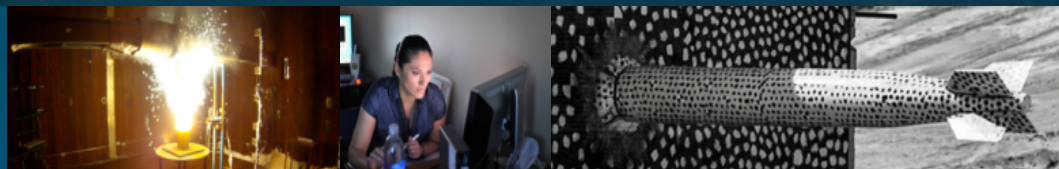




Hydrogen Concentration and Uptake in Additively Manufactured Austenitic Stainless Steels



R. Schaller^{a,b}, A. Imani^b, E. Asselin^b, M. Melia^a, and E. Schindelholz^a

^a Sandia National Laboratories, Albuquerque, NM

^b The University of British Columbia, Vancouver, BC



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Powder Based SLM Processing and Characteristics



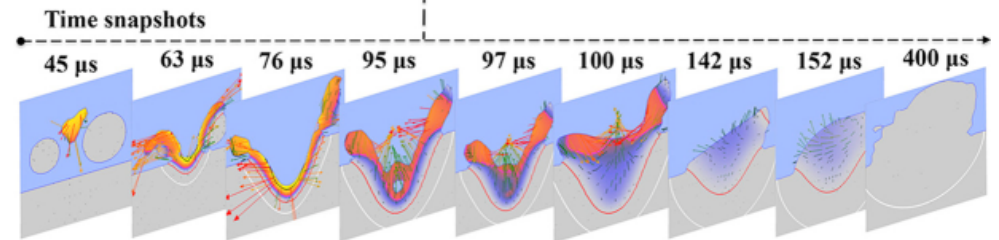
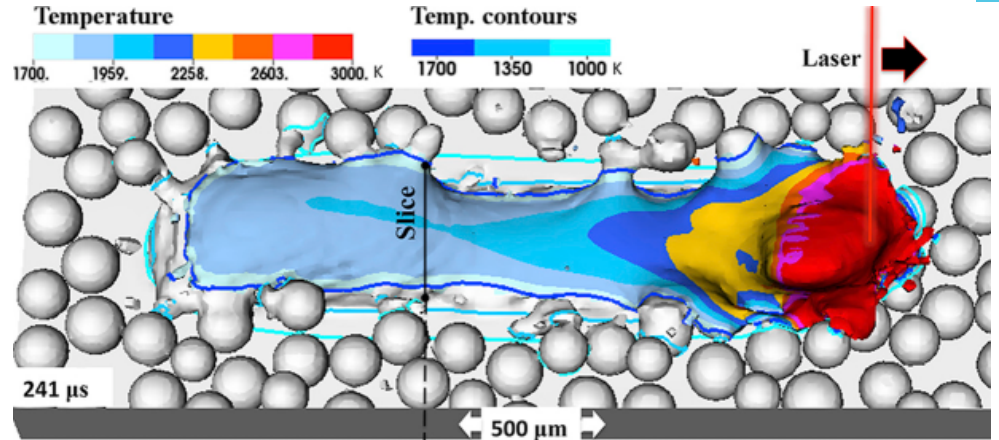
2

SLM Processing:

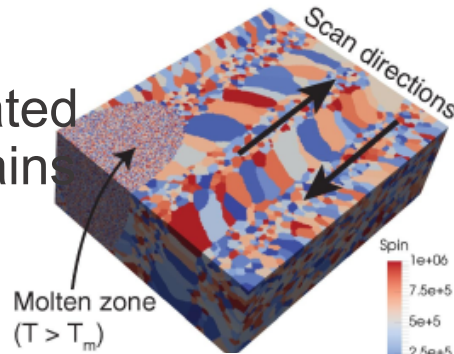
- High thermal gradients
- Fast cooling rates (up to 107K/s)
- High velocity growth vectors (directional solidification)

SLM Materials

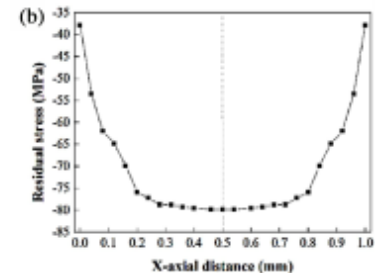
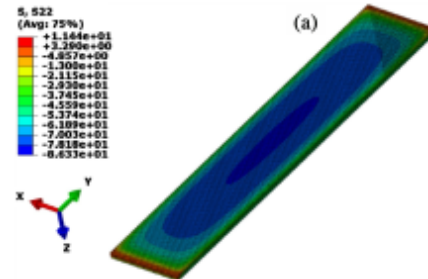
- Heterogeneous mixture of elongated and equiaxed grains with periodicity



Khairallah, 2016



Rodgers, 2017



An, 2017

¹ Khairallah, S. A., Anderson, A. T., Rubenchik, A., & King, W. E. (2016). Laser powder-bed fusion additive manufacturing: Physics of complex melt flow and formation mechanisms of pores, spatter, and denudation zones. *Acta Materialia*, 108, 36-45.

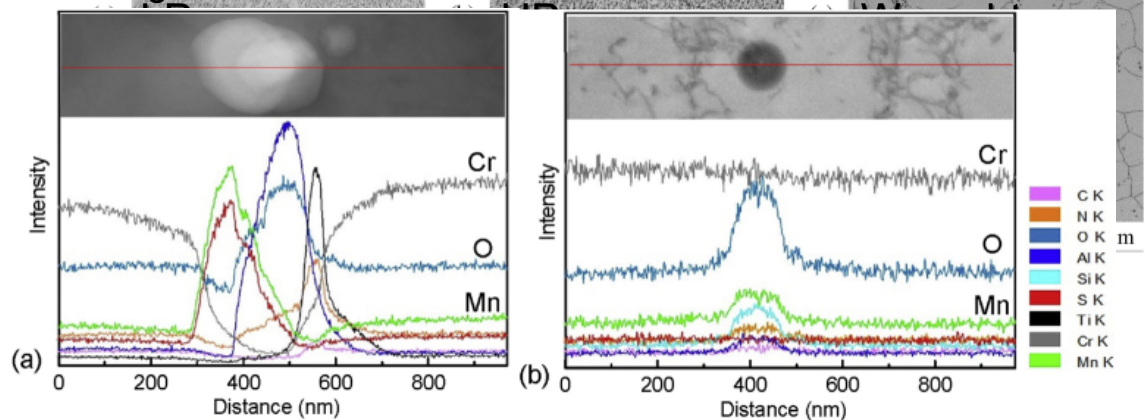
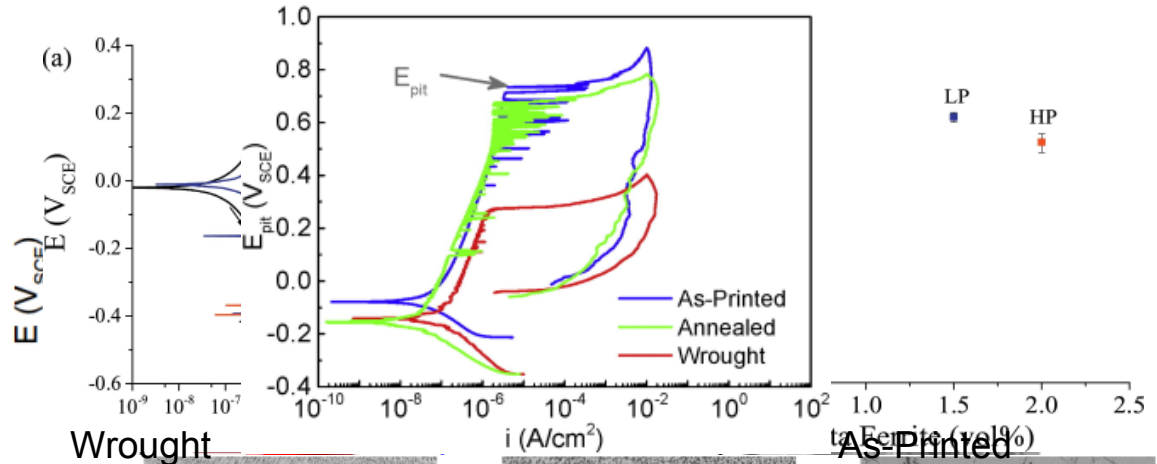
² Rodgers, T. M., Madison, J. D., & Tikare, V. (2017). Simulation of metal additive manufacturing microstructures using kinetic Monte Carlo. *Computational Materials Science*, 135, 78-89.

