in Figure 5.4.
Figure 5.3: DIC maps with corresponding microscopy images of the SLM AlSi10Mg tensile sample showing strain progression within the crack region at the bottom of the pit during the incubation, transient and steady stages of SCC. Strain indicating crack initiation was detected at 300 min. Note that the scale of strain measurements is 0 - 0.1 mm/mm.
Between 25 minutes and 250 minutes (8% to 70% failure load), the number of locations with high strain increased gradually. These locations were consistent between 260 minutes and 275 minutes (71% to 74% failure load). The variations in strains observed in the DIC maps are likely due to pits forming during this time period. The DIC maps continually display this trend until after 297 minutes when the strain begins to localize at the bottom of the electrochemically induced pit. Figure 5.5 takes a closer look at trend in the strain distribution in the DIC maps between 296 minutes and 297.75 minutes. The first observation of localized strain where eventual failure would happen was detected at 297.25 minutes (78% failure load). However, considering that there was a sudden change in strain distribution from 297 minutes to 297.25 minutes, it is likely that the transition was not captured in detail due to the frame rate limitation of the microscope. A smaller frame rate would help validate whether strain begins to localize sooner. To validate that the DIC maps captured the strain progression at the failure region of the sample, the DIC map taken at 637.5 minutes, right before failure, was compared with its corresponding microscopy image and the microscopy image at failure (Figure 5.6).
Figure 5.4: DIC maps with corresponding microscopy images of the SLM AlSi10Mg tensile sample between 25 minutes and 275 minutes (8% to 74% failure load) of the SCC experiment, which is during the incubation and transient stages of SCC. Multiple areas of high strain were detected, which are most likely attributed to formation of pits. Note that the scale of the strain measurements is 0 – 0.03 mm/mm.
Figure 5.5: DIC maps with corresponding microscopy images of the SLM AlSi10Mg tensile sample between 296 minutes and 297.75 minutes of the SCC experiment, which is during the transient stage of SCC. At 297.25 minutes, localized high strain concentration was detected and was within the region where the crack occurred. Note that the scale of the strain measurements is 0 – 0.03 mm/mm.
Figure 5.6: (a) DIC strain map and corresponding microscopy image of the SLM AlSi10Mg tensile sample prior to failure at 637.5 minutes of the SCC test. The yellow box indicates the area in which failure of the tensile sample occurred. The red markers indicate distinctive characteristics of the sample that are within proximity of the failure region. (b) Microscopy image of the sample after failure, which occurred in the same region where high strain was detected from DIC.

Post test microscopy images of the failed SLM AlSi10Mg sample were taken, as shown in Figure 5.7. Along the cross-section where the failure occurred, multiple pits were observed. While the electrochemically induced pit promoted localized strain within the same region of failure, the pits that have formed during the test could have accelerated the damage to the sample, thus, shortening
the incubation time. The sample has a raw matte finish, which gives it a rough granular surface. The texture of an AM aluminum alloy affects its SCC behavior. Leon and Aghion compared unpolished and polished SLM AlSi10Mg samples (made by SLM) to assess the effect of surface roughness on corrosion fatigue performance [111]. The study showed that SLM AlSi10Mg samples with high surface roughness have an excessive amount of cavities and other surface imperfections that can make the material more susceptible to corrosive damage. Such cavities and imperfections can promote electrolyte stagnation in concaved zones, which influences localized corrosion [110]. Not only is surface roughness a contributing factor to the SCC behavior of SLM AlSi10Mg, but also the material content. Al-Si alloys tend to be sensitive to multiple forms of corrosion, such as pitting and stress, and experience premature stress corrosion failure [115]. For AM aluminum alloys, the manufacturing process plays a significant role in their susceptibility to SCC. To further assess how manufacturing affects AM aluminum alloys, the characteristics and SCC behavior of SLM AlSi10Mg was compared with published literature on standard manufactured aluminum alloys of similar characteristics in Section 5.3.
Figure 5.7: Post test microscopy images of the right piece of the SLM AlSi10Mg sample where the failure occurred. Arrows in red indicate multiple locations where pits formed.
5.3 Comparison of stress corrosion crack behavior in additively manufactured AlSi10Mg and standard manufactured aluminum alloy

