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Corrosion Properties of Powder Bed Fusion Additively Manufactured Stainless Steels

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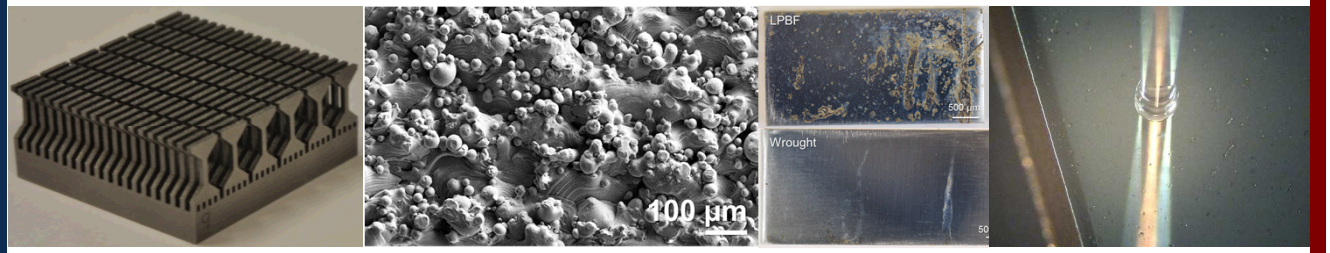
October 6th, 2016



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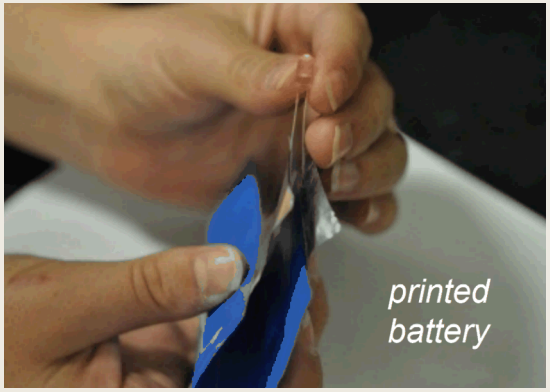
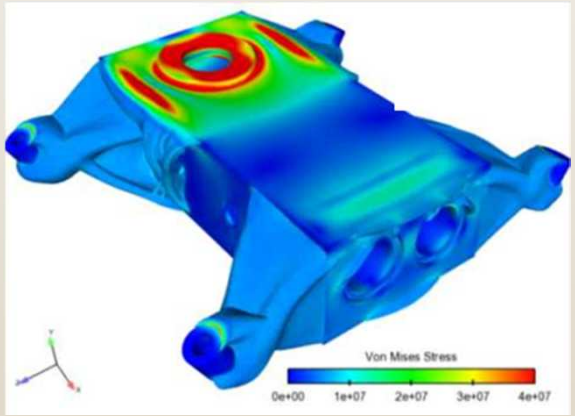
Background:
AM Materials…why?

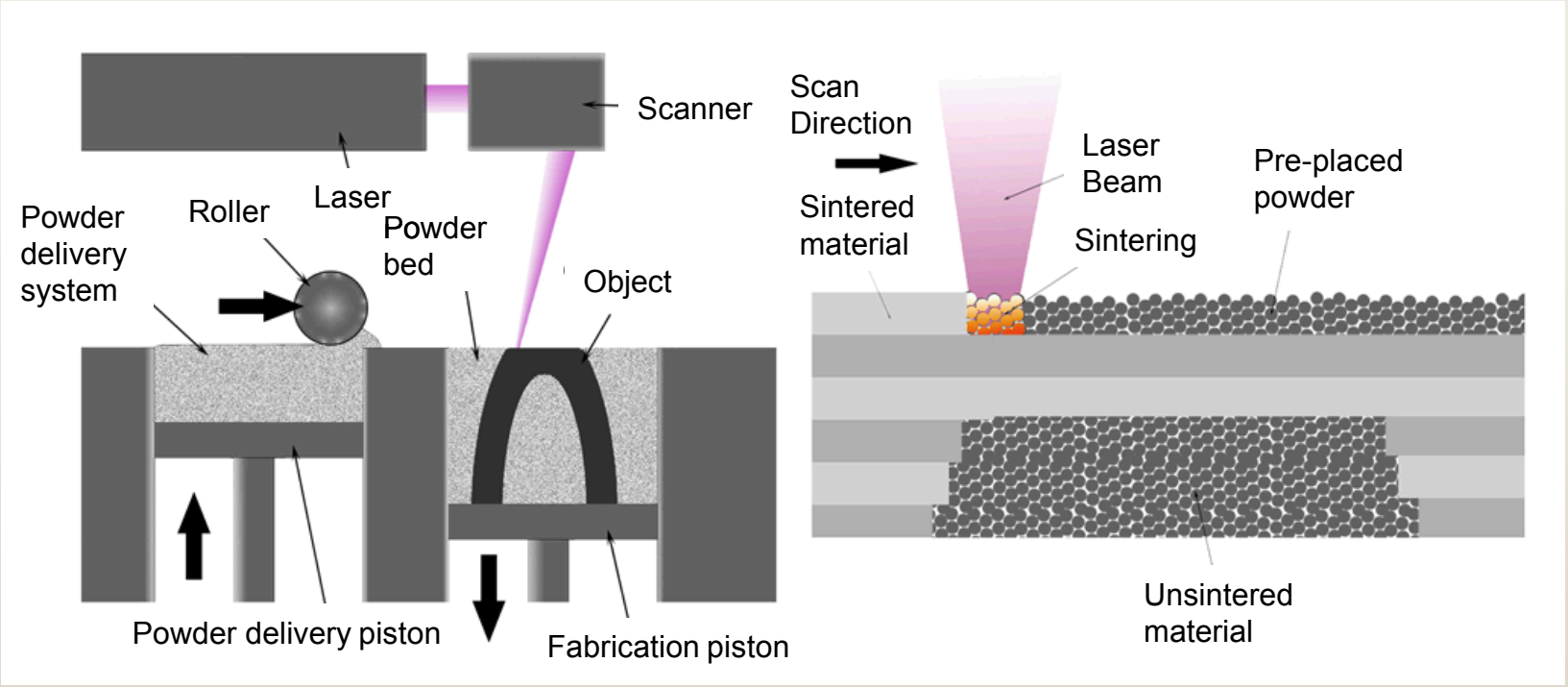
- Additive Manufacturing:
  - Reduce Material Waste
  - Simplify Assembly and Processing
  - Can create complex forms





Background:
LPBF Process

- Laser Powder Bed Fusion:
  - Laser used to selectively melt successive layers of powder material to build a three dimensional structure

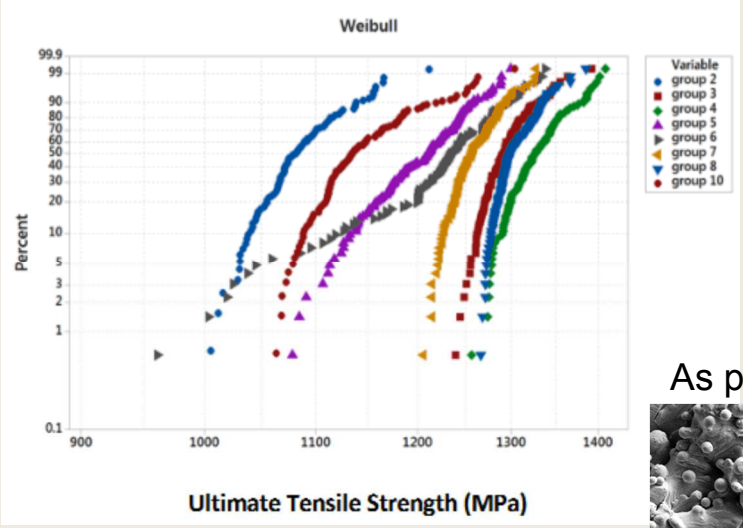


From Wikipedia "selective laser sintering'

