CHARLOTTE

Additive Manufacturing of Ceramics: Can Surface Measurements Provide Processing and Part Integrity Insights?

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THE WILLIAM STATES LEE COLLEGE OF ENGINEERING

Additive Manufacturing of Ceramics

Y. Lakhdar et al.(2021)

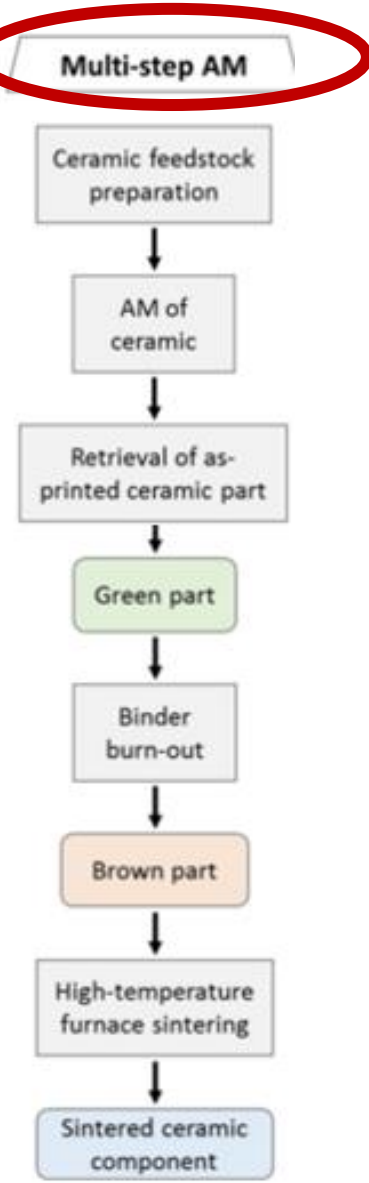
Direct Additive Manufacturing of Advanced Ceramics

***Single-step* processes**		***Multi-step* processes**						
Bedless	***Bed***	***Bed***			***Bedless***			
Directed Energy Deposition	**Powder Bed**	**Fusion**	**Binder Jetting**	**Sheet Lamination**	**Material Extrusion**		**Material Jetting**	**Vat Photopolymerisation**
LENS	Powder- dLS	Powder-iLS	Powder-BJ	LOM	Wax-based	Water-based	Solvent-DIP	SL
	Slurry- dLS	Slurry-iLS	Slurry-BJ	CAM-LEM	FDC	RC / DIW	Wax-DIP	DLP / LCM
					MJS	FEF		SPPW
					T3DP	CODE		2PP
					PHASE*	3DGP		

Available/soon to be at UNC Charlotte
https://camac.charlotte.edu/

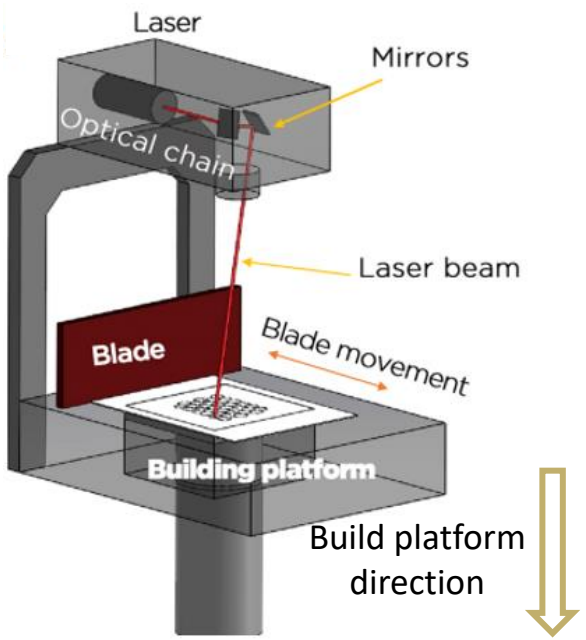
LENS: Laser Engineered Net Shaping
dLS/iLS: direct/indirect Laser Sintering
BJ: Binder Jetting
LOM: Laminated Object Manufacturing
CAM-LEM: Computer-Aided Manufacturing of Laminated Engineering Materials
SL: Stereolithography
DLP: Digital Light Projection
LCM: Lithography-based Ceramic Manufacturing
SPPW: Self-Propagating Photopolymer Waveguide
2PP: Two-Photon Photopolymerisation

FDC: Fused Deposition of Ceramics
MJS: Multiphase Jet Solidification
T3DP: Thermoplastic 3D Printing
PHASE: PHotopolymerisation-ASsisted Extrusion - *suggested acronym
RC: Robocasting
DIW: Direct Ink Writing
FEF: Freeze-Form Extrusion Fabrication
CODE: Ceramic On-Demand Extrusion
3DGP: 3D Gel Printing
DIP: Direct Inkjet Printing



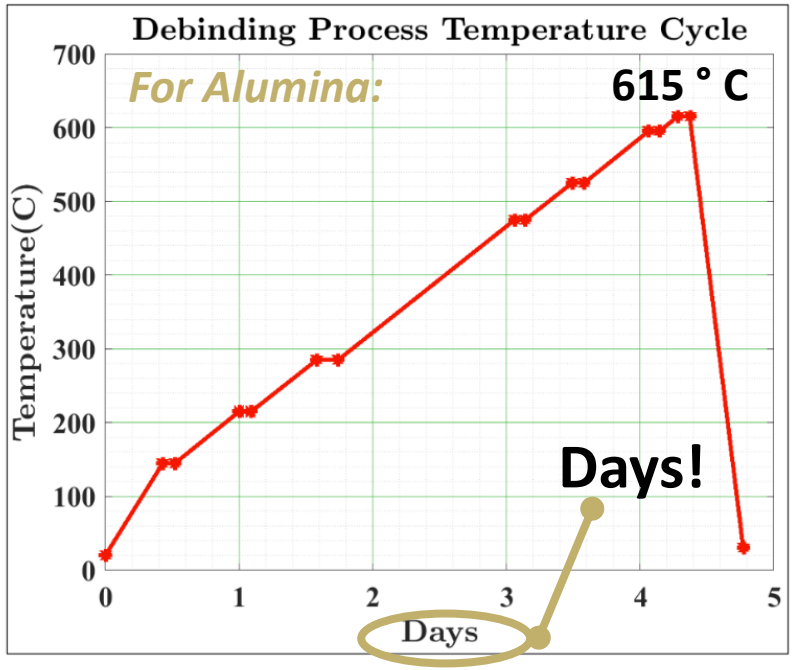
Vat Polymerization – Stereolithography (SLA)

Printing



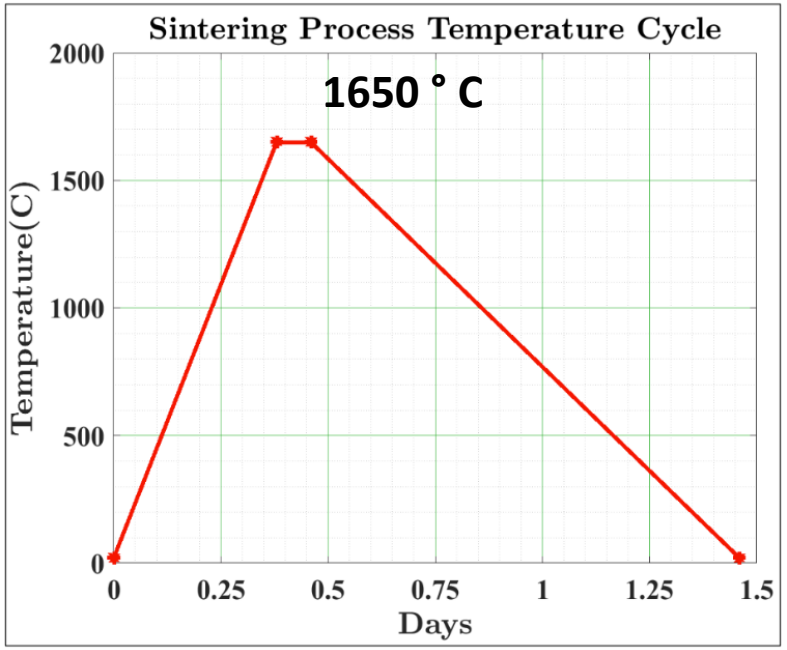
- Photocurable Polymer
- Ceramic powders (Ø< 1μm)
- Layer thickness: 50 - 100 μm
- Laser wavelength: 405 nm
- Laser spot size: ~50 μm

Debinding



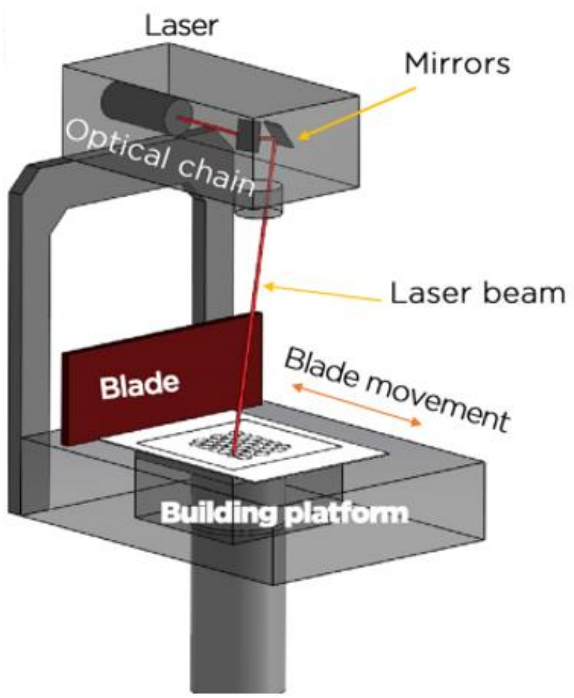
- Limits to feature thickness

Sintering

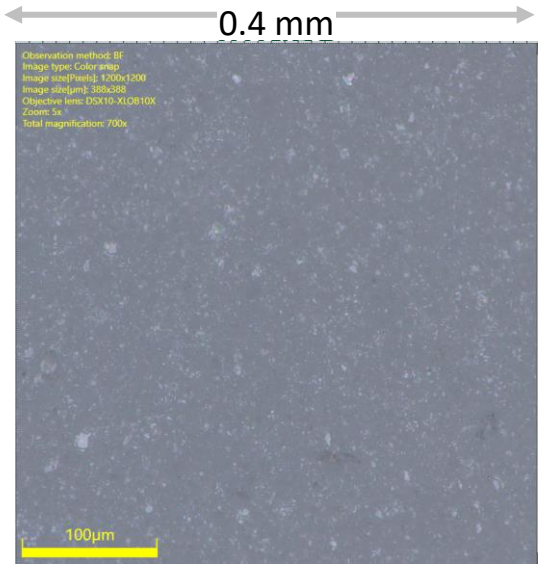


- 20% shrinkage is not uncommon
- Anisotropic shrinkage

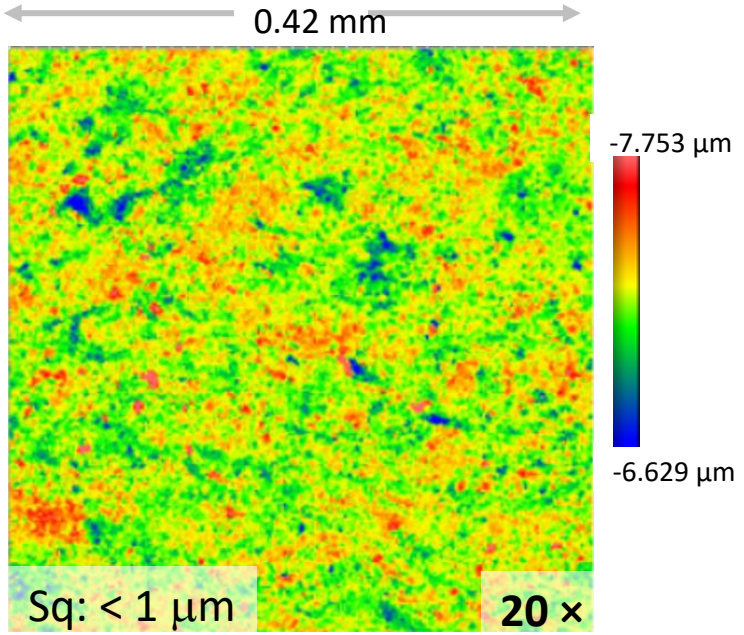
Stereolithography (SLA) - relevant length scales

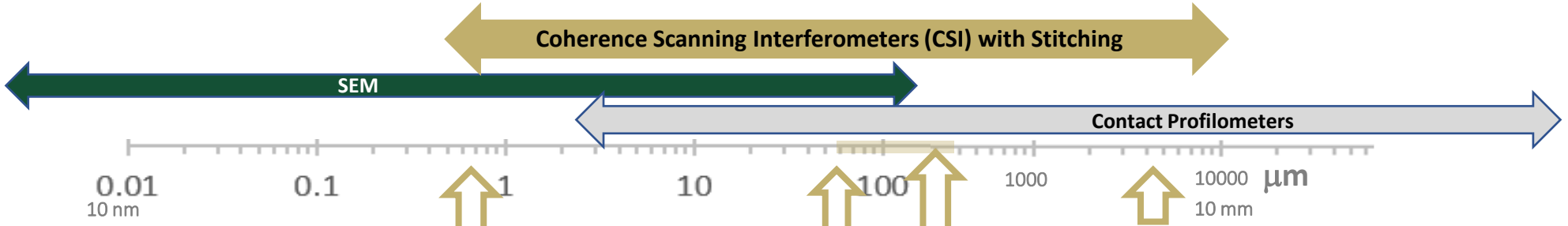


Optical Microscope



CSI Height Map





Max feature thickness

Min printable feature sizes.