³ Wu, L., Wang, L., & An, Y. (2017). Numerical analysis of residual stress evolution of AlSi10Mg manufactured by selective laser melting. *Optik*, 127, 65-70.



Possible Deleterious Corrosion Features Formed from AM Processing:

- Pores: Lack of Fusion
- Elemental Segregation
- Inclusions/ Oxides

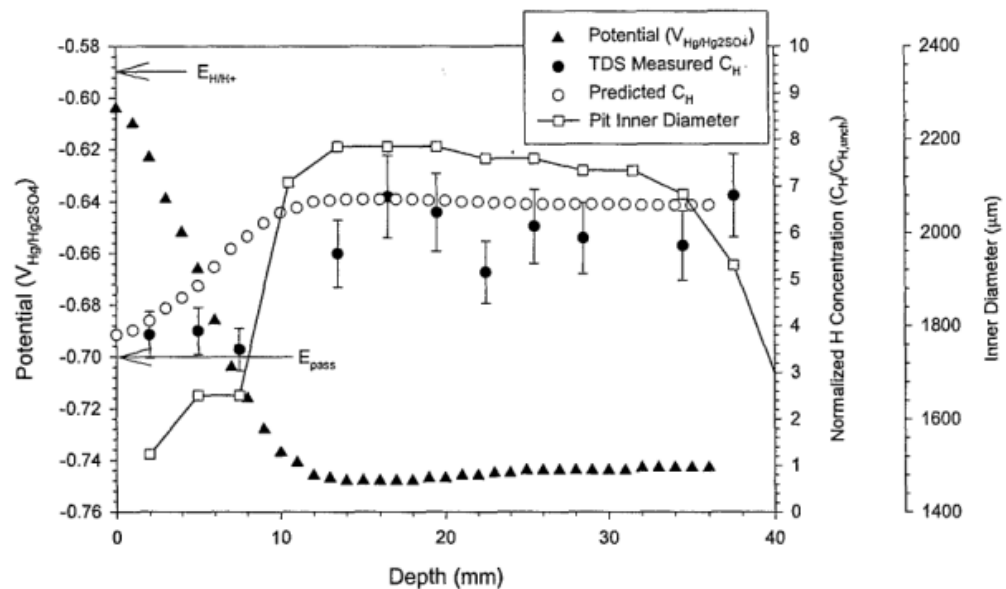
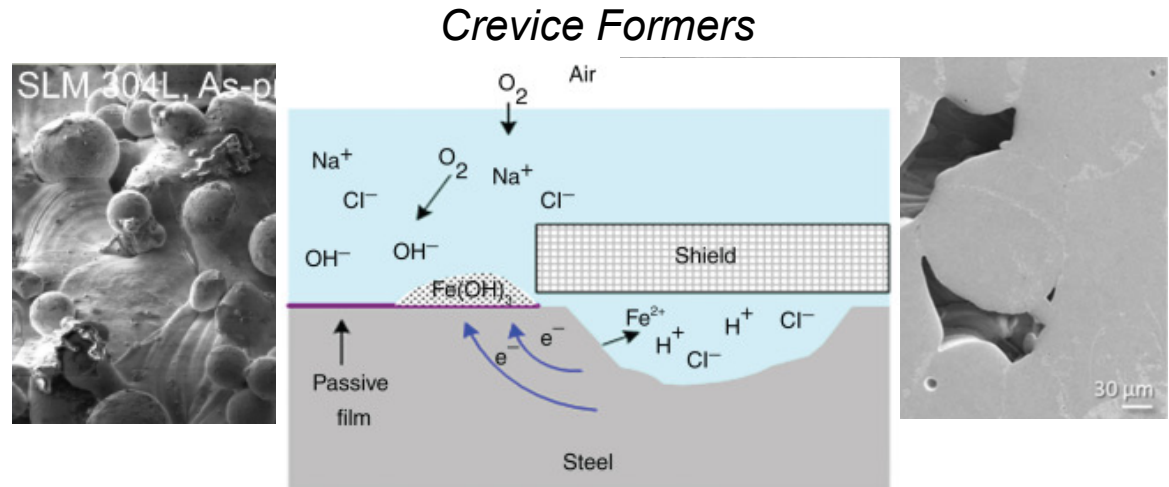


Nanoscale oxides/oxynitrides formed in SLM have a homogeneous Cr composition with the surrounding matrix, but direct energy deposition material observed a reduced E_b → chemical pitting/segregation dictated by cooling rate.



Possible Deleterious Features for HE:

- Surface Roughness/ Pores: lead to acidified regions? (similar to crevice)
- Elemental Segregation: Similar to sensitization? Scale?
- Inclusions/ Oxides
- Residual Stresses
- Dislocations

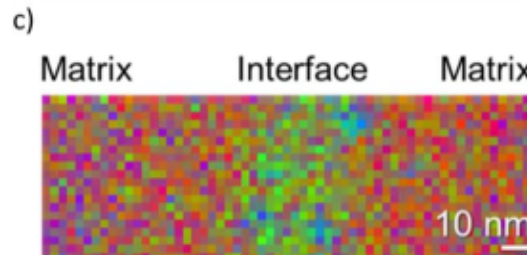
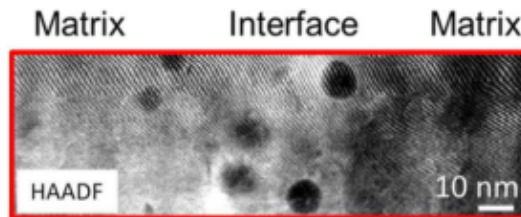
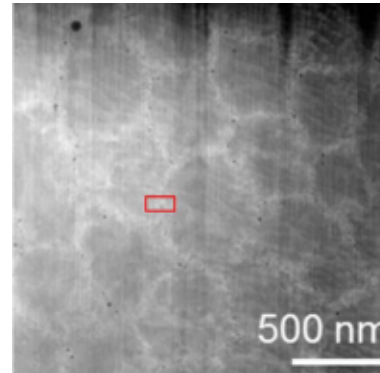




