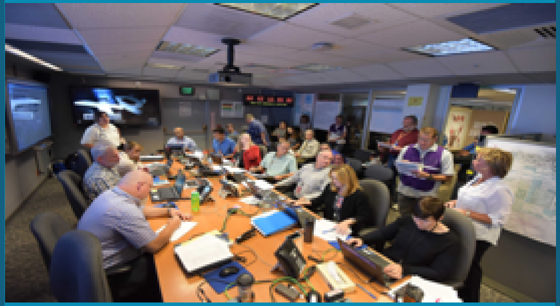
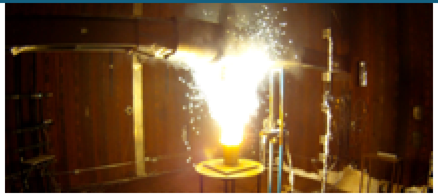
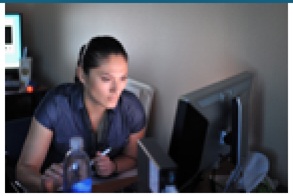
SAND2022-7624C

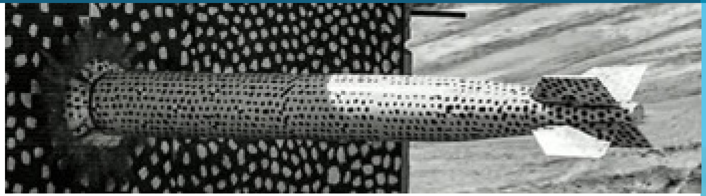
Sandia National Laboratories

Insights from X-ray computed tomography into the effects of pit morphology evolution on pit growth









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U.S. DEPARTMENT OF ENERGY NNSA National Nuclear Security Administration

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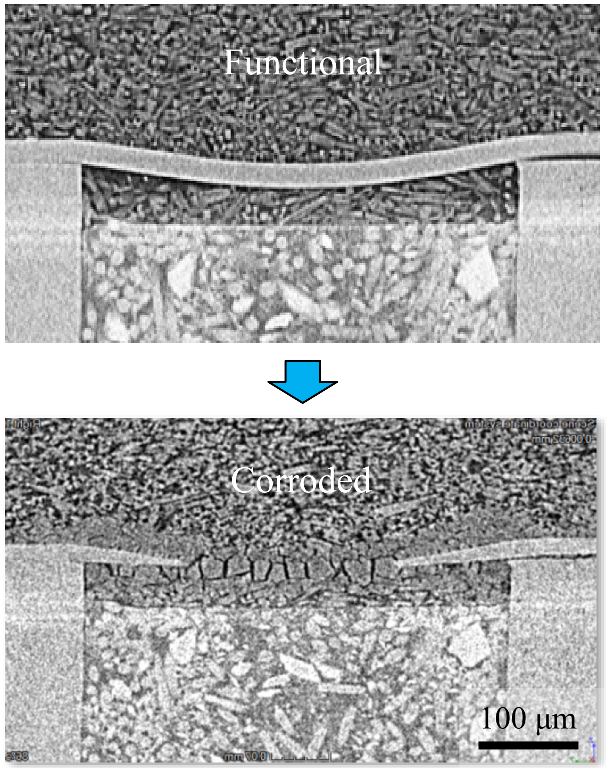
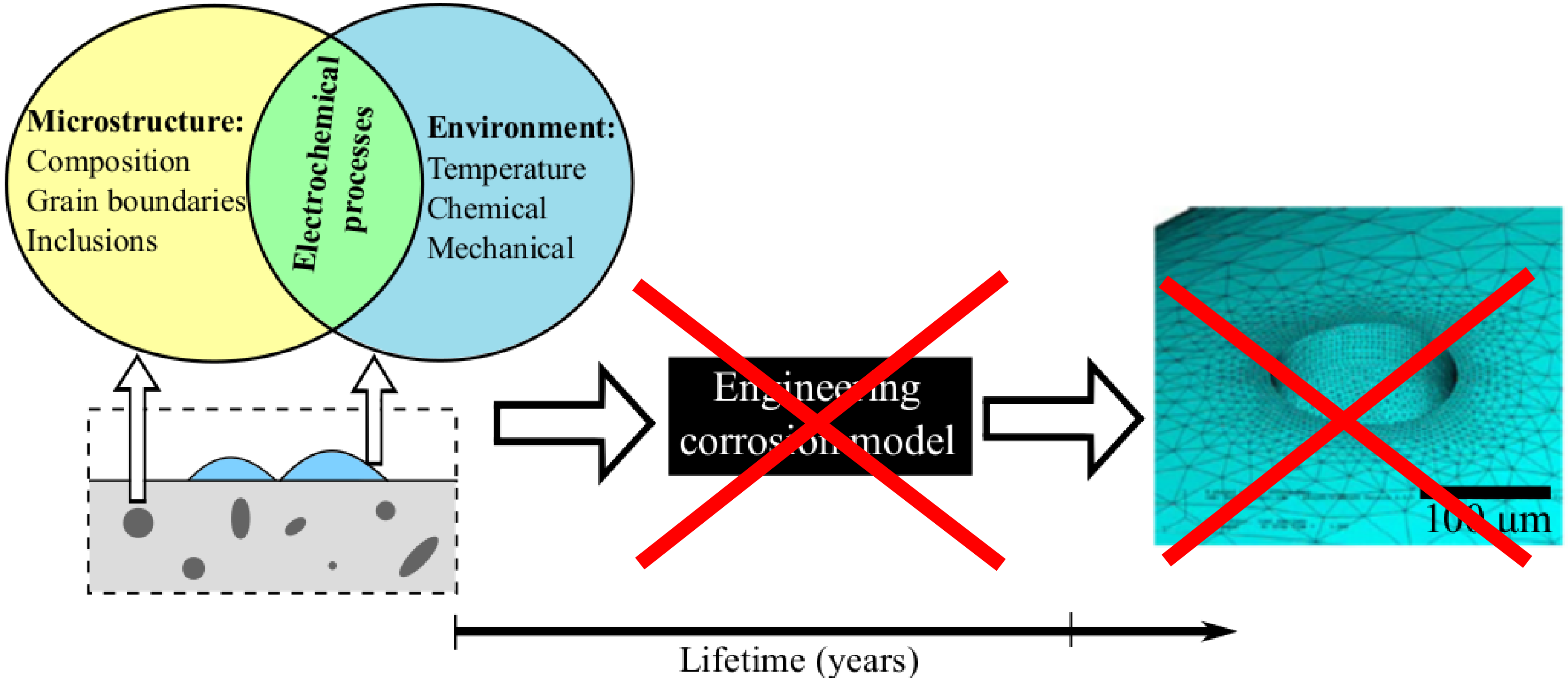
SAND2022-1955 C

What is the useful lifetime of a component in a humid, chloride environment?

