SAND2016-11446C

Material science path toward AM material assurance/qualification

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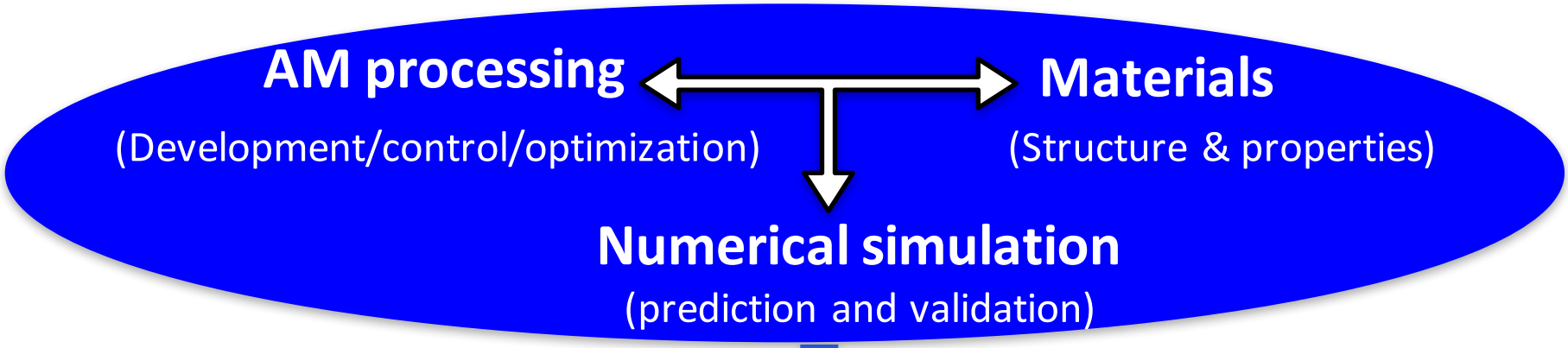
Outline

- Programmatic objective and technical path
- Fundamentals of powder-based 3-D LENS- and PBF- printing
- Material properties investigated for LENS/PBF printed SS316L
- On-going activities and future technical path
- Summary

Technical objective path forward

3-way technical integration



Establish

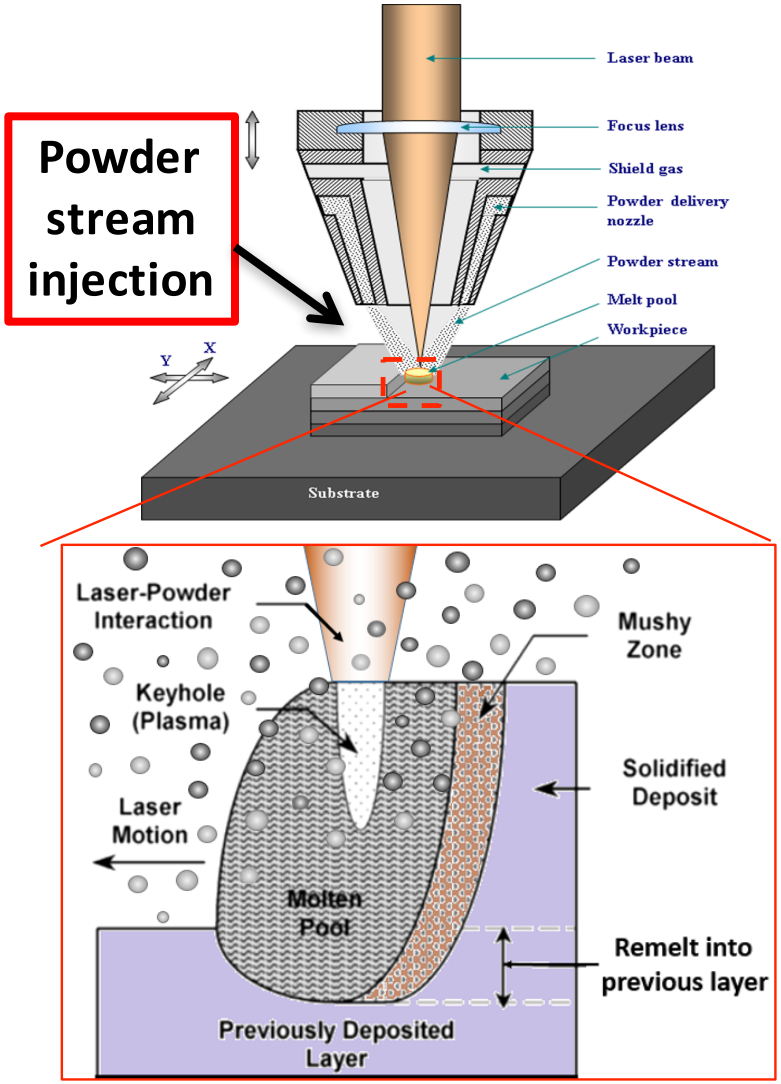
- Process-structure-property relationships
- Geometry/ precision & limitation/ manufacturing constraint
- Material property control and defect mitigation

achieve

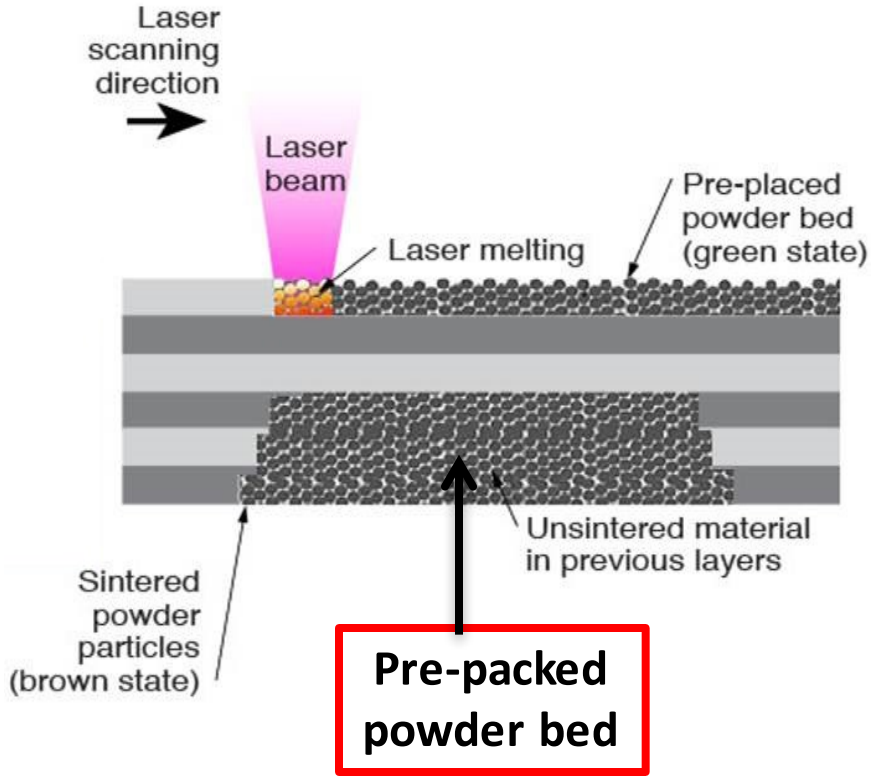
AM Material assurance for W80-4 LEP AM system engineering