Possible Deleterious Features for HE:

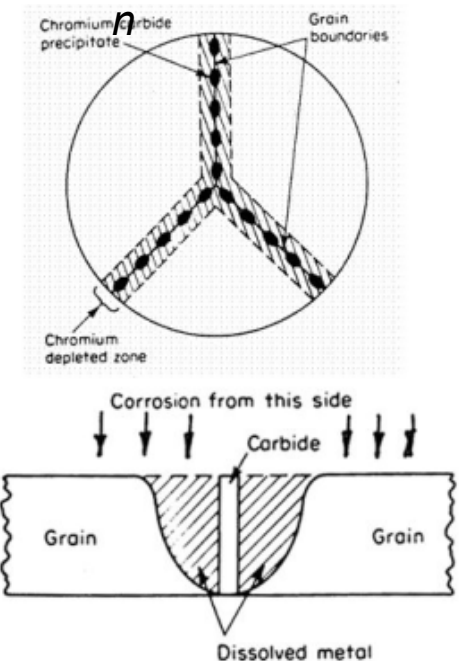
- Surface Roughness/
Pores: lead to acidified regions?
(similar to crevice)
- Elemental Segregation: Similar to sensitization?
Scale?
- Inclusions/ Oxides
- Residual Stresses
- Dislocations

Elemental



- Increased Fe (+ 9%)
- Increased Si
- Increased Cr (+ 4%)

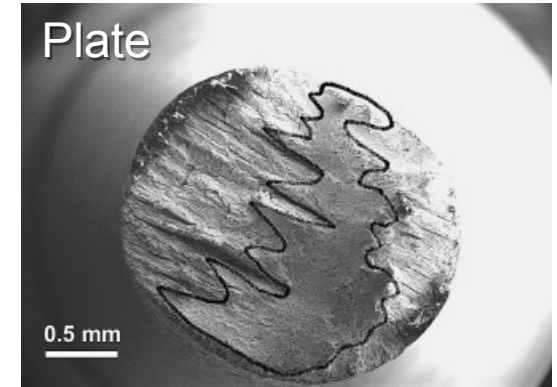
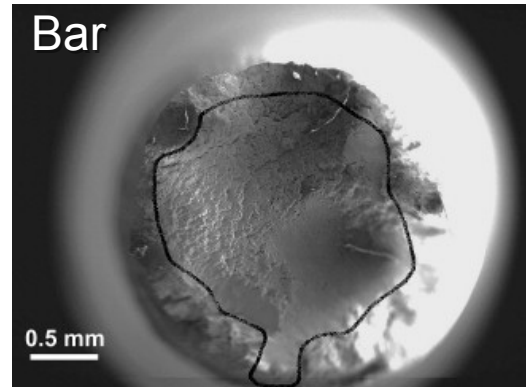
Sensitization





Possible Deleterious Features for HE:

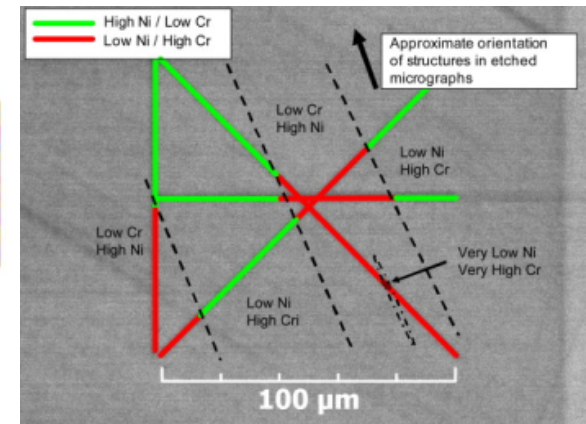
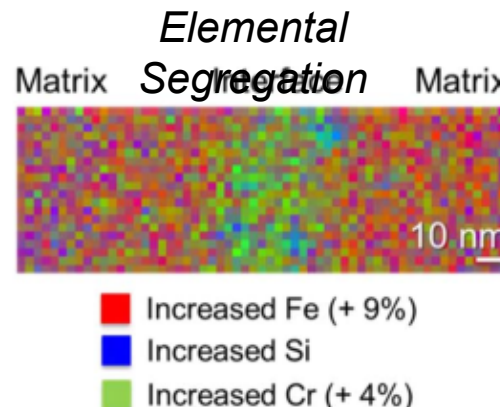
- Surface Roughness/ Pores: lead to acidified regions? (similar to crevice)
- Elemental Segregation: Similar to sensitization? Scale?
- Inclusions/ Oxides
- Residual Stresses
- Dislocations



Overview of 316 SS tensile fractured in H₂. The encircled area represents the maximum penetration of HEE.

Compositional dependence of the martensitic transformation:

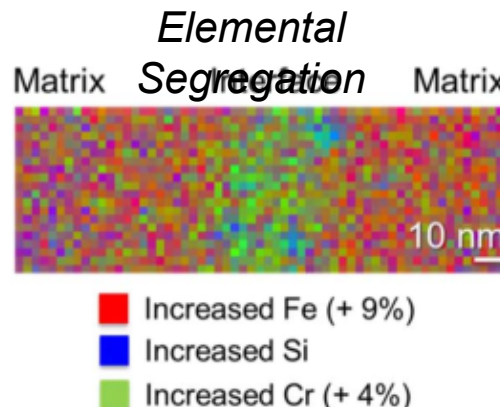
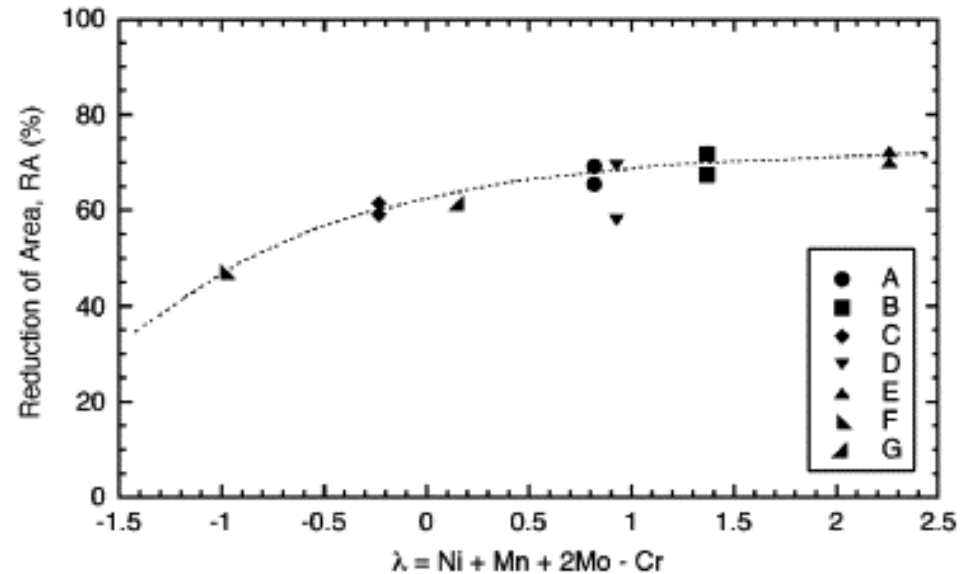
$$M_{d30} = 413 - 462 (C+N) - 9.2 Si - 8.1 Mn - 13.7 Cr - 9.5 Ni - 18.5 Mo$$