The SCC behavior of SLM AlSi10Mg sample was compared to its cast and wrought alloy counterparts, A360 and AA6061-T6, respectively. A360 is a cast alloy with the closest chemical composition to that of AlSi10Mg [112]. It has numerous applications in the automotive industry due to its high strength-to-weight ratio, which is beneficial for enhancing mechanical performance and decreasing fuel consumption [116]. Several studies have compared its mechanical and corrosion properties to those of SLM AlSi10Mg [117, 113, 112]. AA6061-T6 is a Mg-Si based aluminum alloy that is one of the most widely used standard manufactured material that is applicable in aerospace, automotive and other fields, and its mechanical properties have been compared to SLM AlSi10Mg in several studies [118, 119, 120, 121, 122]. The properties of this wrought alloy are attractive to industry due to its medium strength, good corrosion resistance, formability, weldability and low cost [123]. A360 and AA6061-T6 are compared to SLM AlSi10Mg, based on literature findings, in this study due to their similarities in chemical composition (Table 5.1), mechanical properties (Table 5.2), and industrial applications. This was done to determine whether AM-prepared alloys perform better or worse than standard manufactured aluminum alloys when subjected to SCC. The comparisons made here will serve as a basis for future work, which would involve obtaining empirical results using the high-resolution micro-DIC approach developed here on SCC behavior of SLM AlSi10Mg’s counterpart aluminum alloys.
Si content in SLM AlSi10Mg and A360 are nearly equivalent to each other, whereas AA6061-T6 consists of less Si (0.782 wt%). Mower and Long [120] concluded from their study that the fatigue strength of SLM AlSi10Mg is significantly inferior compared to conventional material (AA6061-T6). They attribute this to fatigue fracture initiating both at surface defects and at internal pores and large particles of segregated silicon, regardless of whether the SLM AlSi10Mg sample was polished or not. However, when SLM AlSi10Mg was compared to A360, it exhibited superior mechanical behavior and better strength-to-weight ratio than A360 [124]. Although SLM AlSi10Mg is more brittle than its A360, its tensile strength is higher, especially when printed in the horizontal direction due to less presence of pores, voids and laser splatter [125]. SLM AlSi10Mg’s yield strength and hardness were also found to be more superior to that of A360 depending on the process parameters used.

Table 5.1: Comparison of chemical composition of SLM AlSi10Mg and its counterpart cast and wrought alloys.

<table>
<thead>
<tr>
<th>Material</th>
<th>Al</th>
<th>Mg</th>
<th>Si</th>
<th>Ni</th>
<th>Sn</th>
<th>Pb</th>
<th>Cu</th>
<th>Zn</th>
<th>Ti</th>
<th>Mn</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>SLM AlSi10Mg</td>
<td>Balance</td>
<td>0.2-0.45</td>
<td>9-11</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
<td>&lt;0.1</td>
<td>&lt;0.15</td>
<td>&lt;0.45</td>
<td>&lt;0.55</td>
<td></td>
</tr>
<tr>
<td>A360 [113]</td>
<td>Balance</td>
<td>0.267</td>
<td>10.51</td>
<td>0.012</td>
<td>0.004</td>
<td>0.016</td>
<td>0.124</td>
<td>0.084</td>
<td>0.08</td>
<td>0.134</td>
<td>0.767</td>
</tr>
<tr>
<td>AA6061-T6 [118]</td>
<td>Balance</td>
<td>1.098</td>
<td>0.782</td>
<td>—</td>
<td>0.0012</td>
<td>—</td>
<td>0.117</td>
<td>0.003</td>
<td>0.0138</td>
<td>0.0159</td>
<td>0.240</td>
</tr>
</tbody>
</table>
Table 5.2: Comparison of mechanical properties of SLM AlSi10Mg and its counterpart cast and wrought alloys.

