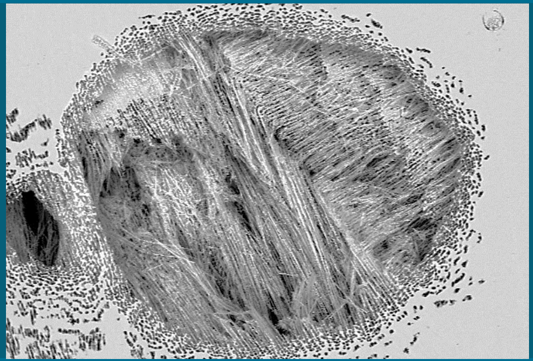
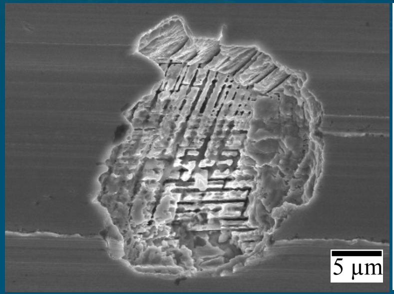
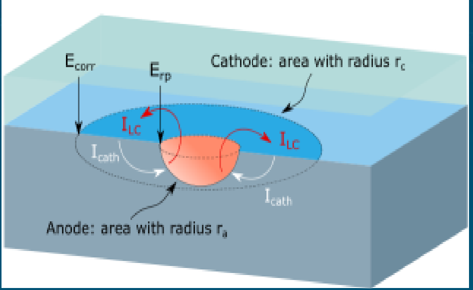
SAND2022-2095C

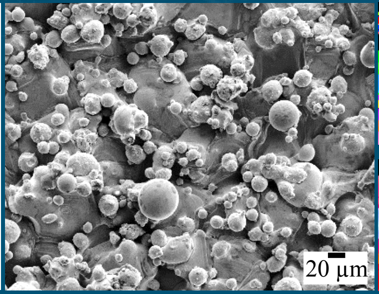
Sandia National Laboratories

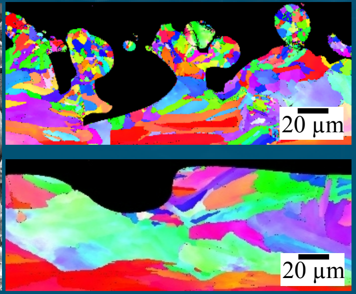
Atmospheric Corrosion Of Laser Powder Bed Fusion 316L Stainless Steel











AMPP 2022 Annual Conference + Expo
March 6th to 10th, 2022 - San Antonio, TX

AMPP™ ANNUAL CONFERENCE + EXPO

PRESENTED BY

Michael A. Melia

Senior Member of Technical Staff - R&D S&E Material Science

Key contributors: Erin Karasz (Postdoc), Kasandra Escarcega-Herrera (Undergrad), Jay Taylor, Rebecca Schaller, Michael J. Heiden, Jeffrey M. Rodelas

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Acknowledgements

Funding: The ACT program, DOD-DOE Joint Munitions Program (JMP), Aging and Lifetime Program.

Many people to thank…

SNL: Kasandra Escarcega-Herrera, Erin Karasz, Jesse Duran, Jason M. Taylor, Rebecca F. Schaller, Jeffrey M. Rodelas, Michael J. Heiden, Bradley H. Jared, Eric J. Schindelholz, Joseph Michael, Paul Kotula, Philip Noell, Daniel Perry, David Saiz, Mark Wilson, Brendan Nation, Joshua Koepke, Christina Profazi, Sara Dickens, Luis Jauregui, Alex Hickman

UVA: Rebecca S. Marshall, Danyil Kovalov, Ryan Katona, Ji Ma, Robert Kelly

LANL: Courtney Clark, Tim Gorey, Jamie Stull, Colt Montgomery, Daniel Hooks

KCNSC: Lucas Rice, Jonathan Dwyer, Andy Deal, Zach Rueger, Francisco Garcia-Moreno

SRNL: Patrick Kuzbary, Savidra Lucatero, Prabhu Ganesan, Hector Colon-Mercado, Paul Korinko

LLNL: Margaret Wu, Seongkoo Cho, Monika M. Biener, Y. Morris Wang, Justin Jones, and S. Roger Qiu, …

Thanks to the symposium organizers for this opportunity.

Publications on topic:

E.J. Schindelholz, et al., Corrosion of Additively Manufactured Stainless Steels—Process, Structure, Performance: A Review, Corrosion, 77 (2021).

M.A. Melia, et al., Marine Atmospheric Corrosion of Additively Manufactured Stainless Steels, Corrosion, (2021).

M.A. Melia, et al., How build angle and post-processing impact roughness and corrosion of additively manufactured 316L stainless steel, npj Materials Degradation, 4 (2020).

M.A. Melia, et al., Corrosion properties of 304L stainless steel made by directed energy deposition additive manufacturing, Corros. Sci., (2019).

R.F. Schaller, et al., The Role of Microstructure and Surface Finish on the Corrosion of Selective Laser Melted 304L, J. Electrochem. Soc., 165 (2018).

R.F. Schaller, et al., Corrosion Properties of Powder Bed Fusion Additively Manufactured 17-4 PH Stainless Steel, CORROSION, 73 (2017).

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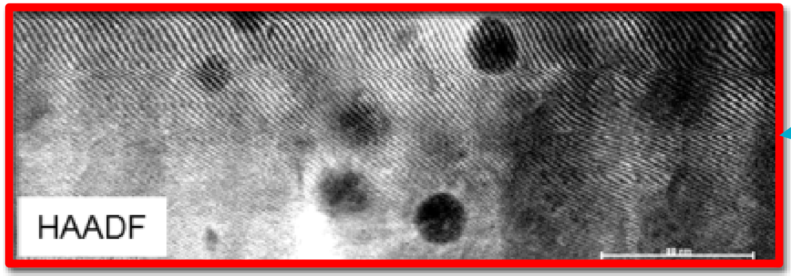
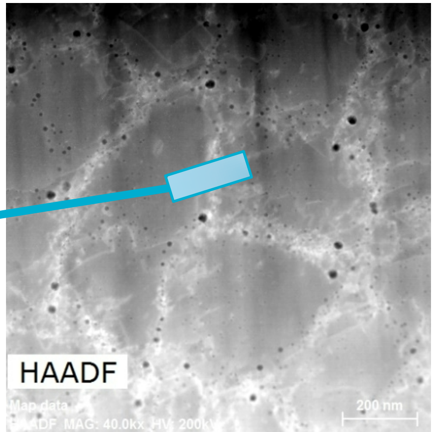
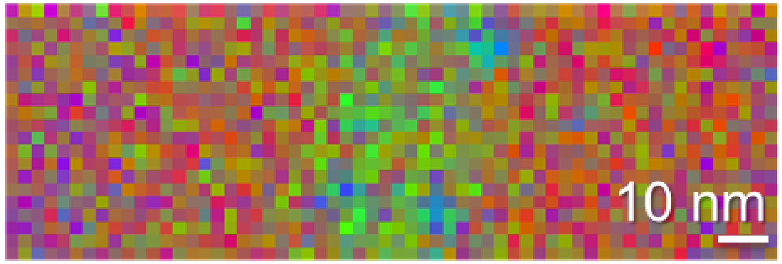
Background - Powder metal additively manufactured (AM) microstructures

Chemical heterogeneities

Cell walls decorated with oxides



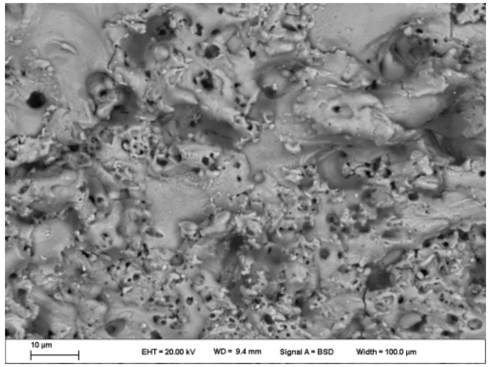




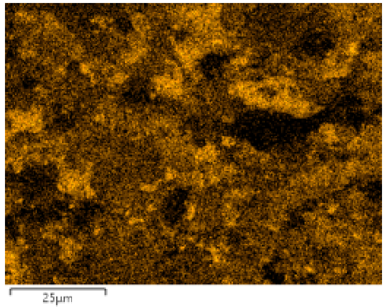
- Increased Fe (+ 9%)
- Increased Si
- Increased Cr (+ 4%)

Adapted from Schaller, 2018

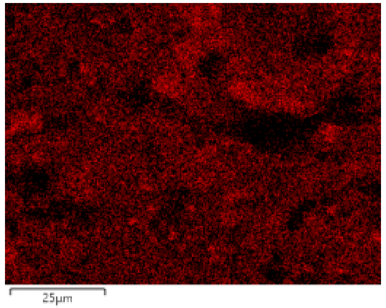
Recast layer from a part removed with wire EDM



Cu map



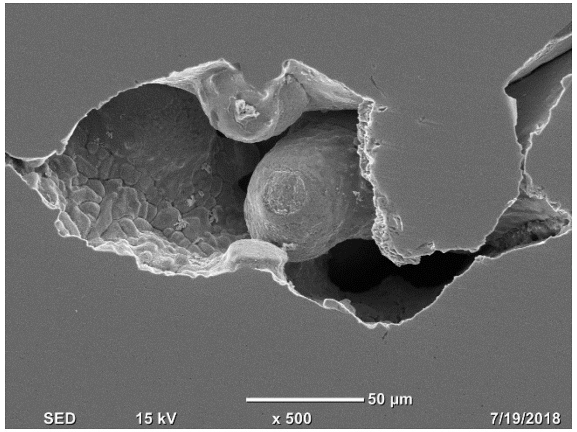
Zn map



Cu and Zn penetrate 5 to 10 μm deep.

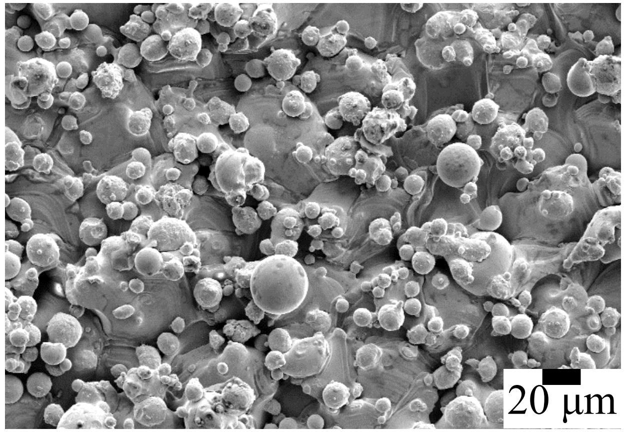
Processing defects

Porosity



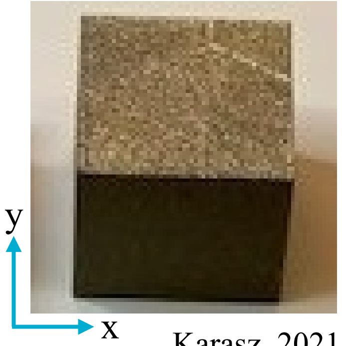
Melia, 2019

Surface roughness



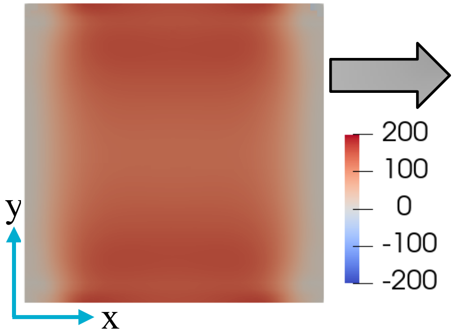
Melia, 2020

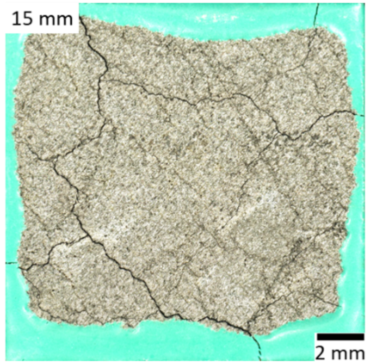
Residual stress of 316L cubes



Karasz, 2021

Stress XX (MPa)





Leads to susceptibility to stress corrosion cracking

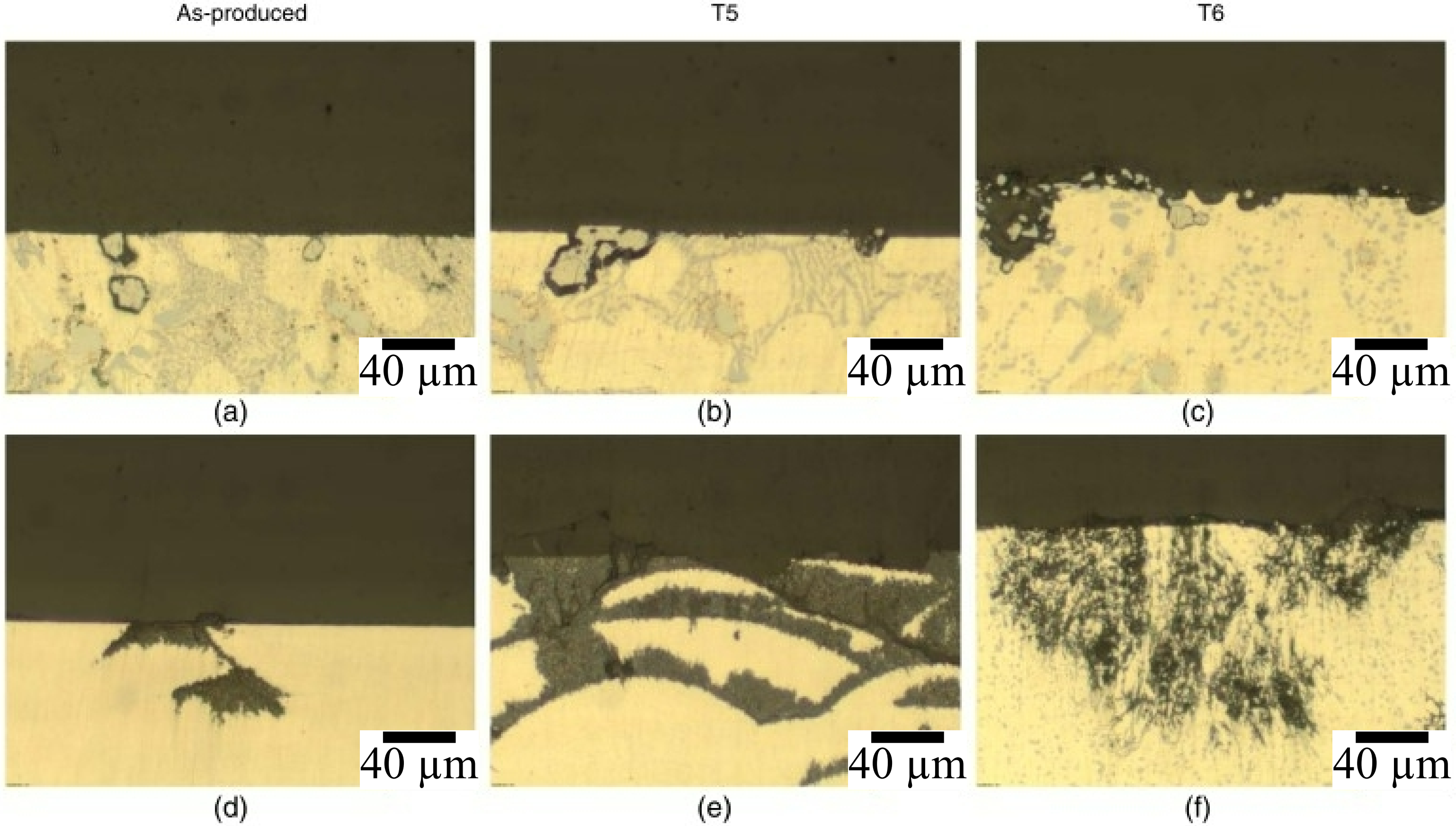
(UUR)

Background – AM Al-10Si-Mg exposed to ASTM G85-A2

Cross-section images after **31 days** of ASTM G85-A2 exposures

Cast Al-10Si-Mg

AM Al-10Si-Mg

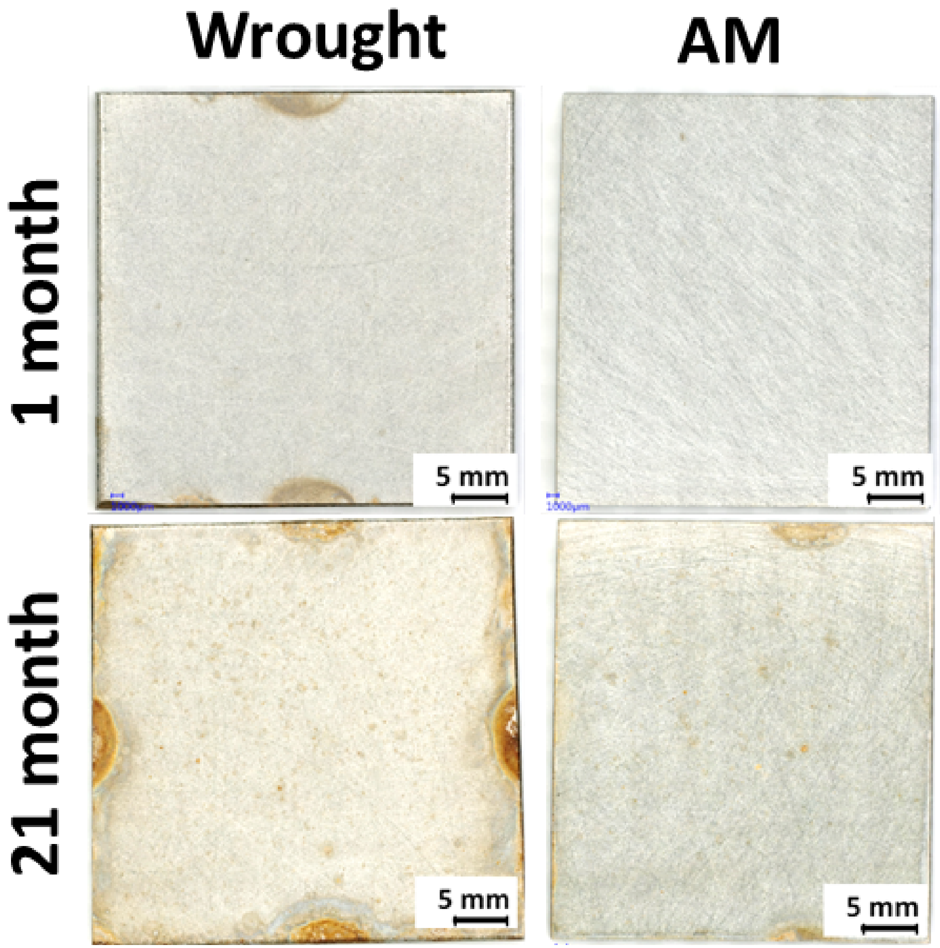


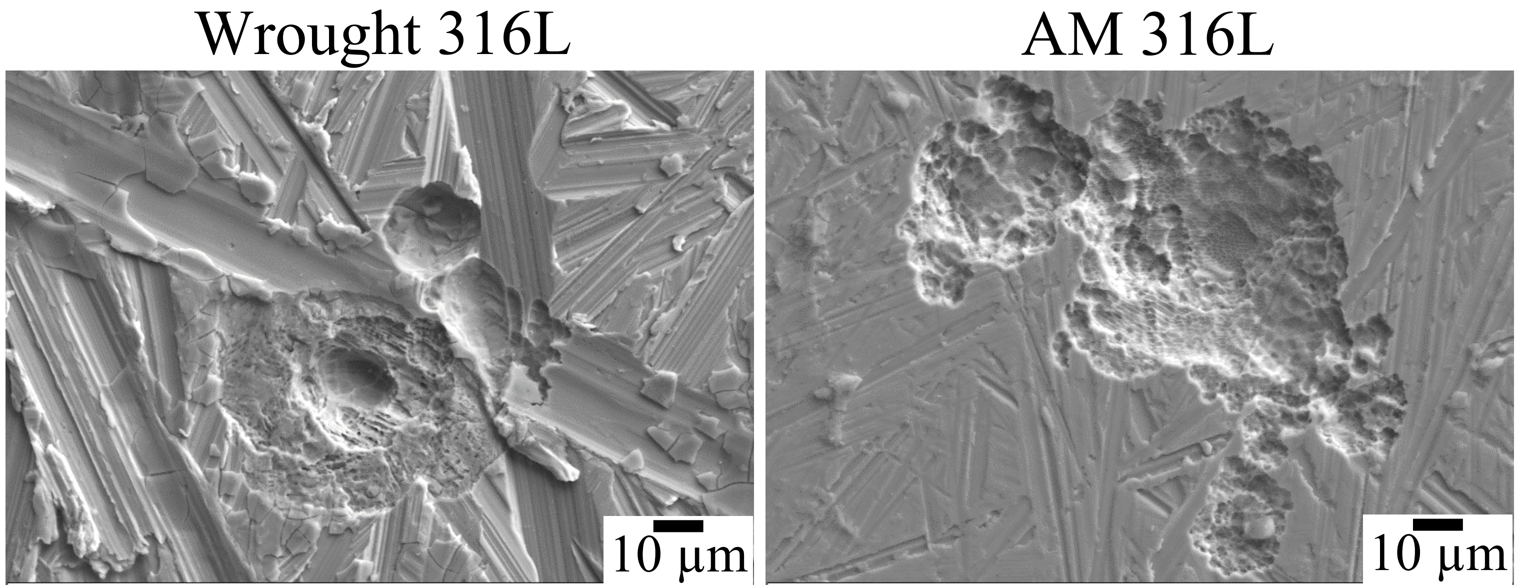
- Corrosion damage morphology was dictated by AM microstructure. They hypothesize that melt pool boundaries act as barriers to corrosion propagation.
- Maximum corrosion depth was greater for AM compared to cast.

Source: Kubacki, CORROSION, 2019.

(UUR)

Background – Coastal exposures of **AM 304L/316L**





Salt loading: 26 ± 18 µg/cm^2/day

- The pit morphology for AM 316L showed significant preferential corrosion attack of the cellular microstructure, the cell boundaries were not as vulnerable.
- Pit depth of AM samples were smaller than wrought samples (often larger pit density).

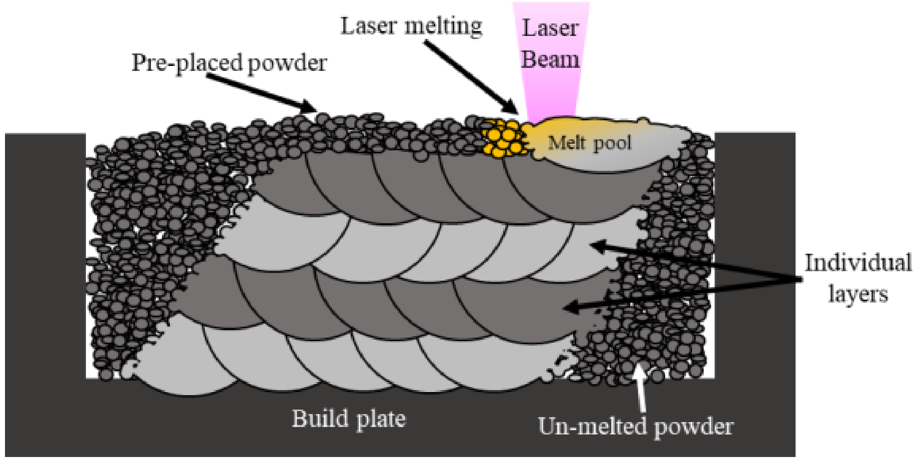
In collaboration with Francisco Presuel-Moreno (FAU)

Source: Melia, CORROSION, 2021.

What will we go over in this talk?

- Constant humidity and contamination exposures of **ground surfaces** for wrought and powder bed fusion (PBF) 316L.
  - Pit morphologies.
  - Pit statistic analysis.
- Explore corrosion behavior of **as-printed** 316L parts when exposed to the accelerated corrosion test, ASTM G85-A2.

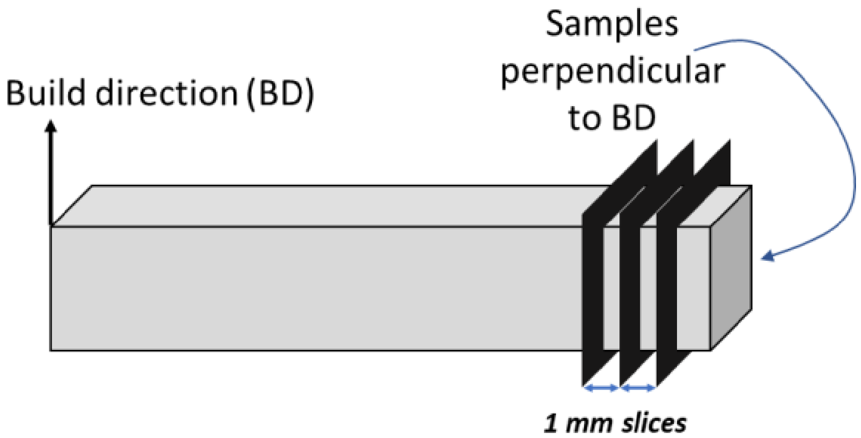
Samples were prepared using 316L powder with a powder bed fusion (PBF) technique.



(UUR)

Constant humidity/contamination exposures

Alloy type	Rel. Hum. (%)		Salt loading (µg/cm²)		Salt		Build	
	76	40	20	300	NaCl	ASW*	Parallel	Perpendicular
304AM	x		x		x			x
	x			x	x			x
	x			x		x		x
	x		x			x		x
		x		x		x		x
		x	x			x		x
		x		x		x	x	
316AM	x		x		x			x
	x			x	x			x
	x			x		x		x
	x		x			x		x
		x		x		x		x
		x	x			x		x
		x		x		x	x	
304W	x		x		x		N/A	
	x			x	x			
	x			x		x		
	x		x			x		
		x		x		x		
		x	x			x		
316W	x		x		x			
	x			x	x			
	x			x		x		
	x		x			x		
		x		x		x		
		x	x			x		



ASW composition	
Composition	**Concentration (g/L)**
NaCl	24.53
$MgCl_2.6H_2O$	5.20
Na_2SO_4	4.09
$CaCl_2$	1.16
KCl	0.695
$NaHCO_3$	0.201
KBr	0.101
H_3BO_3	0.027
$SrCl_2.6H_2O$	0.025
NaF	0.003

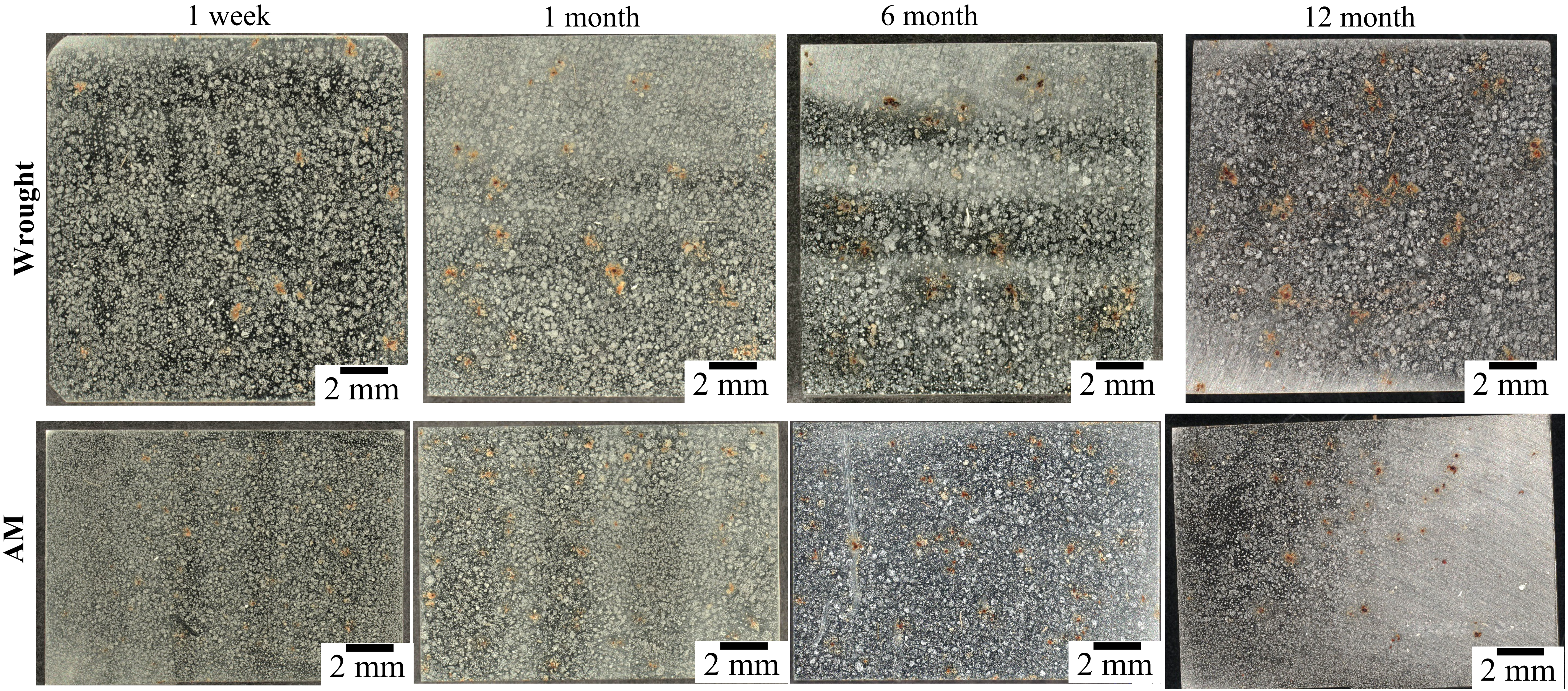
*Artificial Sea Water (ASW)

All samples polished to **600 grit prior to salt deposition.

