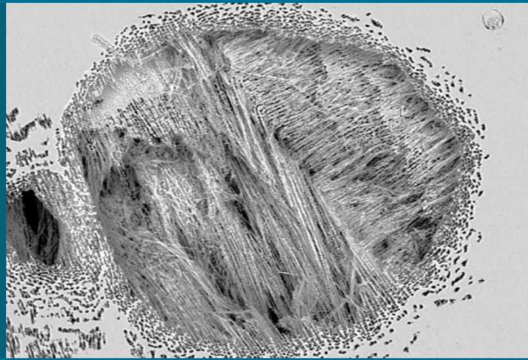
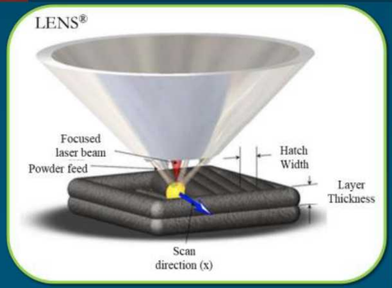
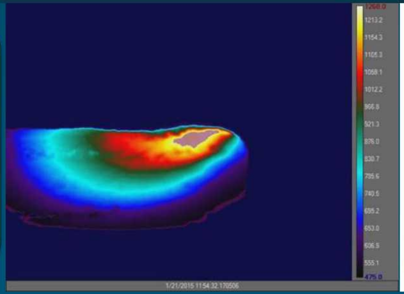
Sandia National Laboratories

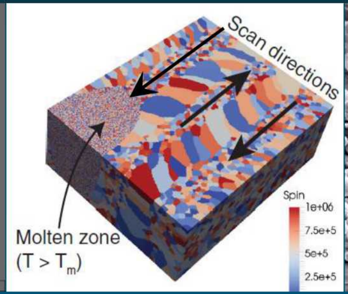
SAND2019-11645C

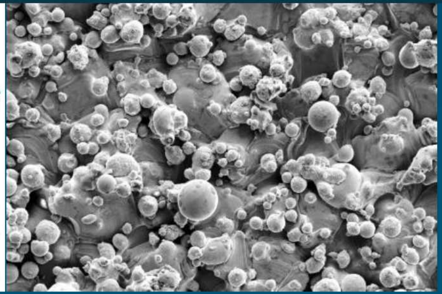
Corrosion of Additively Manufactured Stainless Steels – Process, Structure, and Performance











PRESENTED BY

Presenter: Michael A. Melia

Co-authors: (SNL) Jesse Duran, Jeffery M. Rodelas, Eric J. Schindelholz

U.S. DEPARTMENT OF ENERGY NNSA

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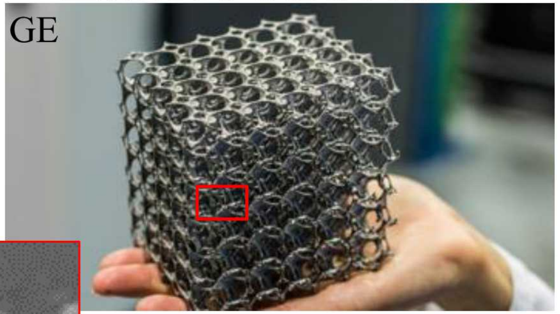
Why AM?

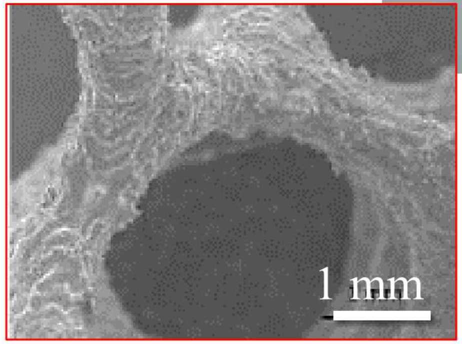
Sophisticated, unconventional 3D geometries

Lattice structure



GE

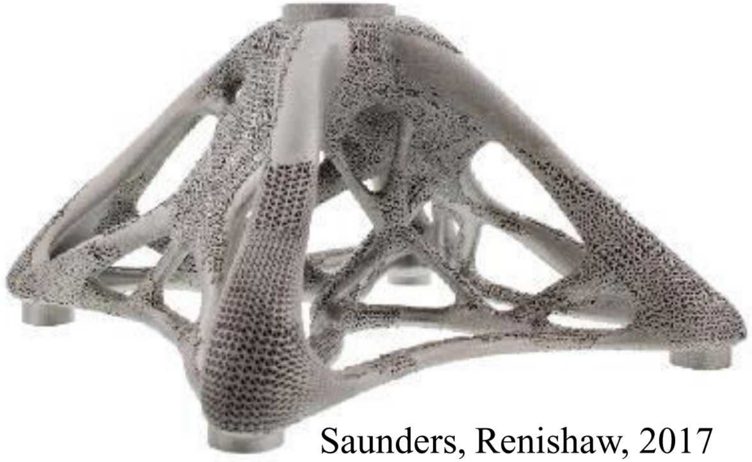
Yan, 2014



Cunningham, 2017

Heat exchanger

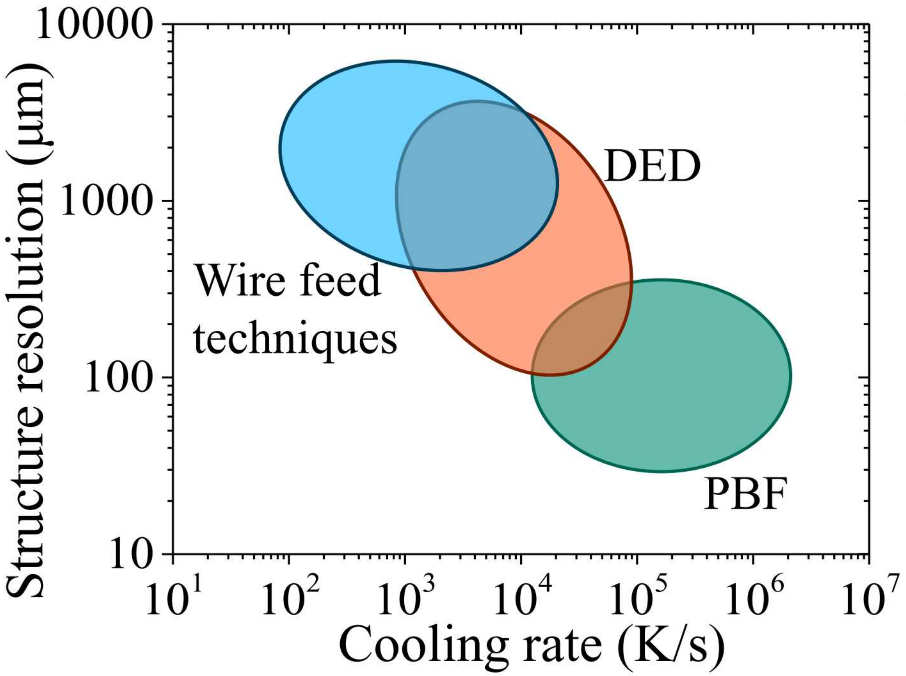
A design/process-pathway to lightweight-high strength parts

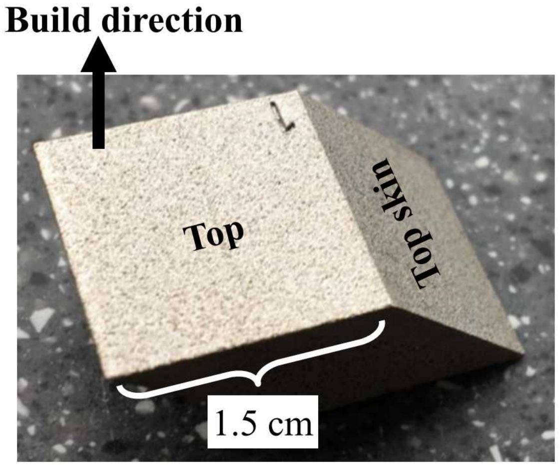


Saunders, Renishaw, 2017

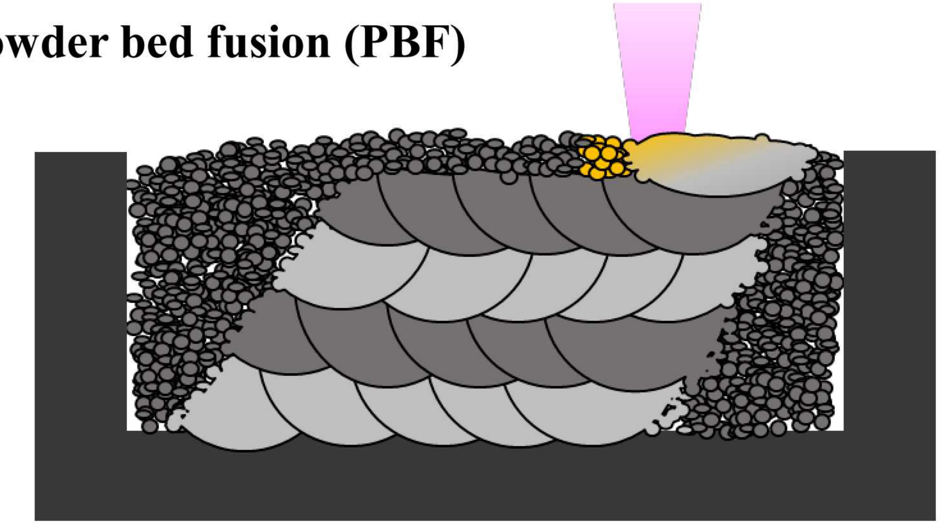
Topology optimized design

Multiple techniques ranging resolution and cooling rate scales

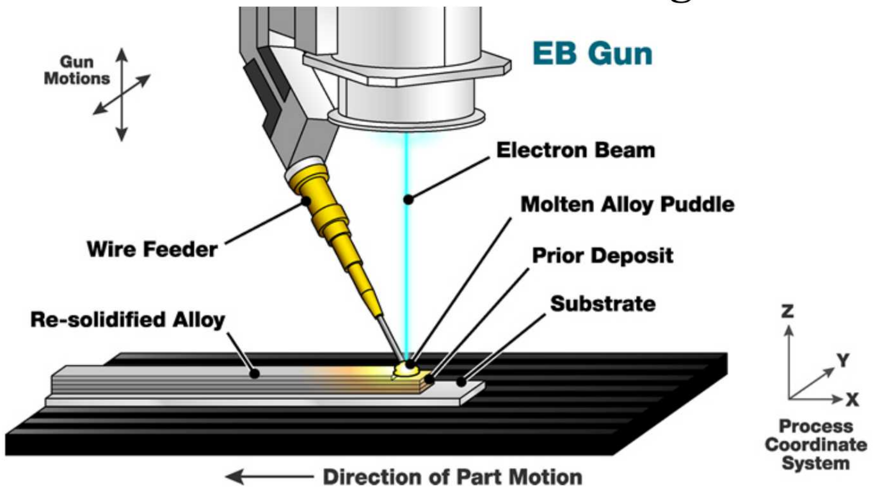




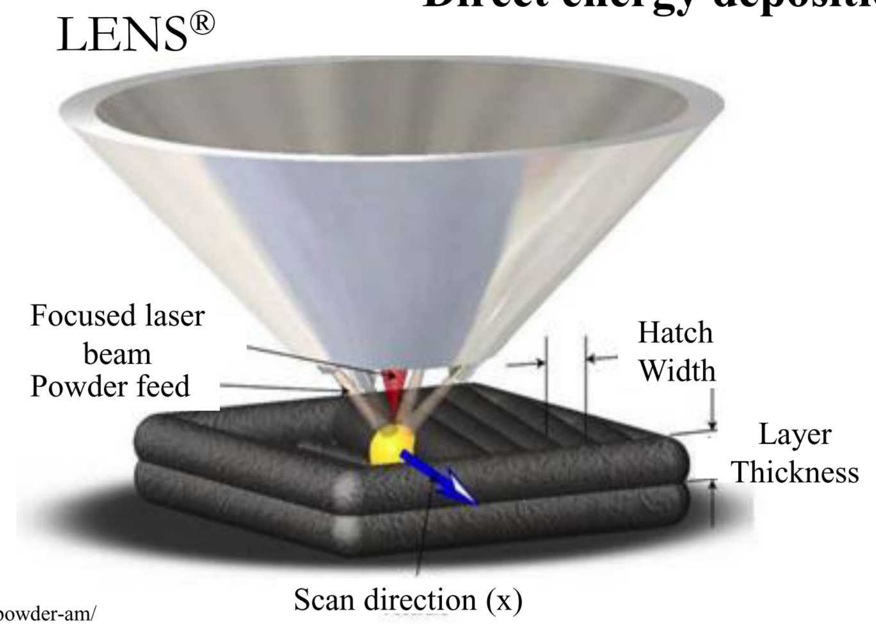
Powder bed fusion (PBF)

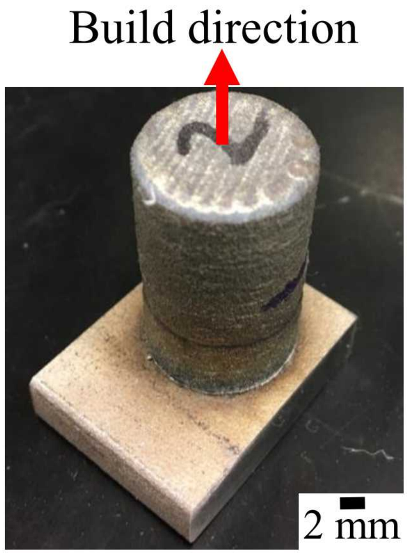


e⁻ beam wire-feed melting



Direct energy deposition (DED)

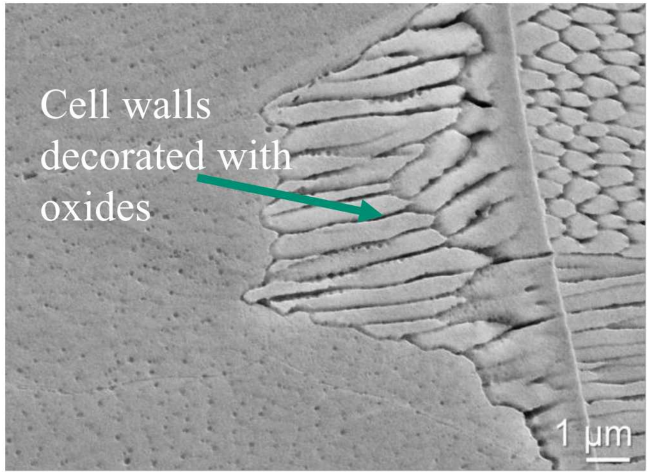


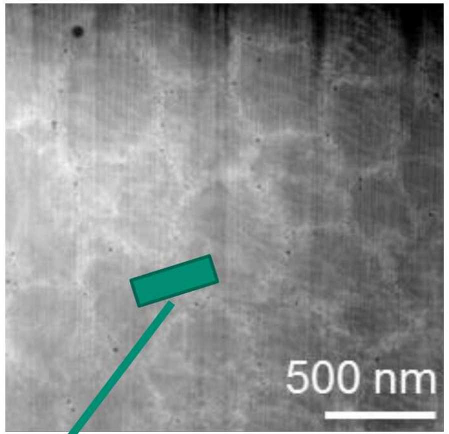


http://additivemanufacturing.com/2015/10/14/electron-beam-additive-manufacturing-ebam-advantages-of-wire-am-vs-powder-am/

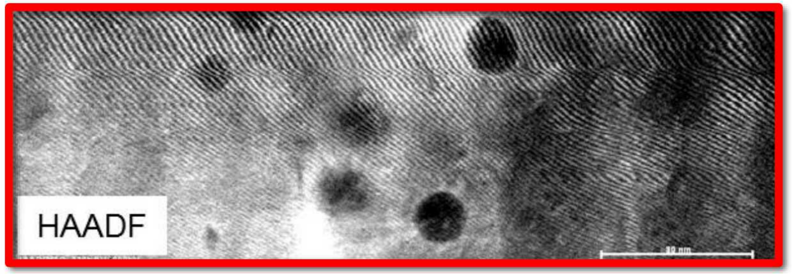
What do all of these techniques have in common?

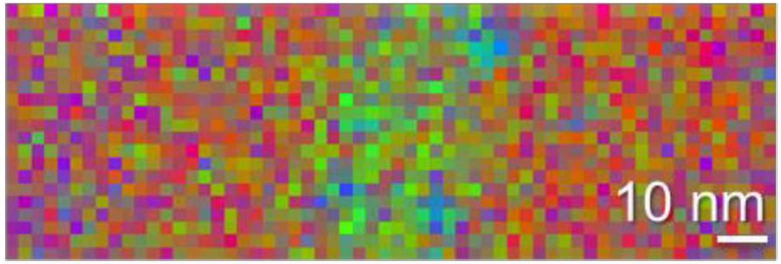
Chemical heterogeneities





Matrix Interface Matrix

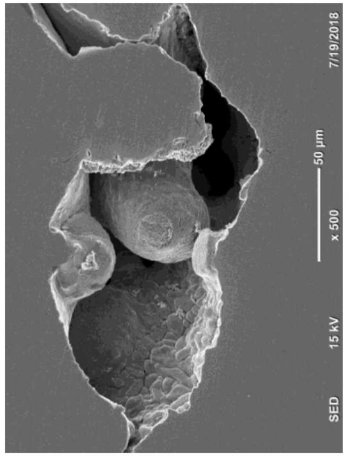


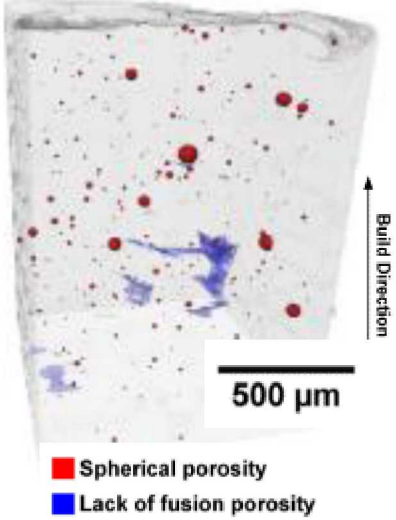


- Increased Fe (+ 9%)
- Increased Si
- Increased Cr (+ 4%)

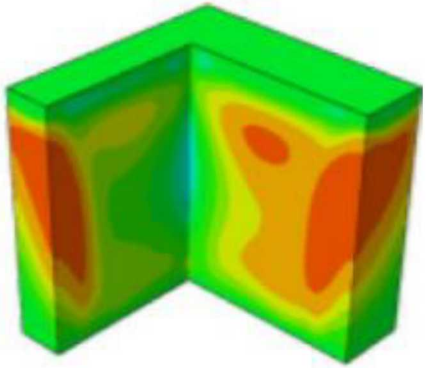
Processing defects

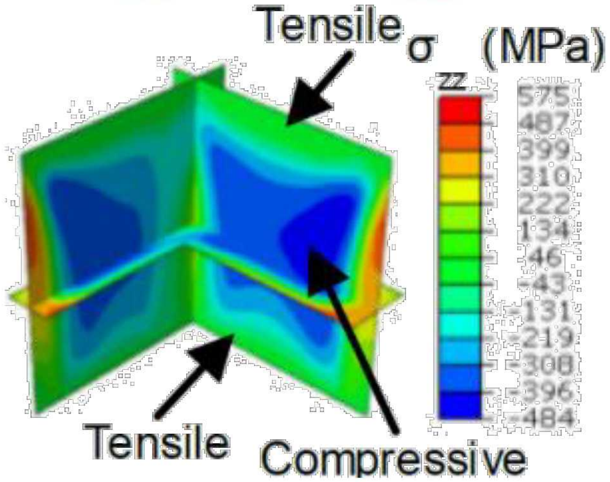
Porosity





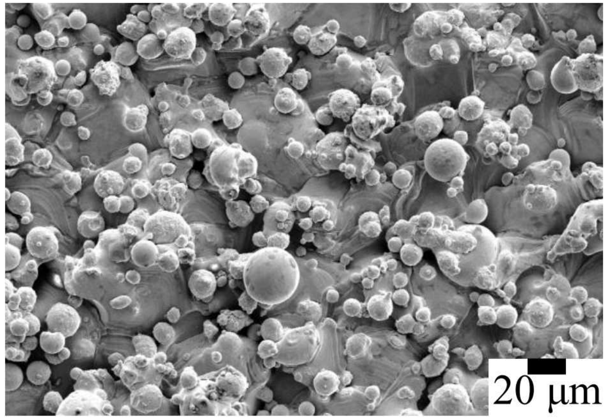
Residual stress





Li, 2018

Surface roughness



Outline

Focus areas

1. Impact of **surface finish** on the initiation of local corrosion and possible mitigation strategies.
   - Surface roughness controls the susceptibility to local corrosion initiation

2. Preferential corrosion attack at **melt pool boundaries (MPBs)**.
   - Solute depletion at MPBs leads to preferential corrosion along them in concentrated oxidizing environments.