<table>
<thead>
<tr>
<th>Material</th>
<th>Orientation</th>
<th>Modulus</th>
<th>Yield</th>
<th>Ultimate tensile strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>SLM AlSi10Mg (as received)</td>
<td>Horizontal</td>
<td>70 GPa</td>
<td>240 MPa</td>
<td>410 MPa</td>
</tr>
<tr>
<td>A360 [126]</td>
<td>—</td>
<td>71 GPa</td>
<td>170 MPa</td>
<td>317 MPa</td>
</tr>
<tr>
<td>AA6061-T6 [120]</td>
<td>Longitudinal</td>
<td>66.5 GPa</td>
<td>293 MPa</td>
<td>310 MPa</td>
</tr>
</tbody>
</table>

Although there have been some studies done on the corrosion behavior of SLM AlSi10Mg, there is very little information on how this material performs when subjected to SCC. While applying micro-DIC is one way to understand the initiation mechanisms of SCC in SLM AlSi10Mg, comparing its SCC behavior to that of its counterpart aluminum alloy can provide a threshold comparison for initiation. This comparison can provide a marker for determining a material’s susceptibility for easier penetration of corrosion as a weakness or accelerated corrosion after penetration as the key mechanism for SCC failure. Consequently, preventative solutions can target the appropriate weaknesses. AA6061-T6 can potentially serve as a base material for comparison with SLM AlSi10Mg since it has similar alloy composition and there have been some studies done to investigate its SCC behavior [127, 123, 128]. Lim et al [127] performed SCC tests on AA6061-T651 and found that the as-received material had a UTS of 298 MPa when subjected to 3.5 wt% NaCl solution. This UTS is 39 MPa less than that of the SLM AlSi10Mg sample that was tested in this work (Figure 5.2), indicating that SLM AlSi10Mg performs better when subjected to SCC than AA6061. In future work, an SCC experiment will be performed on AA6061-T6 using
high-resolution micro-DIC to obtain empirical results on its SCC behavior. In regards to A360, no studies pertaining to its SCC behavior were found. However, A360 will still be considered as a candidate material for future SCC experiments since it is closest to SLM AlSi10Mg in terms of chemical composition. Additionally, studies on its corrosion performance can serve as a basis to assess A360’s SCC behavior in comparison to SLM AlSi10Mg.

5.4 Summary

In summary of this work, micro-DIC was applied to study the SCC behavior of SLM AlSi10Mg in situ since advances in the micro-DIC to detect early SCC initiation was demonstrated in Chapter 4. It was found that, at 78% failure load, the first observation of localized strain where failure would eventually occur on the sample was detected from DIC. A closer look at the DIC maps taken during the incubation and transient stages of SCC revealed multiple regions of high strain indicating the formation of pits. This was validated from the post test optical microscopy images, which showed multiple pit locations across the cross-section of the failure region of the sample. This is likely attributed to the rough granular texture of the sample, making the material more susceptible to corrosive damage. These results show that the manufacturing process affects SCC susceptibility in AM-prepared alloys.