***Held at **35ºC** for all atmospheric exposures.

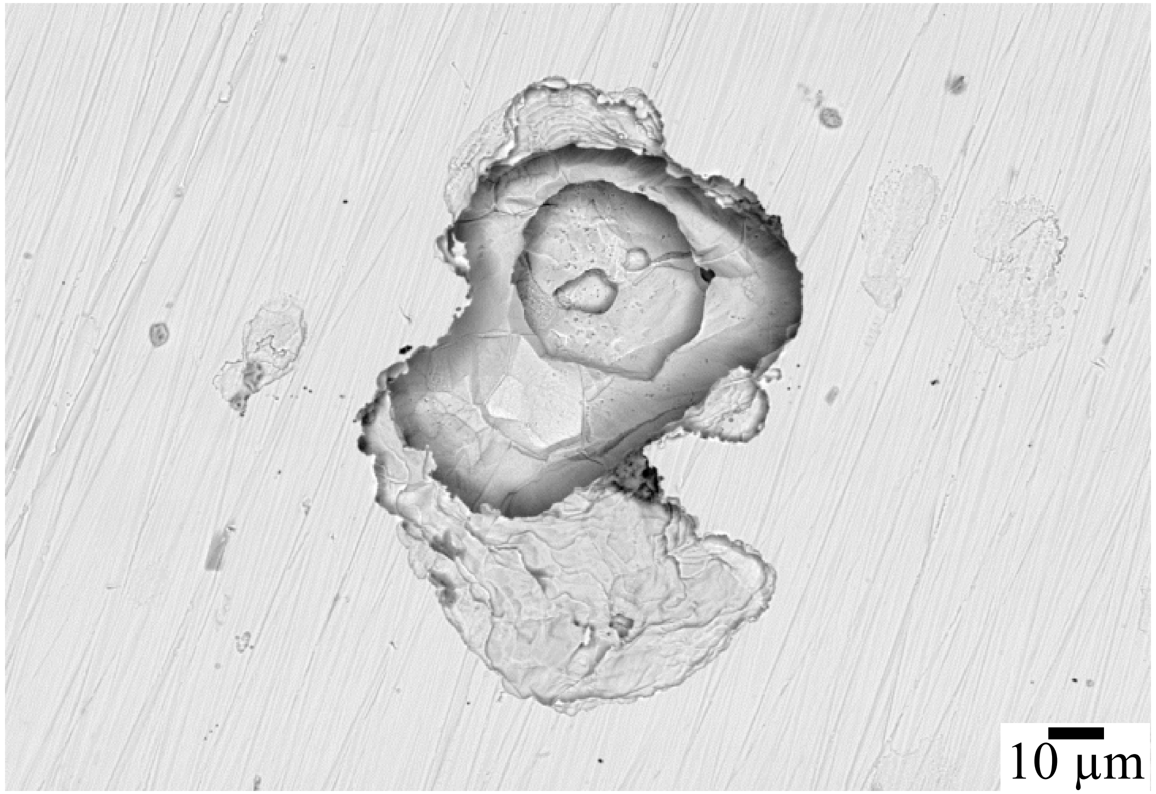
We will mostly show results from 316L material at 40% RH and 300 µg/cm² ASW. (UUR)

Optical images of **316L** specimen with **300 μg/cm^2 ASW at 40% RH**

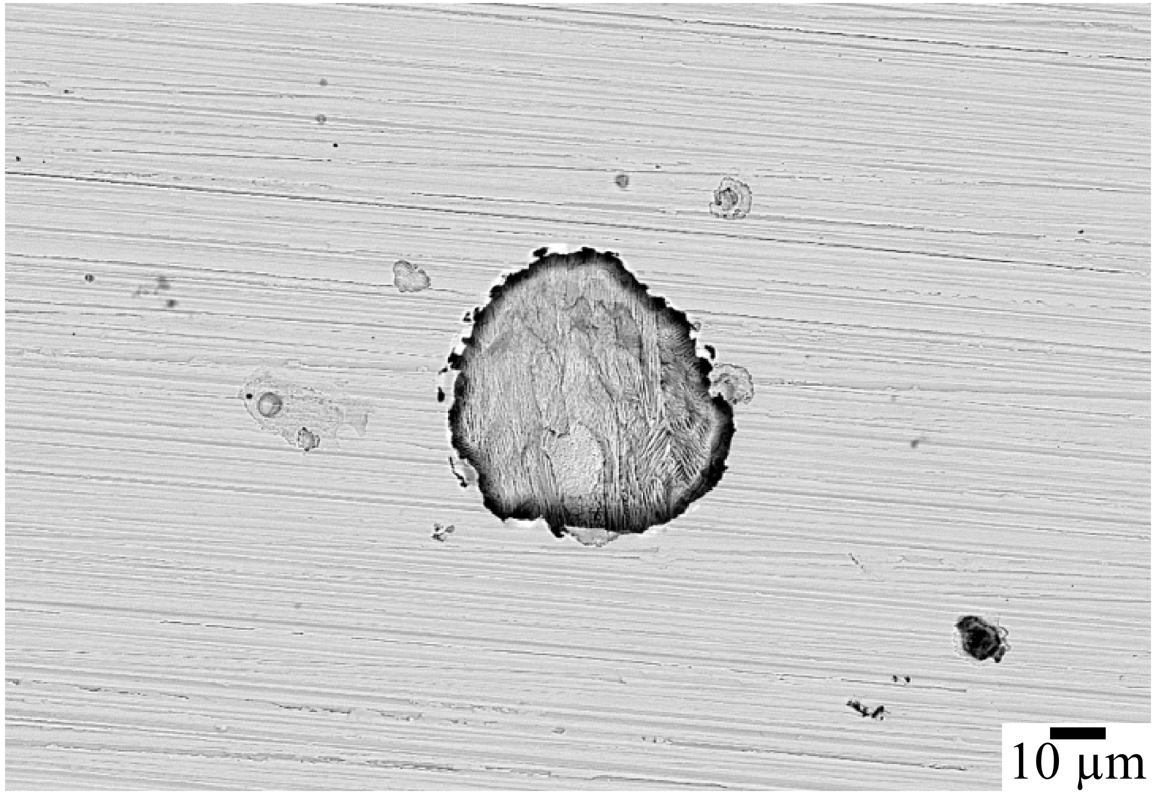


SEM images of **316L** specimen with **300 µg/cm² ASW at 40% RH**

Wrought



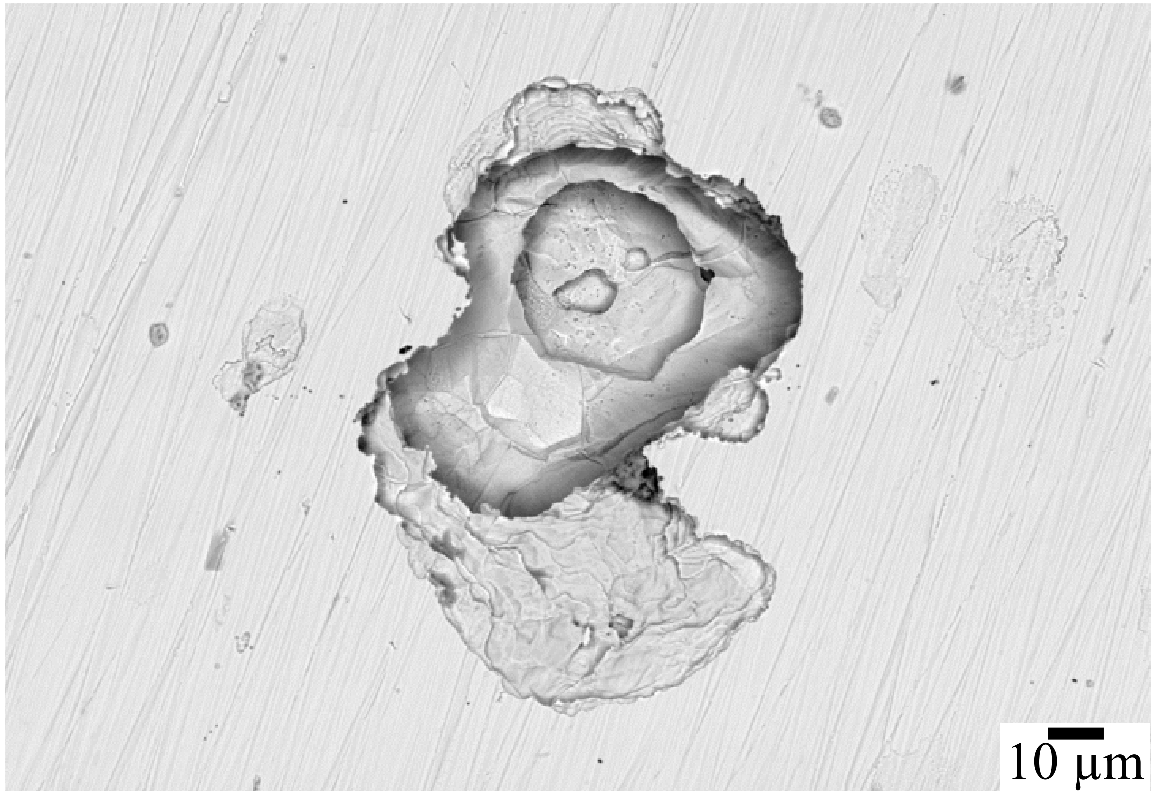
AM



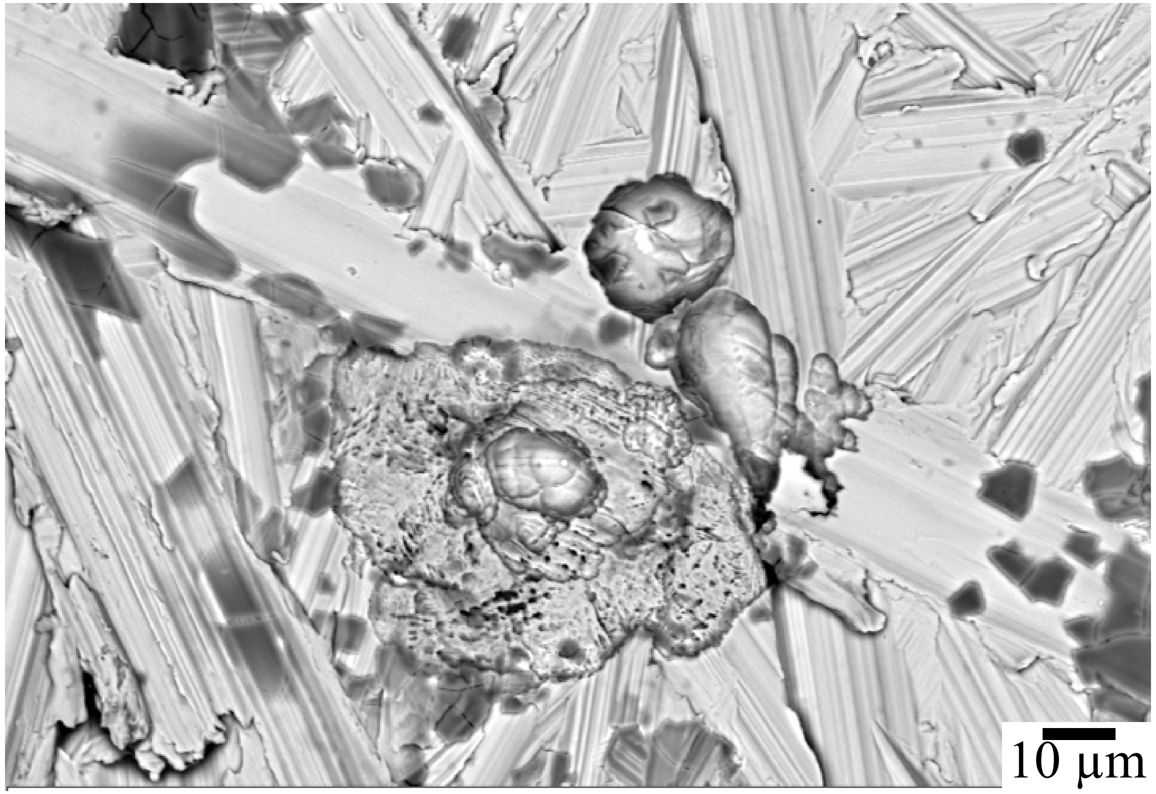
Cleaned surfaces with nitric acid solution.

SEM images of **Wrought 316L** specimen - **Comparison**

40% RH - 300 µg/cm^2 of ASW



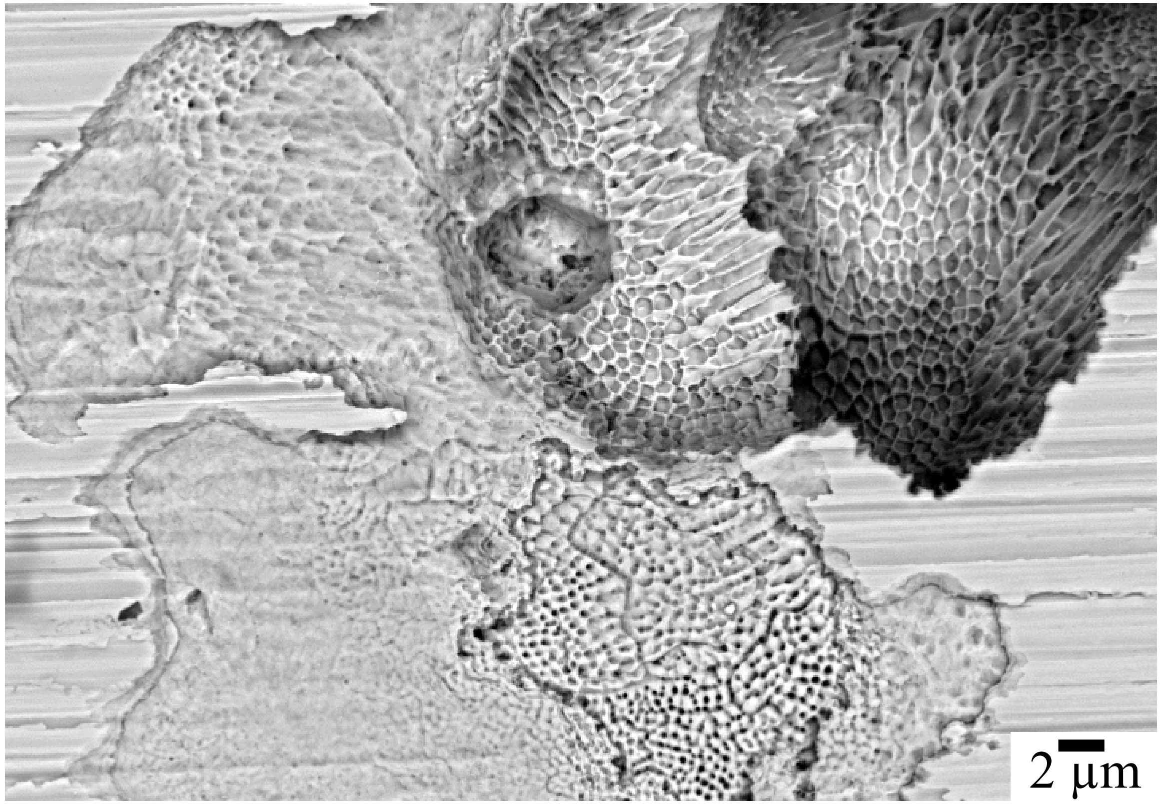
From 21 month exposure off coast of Florida



These samples were prepared to 120 grit ground finish.

SEM images of **AM 316L** specimen - **Comparison**

40% RH - 300 μg/cm² of ASW



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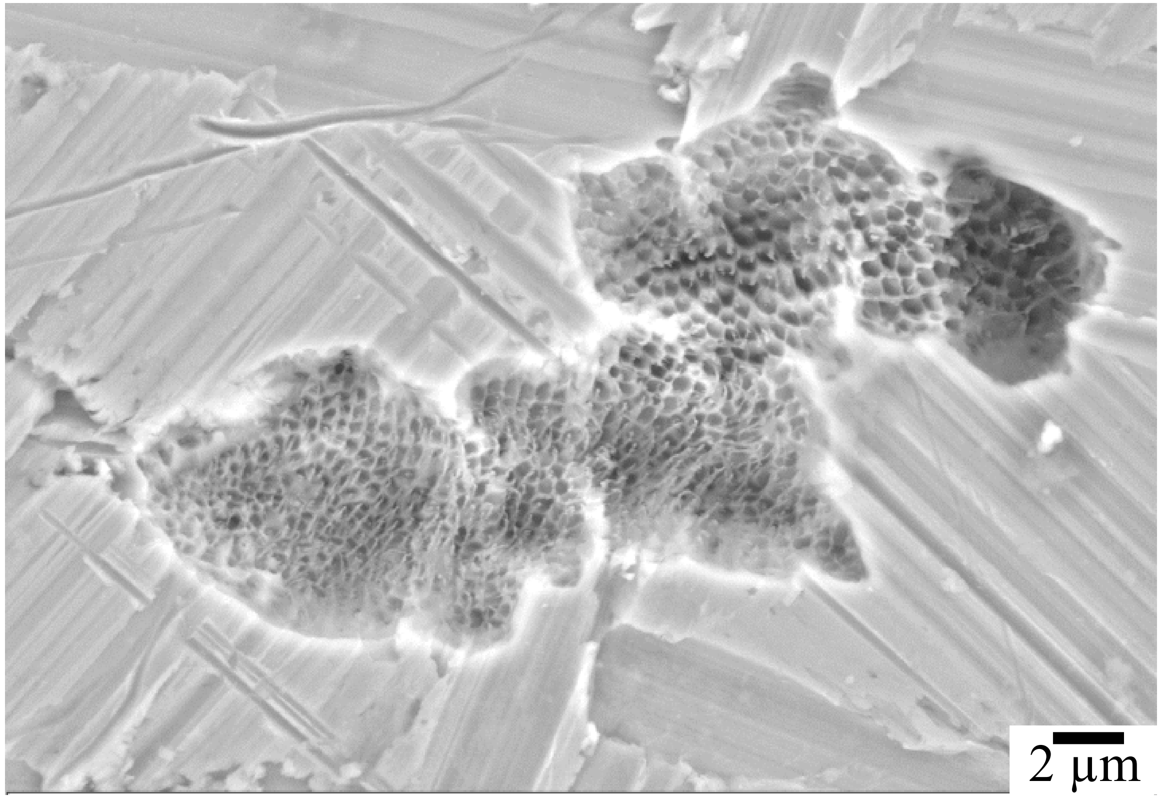

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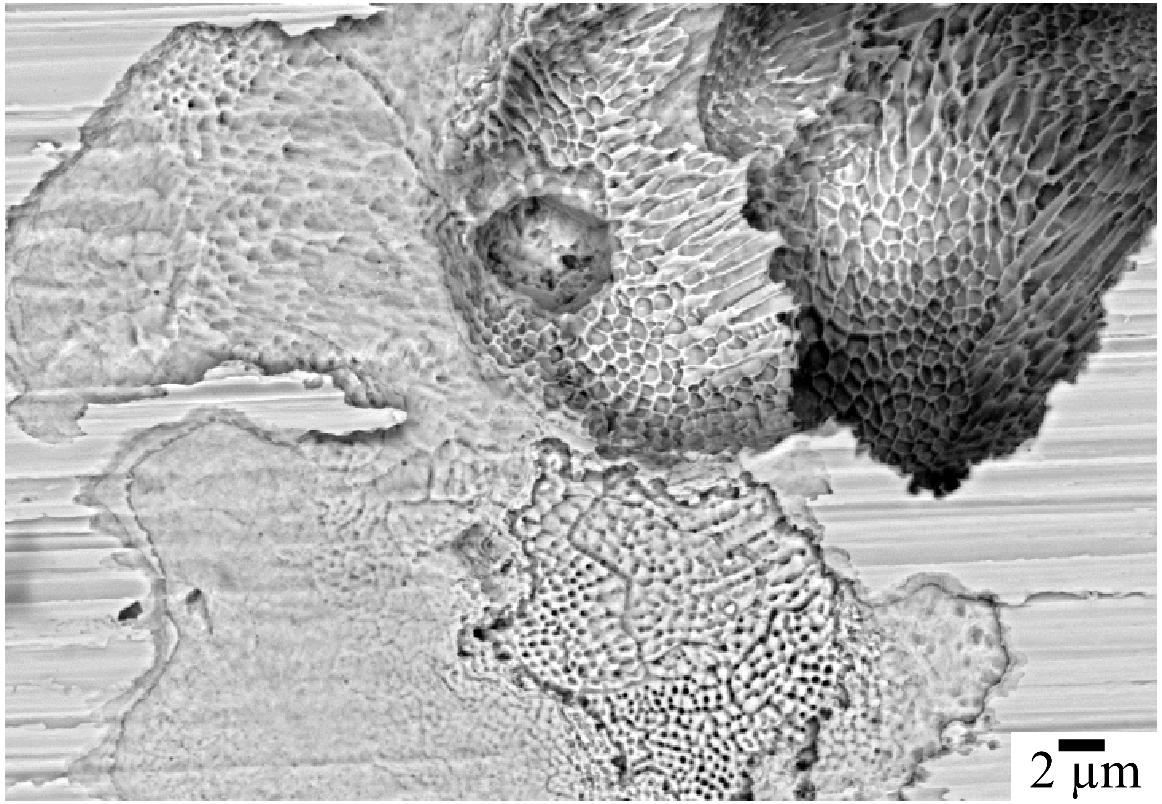
SEM images of **AM 316L** specimen - **Comparison**

40% RH - 300 μg/cm^2 of ASW



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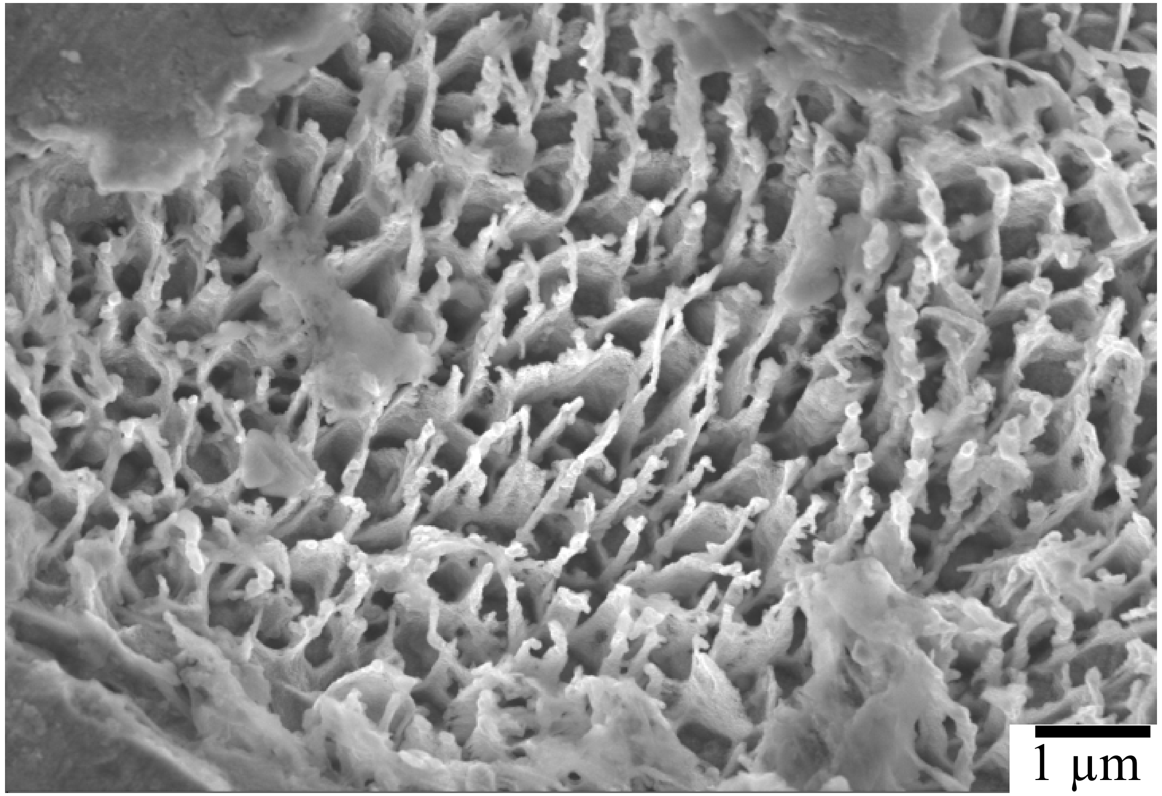
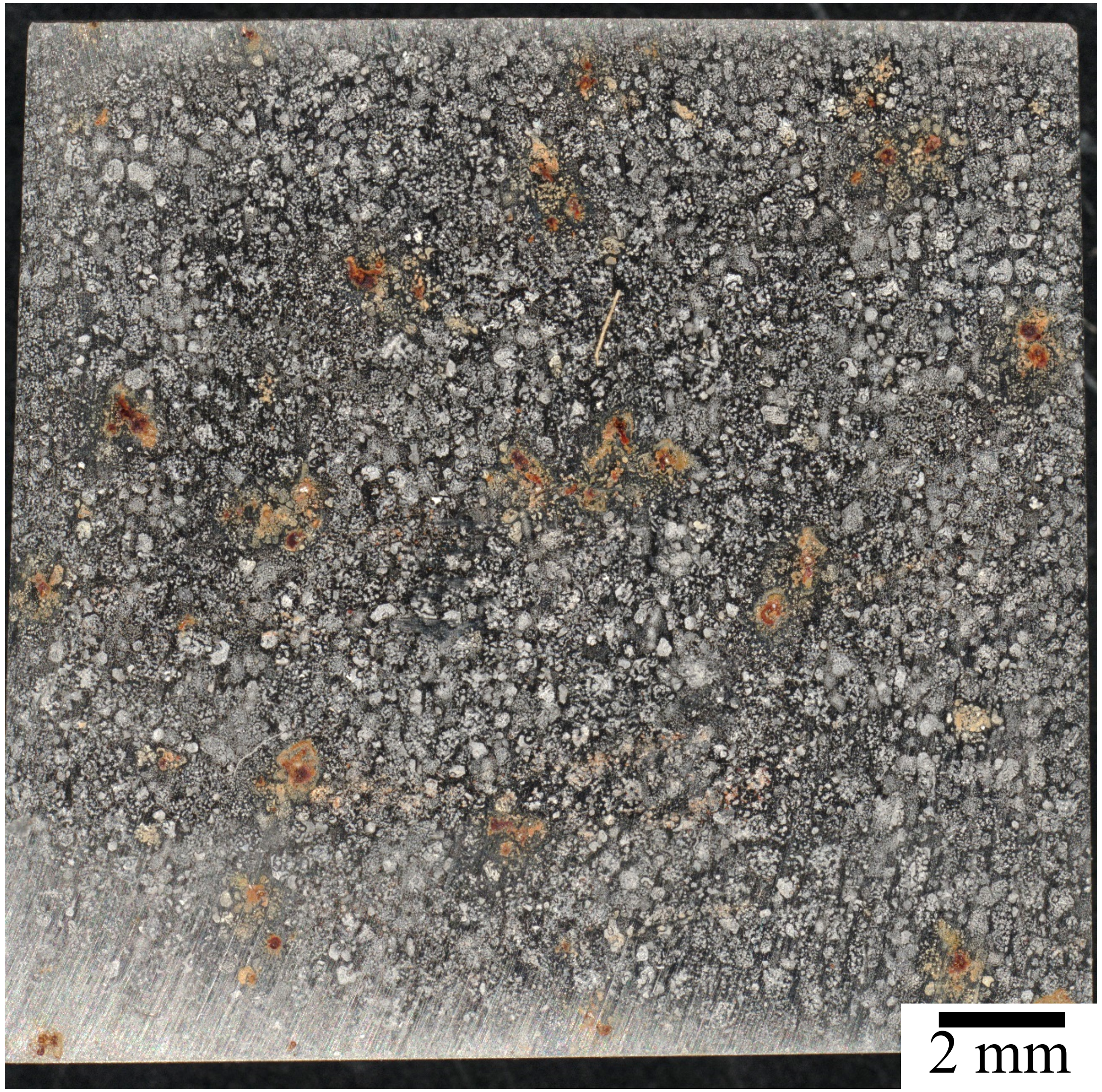


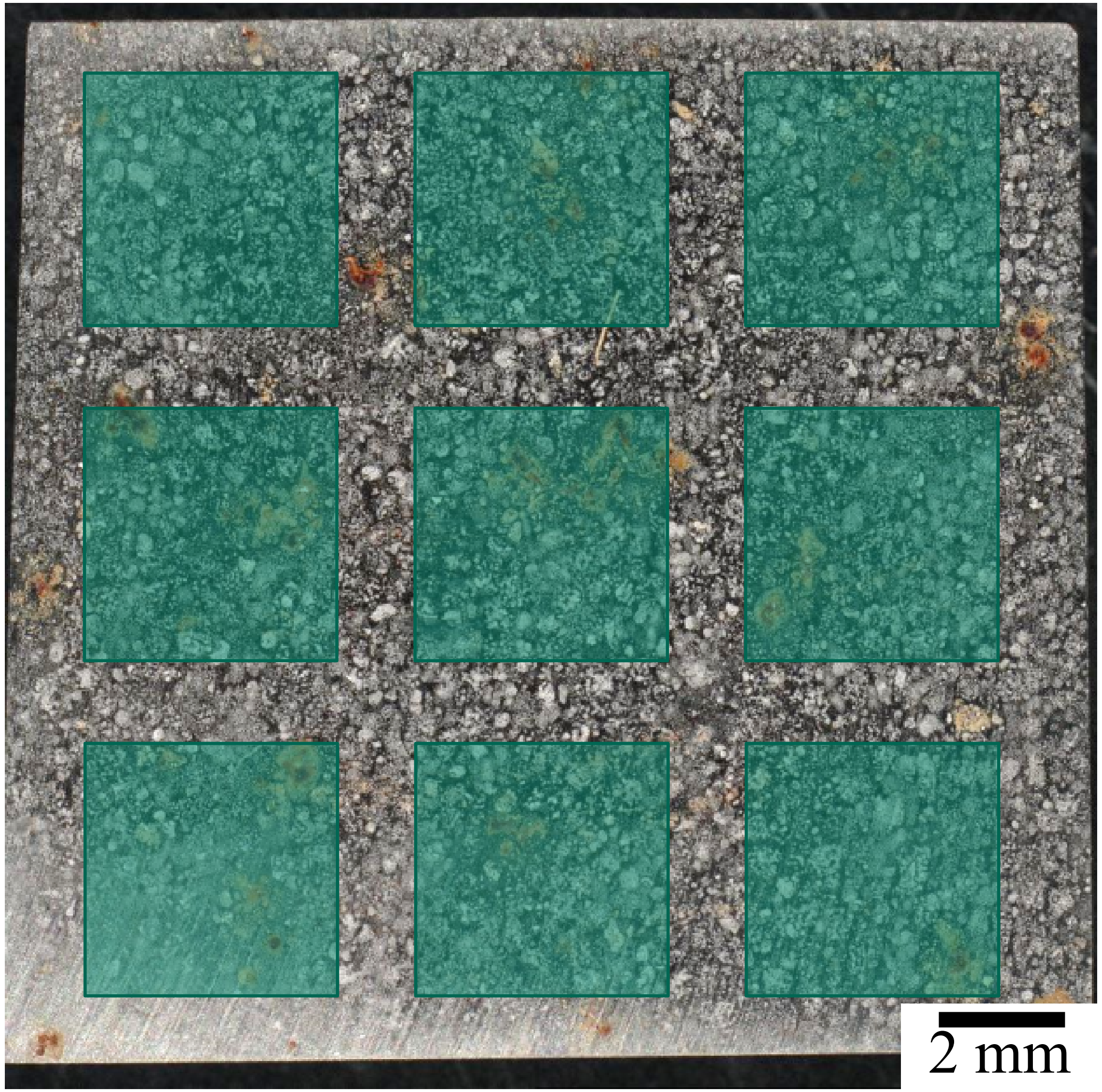
Pit Analysis Process



White light interferometry (Nexview™)

(UUR)