Fundamentals for powder-based 3-D LENS & PBF AM printing

Laser Engineered Net Shaping (LENS)



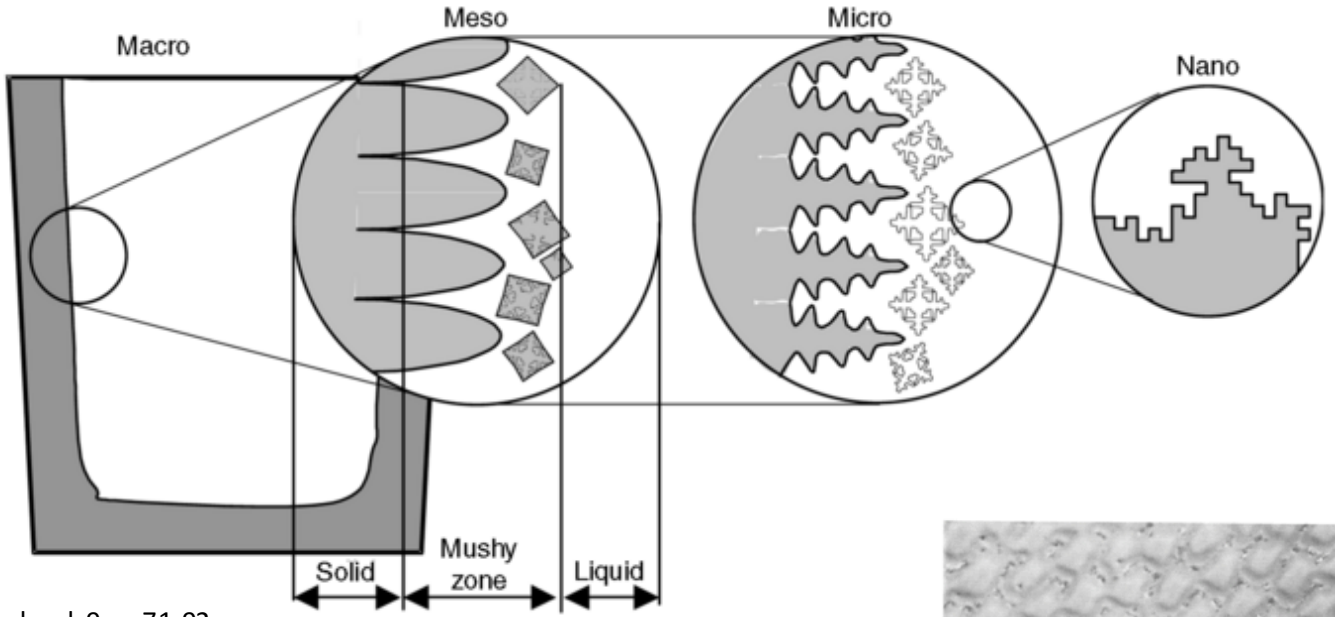
Powder Bed Fusion (PBF)



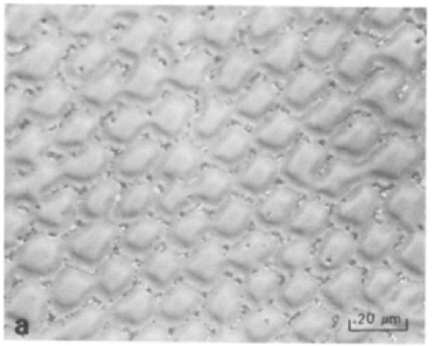
Printing steps common to LENS & PBF

- Powder melting
- Molten metal fusion
- Molten metal solidification

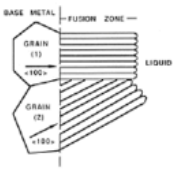
Alloy solidification basic and solidified cell morphology