Possible Deleterious Features for HE:

- Surface Roughness/
Pores: lead to acidified regions?
(similar to crevice)
- Elemental Segregation: Similar to sensitization?
Scale?
- Inclusions/ Oxides
- Residual Stresses
- Dislocations



*Ductility as a function of composition
(with H Pre-charging):*

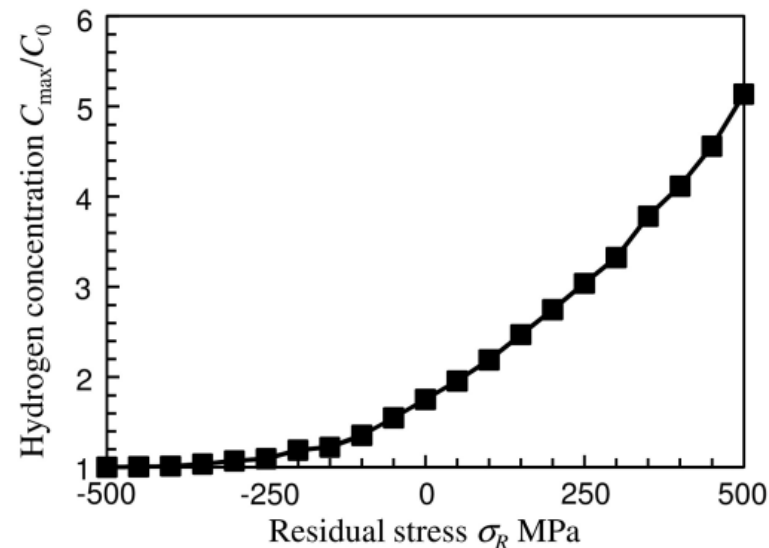
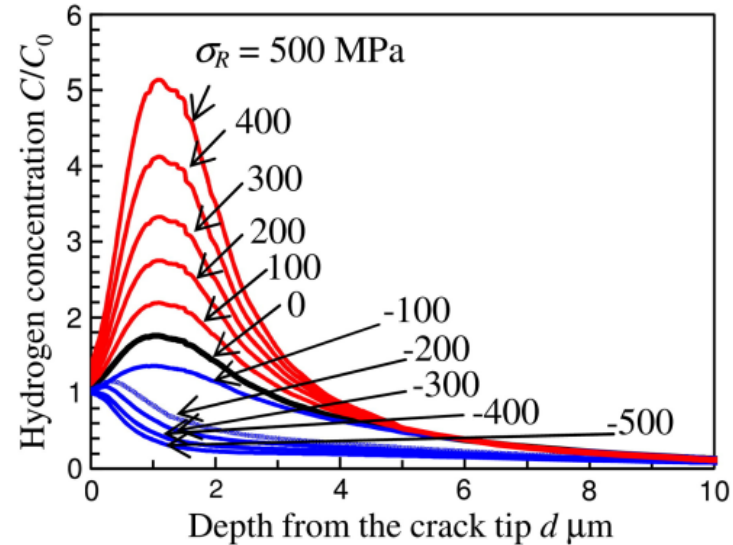
$$\lambda = Ni + Mn + 2Mo - Cr$$



Possible Deleterious Features for HE:

- Surface Roughness/
Pores: lead to acidified regions?
(similar to crevice)
- Elemental Segregation: Similar to sensitization?
Scale?
- Inclusions/ Oxides
- Residual Stresses
- Dislocations

Residual Stresses





- How do the unique microstructural features and surface morphology observed in SLM metals govern hydrogen embrittlement resistance relative to their conventionally processed counterparts?
 - What features exist in SLM Austenitic SS?
 - Initial H uptake and transport measurement

- Specifically, how do these features govern H uptake and transport in SLM materials?
 - Need for correlation/ understanding of the effects of the possible deleterious or advantageous features found in SLM materials to H uptake and transport



Measured 304L Material Composition (Weight %)

	<u>C</u>	<u>Cr</u>	<u>Co</u>	<u>Cu</u>	<u>Nb</u>	<u>Ni</u>	<u>Mn</u>	<u>Mo</u>	<u>P</u>	<u>Si</u>	<u>S</u>	<u>Fe</u>	<u>O</u>	<u>N</u>
Wrought	0.025±	18.16±	0.17±	0.49±	<0.05±	8.10±	1.73±	0.33±	0.034±	0.22±	<0.001±	70.5±	0.003±	0.076±
	0.004	0.363	0.026	0.074	0.008	0.405	0.173	0.049	0.005	0.033	0.000	0.353	0.000	0.011
AM	0.013±	18.89±	<0.03±	<0.1±	<0.05±	10.74±	1.44±	0.014±	0.005±	0.50±	0.005±	68.3±	0.038±	0.011±
	0.002	0.378	0.005	0.000	0.008	0.537	0.144	0.002	0.001	0.075	0.001	0.342	0.006	0.002

Renishaw AM 250 Build Parameters

Parameter	Value
Laser power	200 W
Pulse time	75 μs
Pulse spacing	60 μm
Dwell time	1-3 μs
Hatch spacing	85 μm (Measured 82 ± 15 μm)
Layer thickness	50 μm (Measured 40 ± 10 μm)
Cover gas	Argon

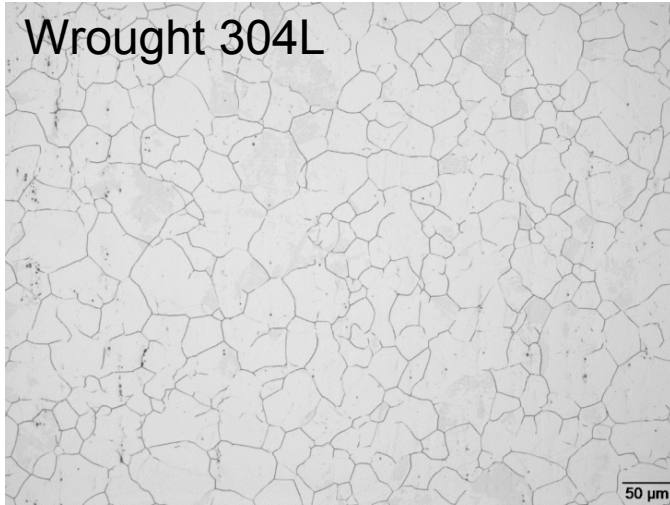
Starting Powder

Ø = 15-45 μm, D50 = 29 μm
Ar-atomized
single use (not recycled)

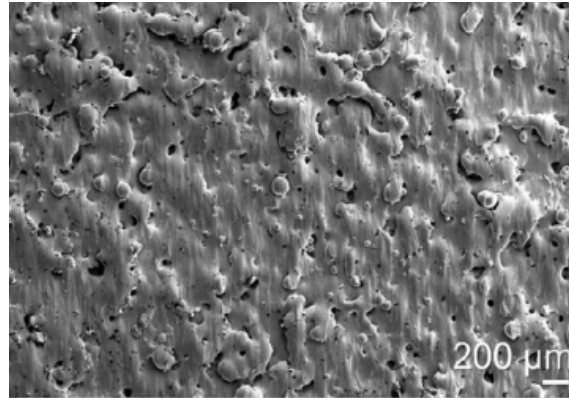
PREN_{austenitic} = %Cr + 3.3%Mo + 16%N → Wrought=20.3, SLM =19.1
M_{d30} = 413 – 462 (C+N) -9.2 Si – 8.1 Mn – 13.7 Cr – 9.5 Ni – 18.5 Mo
 Wrought =18.454, SLM = 29.166