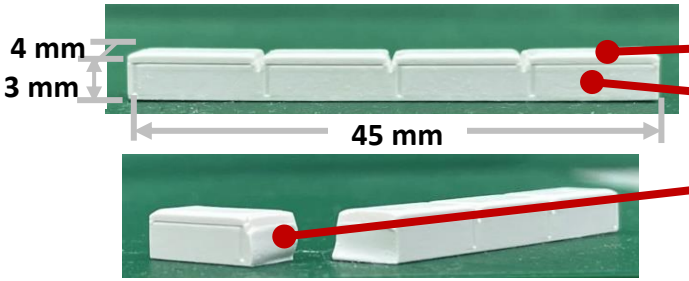
Particle size

Laser Spot size, Layer heights, stepovers, etc.

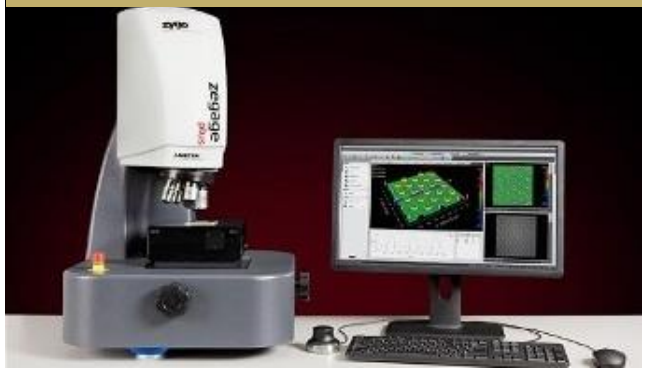
Surface Metrology and Analysis Approaches

SLA Printing

Alumina - printed bars



Surface Measurement



Measurement

- Zygo Zegage
- 20 × (FoV 0.417 μm × 0.417 μm)
- Lateral sampling 0.7 μm

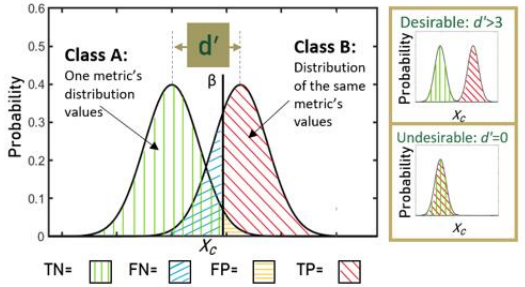
Surfaces measured

- Top surface – last layer
- Side - build direction
- Fractured surface

States

- Green
- Sintered

Analysis



Assessment

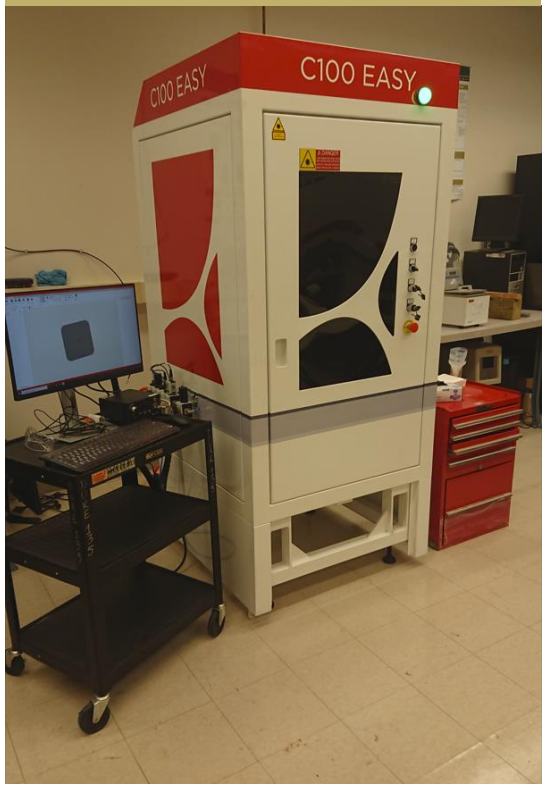
- Process signatures
- Part characteristics

Classification

- SQuID™ - ML approach
- Processing variances

Printing conditions

SLA Printing

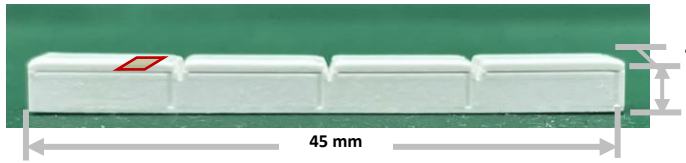


	Ideal	Non-ideal
Slurry	Al_2O_3	Same
Slurry age	1 month old	3 months past expiration date
Pre use	3.5 hrs mixing	Same
Laser spot	~ 50 mm	Same
Wavelength	405 nm	Same
Power	~ 50mW	Same
Layer thickness	50 mm	Same
Mark time	Nominal, **Note:** one test done with a 50% reduction in mark time	Nominal
Time in green- state	~10 days	~42 days
Debinding ramps / max. temp.	Nominal/ 615 ° C	Same
Debinding atmosphere	Nitrogen	Air
Sintering ramps/ max. temp.	Nominal/ 1650 ° C	Same

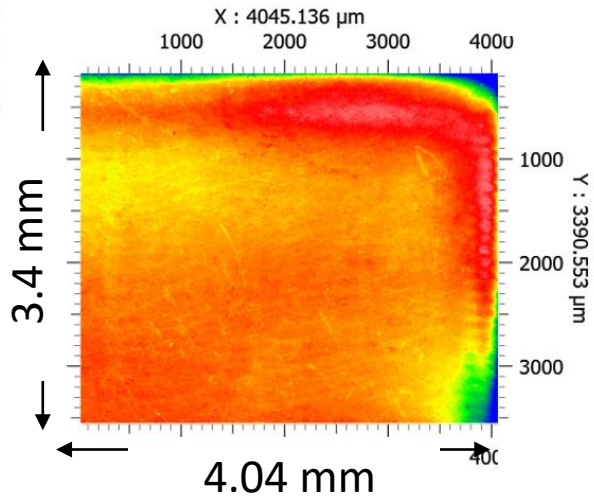
Nominal Build – Green – Top surface

Measurement

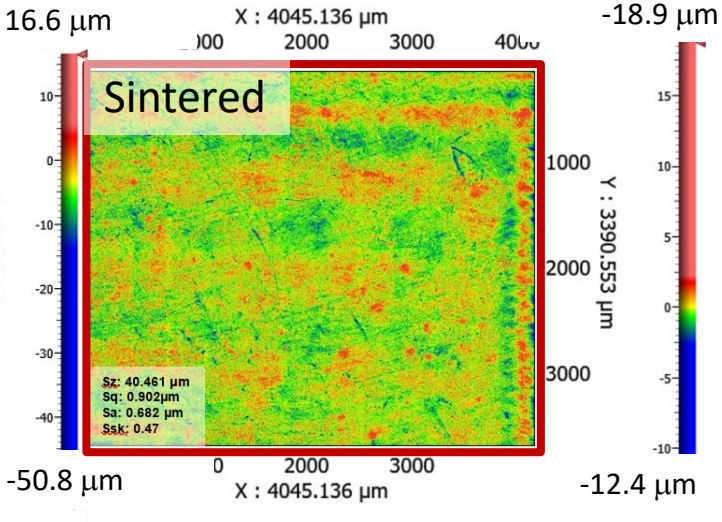
- *CSI: Obj. 20 ×*
- *Stitched: 10 × 12*
- *Removed*
  - *See below*