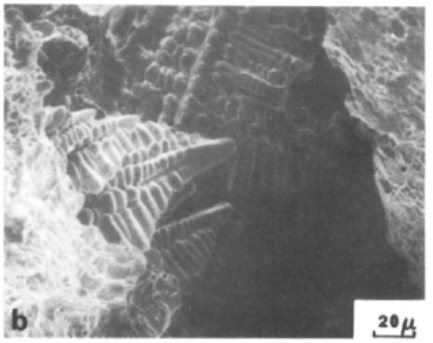


ASM Handbook vol. 9 pg 71-92

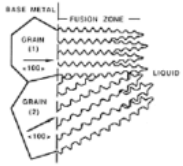


Cellular cell





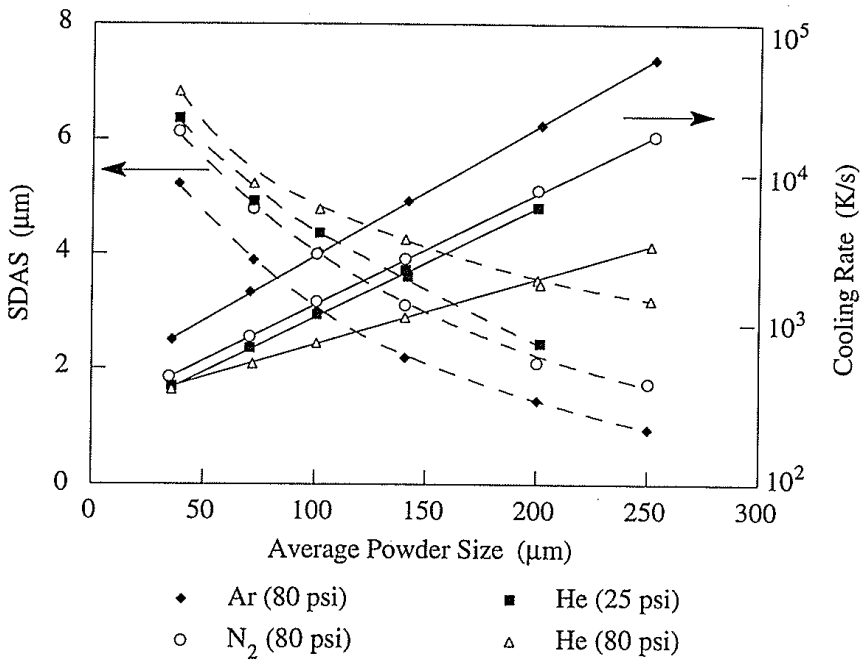
Dendrite

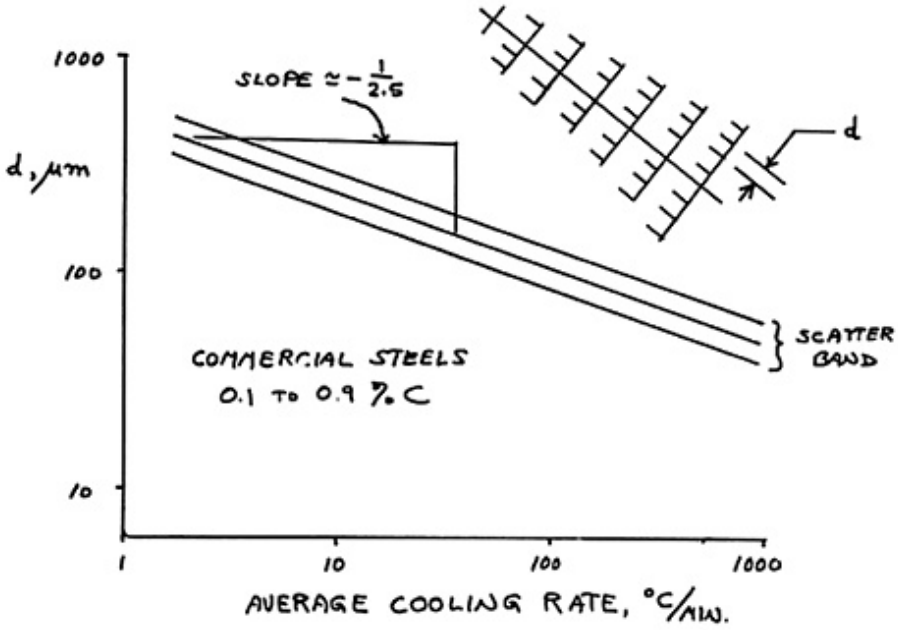


Solidification cooling rate impacts microstructure and chemical uniformity of 3-D AM print.

Secondary dendrite arm spacing (SDAS) decreases with increases in solidification cooling rate

For Al (Lavernia's Hand Book)





Solidification cooling rate impacts microstructure and chemical distribution of the finished components.

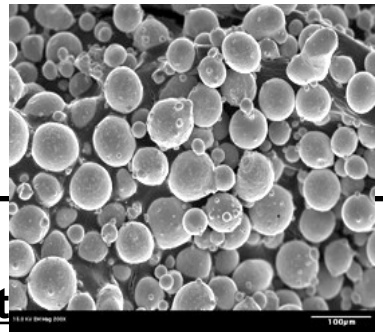
SDAS can be empirically related to cooling rates:

Where

SDAS=secondary dendritic arm spacing
k_c=empirically determined constant
dT/dt=cooling rate
n=empirically determined constant

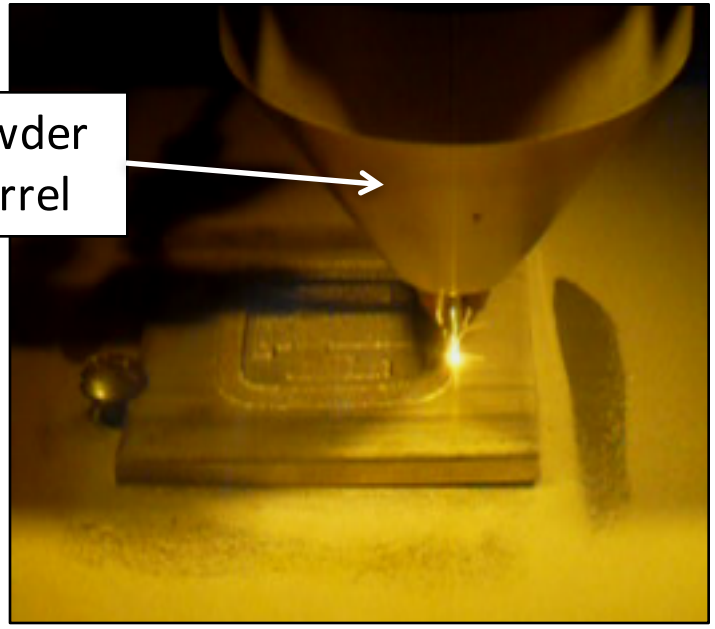
3-D LENS and PBF metal printing demonstration

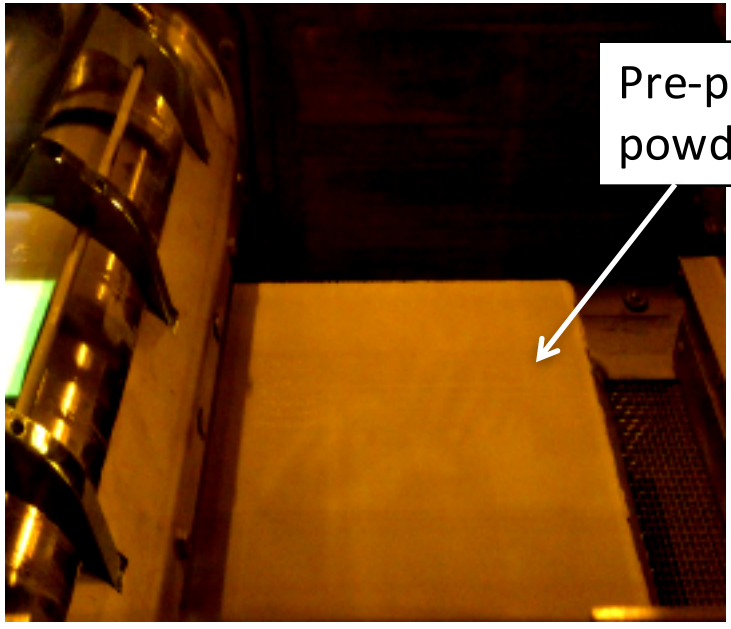
Starting SS316L atomized feedstock powders