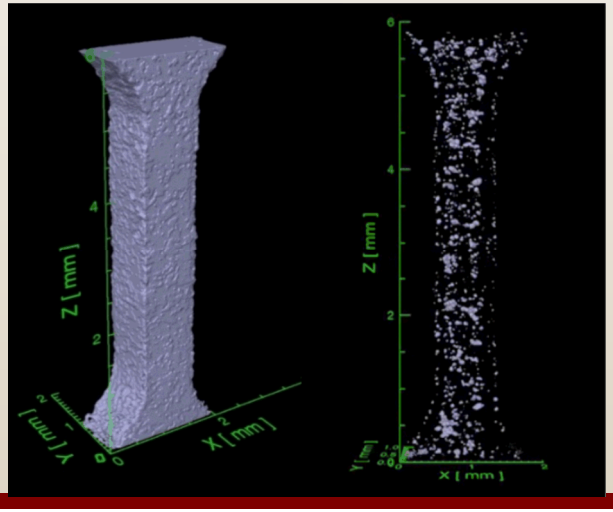
Background:

AM Microstructural Challenges

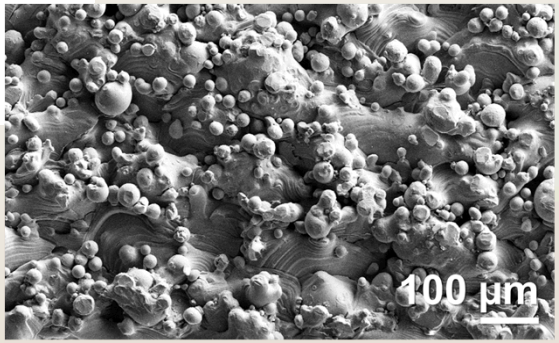
- Chemistry similar to specifications?
  - Segregation of elements?
- Residual oxides from un-melted powder
- Porosity
  - Density, Size, morphology?



17-4PH AM dogbone
CT surface (left) & porosity (right)

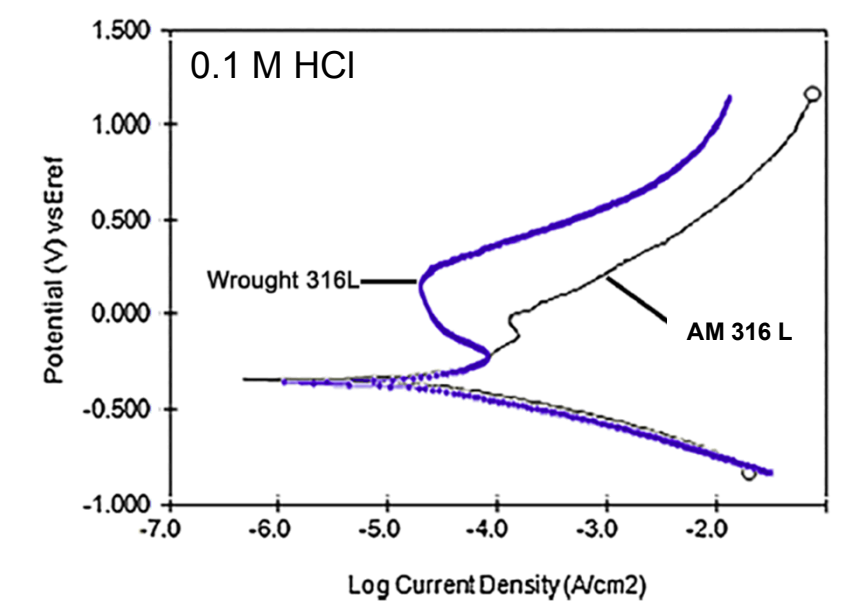


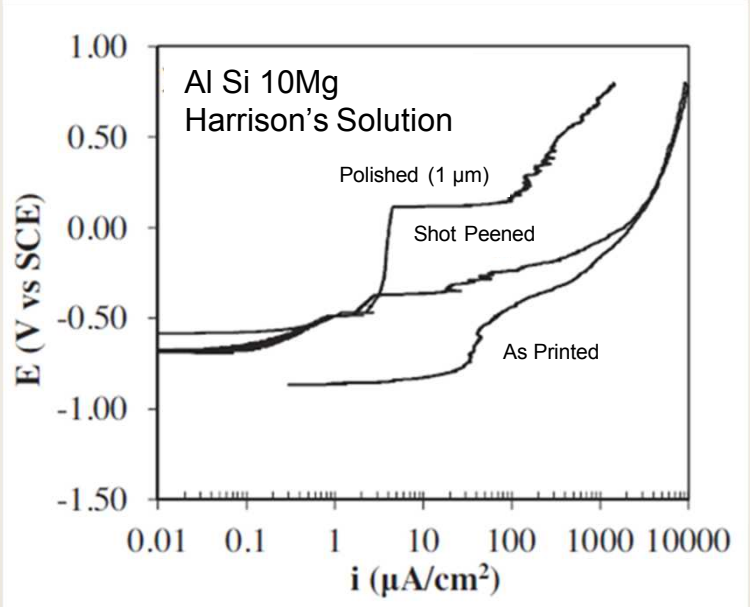
As printed surface of 304L



Background:

Material Challenges and Corrosion





Reduced corrosion resistance of AM alloy attributed to inhomogeneous solute distribution. (Trelewicz, 2016)

Effect of surface finish enhances the corrosion resistance. (Cabrini, 2016)

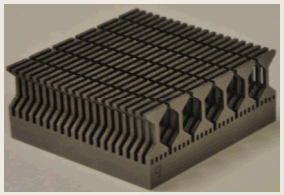
Overall Approach

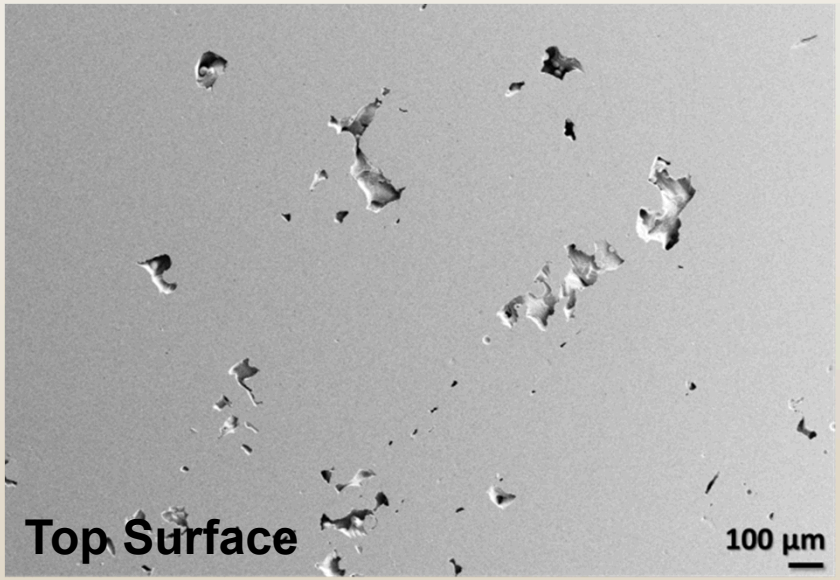
- What are the relative impacts of the microstructural characteristics unique to AM processing on the global scale corrosion properties of PBLF 17-4 PH in NaCl full immersion environments?
  - Solute segregation, Residual oxides, Porosity