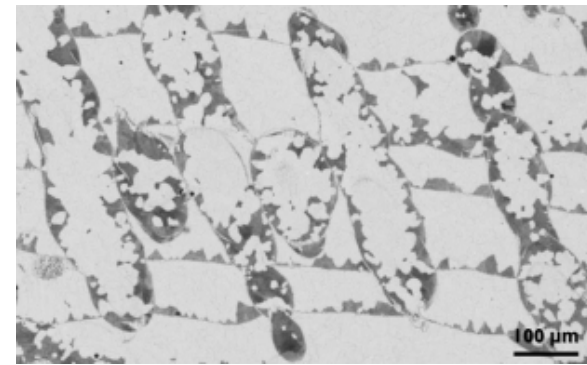
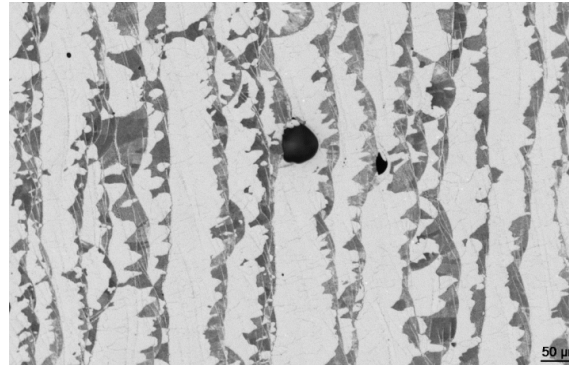
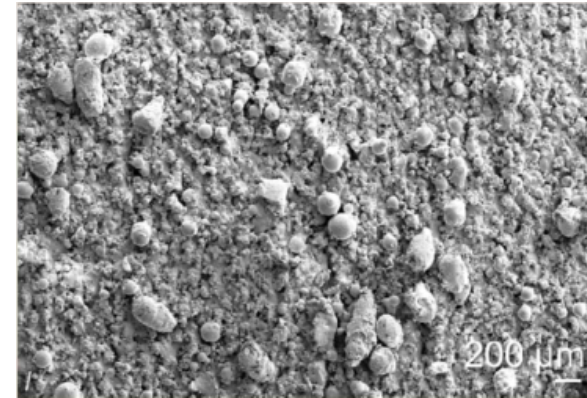
Wrought 304L



SLM Perpendicular to build

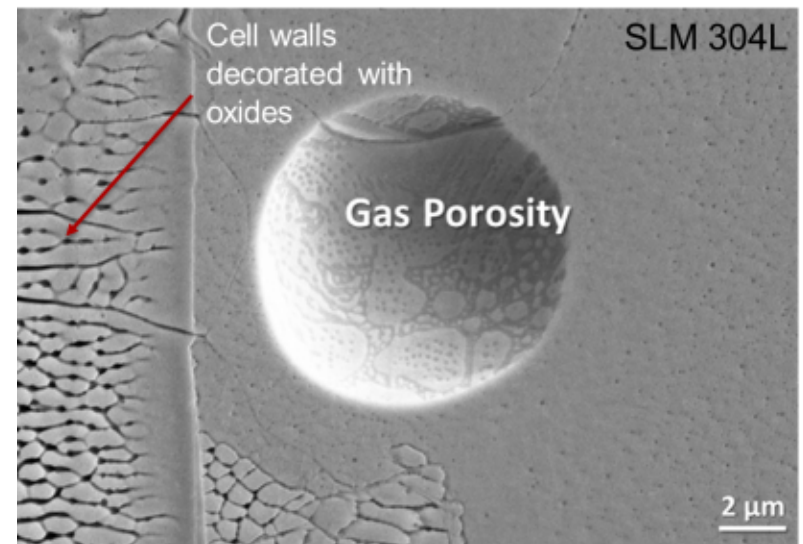
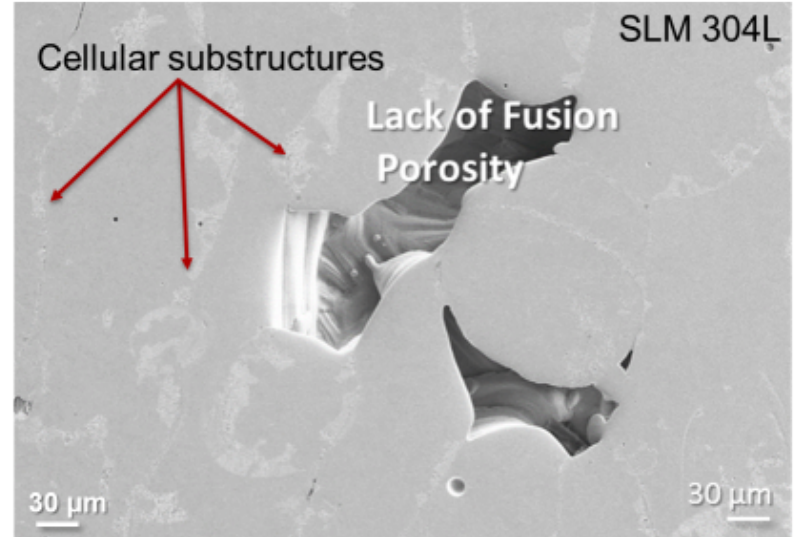
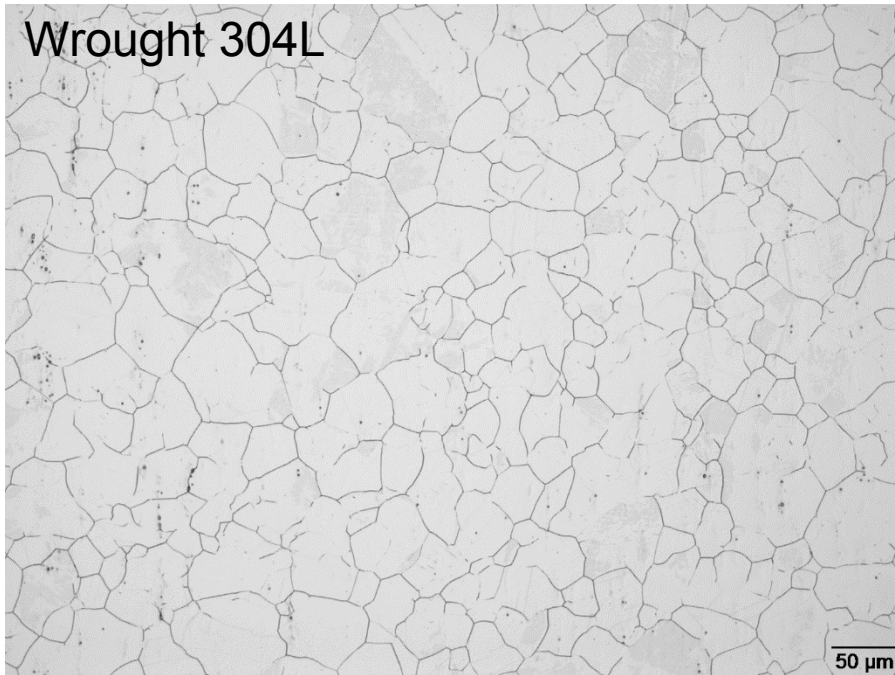