Representative measurement areas on sample

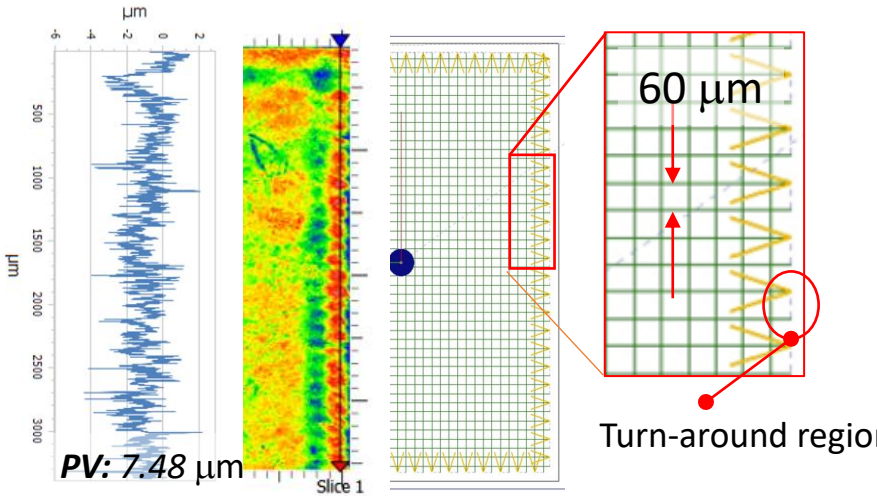


Processing: Plane removed
Sq: 2.7 µm
Sz: 67.5 µm



Processing: 10th order removed
Sq: 0.8 µm
Sz: 31.3 µm

Sq: 0.9 µm
Sz: 40.5 µm



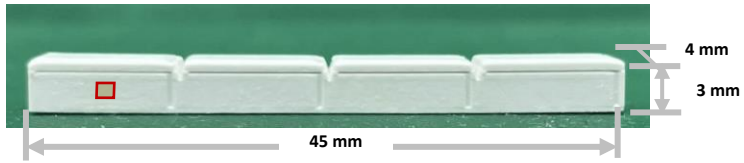
Slice through periodic features

Laser Scan path

Nominal Build –Green & Sintered – Side surface

Measurement

- *CSI: Obj. 20 ×*
- *Removed*
  - *Best fit plane*



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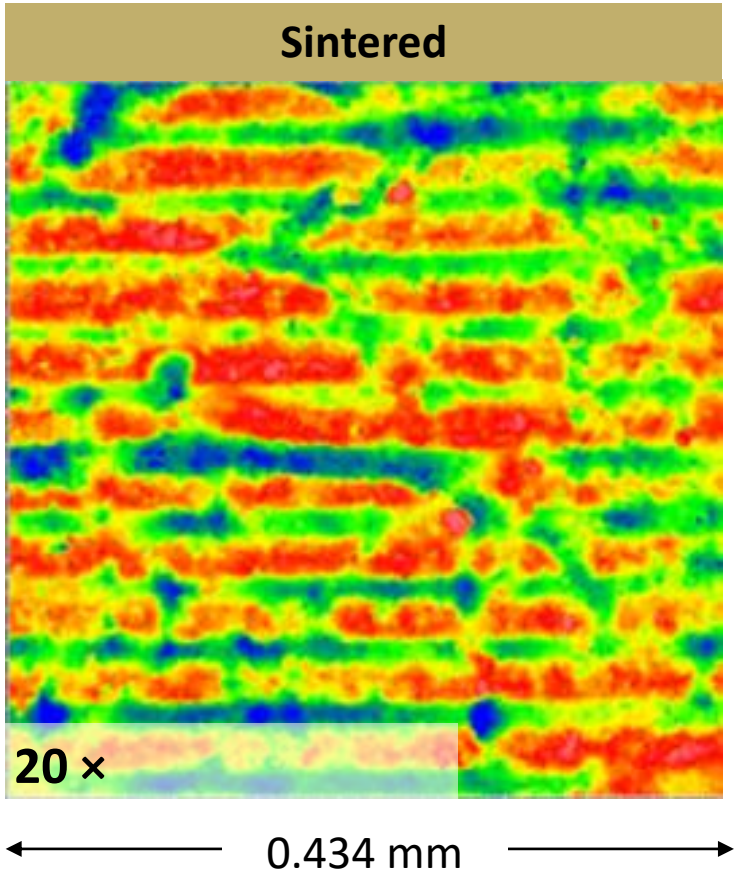
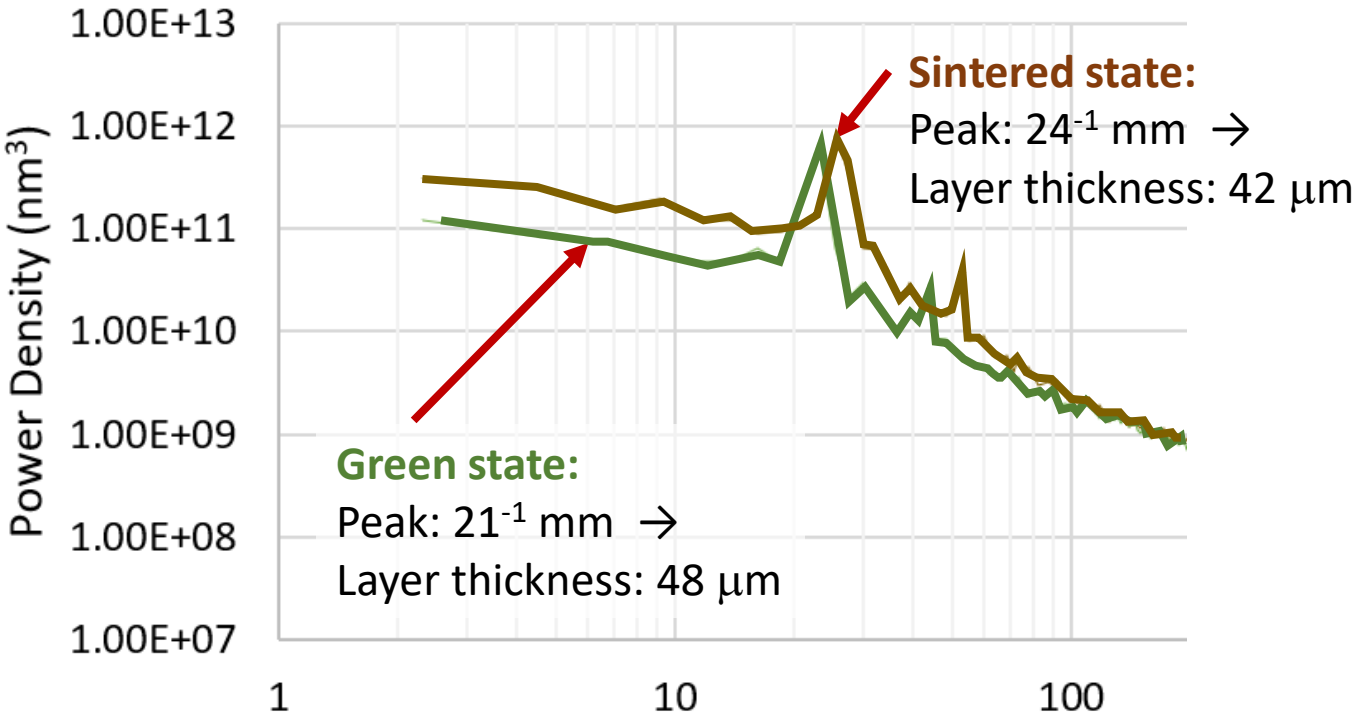
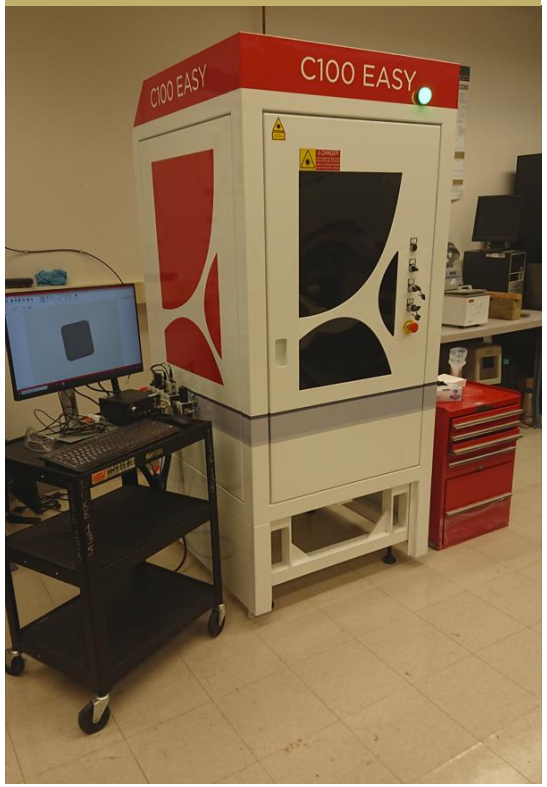




Printing conditions

SLA Printing



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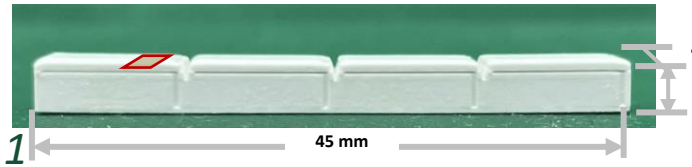
Time in green- state	~10 days	~42 days
Debinding ramps / max. temp.	Nominal/ 615 ° C	Same
Debinding atmosphere	Nitrogen	Air
Sintering ramps/ max. temp.	Nominal/ 1650 ° C	Same

Reduced mark speed- Green - Top surface

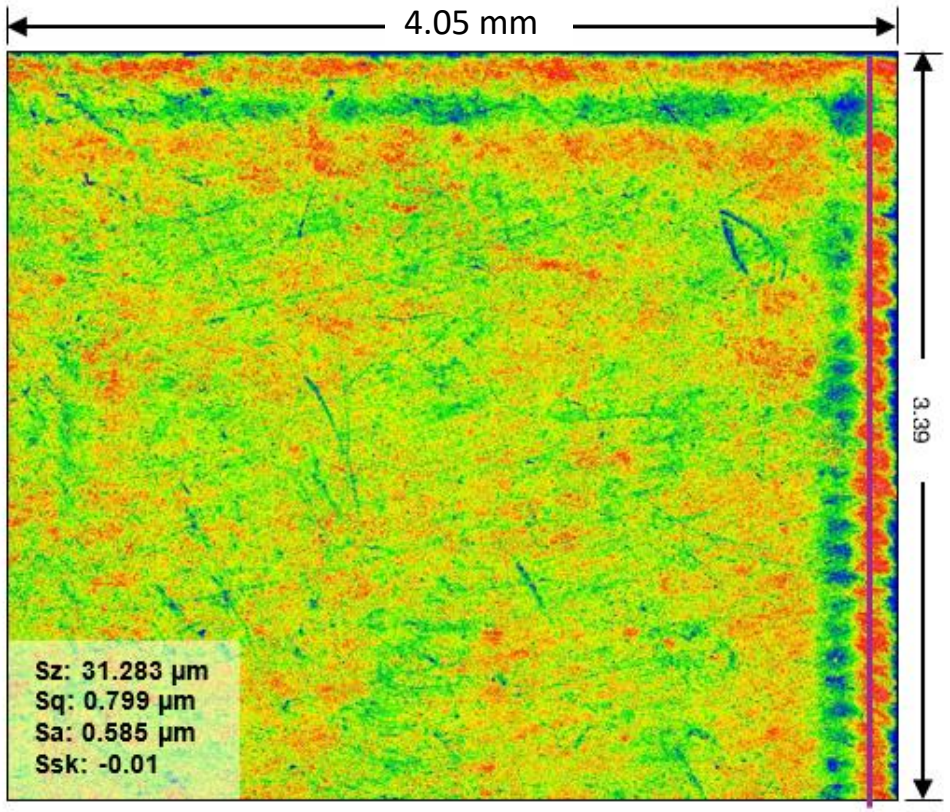
Measurement

- *CSI: Obj. 20 ×*
- *Stitched: 10 × 12 , 4 × 11*
- *Top surface*
- *Removed:*
  - *10th order poly*

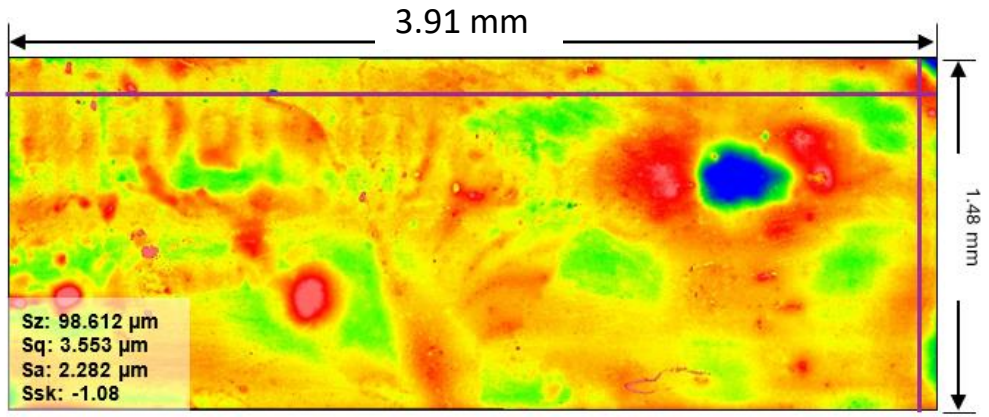


Representative measurement areas on sample

Ideal - Green State



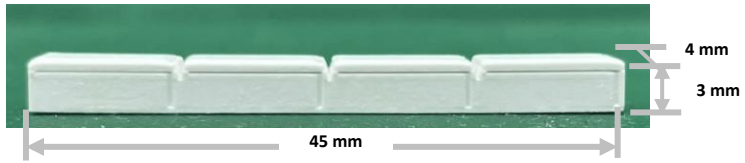
50% Mark time - Green State



Reduced mark speed- Green - Fractured surface

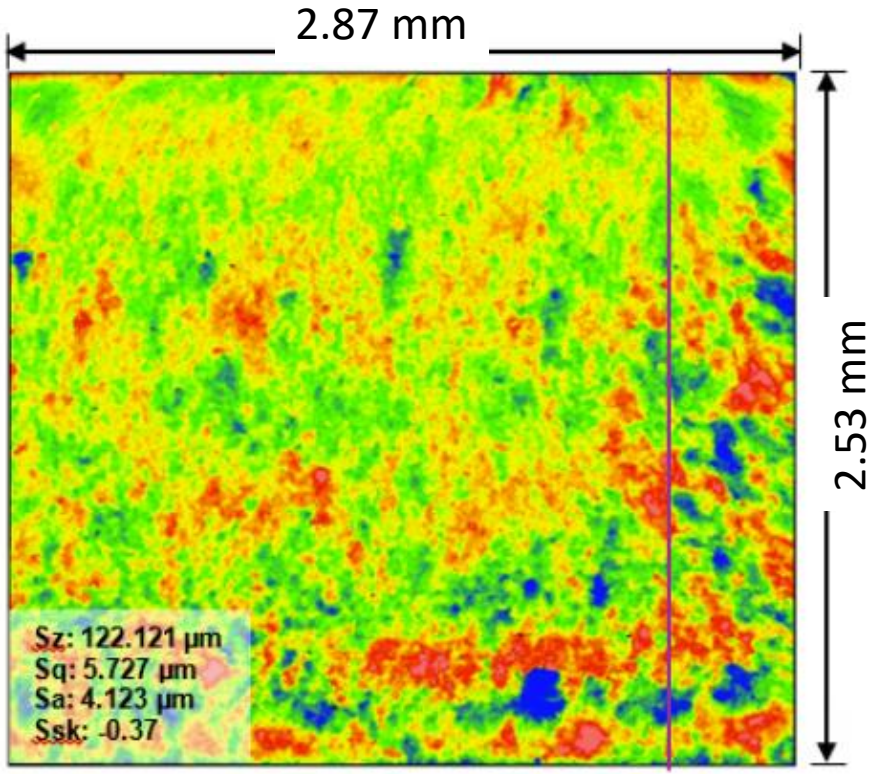
Measurement

- *CSI: Obj. 20 ×*
- *Stitched*
- *Fractured surface*
- *Removed*
  - *4th order poly*