The cost of corrosion in the US is estimated at 1-3% of our GDP, ***yet we lack an ability to predict the distribution and morphology of corrosion damage as a function of time***

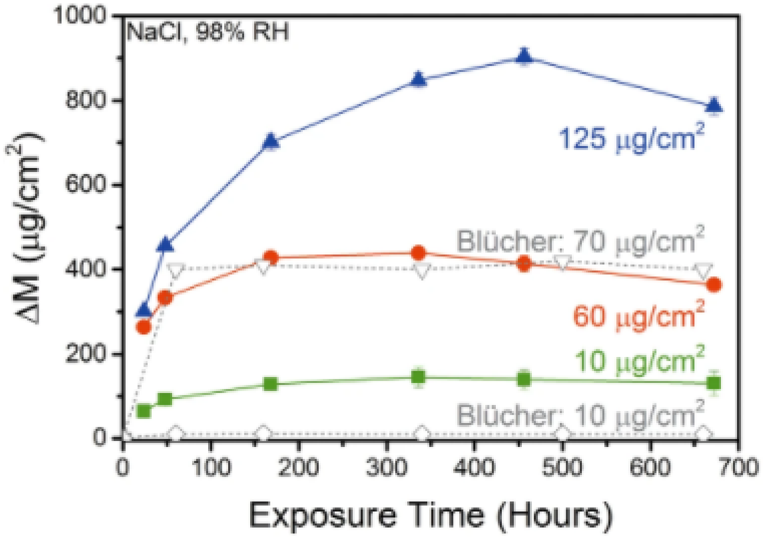


How fast does this happen? Why does it happen in some cases and not others?



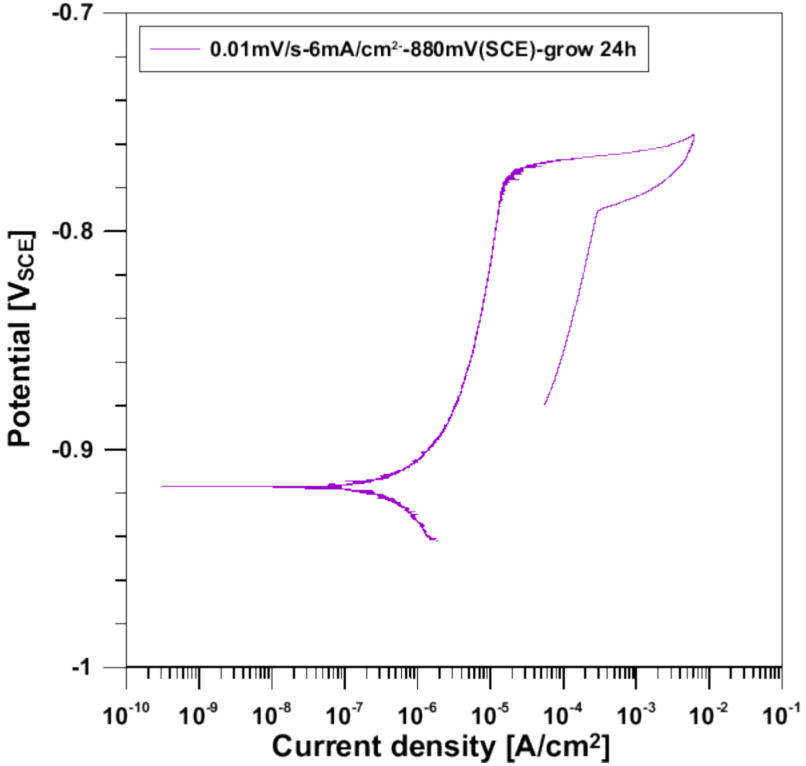
A key challenge for materials science is "the ability to predict the lifetime of metals and components from short-term experimental corrosion data." – E McCafferty, Introduction to corrosion science, 2010

Current approaches to measuring kinetics generally focus on the continuum scale or very controlled environments…

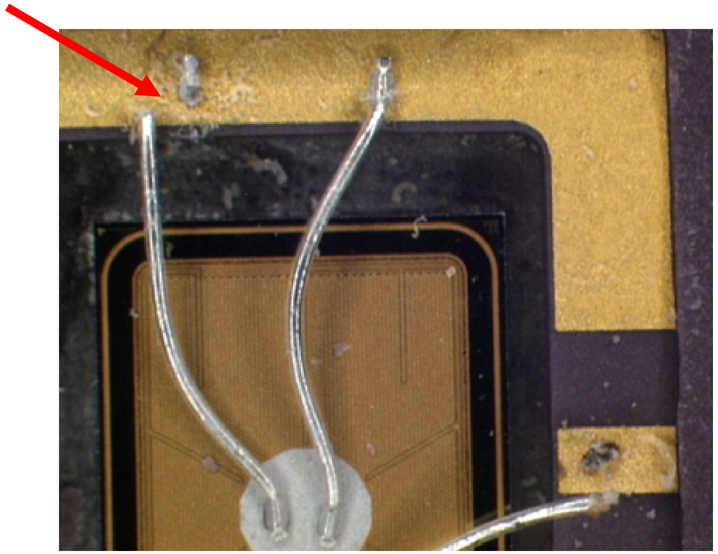


Schaller, R. F., et al. *npj Mat Deg* 2017

Atmospheric test rack from https://www.corrosion-doctors.org/Corrosion-Atmospheric/Corrosion-tests.html



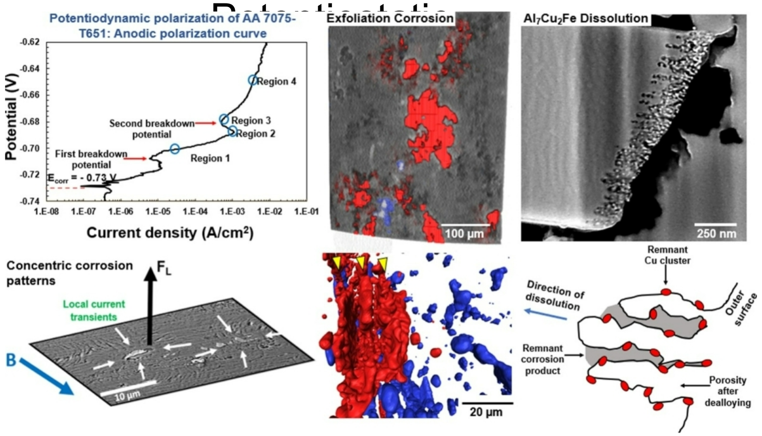
but failure is usually driven by "outliers"



Challenge – we need to characterize the evolving damage in-situ without disturbing the environment
Solution – the field of ductile failure overcame a similar problem using XCT. Why not try this with corrosion?

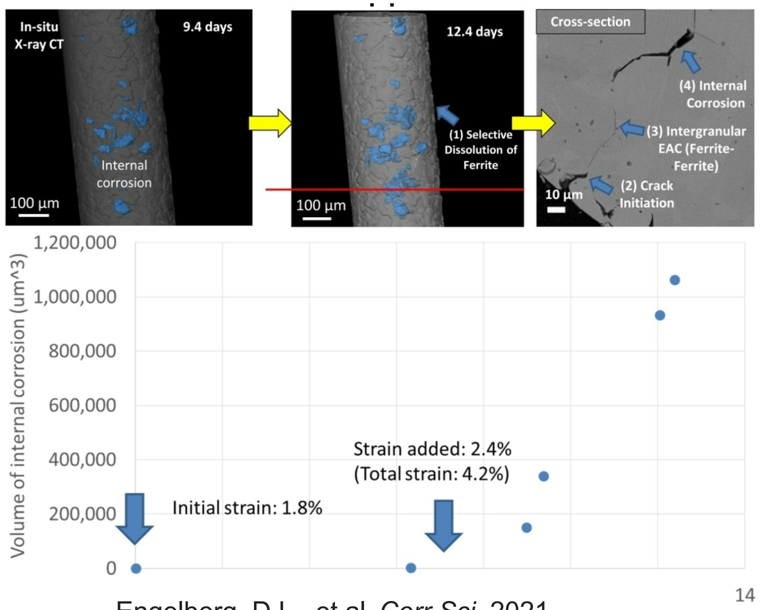
How is XCT being used in the field of corrosion science?