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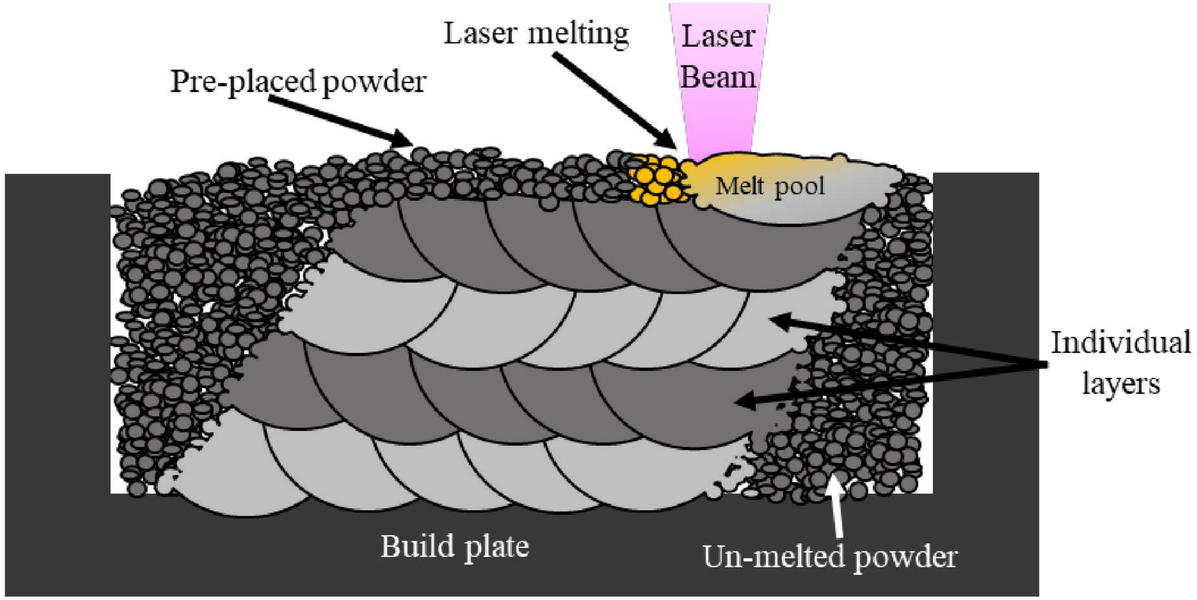
Powder bed fusion 316L samples

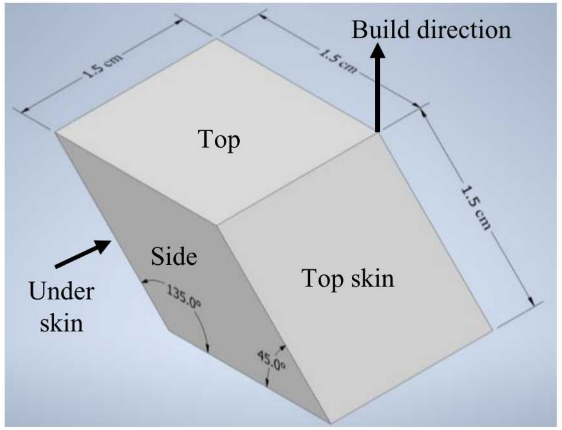
wt.%	C	Cr	Cu	Fe	Mn	Mo	N	Nb	Ni	O	P	S	Si	PREN
PBF 316L as-built	0.013	16.87	0.039	65.5	1.54	2.31	0.078	0.001	12.74	0.055	0.015	0.006	0.71	25.7

Samples were prepared using 316L powder with a powder bed fusion (PBF) technique.

Parameter	Value
Laser power	110 W
Laser velocity	1400 mm/sec
Layer thickness	30 µm
Laser focus offset	+1 mm
Average powder diameter	12 µm
Cover gas	Argon



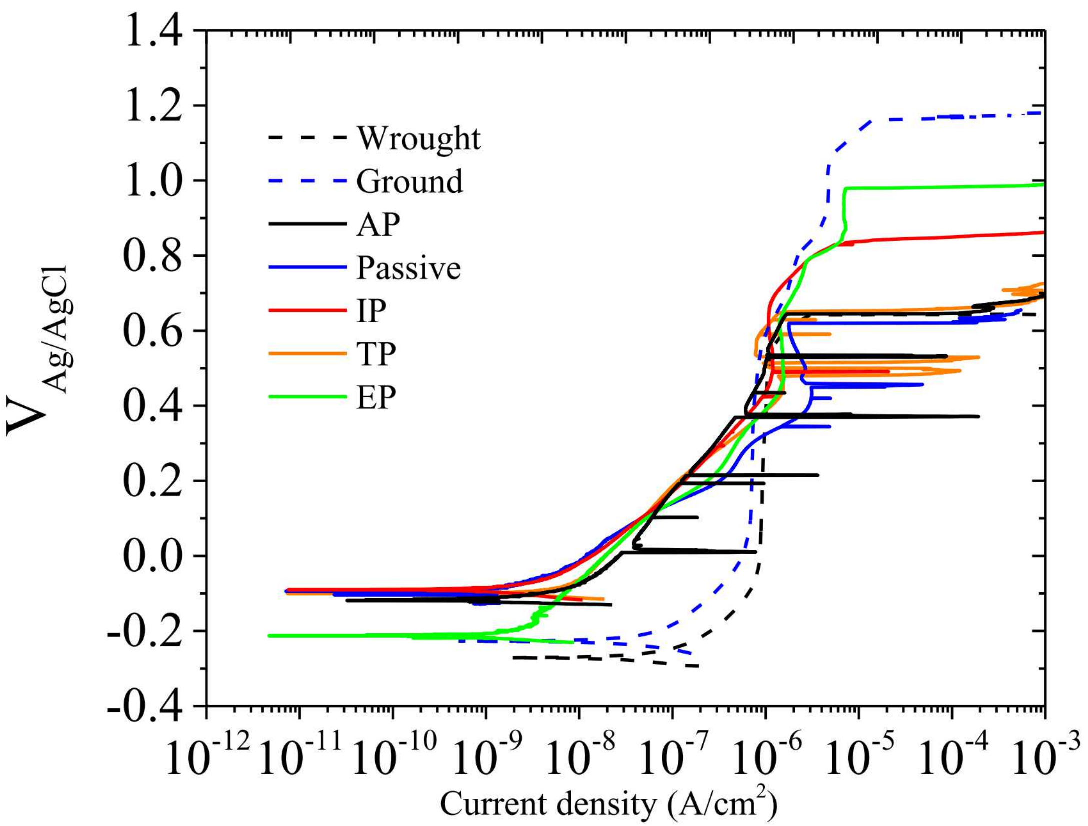
Laser pattern turns 90 degrees every layer and the starting position changes every layer. Identical laser scan pattern occurs every 4th layer.



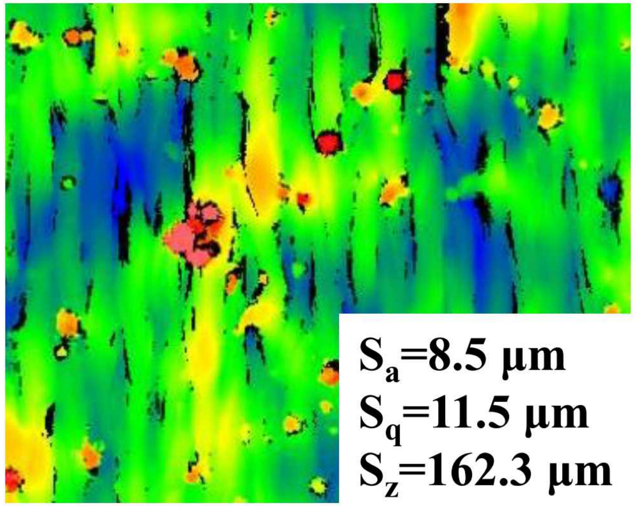
Experimental approach

Surface finishing procedures:

- **As-printed (AP)**
- **Electro-polished (EP)**
  - 50% Phosphoric acid, 20% Sulfuric acid, 30% water.
- **Tumble polished (TP)**
  - Fast cutting ceramic (Triangular) media.
- **In processed laser polishing (IP)**
  - Contour build pattern.
- **Passivation (P)**
  - Immersed in 45% nitric acid for 30 minutes.
- **Grinding with SiC paper to 1200 grit (G)**



Optical white light profilometry and SEM for surface roughness.



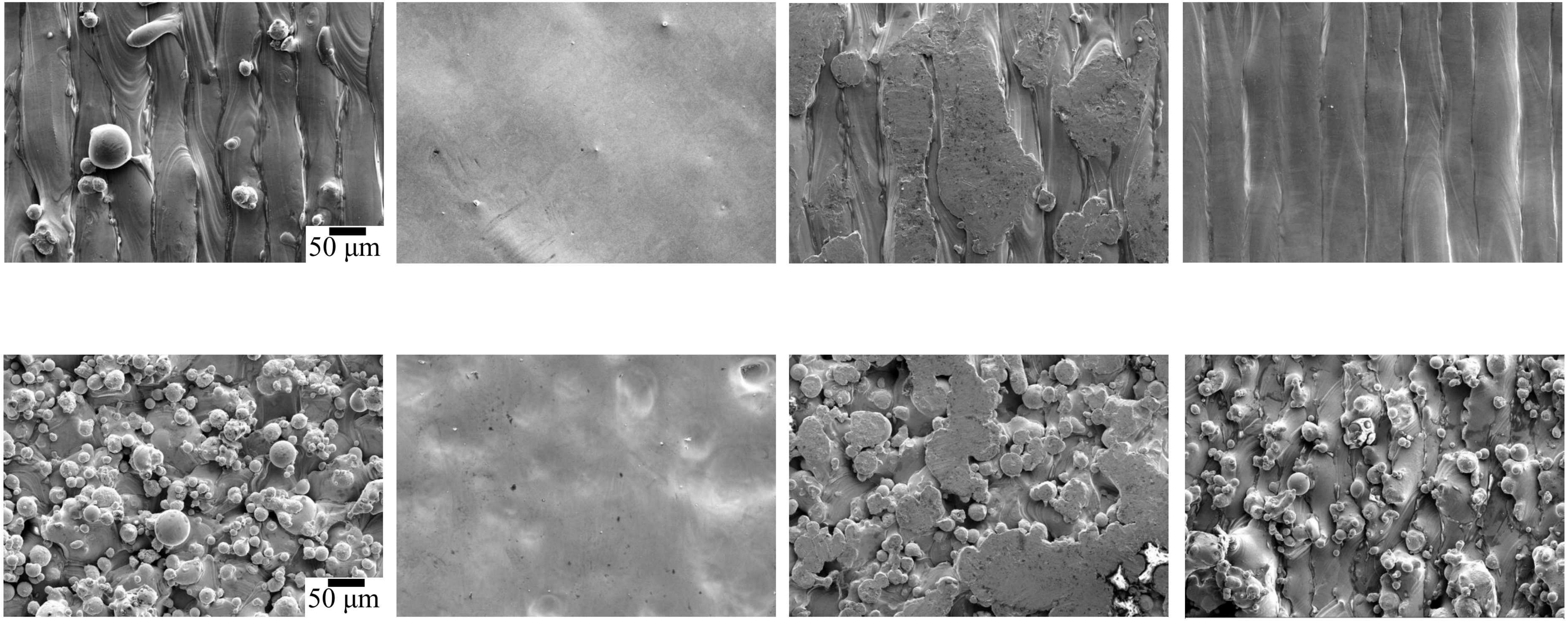
Anodic potentiodynamic polarization in quiescent 0.6 M NaCl – after 1 hour open circuit potential.

Surface images of various surface finishes

As-printed (AP) | Electro-polished (EP) | Tumble polished (TP) | In process laser polish (IP)