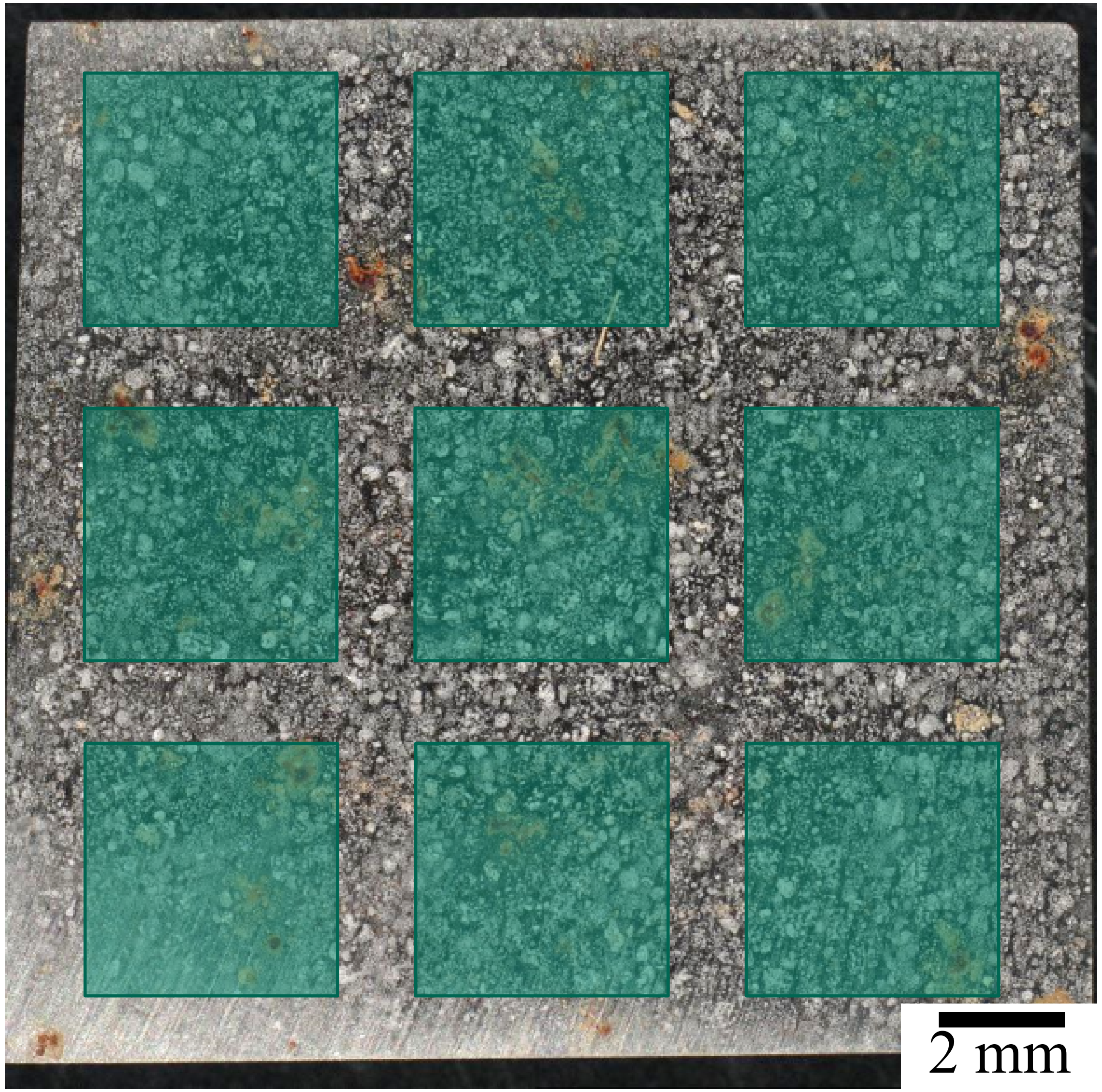
Pit Analysis Process



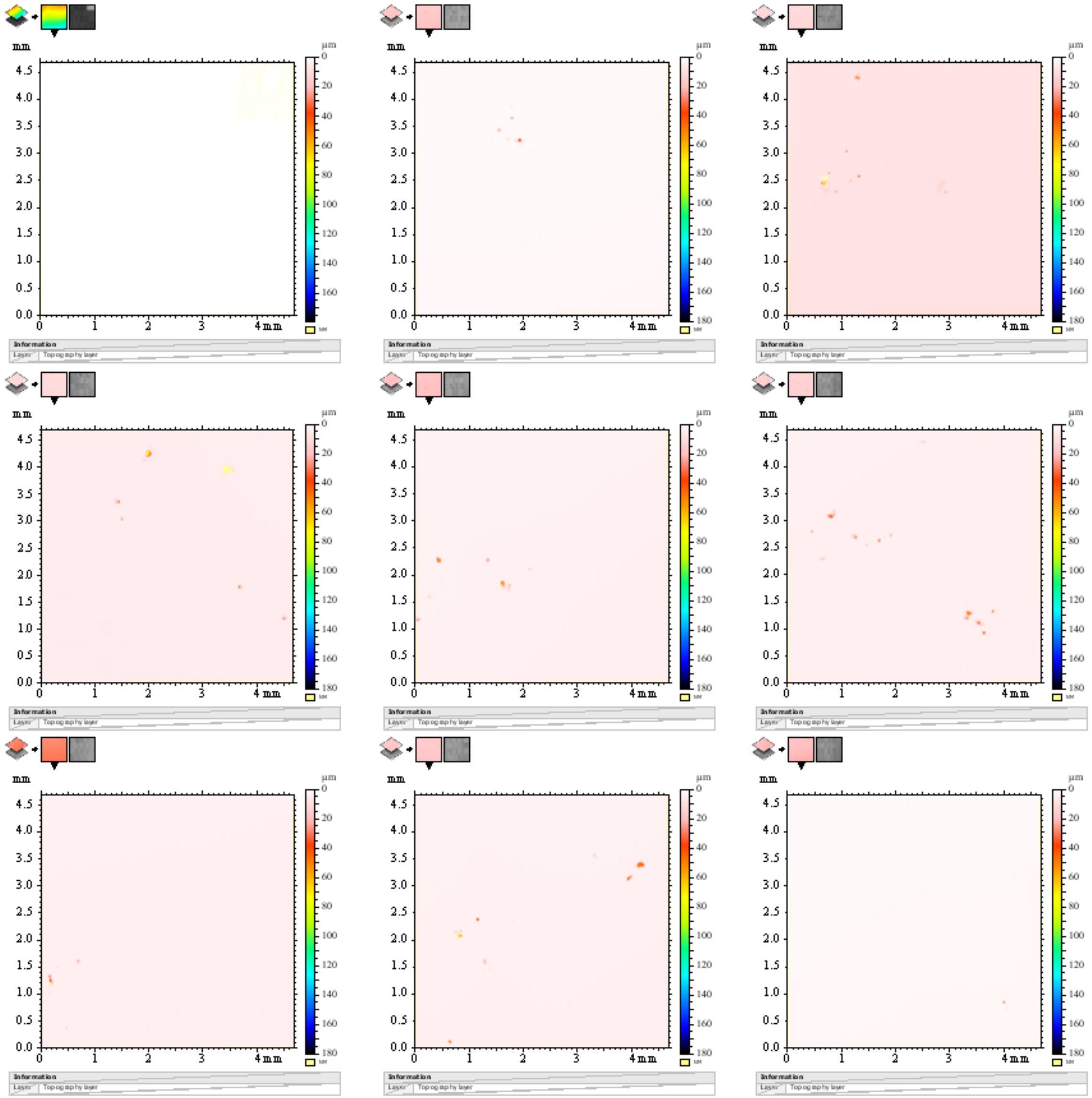
White light interferometry (Nexview^{TM})

(UUR)

Pit Analysis Process



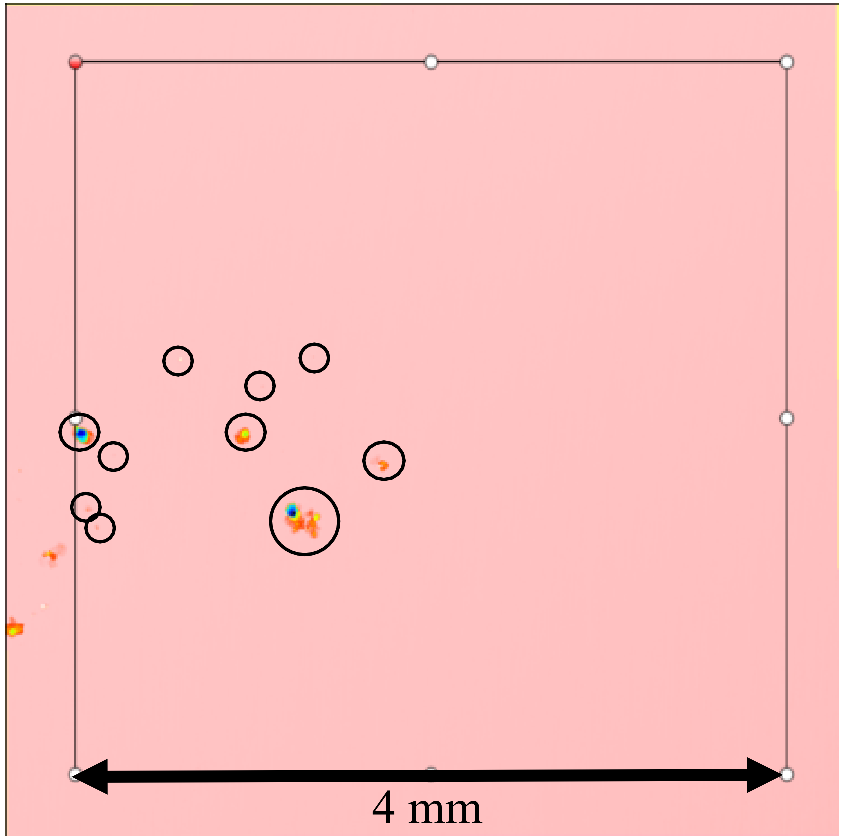
Raw profilometry data



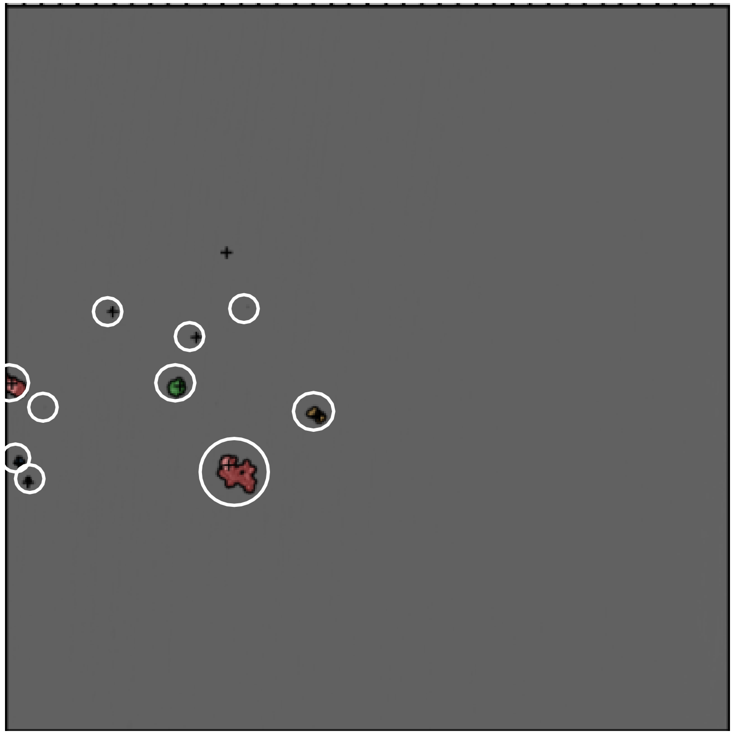
(UUR)

Pit Verification Process

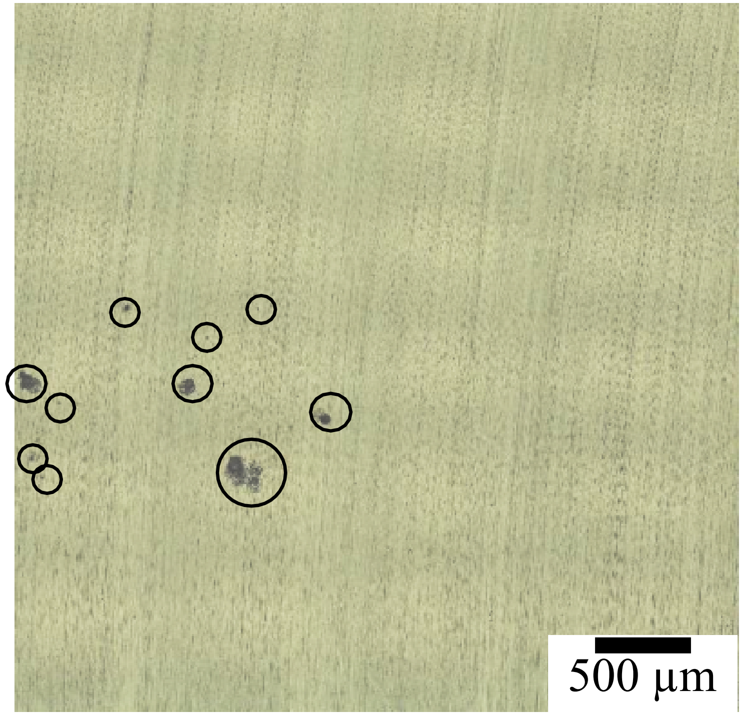
Raw profilometry data



Processed particle data

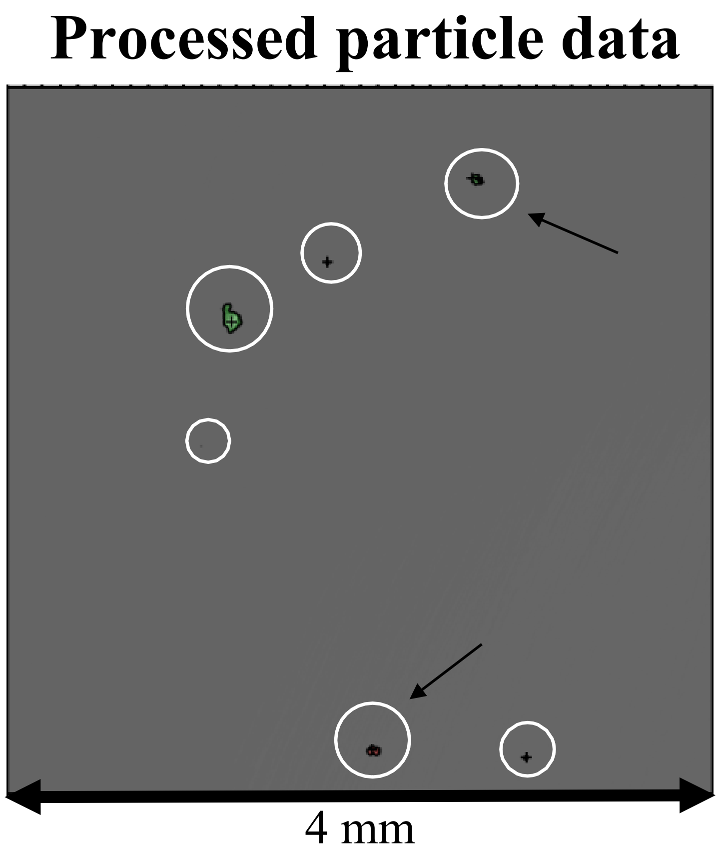


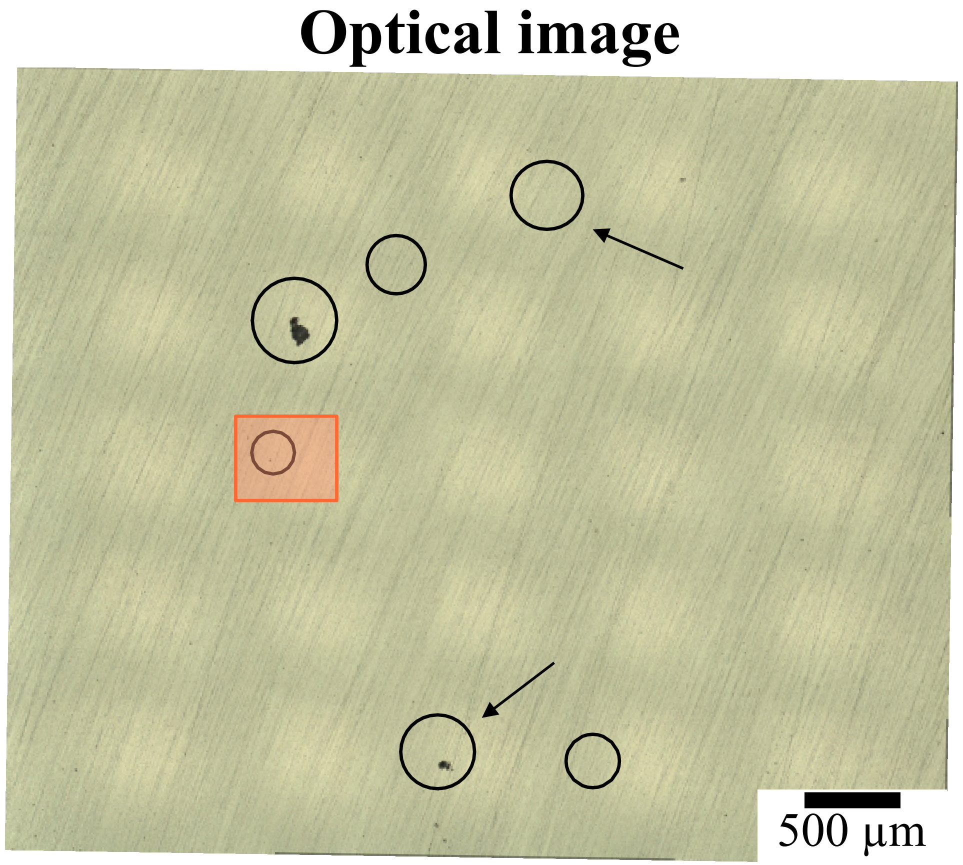
Optical image



- White light interferometry measurements of cleaned surfaces to detect pits were processed to smooth surfaces.
- Mean plane threshold was used to eliminate scratch marks: **0.5 µm below surface.**
- Filtering criteria: **Maximum Height (Depth) > 0.2 µm** and **Volume/Area ratio > 0.2 µm.**
- Each circle contains one pit. *There were a total of 10 pits through the filtering process.*
- Through visual inspection, the particles are verified to be pits.

Pit Verification Process – what are we missing?

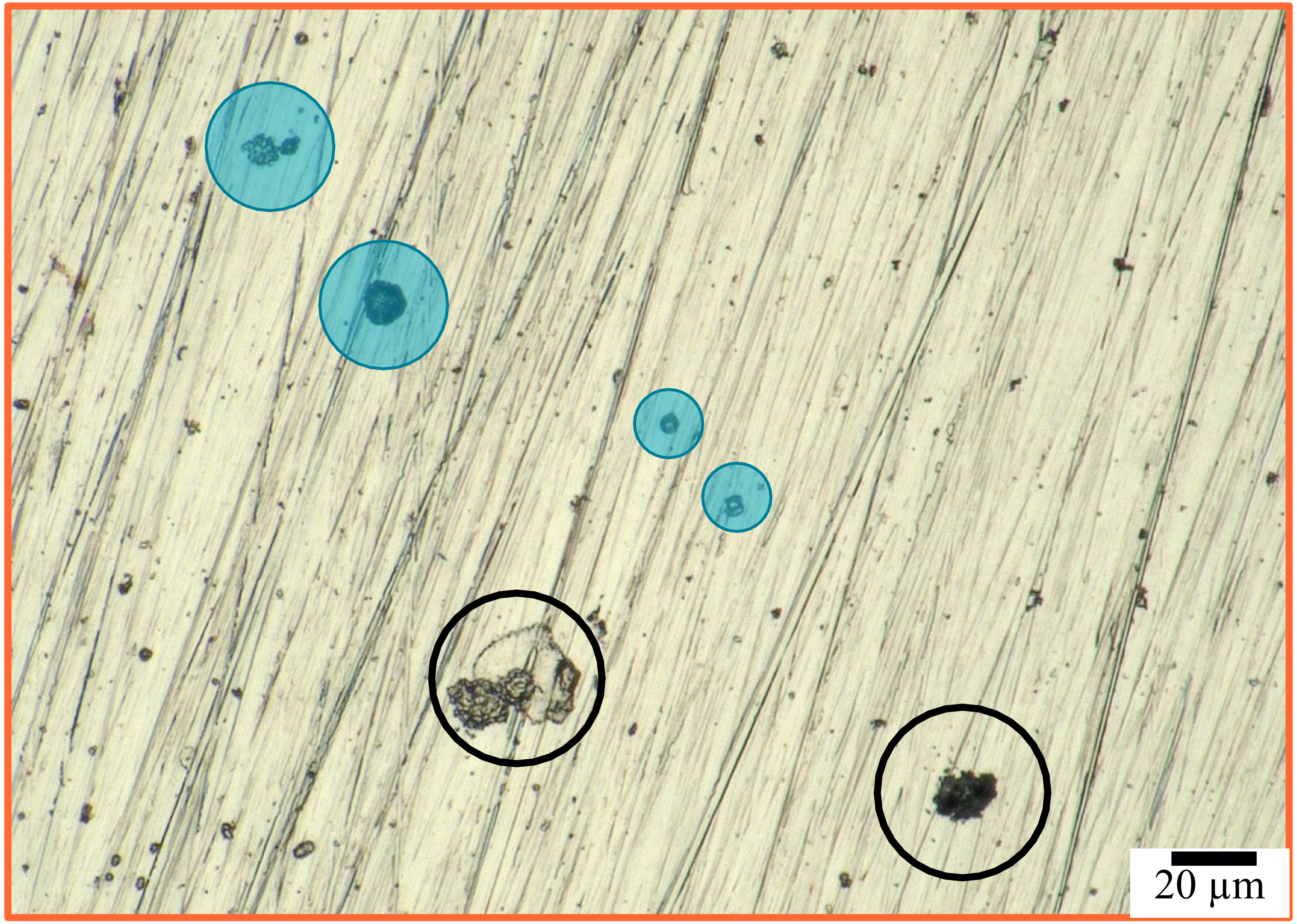




Pit Verification Process – what are we missing?

- Pits not picked up from interferometry analysis because they are **too small**.
- Specific to AM – lack of fusion **porosity** can cause outlier pit information.
- **Contamination** of the surface can lead to unexpected pits – make sure surfaces are clean before measurements!

Optical image



Another filter based on Height/Area ratio was used to eliminate contamination/pore outliers.

(UUR)

Pit statistics of **316L specimen - 300 µg/cm² ASW at 40% RH**

Wrought

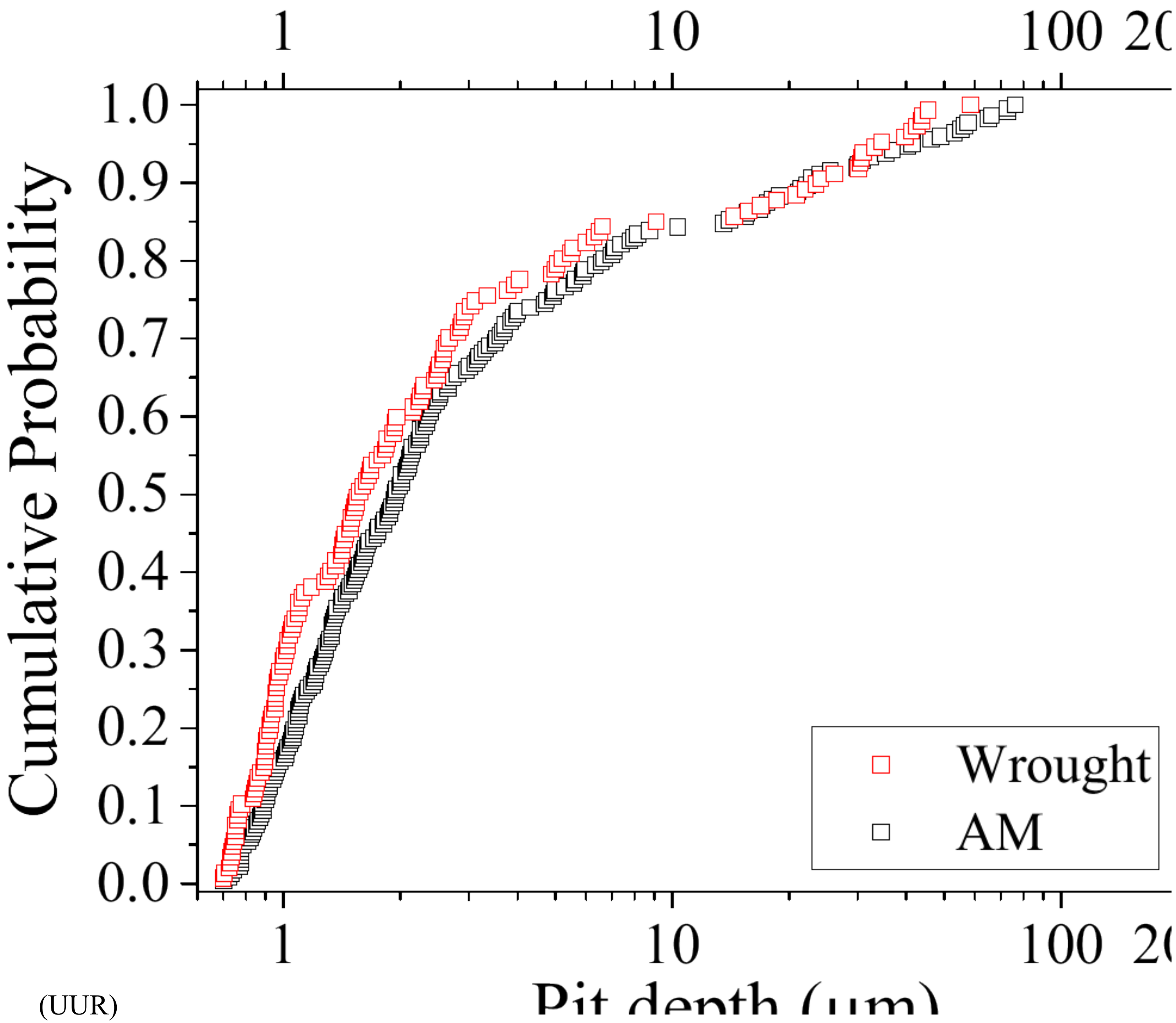
	Average pit depth (µm)	Measured deepest pit (µm)
1 week	2.9	35.1
1 month	7.0	41.8
6 months	6.3	58.5
12 months	11.7	118.1

AM

	Average pit depth (µm)	Measured deepest pit (µm)
1 week	4.0	32.6
1 month	10.1	*210.4* (73.3)
6 months	7.6	76.2
12 months	9.4	80.2

Likely porosity

6 months – Pit depth



(UUR)

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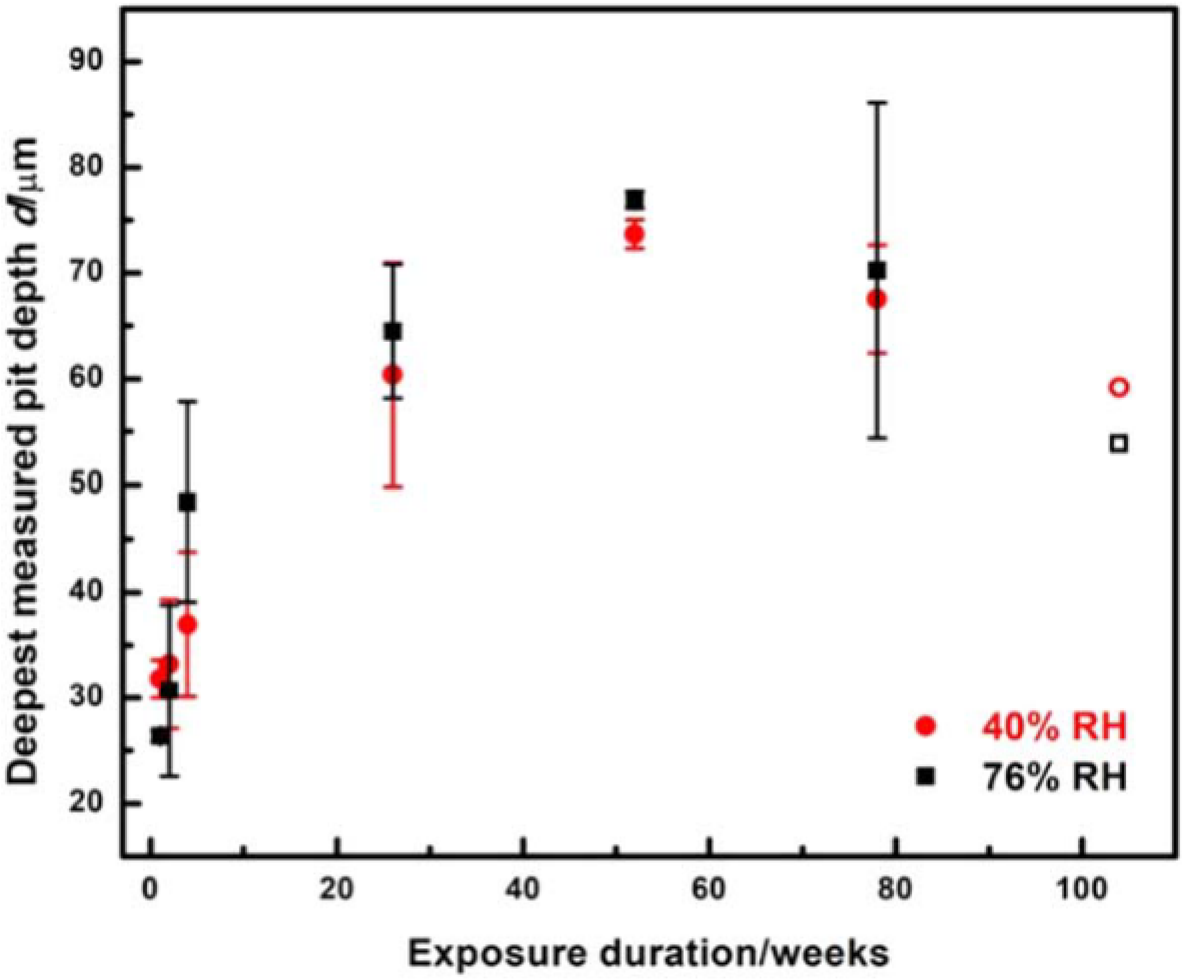
1 week	4.0	32.6
1 month	10.1	*210.4* (73.3)
6 months	7.6	76.2
12 months	9.4	80.2

Likely porosity

10 deepest pits/area from literature – 304L



Source: Srinivasan (2021)

(UUR)

Pit statistics of **316L specimen - 300 μg/cm² ASW at 40% RH**

Wrought

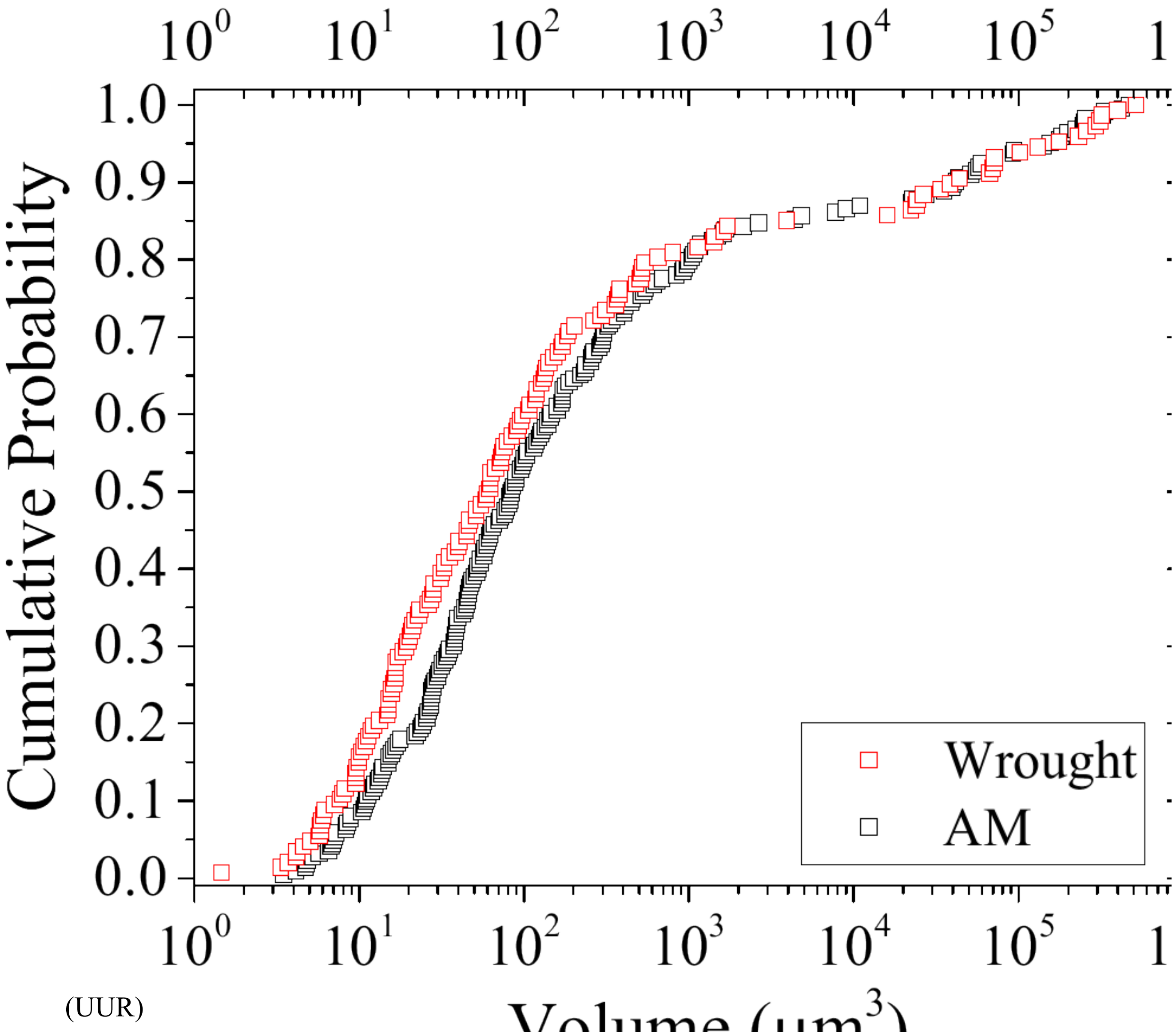
	Volume per area ($\mu m^3/cm^2$)	# of Pits/cm^2
1 week	$1.15x10^5$	50
1 month	$9.28x10^5$	65
6 months	$2.34x10^6$	105
12 months	$2.75x10^6$	96

