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Investigation of Intergranular Phenomena in Σ3^n CSL Grain Boundaries Exposed to Extreme Environments

Christopher M. Barr (1), Gregory A. Vetterick (1), Khalid Hattar (2), Marquis A. Kirk (3), Mitra L. Taheri (1)

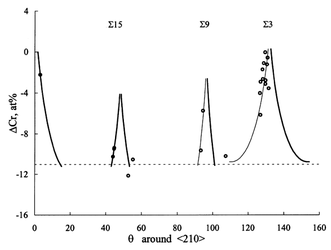
(1) Department of Materials Science and Engineering — Drexel University, Philadelphia, PA
(2) Radiation Solid Interaction, Sandia National Laboratory, Albuquerque, New Mexico
(3) Materials Science Division — Argonne National Laboratory, Argonne, IL

Introduction and Motivation:

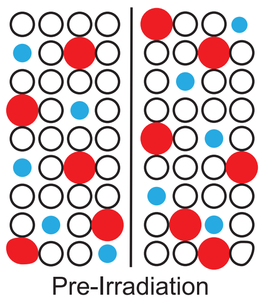
- Examine the role of specific grain boundary structures (Σ3 and Σ9) relevant to grain boundary engineering (GBE) on multiple length scales to two nuclear materials related issues:
  1) Intergranular corrosion (IGC) 2) Radiation Induced Segregation (RIS)
- Study effect of both non-equilibrium thermal segregation and RIS independently and ultimately together with grain boundary type

Project Background:

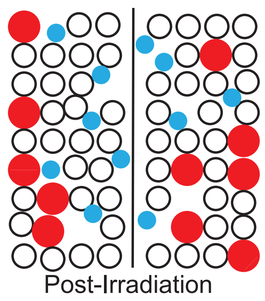
- Understanding and tailoring the GBCD under extreme environments (irradiation, high temperature, and corrosion) can provide improved material performance for Fe-based alloys (e.g 316L)
- Interaction of solutes with specific grain boundary structure under irradiation and non-equilibrium thermal segregation is heavily dependent on the GB structure



Variation in Cr RIS with misorientation angle [1]



Pre-Irradiation



Post-Irradiation

- GBE is associated with the increase in specific low coincidence site lattice (CSL) boundaries (e.g. coherent twins and twin variant boundaries) that break up the HAGB network connectivity.
- GB energy computations indicate a large variation in energy within same Σ value: examine this variation in the environments relevant to nuclear materials

Methodology:

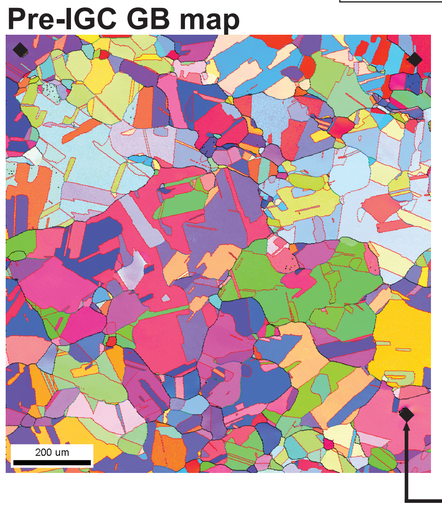
1. GBE: Single-step optimized thermomechanical process: 5% rolling reduction and 1000˚C anneal for 316L

- Meso-scale grain boundary population and connectivity: Σ3n length fraction and triple junction distribution
- Preliminary 5-parameter stereological grain boundary character distribution (GBCD) for grain boundary plane distribution [2]

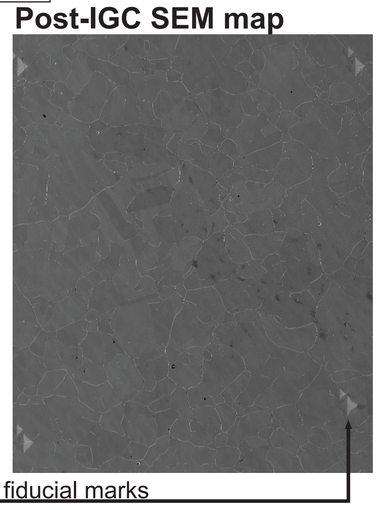
2. Thermal segregation and precipitation: Site-specific EBSD/IGC experiment
➡sensitized 316L in high temperature acid test

GB Specific IGC

Pre-IGC GB map



Post-IGC SEM map

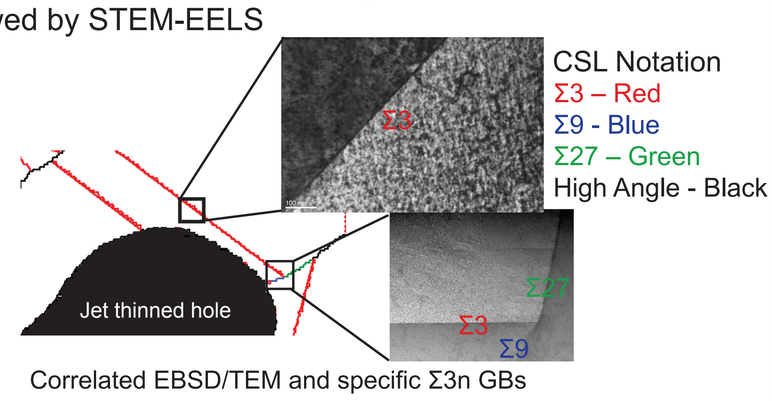


Correlated fiducial marks

3. GB specific in-situ TEM Irradiation

- Preliminary site-specific in-situ TEM irradiation/heating and correlated EBSD for boundary character followed by STEM-EELS