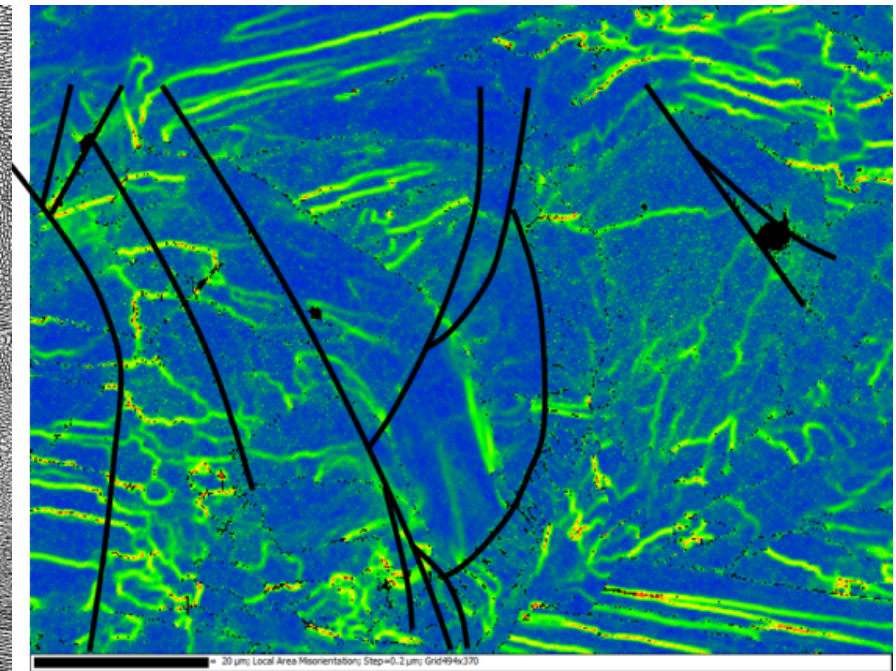
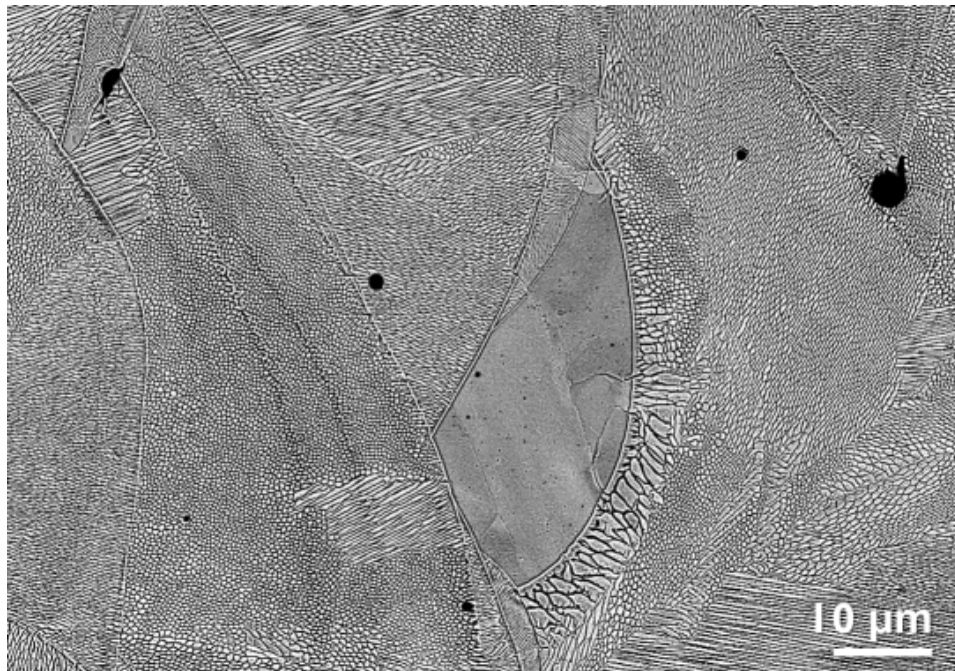
SLM Parallel to build





Wrought 304L

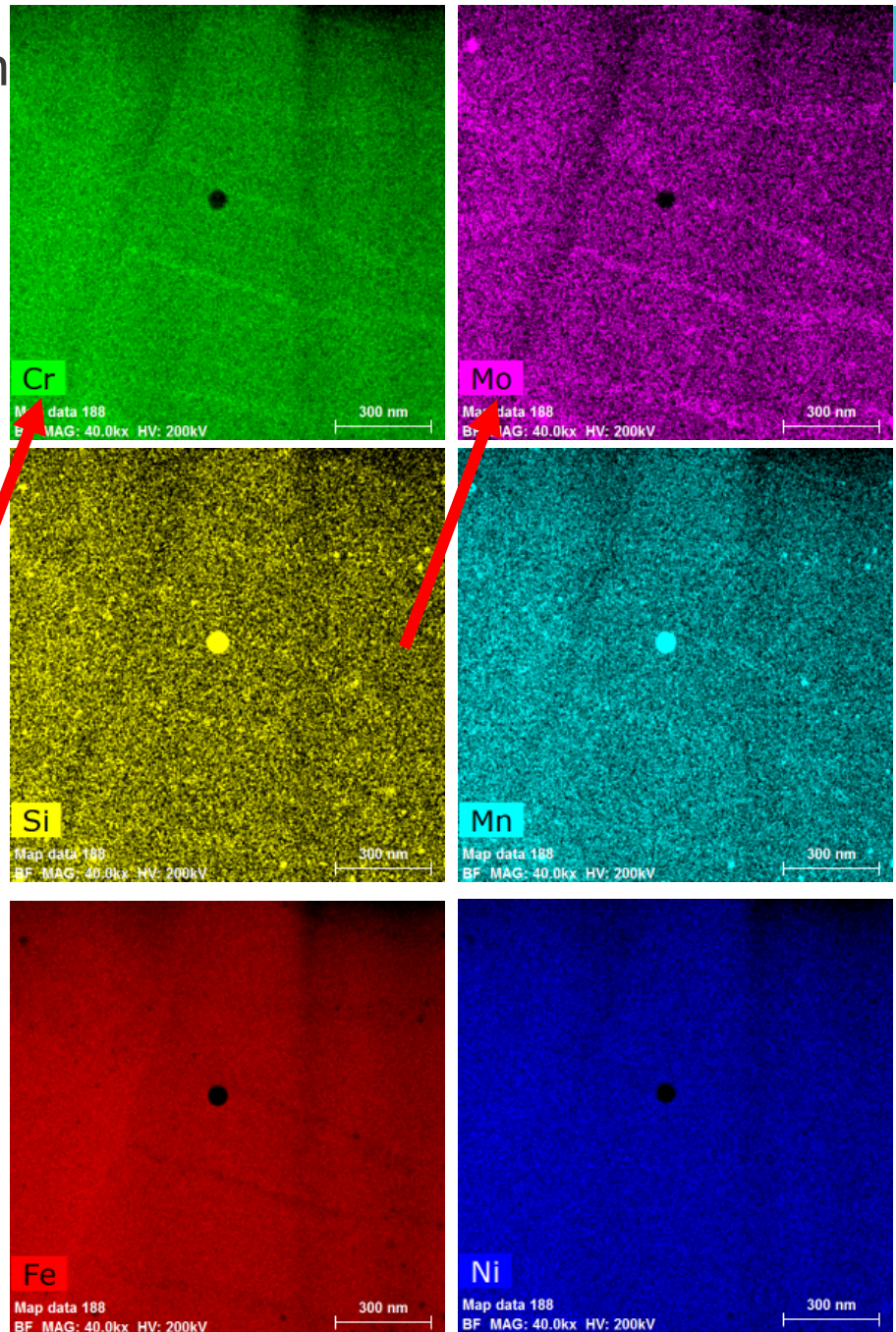
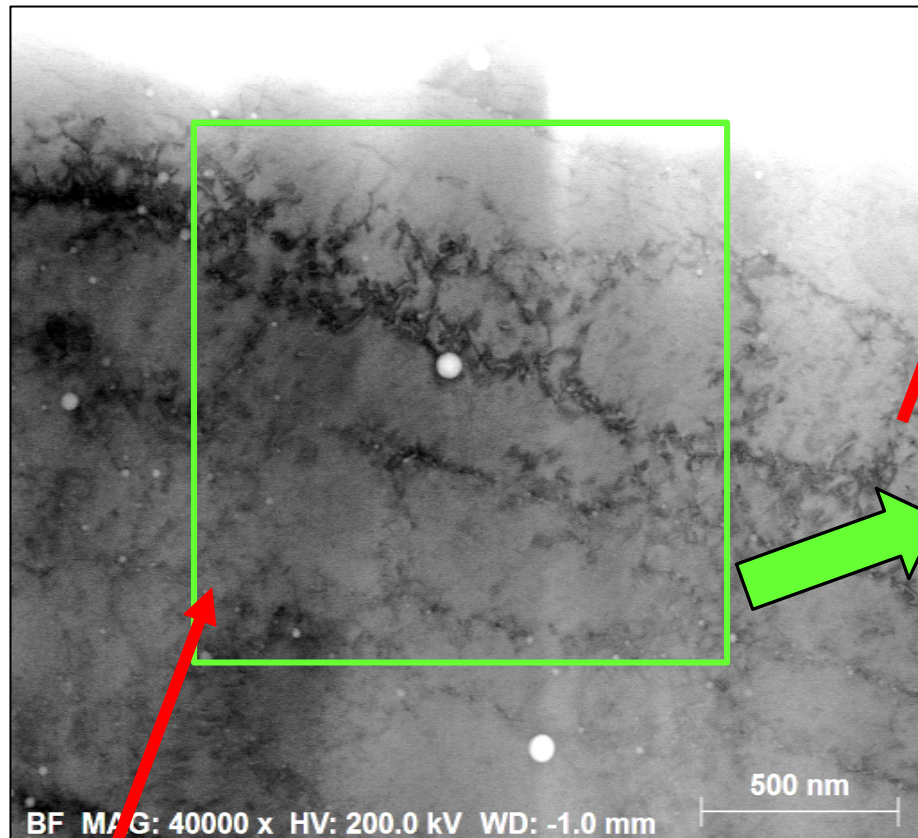