Immersed conditions,



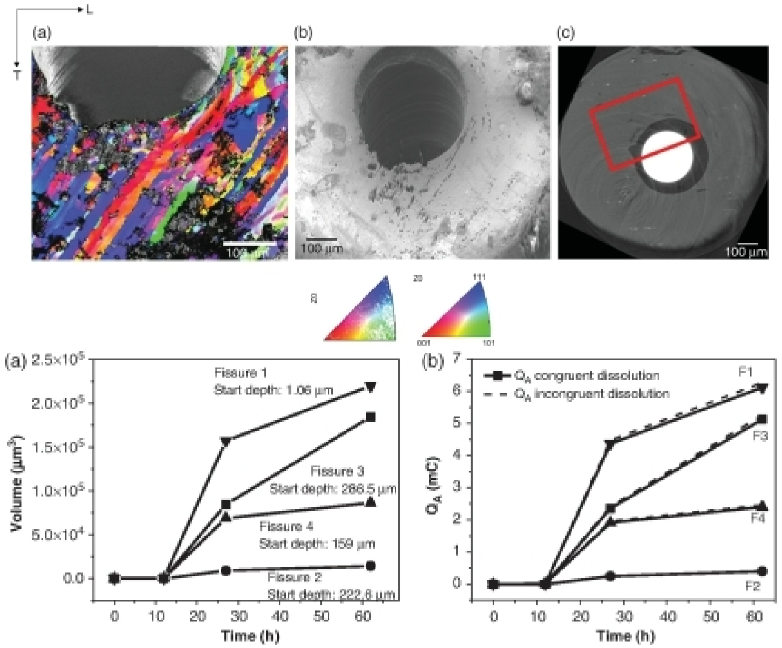
Chawlam, N, et al. *Corr Sci,* 2021

Environmentally-assisted



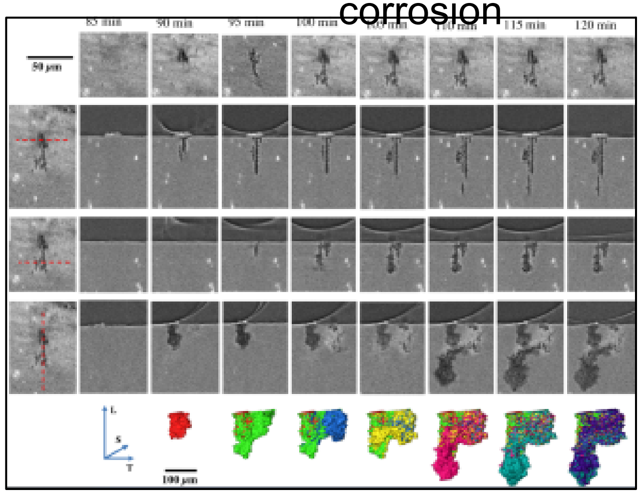
Engelberg, D.L., et al. *Corr Sci,* 2021