3-D LENS deposit

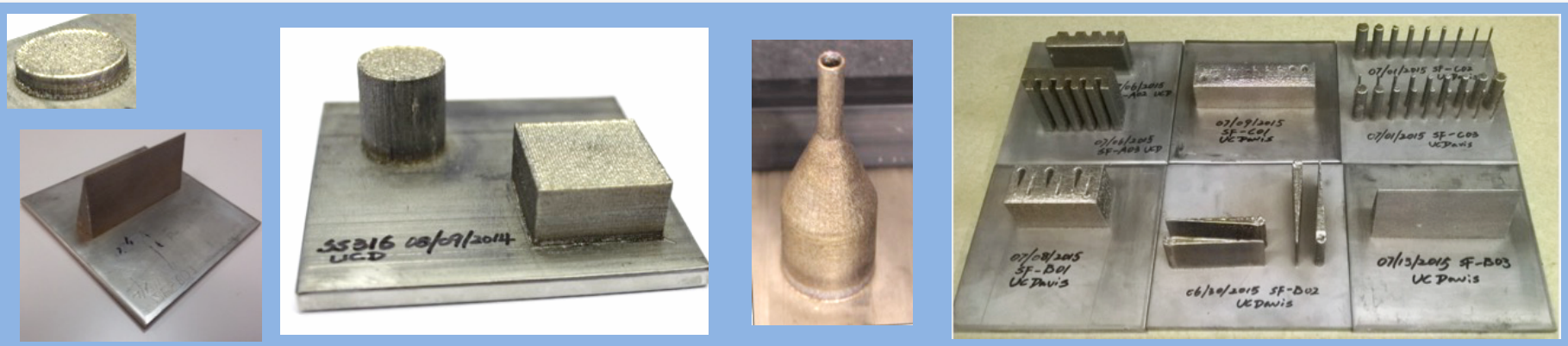
3-D PBF printing





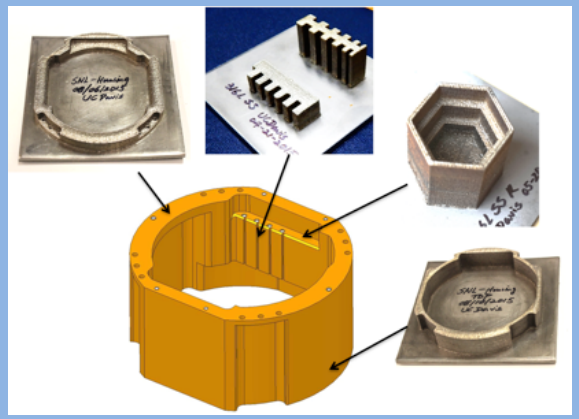
3-D LENS and PBF prototypes printed at UCD/UCI and SNL, CA

3-D LENS prints for material science R&D

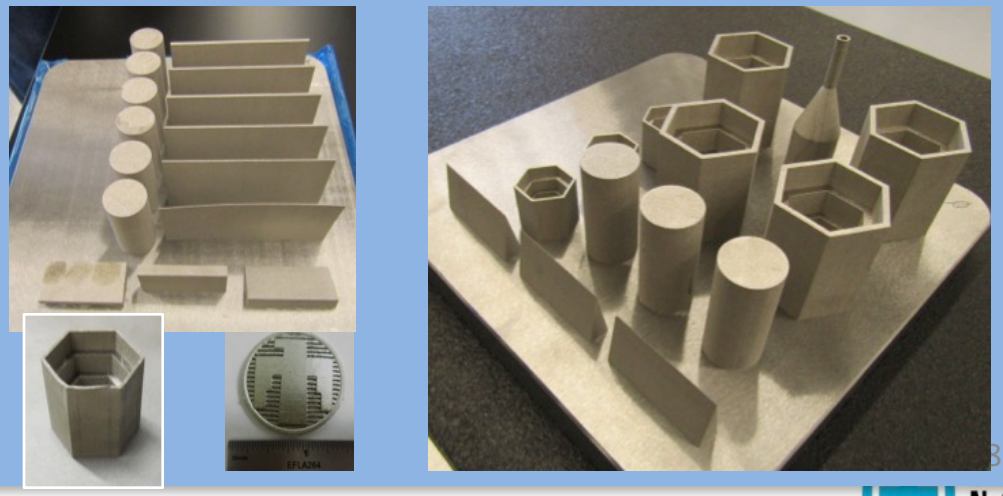


3-D AM for prototyping feasibility

3-D LENS prints using Optomec



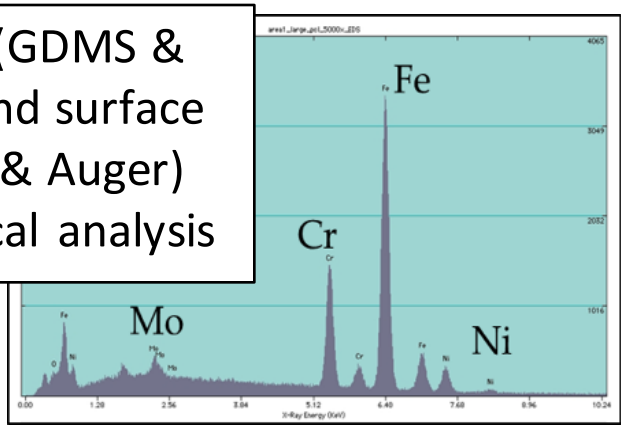
3-D PBF print by ProX300 system



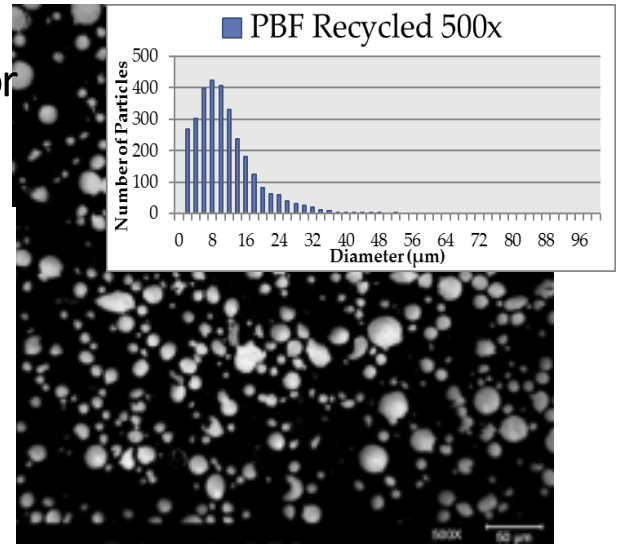
Processing and/or product screening/QA/QC analytical tools