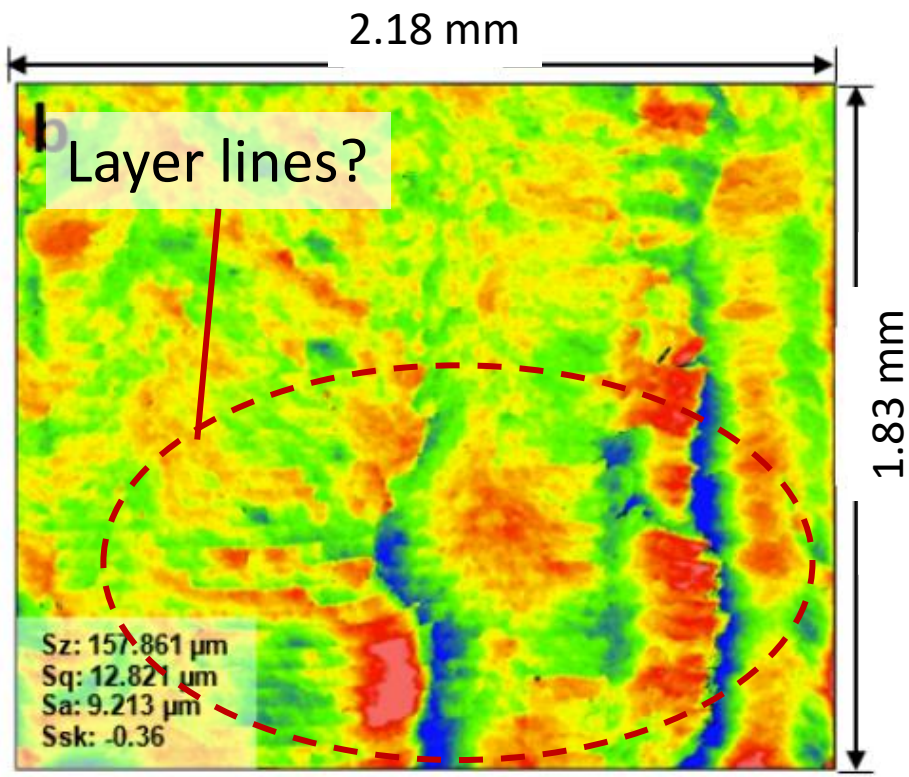


Representative measurement areas on sample

Nominal build

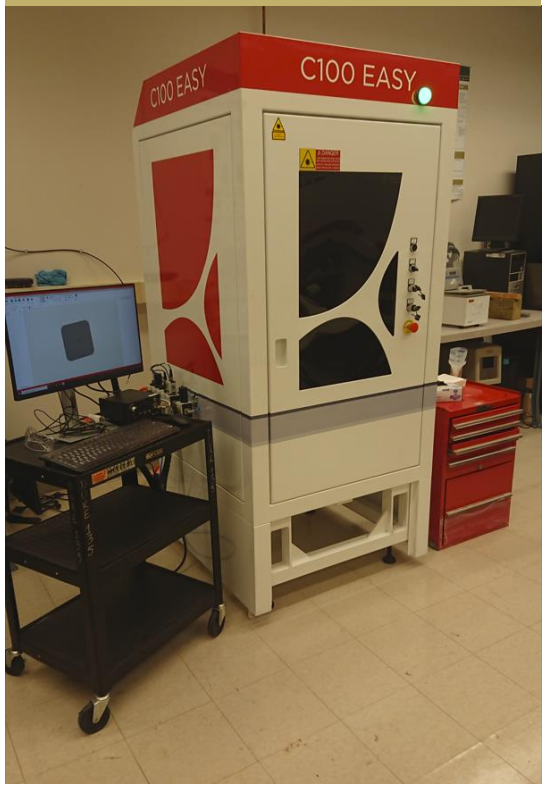


50% reduction in Mark Time



Printing conditions

SLA Printing



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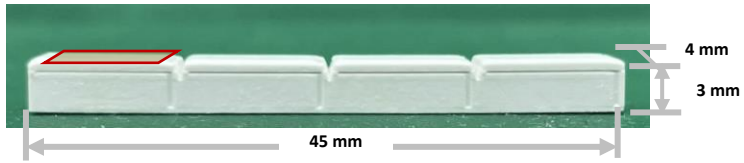
Ideal Vs Non ideal – Green State

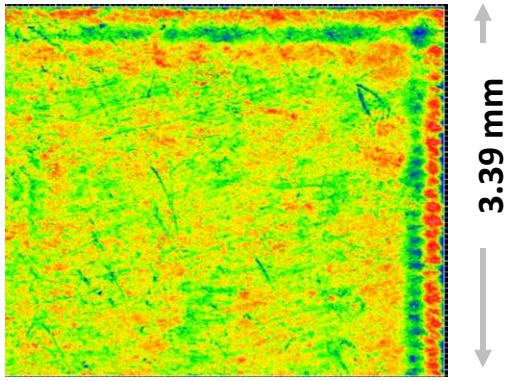


Representative measurement areas on sample

Ideal - Green State

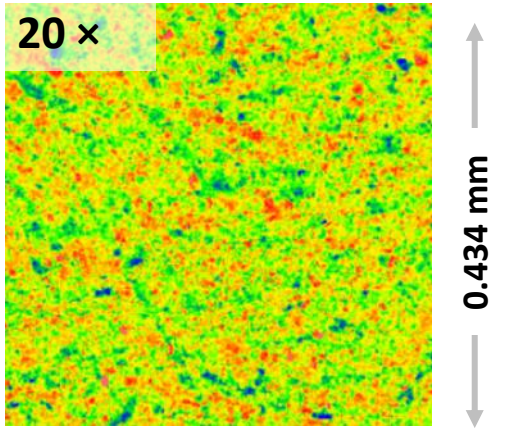
CSI Measurement
- 20 ×
- Stitched
- 20% overlap

Removed
- 10th order poly

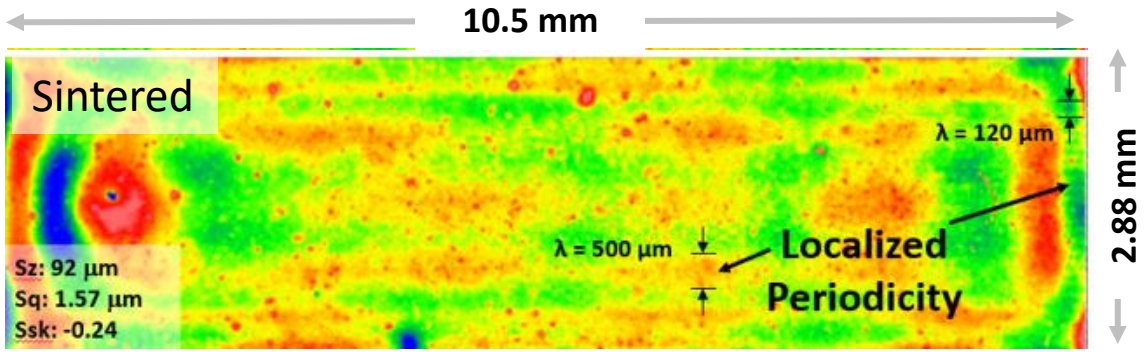


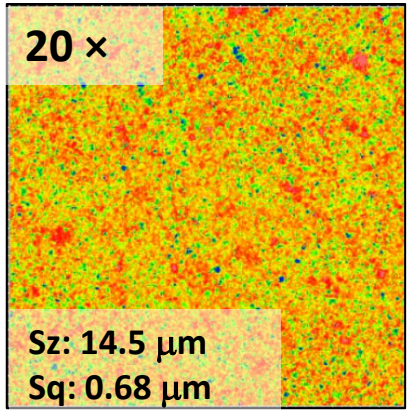
CSI Measurement
- 20 ×

Removed
- Best Fit Plane



Non-Ideal - Green State

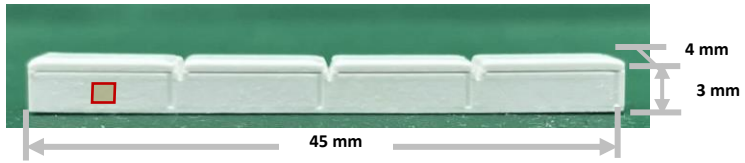




Non ideal – Green and Sintered States

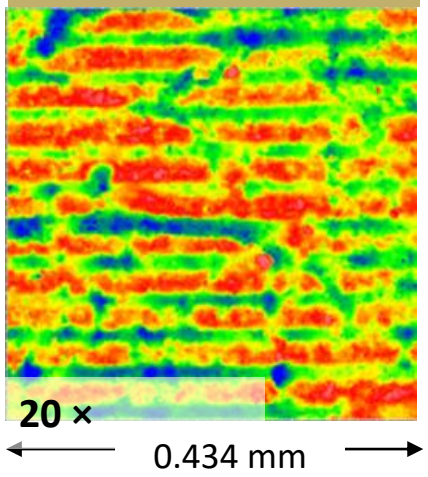
Measurement

- *CSI: Obj. 20 ×*
- *Side surface*
- *Removed:*
  - *4th order poly*

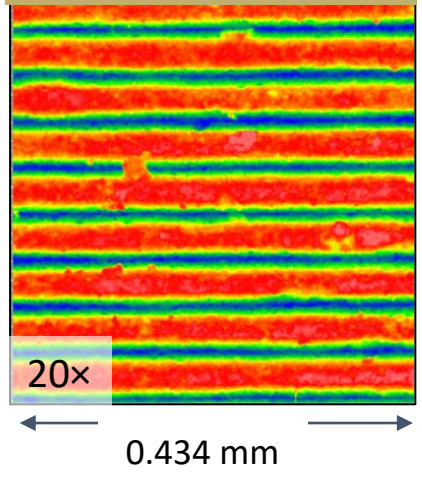


Representative measurement areas on sample

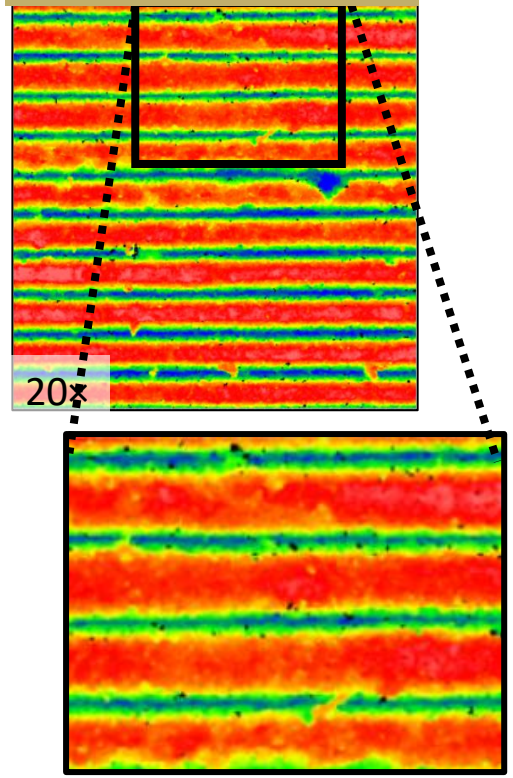
Ideal Green



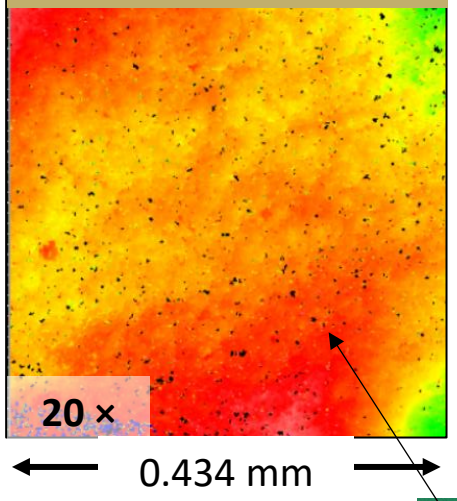
Non-ideal Green



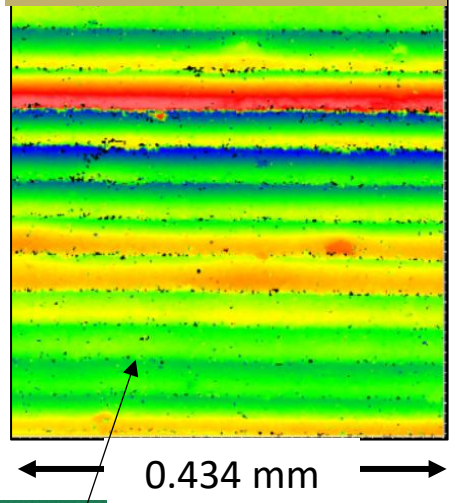
Non-ideal Sintered

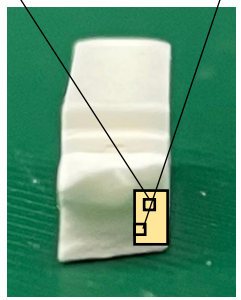


Non-ideal Sintered – Fracture



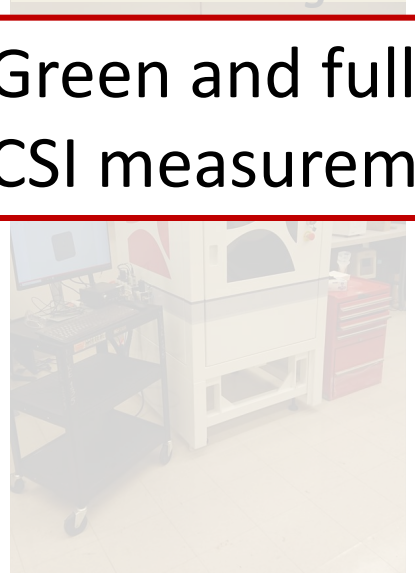
Non-ideal Sintered – Fracture



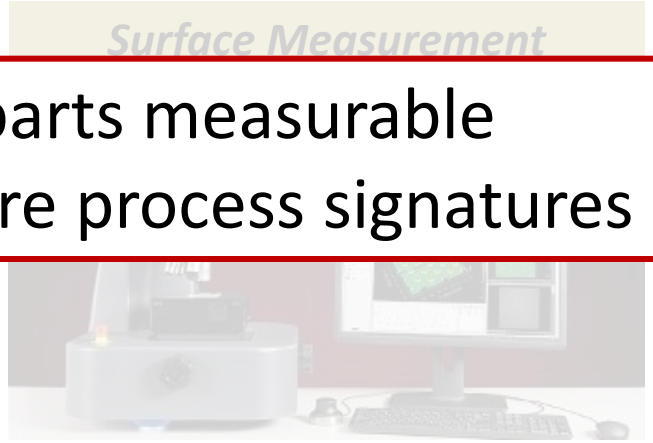


Surface Metrology and Analysis Approaches

SLA Printing



Surface Measurement



Measurement
- Zygo Zegage
- 20 × (FoV 0.417 μm × 0.417 μm)
- Lateral sampling 0.7 μm

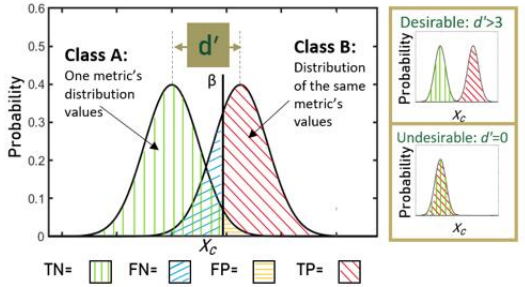
- Sintered

- Green and full-strength parts measurable
- CSI measurements capture process signatures

- Need into quantify observed differences
  - Which metrics track with observed variances?