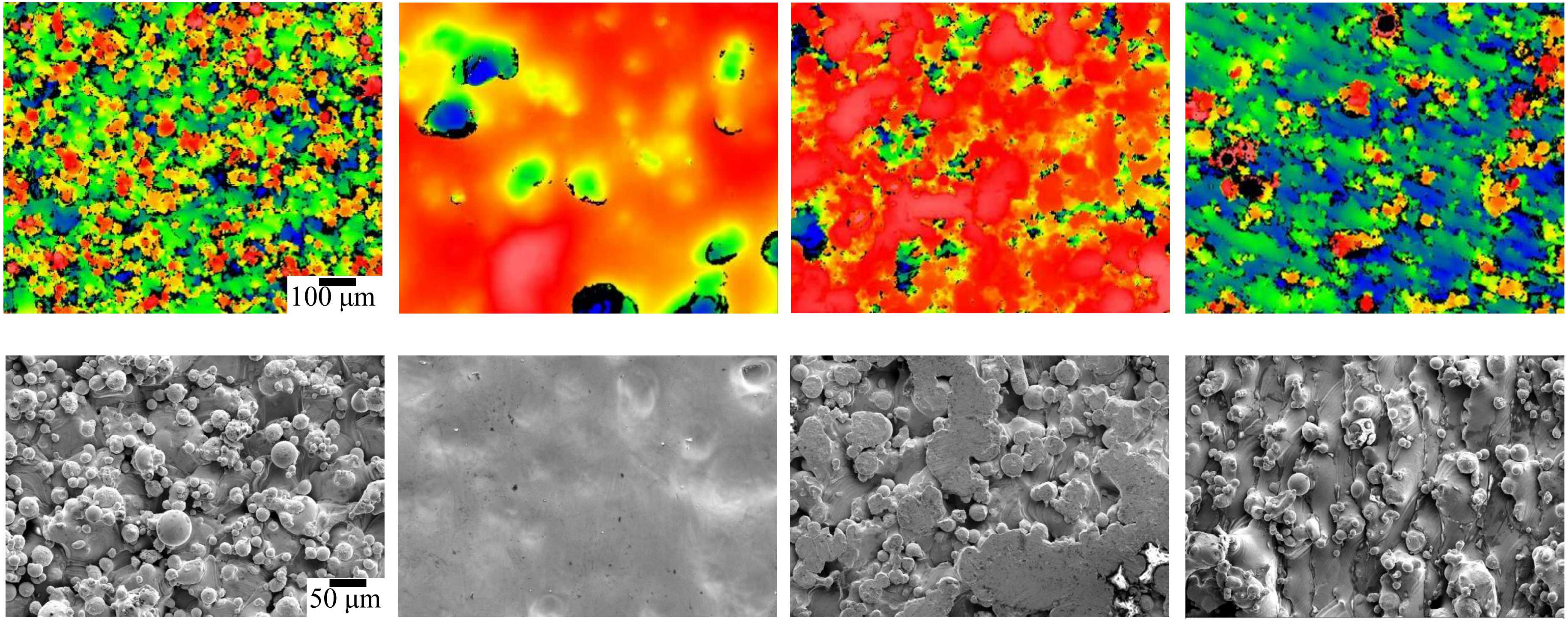
Top

Side

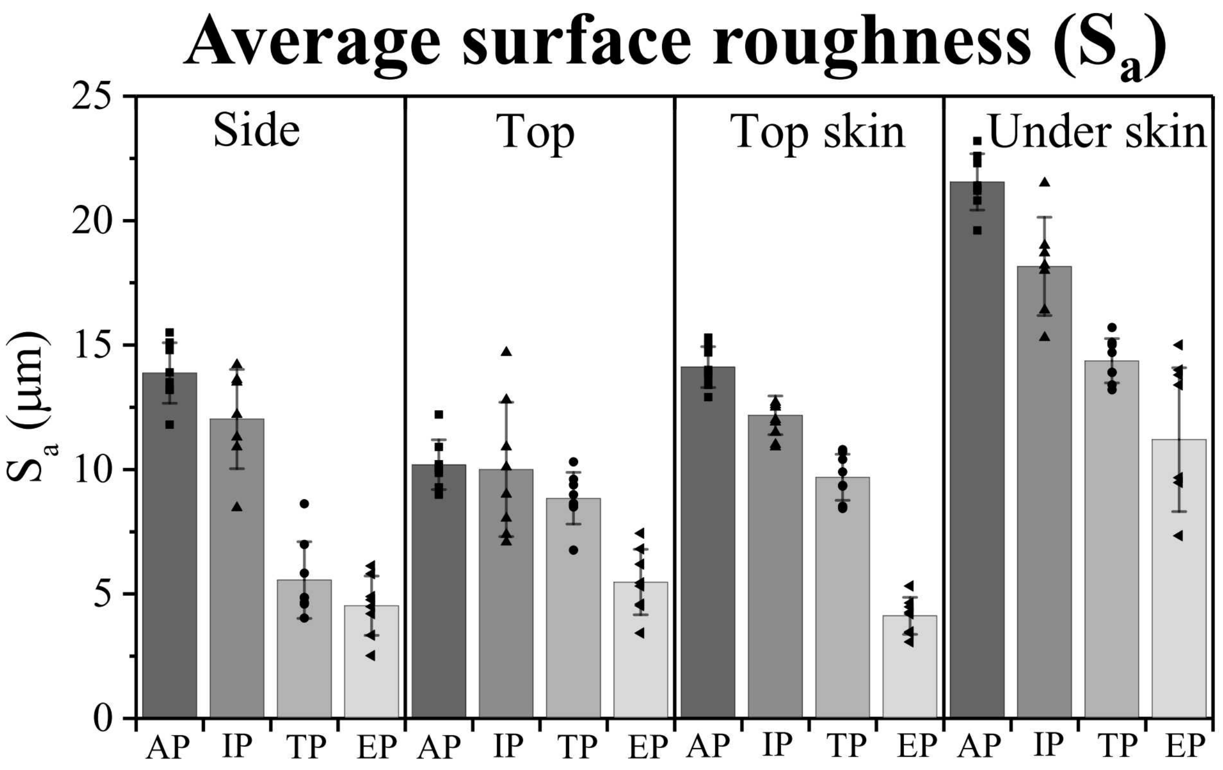


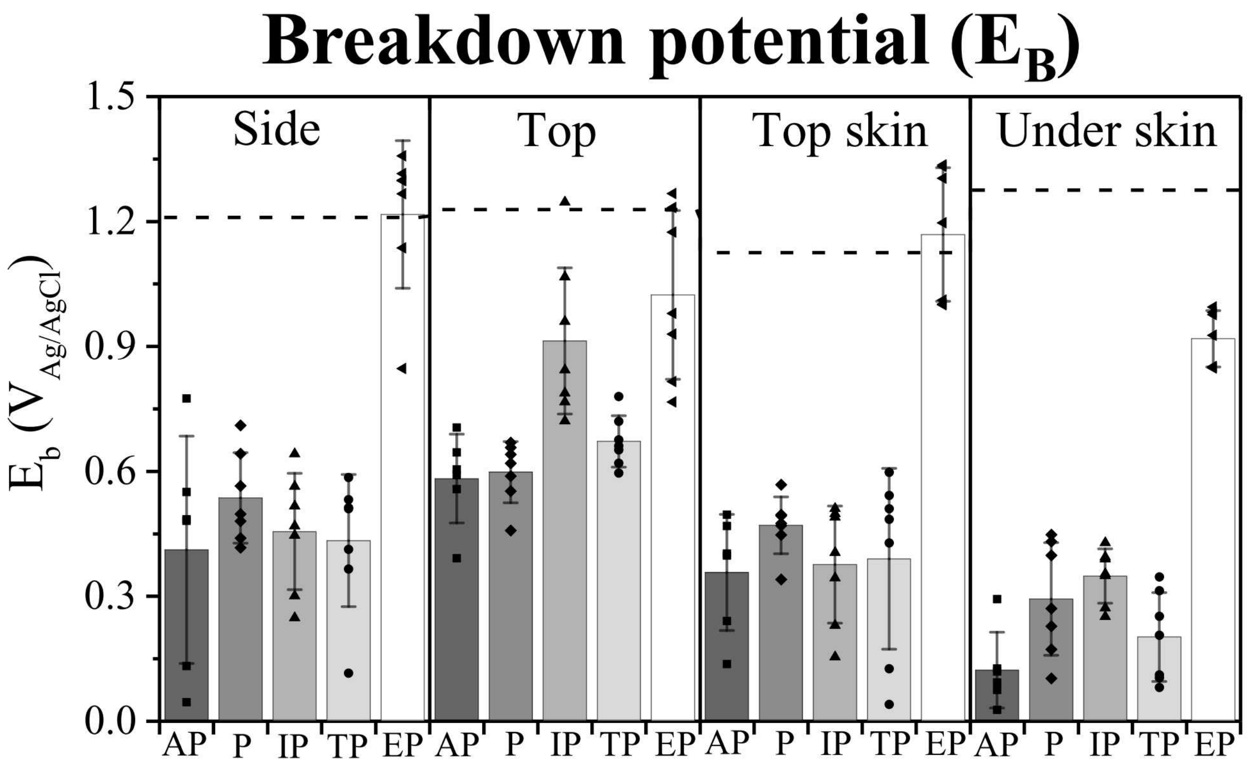
Roughness of various surface finishes – Side orientation

As-printed (AP) | Electro-polished (EP) | Tumble polished (TP) | In process laser polish (IP)



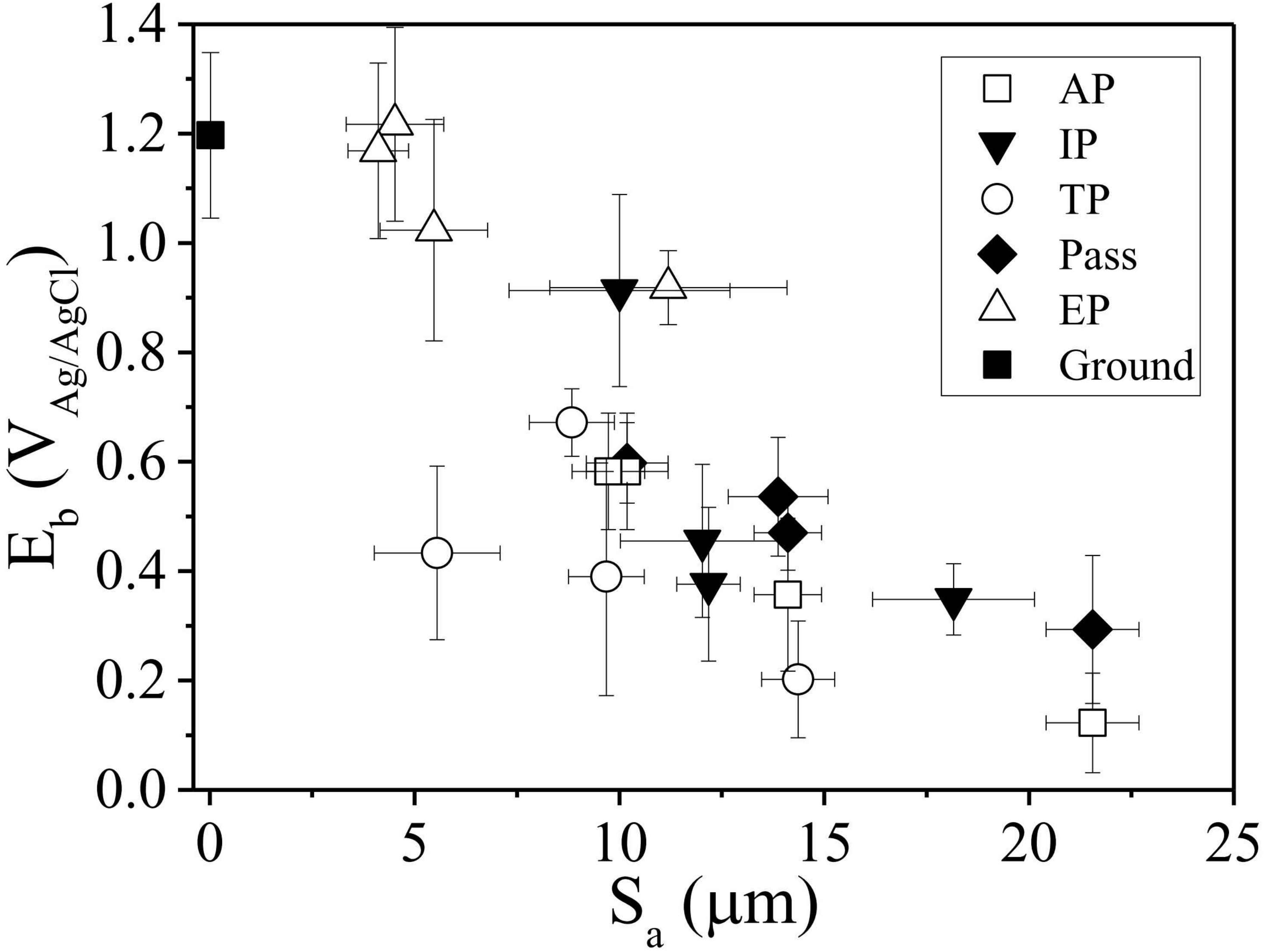
Average and range of S_a and E_b





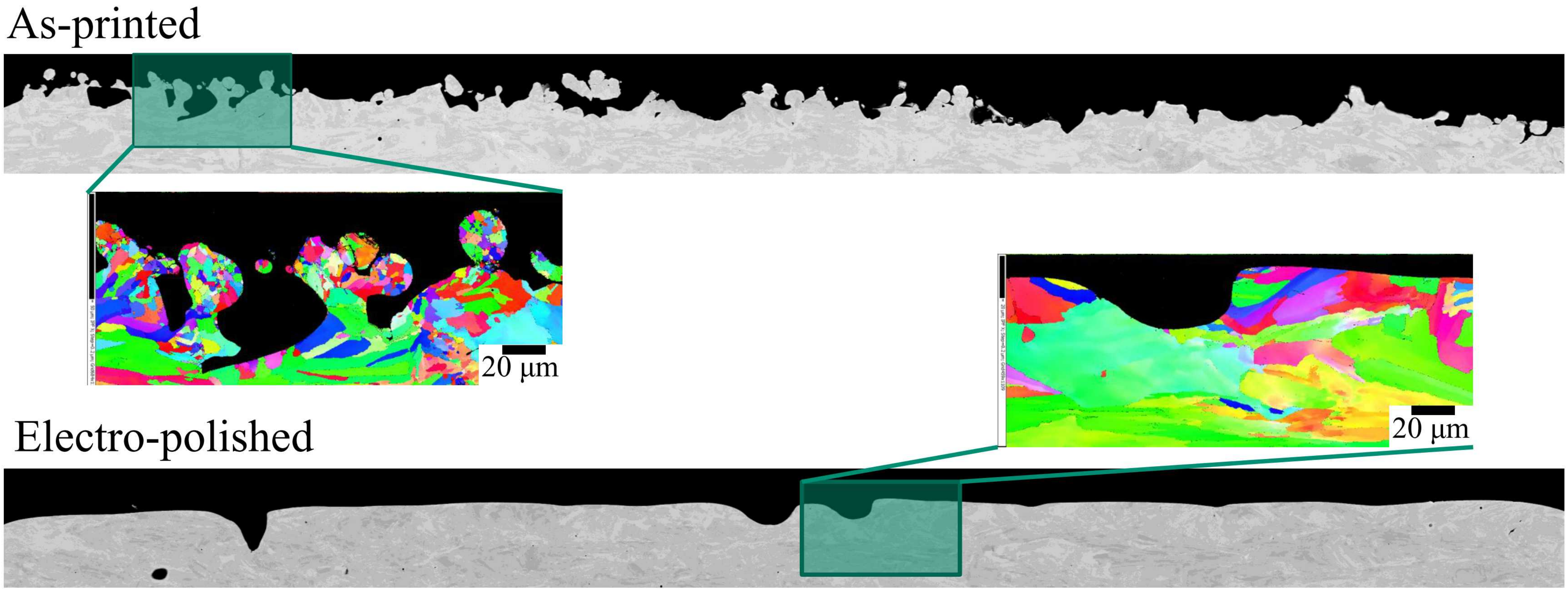
The horizontal dashed lines represents the E_b for the ground condition.

Comparison between S_a and E_b



Scatter plots of E_b with respect to roughness measurements S_a. Error bars represent one standard deviation for all measurements.

Besides roughness, what will be other controlling factors of corrosion initiation?

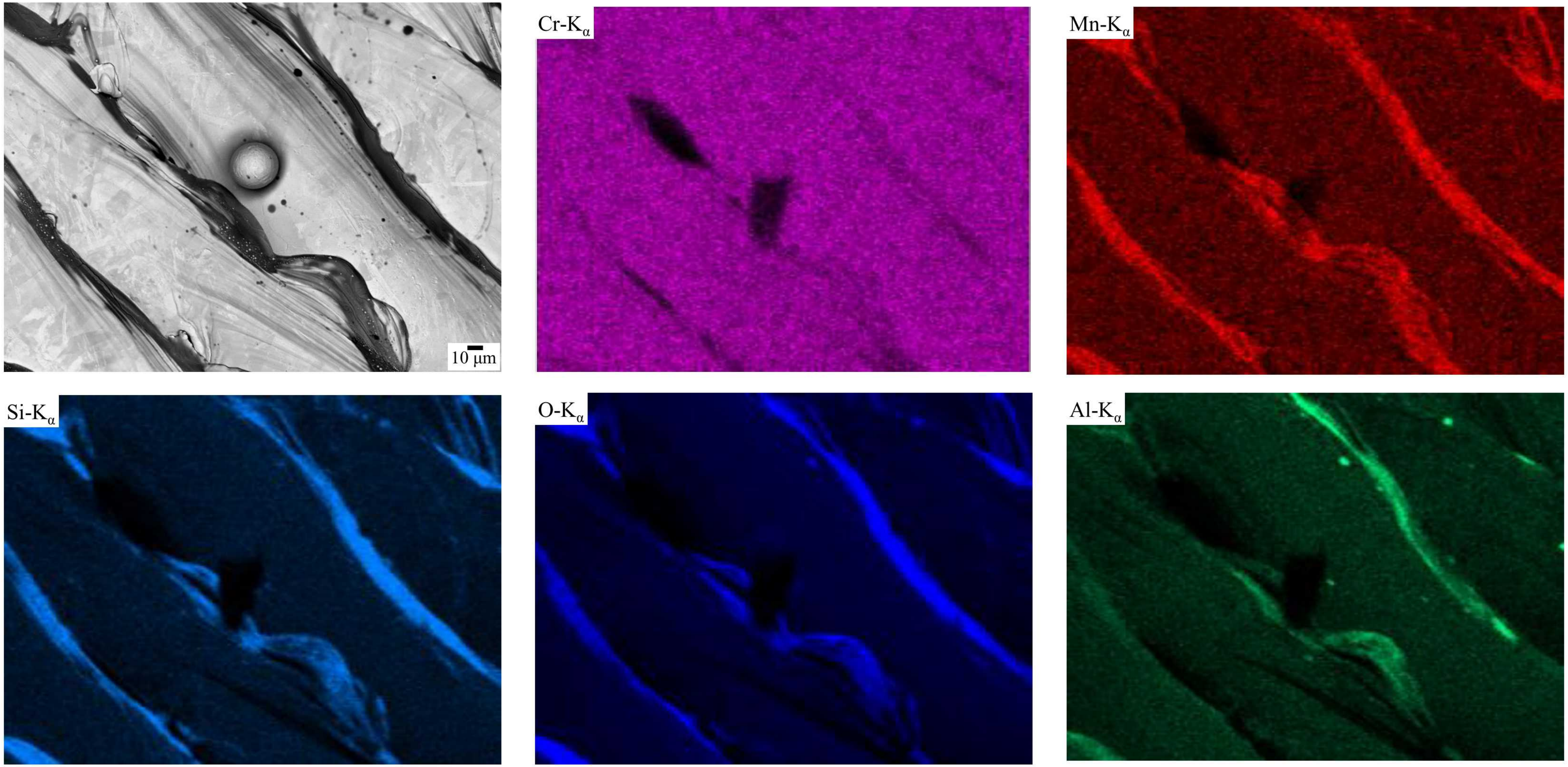


Microstructural differences.

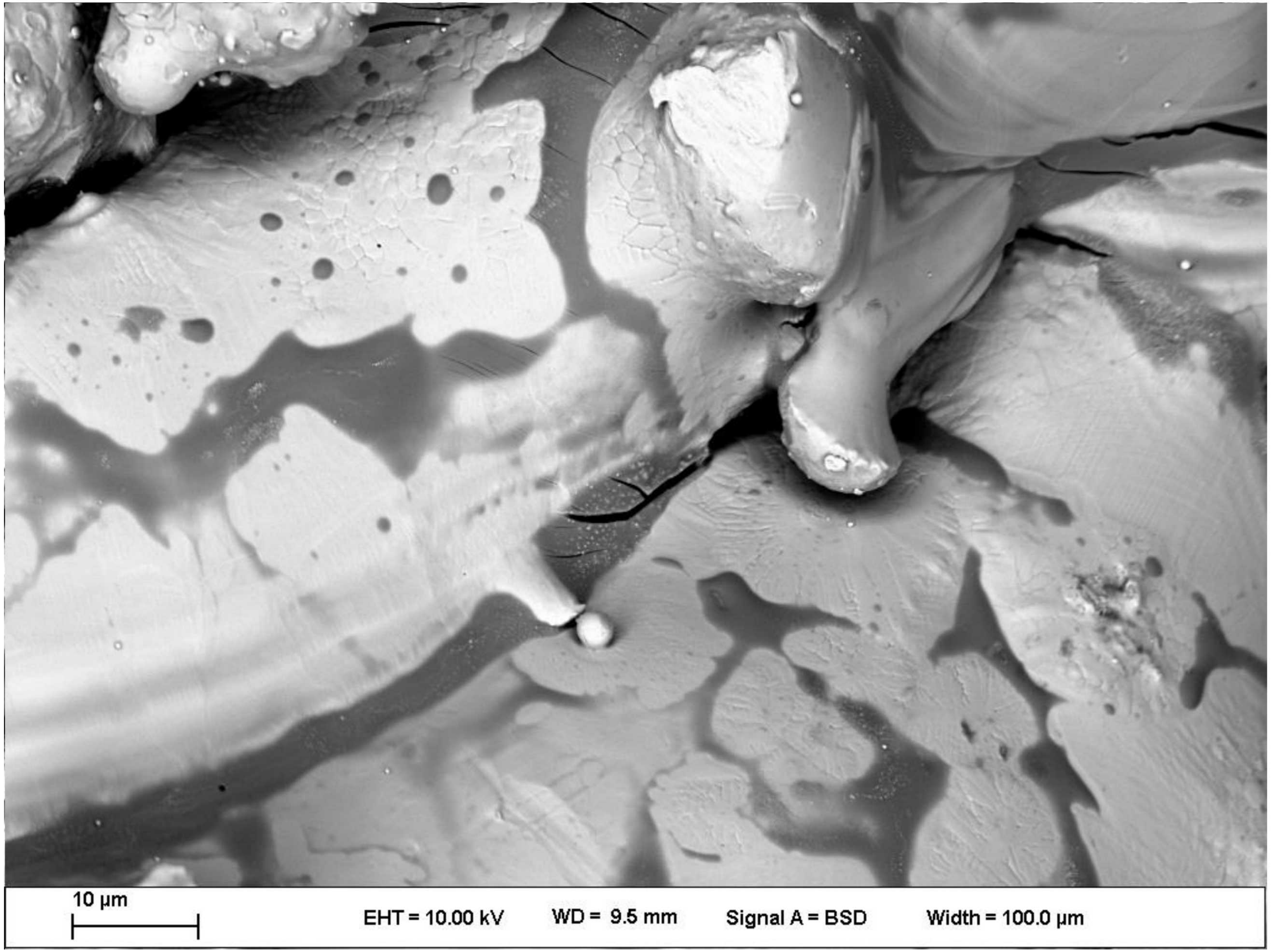
Will tortuosity more accurately predict corrosion susceptibility?

- More accurate depiction of roughness.

EDS maps of **AP Top** surface showing oxide in between layers.