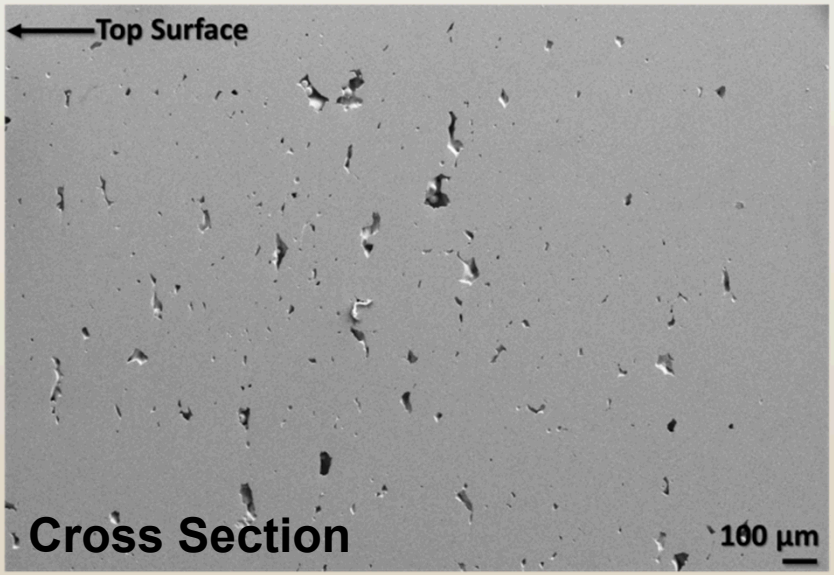
- Experimental Approach:
  - Material Characterization:
    - Compositional and Microstructural analysis
  - Global and Local Scale Electrochemistry
  - Exposures: Full immersion and salt fog

Material Characterization:

17-4 PH LPBF, Surface Analysis



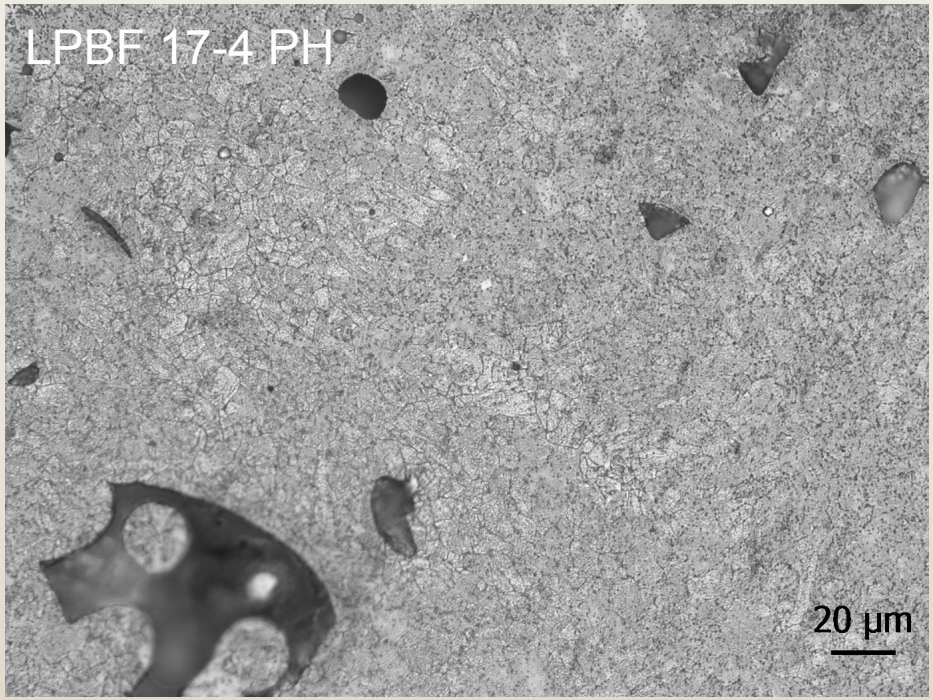




Material Characterization:

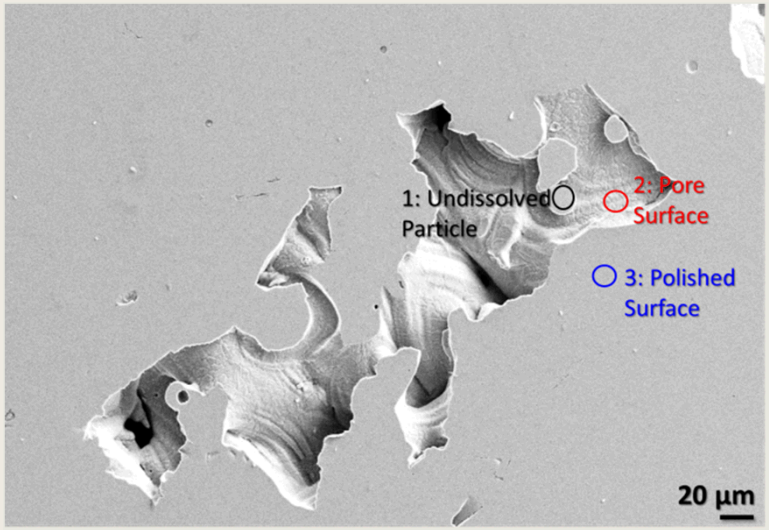
17-4 PH Wrought vs LPBF, Compositional Analysis

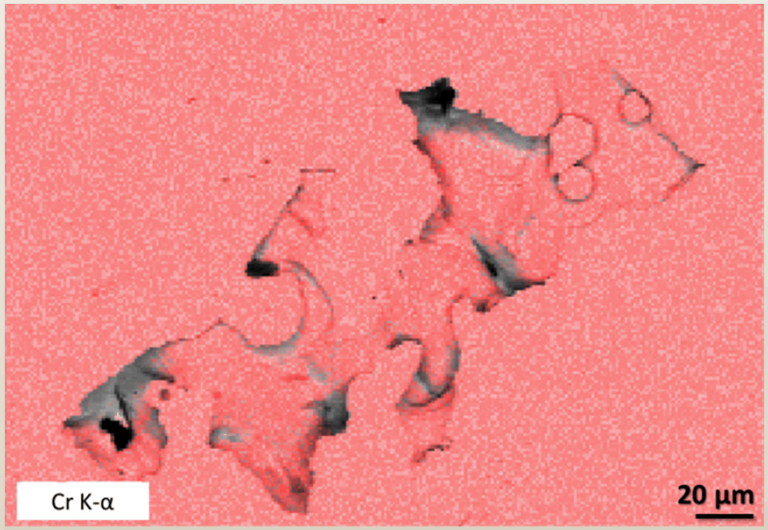
Sample	C +/- 0.004	Nb	Cr	Cu	Fe	Mn	Mo +/- 0.011	N	Ni	O +/- 0.008	P	S	Si	Ta
Wrought	0.047	0.23	15.19	3.21	75.6	0.53	0.23	0.023	4.54	0.014	0.022	<0.001	0.17	<0.001
LPBF	0.017	0.29	16.02	3.95	74.9	0.22	0.025	0.036	4.12	0.068	0.013	0.002	0.33	<0.001