Link with process physics, slurry condition etc.

Analysis



Assessment
- Process signatures
- Part characteristics

Classification
- SQuID™ - ML approach
- Processing variances

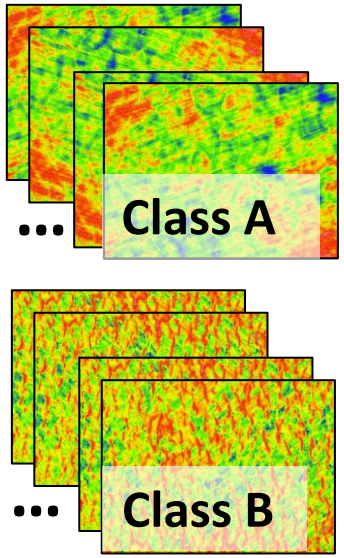
Systematic approach to the problem

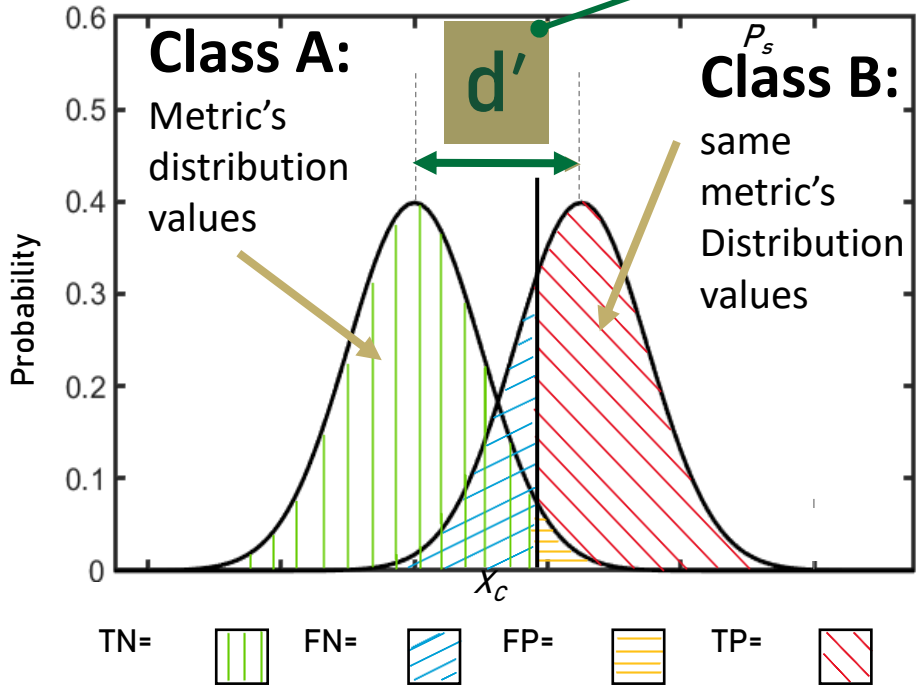
Systematic approach

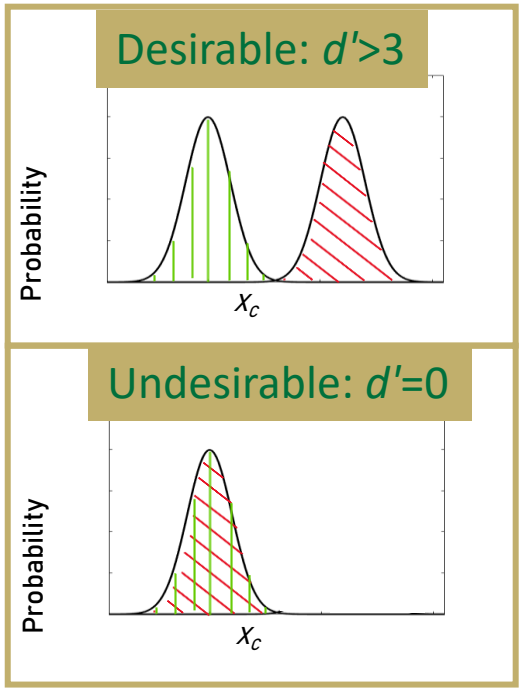
- Analytical Framework: **S**urface **Qu**ality and **I**nspection **D**escriptors (SQuID™)
  - Automatic isolation of suitable classification metrics
  - Simple, interpretable decision tree outputs

$$d' = \frac{|\mu_A - \mu_B|}{\sqrt{\frac{(\sigma_A^2 + \sigma_B^2)}{2}}}$$

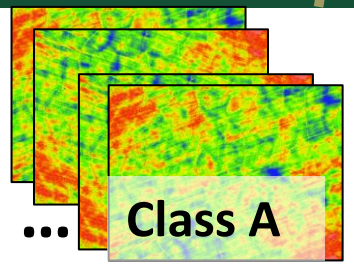
Classification features?

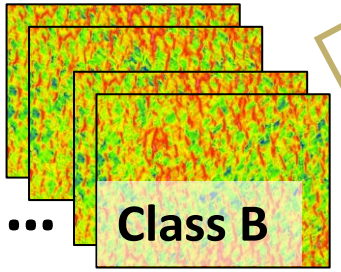






Data flow chart and SQuID™ output

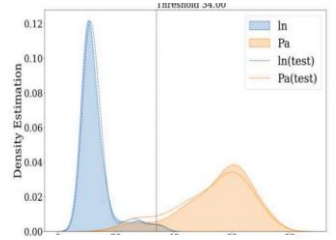




Data processing
- Fifteen 20× measurements from each surface
- Best fit plane subtraction
- Calculate ISO 25178-2 metrics – using 21 of them

SQuID™
See GitHub
https://jesse-redford-squid-main-otrfh7.streamlit.app/

d' Values
Associated with each ISO metric



Metric values

d´ values

Sq	4.7
Sku	3.6
Ssk	1.5
Sdq	.9
⋮	⋱
Sal	0.9
Str	1.2

A vs B

Down selection of optimal metric(s)

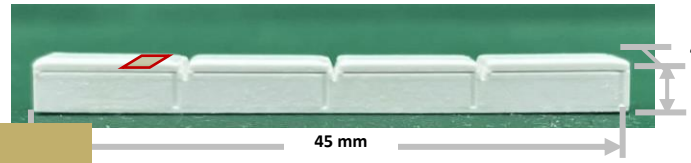
Sq 4.7

When using multiple metrics, a decision tree can be generated to outline classification decision

Data collection
- Multiple height maps per class
- Will also work for jpeg, bmp , etc.

Full details:
J. Redford et al.
Journal of Manufacturing Systems 71 (2023) 131–143

Ideal Vs Non ideal – Green State

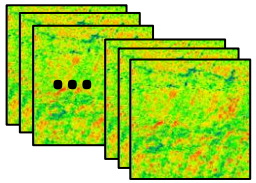


Representative measurement areas on sample

Ideal - Green State

Nominal Conditions

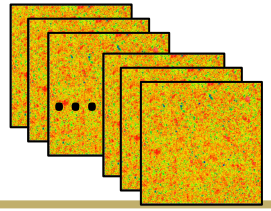
- 15 CSI measurements: 20×
- Best fit plane removed
- Top surface



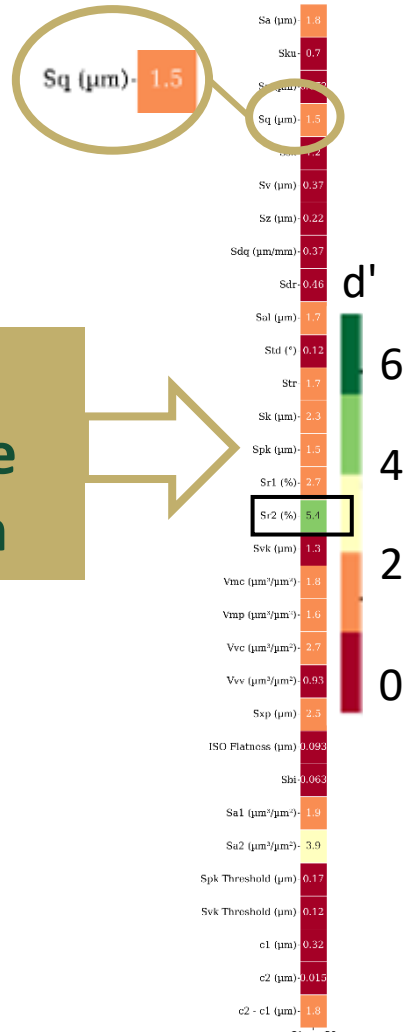
Non-Ideal - Green State

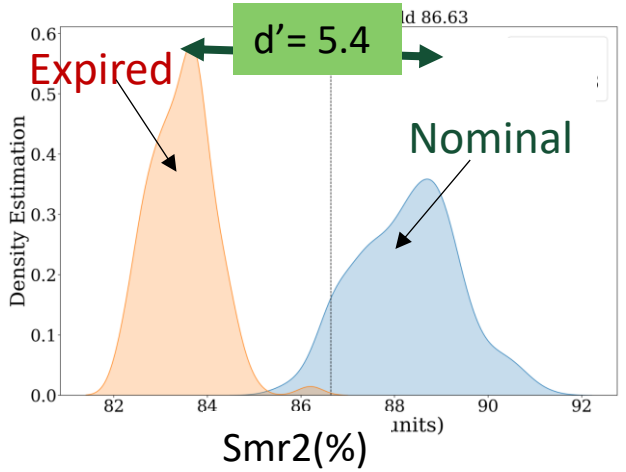
Old Slurry

- 15 CSI measurements: 20×
- Best fit plane removed
- Top surface



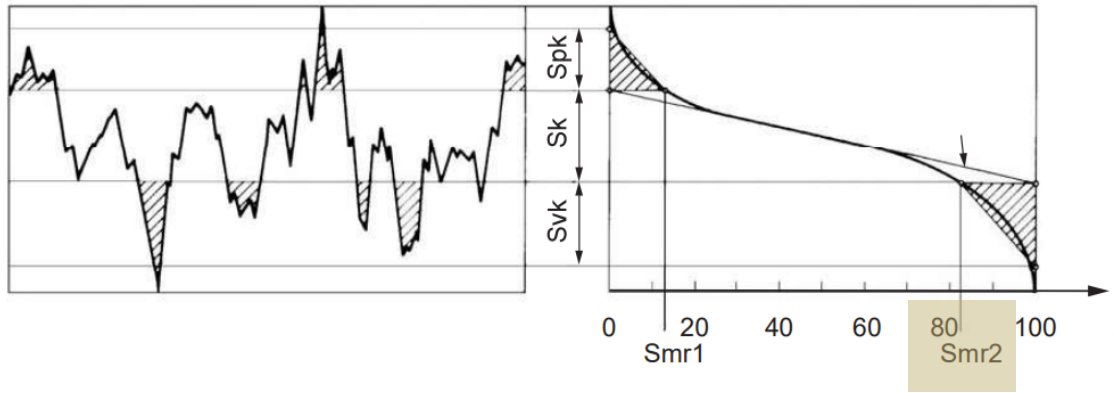
SQuID™ Software Platform



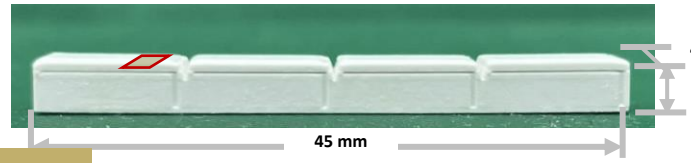


Material probability curve and its plateau and dale regions

- Related to the extent of valleys/voids below the surface plateau



Mark Speed reduction – Green State

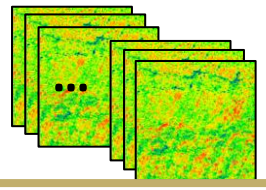


Representative measurement areas on sample

Ideal - Green State

Nominal Mark Speed

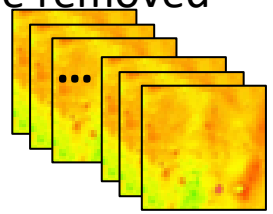
- 15 CSI measurements: 20×
- Best fit plane removed
- Top surface



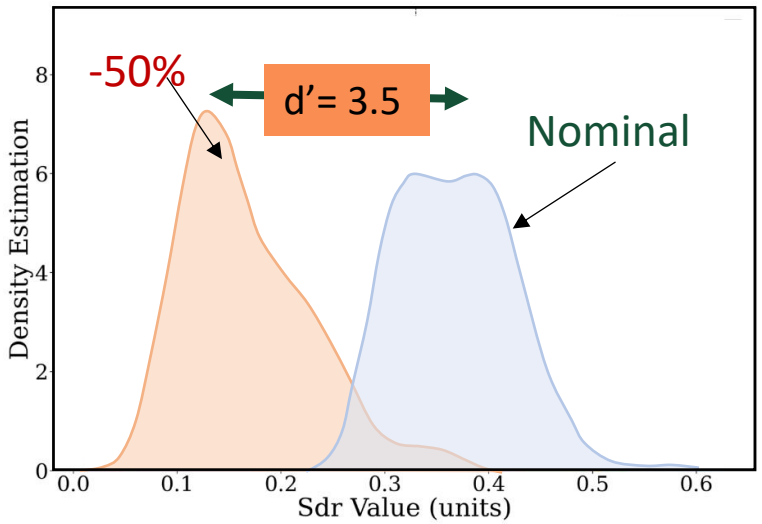
50% reduction - Green

50% reduction in Mark Speed

- 15 CSI measurements: 20×
- Best fit plane removed
- Top surface

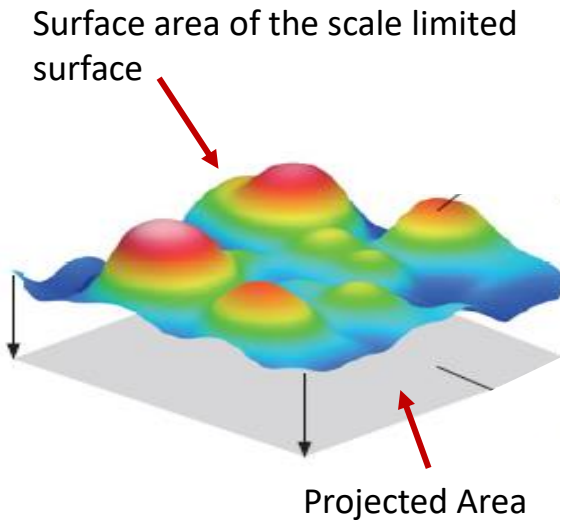


SQuID™ Software Platform



Developed Interfacial Area Ratio (Sdr)

- difference between the projected surface and the actual surface



Can Surface Measurements Provide Processing & Part Integrity Insights?