Local strain is associated with solidification structure not MPBs.

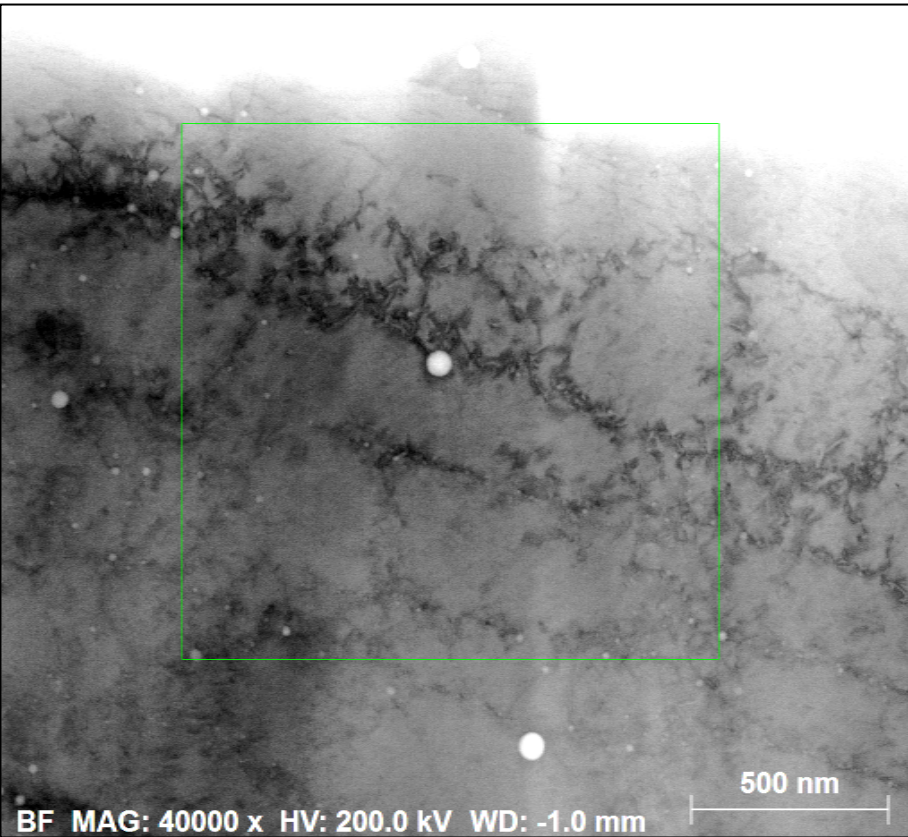
- Mostly associated with sub-grain boundaries.

SLM SS Elemental Distribution



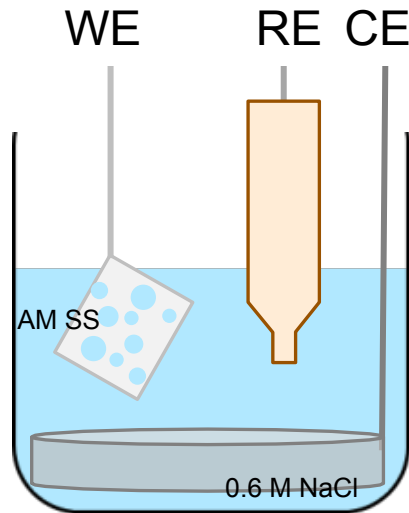
Melt Pool Boundary: Displays depletion of Cr, Mo, Mn, Si here and enrichment of

M_{d30}^{Fe} → SLM = 29.166 to 18.8
(with a 4% change in Cr)



Melt Pool Boundary: Displays depletion of Cr, Mo, Mn, Si here and enrichment of Fe.