For Powder

Bulk (GDMS & ICP) and surface (EDS & Auger) chemical analysis

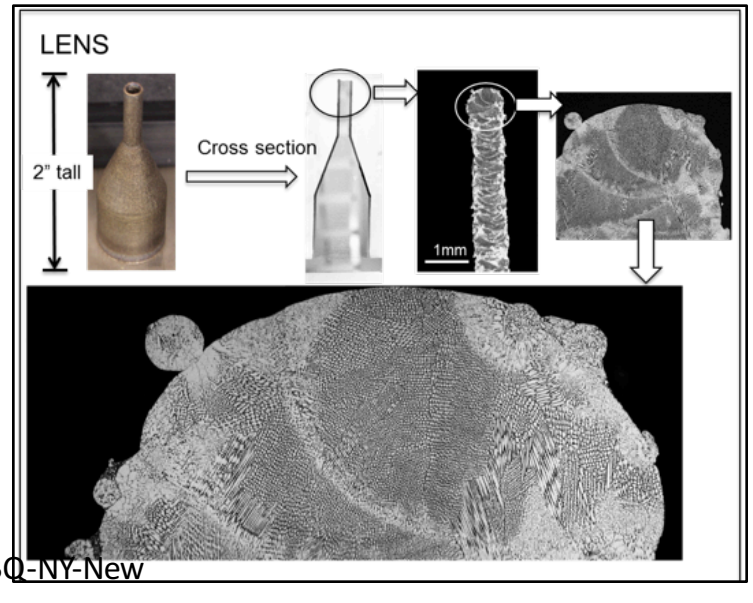


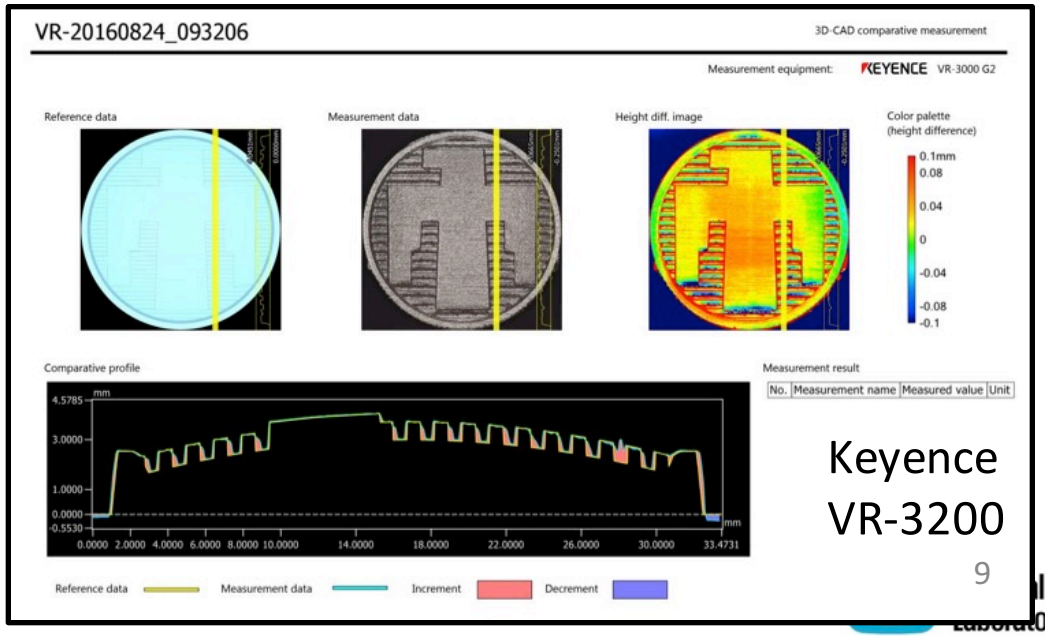
Particle Insight for size & shape



For Printed Part

Metallography for microstructure





Keyence VR-3200

Material properties investigated for AM printed SS316L investigated

- **3-D LENS and PBF printing induced physical metallurgy & structural irregularities**
  - ✓ *Surface topography and contour*
  - ✓ *Metallurgical characteristics*
  - ✓ Solidification microstructure evolution and geometry factors
  - ✓ Structural irregularity and its origin or root cause
  - ✓ Mechanical behavior & engineering properties of AM SS 316L
- **Summary Conclusion**

Subjects for discussion

- **3-D LENS and PBF printing induced physical metallurgy & structural irregularities**

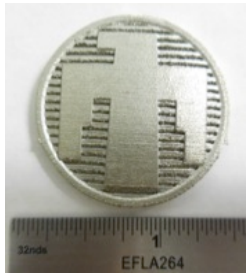
  ✓ *Surface topography and contour*
  ✓ *Metallurgical characteristics*
  ✓ Solidification microstructure evolution and geometry factors
  ✓ Structural irregularity and its origin
  ✓ Mechanical behavior & engineering properties of AM SS 316L

Unmelted powder deposition is a common characteristics on the surface of all PBF- & LENS- prints