IVEM-Tandem Facility



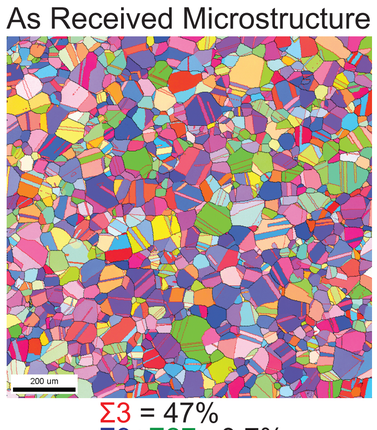
CSL Notation
Σ3 – Red
Σ9 - Blue
Σ27 – Green
High Angle - Black

Correlated EBSD/TEM and specific Σ3n GBs

Results:

Grain Boundary Structure and Population:

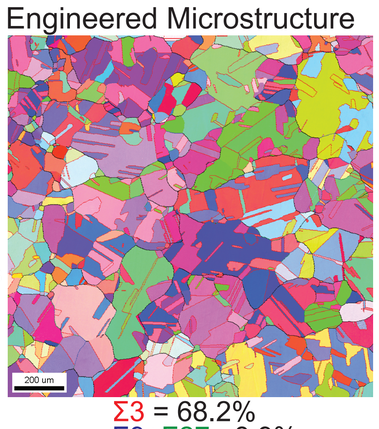
As Received Microstructure



Σ3 = 47%
Σ9+Σ27= 0.7%

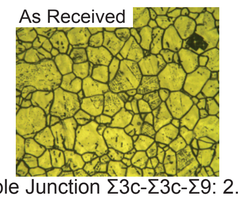
GBE Process

Engineered Microstructure



Σ3 = 68.2%
Σ9+Σ27= 8.9%

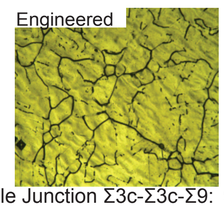
As Received



Triple Junction Σ3c-Σ3c-Σ9: 2.6 %

Reduced Connectivity of Corroded Boundaries

Engineered

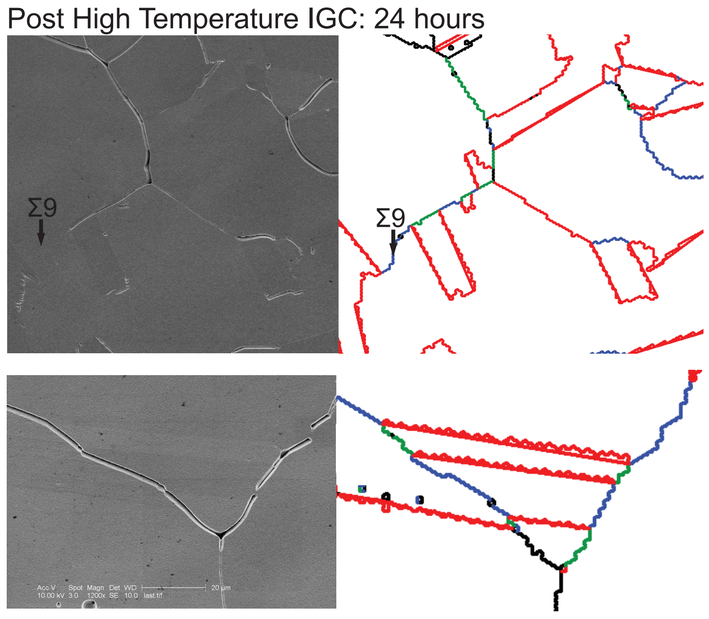


Triple Junction Σ3c-Σ3c-Σ9: 9.5 %

- GBCD study: Most twin boundaries introduced are not coherent
  ➡ large increases in MRD intensity associated with non-coherent twin planes
- GBE Σ9 shows preference for both symmetric (114/114) and asymmetric tilt (e.g.: 111/115) grain boundaries

Site-Specific Integranular Corrosion:

Post High Temperature IGC: 24 hours



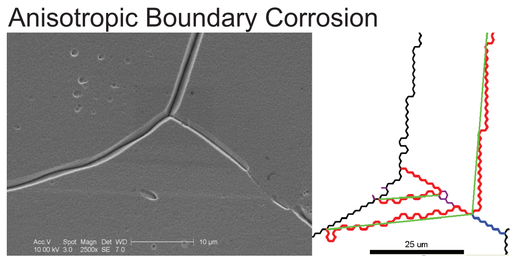
- Coherent Σ3 showed complete IGC resistance while non-coherent Σ3 illustrated wide spectrum (resistant to susceptible) of IGC
- Anisotropic IGC also indicated by high degree of variation in IGC within the same CSL notation: Σ9, Σ3
- Indicates overall importance of GB plane and coherency compared to misorientation based description

Project Summary and Future Work:

Site-specific high temperature corrosion indicate anisotropic nature IGC of GBs within the same CSL value (e.g. Σ3 and Σ9)

➡ Variation of grain boundary plane must be accounted: full GBCD necessary
➡ Coherent twin showed the highest resistance - consistent with GB energy

Anisotropic Boundary Corrosion



RIS studies are preliminary but do indicate a clear methodology while hinting at anisotropy in boundary structure

Future Experiments:

1. RIS analysis of specific boundaries: Atom Probe Tomography and HREM
2. ATP of thermal sensitized and nuetron irradiated samples

References and Acknowledgements:

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[1] Duh, T.S., Kai, J.J., Chen, F.R. J. Nucl. Mater. 283-287 (2000) 198-204
[2] GBCD program used in this work was developed by the MRSEC at Carnegie Mellon University