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***In Situ* Investigation of High Humidity Stress Corrosion Cracking of 7075 Aluminum Alloy by Three-Dimensional (3D) X-ray Synchrotron Tomography**

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In situ X-ray synchrotron tomography was used to investigate the stress corrosion cracking behavior of under-aged Al–Zn–Mg–Cu alloy in moisture. The discontinuous surface cracks (crack jumps) mentioned in the literature are actually a single continuous and tortuous crack when observed in three dimension (3D). Contrary to 2D measurements made at the surface which suggest non-uniform crack growth rates, 3D measurements of the crack length led to a much more accurate measurement of crack growth rates.

Keywords: Stress Corrosion Cracking, X-ray Tomography, Moisture, Aluminum Alloys

Al 7075 alloys are used extensively due to their high strength-to-weight ratio.[1] However, they are susceptible to stress corrosion cracking (SCC) and show a significant increase in crack growth rate in the presence of moisture, especially in the peak-aged and under-aged condition.[2,3] Hydrogen embrittlement has been attributed as a possible mechanism for this increase in crack growth rate.[3,4]

Several studies have looked at the crack growth behavior of aluminum alloys. Discontinuous surface cracks (crack jumps) were observed during SCC of aluminum alloys.[5] These discontinuous surface cracks have also been observed in steel during SCC.[6,7] It was suggested that the cracking is discontinuous where surface cracks nucleate in front of the main crack, grow separately and then combine with the main crack.[5,7] Many studies have used surface measurements (using optical techniques, for example) to quantify SCC behavior.[8–10] This approach may be inaccurate since the crack growth behavior in the interior of the specimen and at the surface may be quite different due to differences in stress state.[11]

It is clear that a thorough understanding of the cracking behavior in SCC requires a quantification of the crack growth behavior in three dimensions (3D). Furthermore, the experiments must be carried out *in situ* to verify whether the ‘crack jumps’ are indeed jumps. X-ray tomography is an excellent technique to study

the behavior of materials under mechanical loading in 3D and 4D (the fourth dimension here is time). In particular, *in situ* mechanical testing is more promising where visualization and quantification of the microstructure changes can be performed with time. *In situ* experiments under tension,[12] cyclic loading,[13,14] corrosion-fatigue,[15] and SCC [16–18] have been conducted using X-ray tomography.

In this study, we have used X-ray synchrotron tomography to study the crack growth behavior of under-aged AA7075 in moisture. We were able to unequivocally determine whether the SCC cracks were discontinuous or simply a single tortuous crack. The SCC crack growth rates were measured and compared using the surface measurement (2D) and bulk measurement (3D). By using the *in situ* tomography approach it was possible to quantify the evolution of crack growth under SCC.

X-ray synchrotron tomography was performed at the 2-BM beamline of the Advanced Photon Source (APS) at Argonne National Laboratory. The details of the tomography system at 2-BM have been described elsewhere.[19,20] A monochromatic beam with energy of 24.3 keV was focused on the specimen. A LuAG:Ce scintillator screen was used to convert the transmitted X-rays to visible light. This was coupled with a 5× objective lens and a CCD camera (PCO Tech edge camera, Romulus, Michigan) to achieve pixel sizes of 1.3 μm. 2D projections were collected at an angular increment

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of 0.12° for a total of 1500 projections. These 2D projections were then reconstructed using the Gridrec fast Fourier transform-based algorithm. The grayscale images obtained after reconstructions were segmented using conventional thresholding using image analysis software (ImageJ, Bethesda, MD). 3D rendering of these segmented SCC cracks was performed in Avizo® Fire (VSG, Burlington, MA) for visualization purposes.