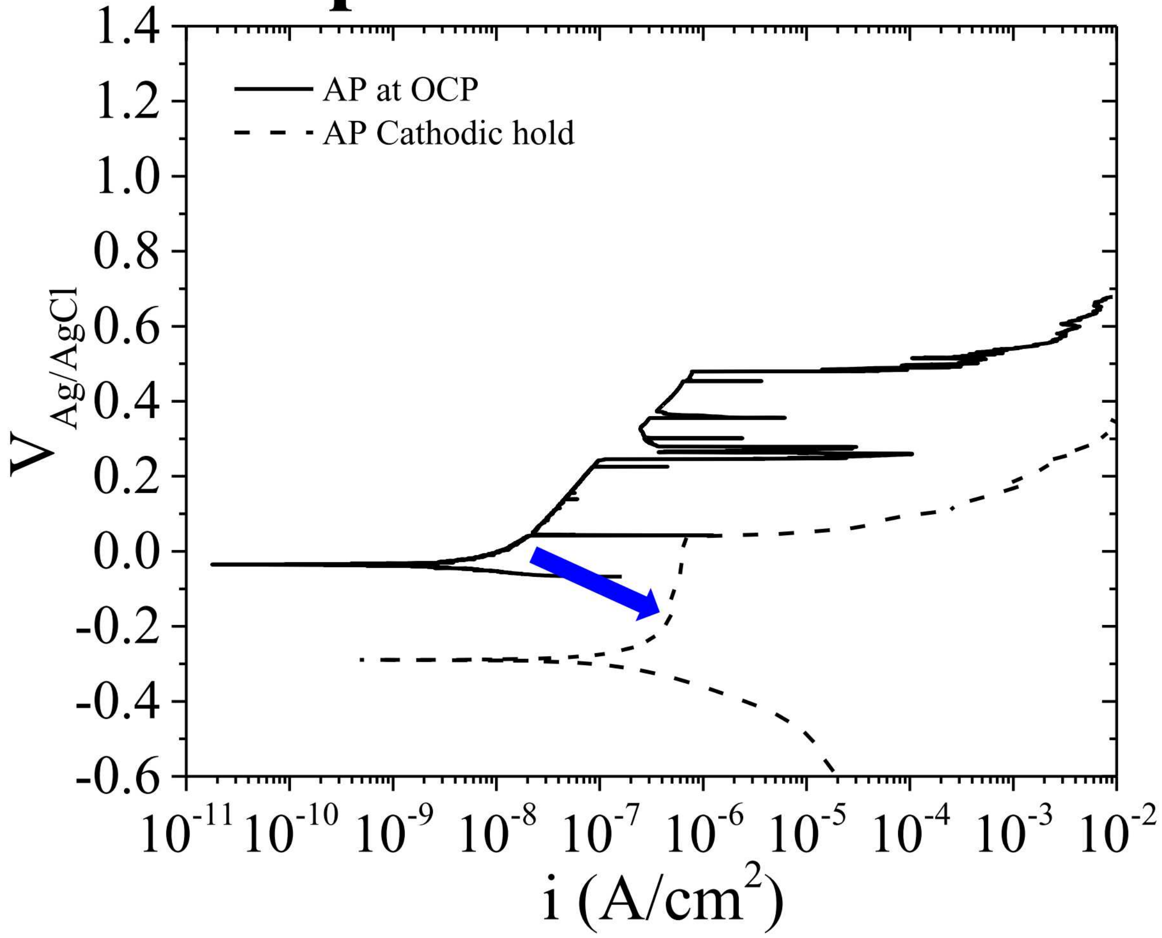


Oxide formation on Underskin orientation

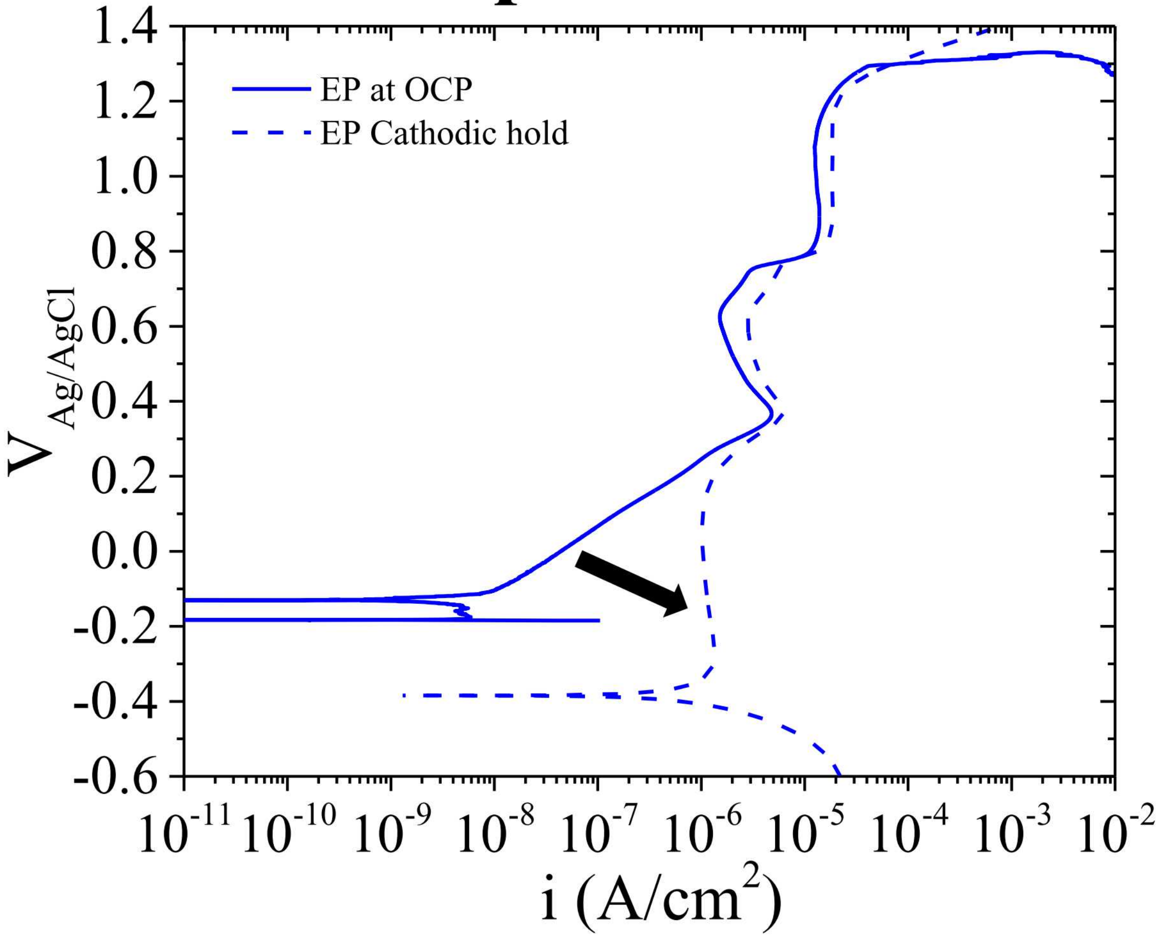


E_b after removing oxides

As-printed



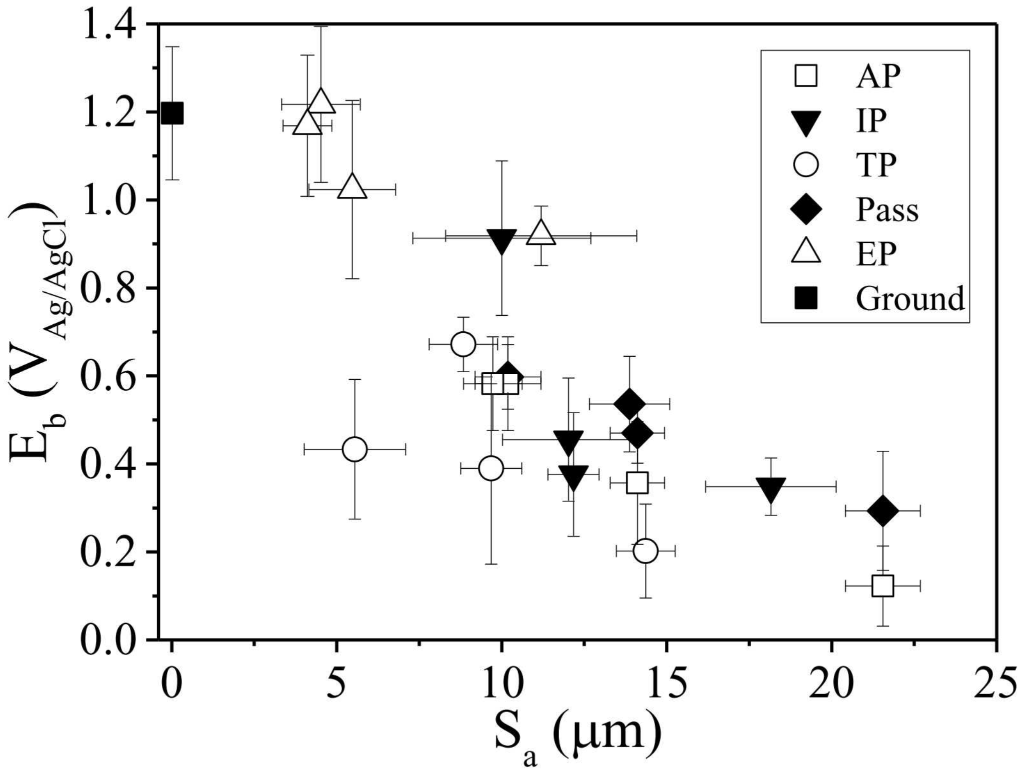
Electro-polished

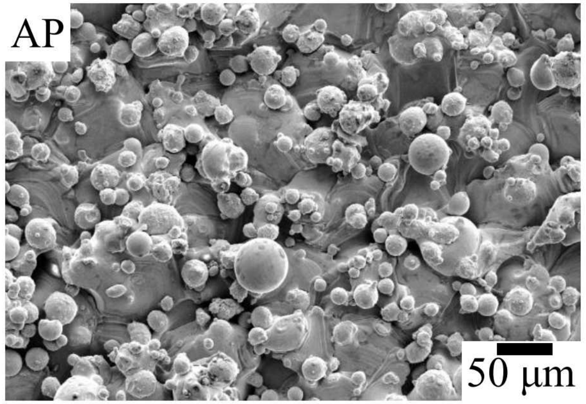


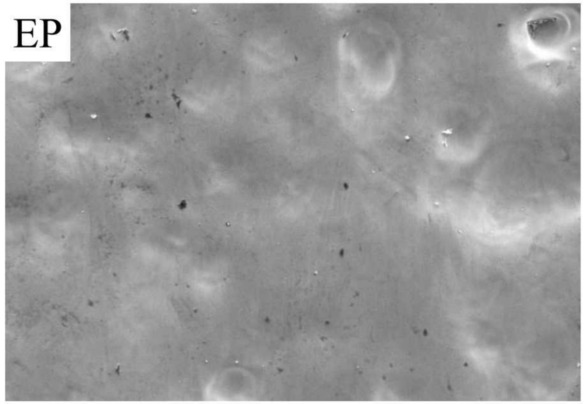
Sample orientation: **Side**

Surface finish conclusions

- Surface roughness is controlling the initiation of pitting to the first order with good correlation between S_a and E_b.
- EP increased the E_b and reduced the S_a for all build orientations compared to AP.
- IP, TP, and passivation provide no significant increase to E_b.
- Surface roughness/tortuosity effects dominate pit initiation compared to the improvement from oxides formed during processing.







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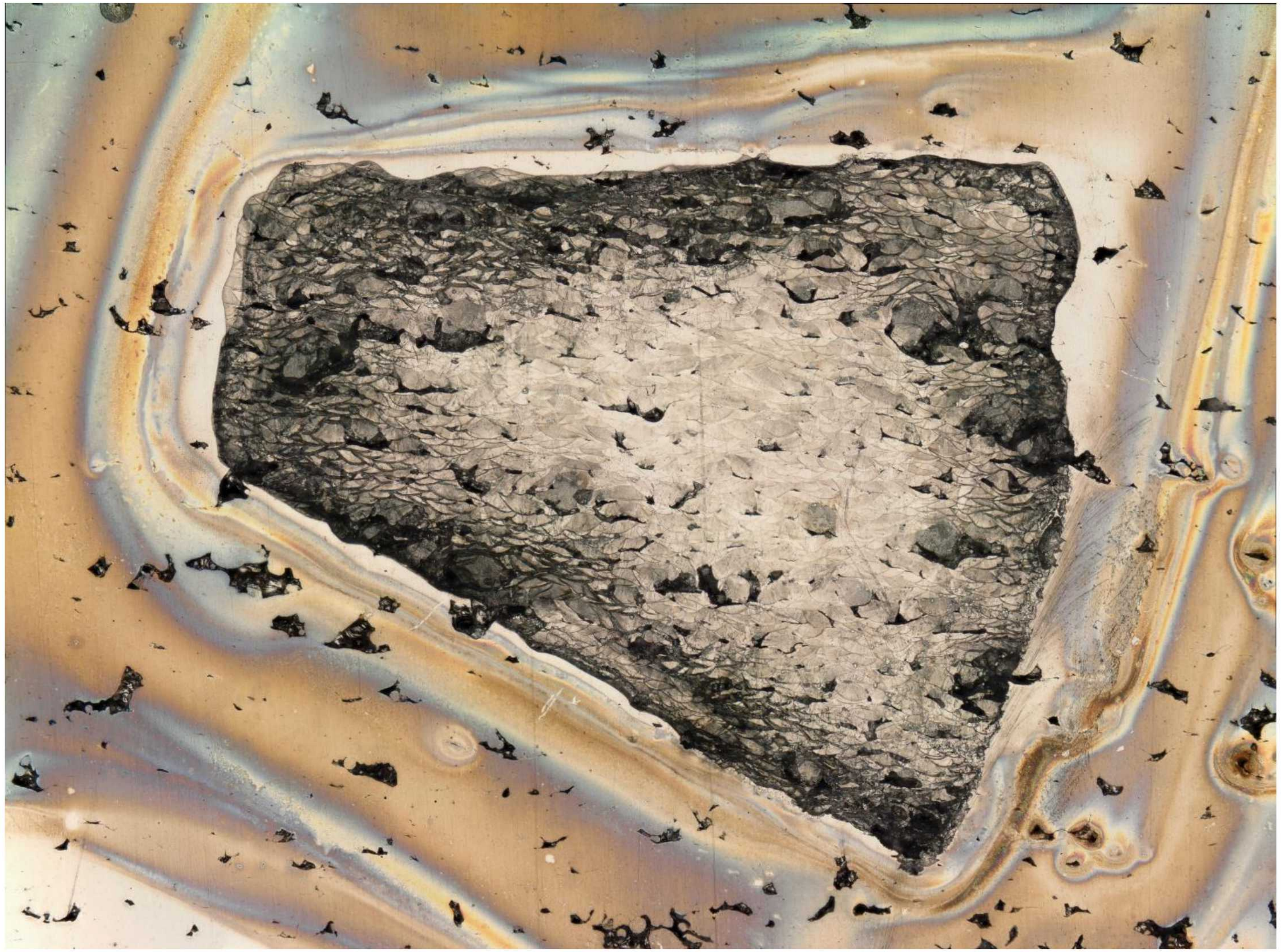
2. Preferential corrosion attack at **melt pool boundaries (MPBs)**.
    - Solute depletion at MPBs leads to preferential corrosion along them in concentrated oxidizing environments.

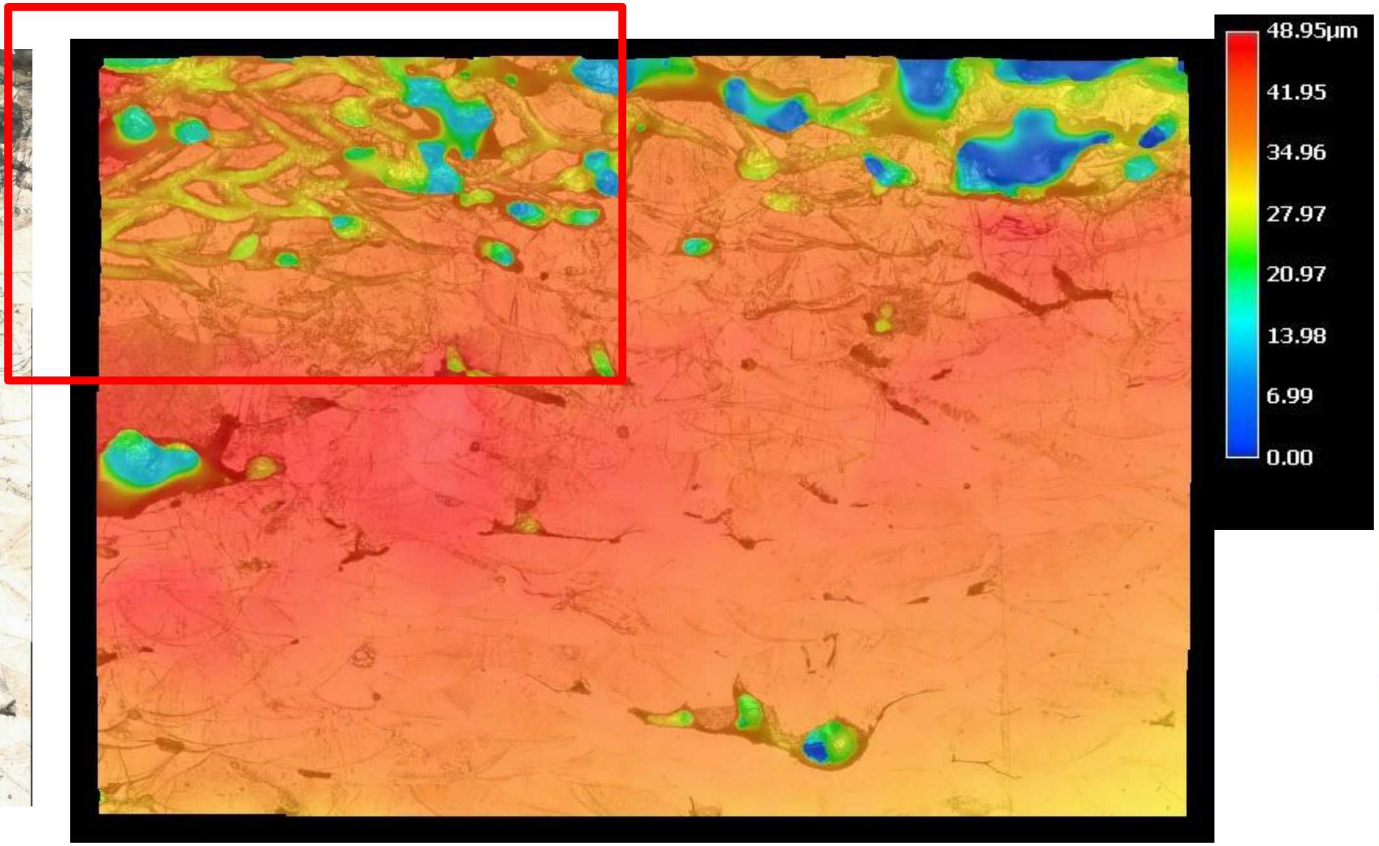
Why we initially looked into melt pool boundary attack: **crevice corrosion of PBF 316L**