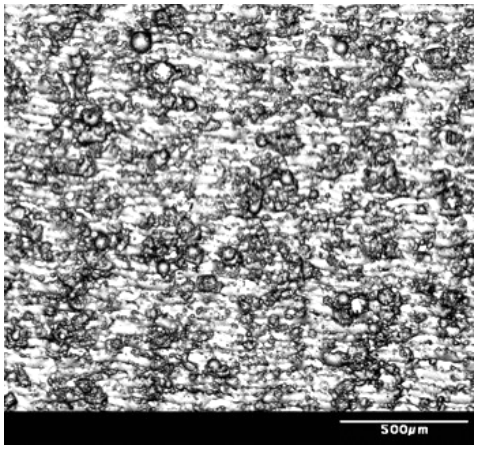
PBF by GPI

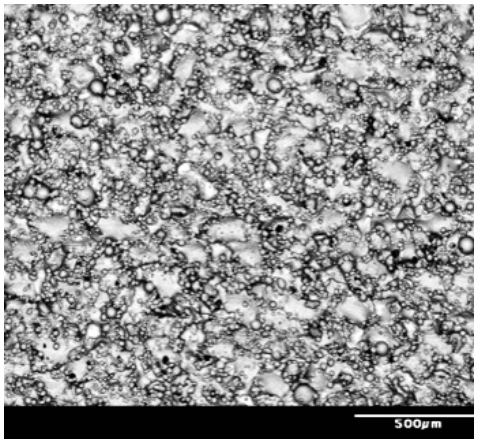
PBF by SNL,CA

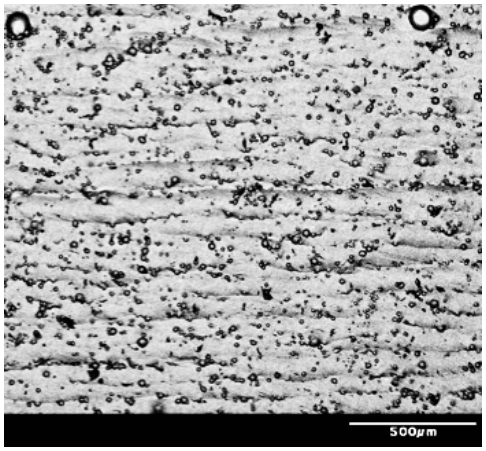
PBF by SNL, CA

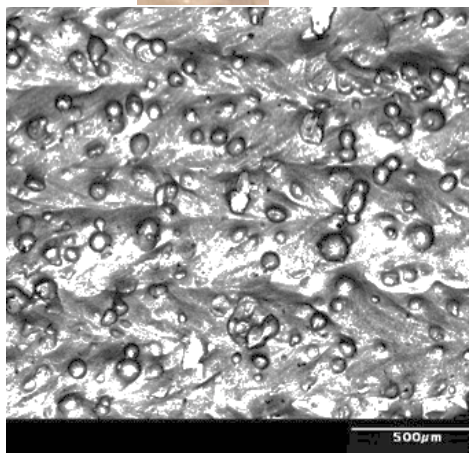


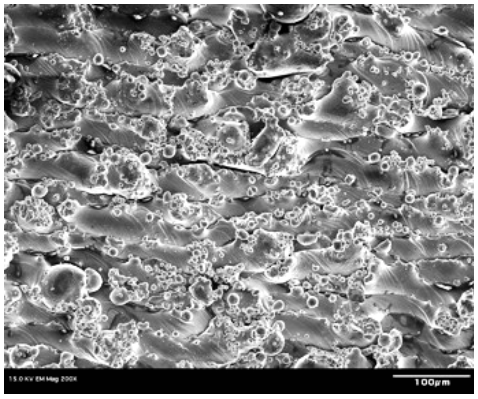
LENS by UCD

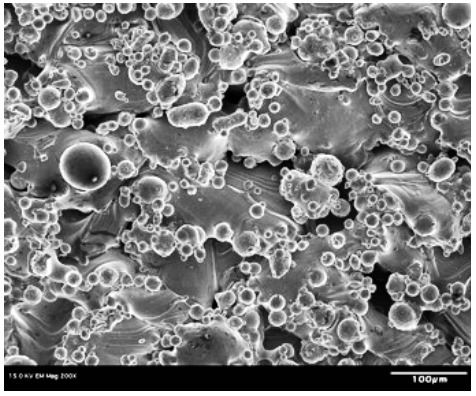


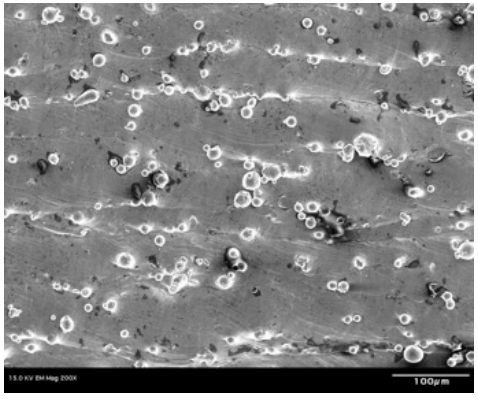


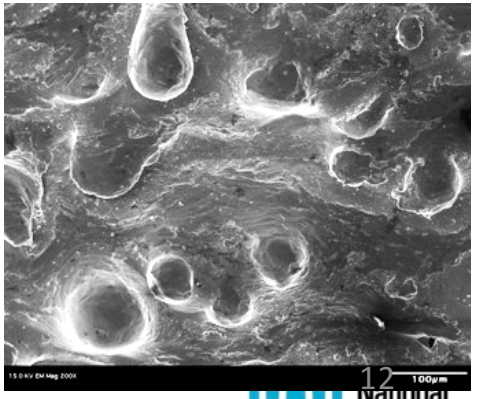








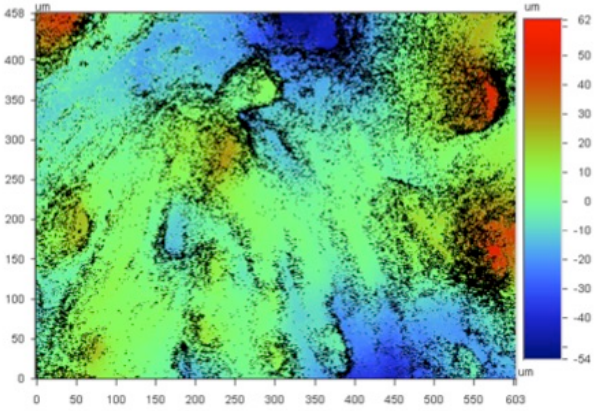




Presence of unbelted powder results in Irregular surface morphology & contour on LENS- and PBF- print

LENS Funnel (sidewall)

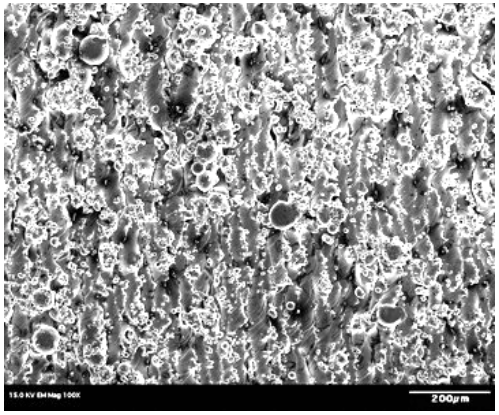
Printing direction ↑

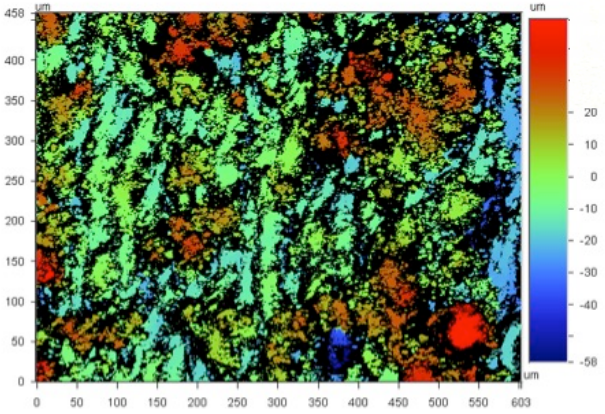


Peak/Valley

Rz ~110µm

PBF Hexagon (sidewall)



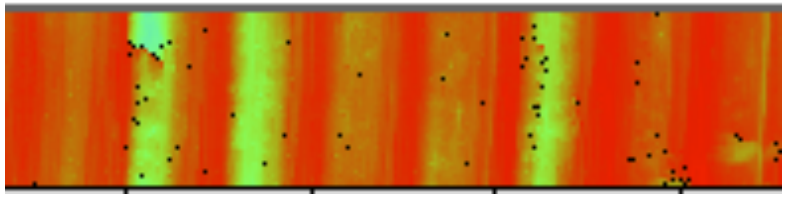


Peak/valley

Rz~100µm

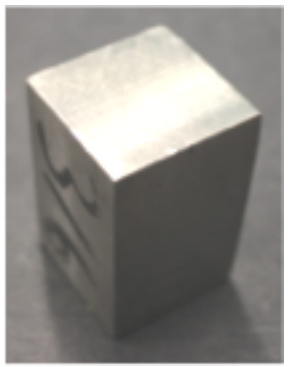
Surface topography of LENS print is less consistent or unpredictable than those by mechanical machining