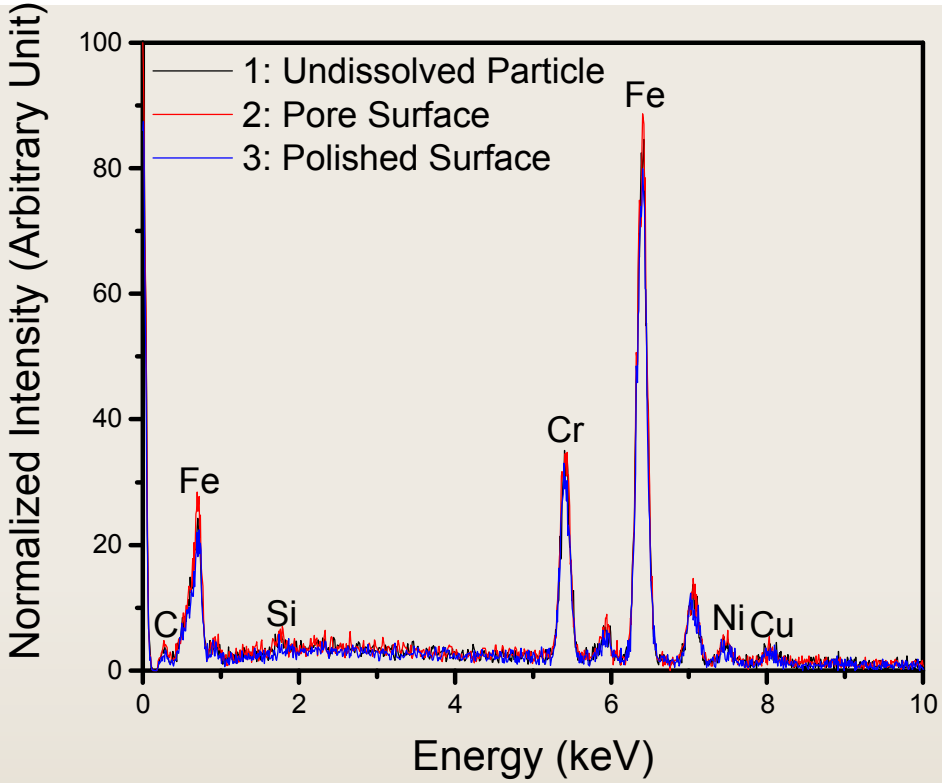


- Heat treated:
  - AMS 5604:1050°C, 60 min in Ar, cooled to RT
  - Age hardened H900: 482°C 60 min in air
- No evidence of solidification substructure can be observed
- Solutionization heat treatment resulted in recrystallization and homogenization of solute microsegregation

Materials Characterization:
17-4 PH LPBF



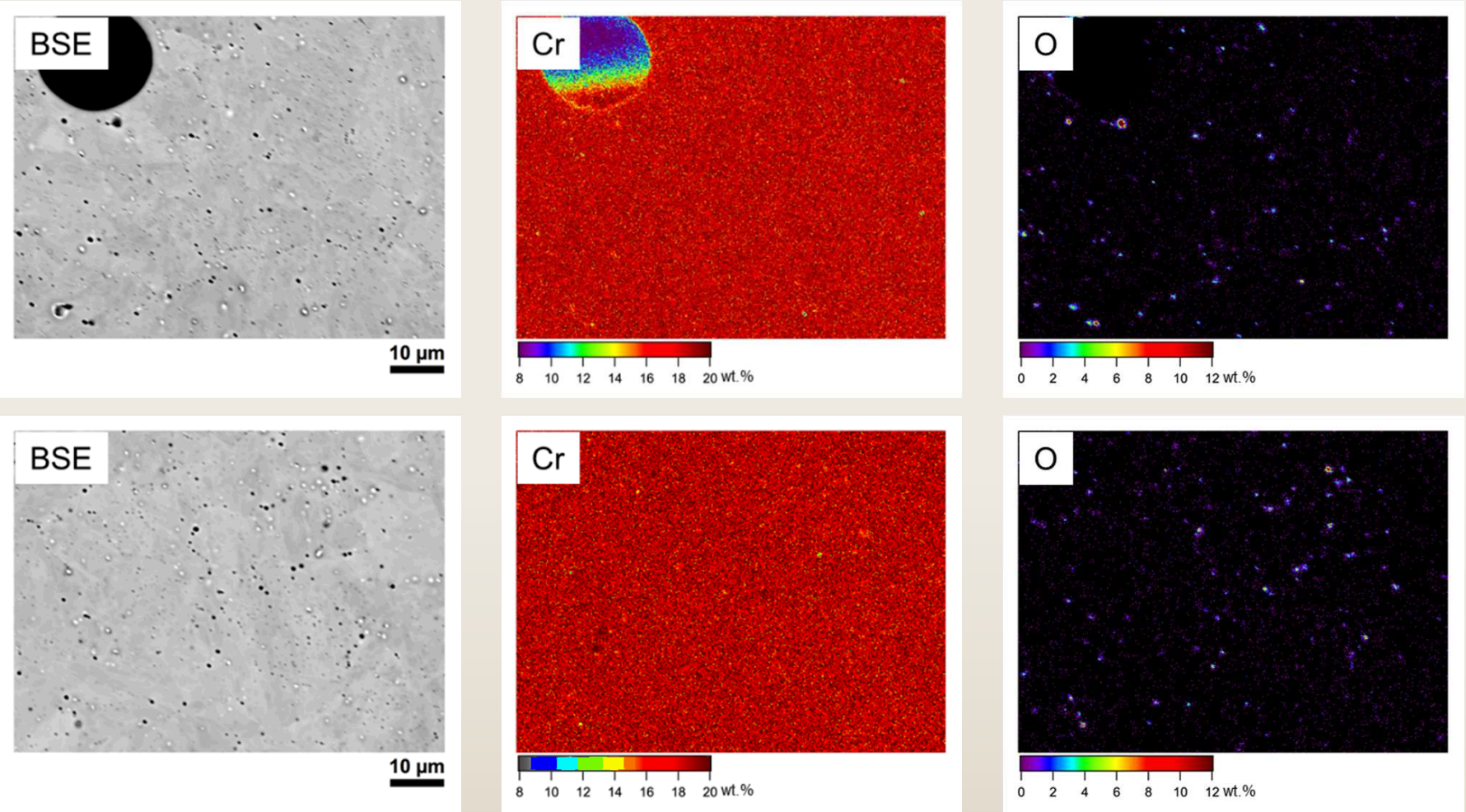




- SEM and EDS of unexposed LPBF 17-4 PH: undissolved particle, pore surface, and polished surface
- No significant large scale segregation.

Materials Characterization:

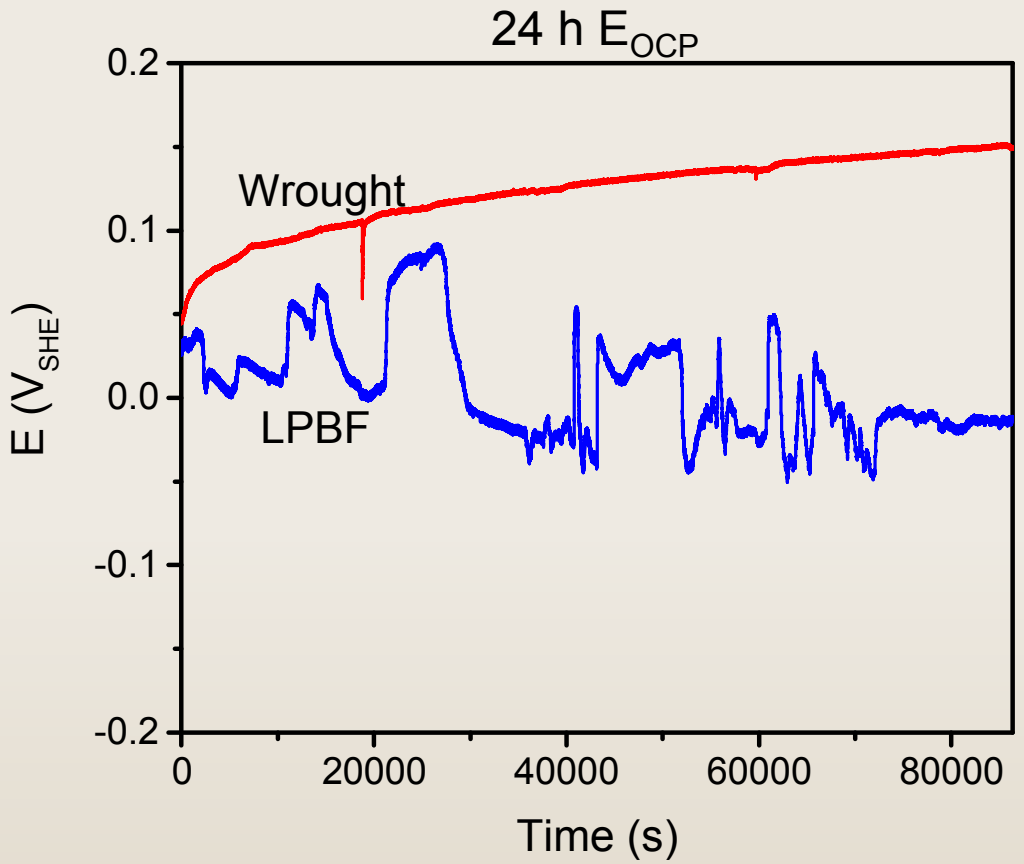
17-4 PH LPBF



X-ray wavelength dispersive spectroscopy (WDS) of a pore and a non-porous area. Solutionization resulted in homogenization of solute microsegregation. Some local enrichment of Nb and Si/O corresponding to Nb-carbonitrides and Si-rich oxides,

Global Scale Electrochemistry:
17-4 PH Wrought and LPBF

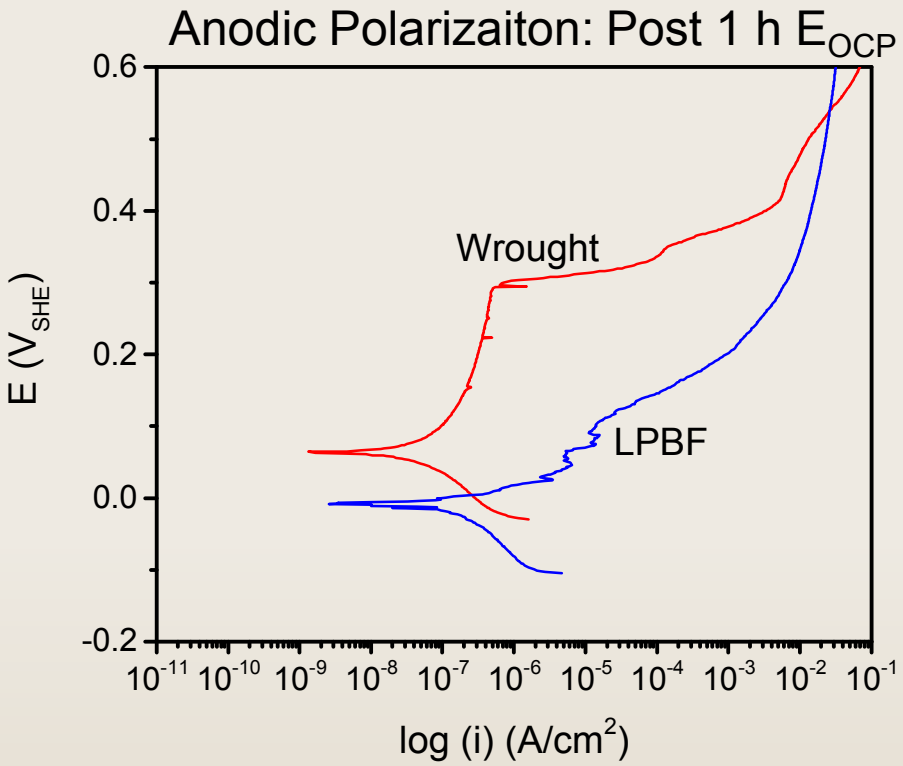
- Global Scale Electrochemistry:
  - 0.6 M ambiently aerated NaCl solution
  - 1 and 24 h E_{OCP}
  - Potentiodynamic Polarizations

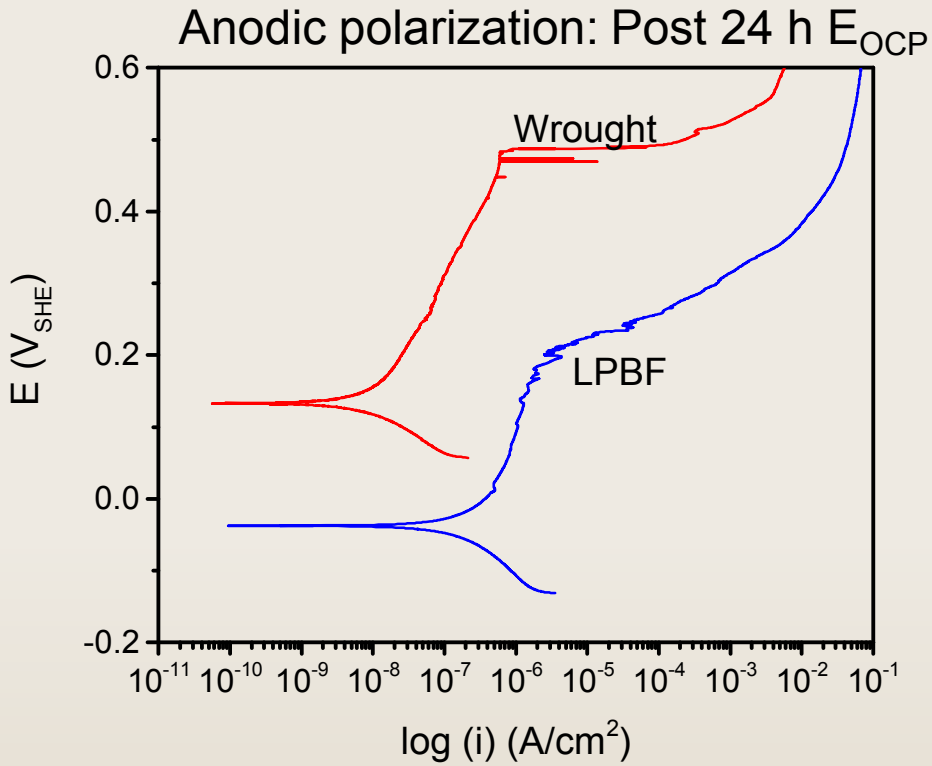


- Instabilities in the E_{OCP} of the LPBF samples:
  - Solution ingress into the pores
  - Active corrosion/pitting

Global Scale Electrochemistry:

17-4 PH Wrought and LPBF

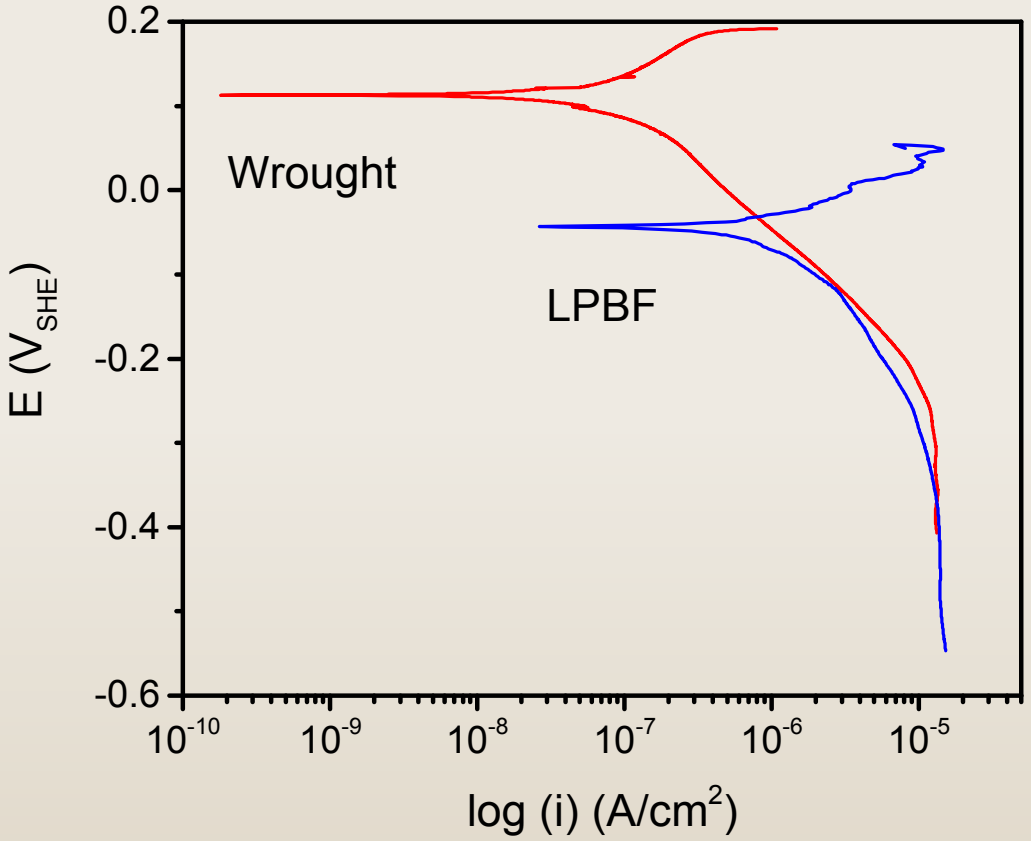




- LPBF displays a lower E_{OCP} than wrought 17-4 PH
- LPBF displays a diminished passive region post 1 h E_{OCP}, whereas post 24 E_{OCP} there is a larger passive region
  - Possibly indicative of the pores passivating with time

Global Scale Electrochemistry:
17-4 PH Wrought and LPBF

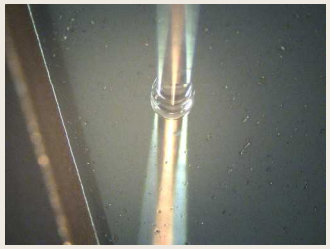
- Again, LPBF displays a lower E_{OCP} than wrought 17-4 PH
- Both LPBF and wrought 17-4 PH display similar cathodic kinetics

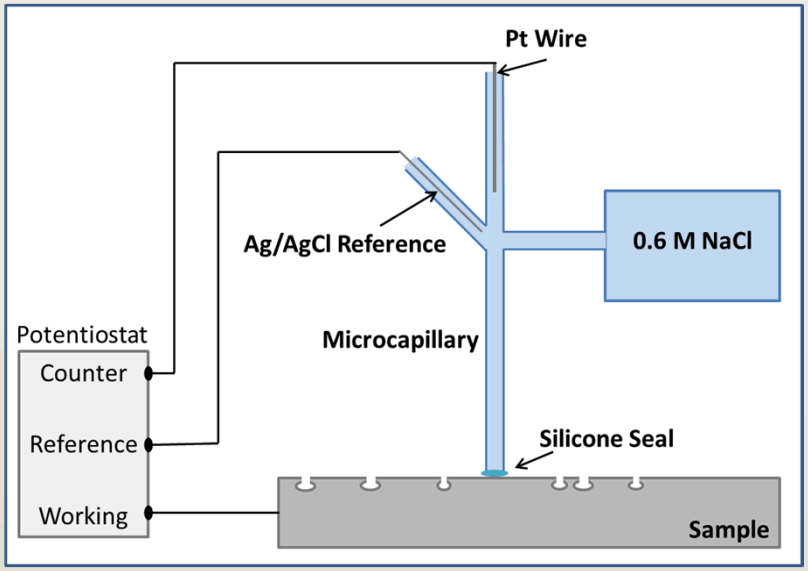


Local Scale Electrochemistry:
17-4 PH Wrought and LPBF

Micro-electrochemical cell:

- 0.6 M ambiently aerated NaCl solution
- 1 h E_{OCP}
- Potentiodynamic Polarizations:
  - Scan rate of 1 mV/s from -200 to +500 mV vs E_{OCP}
- 380 µm diameter capillary
- Tested pores ≥ 50 µm and areas with pores ≤ 10 µm