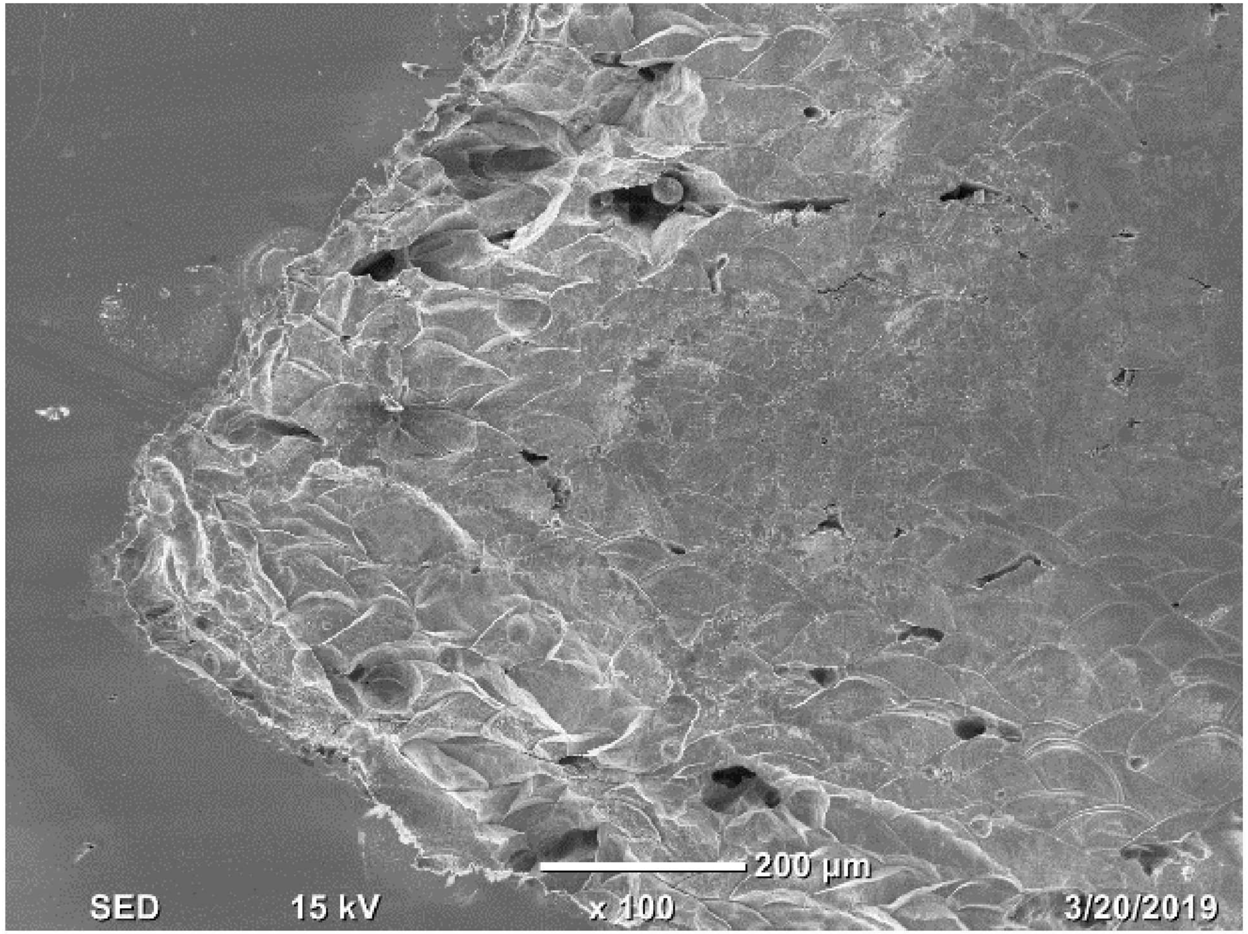
Observed local corrosion at crevice sites, this is from the high porosity sample, but melt pool boundary (MPB) attack was seen for both dense and porous samples under an alumina crevice former.

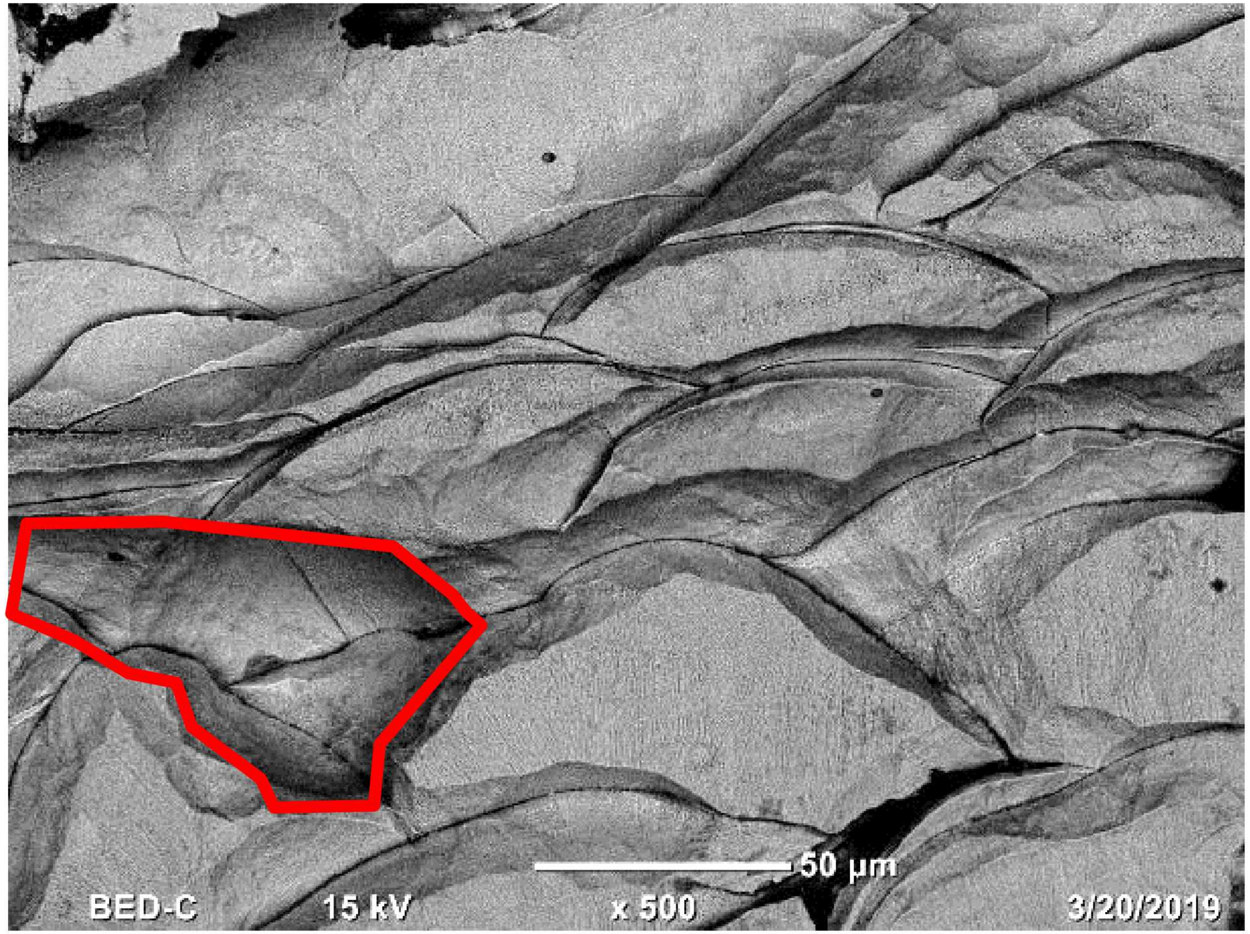
Crevice corrosion of PBF 316L

Local corrosion at melt pool boundaries of AM material, primarily located in deep crevice region (not center of tooth).



Crevice corrosion of PBF 316L

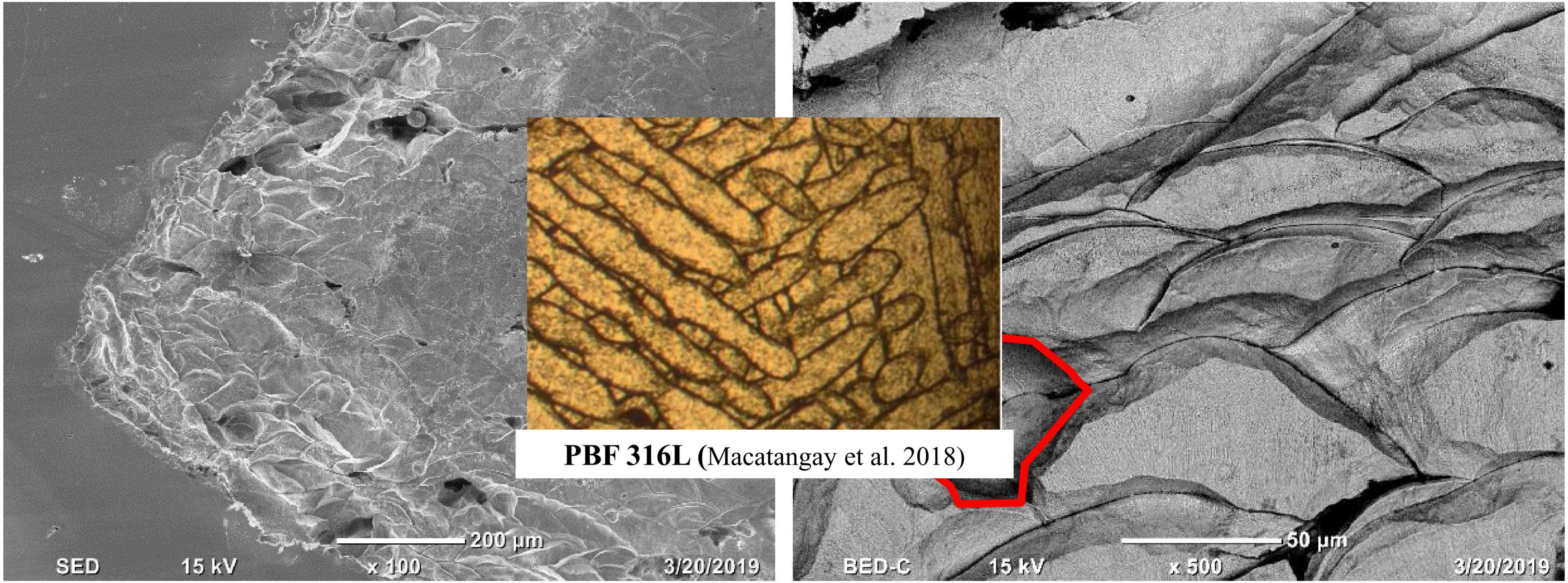




Is there melt pool ("grain") fall out for SLM material?

Local corrosion at melt pool boundaries of AM material, primarily located in deep crevice region (not center of tooth). Has been seen in other studies.

Crevice corrosion of PBF 316L



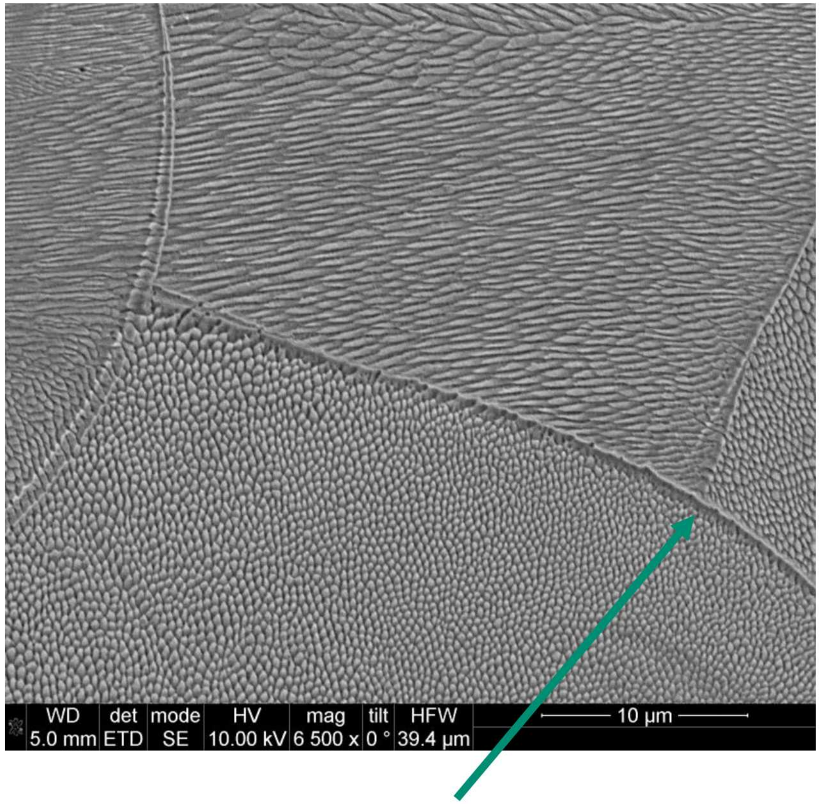
PBF 316L (Macatangay et al. 2018)

Is there melt pool ("grain") fall out for SLM material?

Local corrosion at melt pool boundaries of AM material, primarily located in deep crevice region (not center of tooth). Has been seen in other studies.

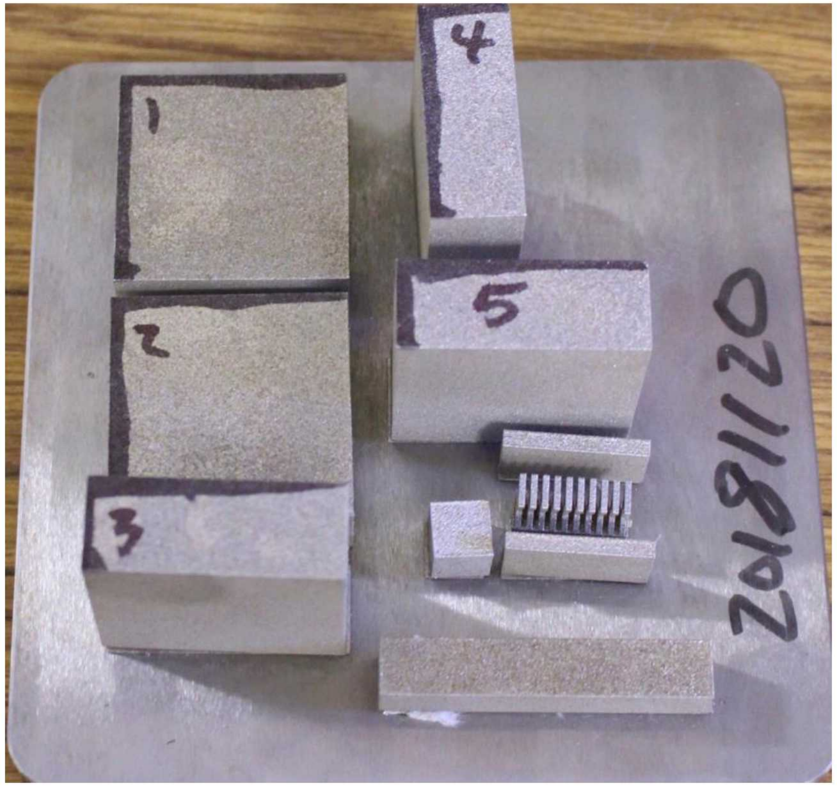
Why are these MPBs showing preferential attack in aggressive corrosion environments?

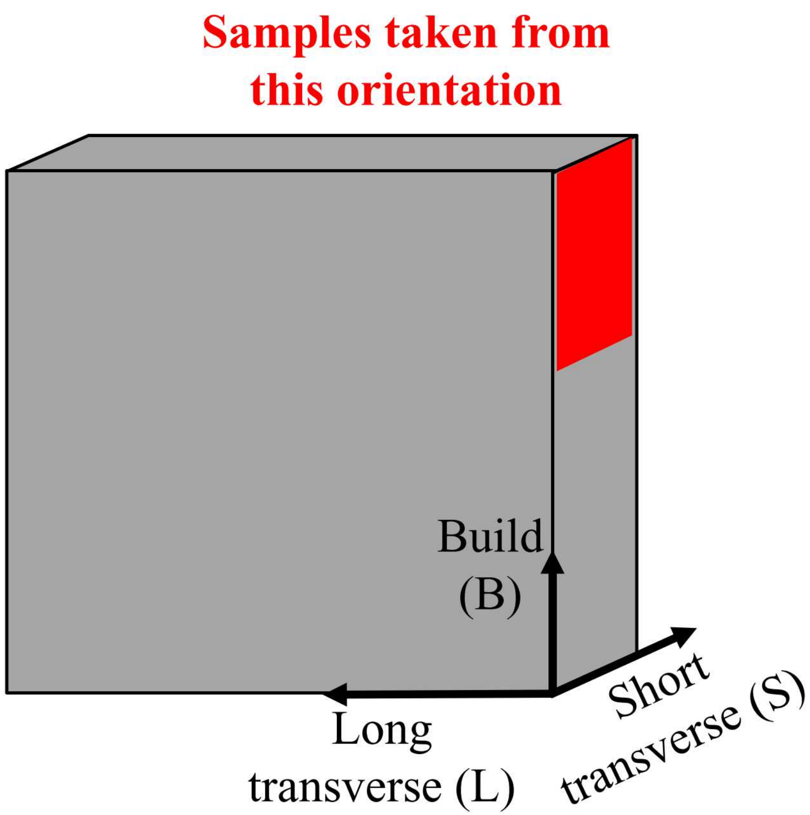
1. Composition? Solute depletion.
2. Local strains?
3. Are the boundaries decorated with oxides causing preferred initiation?
4. Preferred grain orientation attack?
   - Impacts from epitaxial growth?



- MPB are raised (etch at slower rate) after electro-etching in nitric acid. This may suggest it is enriched in Fe and/or depleted in Cr and Mo, based on Cr/Mo oxides being unstable at high potentials.
- Where Cr/Mo is enriched (dislocation cell boundaries) there is preferential etching.