SLM AlSi10Mg was compared with published literature on standard manufactured aluminum alloys with similar characteristics. While both A360 and AA6061-T6 have similar chemical composition as AlSi10Mg, A360 is the closest to AlSi10Mg in terms of Si content (∼10 wt%). In terms
of mechanical properties, AlSi10Mg tensile strength, yield strength and hardness are better than that of A360, especially when printed in the horizontal direction. However it is inferior in fatigue strength compared to AA6061-T6. From this work, SLM AlSi10Mg seems to show slightly better response than AA6061-T6 when subjected to SCC since its UTS is 39 MPa greater than the UTS of AA6061-T6 reported in literature. This has yet to be validated by performing an SCC experiment on both AA6061-T6 and A360 using micro-DIC, which will be done in future work.
This chapter discusses the efforts to elucidate the mechanisms contributing to the initiation of stress corrosion cracking (SCC) in aluminum alloys based on x-ray synchrotron tomography measurements. The results presented in this chapter were obtained from an SCC experiment that was done using x-ray synchrotron tomography at the Advanced Photon Source (APS) beamline 2-BM. The first section provides qualitative information on hydrogen bubble formation and growth during SCC testing on the AA7075-T651 sample with a square cross-section and an electrochemically induced pitted notch (named N2). Avenues for quantifying the bubble formation and growth and correlating that with SCC initiation are discussed. The second section presents crack growth measured post initiation from three AA7075-T651 samples, one of which has a square cross-section with an electrochemically induced pitted notch (named N2) and the other two with a circular cross-section and an electrochemically induced through-hole pit (named C1 and C5). It was found that sample cross-section shape and load concentrating artificial feature did not matter since noticeable evolution of SCC was observed in different sample configurations. Based on the crack area
measurements, the crack growth trend varied for each sample, and the corrosive damage on the samples were potentially due to intergranular cracking.

6.1 Application of x-ray synchrotron tomography approach to the study of stress corrosion cracking initiation in aluminum alloy 7075

X-ray synchrotron tomography is a non-destructive technique that involves rotating a sample 360° to obtain a series of 2D x-ray projections. These projections are reconstructed in produce a 3D representation of the sample in which the differences in phase contrast can reveal defects and damage within the sample. Due to this technique’s capability of discerning sub-micron details within a tested sample, it is applied to this work to better understand the key initiation mechanisms of SCC initiation. In Chapters 4 and 5, crack initiation was assessed based on 2D strain measurements from micro-DIC, which provided information on the samples’ SCC behavior on the surface and microscale level. With x-ray synchrotron tomography, SCC phenomena within the sample can be detected.

There have been many studies on the SCC behavior of AA7075 using x-ray synchrotron tomography, mainly by Singh et al. and Stannard et al. [42, 43, 44, 32, 45]. Mechanical characterization of corrosion-induced damage and assessment of fatigue crack initiation and growth from corrosion in AA7075 were also studied using x-ray synchrotron tomography [46, 47]. Studies done by Singh et al. [42, 43, 44, 32] have shown that crack propagation trend differs when observing cracks in 2D and 3D perspective. Discontinuous surface cracks that form during SCC were found to be a
single crack connected through the thickness of the AA7075 sample when viewed in 3D. Measurements in 2D led to more variation in the crack growth rates than when using average crack lengths through the thickness. It was also found that stress plays a role in hydrogen bubble formation and corrosion rate of Mg$_2$Si particles during SCC of AA7075. Stannard et al. [45] found that cracking may be due to preferential intergranular corrosion attack, but noted that further work is needed to validate this. It was also inferred that the evolved hydrogen bubbles may have mainly originated from preferential dissolution of Mg from Mg$_2$Si particles, indicating chemical changes in the sample during corrosion fatigue. From these studies, it is evident that x-ray synchrotron tomography offers valuable insights on various phenomena that occur during SCC. However, further work needs to be done to better understand the role of stress, hydrogen bubble formation and Mg$_2$Si particles in SCC as well as better quantifying the causes of cracking.

The motivation to use x-ray synchrotron tomography was due to its potential to reveal SCC phenomena that cannot be discerned by 2D and surface level means. As shown in Chapters 4 and 5, micro-DIC offers information on crack initiation and propagation based on strain measurements. However, those results can be further supported with x-ray synchrotron tomography to better understand how other SCC phenomena play a role in SCC initiation. In this work, x-ray synchrotron tomography measurements were used to assess the trend in crack growth over time and the characterize the hydrogen bubbles and precipitates that were observed.
6.2 Monitoring hydrogen bubbles in tomographic images of aluminum alloy 7075 subjected to stress corrosion cracking