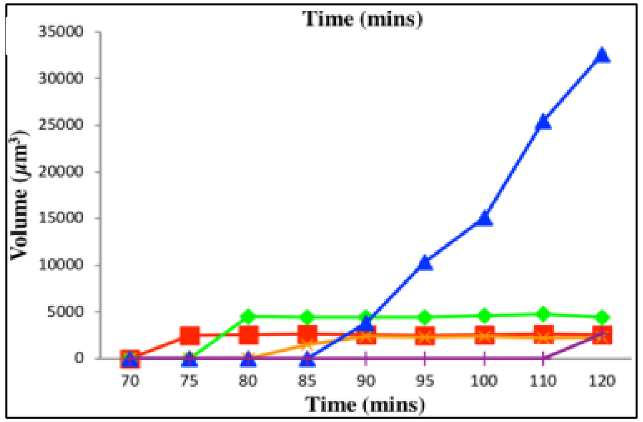
Galvanic Corrosion



Davenport, A.J.., et al. *Corrosion,* 2018

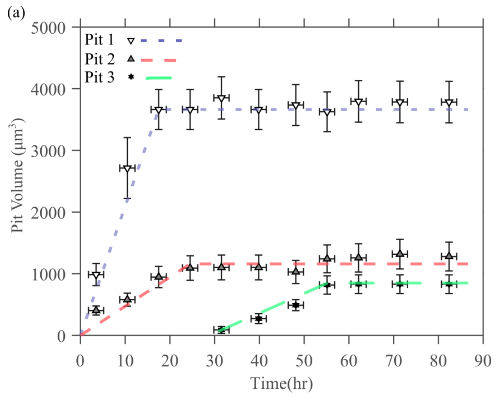
Short-term atmospheric corrosion

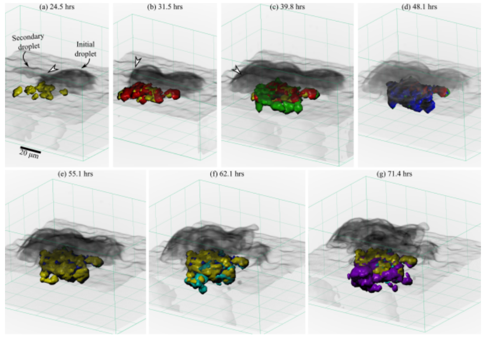




Davenport, A.J.., et al. *npj Mat Deg,* 2021

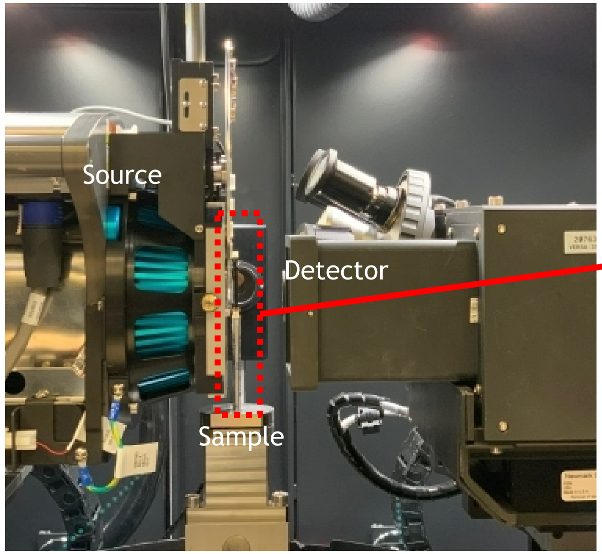
Long-term atmospheric corrosion

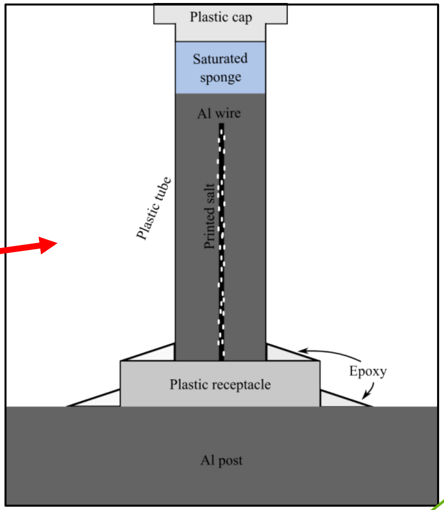


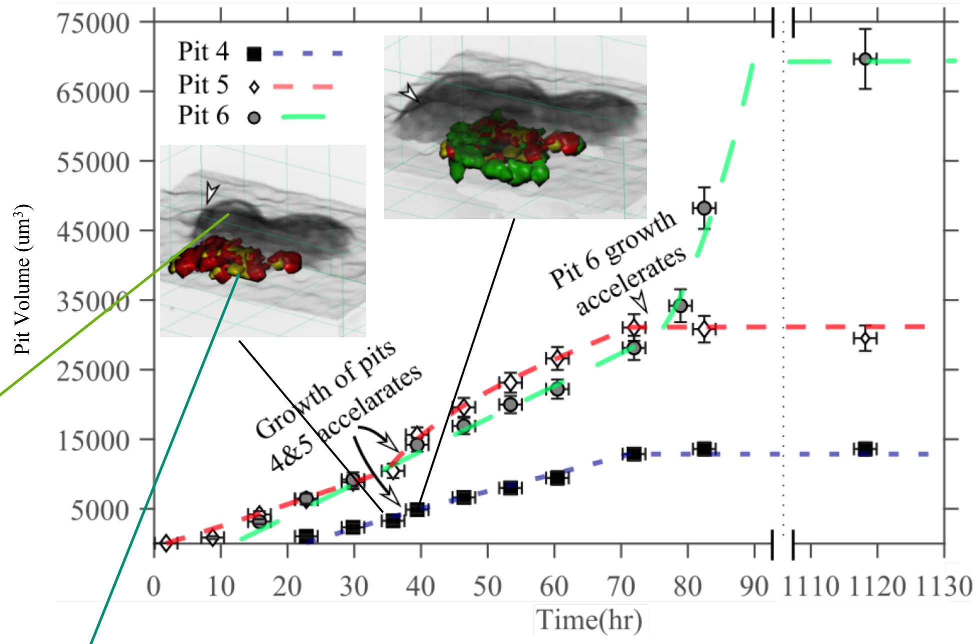


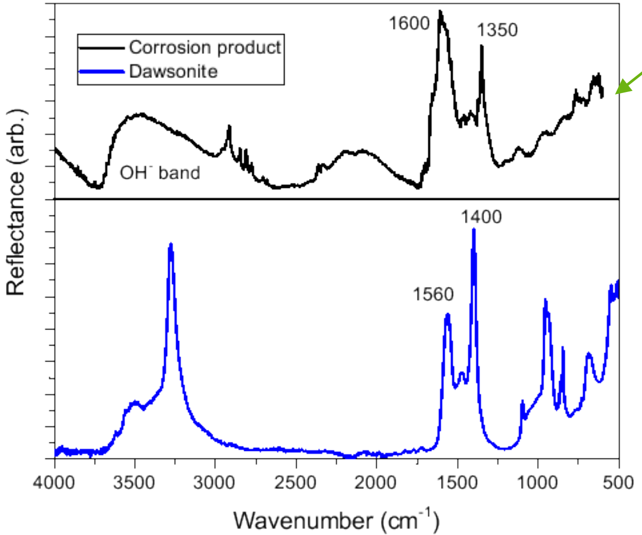
Noell, P.J.., et al. *npj Mat Deg,* 2020

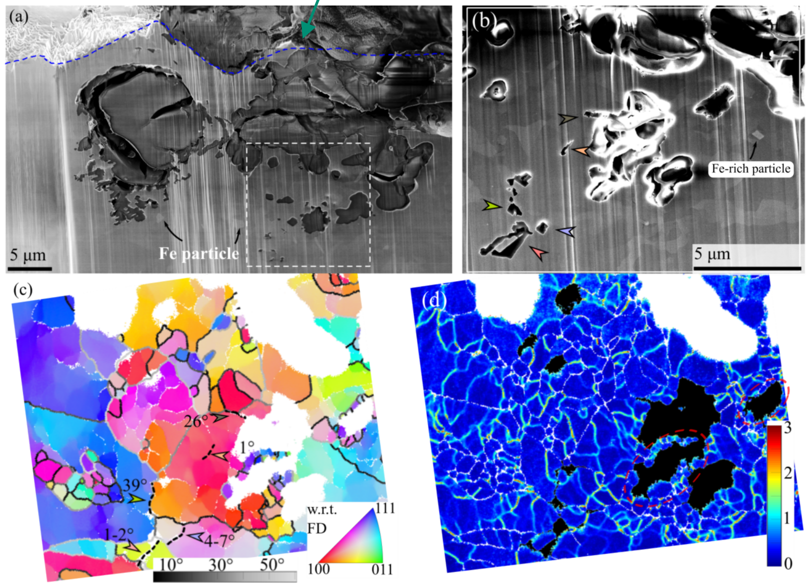
Combining in-situ XCT with ex-situ characterization techniques can provide new insights into pit growth kinetics and controlling factors