A commercially available AA7075-T651 was solution-heat-treated at 475°C for 2 h followed by quenching in water and then aging for 80 min at 120°C to obtain an under-aged condition (Vickers hardness of $\sim 138\text{ HV}$). Single-edge-notch specimens ($20\text{ mm} \times 2.8\text{ mm} \times 0.8\text{ mm}$) were machined by electro-discharge machining such that the loading direction was along the short-transverse (S-T) orientation. The longest dimension (20 mm) is along the loading direction and the lowest dimension (0.8 mm) is the thickness. A width of 2.8 mm was chosen due to limited field of view ($\sim 3\text{ mm}$, necessary to achieve a $2\text{ }\mu\text{m}$ resolution at the APS). The thickness of 0.8 mm was chosen on the basis of two factors: The ability to easily nucleate a crack on the microforce testing system (MTS Tytron 250) having a maximum capacity of $\sim 240\text{ N}$ and the ability to observe the long growth of the crack while maintaining the diagonal dimension of the sample to be less than 3 mm. Fatigue precracking was performed *ex situ* on a microforce testing system (MTS Tytron 250) in tension–tension fatigue (4 Hz, $R = 0.1$). The precrack length was about 1 mm. The SCC experiments were performed in the *in situ* loading stage as shown in Figure 1(a). The upper grip was attached to the stepper motor with a linear actuator, which imposes a displacement on the sample to achieve the desired load. The linear actuator was capable of $8\text{ }\mu\text{m}$ per step and a total stroke of 25 mm. The load cell had a capacity of 500 N. The load was transmitted from the top of the stage to the bottom through a polymer sleeve (Poly methyl methacrylate or PMMA). The PMMA material was chosen due to its transparency to X-rays. The actuator can

be controlled automatically using feedback from the load cell.

A schematic of the *in situ* loading stage to perform the SCC experiments in moisture is shown in Figure 1(b). The PMMA sleeve has not been shown here for clear visualization of the arrangements inside the stage. The sample was loaded on both sides using grips attached to the actuator on one side and the load cell on the other. To introduce moisture into the cell, an annular (ring shape) wet sponge was placed around the load cell at the base of the loading stage. Since SCC experiments require long exposures to moisture, maintaining a constant humidity inside the chamber is a big challenge as fluctuations can lead to variability in results. In order to minimize the loss of moisture and to maintain constant relative humidity, the top part of the loading stage was covered with a plastic wrap as shown in Figure 1(b). A humidity sensor was placed in front of the notch to measure the relative humidity throughout the test. These arrangements led to the constant humidity of 95–96% inside the system throughout the test. Samples were placed inside the deionized water for about a week before starting the SCC test in moisture at Argonne National Laboratory.

Figure 2 shows the 3D rendering of the SCC crack in moisture, where each color represents time (cumulative) during which the stress corrosion cracks grew at a constant load of 110 N. Two independent cracks (not connected in 3D) were observed, where the bottom crack almost covers the entire thickness. It is interesting to note that there is a significant variation in the crack length through the thickness and therefore variation in crack growth rate.

The discontinuous cracks on both sides of the specimen can clearly be seen from the 2D slices obtained from X-ray tomography after about 25 h of SCC, as shown in Figure 3(a) and 3(b). These 2D images indicate that the second crack might have initiated in front of the first crack and then grew separately. However, 3D rendering of the crack in Figure 3(c) and 3(d) actually show that these discontinuous surface cracks were connected in three dimensions. Figure 3(e)–(i) shows the step-wise growth of the crack and the appearance of discontinuous surface cracks, observed in Figure 3(a). The time interval is shown in the legend. It can be seen that the main crack grew from inside toward the surface (Figure 3(e) and 3(f)) and finally appeared as a discontinuous crack on the surface (Figure 3(g)), and then continued to grow. The appearance of discontinuous surface cracks can clearly be seen in Figure 3(j), which was not in Figure 3(i). Both, Figure 3(i) and 3(j) are the top views of Figure 3(f) and 3(g).