The experimental setup for running the SCC experiment with x-ray synchrotron tomography is described in Section 3.2. Hydrogen bubble formation was observed in the N2 sample as shown in Figure 6.1. Between 0 minutes and 40 minutes, prior to the crack forming, hydrogen bubbles were already appearing, especially near the precipitate location. Multiple bubbles were also appearing near the region where eventually the crack would form. After 40 minutes, as the crack propagates, additional bubbles form, and growth of the bubble on the left side of the crack region was also observed. This could suggest that, not only stress plays a role in hydrogen bubble formation, but it could also be due to the preferential dissolution of Mg from Mg$_2$Si as pointed out by Stannard et al. [45]. The appearance of hydrogen bubbles prior to crack initiation in the N2 sample indicates the potential to use the hydrogen bubbles as a means to assess the pre-initiation and initiation stages of SCC. Additionally, the hydrogen bubble formation and growth, both at the crack location and precipitate region, needs to be quantified to better understand how each of those factors contribute to hydrogen bubble formation. The rate at which tomographic images are taken also need to be considered as there are possibly valuable information that could be missed during the SCC experiment. As seen in Chapters 4 and 5, the frame rate for obtaining DIC images plays a role in how early initiation detection can be achieved. Thus, reducing the rate at which tomographic images would be beneficial in better capturing the initiation stages of SCC. Furthermore, the correlation of results from micro-DIC and x-ray synchrotron tomography can be further improved if they were
used simultaneously in an SCC experiment, as this can provide both mechanical and chemical information on SCC initiation in real time. This would be similar to the experiment setup that was done by Rossmann et al. [129], where DIC was performed while synchrotron measurements were taken under thermomechanical conditions. Using micro-DIC along with x-ray synchrotron tomography can also provide information in between tomographic images, if the rate of tomographic images cannot be adjusted, since the frame rate for taking DIC images can be shortened as needed. This synergy in microscale techniques can improve the capability of resolving the mechanisms involved in the initiation stage of SCC and potentially the pre-initiation stage as well.
Figure 6.1: Reconstructed images of the N2 sample cross-section over time showing hydrogen bubble formation, crack propagation and precipitates near the crack location.
6.3 Measuring crack area in tomographic images of aluminum alloy 7075 subjected to stress corrosion cracking