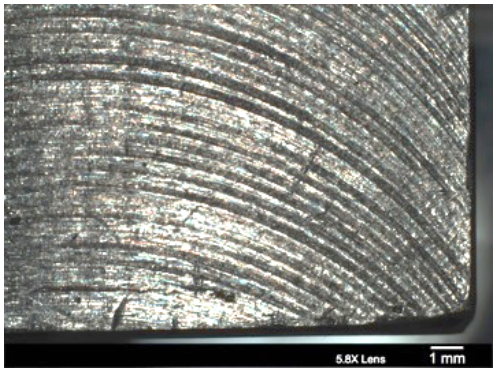
Mechanically machined

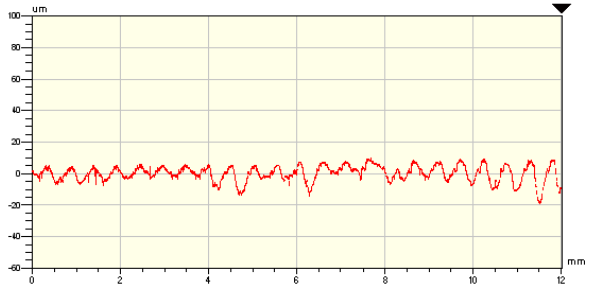


Rz: 37.60 um (Average)

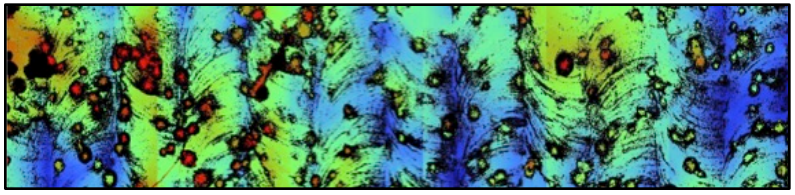
Rt: 58.54 um (Peak –valley)





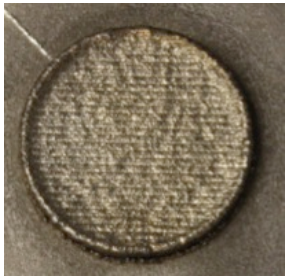


LENS printed



Rz: 326.24 um (Average)

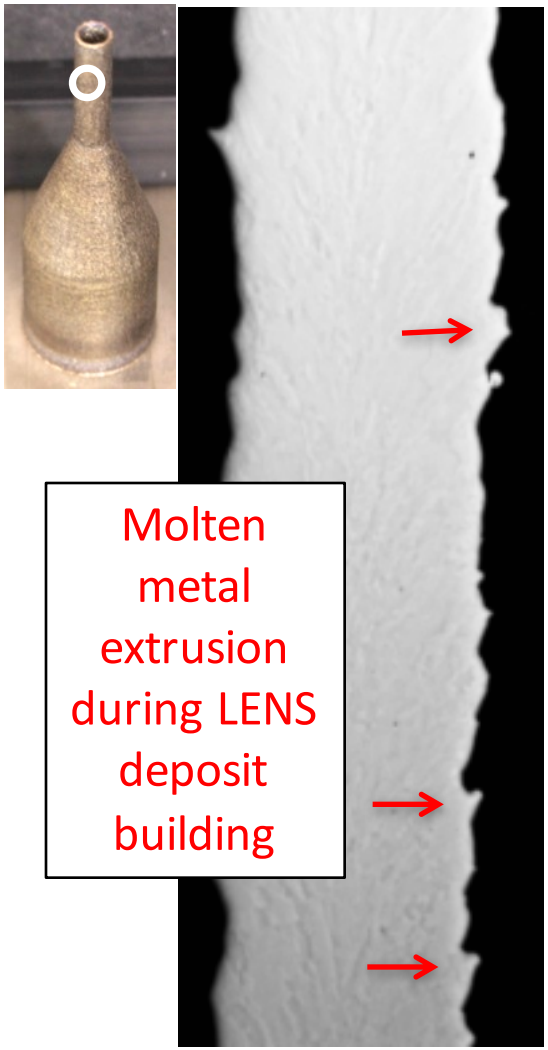
Rt: 418.07 um (Peak –valley)



Polished cross sections show different surface contour between LENS and PBF print

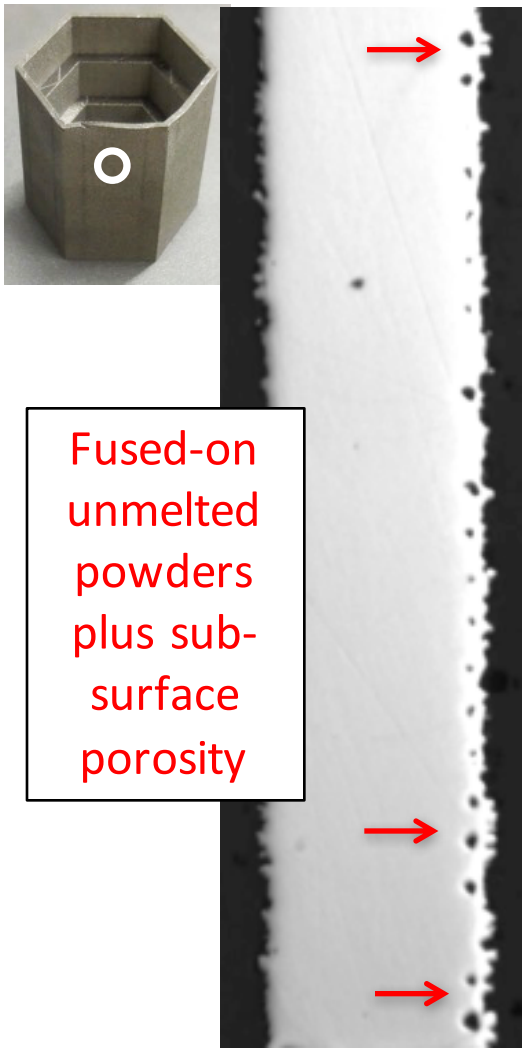
Thin sidewall cross section, ~1mm thick

UCD LENS funnel)



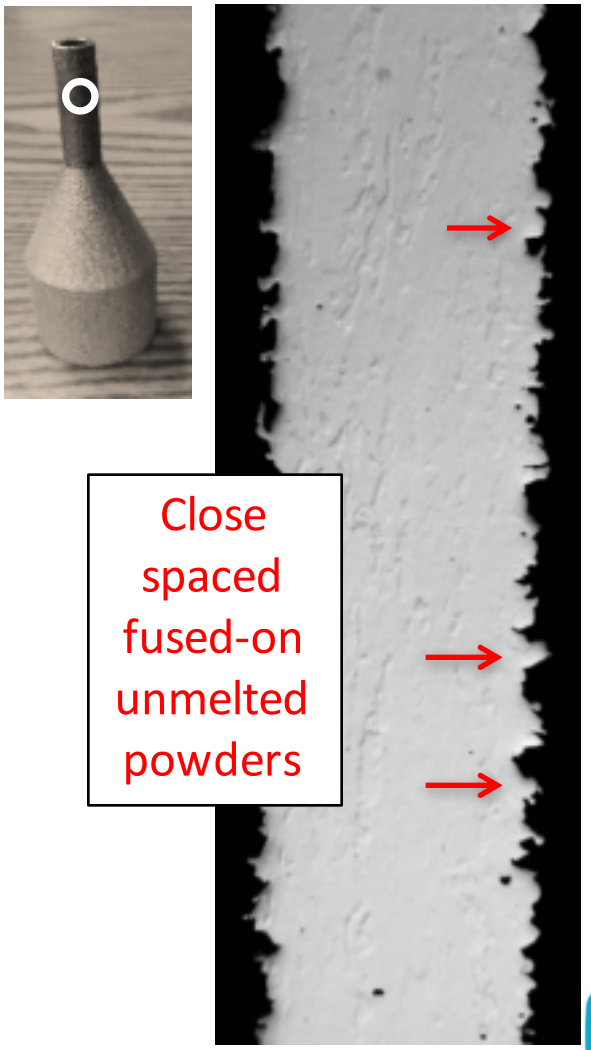
Molten metal extrusion during LENS deposit building