Summary

- CSI measurements capture expected and unexpected features
- Surface metrics can detect process drift
  - Green strength can be used to assess process state

Repeatability:

- Needs more work beyond that shown here, but initial results are promising

Predictability:

- More work (much more) needed to link the process output to process physics and performance

Acknowledgements

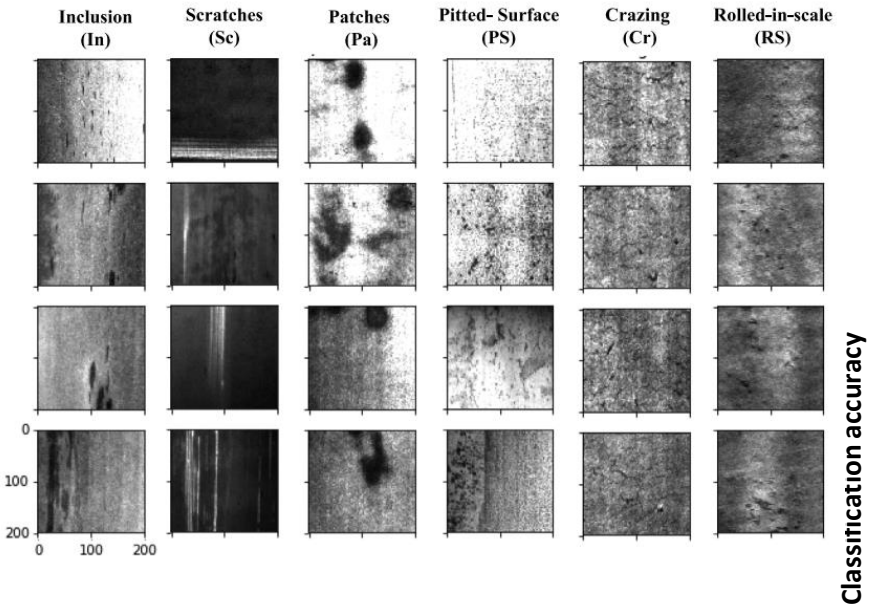
- Tien Herd
- Taylor Barrett
- Jesse Redford
- Center for Precision Metrology (CPM)
- Center for Additive Manufacture of Advanced Ceramics (CAMAC)

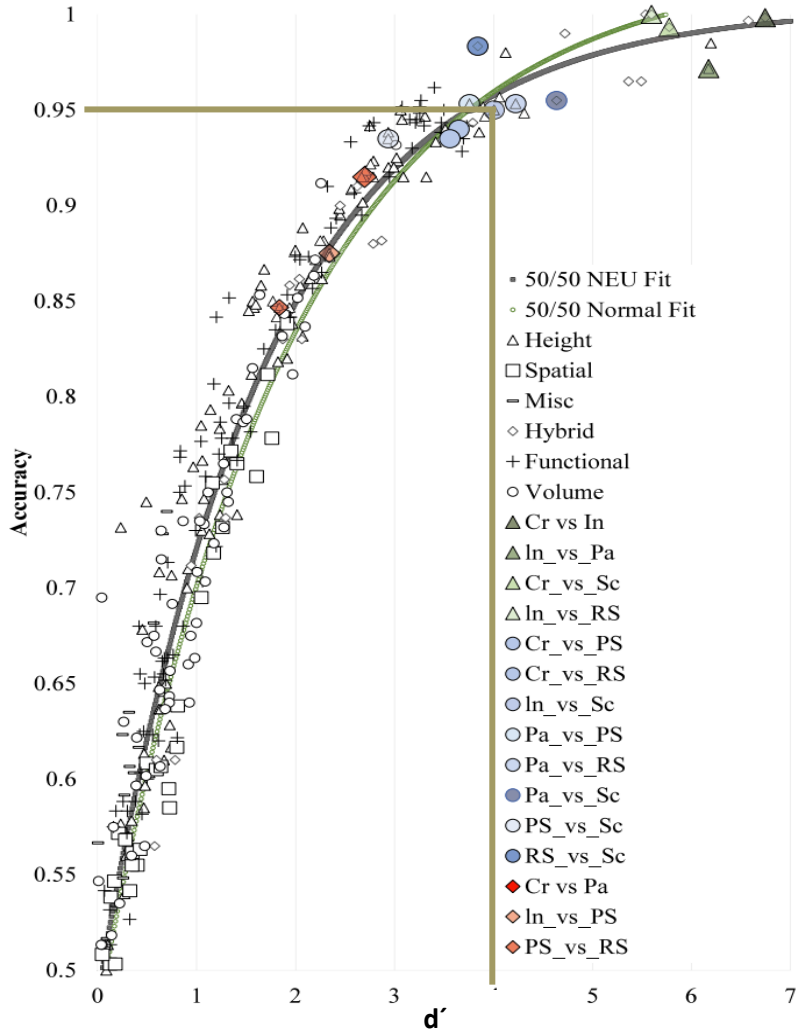
THE UNIVERSITY OF NORTH CAROLINA SYSTEM

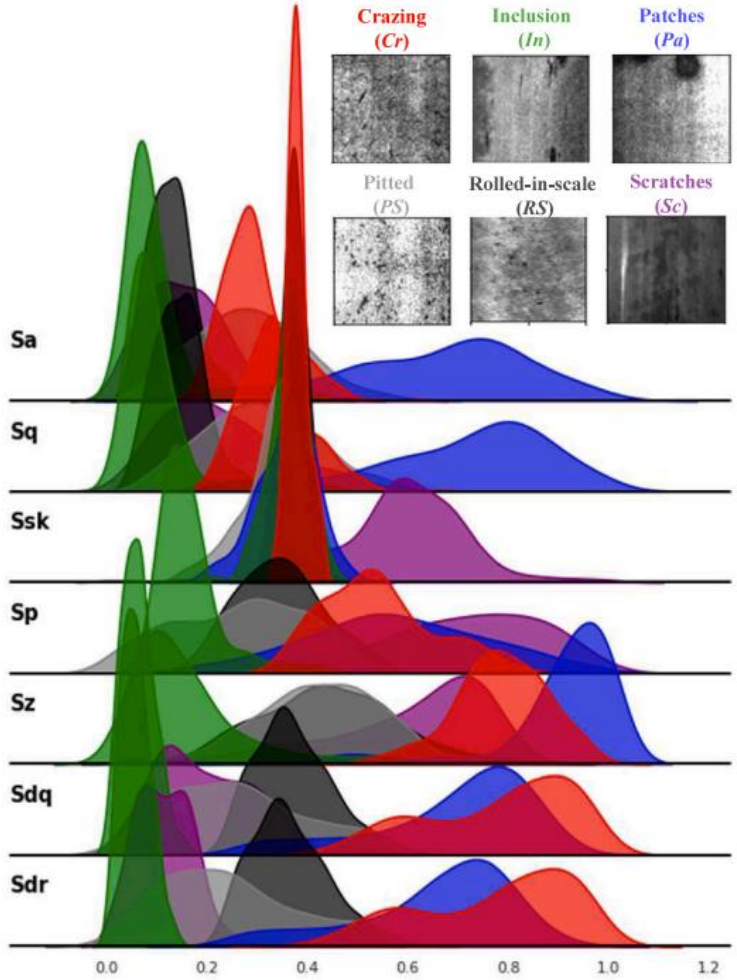
Questions?

bamullan@charlotte.edu

SQuID™


Inclusion (In)
Scratches (Sc)
Patches (Pa)
Pitted- Surface (PS)
Crazing (Cr)
Rolled-in-scale (RS)
0
100
200
0
100
200


Classification accuracy
Accuracy
d´
1
0.95
0.9
0.85
0.8
0.75
0.7
0.65
0.6
0.55
0.5
0
1
2
3
5
6
7
50/50 NEU Fit
50/50 Normal Fit
Height
Spatial
Misc
Hybrid
Functional
Volume
Cr vs In
ln_vs_Pa
Cr_vs_Sc
ln_vs_RS
Cr_vs_PS
Cr_vs_RS
ln_vs_Sc
Pa_vs_PS
Pa_vs_RS
Pa_vs_Sc
PS_vs_Sc
RS_vs_Sc
Cr vs Pa
ln_vs_PS
PS_vs_RS


Crazing (Cr)
Inclusion (In)
Patches (Pa)
Pitted (PS)
Rolled-in-scale (RS)
Scratches (Sc)
Sa
Sq
Ssk
Sp
Sz
Sdq
Sdr
0.0
0.2
0.4
0.6
0.8
1.0
1.2