The experiment provided an opportunity to study crack growth for post initiation monitoring of SCC. Three AA7075-T651 samples are presented here to show the crack growth trend over time when they are subjected to 3.5 wt% NaCl solution and tensile load. One sample has has a square cross-section with an electrochemically induced pitted notch (named N2). The other two samples have a circular cross-section and an electrochemically induced through-hole pit (named C1 and C5). These samples were loaded in the short-transverse direction. Figures 6.2, 6.5 and 6.8 show the geometries of the samples as well as the location within their cross-sections where crack growth was observed and assessed. The cross-section shown for each sample correspond to one slice of the tomographic images collected where the pitted notch or hole was located. To quantify the crack growth, Matlab (The Mathworks, Inc.) was used to define the location of the crack, binarize the images of the crack location and black pixel count corresponding to the crack location was plotted with respect to time. The optimal threshold of the binarized images were automatically determined using the "graythresh" function in Matlab to eliminate as much noise as possible from the images. The resulting binarized images for each sample are shown in Figures 6.3, 6.6 and 6.9. The black pixel count was converted to microns, based on the x-ray synchrotron tomography system’s pixel resolution of 0.67 µm/pixel, to determine the crack area. The crack area was then plotted with respect to time for each sample, as shown in Figures 6.4, 6.7 and 6.10.
The crack growth for N2 (Figure 6.4) and C1 (Figure 6.7) exhibited a trend that is nearly logarithmic, while the crack growth trend for C5 (Figure 6.10) appeared to be exponential. The crack growth trend for C5 seems to support the trends observed in Chapters 4 and 5 when compared to the strain vs. time and crack length vs. time trends. However, N2 and C1 differed from those trends. Considering that the crack area was assessed from one slice in each sample, the results of this work further support the literature in which 2D crack propagation would reveal varying trends. Additionally, it is possible that crack propagation was occurring through the thickness of each sample. This is further supported by findings from a similar study by Knight et al. [130]. In this study, x-ray computed tomography was used to assess intergranular corrosion in aircraft aluminum alloys. The trends observed in this study were similar to trends observed in this work in which cracks were detected in multiple directions and locations within each sample. The possibility of intergranular cracking was also pointed out by Stannard et al. [130]. Further work will need to be done to determine whether intergranular cracking was occurring in these samples. This can be supported by implementing simultaneous use of micro-DIC and x-ray synchrotron tomography, as mentioned in Section 6.2 of this chapter, which may aid in using crack growth as a means to study SCC initiation.
Figure 6.2: (a) Tomographic image of the notched, square AA7075 sample (N2) cross-section (top-down view) showing hydrogen bubbles, precipitates and crack growth. (b) A schematic showing the location of the sample where the image was taken.
Figure 6.3: Tomography image of the notched, square AA7075 sample (N2) cross-section with its corresponding binarized images of the crack propagation. The location where the binarized images were taken is boxed in red in the tomographic image.
Figure 6.4: Crack growth trend for the notched, square AA7075 sample (N2). Crack area was based on number of black pixels within the binarized images and pixel resolution (0.67µm/pixel). Boxed in red is the location on the sample cross-section where the crack area measurements were taken.
Figure 6.5: (a) Tomographic image of the open hole, cylindrical AA7075 sample (C1) cross-section (top-down view) showing hydrogen bubbles, precipitates and crack growth. (b) A schematic showing the location of the sample where the image was taken.
Figure 6.6: Tomography image of the open hole, cylindrical AA7075 sample (C1) cross-section with its corresponding binarized images of the crack propagation. The location where the binarized images were taken is boxed in red in the tomographic image.
Figure 6.7: Crack growth trend for the open hole, cylindrical AA7075 sample (C1). Crack area was based on number of black pixels within the binarized images and pixel resolution (0.67µm/pixel). Boxed in red is the location on the sample cross-section where the crack area measurements were taken.
Figure 6.8: (a) Tomographic image of the open hole, cylindrical AA7075 sample (C5) cross-section (top-down view) showing hydrogen bubbles and crack growth. (b) A schematic showing the location of the sample where the image was taken.
Figure 6.9: Tomography image of the open hole, cylindrical AA7075 sample (C5) cross-section with its corresponding binarized images of the crack propagation. The location where the binarized images were taken is boxed in red in the tomographic image.
Figure 6.10: Crack growth trend for the open hole, cylindrical AA7075 sample (C5). Crack area was based on number of black pixels within the binarized images and pixel resolution (0.67μm/pixel). Boxed in red is the location on the sample cross-section where the crack area measurements were taken.

6.4 Summary

This chapter presents the initial effort to qualify and quantify the SCC phenomena in AA7075-T651 using x-ray synchrotron tomography. Observation of the hydrogen bubbles in the N2 sample showed that there are possibly both mechanical and chemical aspects that contribute to the formation of the bubbles. The crack measurements from the N2, C1 and C5 samples prove that crack growth trend can vary due to the possibility of crack propagation happening through the thickness
of the sample. Additionally, further work needs to be done to validate whether the crack propagation is due to intergranular cracking and to utilize crack growth to study the initiation stage of SCC. Additional analysis on the x-ray synchrotron tomography measurements will help better resolve the causes of SCC initiation. Furthermore, to better correlate SCC phenomena obtained from x-ray tomography with surface-level crack initiation and propagation captured from micro-DIC, it would be beneficial to utilize micro-DIC simultaneously with x-ray synchrotron tomography.
Aluminum alloys are widely used in the aerospace industry since they have exceptional strength-to-weight ratio, formability and machinability. While they have desirable mechanical properties to ensure structural integrity of aerospace structures, they tend to be vulnerable to corrosion, especially when frequently exposed to marine environments. Corrosion-based damage is costly and can lead to loss in flight hours from inspection and maintenance. Under stress conditions, high strength alloys become quickly and destructively corroded, often with little visual warning. Research has been done to elucidate and mitigate stress corrosion cracking, but capturing the initiation stages of corrosion is challenging due to the scale of characterization being too large. Techniques, such as vertical scanning interferometry (VSI), correlated energy-dispersive x-ray spectroscopy (EDAX) and direct current potential drop (DCPD) to name a few, have been used to study SCC. However, they are not frequently linked together to understand the multi-scale and field interactions of SCC.