Dislocation
substructure
decorated with oxides.



$$C_{H,diff} = \frac{i_t}{zF} \left(\frac{D_{H,eff}}{\pi t} \right)^{-0.5}$$

Assuming:

$$D_{H,eff} = 4.6 \times 10^{-12} \frac{cm^2}{s}$$

$$t = 1800 s$$

Electrochemical Pre-charging:

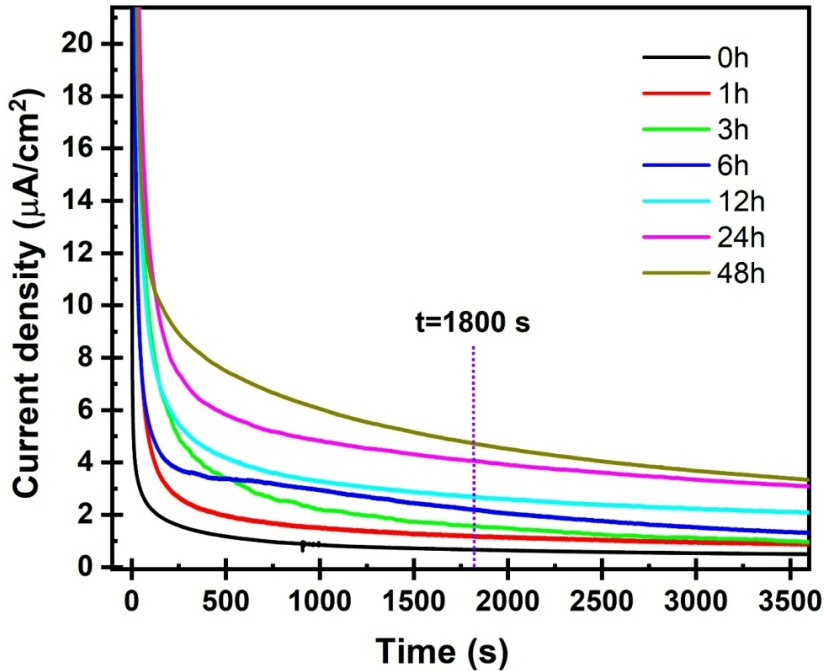
- 0.6 M NaCl Solution
- Cathodic hold at $-1.2 V_{SHE}$
- Time: Varying 0 to 48 h

Electrochemical Extraction:

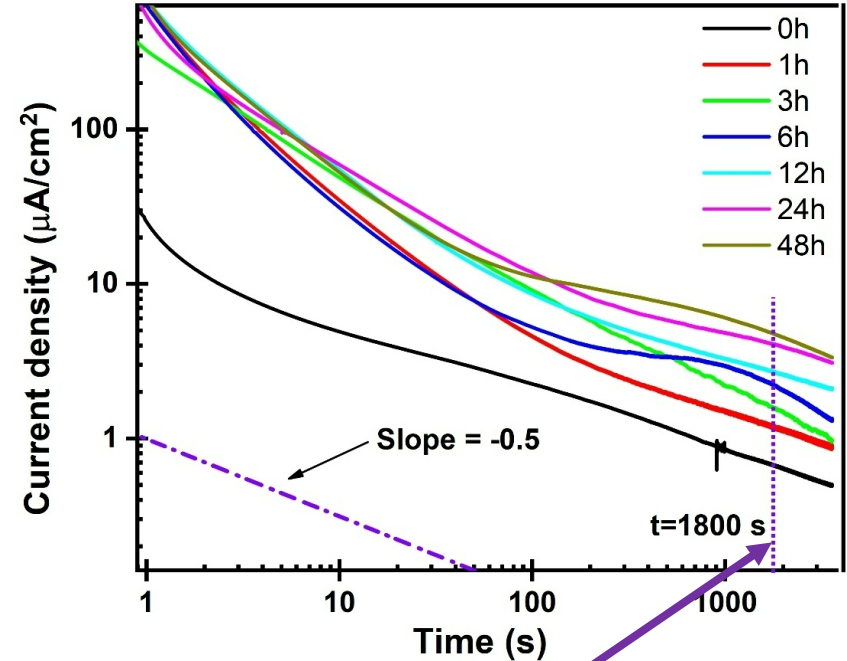
- 0.1 M NaOH Solution
- Deaerated (N_2 purging, 1 h prior and continuous)
- Anodic hold at $+0.33 V_{SHE}$
- Time: 1 h



H extraction/oxidation currents

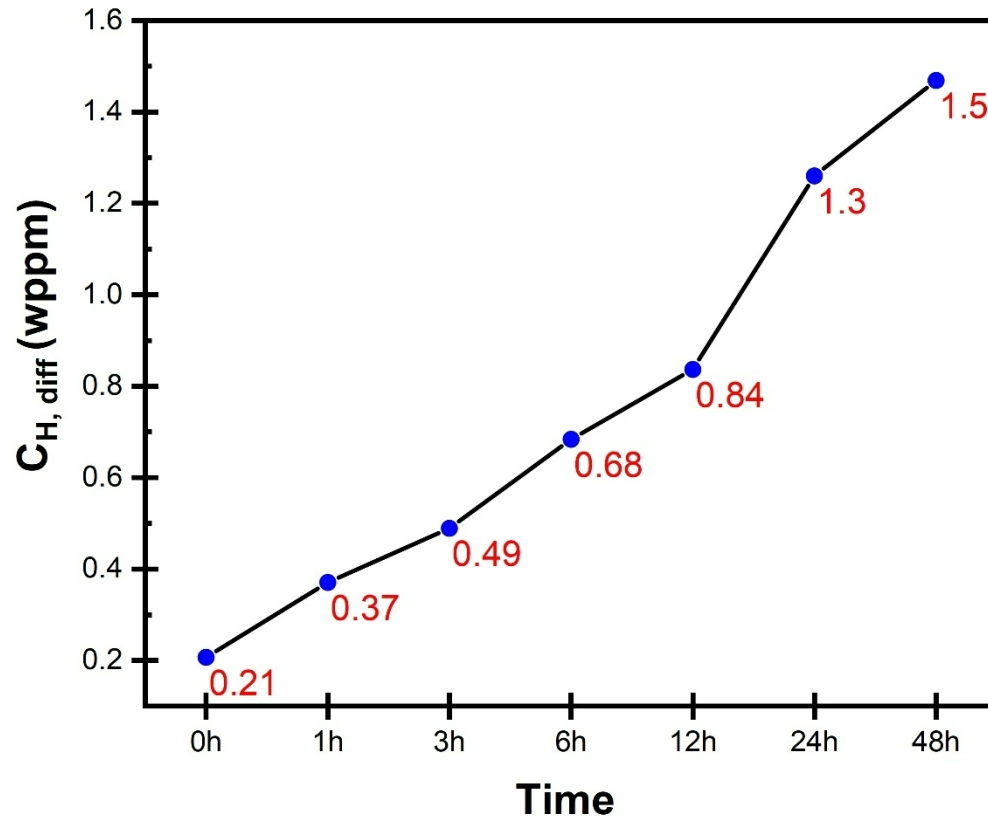


Comparison to Theoretical Slope



$$C_{\text{H,diff}} = \frac{i_t}{zF} \left(\frac{D_{\text{H,eff}}}{\pi t} \right)^{-1/2}$$

H oxidation current at $t = 1800$ s is used to evaluate $C_{\text{H,diff}}$



- In SLM SS, $C_{H, \text{diff}}$ increases with increased charging time associated with the controlled cathodic charging
- Can measure a significant increase in H above background ($t = 0$)



- Need comparison to traditional wrought materials
- H measurements at the global not local scale
 - Microstructural/ compositional effects may influence local H concentrations more drastically
- Need to further not only concentration measurements, but H transport measurements as well
 - Dependent on direction, microstructure, composition, etc. ?
- Knowing this, can we take advantage of AM to “tune” these conventional material properties?



Questions?

Acknowledgements Paul Kotula (SNL), Bonnie McKenzie (SNL)