AM

	Volume per area ($\mu m^3/cm^2$)	# of Pits/cm^2
1 week	$3.66x10^5$	89
1 month	$9.35x10^5$	88
6 months	$2.97x10^6$	155
12 months	$1.74x10^6$	53

Total area analyzed: 1.3 to 1.44 cm²

6 months – Pit volume



(UUR)

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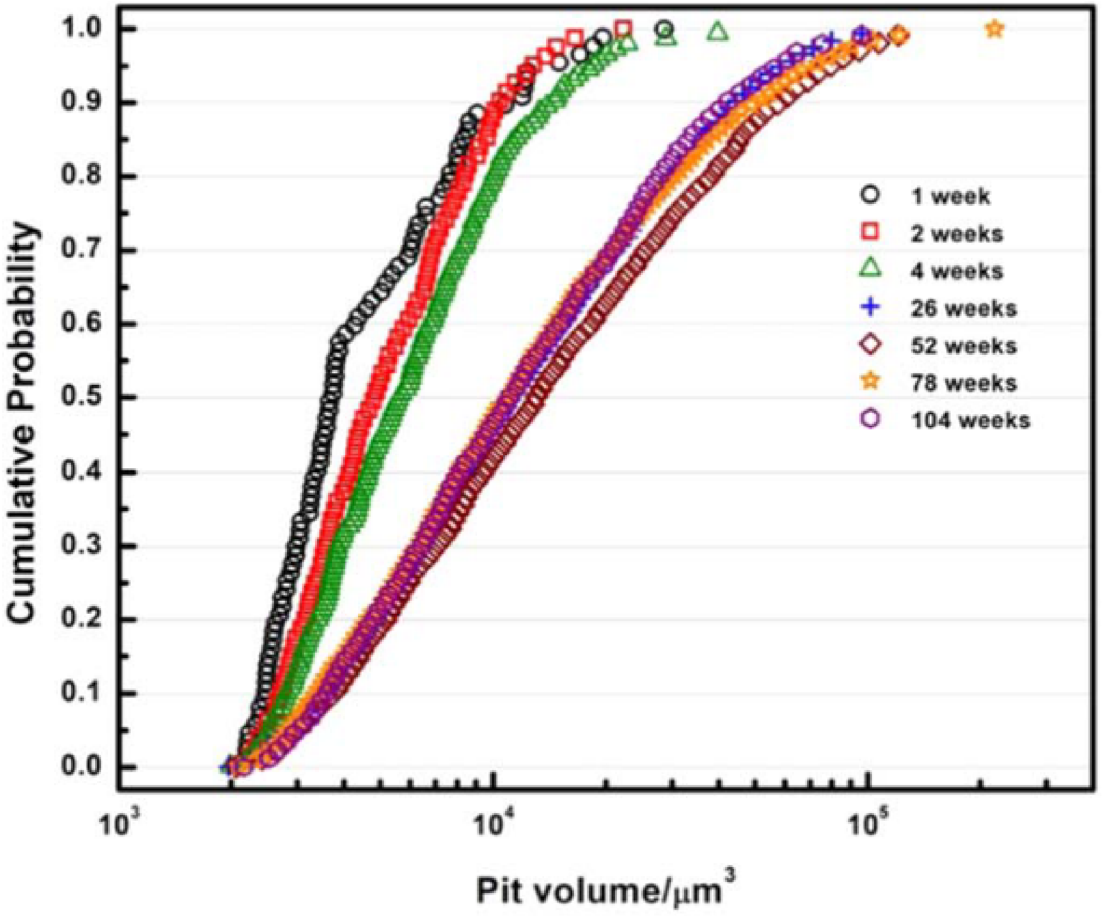
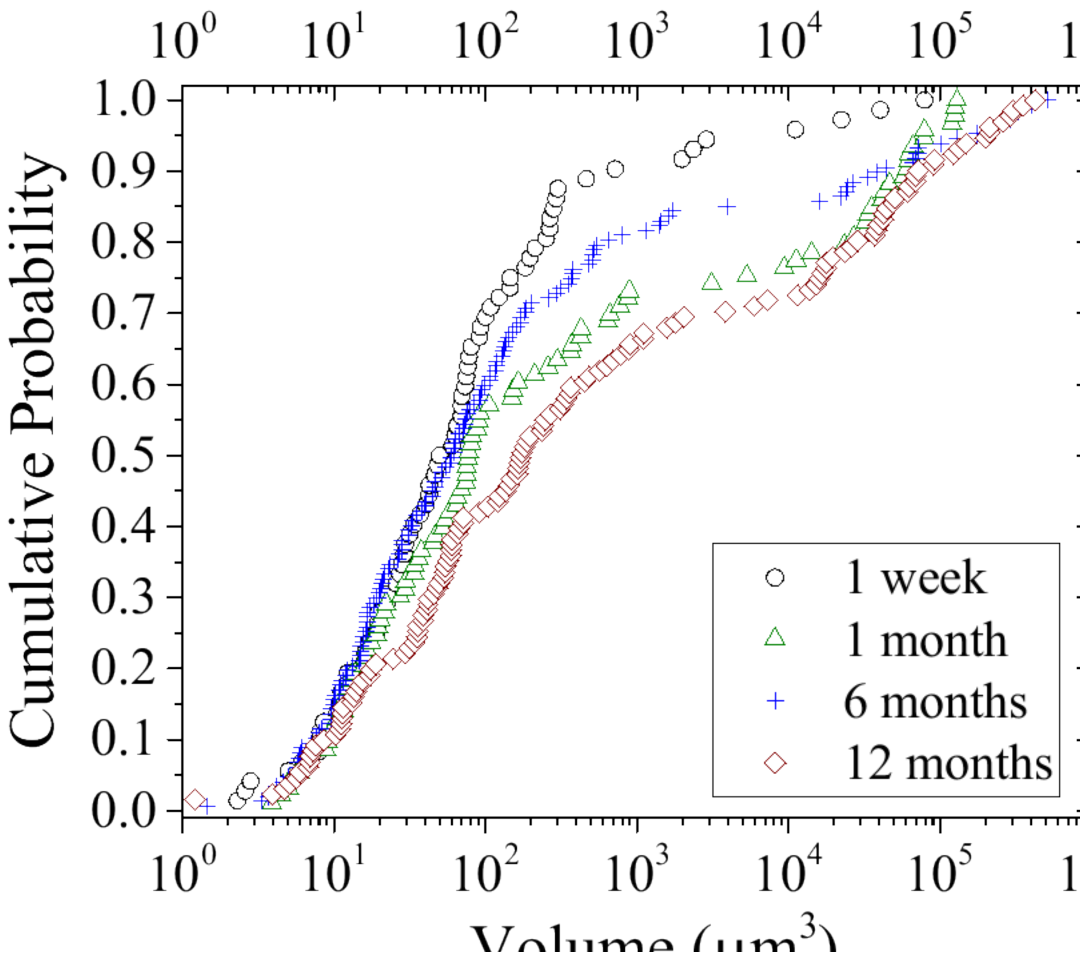


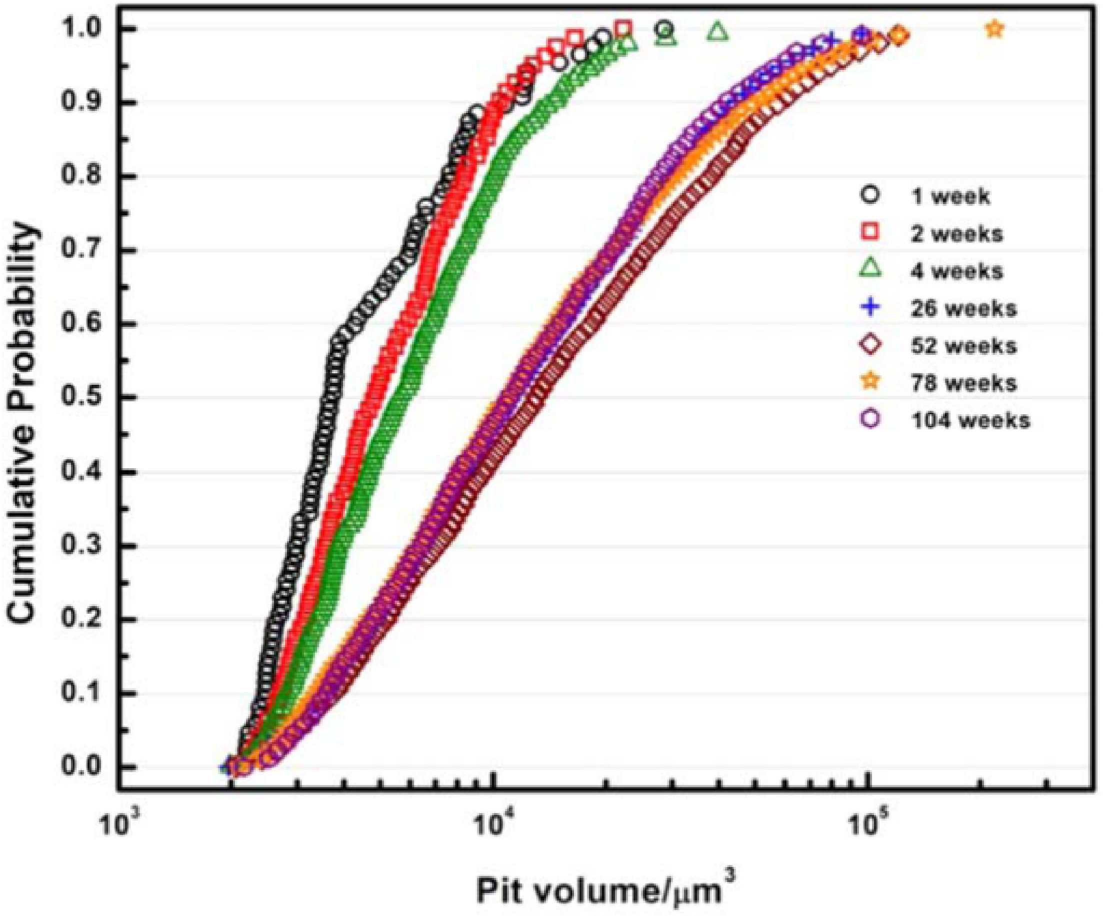
J. Srinivasan, et al,, Long-Term Effects of Humidity on Stainless Steel Pitting in Sea Salt Exposures, J. Electrochem. Soc., 168 (2021).

Wrought pit volume – with time



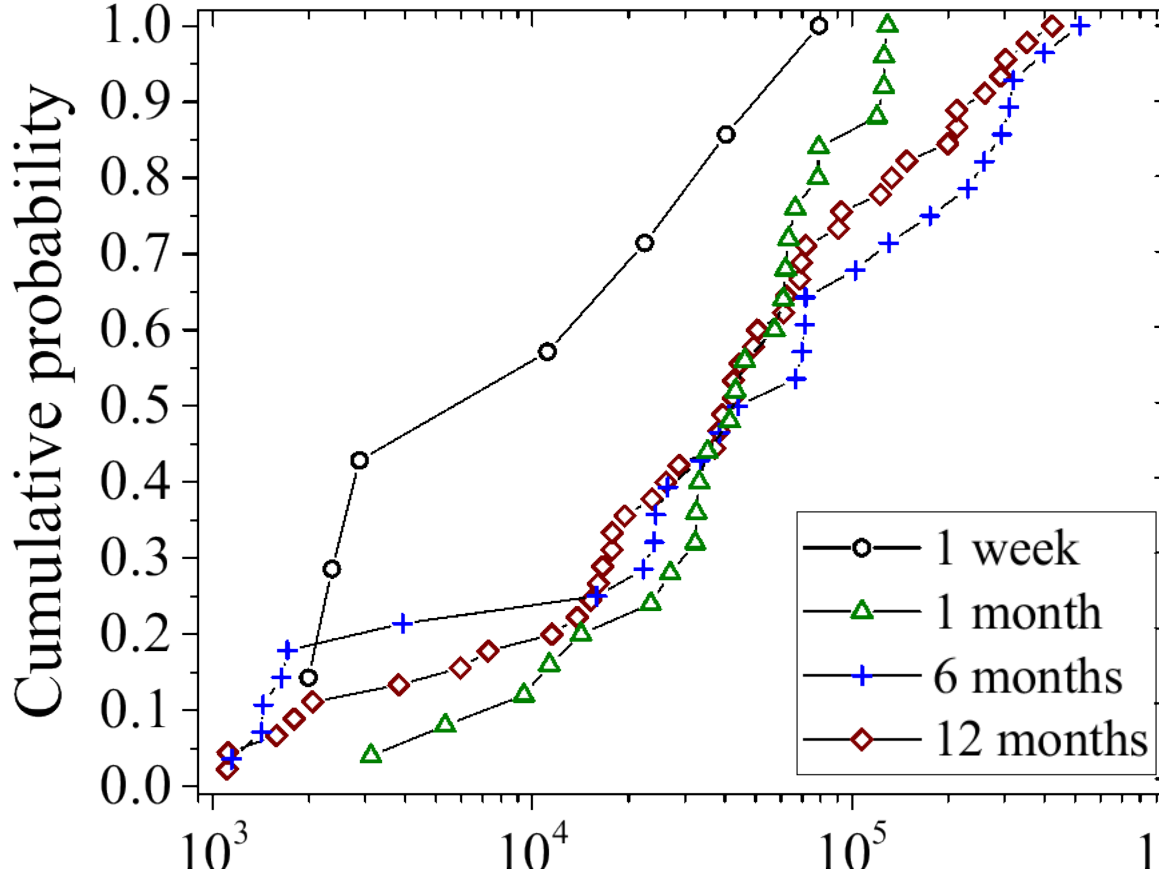
Pit statistics of **316L** specimen - **300 µg/cm² ASW at 40% RH**

Total measured area = 6 cm²



J. Srinivasan, et al,, Long-Term Effects of Humidity on Stainless Steel Pitting in Sea Salt Exposures, J. Electrochem. Soc., 168 (2021).

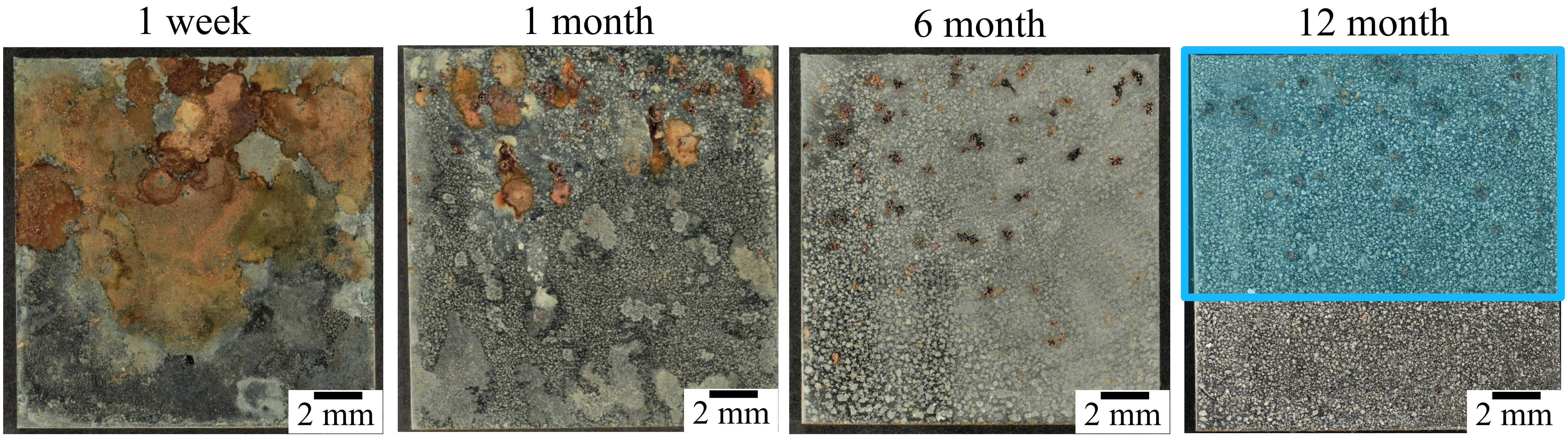
Wrought pit volume – with time



Total measured area = 1.3 cm²

(UUR)

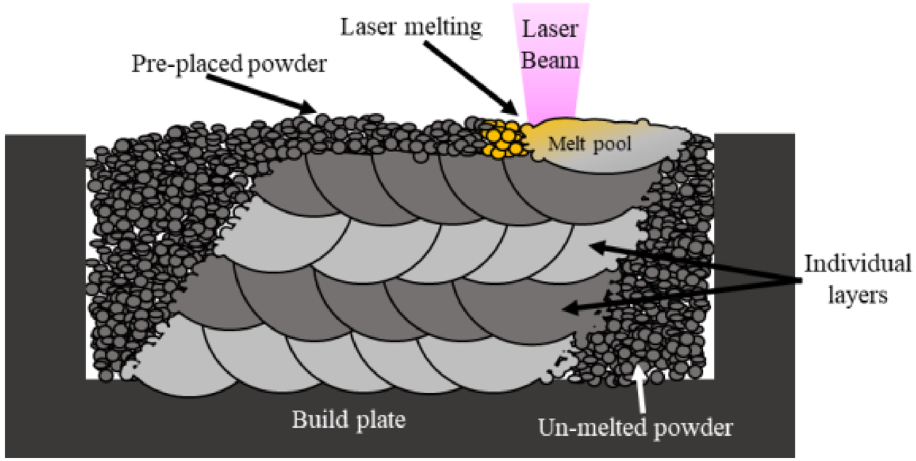
Optical images of **AM 304L** specimen with **300 µg/cm² ASW at 76% RH**



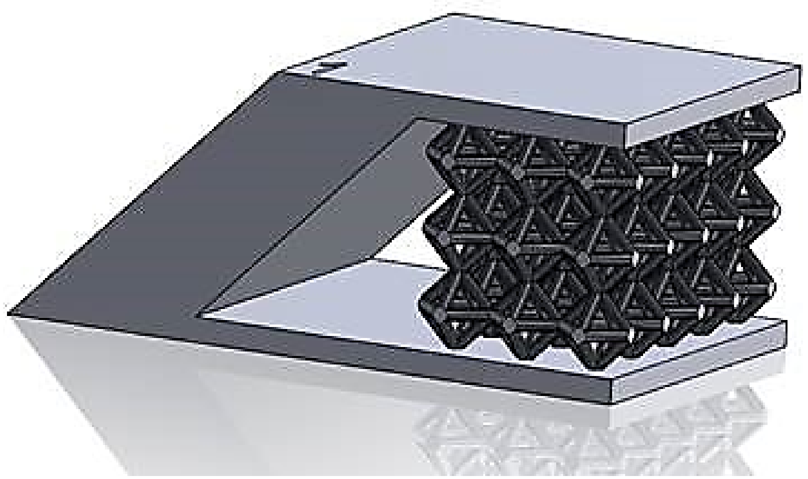
Constant humidity/contamination experiments

- Pit morphologies were similar to coastal exposures, besides the generally deeper pits for the constant humidity exposures.
- Pit density seems to be higher for AM samples, working on three potential hypotheses:
  - More initiation because of more preferential sites (oxides, cell boundaries, melt pool boundaries, etc.).
  - More initiation because of more efficient cathode (heterogeneous microstructure effects).
  - Or, more pits die (stop corroding) faster because of the heterogeneous microstructure (Cr/Mo enriched cell boundaries block propagation).
- Max pit depth was greater for AM at early times, but switched to Wrought after 12 month exposures.
- Initiation of local corrosion is most likely to occur in lack of fusion pores for samples at elevated humidity (≥76%). Most obvious for AM 304L samples.

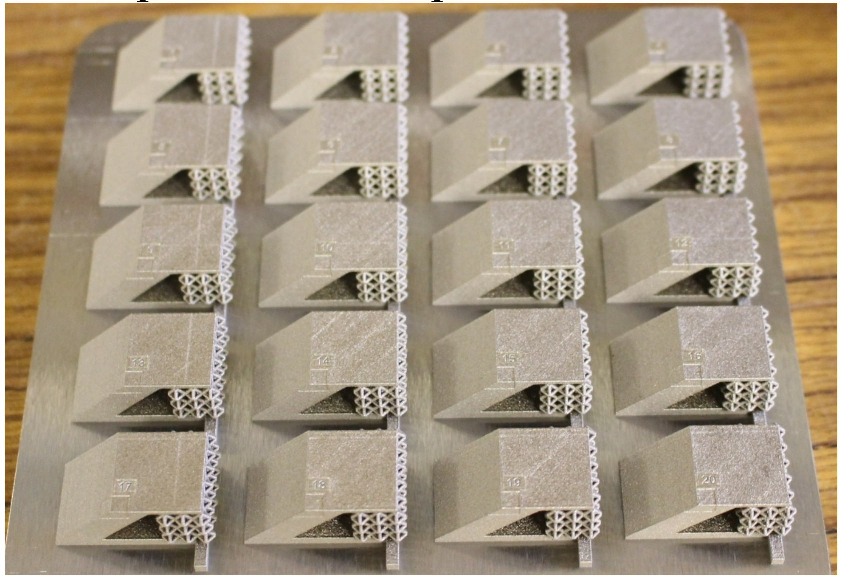
Powder bed fusion 316L samples



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



Build plate of 20 samples

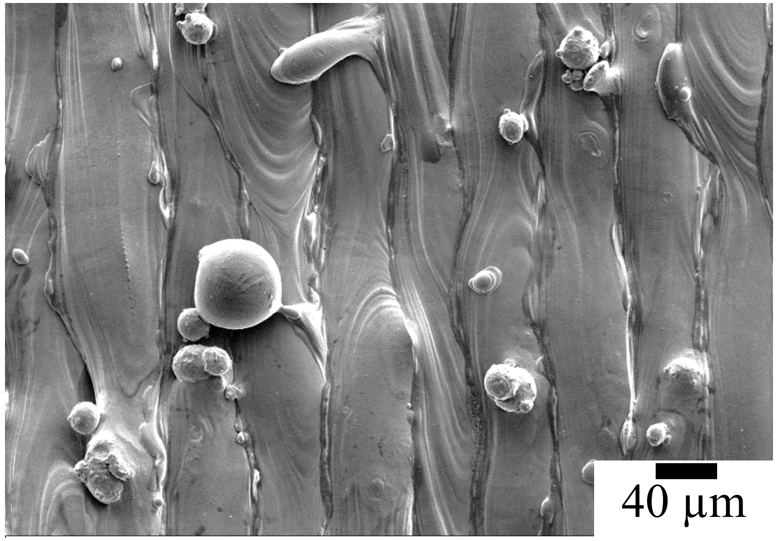


- Samples were printed on 5 different PBF machines, all with the machines optimized parameters for part density.

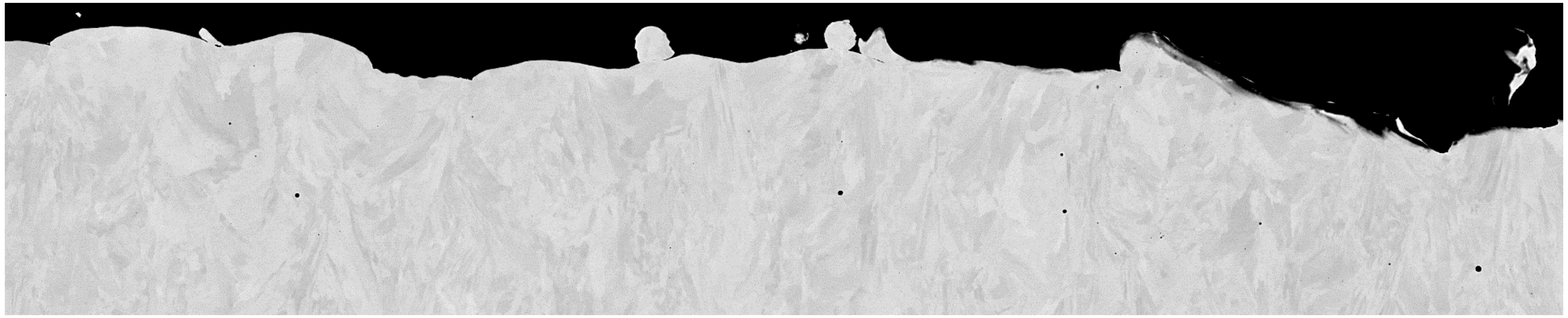
- SEM images of powder.
- Composition determined by ICP-MS and LECO.
- Powder analysis with laser diffraction.

(UUR)

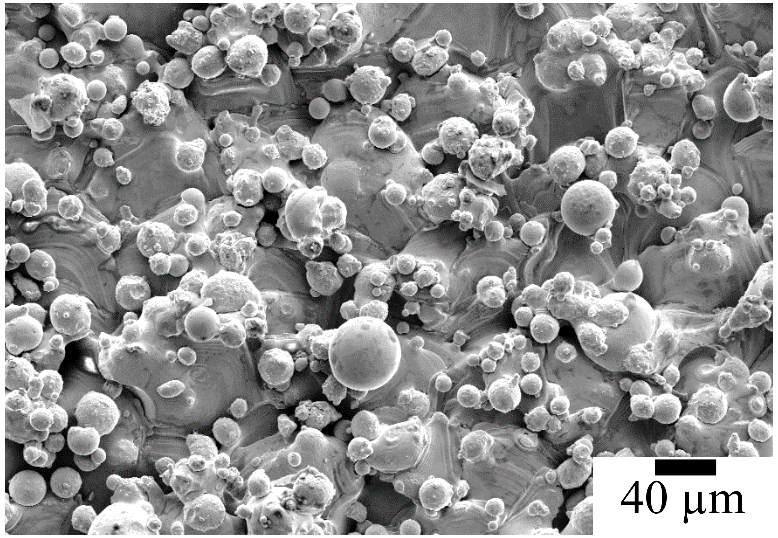
What do as-printed surfaces look like?



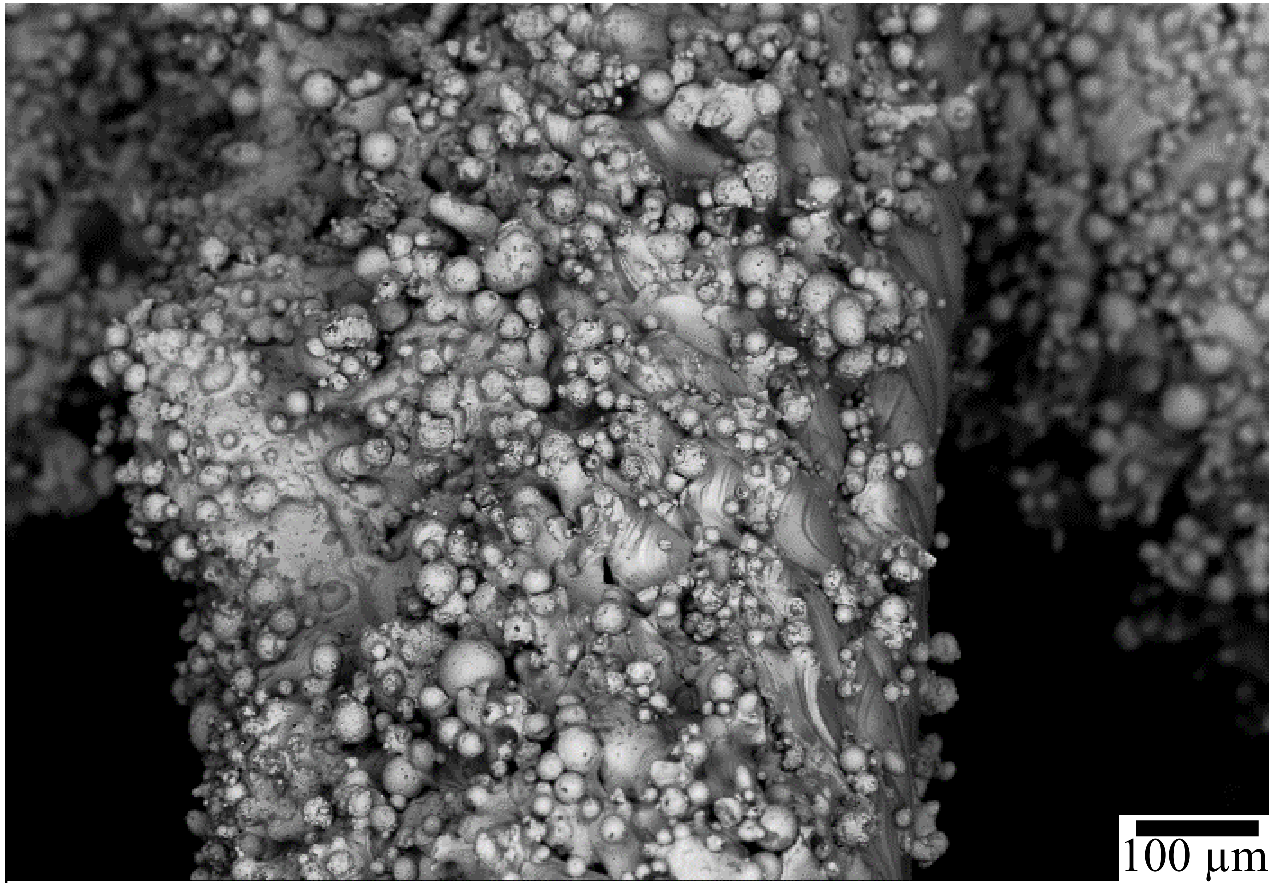
Top surface



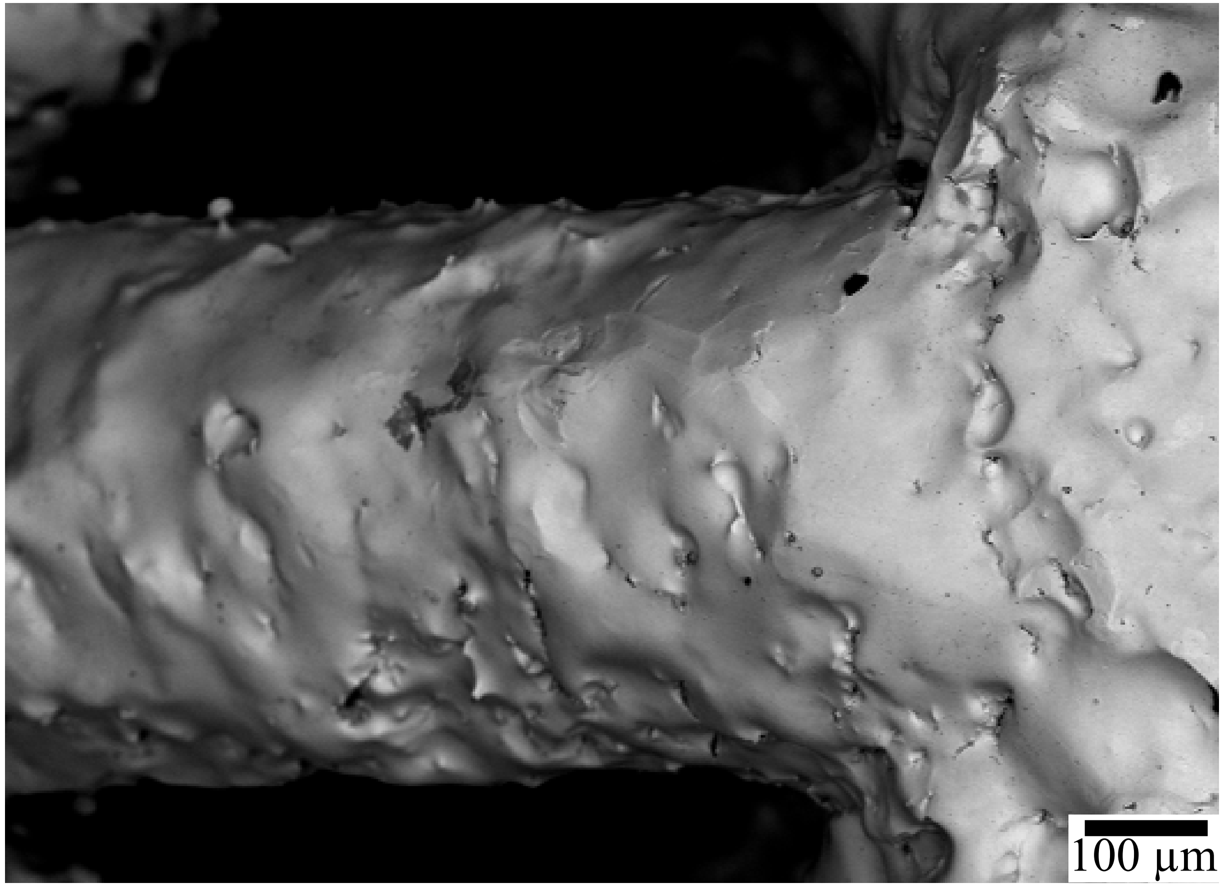
Side surface



As-printed lattices?