The crack growth rates were measured using the average of the crack lengths at both sides (2D measurement) and also the average crack lengths through thickness (3D measurement), as shown in Figure 4. Using 2D slices of the 3D crack, the crack length increment (da) was measured. This increment in crack length was then divided

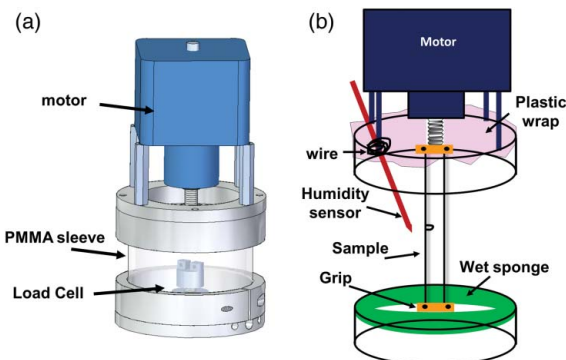


Figure 1. (a) *In situ* mechanical loading stage, and (b) schematic of *in situ* stage for SCC experiments in moisture. The PMMA sleeve is not shown in (b) to show the arrangements clearly.

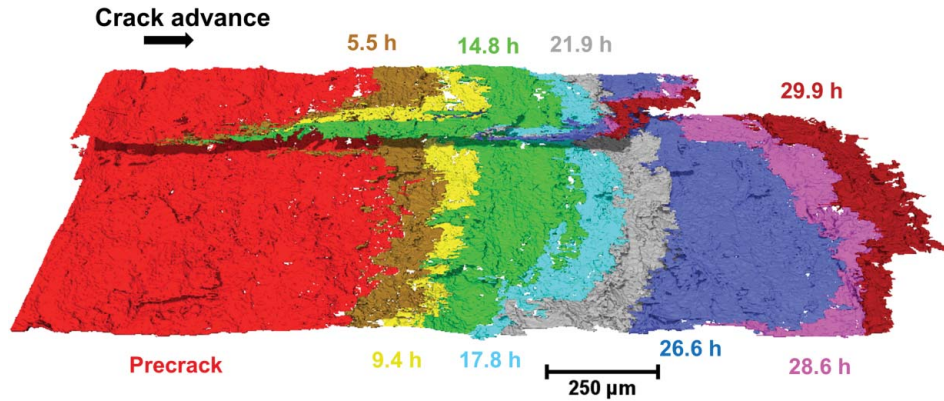


Figure 2. 3D SCC crack growth profile in moisture as a function of time. Significant variation of crack length through the thickness was observed.