This study is motivated by the need to fundamentally understand the mechanisms behind SCC initiation and propagation, which were characterized through microscale characterization techniques. This includes detecting the earliest stage of crack initiation, determining the role of hydrogen bubbles and precipitates in SCC, and quantifying their effects. SCC experiments on aluminum...
alloy samples were performed using micro-DIC and x-ray synchrotron tomography to capture crack initiation and propagation and SCC phenomena at the microscale level.

Three SCC experiments were run on AA7075-T6 tensile samples using micro-DIC to obtain strain measurements. From these experiments it was found that the inclusion of a window on the corrosion chamber helped reduce image distortion, and using a smaller pixel resolution, speckle size and frame rate produced strain maps that show high strain regions that correspond to the crack locations. High strain localizing at the crack region was detected at 274 minutes (58% failure load) with micro-DIC. When the results were compared with literature, it was found that, by using a combination of small pixel resolution, small frame rate, and pre-initiation of the pit, the ability to detect SCC initiation at an earlier time and measure small crack lengths has been improved. However, from literature, SCC tests performed using synchrotron-based holotomography and corrosion characterization using SEM have shown to detect crack lengths as small as 5 µm at the sub-micron scale. Although micro-DIC is capable of predicting the region of eventual cracking based on strain and displacement measurements, the resolution can be improved if it is combined with SEM, which is to be considered for future work.

In light of the advances in the micro-DIC technique to capture early stages of SCC initiation, this technique was applied to study the SCC behavior of a SLM AlSi10Mg tensile sample. It was found that, at 78% failure load, the first observation of localized strain where failure would eventually occur on the sample was detected from DIC. It was found that, at 78% failure load, the first observation of localized strain where failure would eventually occur on the sample was detected from DIC. A closer look at the DIC maps taken during the incubation and transient stages
of SCC revealed multiple regions of high strain indicating the formation of pits. This was validated from the post test optical microscopy images, which showed multiple pit locations across the cross-section of the failure region of the sample. This is likely attributed to the rough granular texture of the sample, making the material more susceptible to corrosive damage. These results show that the manufacturing process affects SCC susceptibility in AM-prepared alloys. From this work, SLM AlSi10Mg seems to perform slightly better than AA6061-T6 when subjected to SCC since its UTS is 39 MPa greater than the UTS of AA6061-T6 reported in literature. This has yet to be validated by performing an SCC experiment on both AA6061-T6 and A360 using micro-DIC, which will be done in future work.

SCC experiments were performed on AA7075-T651 samples with varying geometries to assess hydrogen bubble and precipitate formation and crack growth via x-ray synchrotron tomography. Observation of the hydrogen bubbles in the N2 sample showed that there are possibly both mechanical and chemical aspects that contribute to the formation of the bubbles. The crack measurements from the N2, C1 and C5 samples prove that crack growth trend can vary due to the possibility of crack propagation happening through the thickness of the sample. Additional analysis on can validate whether crack propagation is due to intergranular cracking and better resolve the causes of SCC initiation. Furthermore, to better correlate SCC phenomena obtained from x-ray tomography with surface-level crack initiation and propagation captured from micro-DIC, it would be beneficial to utilize micro-DIC simultaneously with x-ray synchrotron tomography.
LIST OF REFERENCES


[74] ——, “Standard practice for slow strain rate testing to evaluate the susceptibility of metallic materials to environmentally assisted cracking g129 21,” 2021.


