

## Multimodal 3D Time-Lapse Studies of Corrosion Pitting and Corrosion-Fatigue Behavior in 7475 Aluminum Alloys

Tyler Stannard<sup>1</sup>, Hrishikesh Bale<sup>2</sup>, Thomas Chengattu<sup>1</sup>, Sridhar Niverty<sup>1</sup>, Jason Williams<sup>1</sup>, Xianghui Xiao<sup>3</sup>, Arno Merkle<sup>2</sup>, Erik Lauridsen<sup>4</sup>, Nikhilesh Chawla<sup>1</sup>

<sup>1</sup>. Center for 4D Materials Science, Arizona State University, Tempe United States.

<sup>2</sup>. Carl Zeiss X-Ray Microscopy, Pleasanton United States.

<sup>3</sup>. Advanced Photon Source, Argonne National Laboratory, Lemont United States.

<sup>4</sup>. Xnovo Technology ApS, Koege Denmark.

Aluminum alloys are frequently exposed to harsh chemical and mechanical environments in service. For example, naval aircraft are exposed to corrosive saltwater spray when parked on the aircraft carrier deck and then fatigue loads during flight. These complex service environments are conflated with microstructural material variations, complicating the creation of models for the prediction of aircraft and leading to unpredicted component failures. Numerous factors including the material impurities, defects, precipitation hardening parameters, and crystallographic orientation of the grains can play a significant role in corrosion-related fracture of high strength aluminum alloys [1]. In order to create the most accurate models possible, four-dimensional (three dimensions + time) studies of the alloys are required. Ideally every microstructural, chemical, and mechanical detail would be known about a sample for the most accurate understanding possible. This work presents the latest efforts in achieving 3D microstructural information.

Specifically, non-destructive tomography-based studies were employed to study the failure mechanisms of the high strength aluminum alloys. The first study involved soaking polished peak-aged 7475 samples in uncovered 3.5 wt.% NaCl solution for fifteen days, then fatigue testing them *in situ* in solution in the Advanced Photon Source synchrotron at Argonne National Laboratory. The *in situ* test data showed that the corrosion-fatigue cracks initiated at pre-existing “mud cracks” in the corrosion products of the corrosion pits. Moreover, corrosion bubble evolution during the fatigue test was monitored by performing 3D tomography scans at multiple loads within the fatigue cycles. The changes in the bubbles were analysed in 3D to better understand synergistic corrosion-fatigue mechanisms. Figure 1 shows preliminary results of the 4D crack growth analysis.

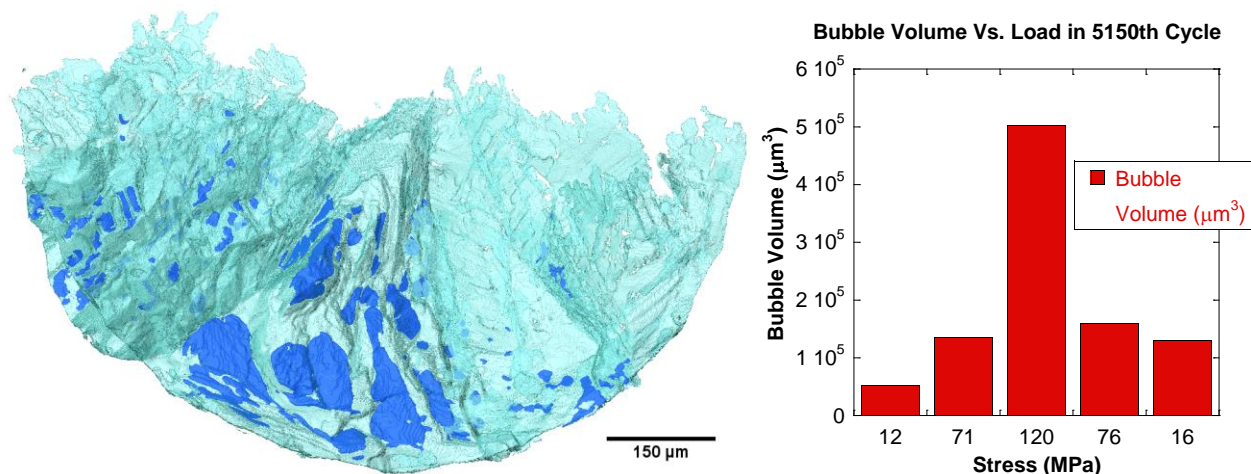
Additionally, most corrosion pits were found to grow surrounding the white iron-bearing inclusions. This was expected since the iron-bearing inclusions are cathodic with respect to the aluminum matrix, leading to preferential dissolution of the matrix surrounding the inclusions via microscopic galvanic corrosion reactions. Surprisingly, corrosion pits only formed around a fraction of the iron-bearing inclusions near the sample surface. Therefore, a time-lapse study examining both 3D phase information and 3D crystallographic information was employed to further probe the underlying corrosion mechanisms. The 3D crystallographic information was captured non-destructively on a pristine, gently mechanically polished Al7475 sample using a newly developed technique called Laboratory-based diffraction contrast tomography (DCT) [2]. The non-destructive nature of the technique allowed further experimentation in which the same sample was submerged in 3.5 wt. % NaCl solution and periodically tomography scanned. The combined diffraction contrast tomography and absorption tomography provides comprehensive information of the grains containing orientation,