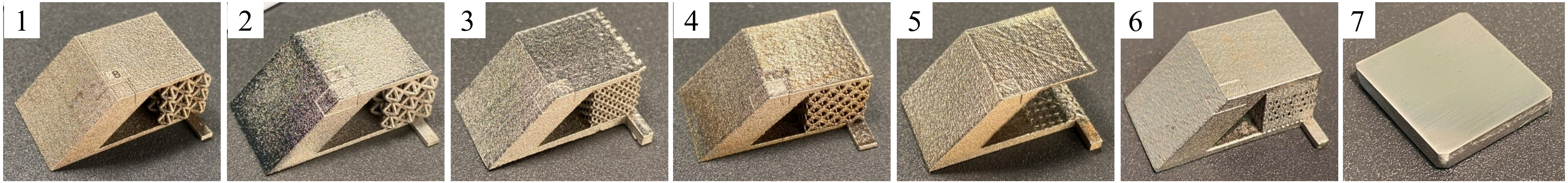
As-printed



DC Electropolished

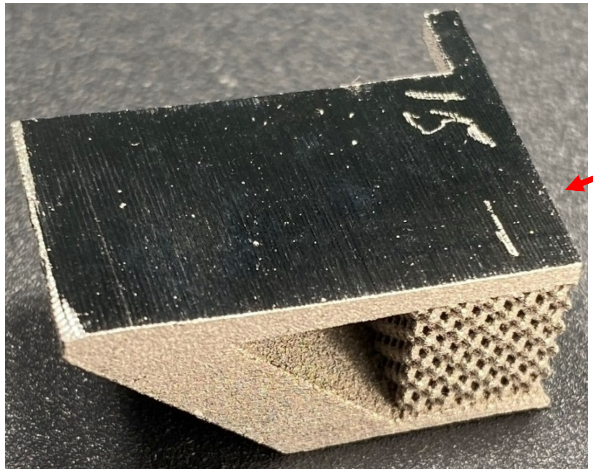
ASTM G85-A2: Sample ID

1st set – ProX200 As-printed – *Wire EDM cut surface*
2nd set – ProX200 – *Electropolished*
3rd set – EOS M290 As-printed – *Bandsaw cut surface*
4th set – Renishaw AM250 As-printed – *Ground with 600 grit sand paper*
5th set – SLM280 As-printed – *Wire EDM cut surface*
6th set – Concept Laser M2 As-printed – *Bandsaw cut surface*
7th set – Wrought 316L – *Ground with 600 grit sand paper*



 Courtesy of Danyil Kovalov (UVA)

ASTM G85-A2: Sample ID

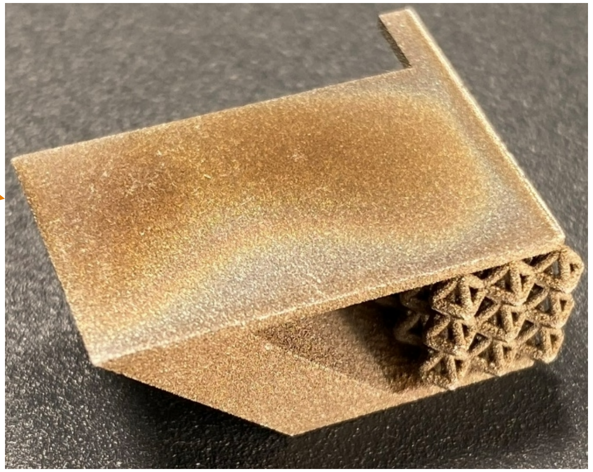


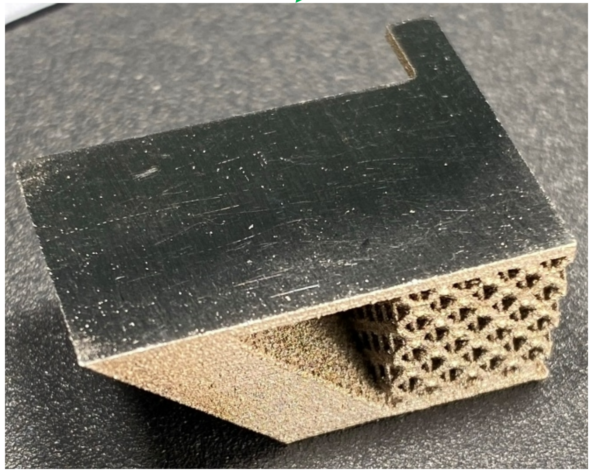
As-cut Bandsaw

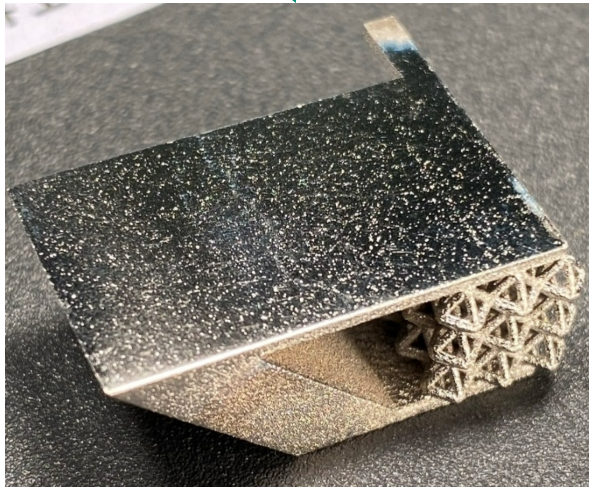
As-cut wire EDM

Electropolishing

Planar grinding





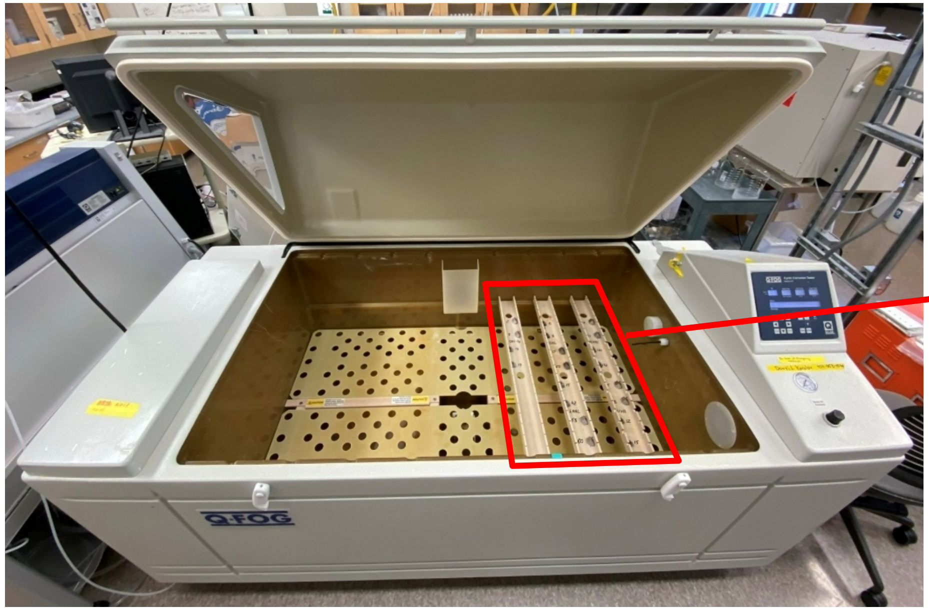


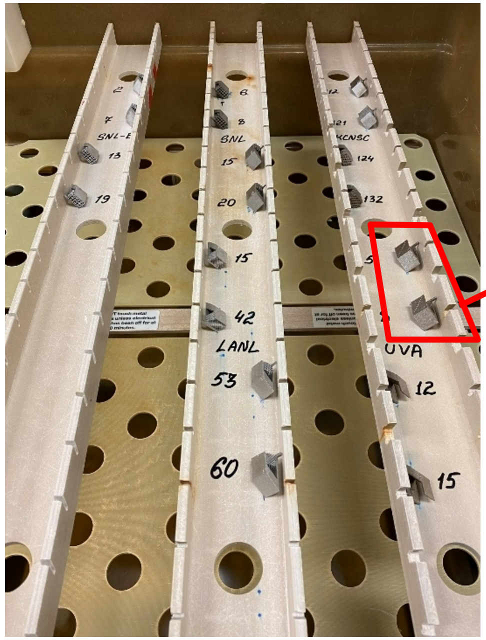
(UUR)

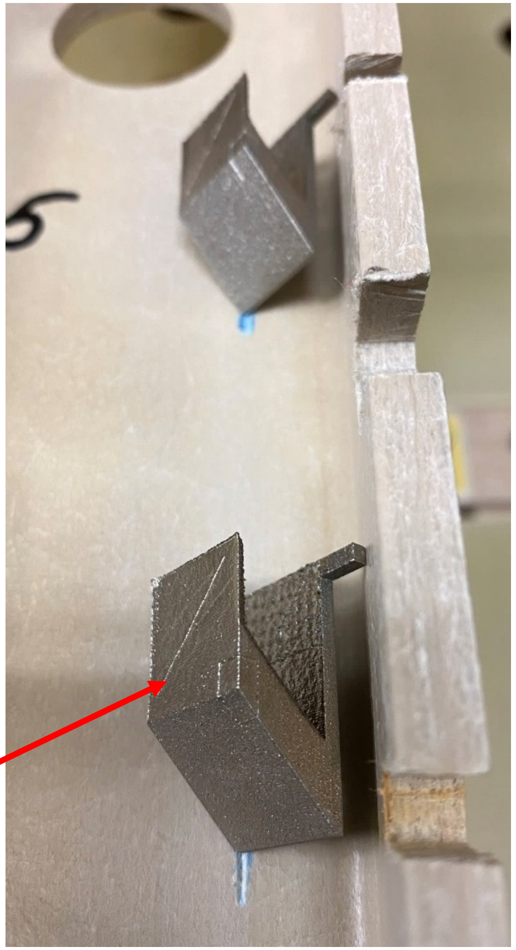
Courtesy of Danyil Kovalov (UVA)

ASTM G85-A2: Setup

- **ASTM for sample testing: G85 A2 with wet bottom**
- Equipment: Q-fog cyclic corrosion tester.
- Solution: 0.9M NaCl acidified to pH 3 with acetic acid.
- Test temperature: 49^{o}C.
- Duration: 2000 hr.
- Sample holder: PEEK rack with milled paths to place AM 316 samples.

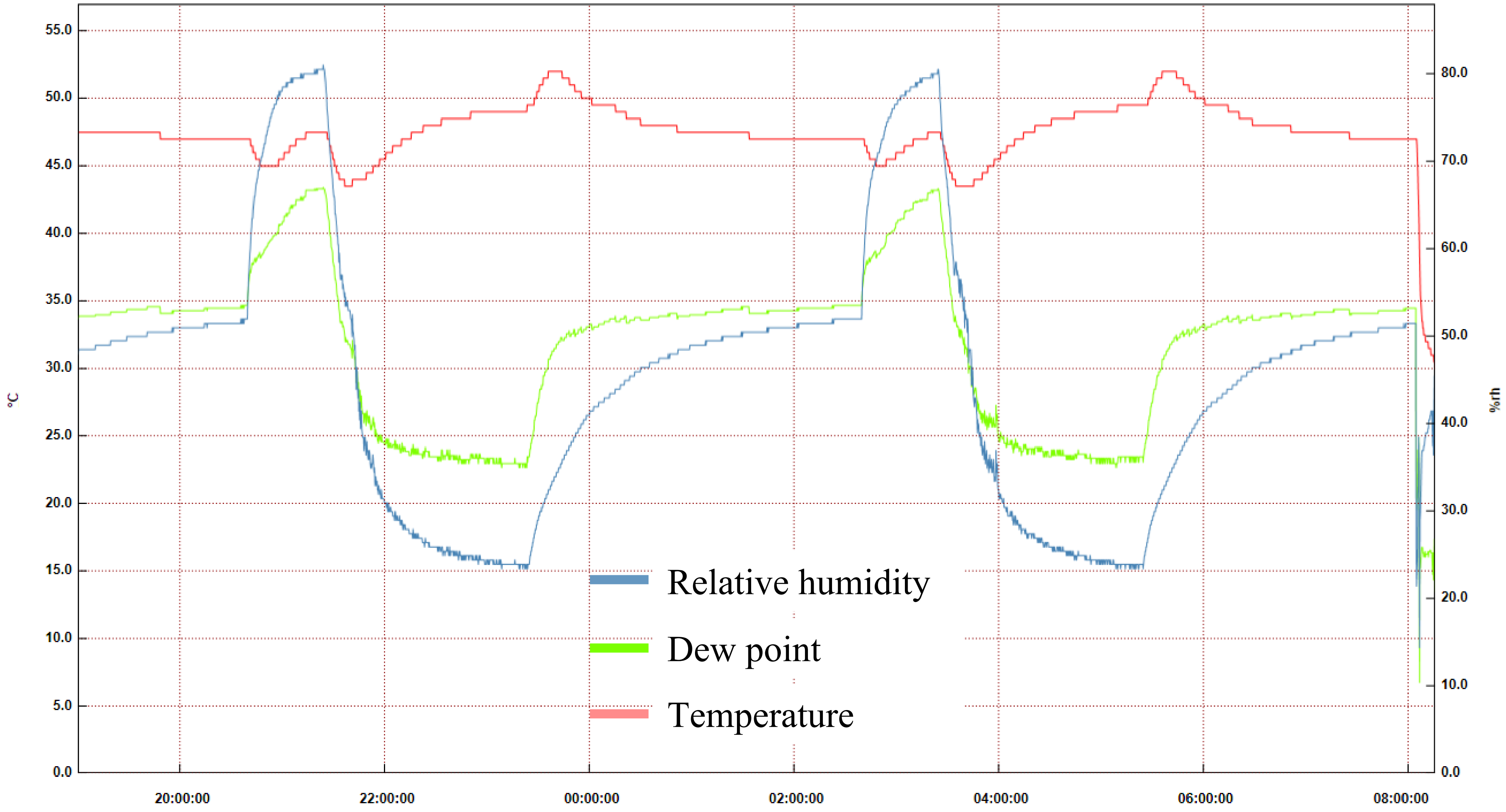






(UUR) Courtesy of Danyil Kovalov (UVA)

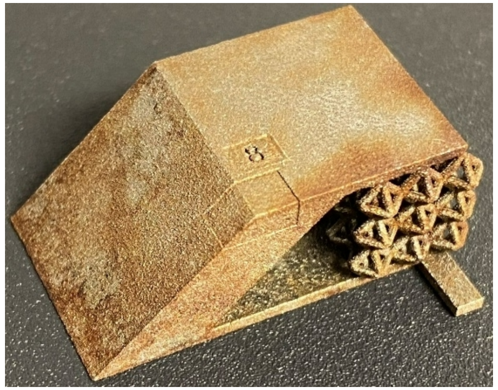
ASTM G85-A2: RH and Temp.



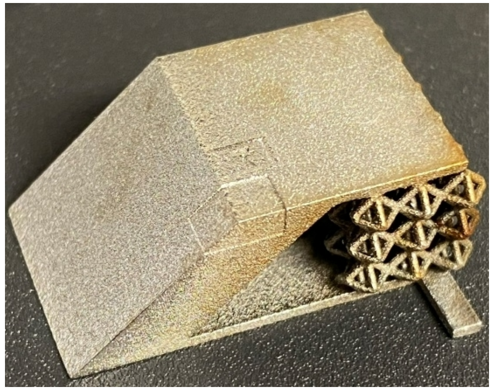
(UUR) Courtesy of Danyil Kovalov (UVA)

Samples after 2000 hours – Top down

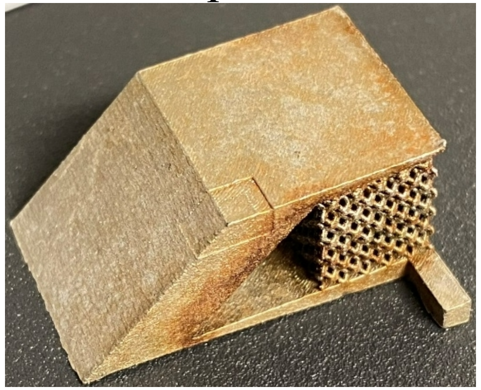
ProX 200
As-printed



ProX 200
Electropolish

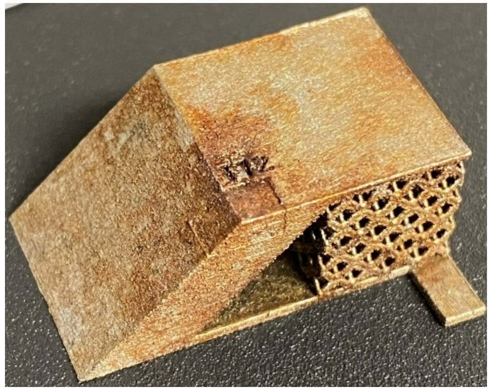


EOS M290
As-printed

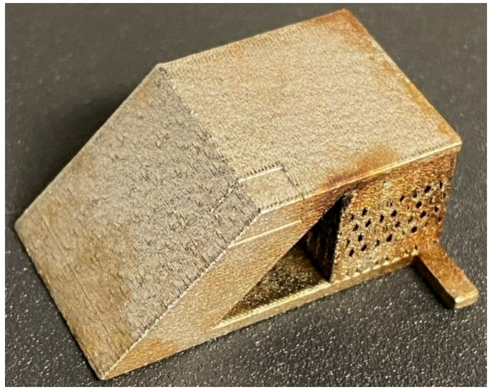


Wrought surface
was polished

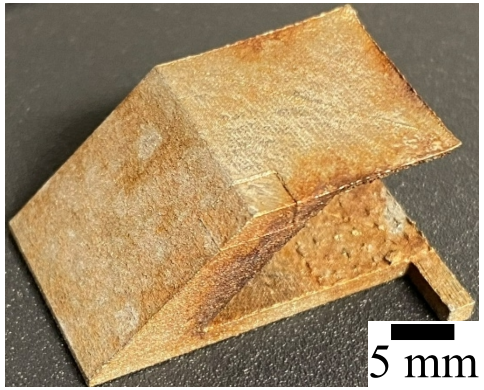
Renishaw
As-printed



Concept M2
As-printed



SLM®280
As-printed

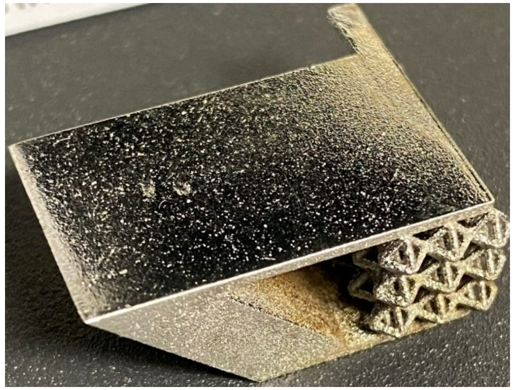


(UUR) Courtesy of Danyil Kovalov (UVA)

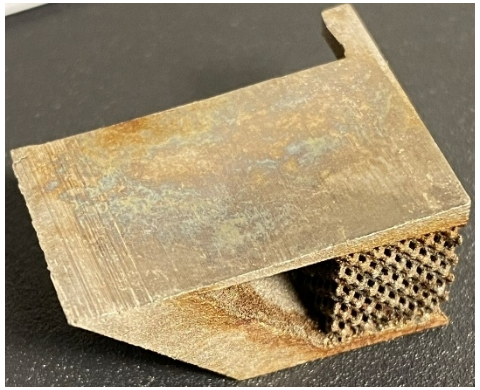
Samples after 2000 hours – Cut surface

ProX 200
As-cut wire EDM

ProX 200
Electropolish

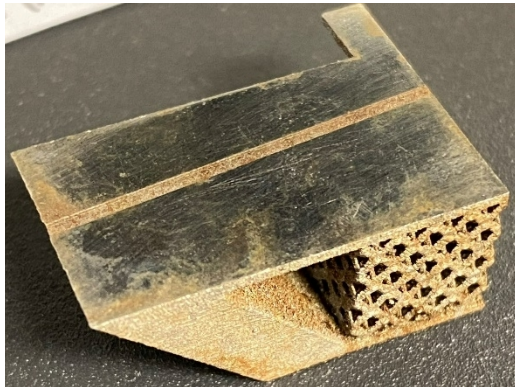


EOS M290
As-cut bandsaw

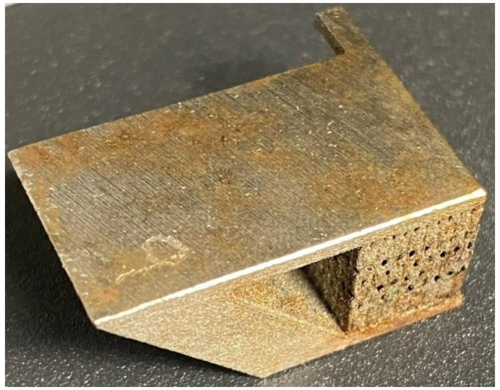


Wrought
Ground to 600 grit

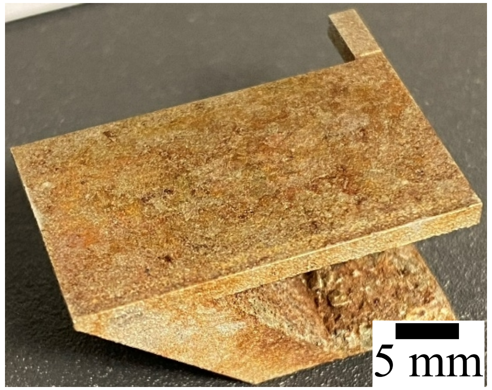
Renishaw
Ground to 600 grit



Concept M2
As-cut bandsaw



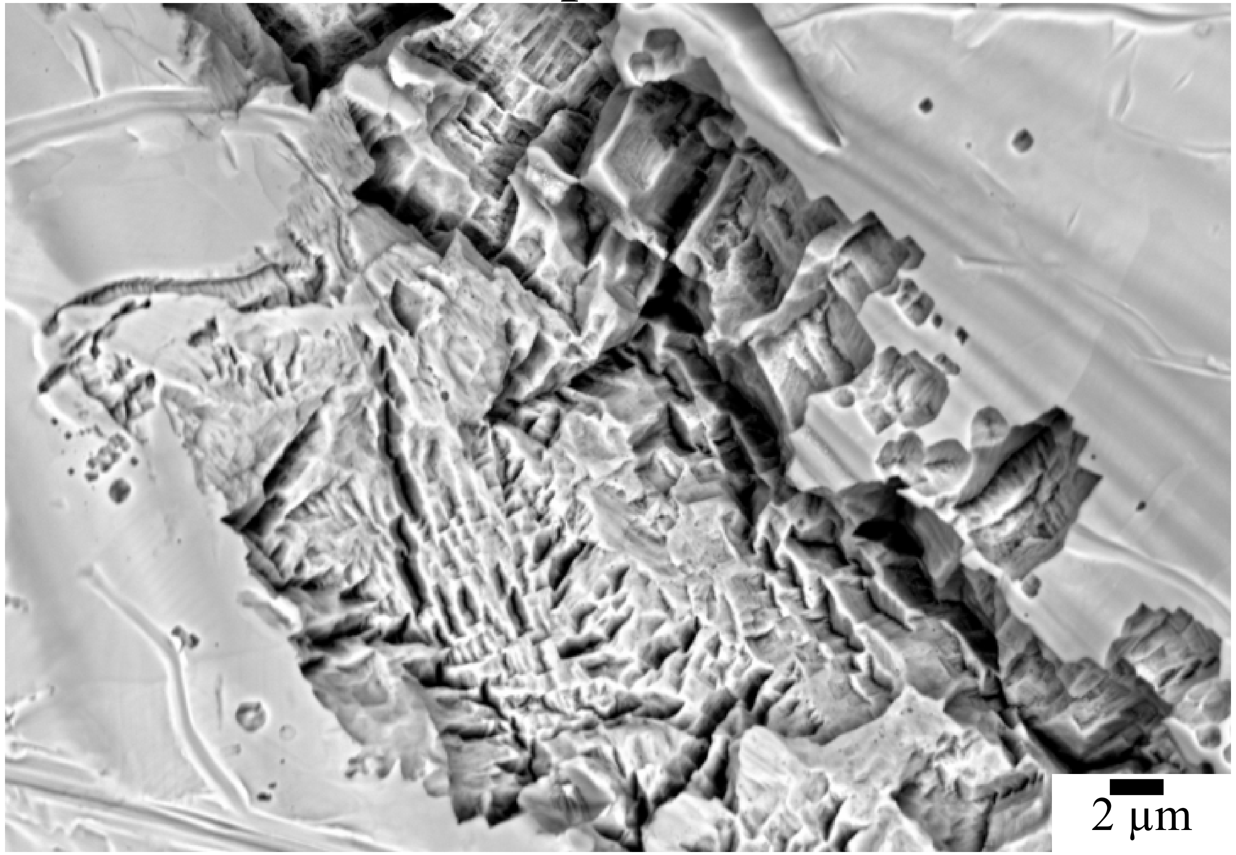
SLM®280
As-cut wire EDM



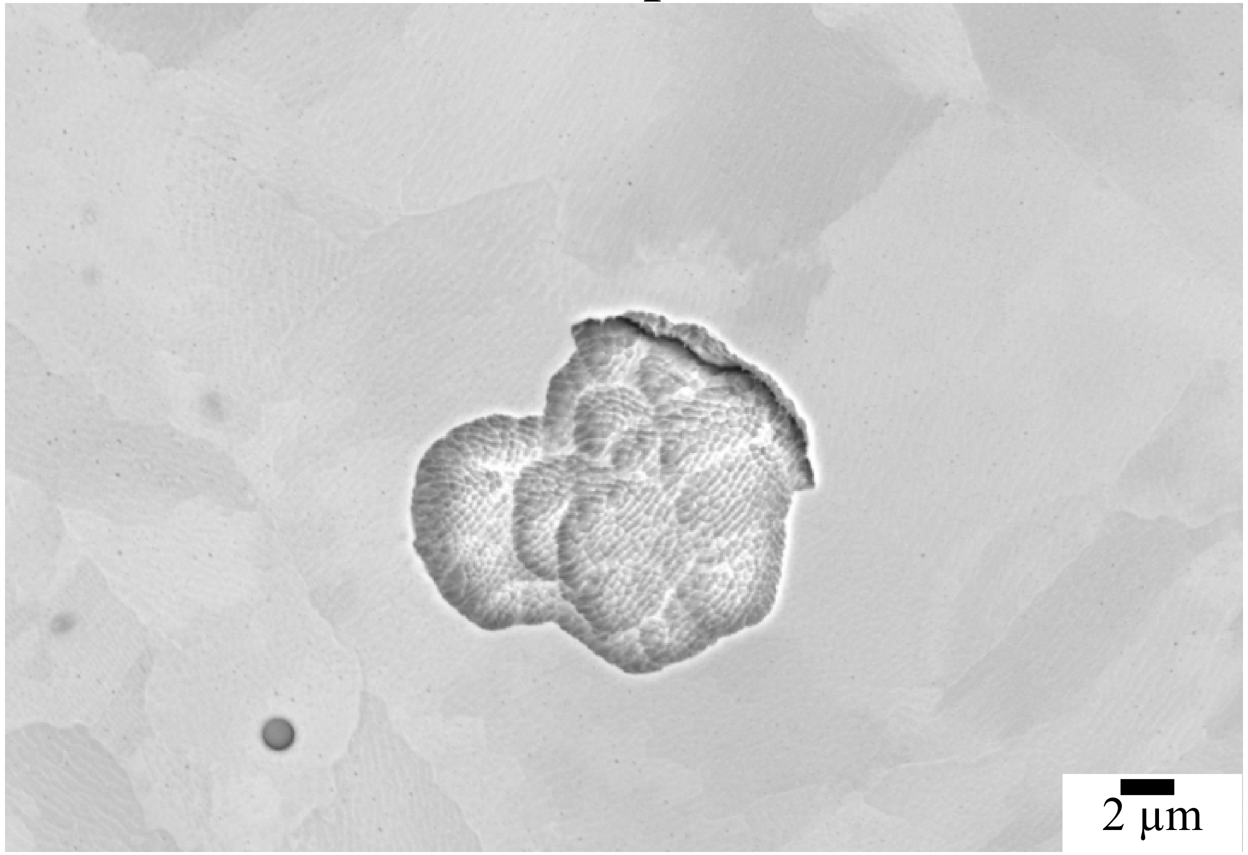
(UUR) Courtesy of Danyil Kovalov (UVA)