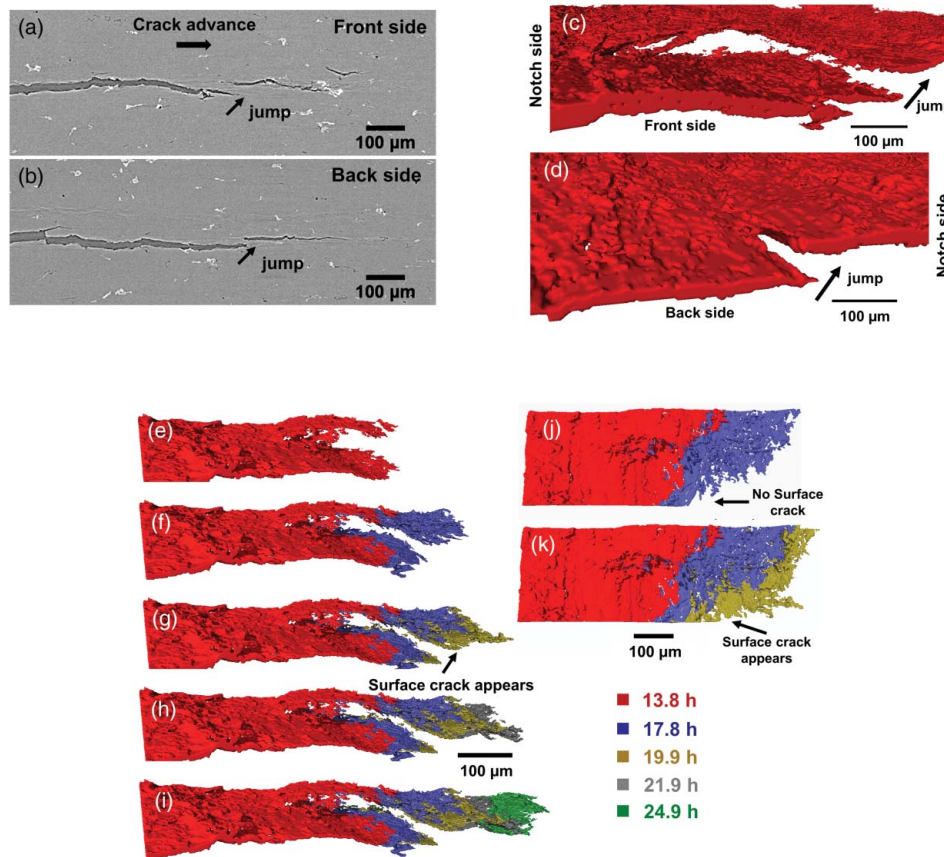


Figure 3. (a) and (b) are 2D slices of the sides of the specimen showing the discontinuous cracks (crack jump), (c) and (d) are the 3D reconstructions of part of the crack showing that the discontinuous crack at the surface are actually connected in 3D, (e)–(i) step wise growth of crack showing the appearance of the discontinuous crack (g) and then further growth, (j)–(k) top view of (f) and (g) showing that the surface crack does not appear in (j) but appears at (k).

by the time increment (dt) to obtain the crack growth rate. The same time interval (dt) was used for both types of measurement. It can be seen that the crack growth rate has a significant amount of variability when surface crack lengths were used for the measurement (2D) compared to the growth rate when the average crack length through thickness was used (3D). Moreover, a large deviation in the surface crack lengths resulted in significant differences in stress intensity factor, as shown as error

bars in Figure 4. These can be attributed to the highly non-uniform growth of the SCC crack at the surface which includes formation of discontinuous cracks and independent growth of the two cracks. The stress intensity factor (K) was calculated using the equations derived by Ahmad et al. [21] for single-edge-notched specimens with fixed grip loading. Since the SCC experiment was performed at constant load, the stress intensity factor increases with an increase in the crack length. For a

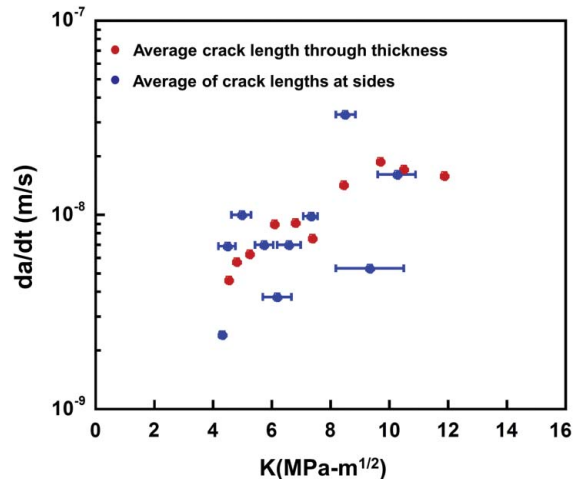


Figure 4. Crack growth rate measurement from average of the crack lengths (2D) and average of the crack length through thickness (3D). The 3D crack growth rate is quite uniform than that of the measurement based on 2D.

particular crack growth rate value (da/dt), the stress intensity factor value (in Figure 4) was taken as the average of the stress intensity factor values corresponding to the initial and final crack lengths for that particular time interval.

In summary, using the X-ray synchrotron tomography, we have shown that the discontinuous surface cracks formed during SCC of AA7075 in moisture were actually a single crack connected in three dimensions. These discontinuous surface cracks were formed when the internal main crack grew from inside the specimen and appeared on the surface. The crack growth rates as determined by measured crack lengths at the surface (2D) led to highly variable crack growth rates than when measured using average crack lengths through thickness (3D). The results obtained here show the importance and necessity of 3D techniques, such as X-ray synchrotron tomography, to study the crack growth behavior during SCC.

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