PBF 316L samples





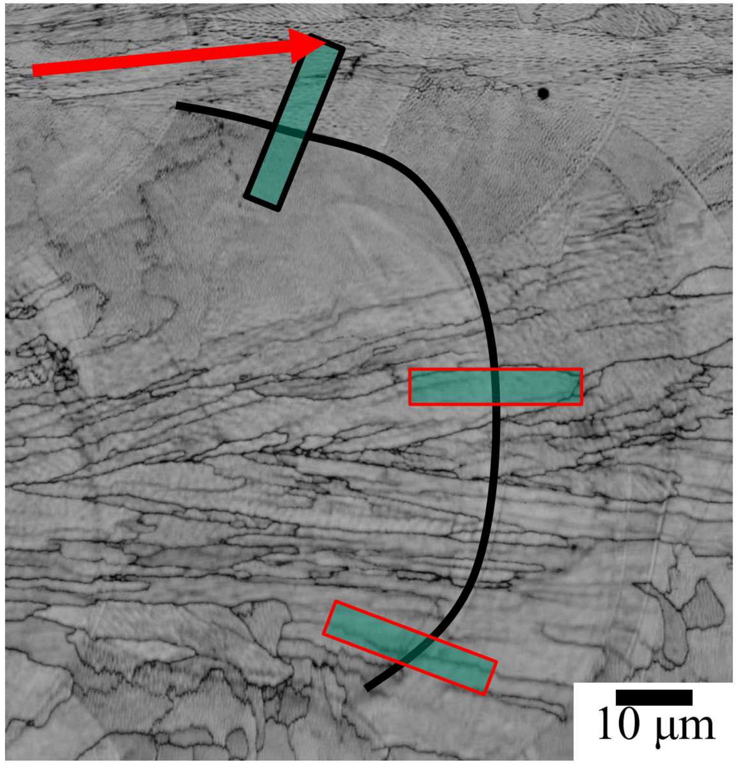
wt.%	C	Cr	Cu	Fe	Mn	Mo	N	Nb	Ni	O	P	S	Si
AM316L - Dense	0.016	17.0	0.14	67.8	1.06	2.1	0.093	0.008	10.86	0.065	0.017	0.009	0.64

	YS (MPa)	UTS (MPa)	Elongation to failure (%)	Density (g/mL)	Hardness (B)	Charpy Toughness (ft-lb)
AM316L - Dense	430.1	575.0	61.3	7.94	92.3	79

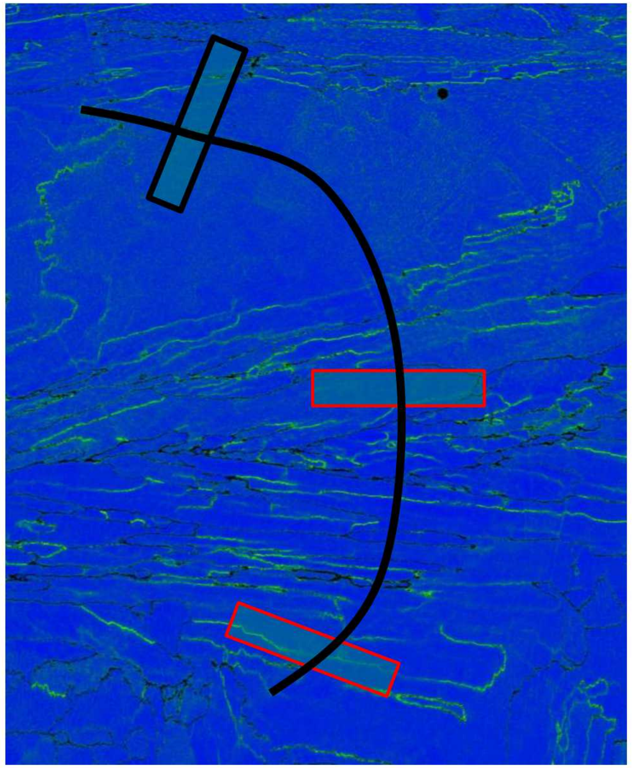
EBSD prior to FIB of samples.

This side is melted into already built AM part.

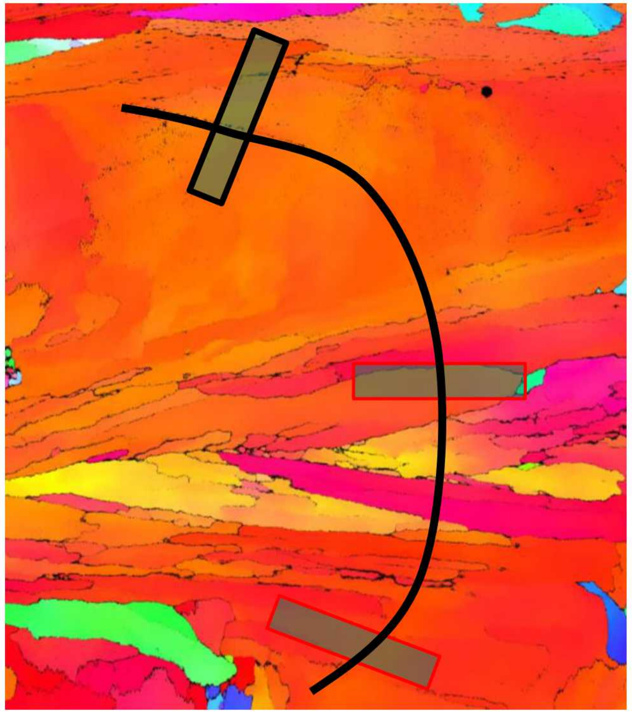
Top



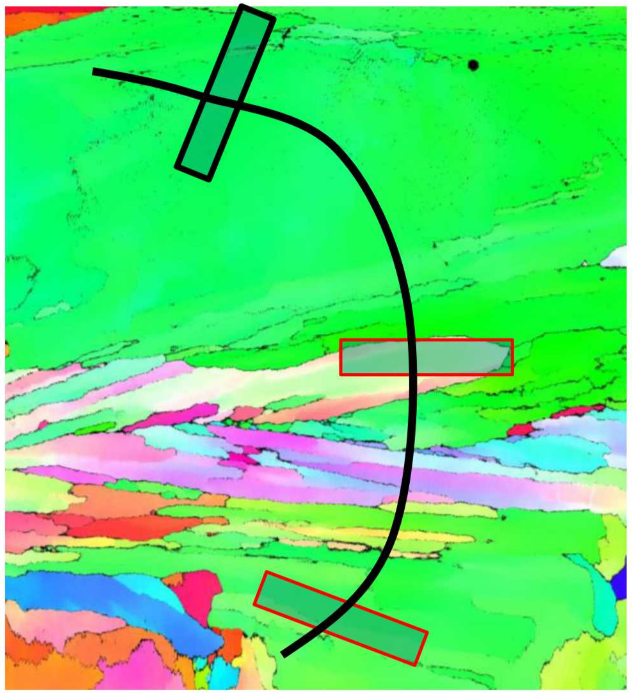
Band Contrast



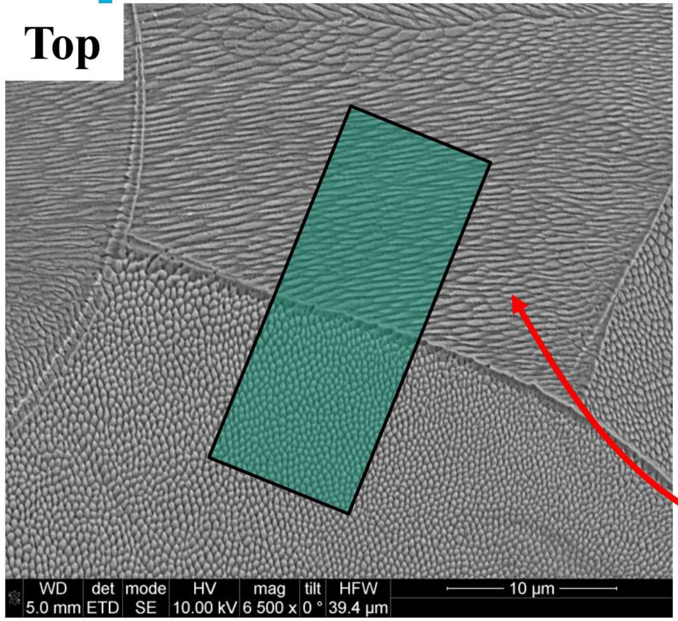
Kernal average misorientation

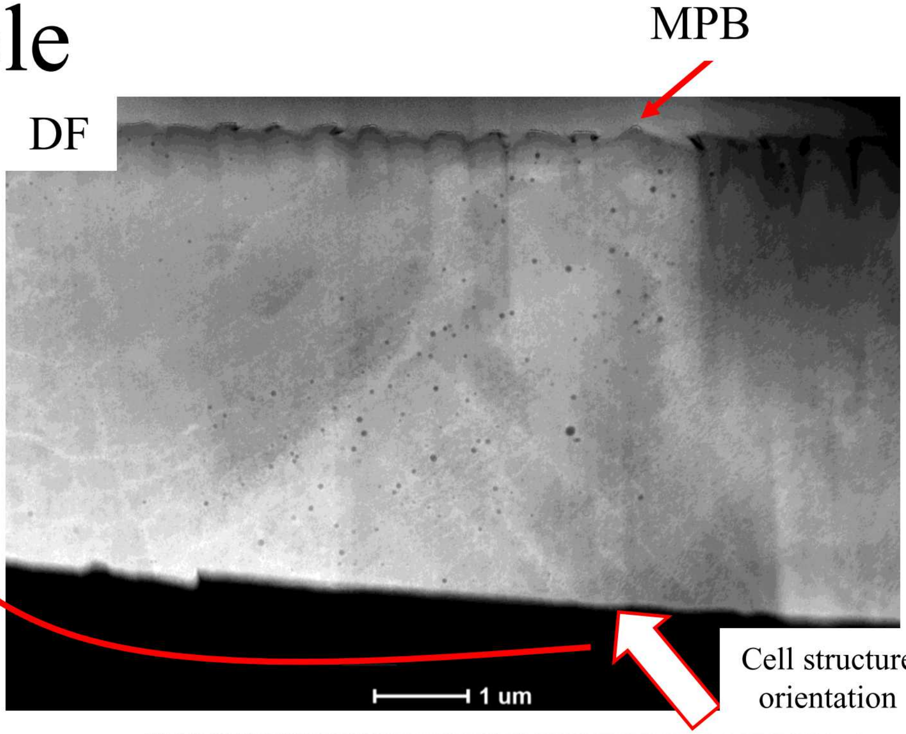


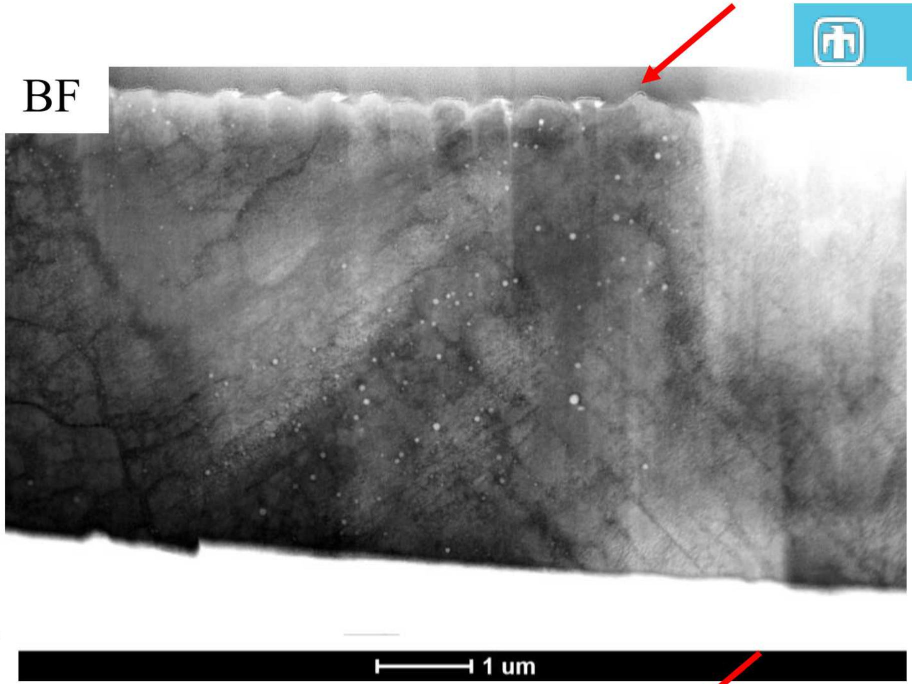
Build direction

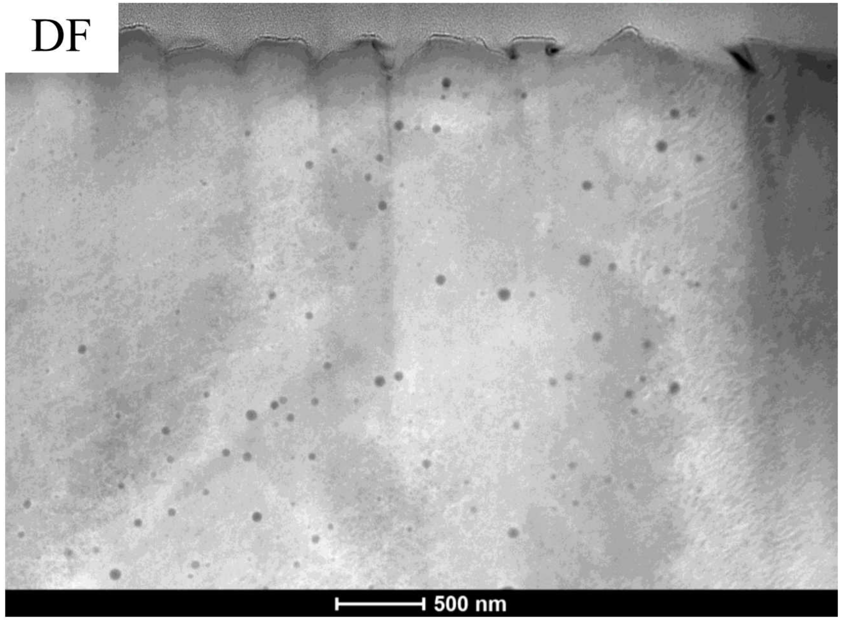


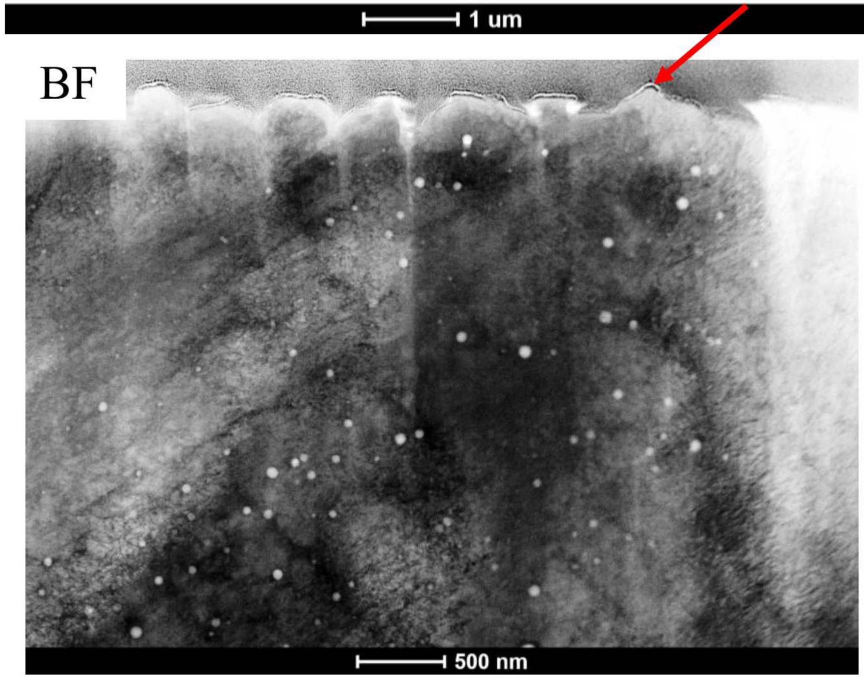
Top FIB sample





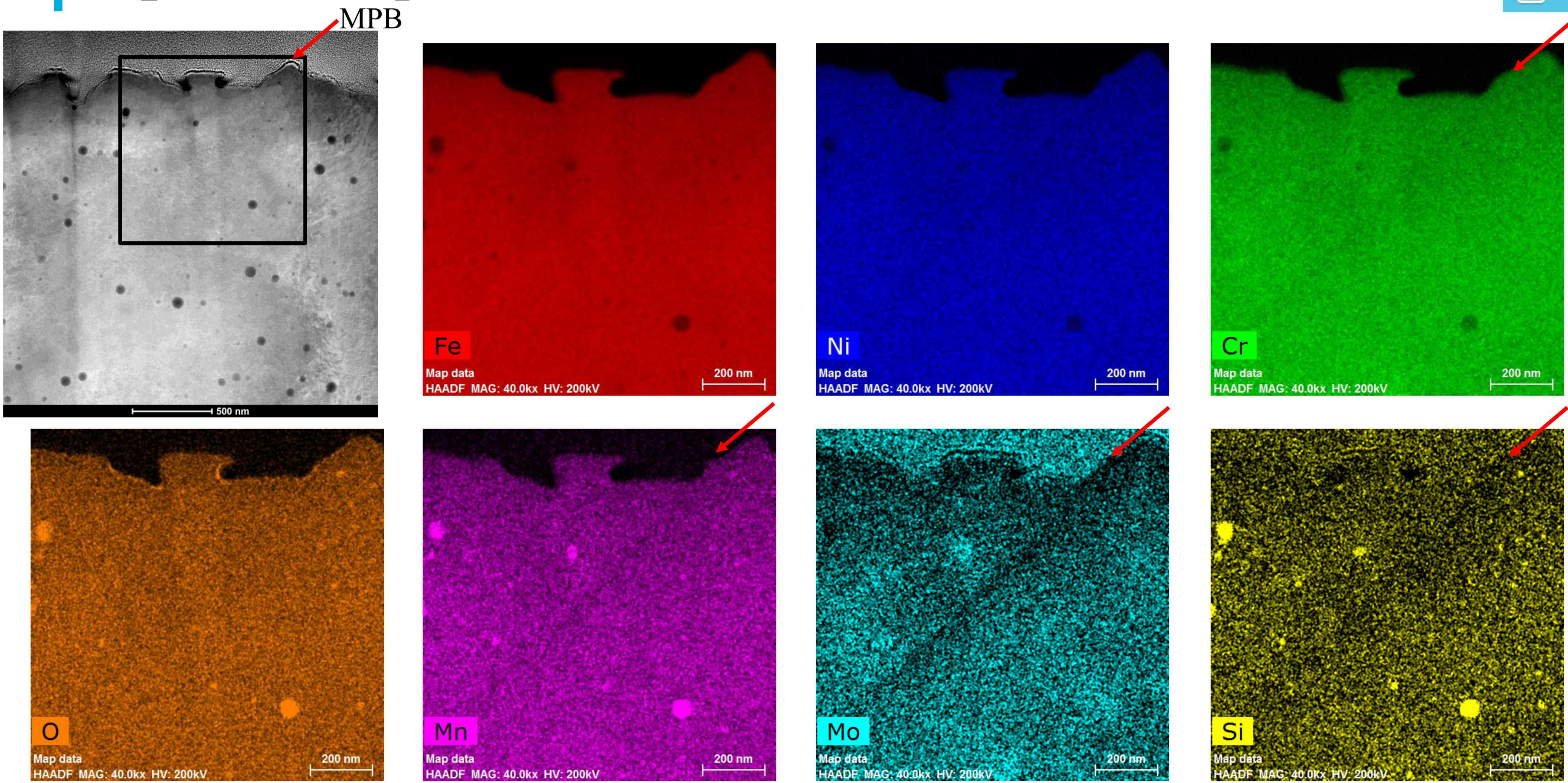






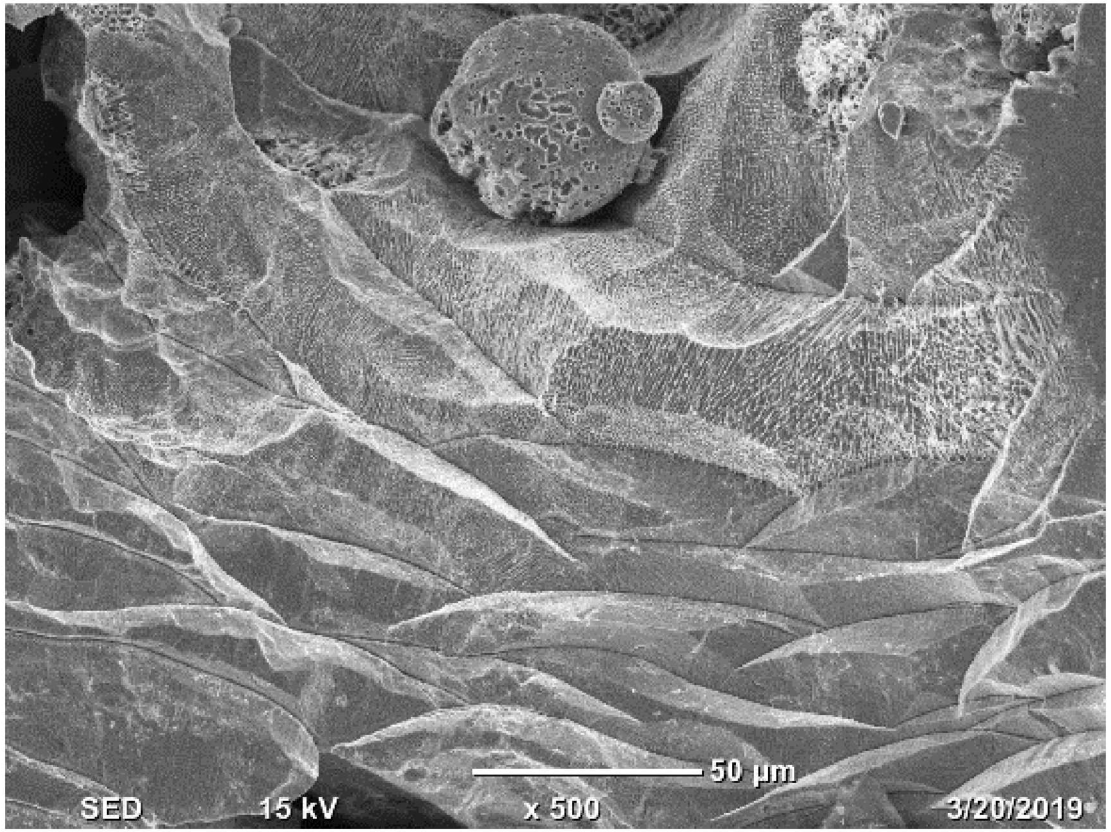
Red arrow indicates suspected MPB.