Pit morphology – Top surface

ProX 200
As-printed



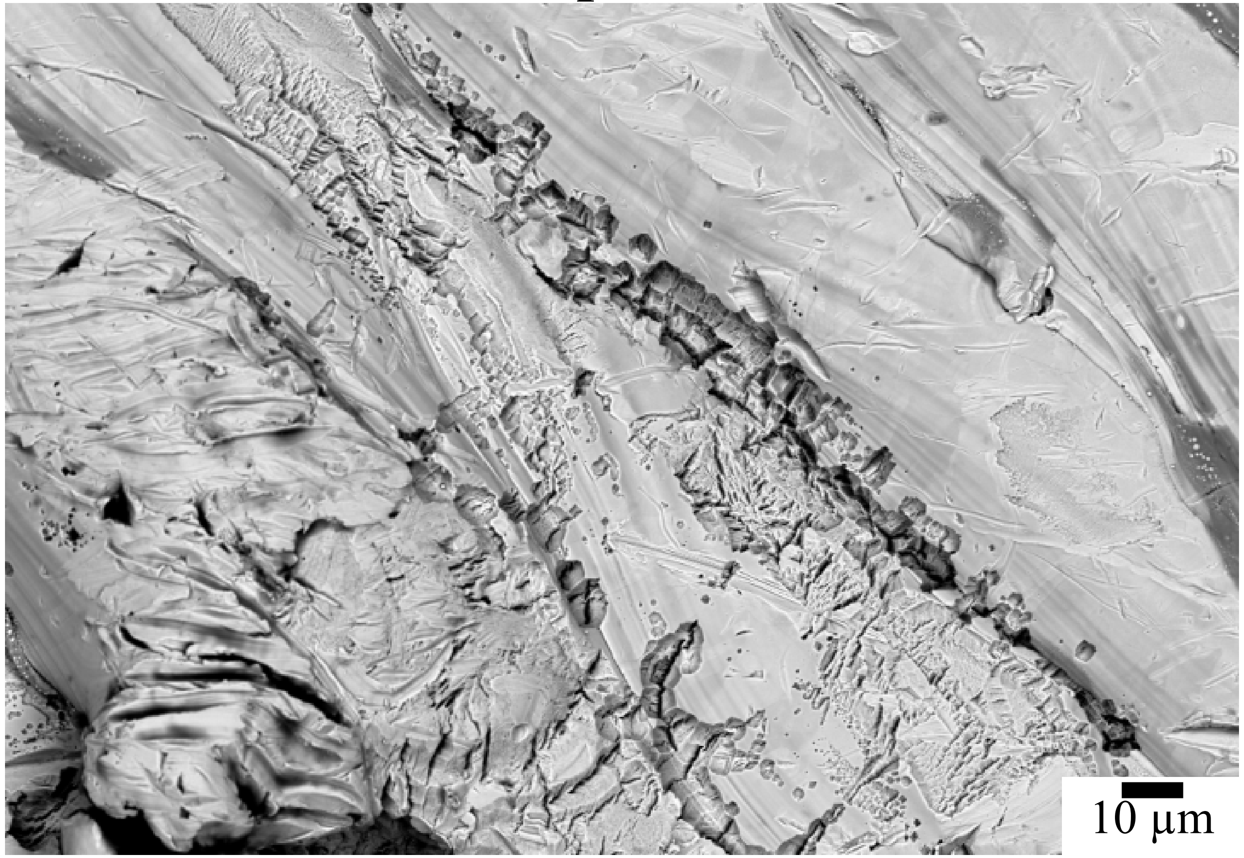
ProX 200
Electropolish



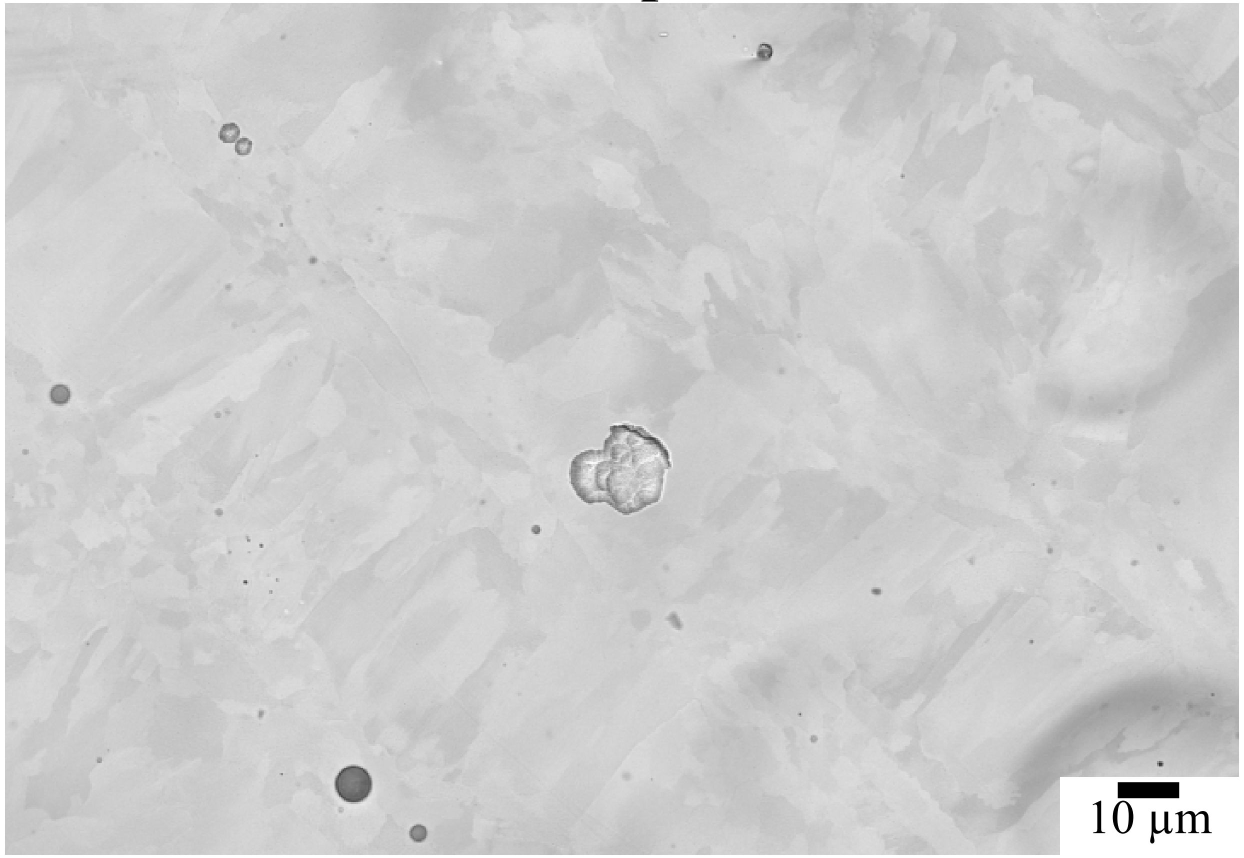
(UUR) Courtesy of Luis Jauregui (SNL)

Pit morphology – Top surface

ProX 200
As-printed



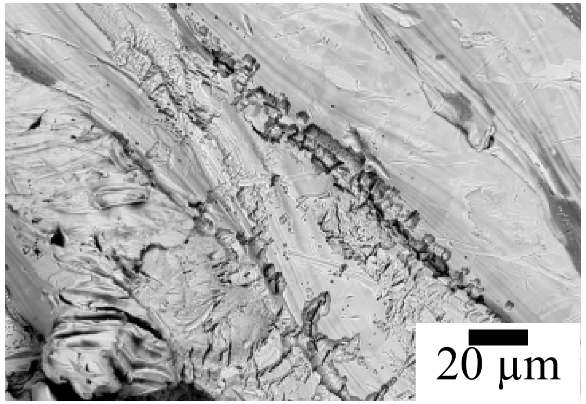
ProX 200
Electropolish



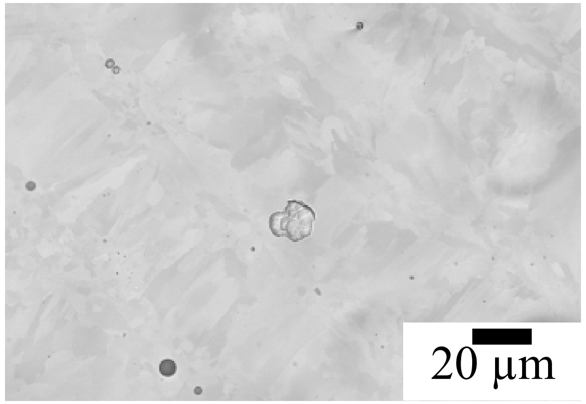
(UUR) Courtesy of Luis Jauregui (SNL)

Pit morphology – Top surface

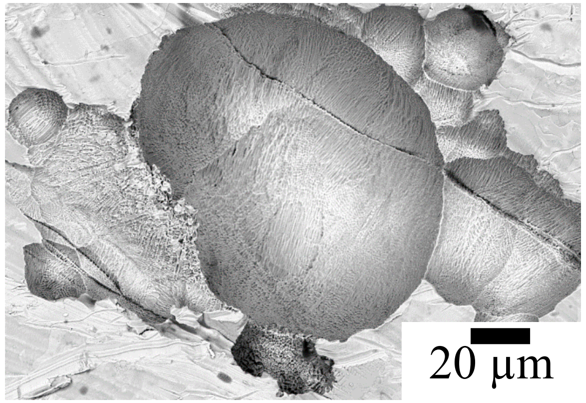
ProX 200
As-printed



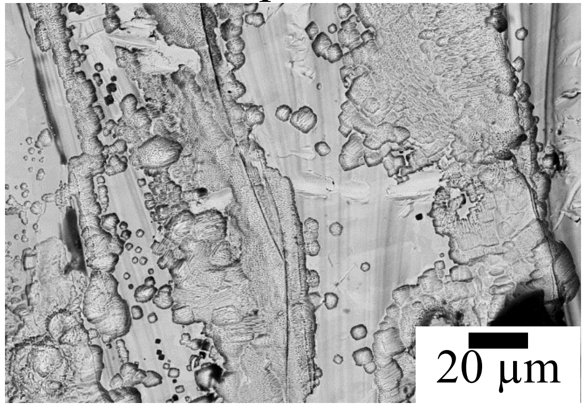
ProX 200
Electropolish



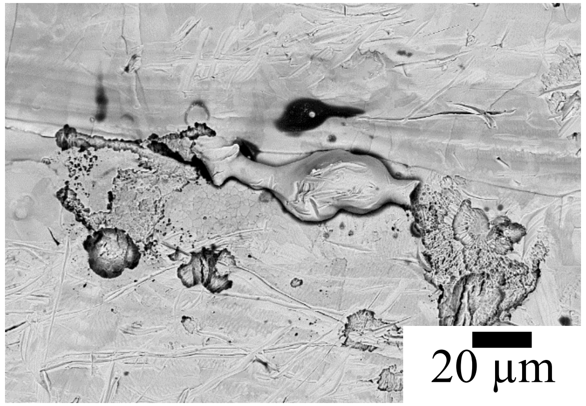
EOS M290
As-printed



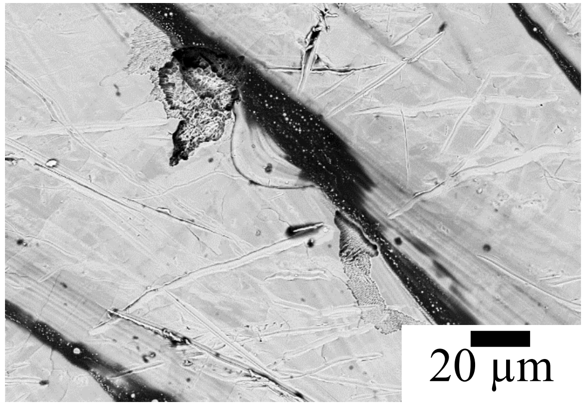
Renishaw
As-printed



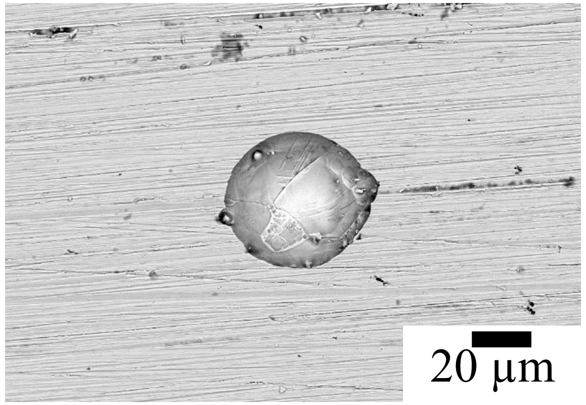
Concept M2
As-printed



SLM®280
As-printed



Wrought surface
was polished



(UUR) Courtesy of Luis Jauregui (SNL)

ASTM G85-A2 conclusions (so far)

- Wire EDM as-cut surface had the most aggressive corrosion based on optical analysis.
- Electropolished surfaces were least susceptible to corrosion.
- As-printed samples had different quantities of corrosion based on optical, but similar morphology (many pits appeared to initiate from melt pool boundaries).

Current results correlate well with electrochemical experiments (breakdown potential, etc.).

Thank you for your attention!

Questions…?

Please contact with questions/comments:

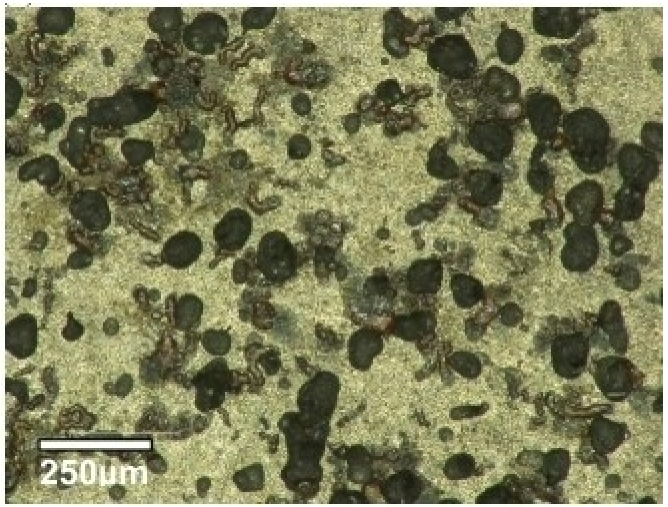
Michael Melia

mamelia@sandia.gov

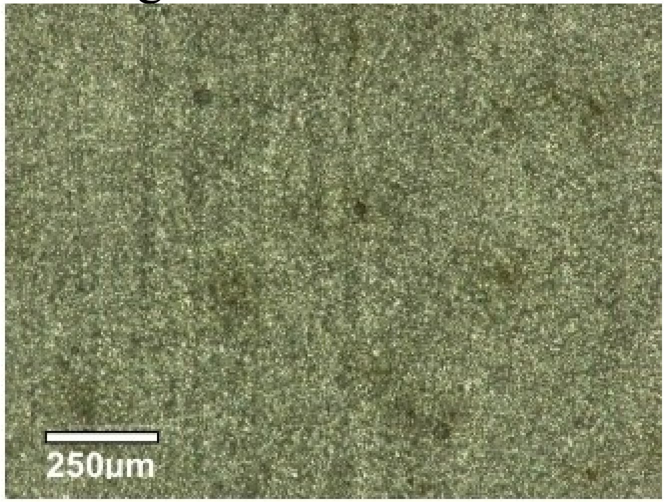
Background – AM 4340 exposure to ASTM-B117

Images after electrochemical tests

AM4340

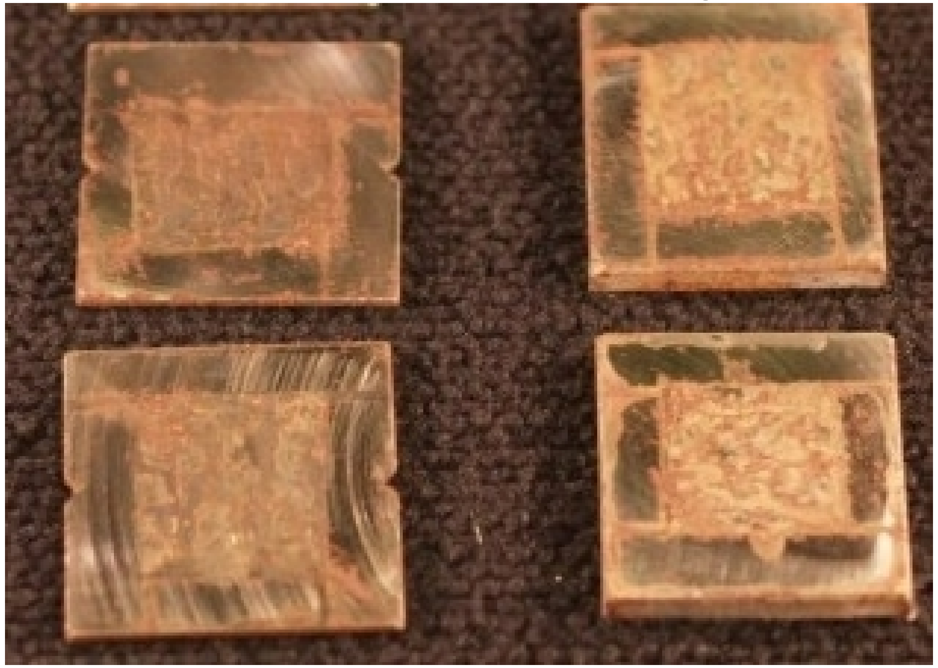


Wrought 4340



Images after 48 hrs of ASTM B117 exposures

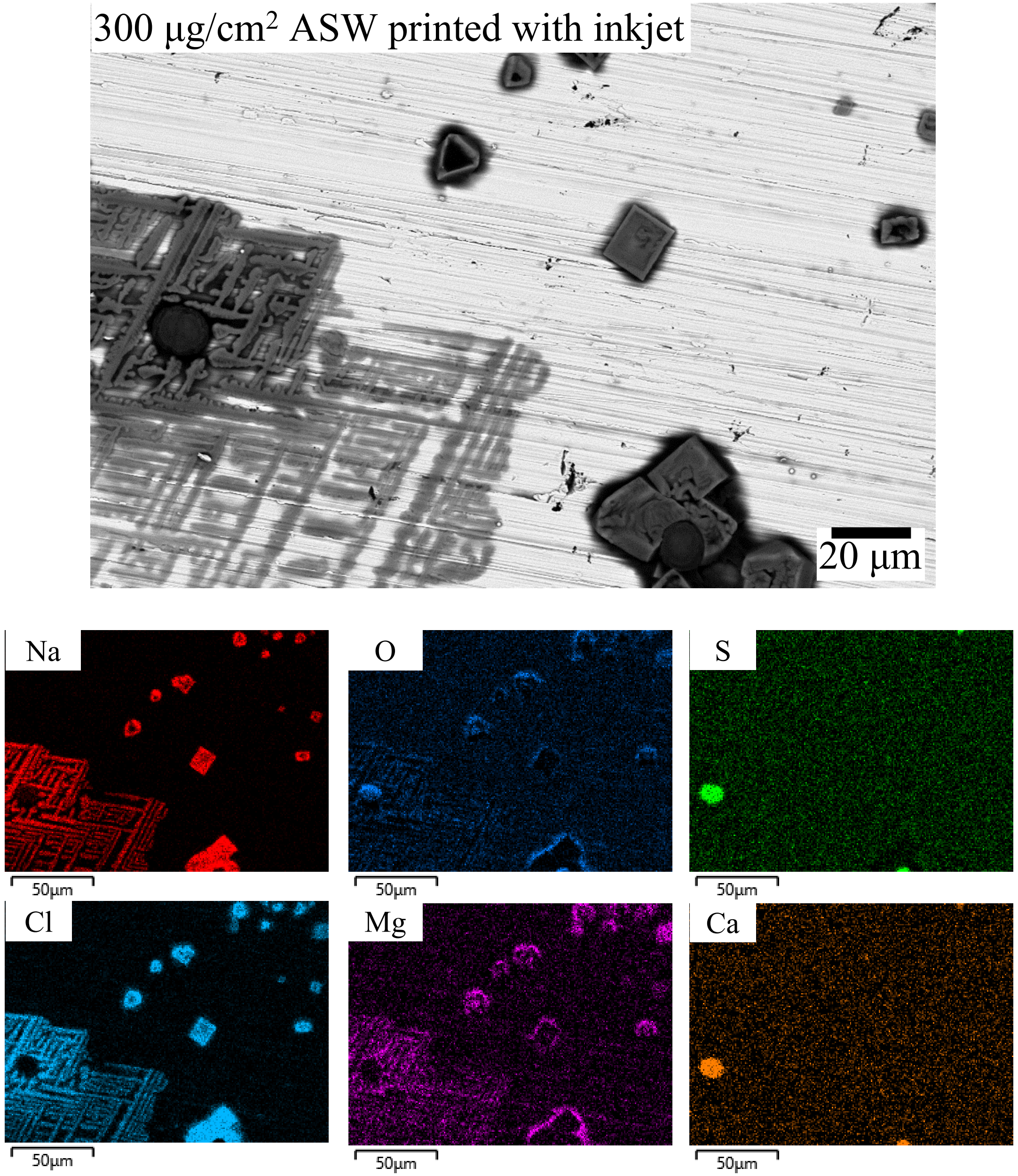
AM4340 Wrought 4340

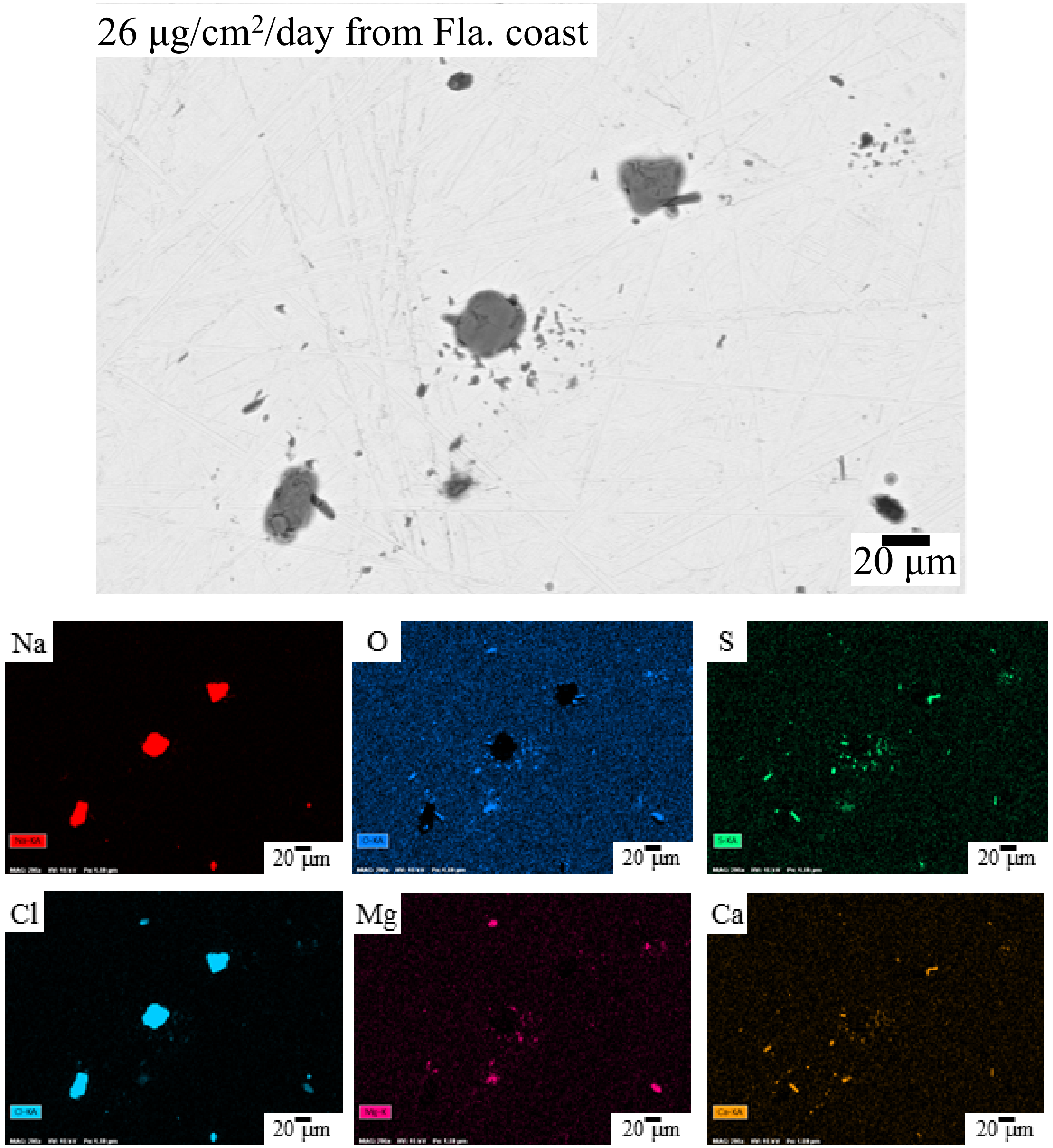


- No noticeable difference in electrochemical response, but surface corrosion morphology was dramatically different.
- Initial ASTM B117 results suggest minimal difference (only analysis was with optical imaging).

Source: Schmidt, MS&T Proceedings, 2015.

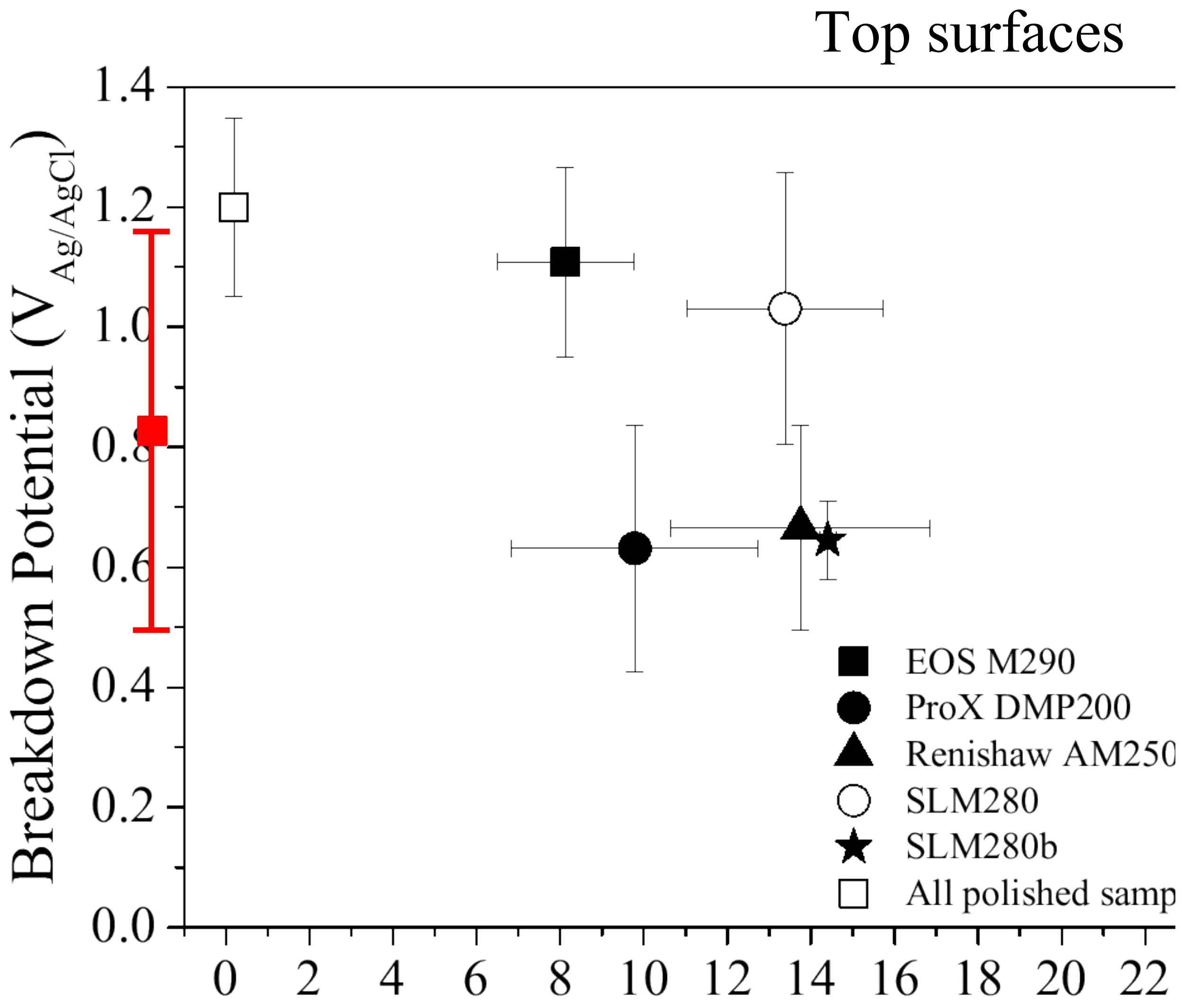
Salt deposited on samples





(UUR)

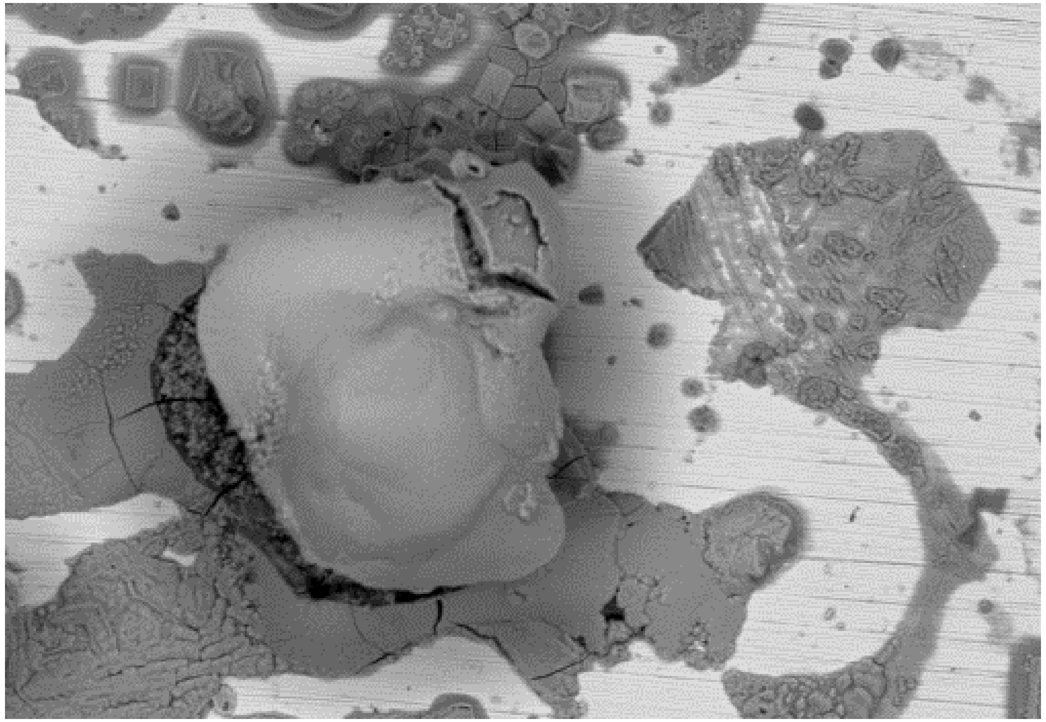
Summary of data from all sites



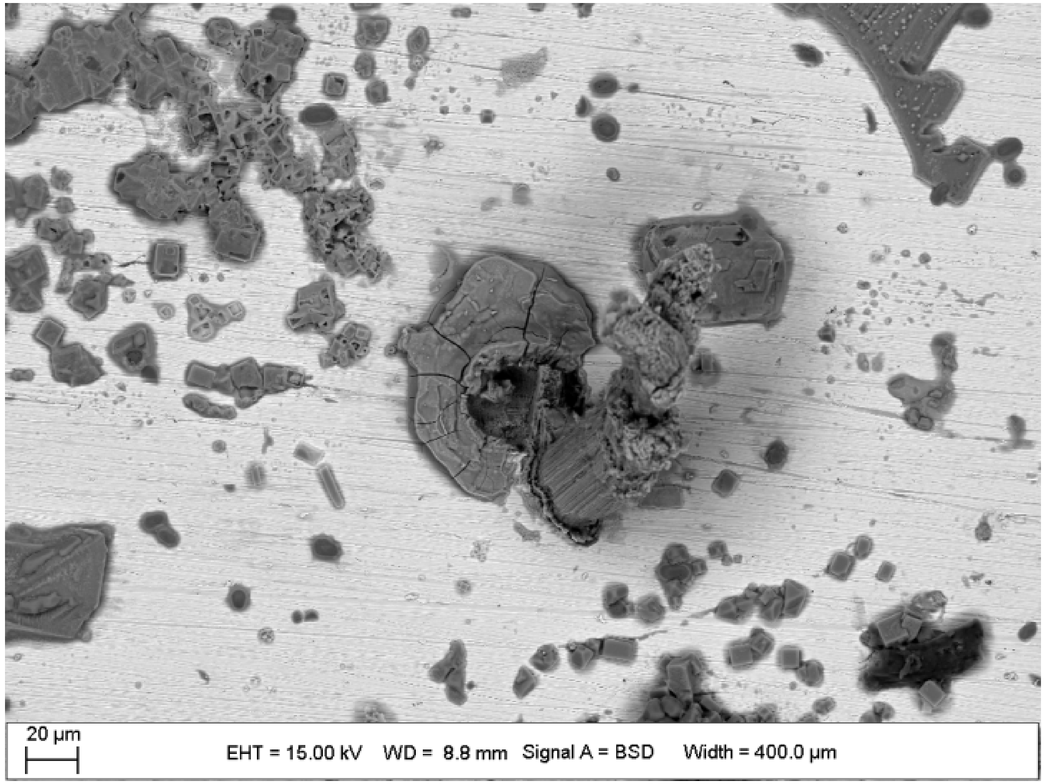
(UUR) Courtesy of Courtney Clark (LANL)