CSI measurements

Objective: 2.75 ×

Tube Lens: 0.5 ×

FoV 6.05 mm × 6.05 mm

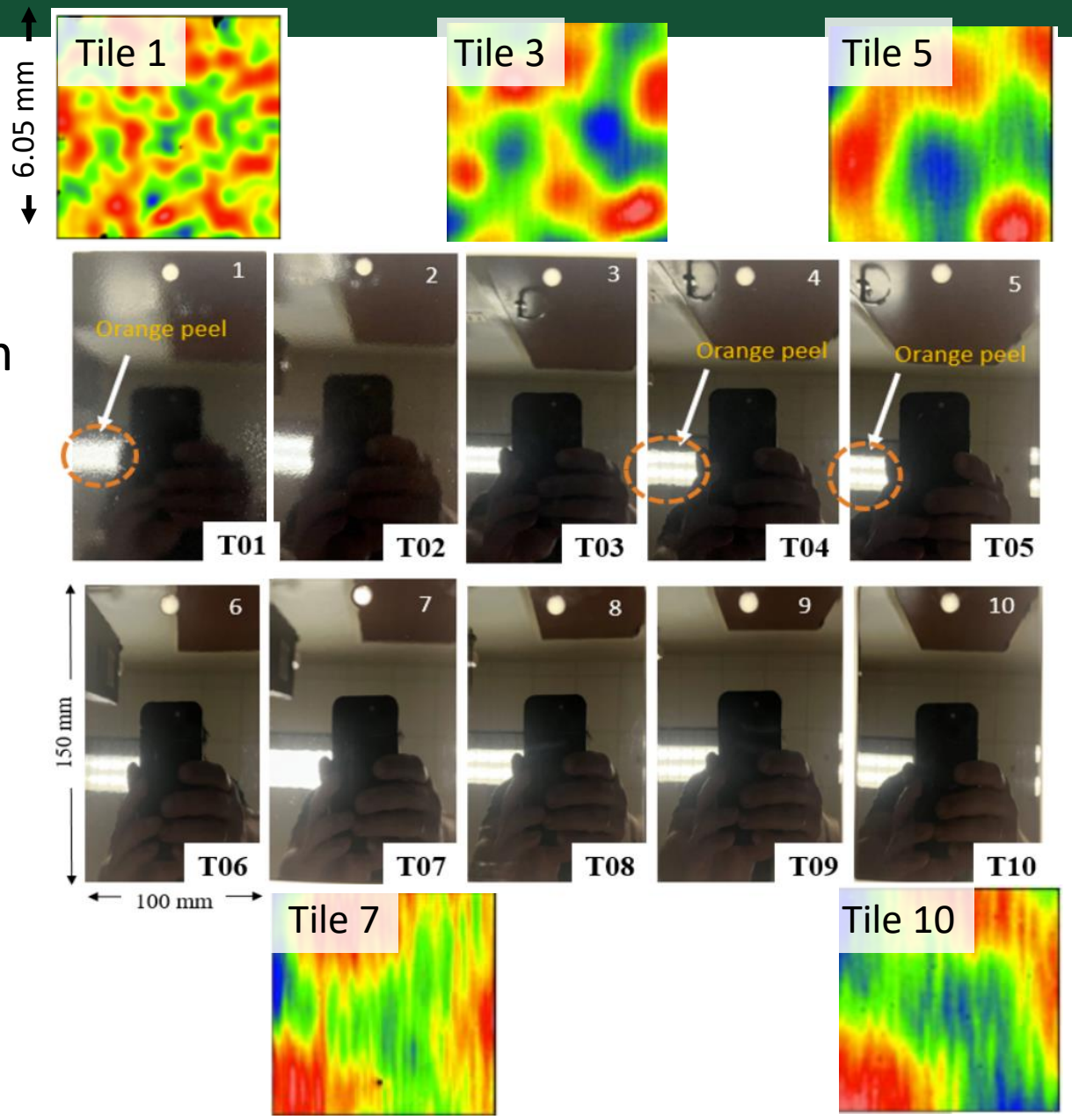
Spatial sampling: 5.9 μm

Removed: Best fit plane

Measurements: 36/tile

Metrics: 22 ISO metrics

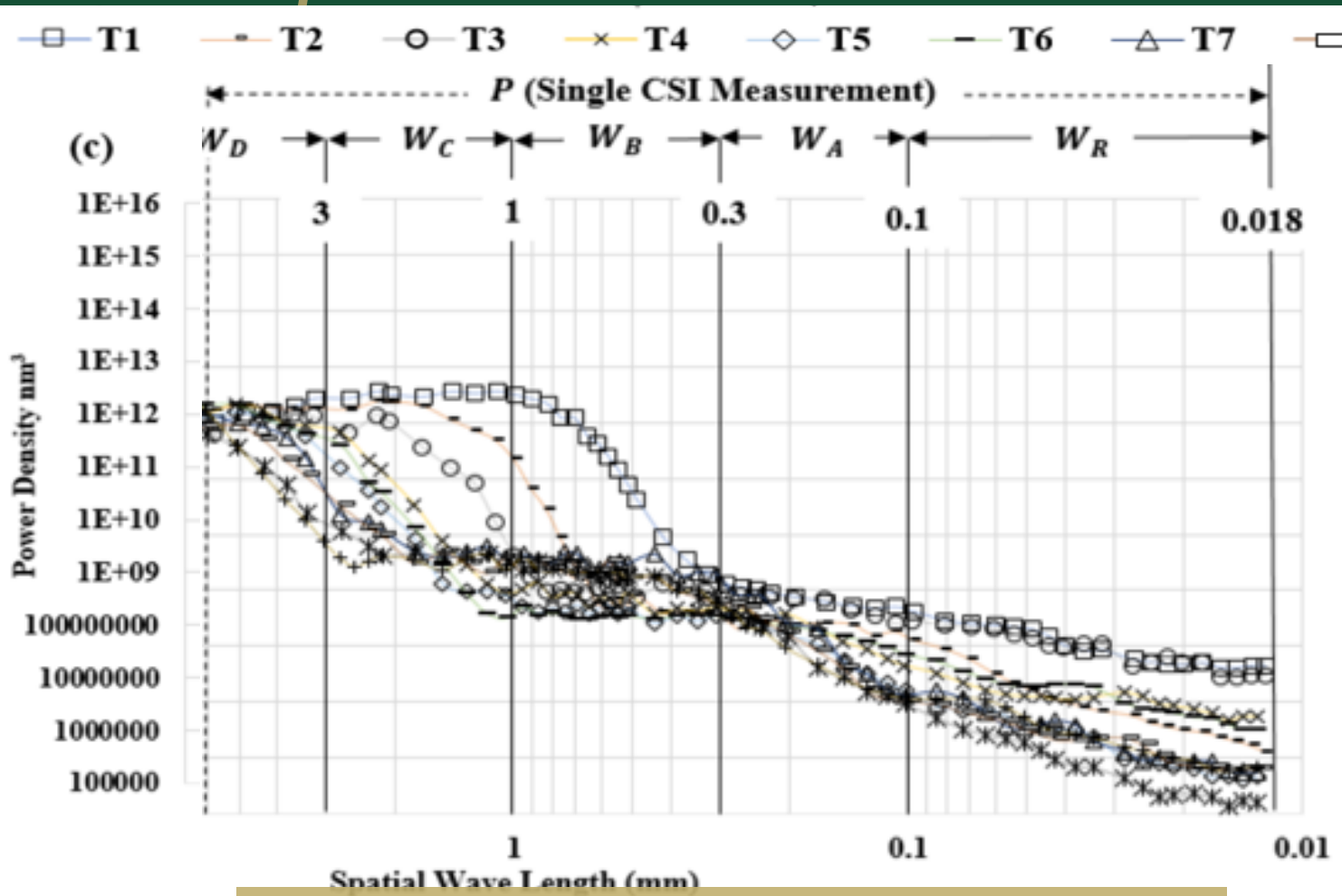
$$Sa, Sq, Ssk,$$
$$Sku, Sp, Sv, Sz,$$
$$Sdq, Sdr,$$
$$Sal, Str,$$
$$Sk\ Spk,$$
$$Sr1, Sr2\ Svk$$
$$Vmc, Vmp,$$
$$Vvc, Vvv, Sxp$$



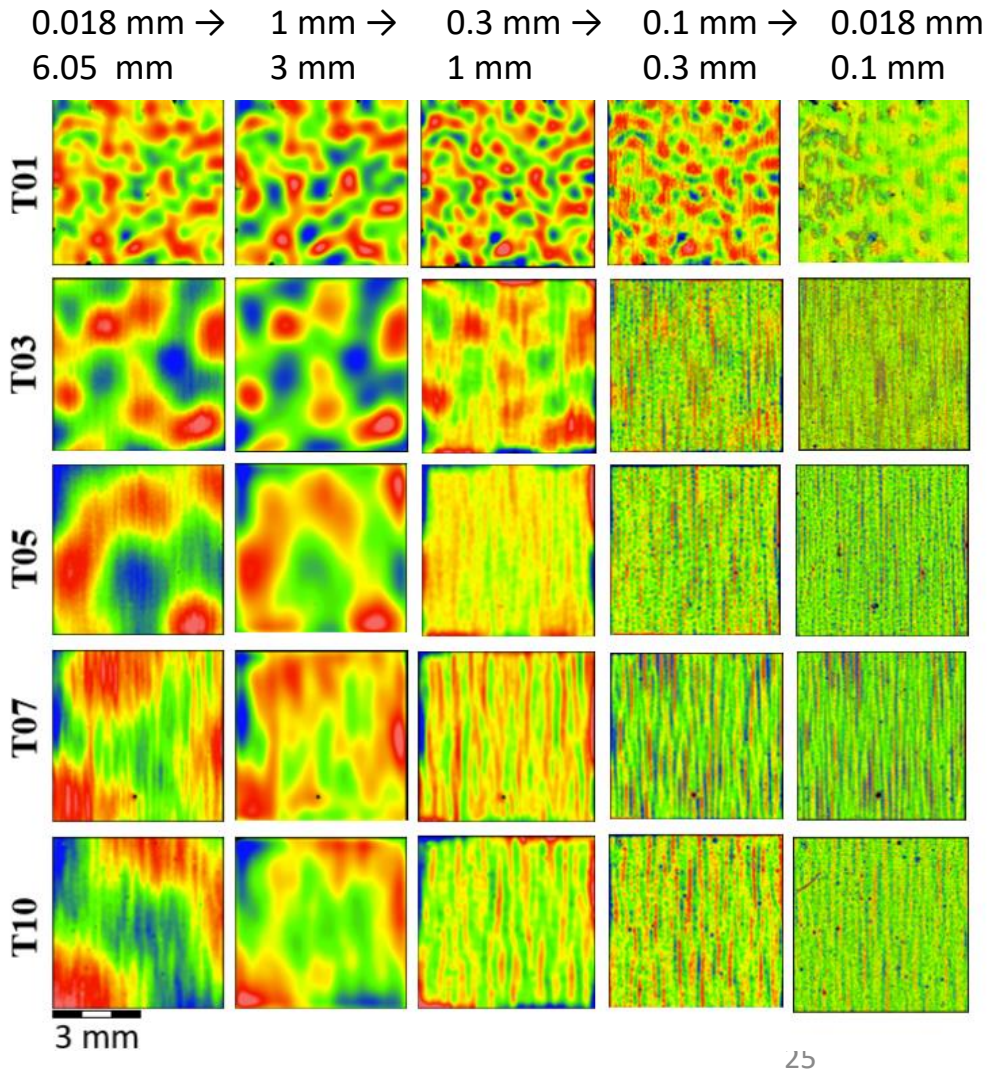
SQuID performance

- 50/50: test/train
- Classification Accuracy*: 65%

*Accuracy: (TP+TN)/(TP+TN+FP+FN)