Top FIB sample



MPB conclusions

- Solute (Cr/Mo) depletion is likely the reason for MPB corrosion susceptibility.
- Minimal grain orientation change was shown across MPBs, epitaxy across these boundaries was very common.
- Few nanoscale oxide inclusions were found on the MPBs.
- No localized strain associated with MPBs.



PBF crevice corrosion

Acknowledgements

- SEM and EBSD by Sara Dickens and Joe Michael.
- FIB by Chris Barr.
- STEM-EDS by Paul Kotula.

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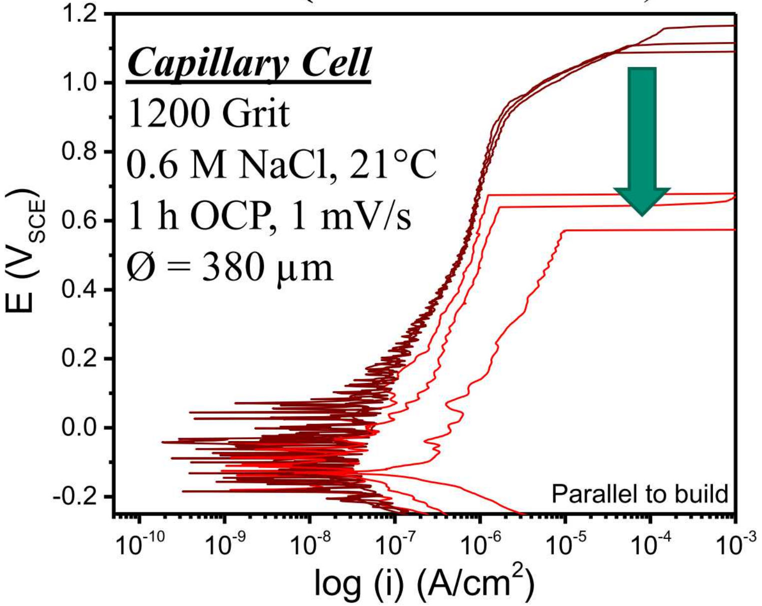
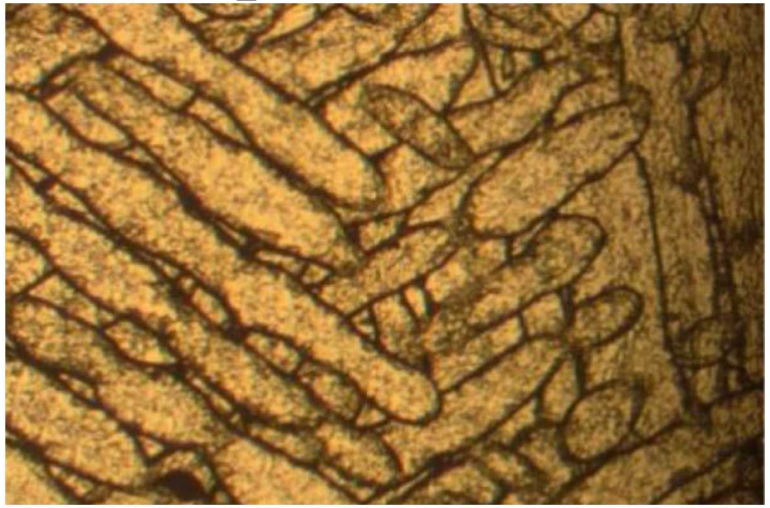
Extra slides

What features lower corrosion resistance of AM stainless steel?

SLM 304L (Schaller et al. 2018)

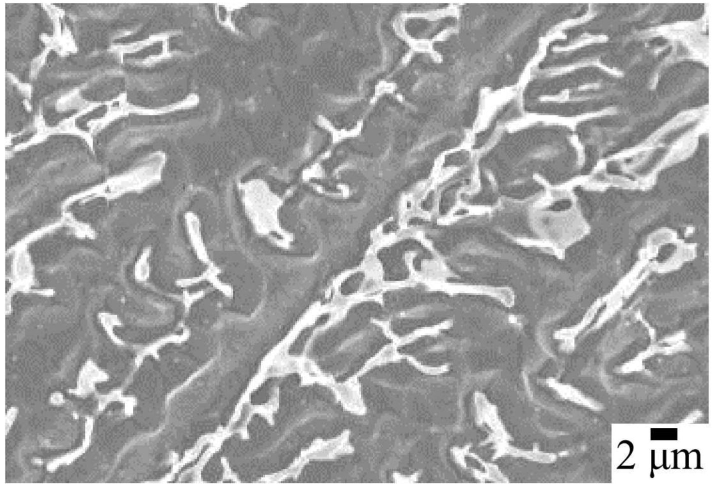


Melt pool interfaces

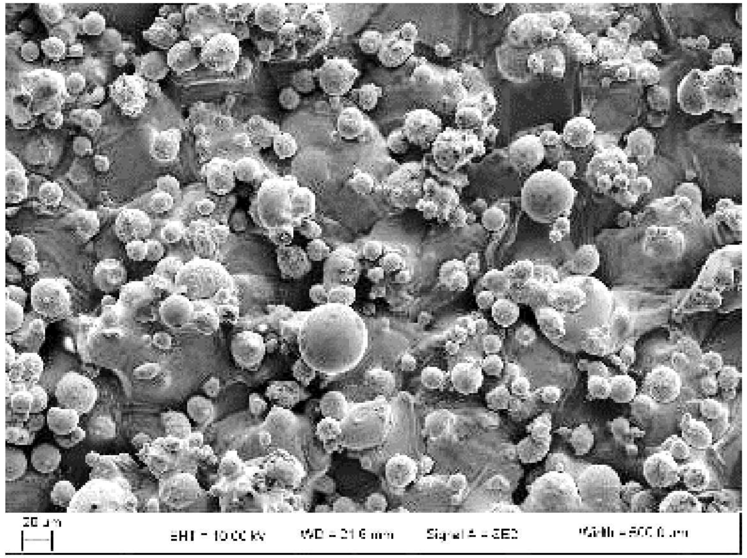


SLM 316L (Macatangay et al. 2018)

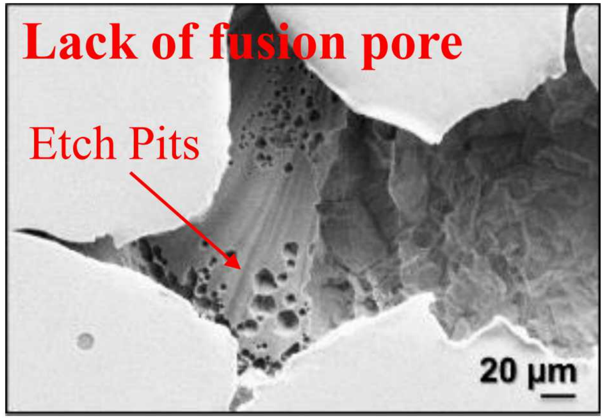
Secondary phase formation



As-built surface roughness



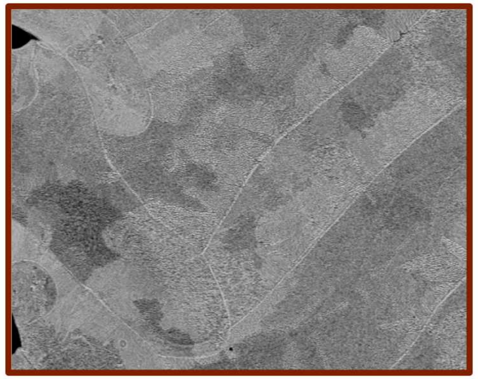
Lack of fusion

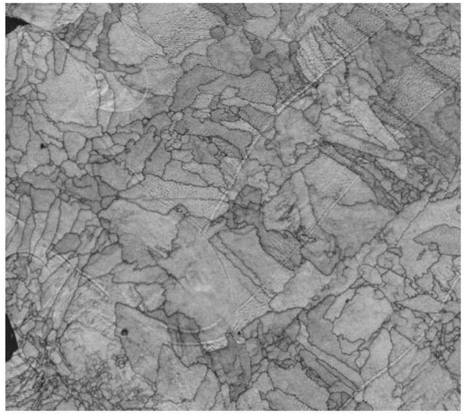


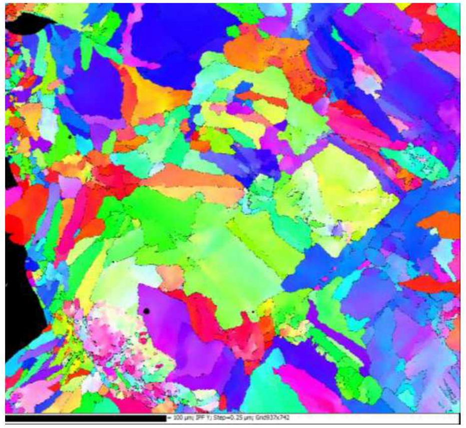
What else:

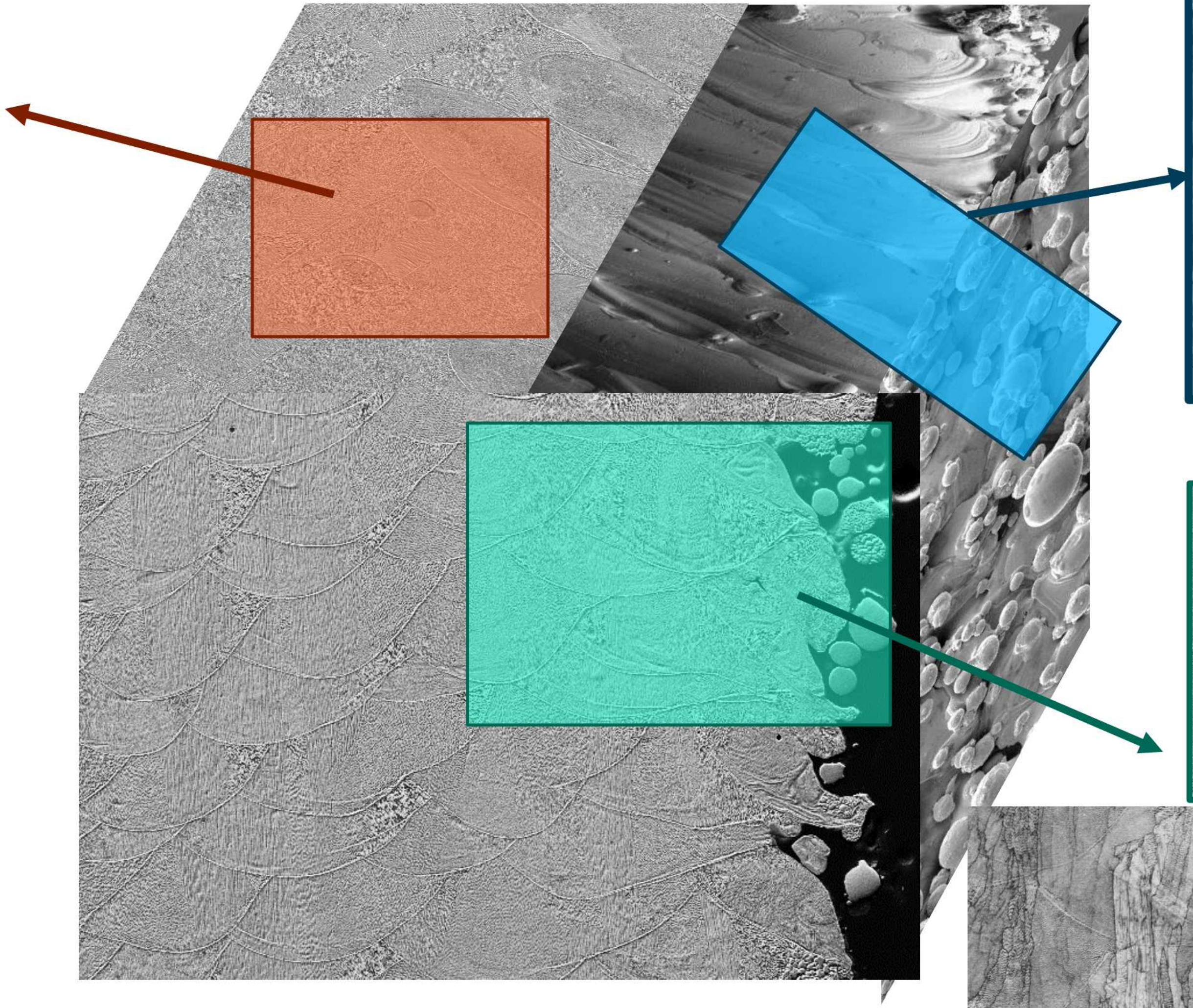
- **Residual stress?**
- **Micro cracks?**
- **Non-metallic inclusions?**

General microstructure of PBF 316L

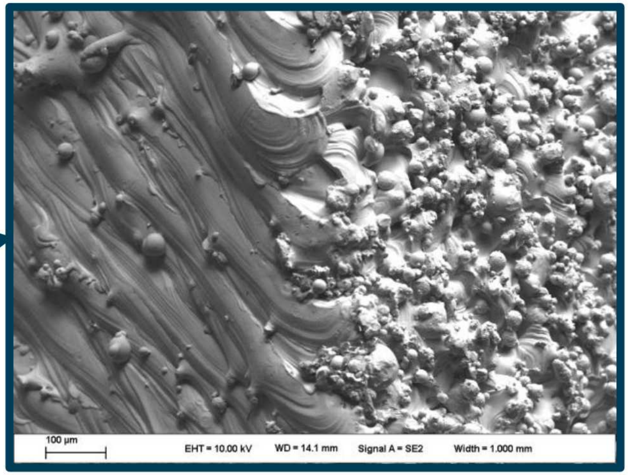


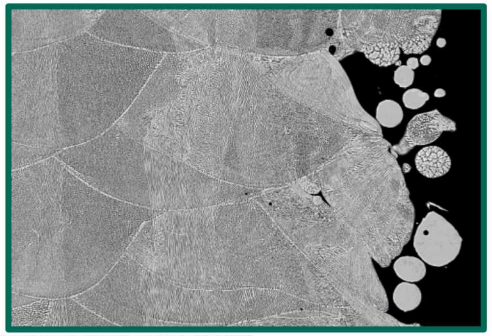




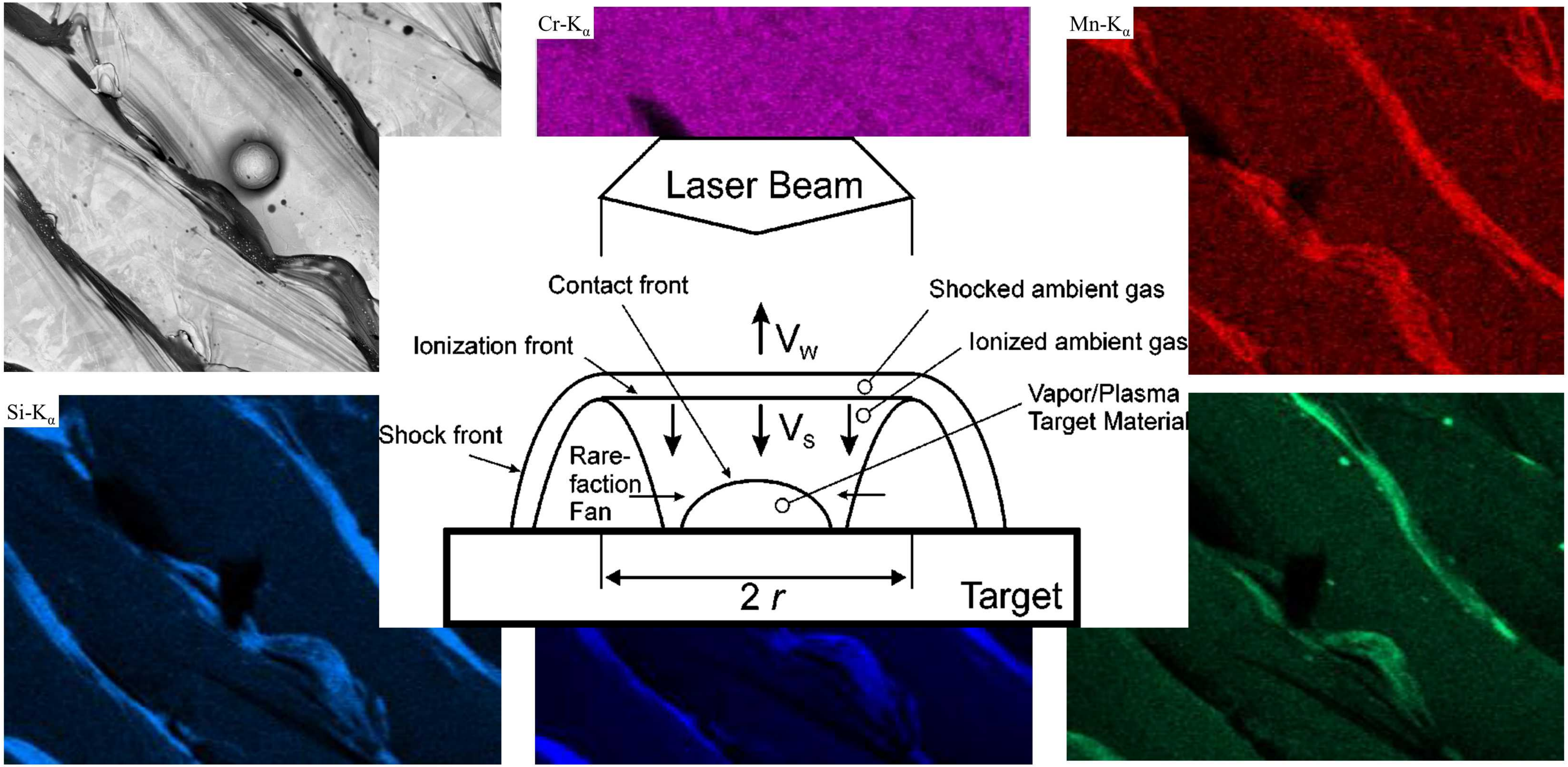


Along the edge.





