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Sandia Academic Alliance Spring 2021 Georgia Tech LDRD Virtual Poster Session

20-0050/Correlating pit nucleation to dislocation network and microstructural defects by high-throughput scanning electron microscopy

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Introduction / Motivation

Motivation

- Pitting Corrosion caused by buildup of ions is hard to detect and can catalyze further localized corrosion
- Investigation of internal factors that contribute to pitting propagation is useful to build information database to aid in material selection and design
- High purity aluminum is corrosion resistant and has high electrical conductivity and is commonly used in sputtering and thin films. It also provides a relatively uncomplicated test case for method refinement

Introduction

- Sandia Project Team is using Micro CT scanning to develop 3D tomographs of corrosion pits in unpolished high purity aluminum samples
- EBSD provides microstructural-sensitive input on local influences on pit formation
- The data generated will be used to tune and guide a phase field model currently under development at Sandia

Approach

Sample Preparation

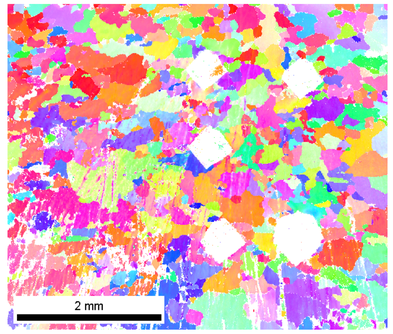
- High purity Al samples prepared for corrosion and EBSD by mechanical grinding, followed by ion milling
- Optical imaging and secondary electron imaging used to determine the initial surface damage state

Experiment

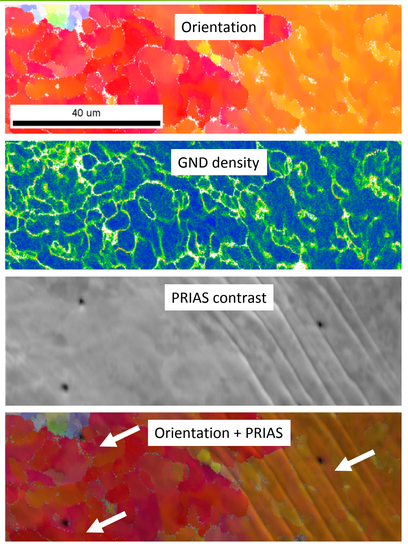
- Samples immersed in 3.0 M NaCl solution and removed at regular intervals of time to detect pit formation and growth

Sample Analysis

- Samples analyzed by EBSD after corrosion to identify orientation, grain boundaries, and local geometrically necessary dislocation density
- PRIAS maps (EDAX/TSL) used to identify pits and correlate with local microstructure



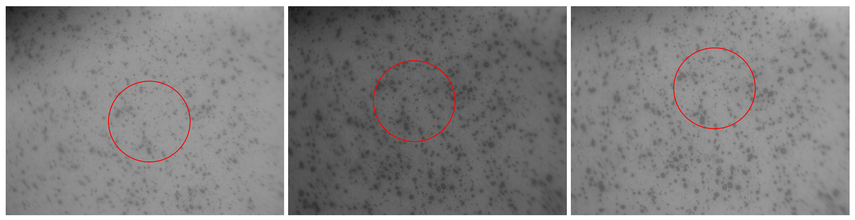
Above: low mag EBSD scan showing microstructure overview
Right: high mag EBSD scan showing defect structure and pits



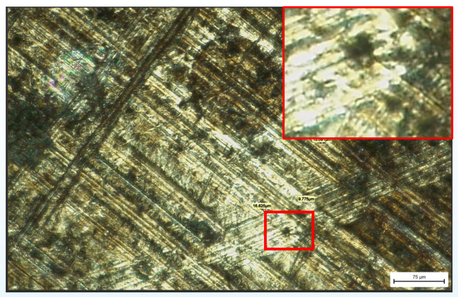
Current Status/ Results (If any)

Status

- Sample Preparation procedure generally defined, needs improvements to minimize surface scratching
- EBSD imaging proved viable for measurements post-corrosion
- Corrosion Pitting observed in as received (not polished) 1100 Al
- No obvious pitting in polished high purity Al or 1100 Al over similar time frames
- Currently performing multiday corrosion experiments on high purity Al

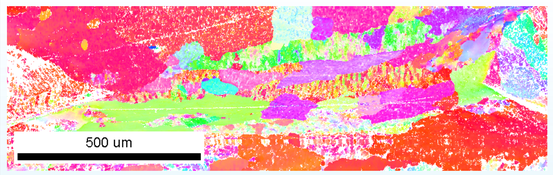


Above: Corrosion over the course of 14 hours of polished 1100 Al showing little to no pitting (50x mag)
Right: Corrosion over the course of 24 hours of as-received 1100 Al showing extensive pitting



Challenges

- Chemical-Mechanical Polishing of Soft Sample without introducing defects remains a challenge, though sample preparation is being standardized
- Differentiating corrosion pits from pre-existing defects
- Establishment of accurate timeline of pit development in polished, high purity materials



EBSD orientation map showing scratches in polished sample

Next Steps/ Future Work

Next Steps

- Continue timed corrosion of polished high purity Al samples
- Refine Sample Preparation procedure to obtain samples with minimal defects
- Use high magnification camera to record corrosion while in solution.
- Once corrosion rates established, run correlation experiments linking corrosion pit formation to orientation, grain boundary proximity, and local GND density

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