grain volume and morphology, and location of impurity inclusions within the samples before and after corrosion. With this novel multimodal 3D time-lapse data, the interactions between corrosion pits, grain boundaries, grain orientations, and inclusions were analyzed. Figure 2 shows a comparison of the DCT grain map reconstruction results, along with absorption scans before and after. Comparing the corroded scan with the grain map, it appears that two near-surface inclusions in different grains may have different corrosion rates.

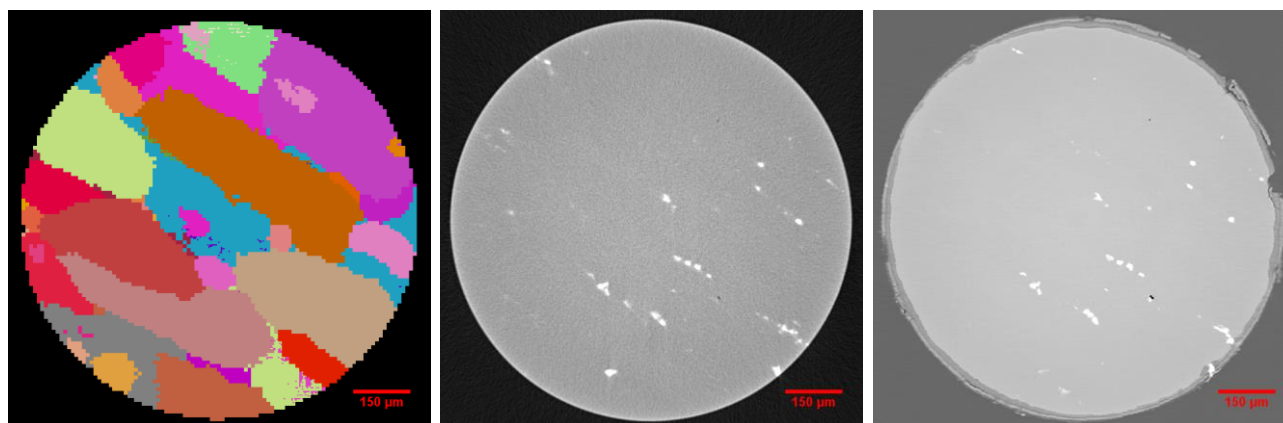
References:

[1] S.S. Singh, *et al*, Mater. Charac., **118** (2016), 102.

[2] C. Holzner *et al*, Microscopy and Microanalysis **22**: (2016), 1970.



**Figure 1.** (Left) Top-down view 3D rendering of a fatigue crack in a peak-aged 7475 sample showing the crack in light blue and the bubbles in dark blue after 5150 cycles of fatigue in 3.5 wt.% NaCl solution at R=0.1,  $\sigma_{\max}$ =120 MPa. (Right) Bubble volume within the crack vs. applied stress in the 5150th fatigue cycle for the same sample.



**Figure 2.** (Left) Virtual slice of a DCT reconstruction of the peak-aged 7475 sample before corrosion with each grain in a different color. The grain boundaries can be clearly distinguished using the DCT technique and Xnovo Grainmapper v2.0 DCT reconstruction software. (Center) Absorption tomography virtual slice of the same section before corrosion. (Right) Absorption tomography virtual slice of the same section after 60 days corrosion in 3.5 wt.% NaCl. Note the near-surface inclusions showing different depth of corrosion.