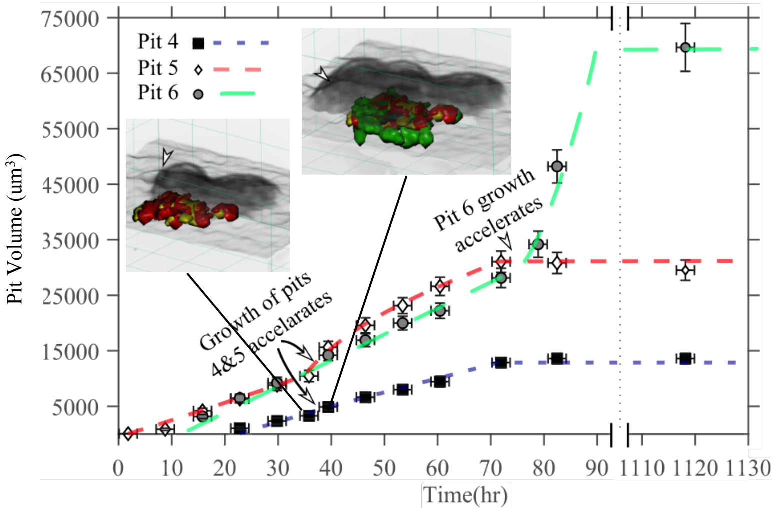


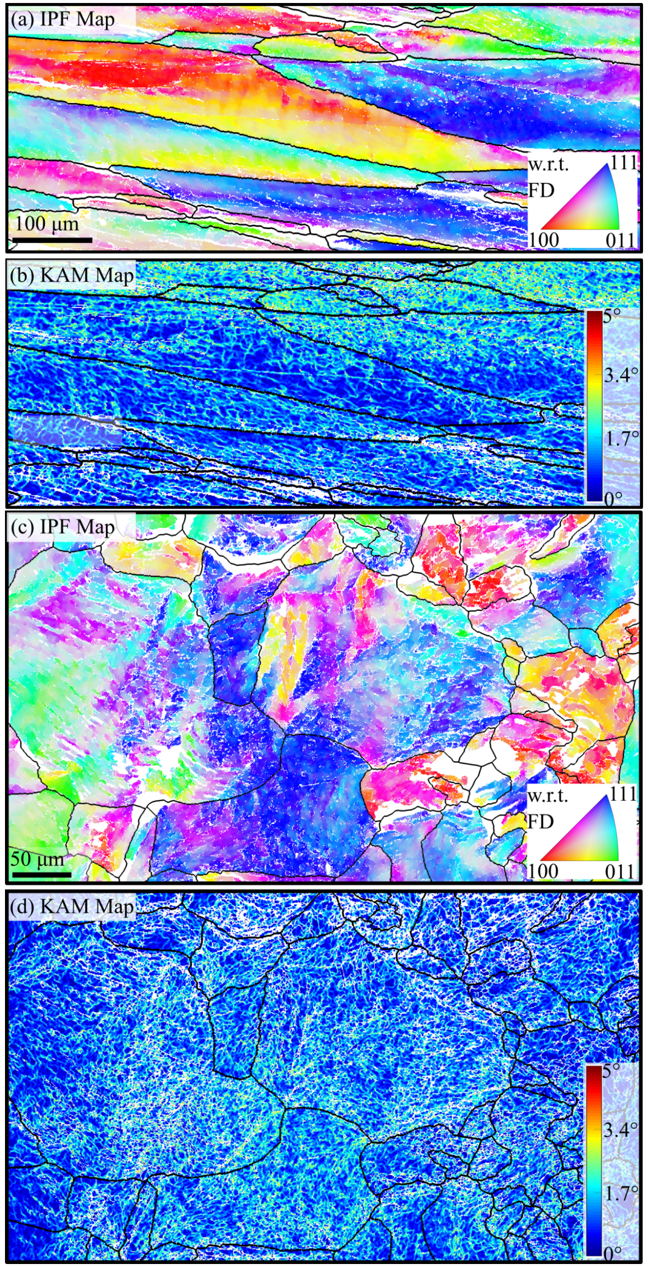


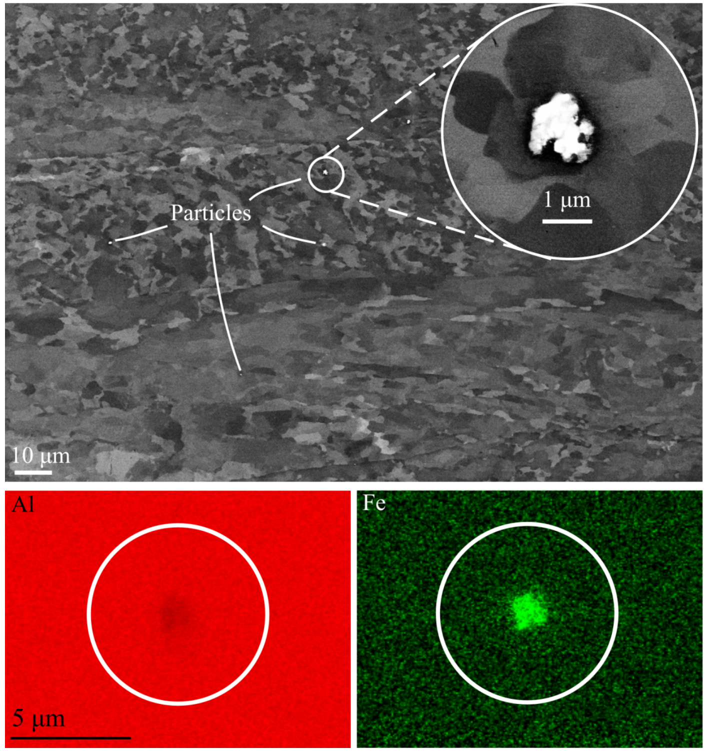


Noell, PJ, et al. *npj Mat. Deg.*, 2020

How do pit growth kinetics evolve in 4N-Al?







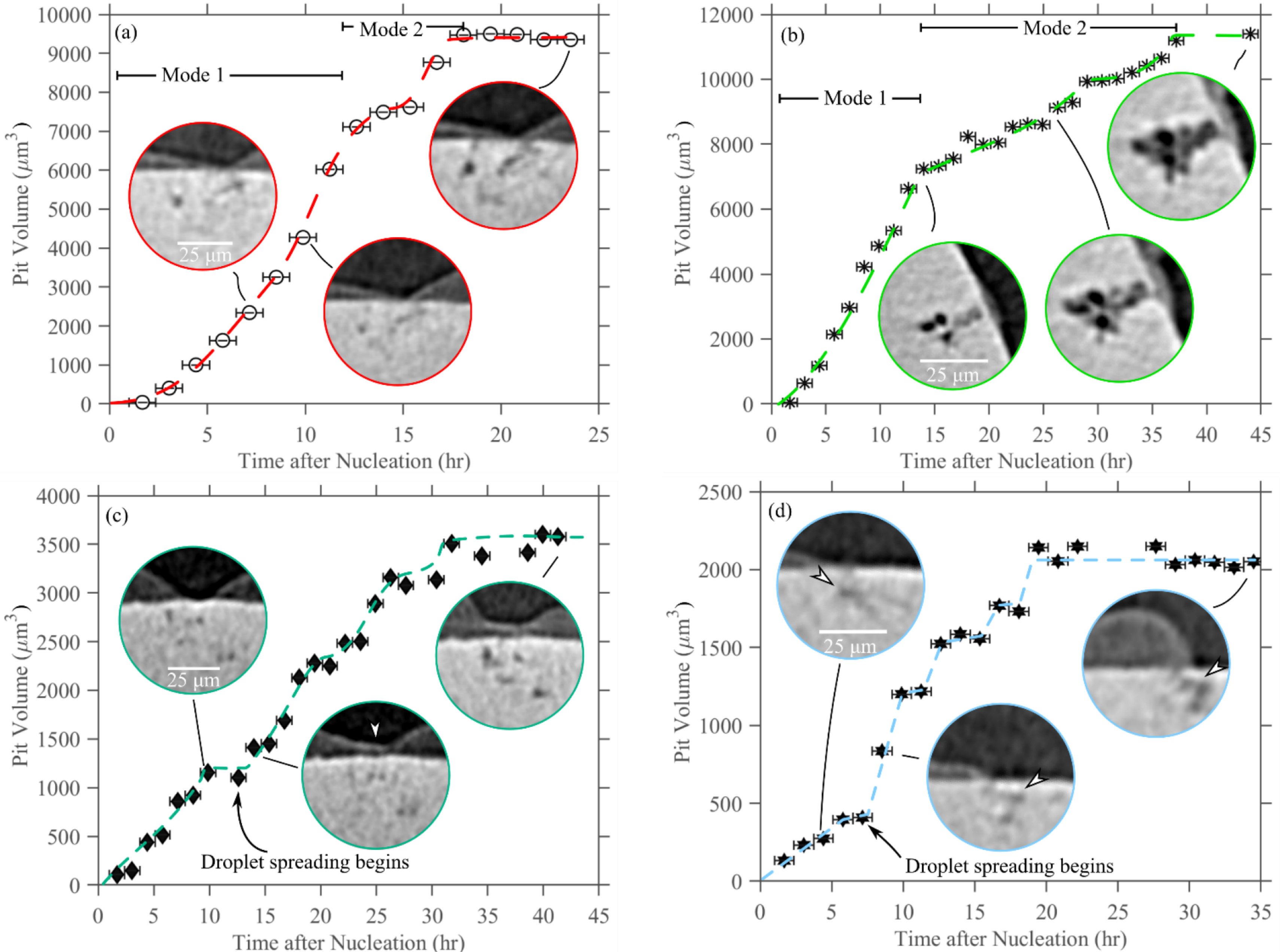
Material and Environment

- Material – 1.02 mm diameter Al wire (99.99% Al)
- Printed with NaCl at 200 ug/cm^2
- Humidity - 84 RH