SEM images of **316L** specimen with **300 µg/cm² ASW at 40% RH**

Wrought



AM

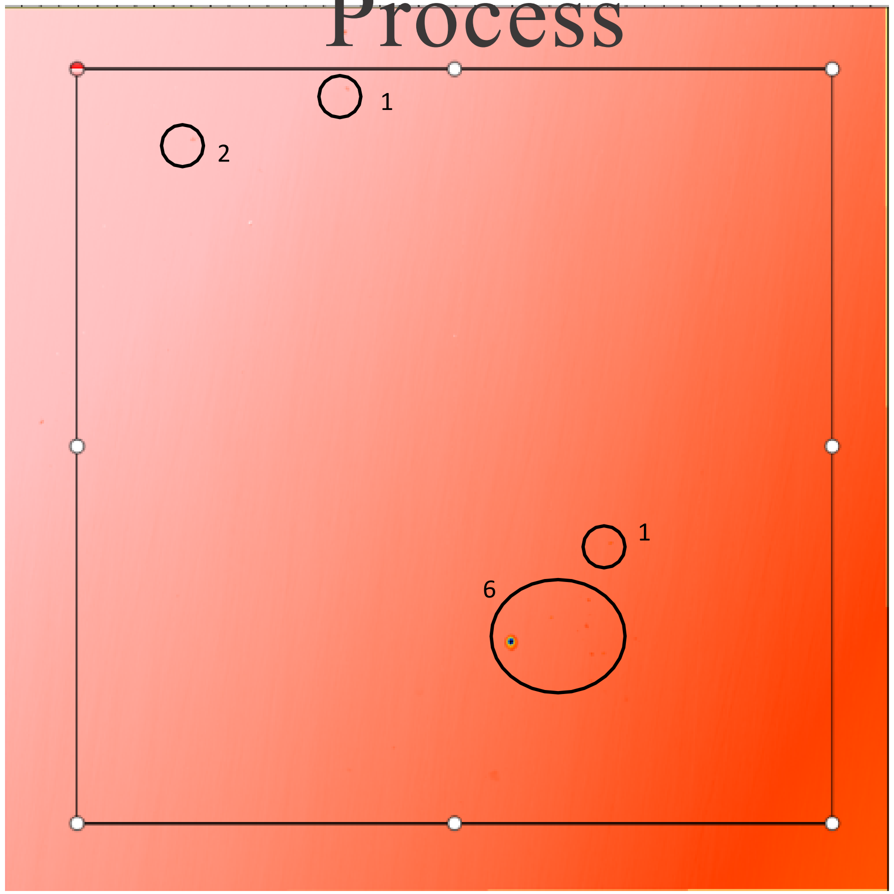


Pit Verification Process

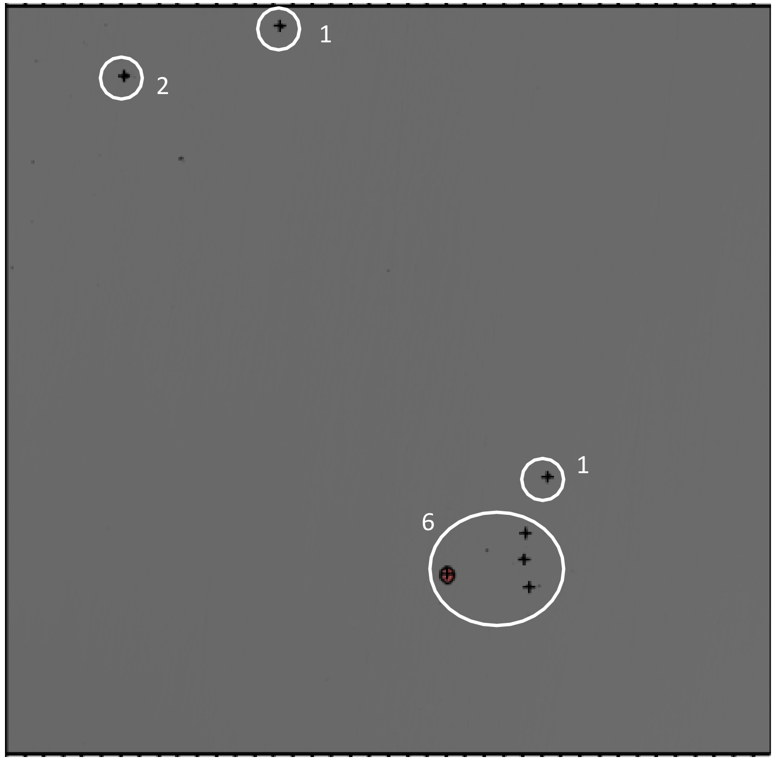
316L_W_35C_40RH_300ug ASW_Perp_1wk-Sample51

Mid Mid

Raw Data



Processed Data



The process for pit verification begins by filtering data taken from a Particle Analysis in MountainsMap. The filtering criteria depends on the Maximum Height (Depth) of a pit exceeding 0.2 μm and the Volume/Area ratio exceeding 0.2 μm.

Once the data is filtered, the remaining particles are verified by comparing the raw data to the processed data. Through visual inspection, the particles are verified to be pits.

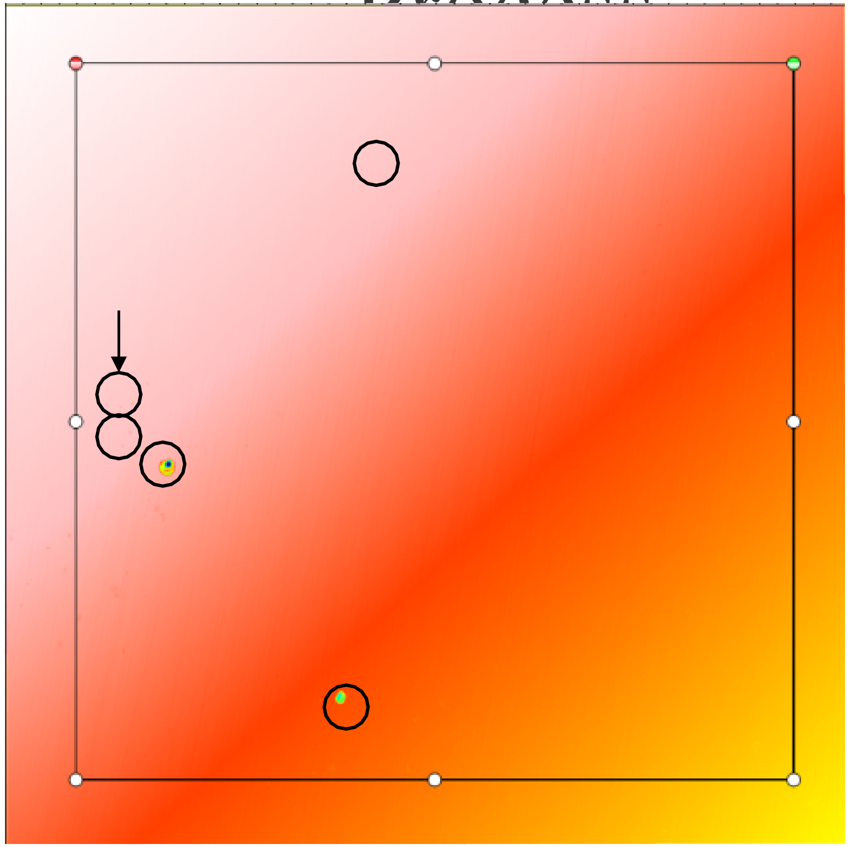
The particles that were considered pits through the filtering process are outlined in the above images. The numbers next to the circles indicate the amount of pits located within the circle. For example, the largest circle contains 6 pits. This results in a total of 10 pits.

Pit Verification Process

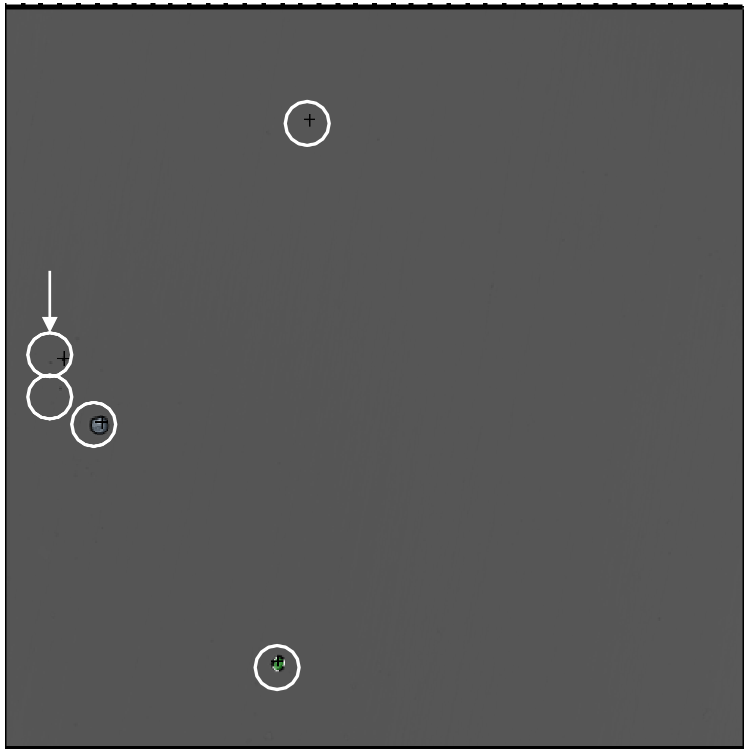
316L_AM_35C_40RH_300 ASW_Perp_1wk-Sample116

Mid Mid

Raw Data



Processed Data



The process for pit verification begins by filtering data taken from a Particle Analysis in MountainsMap. The filtering criteria depends on the Maximum Height (Depth) of a pit exceeding 0.2 µm and the Volume/Area ratio exceeding 0.2 µm.

Once the data is filtered, the remaining particles are verified by comparing the raw data to the processed data. Through visual inspection, the particles are verified to be pits.

The particles that were considered pits through the filtering process are outlined in the above images. The circle with the arrow pointing to it contains two pits, all others contain 1 pit. This created a total of 6 pits.

Pit statistics of **316L specimen - 300 μg/cm² ASW at 40% RH**

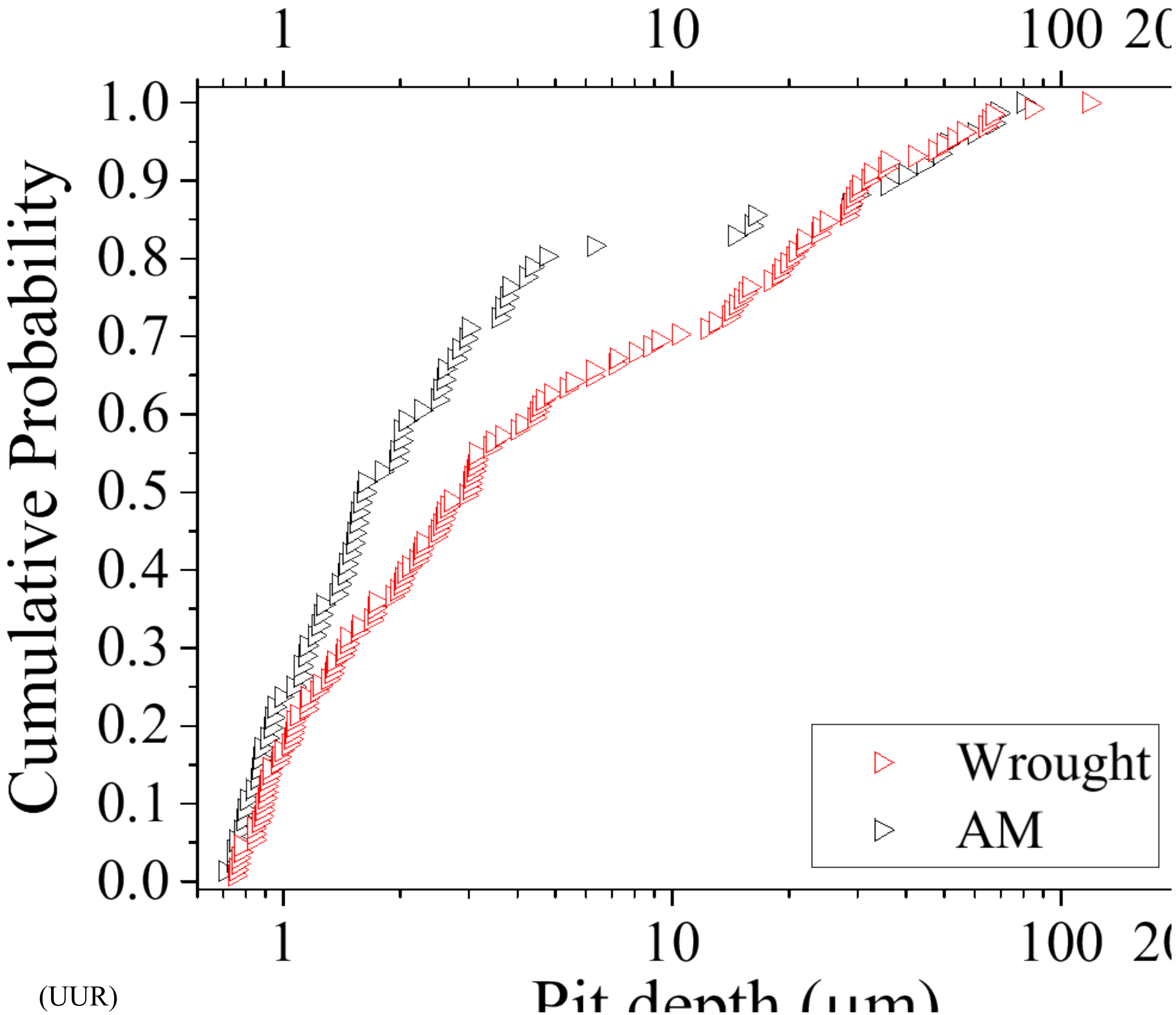
Wrought

	Average pit depth (μm)	Max pit depth (μm)
1 week	2.9	35.1
1 month	7.0	41.8
6 months	6.3	58.5
12 months	11.7	118.1

AM

	Average pit depth (μm)	Max pit depth (μm)
1 week	4.0	32.6
1 month	10.1	210.4 (73.3)
6 months	7.6	76.2
12 months	9.4	80.2

12 months – Pit depth



Pit statistics of **316L specimen - 300 µg/cm² ASW at 40% RH**

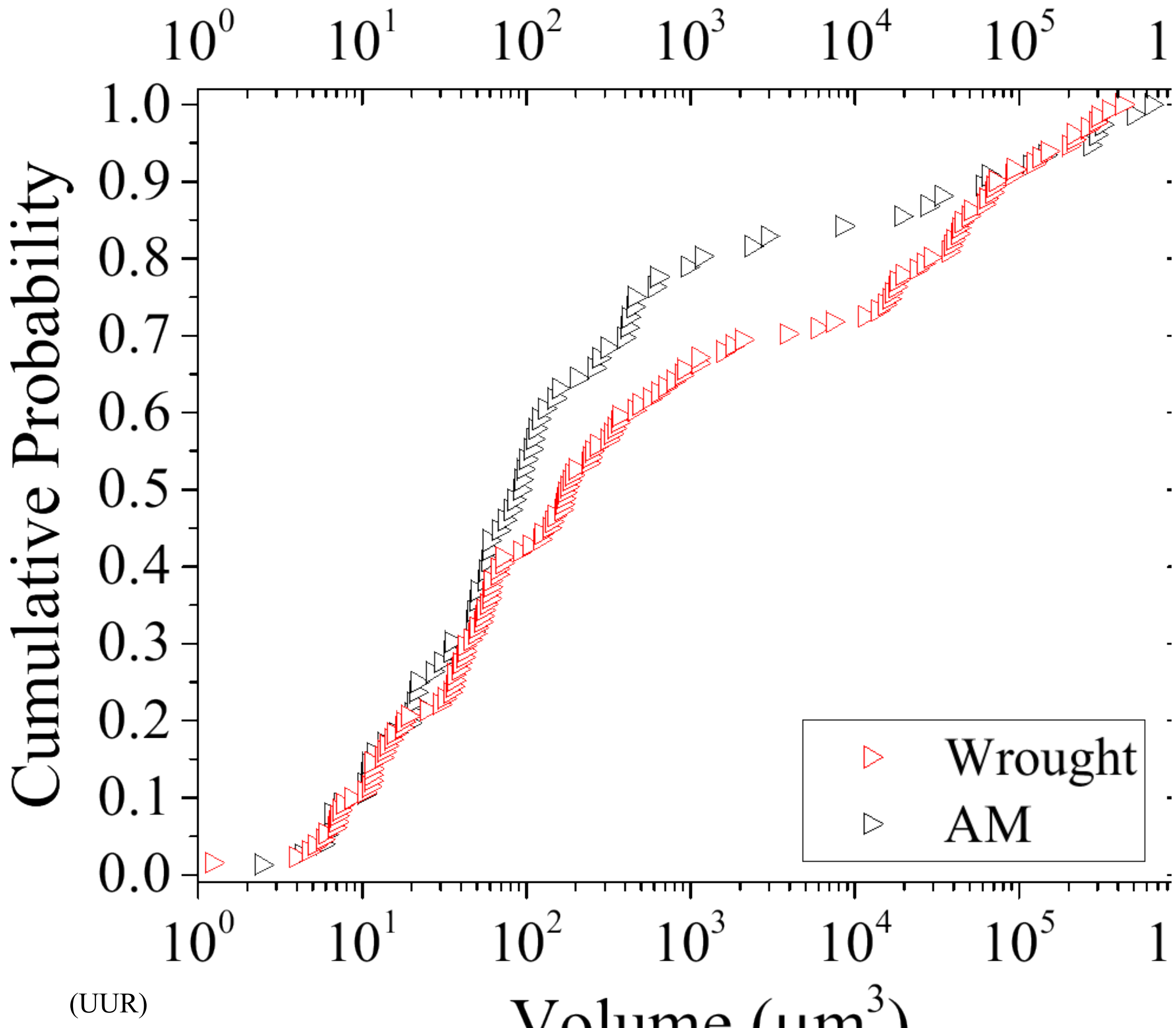
Wrought

	Volume per area ($\mu m^3/cm^2$)	# of Pits/cm^2
1 week	1.15×10^5	50
1 month	9.28×10^5	65
6 months	2.34×10^6	105
12 months	2.75×10^6	96

AM

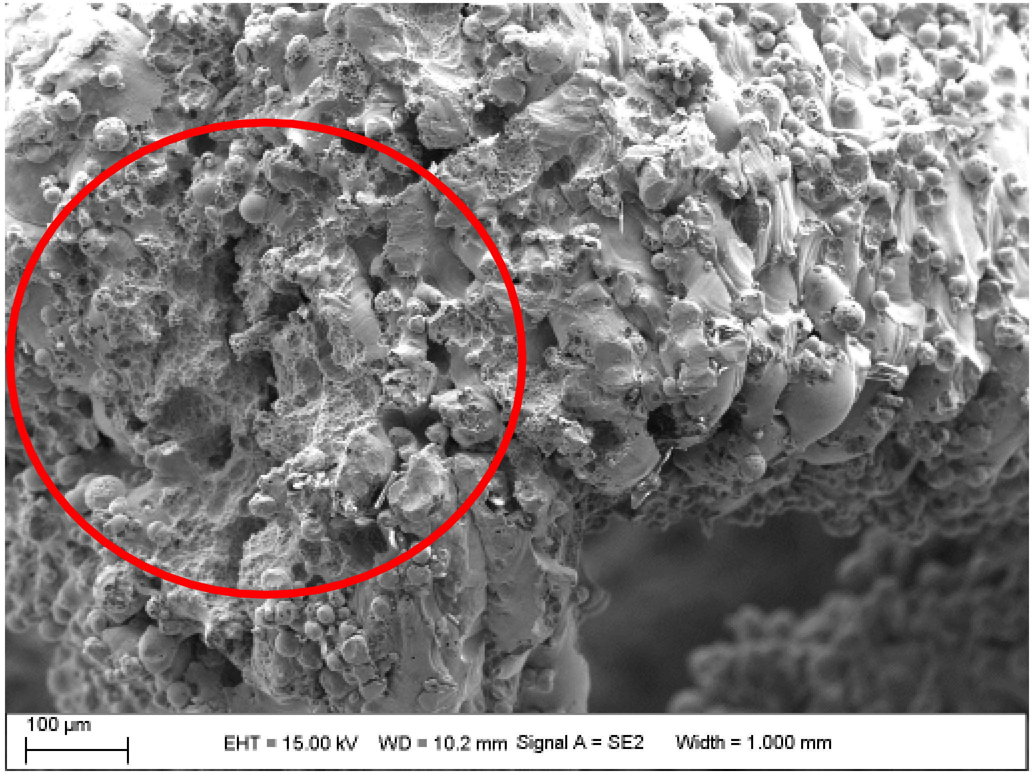
	Volume per area ($\mu m^3/cm^2$)	# of Pits/cm^2
1 week	3.66×10^5	89
1 month	9.35×10^5	88
6 months	2.97×10^6	155
12 months	1.74×10^6	53

12 months – Pit volume

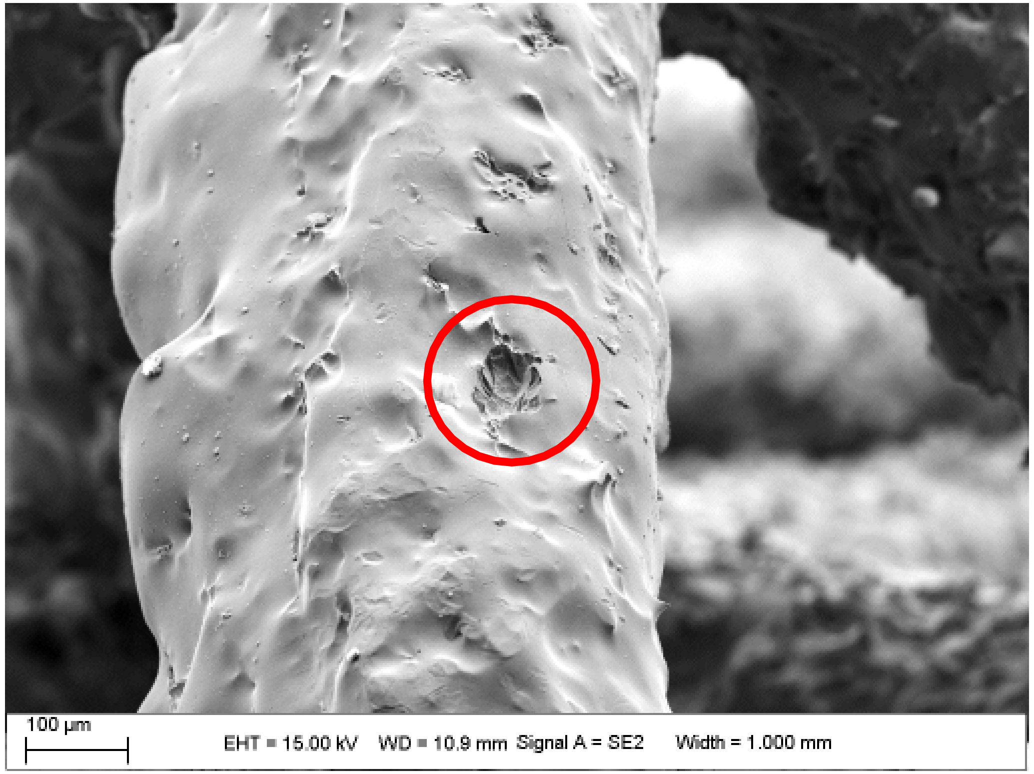


Pit morphology - Lattices

ProX 200
As-printed

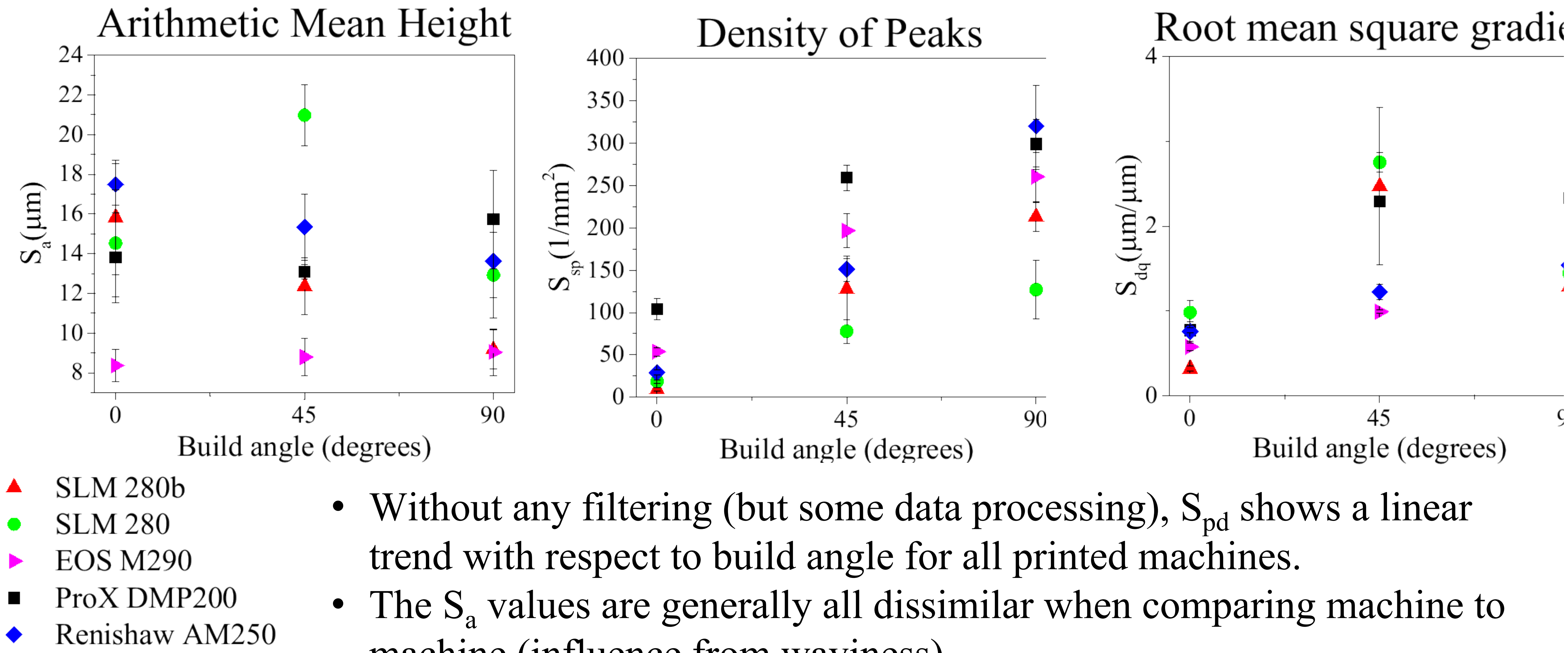


ProX 200
Electropolish



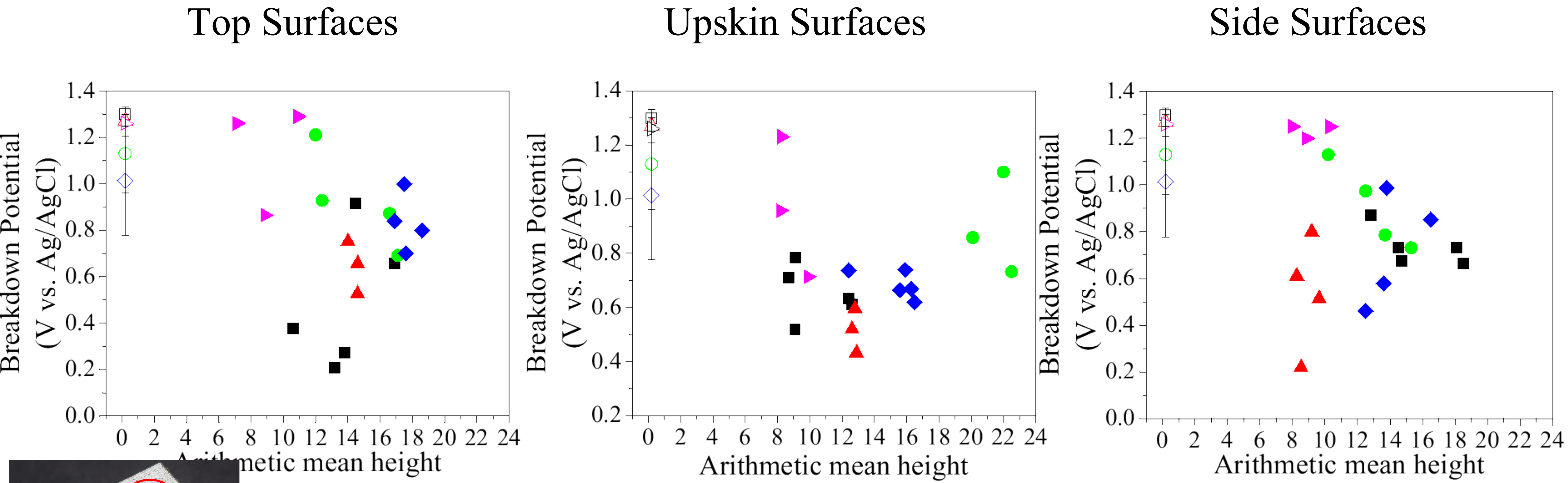
(UUR) Courtesy of Luis Jauregui (SNL)

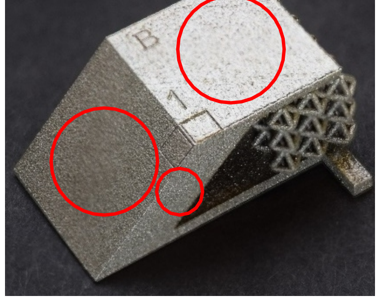
Zygo measurements from all material



- Without any filtering (but some data processing), S_{pd} shows a linear trend with respect to build angle for all printed machines.
- The S_a values are generally all dissimilar when comparing machine to machine (influence from waviness).