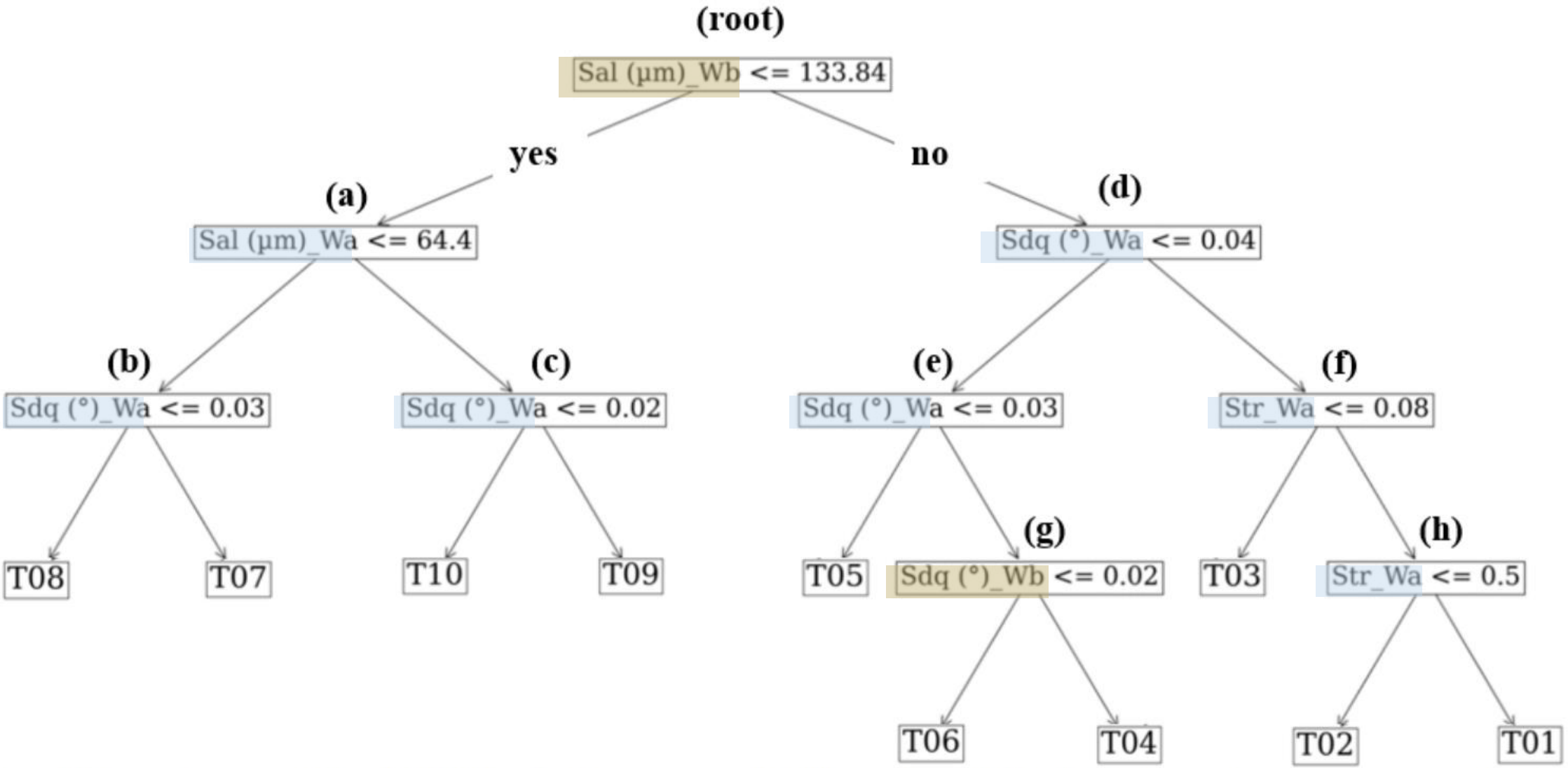
Bandwidth filter the CSI Measurements



SQuID performance

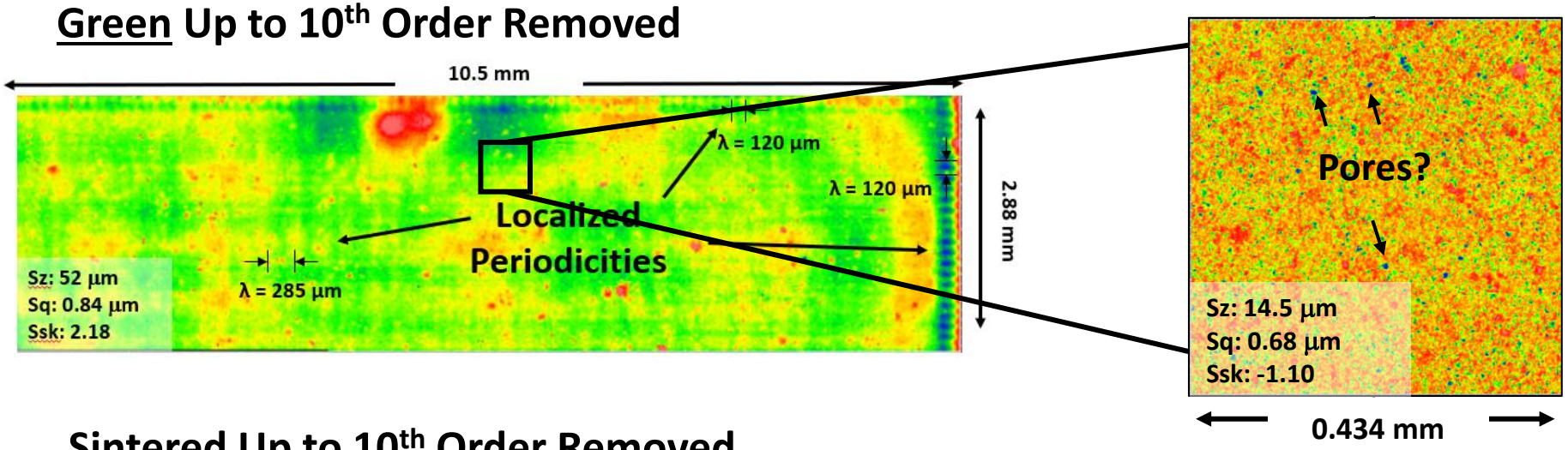
- Isolates top metrics (5) from 88
- Classification Accuracy = 99%

SQuID™

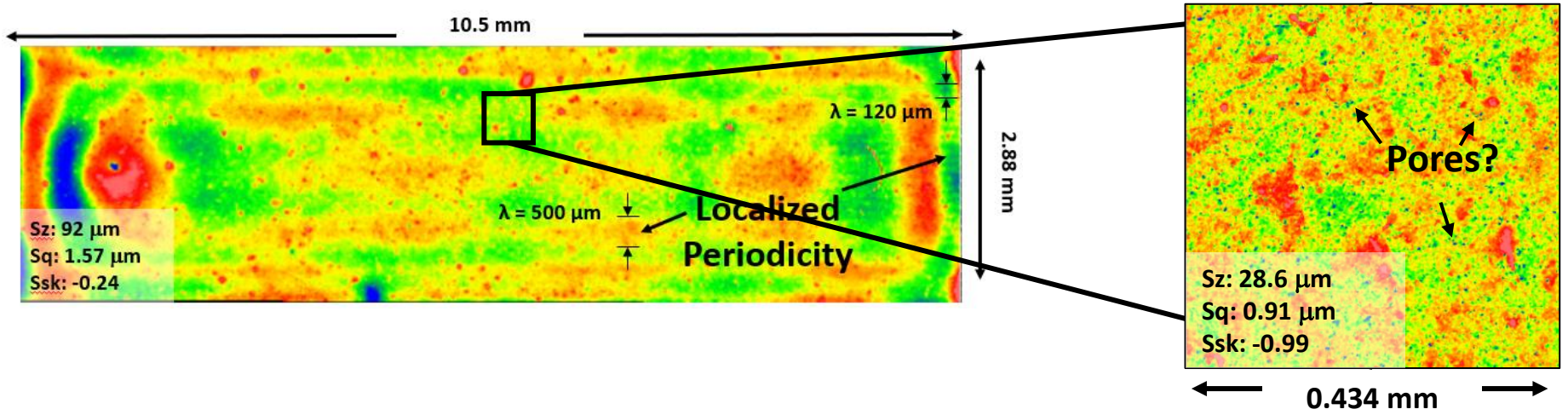

(root)
Sal (µm)_Wb <= 133.84
yes
no
(a)
Sal (µm)_Wa <= 64.4
(d)
Sdq (°)_Wa <= 0.04
(b)
Sdq (°)_Wa <= 0.03
(c)
Sdq (°)_Wa <= 0.02
(e)
Sdq (°)_Wa <= 0.03
(f)
Str_Wa <= 0.08
T08
T07
T10
T09
T05
(g)
Sdq (°)_Wb <= 0.02
T03
(h)
Str_Wa <= 0.5
T06
T04
T02
T01

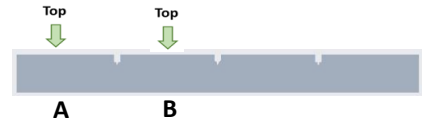
Non ideal Top Surface: Green and Sintered

Green Up to 10th Order Removed



Sintered Up to 10th Order Removed



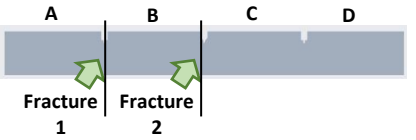


Observations:

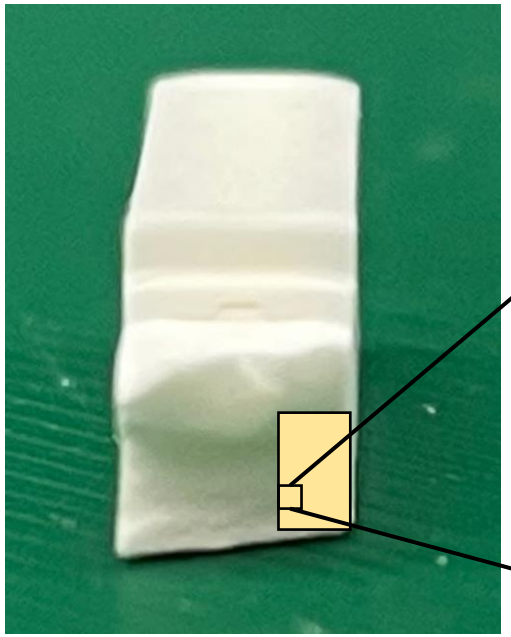
- Potential pores on both surfaces
- Future thresholding segmentation analysis with surface parameter investigations to pursue.
- Sq: 0.68 µm → 0.91 µm

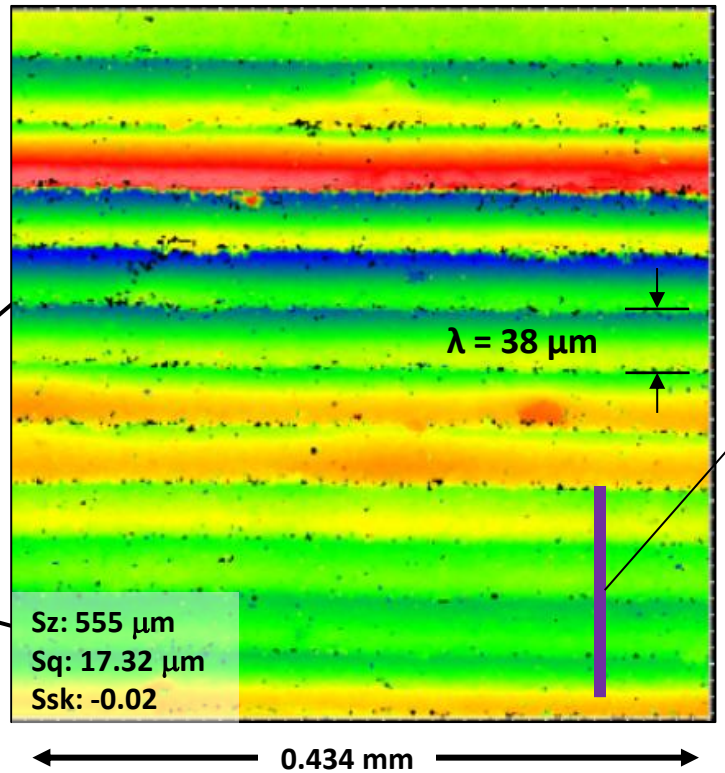
*NOTE: These are not measurements of the same segments.

Non-ideal – Sintered



Sintered
(Fracture 1)





Periodicity Amplitude of Slice

Observations:

- **Height Parameter Values are Affected by Features in Measured Region**

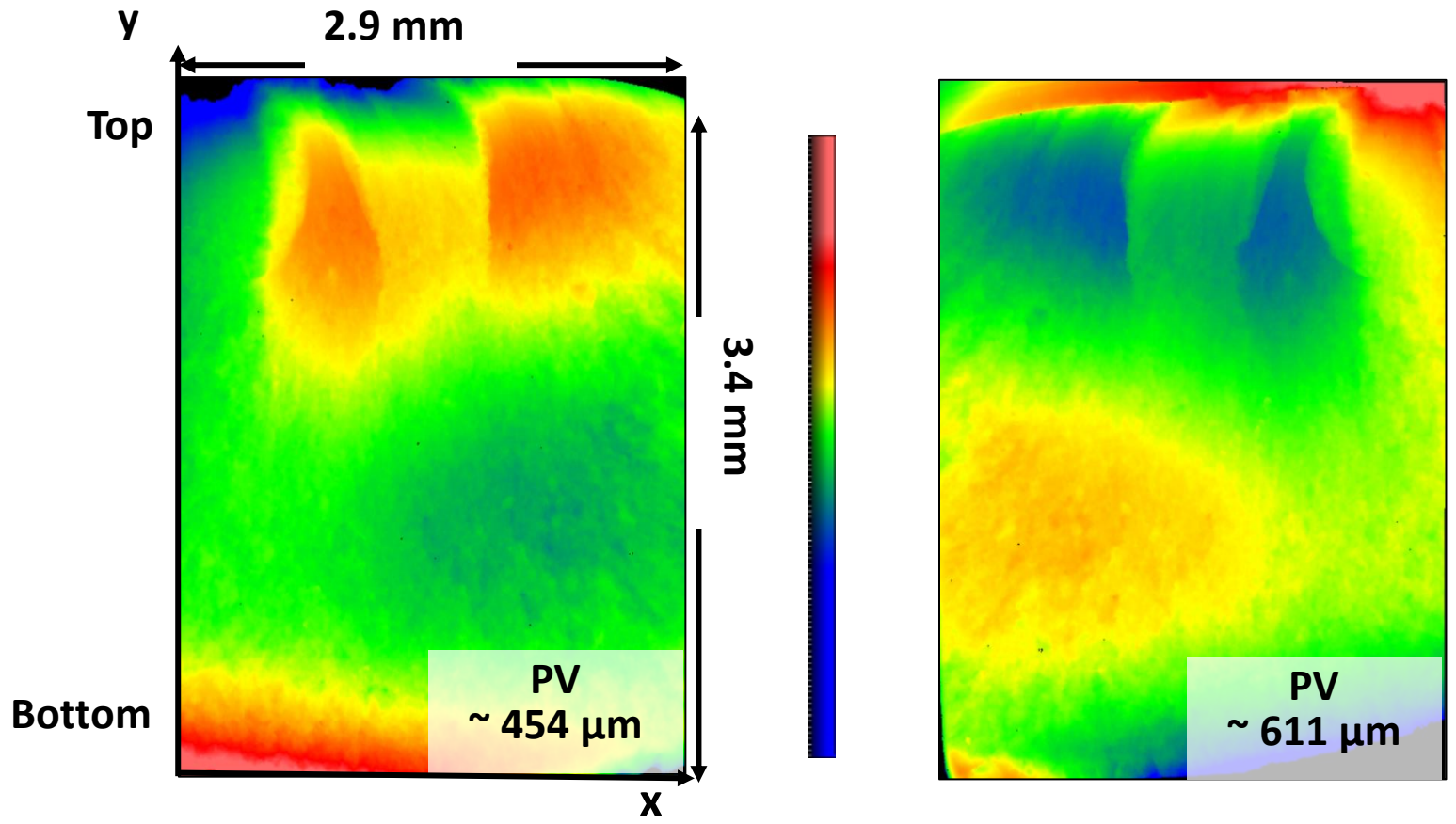
Results of Slice:

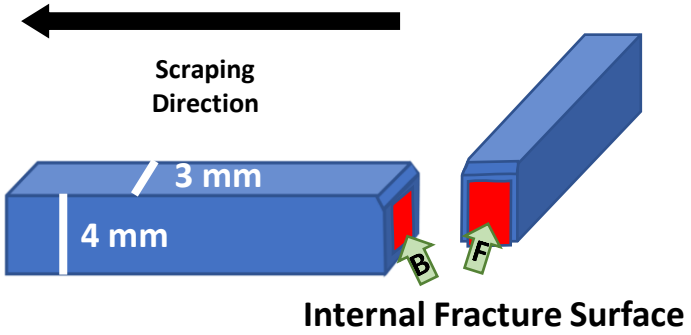
- Sz: 34.44
- Sq: 8.013

28

Non-ideal – Green

20X





No obvious indication of 50 µm layers