XCT Characterization Methods

- 1.25 mm length of wire imaged with XCT periodically using a 1.25 um voxel size (15.6 <u>um^3 minimum feature size</u>)
- 4 Samples exposed under these conditions
- Each sample scanned every **1.3 hours** for at least the first 90 hrs. after exposure, then periodically for the next year

Relatively continuous growth is followed by stepped periods



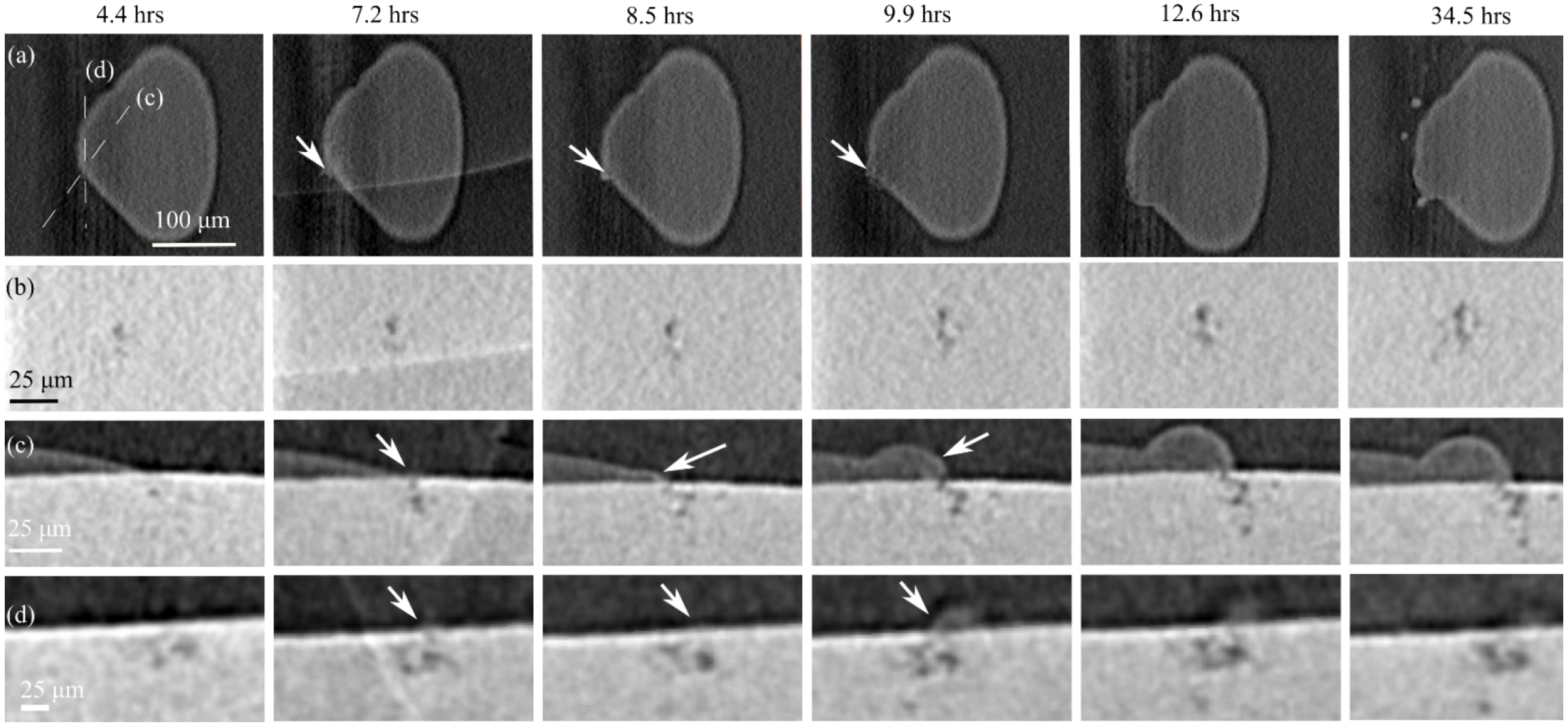
11 pits observed varying in size between ~400 and ~11,000 um^3

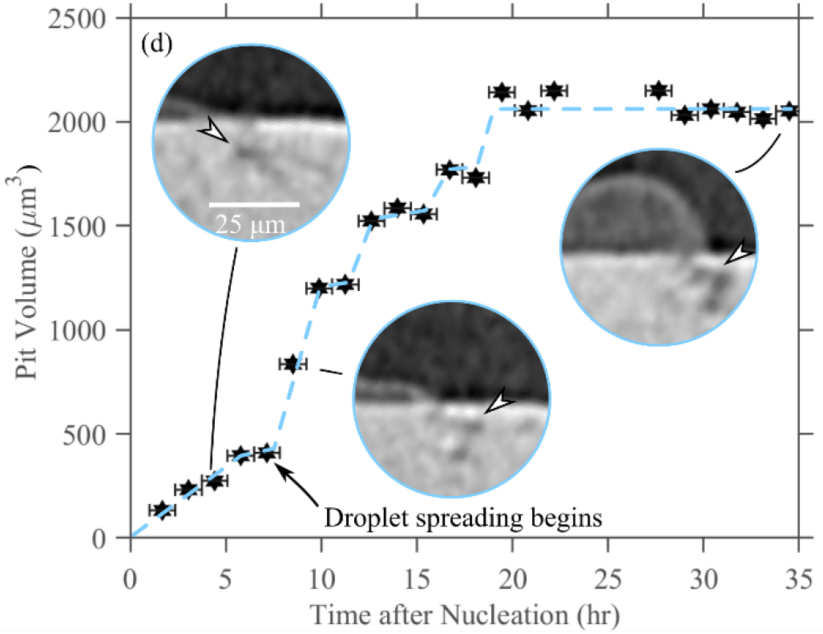
What factors/events are behind this transition from continuous to stepped growth?

Droplet spreading affects pit growth kinetics

	Pit 3	Pit 4	Pit 5	Pit 7
Growth Rate (µm³/hr)	134	61	80	31
Post-Spreading Growth Rate (µm³/hr)	121	115	92	59
Growth duration (hr)	31.8	19.5	30.4	19.5
Final pit volume (µm³)	3601	2166	1856	839

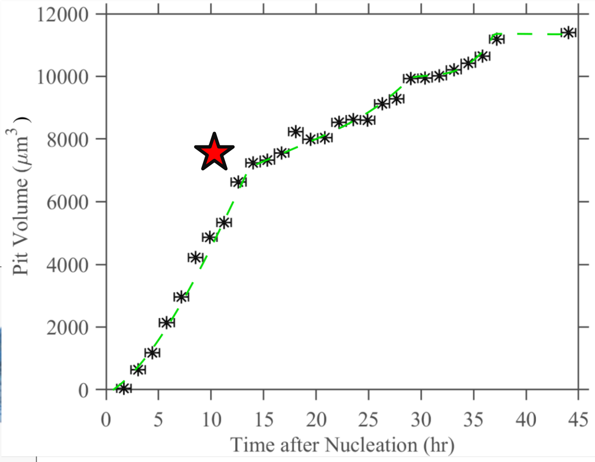
- Droplet spreading, caused by oxygen reduction or metal ion production, was observed for four pit
- This likely increased cathode size and/or local efficiency, changing the rate of pit growth