Summary of Breakdown potential (E_B)





Pit statistics of **316L specimen - 300 μg/cm² ASW at 40% RH**

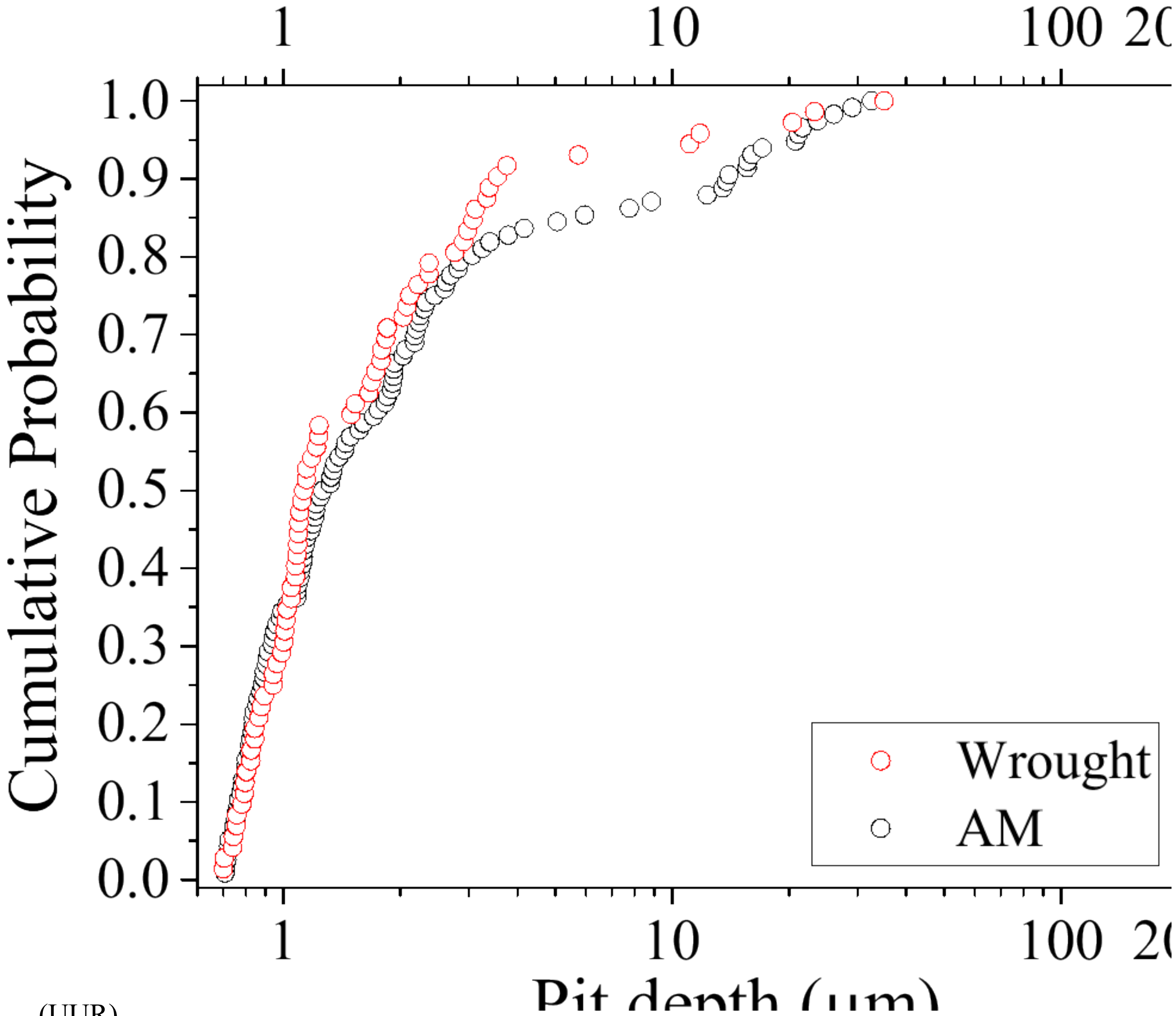
Wrought

	Average pit depth (μm)	Max pit depth (μm)
1 week	2.9	35.1
1 month	7.0	41.8
6 months	6.3	58.5
12 months	11.7	118.1

AM

	Average pit depth (μm)	Max pit depth (μm)
1 week	4.0	32.6
1 month	10.1	210.4 (73.3)
6 months	7.6	76.2
12 months	9.4	80.2

1 week – Pit depth



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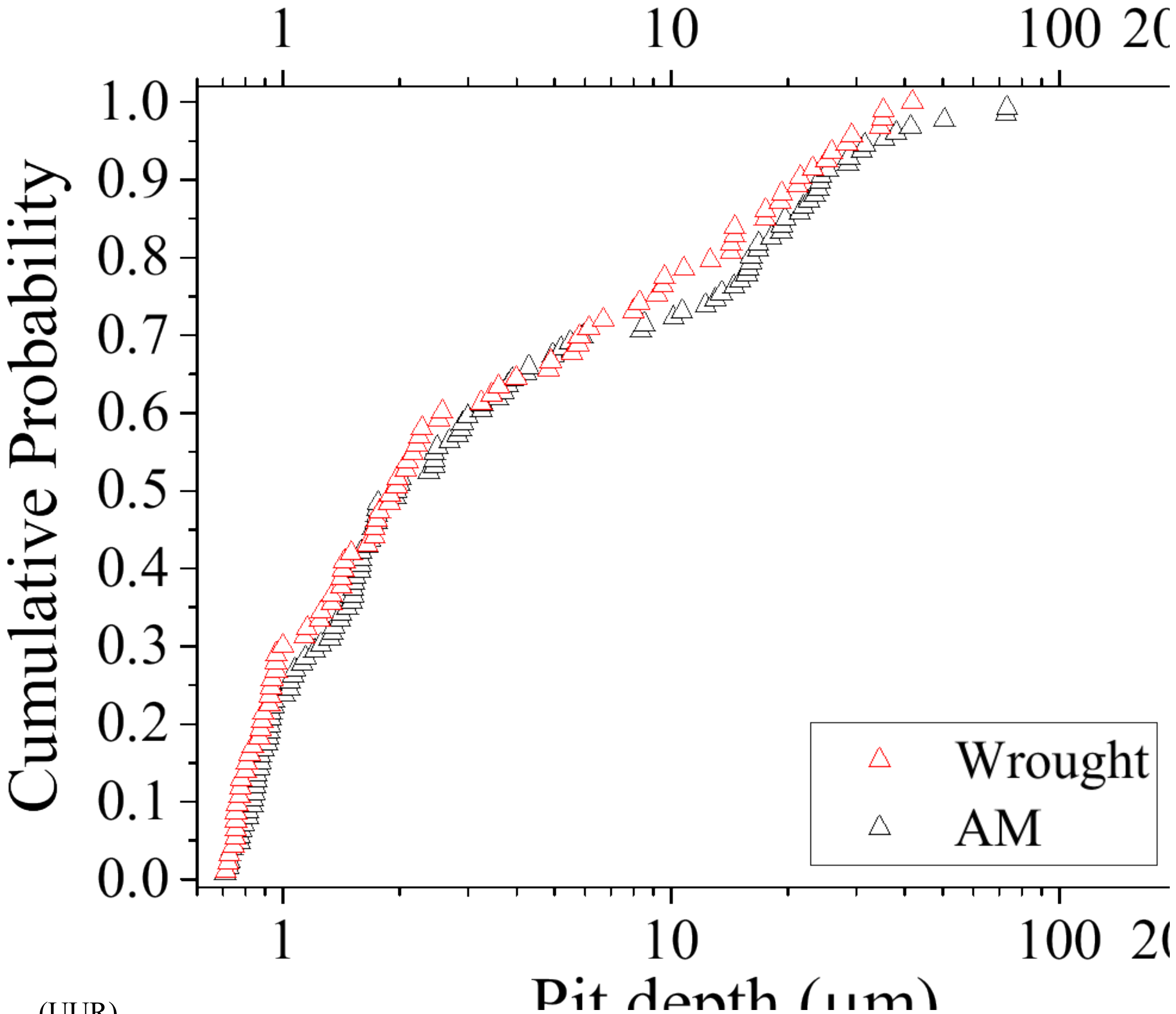


Pit statistics of **316L specimen - 300 µg/cm² ASW at 40% RH**

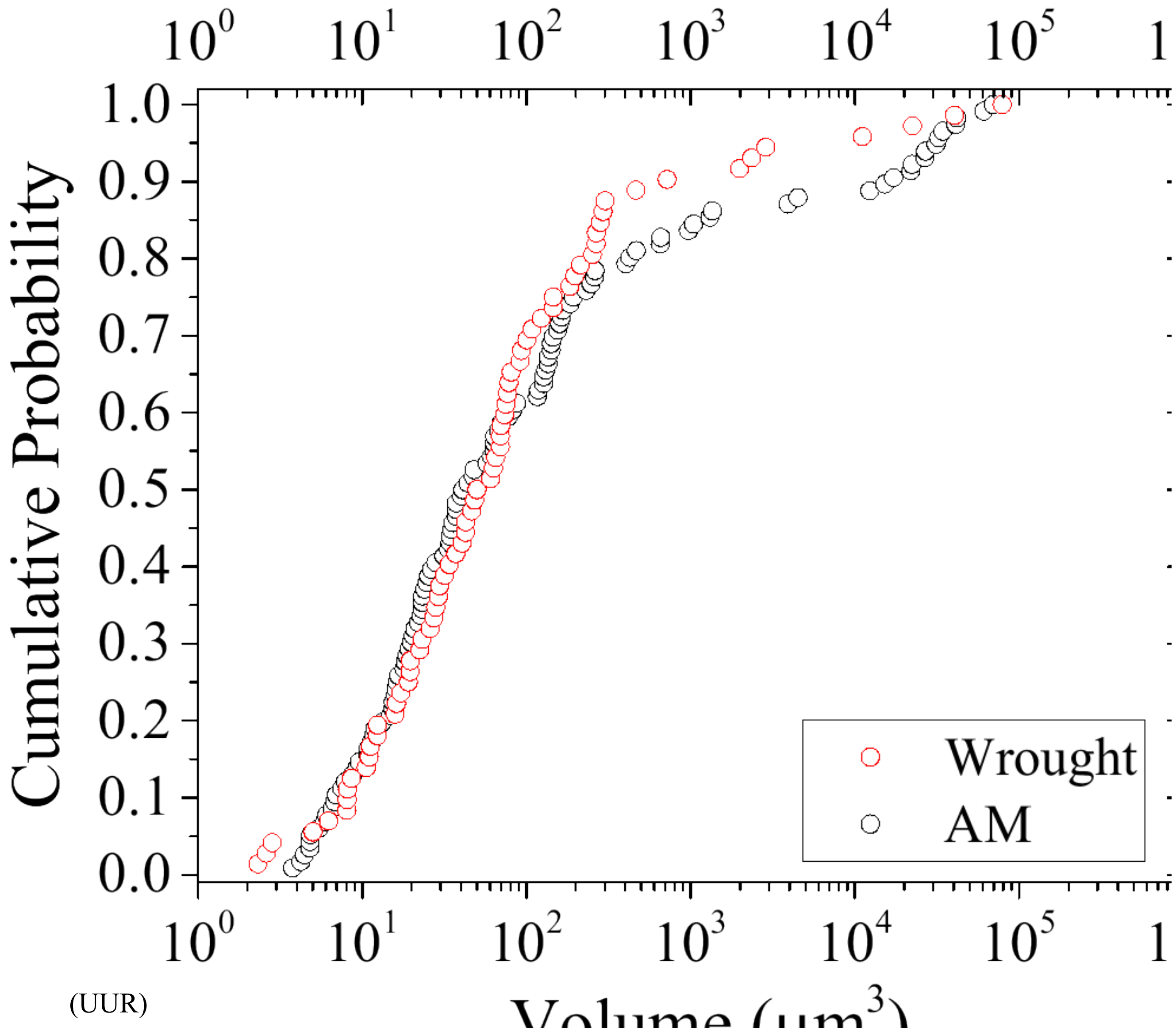
Wrought

	Volume per area (µm³/cm²)	# of Pits/cm²
1 week	1.15×10^5	50
1 month	9.28×10^5	65
6 months	2.34×10^6	105
12 months	2.75×10^6	96

AM

	Volume per area (µm³/cm²)	# of Pits/cm²
1 week	3.66×10^5	89
1 month	9.35×10^5	88
6 months	2.97×10^6	155
12 months	1.74×10^6	53

1 week – Pit volume



(UUR)

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1 month – Pit volume

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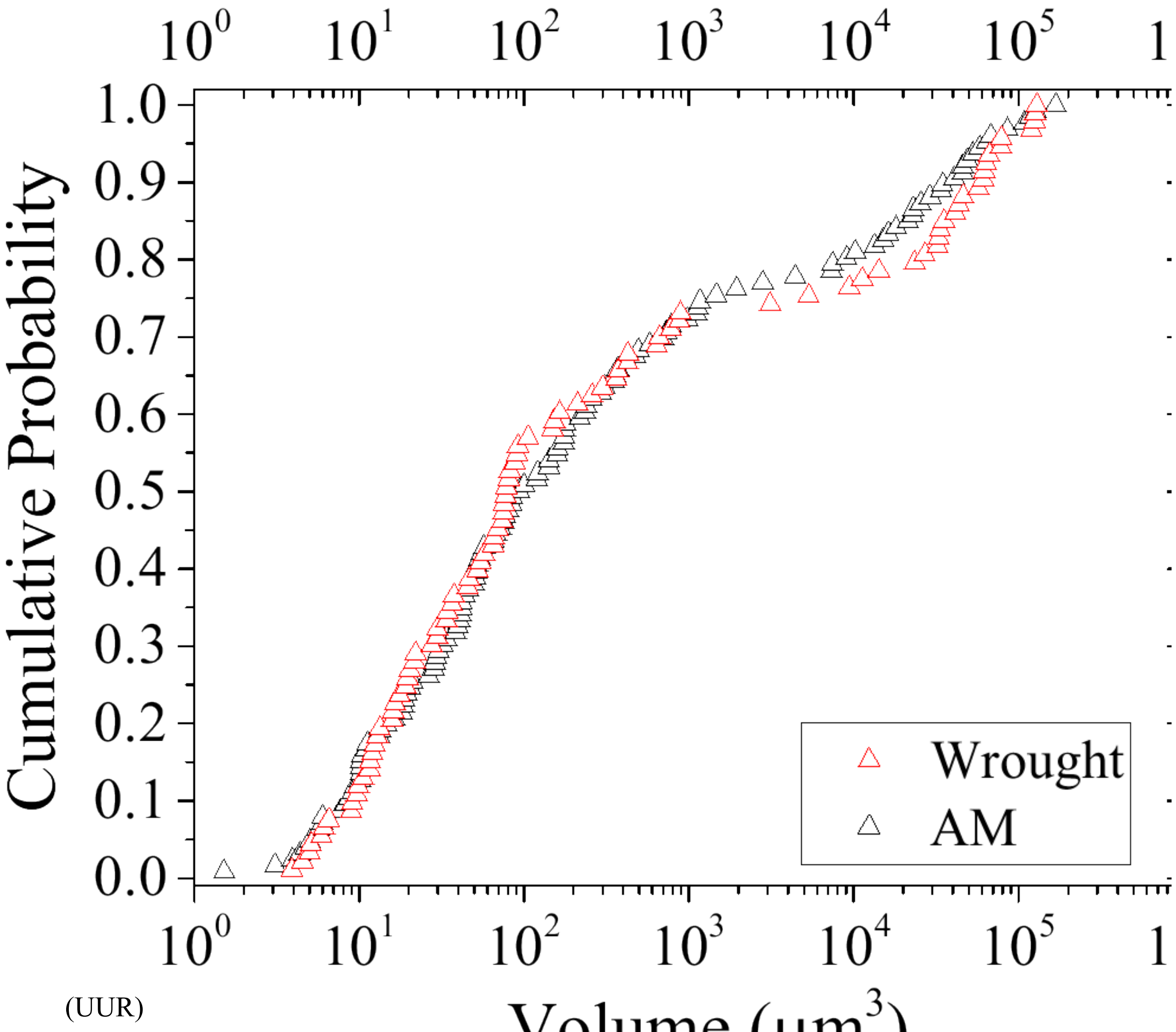
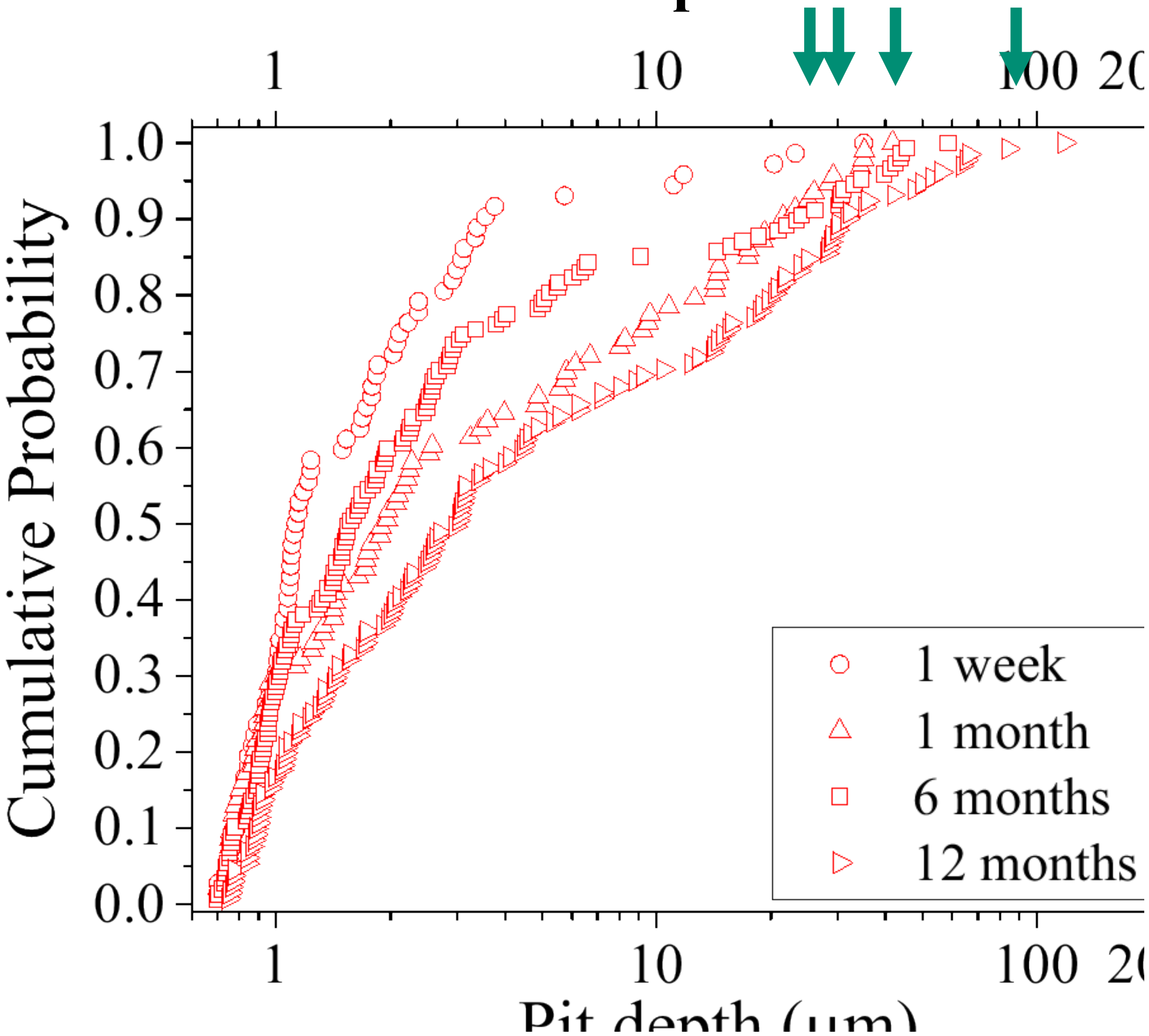
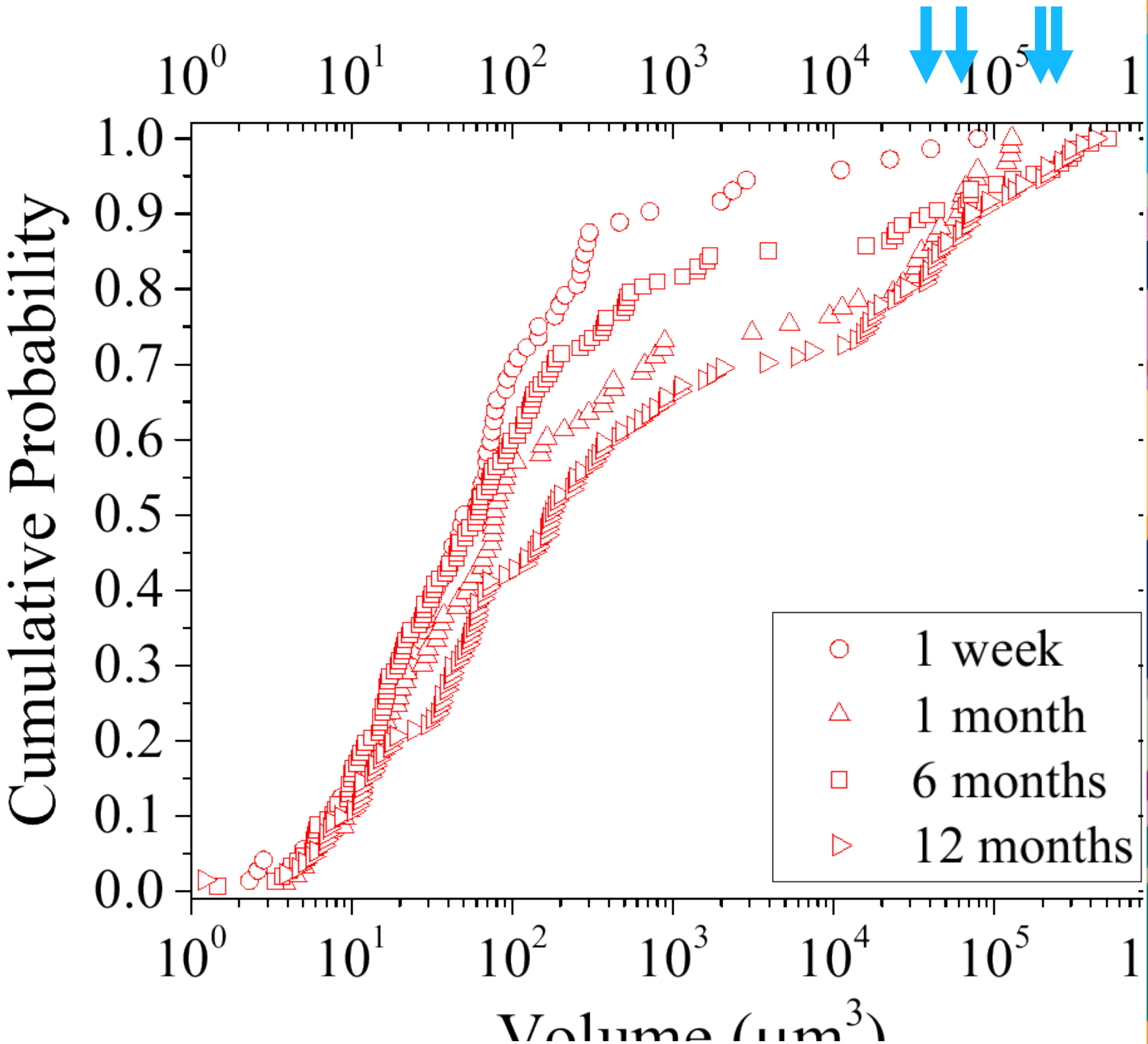


(UUR)

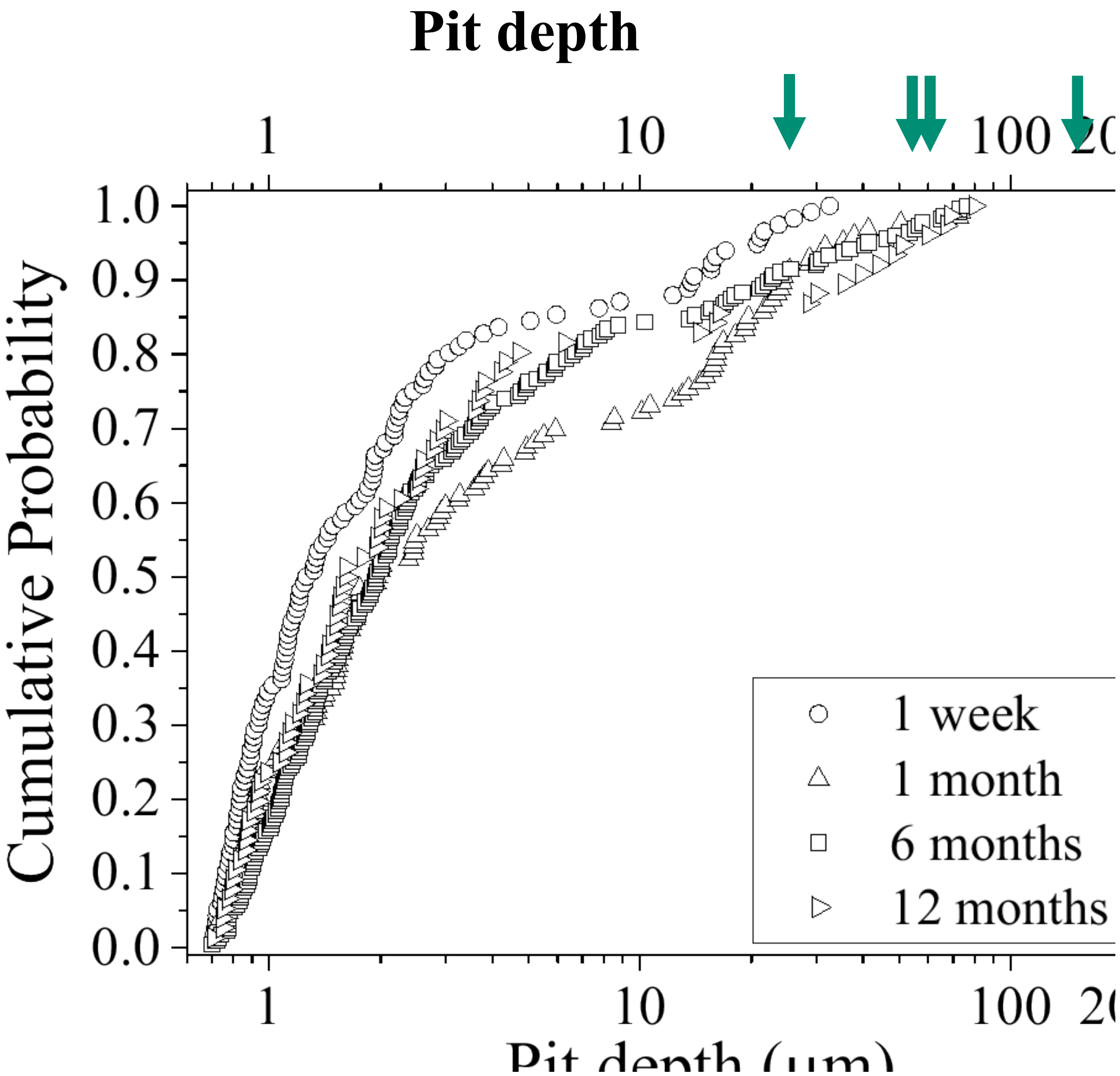
Pit statistics of **316L specimen** - **300 µg/cm² ASW at 40% RH**

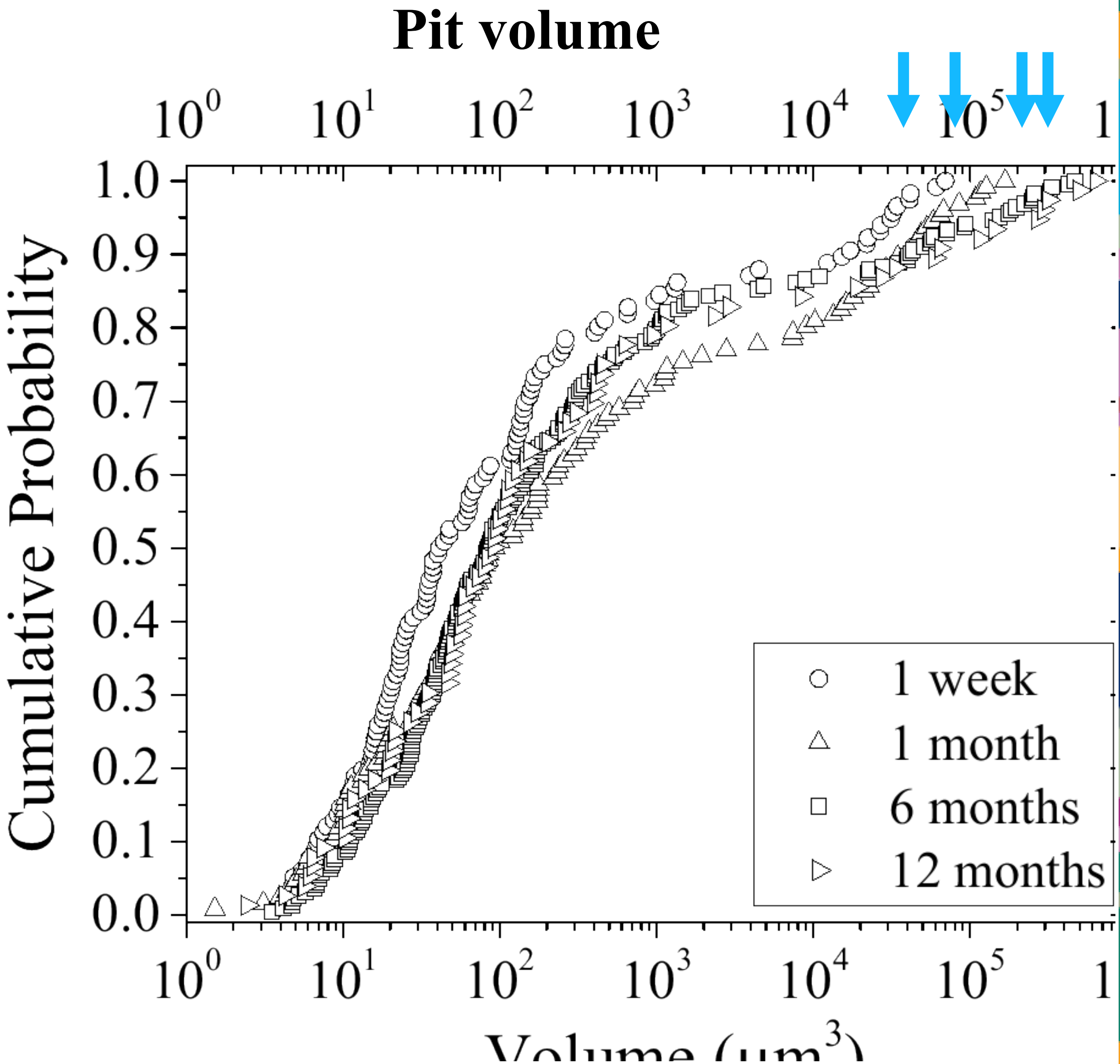




Wrought

Pit statistics of **316L specimen** - **300 μg/cm² ASW at 40% RH**





AM