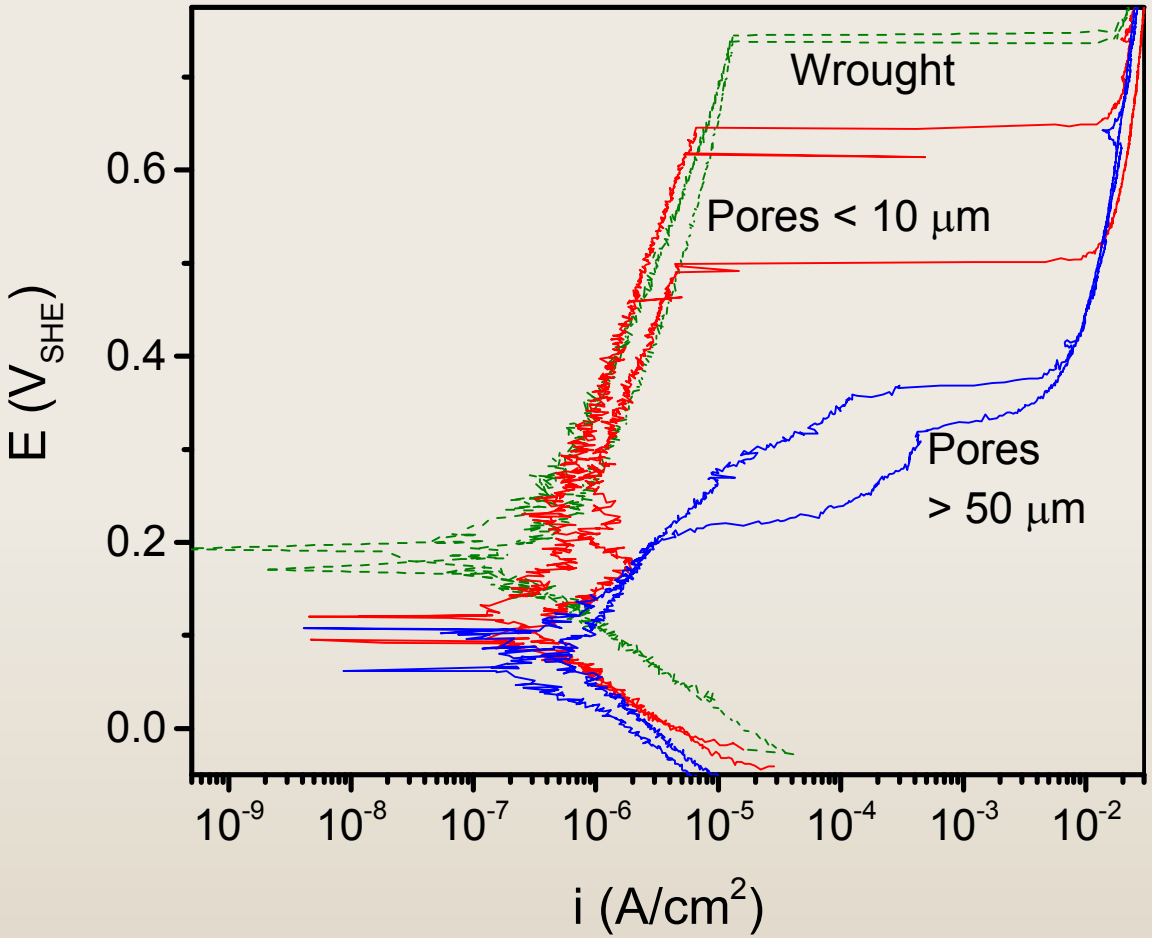




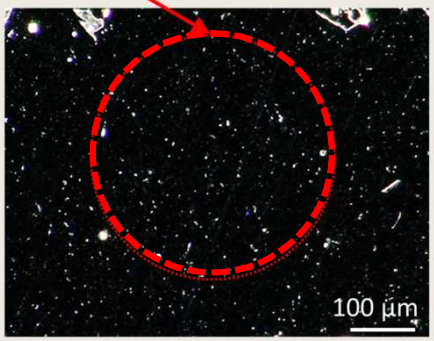
(Birbilis, 2005, Birbilis, 2009, Lohrengel, 2004, Bucheit, 2001, Suter, 2001)

Local Scale Electrochemistry:

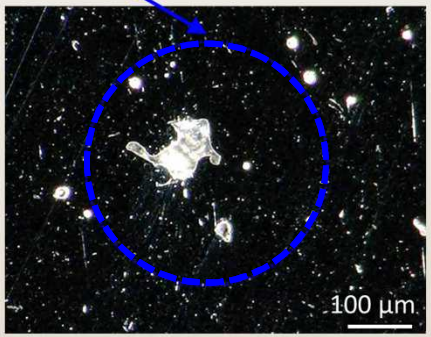
17-4 PH Wrought and LPBF



Pores < 10 μm
Micro-electrochemical Cell Area



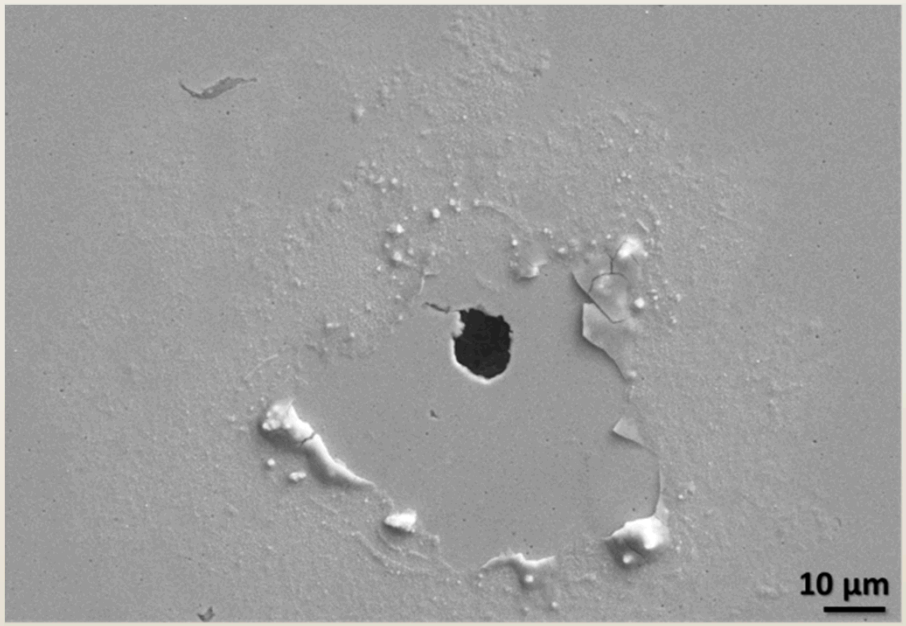
Pores > 50 μm
Micro-electrochemical Cell Area

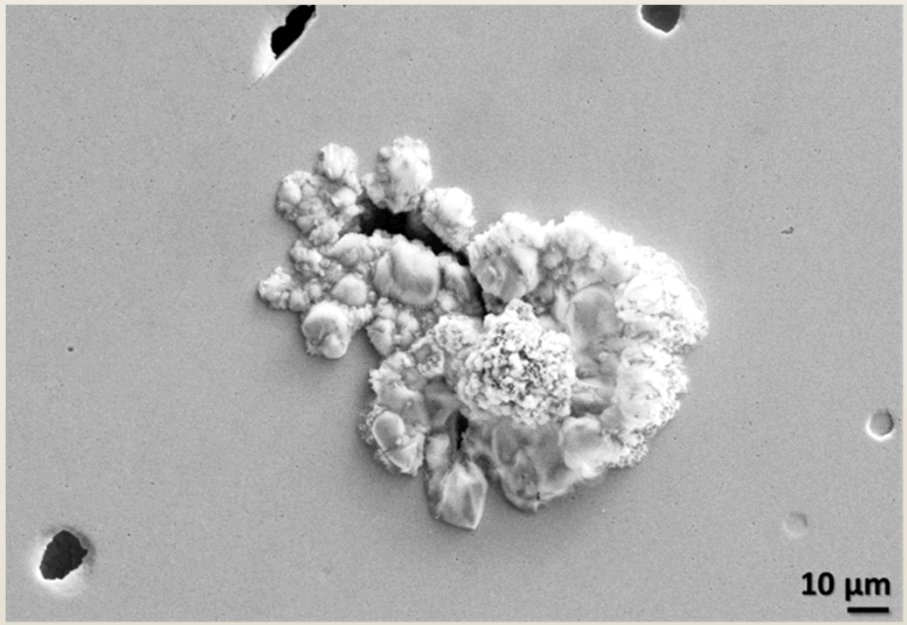


Pores > 50 μm display a decreased E_{OCP} and passive region compared to the pores < 10 μm and the wrought material.

Full Immersion Exposure: 17-4 PH LPBF

LPBF 17-4 PH after a 7 day open circuit exposure in quiescent 0.6 M NaCl



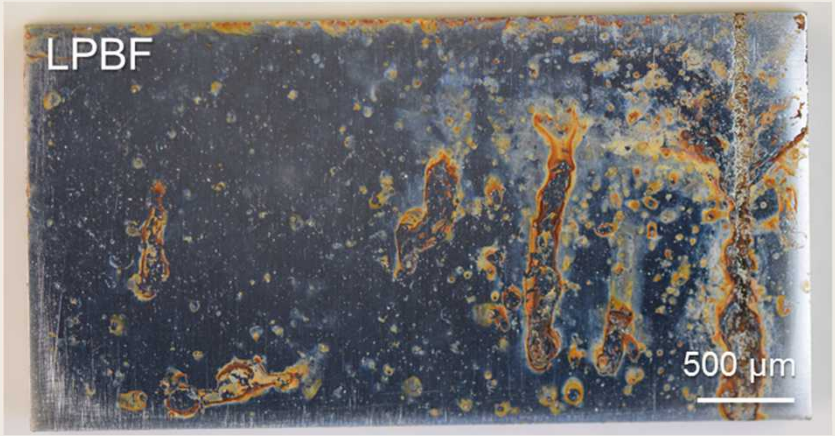


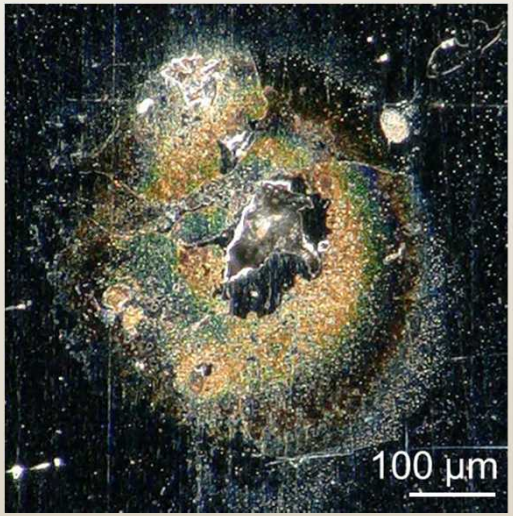
SEM micrographs illustrating area where corrosion product build up has flaked off revealing pore beneath (left) and corrosion product build up over a pore (right).