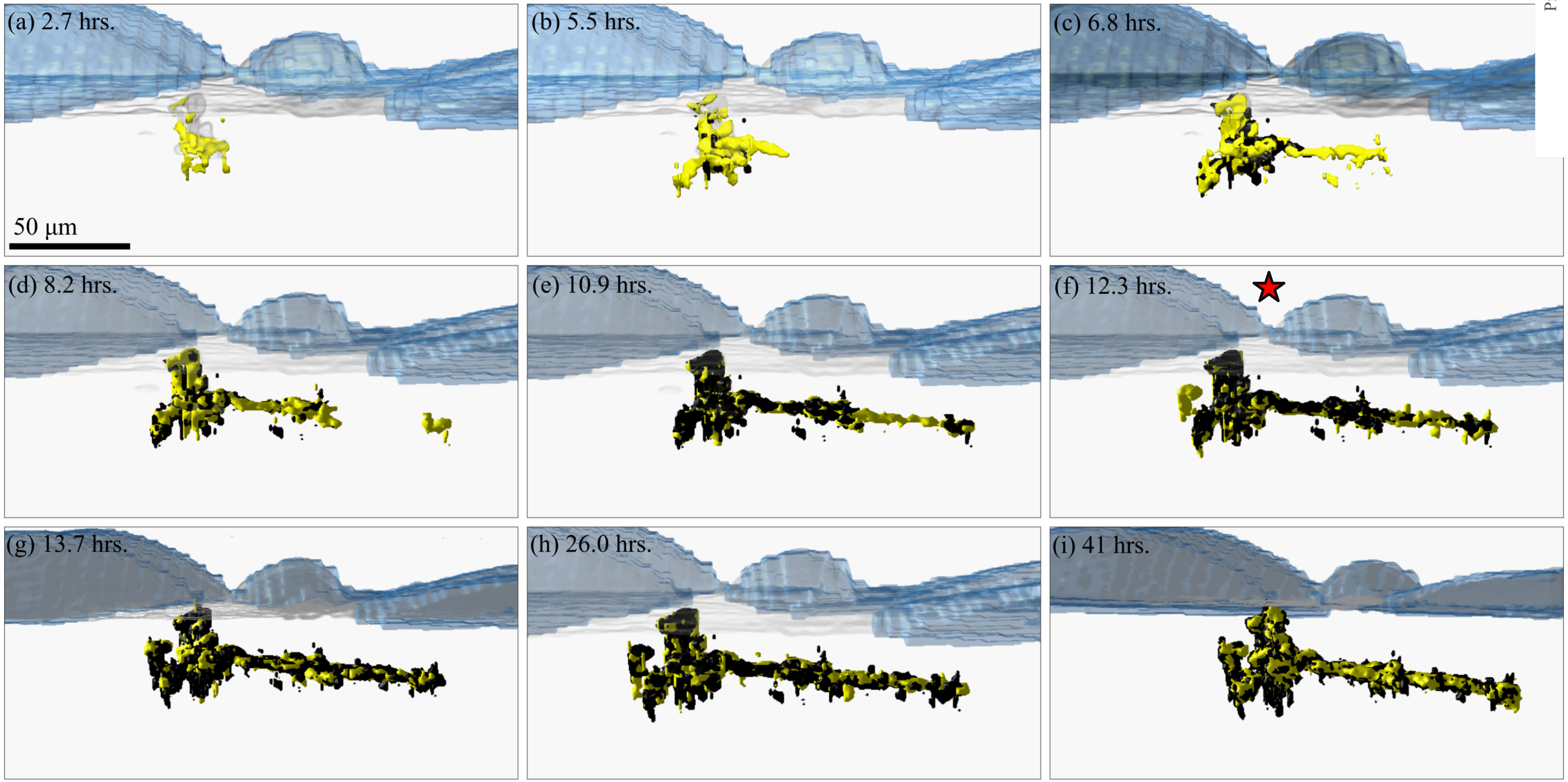




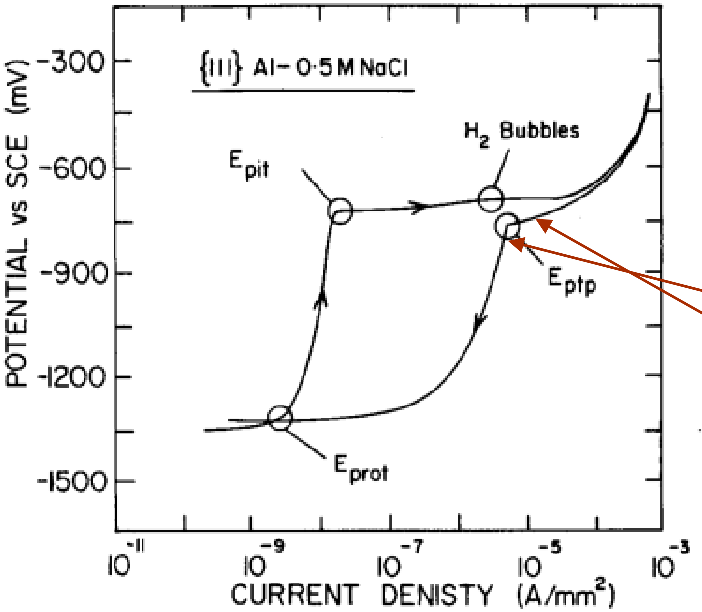
The mode of pit growth can also change, leading to changes in growth kinetics

Mode 1 – pits primarily add volume by some combination of creating new tendrils and the lengthening of pre-existing tendrils.
Mode 2 – existing tendrils expand radially, no new tendrils form

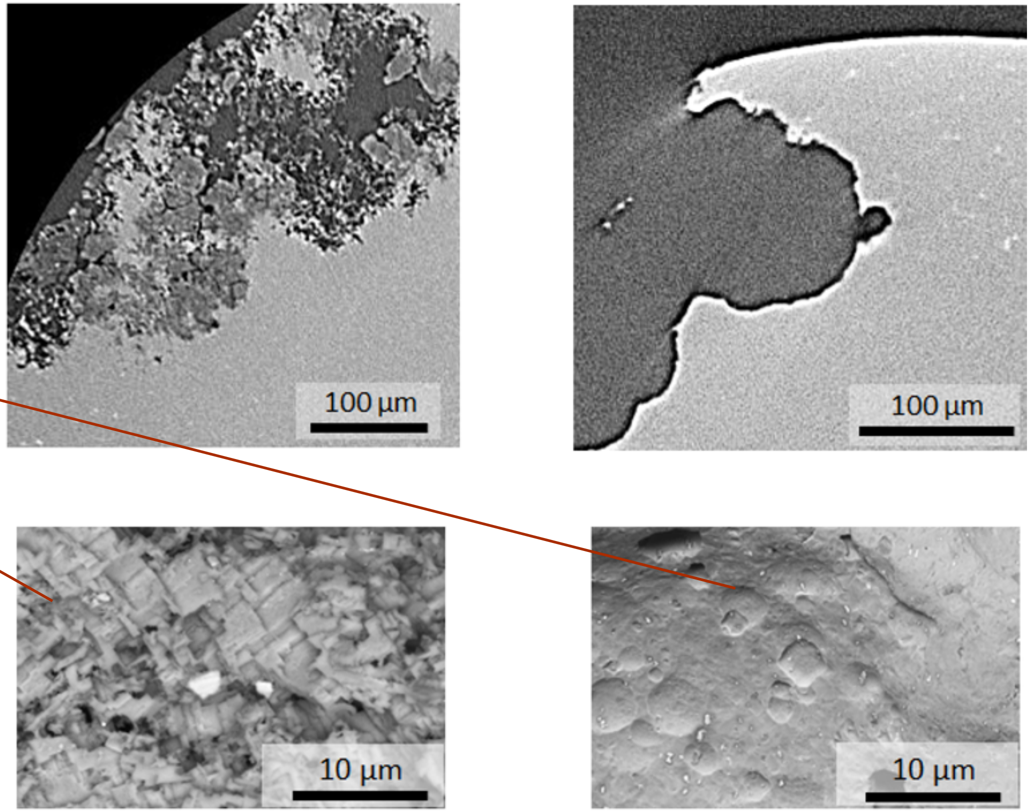




Why two different growth modes?