EDS maps of **AP Top** surface showing oxide in between layers.



M. Han, K.P. Lieb, E. Carpene, P. Schaaf, Laser–plume dynamics during excimer laser nitriding of iron, Journal of Applied Physics, 93 (2003) 5742-5749.10.1063/1.1563814

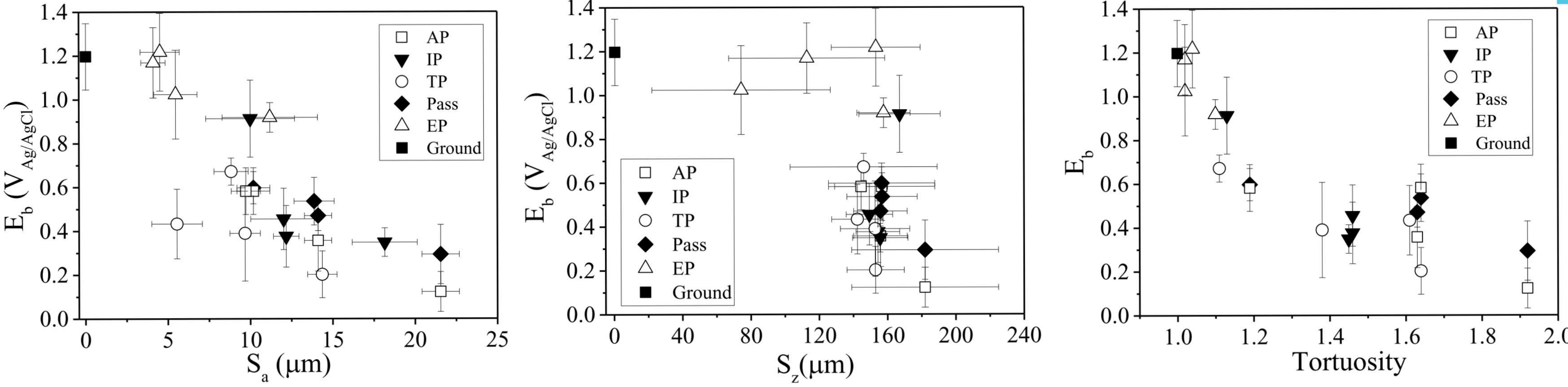
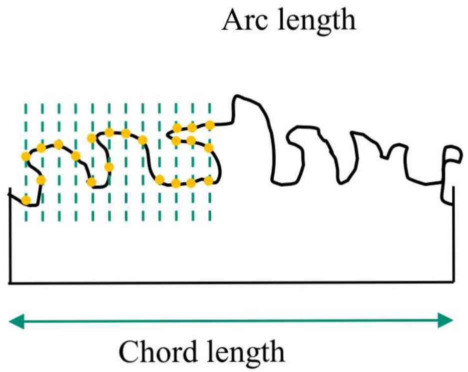
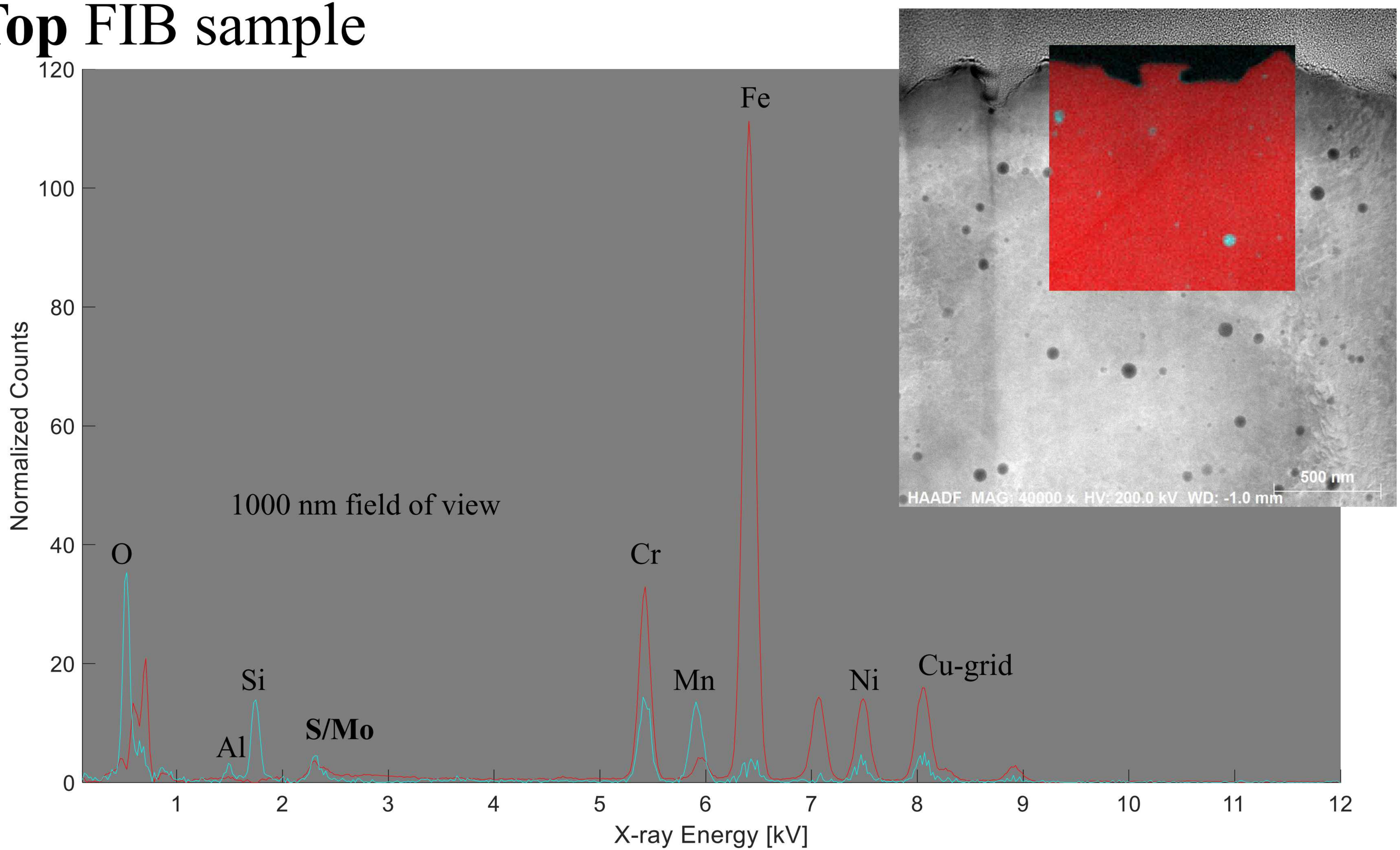


Figure 6: Scatter plots of E_b with respect to roughness measurements, (a) S_a and (b) S_z. Error bars represent one standard deviation for all measurements.

This is still only useful for the case of these surfaces. If the surface has curvature but does not overlap itself (many other surfaces) then this wouldn't be picked up. I guess by arc/chord a little but not entirely.

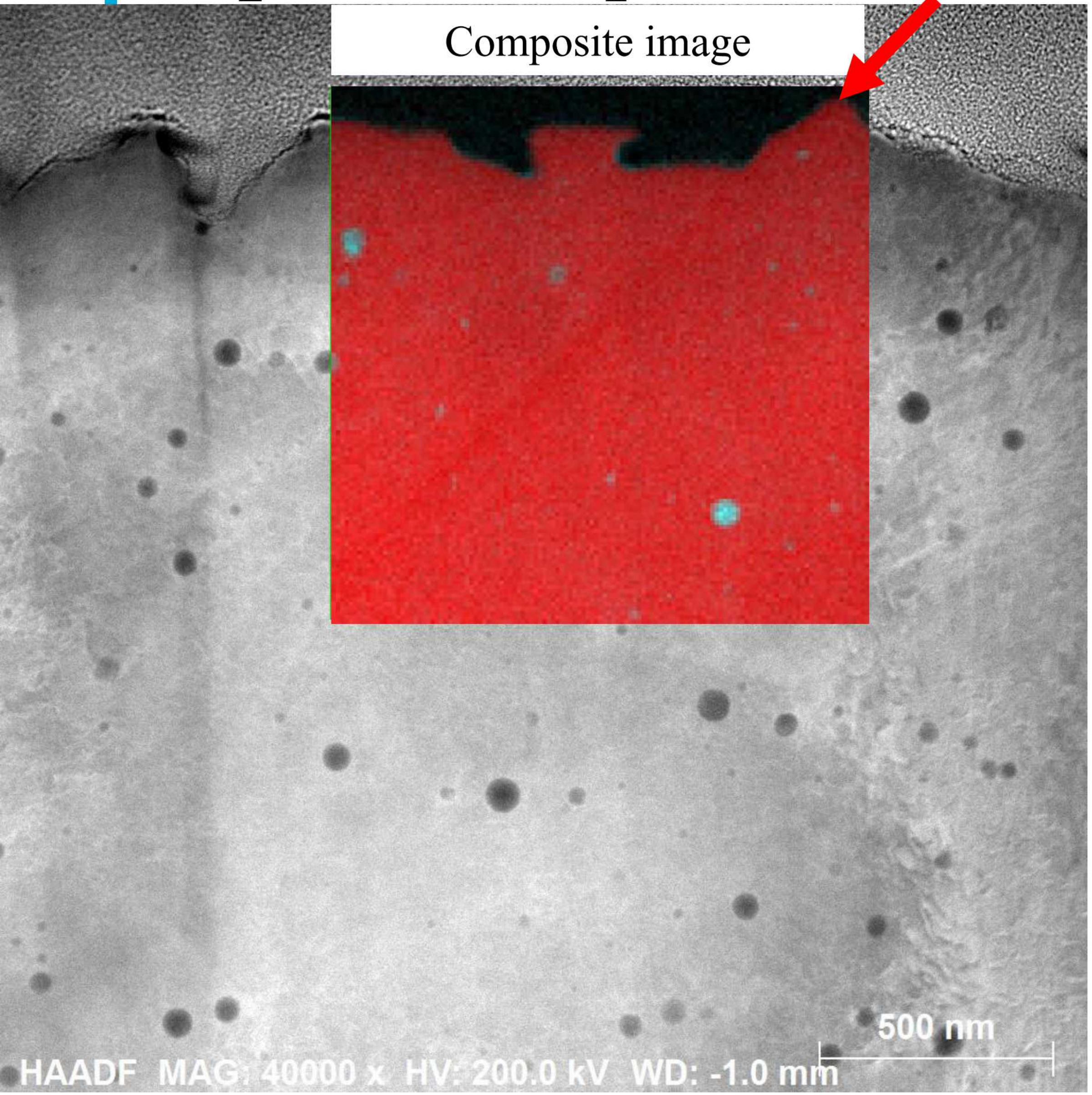


Top FIB sample

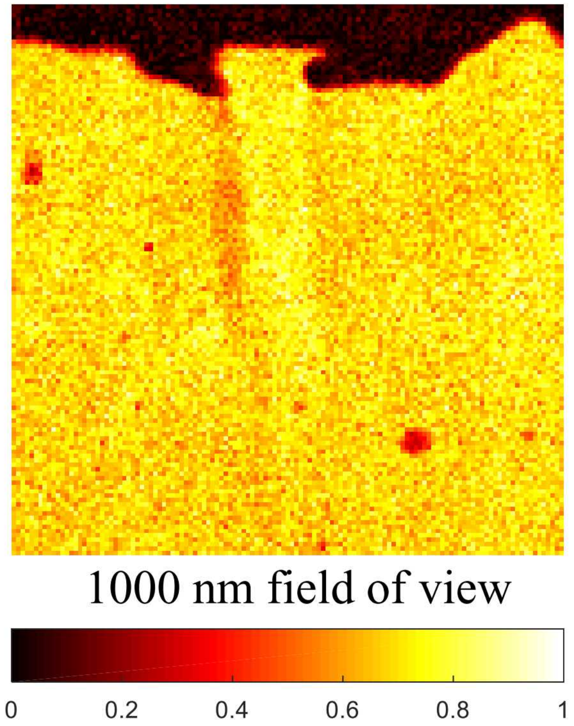


Top FIB sample

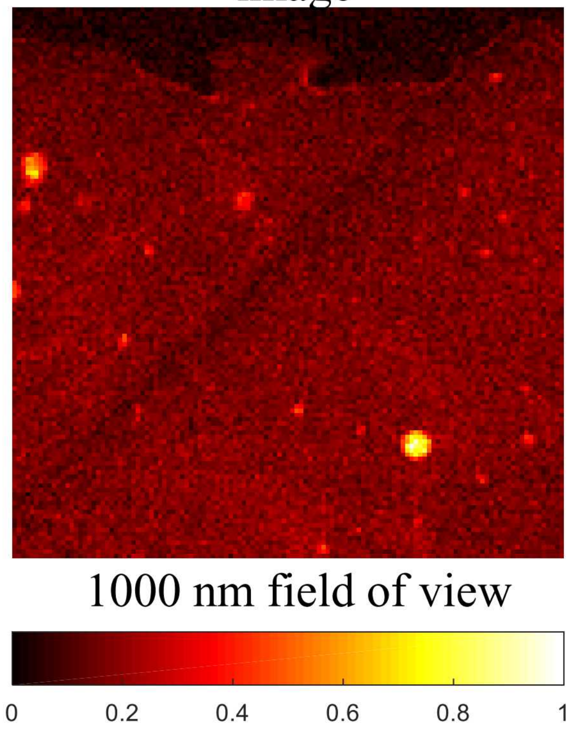
MPB



Matrix component image

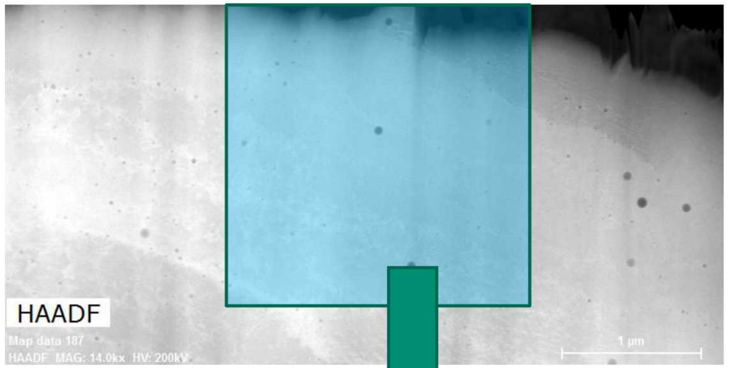


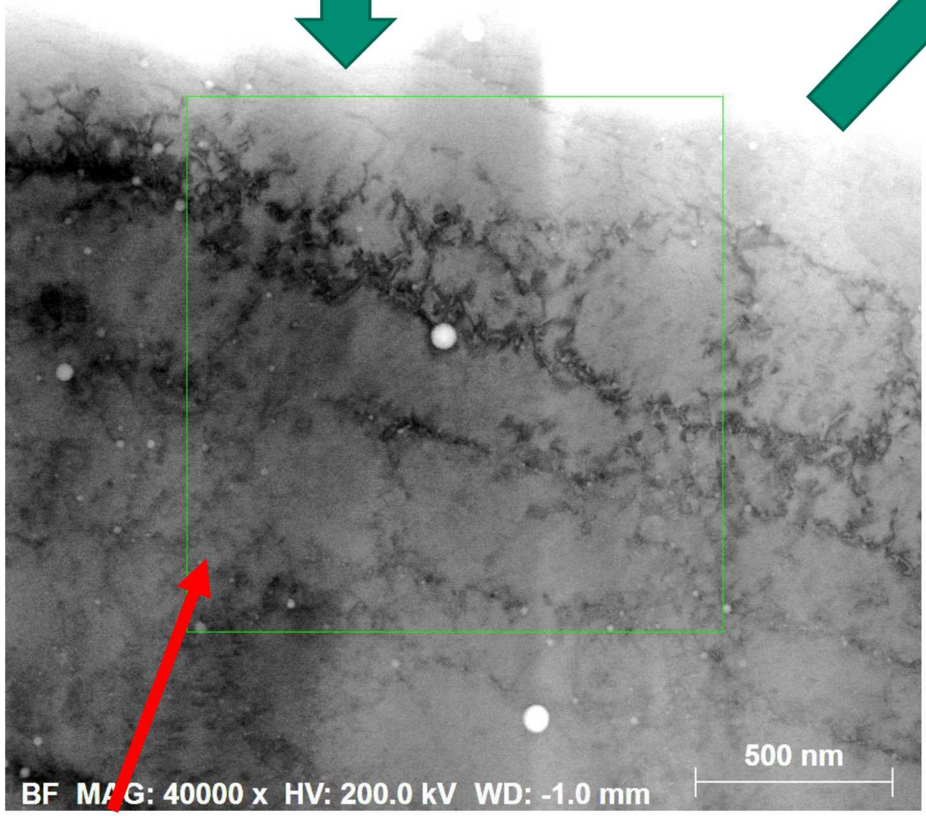
Cr-Mn-S-Mo-Si-Al-O component image

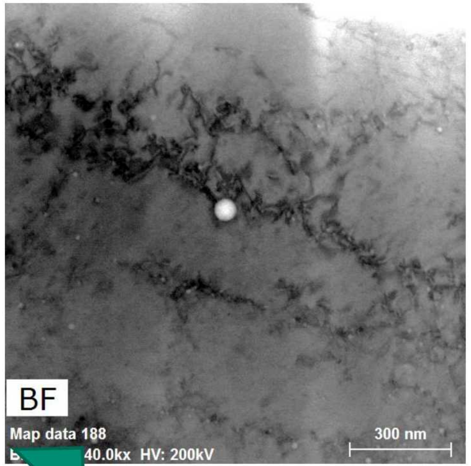


The composite images suggest depletion of solute along this MPB.

STEM-EDS maps of another MPB







We think this is where the MPB is for this sample.

There is depletion of Cr, Mo, Mn, Si here and enrichment of Fe.

Also shown for another MPB back in May.