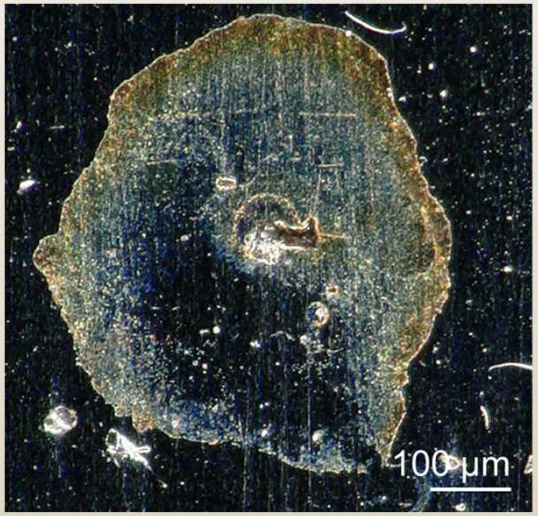
Atmospheric Exposure:

17-4 PH Wrought and LPBF

ASTM B-117: 2 week Exposure







Corrosion near pores post ASTM B-117

Conclusions

- LPBF 17-4 PH versus conventional wrought material exhibited decreased corrosion resistance through electrochemical testing.
-
  - Micro-electrochemical cell experiments established active corrosion above the large pores (d ≥ 50 μm) rather than the passive behavior displayed above regions with smaller pores (d ≤ 10 μm) and wrought material.
  - Further evidence of enhanced corrosion at pores was confirmed by post-exposure analysis of full immersion exposures, where corrosion appeared to initiate at or near pores.
- As pores were found the primary influence on corrosion, Future work should focus on the effects of pore size and aspect ratio on the corrosion properties of the material.
  - An empirical or theoretical model relating pore size to corrosion susceptibility would improve understanding of morphological effects.
  - With the current results, a decrease in surface porosity, either through processing or post-processing treatments would be expected to enhance the corrosion properties of AM 17-4 PH.

Acknowledgements

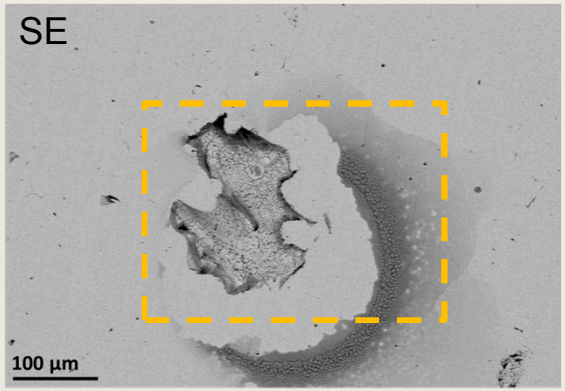
- Bonnie McKenzie, Brendan Nation, Alice Kilgo, Richard Grant

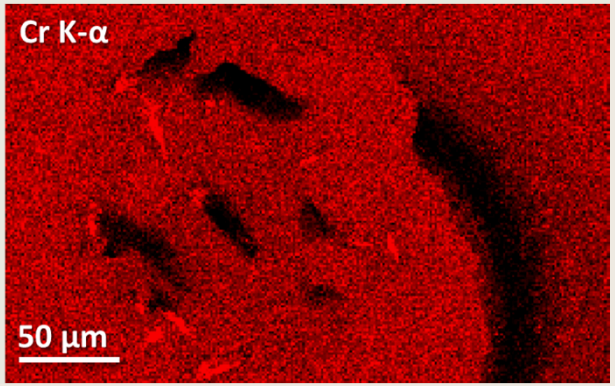
- Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

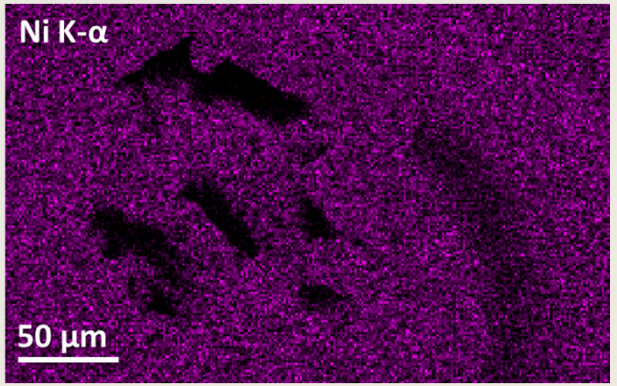
- Questions?

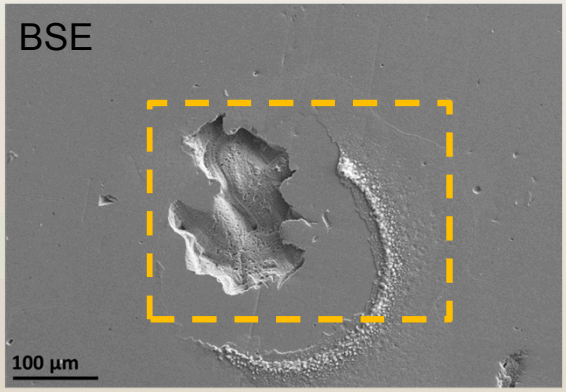
Atmospheric Exposure:
17-4 PH LPBF

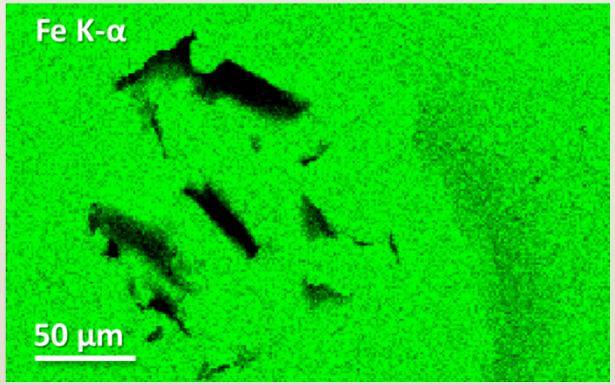
ASTM B-117: 2 week Exposure

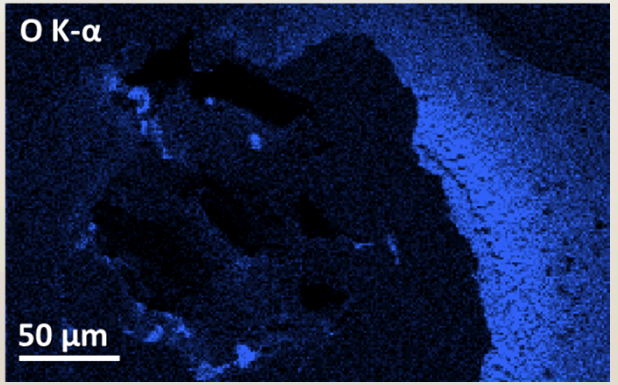












Corrosion occurred at or near pores.