Yasuda et al., J Echem. Soc, 1990



(a) -740 mV_{SCE} (E_{ptp}+50mV)

(b) -790 mV_{SCE} (E_{ptp})

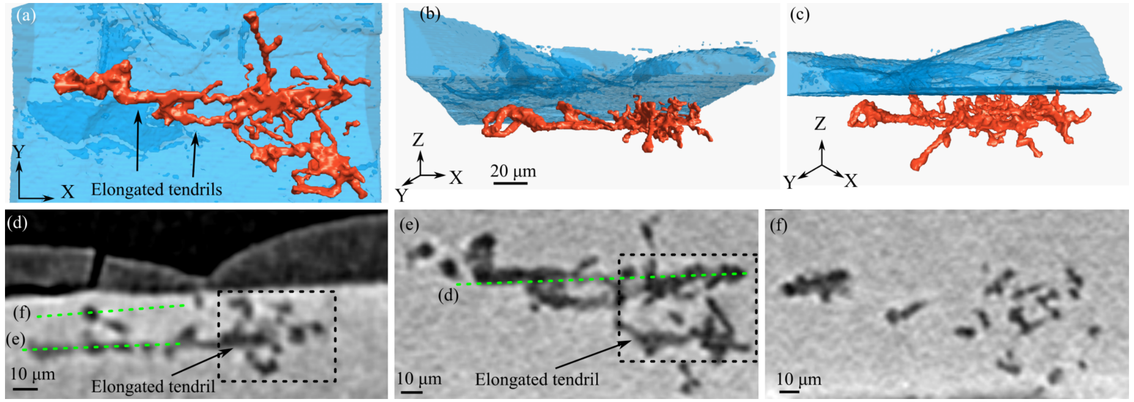
Cyclical polarization experiments show that pitting in Al (and some other passive alloys) is associated with three critical potentials: E_{pit}, E_{ptp}, and E_{prot}

Early work showed that E_{ptp} is related to repassivation but it remains obscure

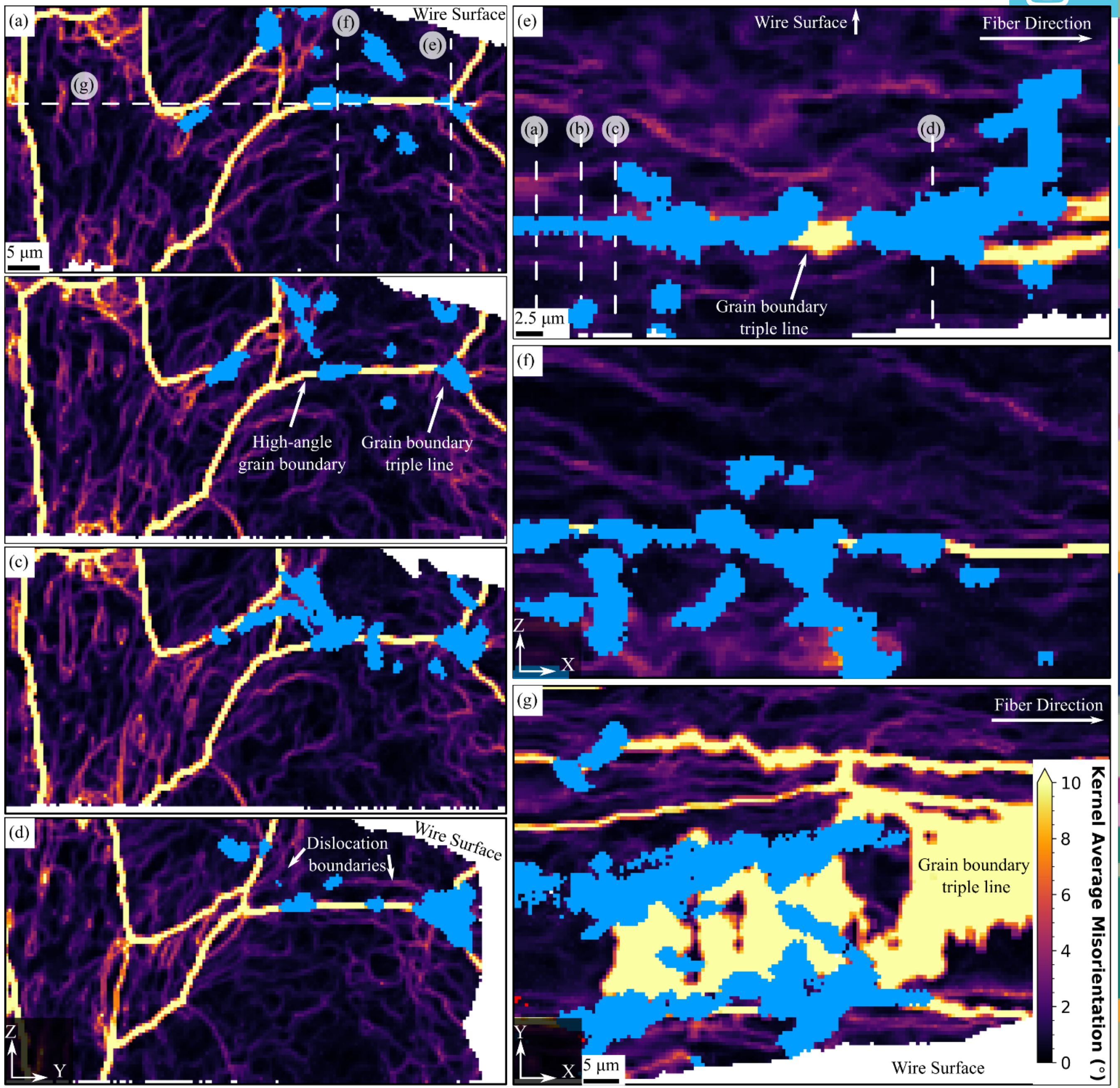
Potentiostatic holds above and below E_{ptp} in a similar Al material show that pit morphology above and below E_{ptp} resembles those observed for mode 1 and mode 2 growth

We speculate that the two growth modes result from the potential within the pit dropping below E_{ptp}

Pit morphology appears to be influenced strongly by the microstructure



- Serial sectioning of a pit after repassivation showed that tendrils grow along grain boundaries and dislocation boundaries
- Large, elongated tendrils followed a grain boundary triple junction and a high-angle boundary



Conclusions

- Droplet spreading altered the rate of pit growth
- Two growth modes identified:
  - Mode 1 – pits primarily add volume by some combination of creating new tendrils and the lengthening of pre-existing tendrils.
  - Mode 2 – existing tendrils expand radially, no new tendrils form
- A clear decrease in the rate of pit growth was observed during the transition from mode 1 to mode 2 growth
- 3D serial sectioning demonstrated that pit morphology strongly depends on local microstructure

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