GPI PBF hexagon



Fused-on unmelted powders plus sub-surface porosity

SNL,CA PBF funnel

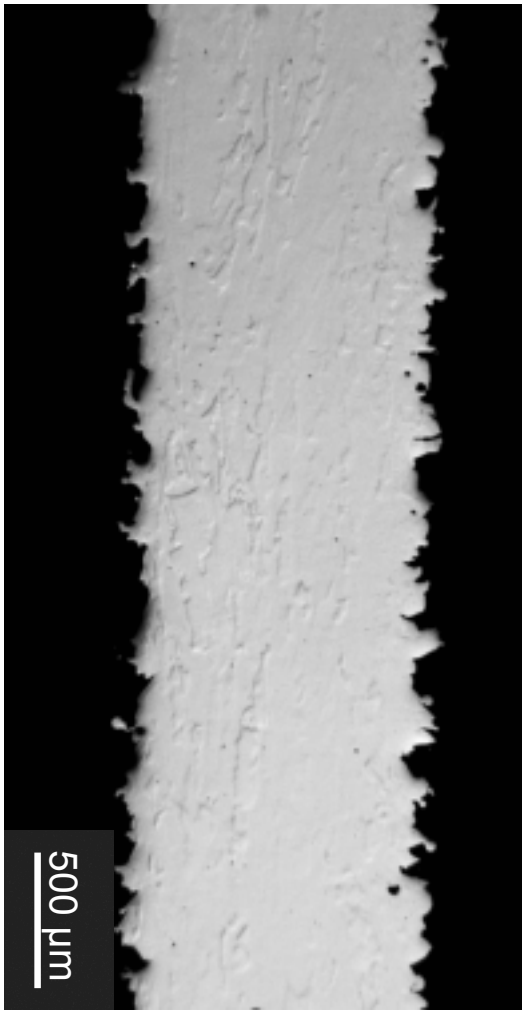


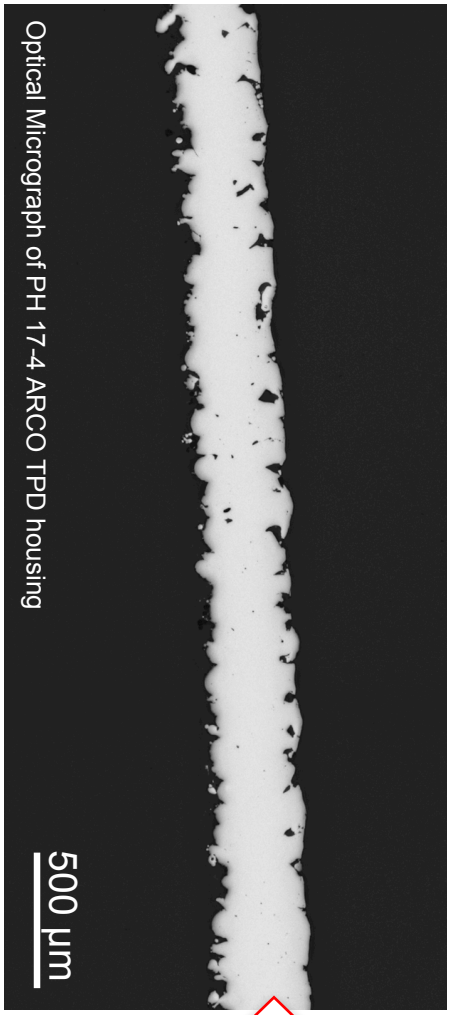
Close spaced fused-on unmelted powders

Building direction

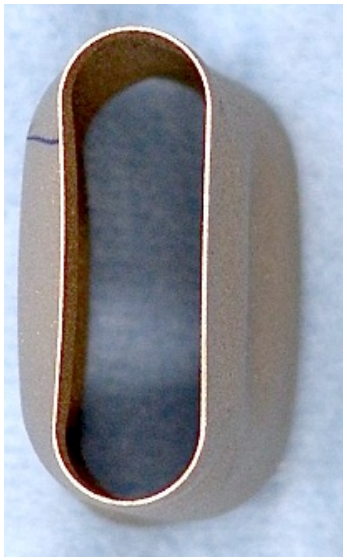
Rough PBF surface contour increases its proportion, therefore, raises its influence on the print property.

SS3116L





17-4 thin



J. Rodelas, 2015

At some locations, the rough surface contour took up ~30-40 % of the sidewall thickness.

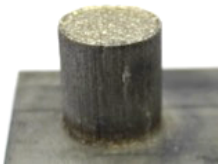
Subjects for discussion

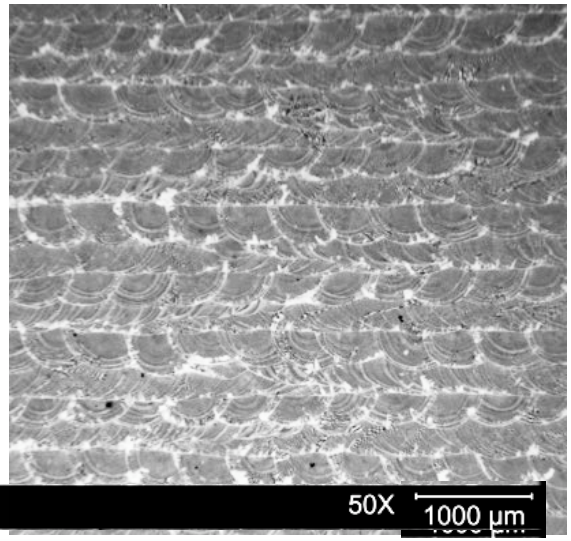
- **3-D LENS and PBF printing induced physical metallurgy & structural irregularities**

  - ✓ *Surface topography and contour*
  - ✓ *Metallurgical characteristics*
  - ✓ Solidification microstructure evolution and geometry factors
  - ✓ Structural irregularity and its origin
  - ✓ Mechanical behavior & engineering properties of AM SS 316L

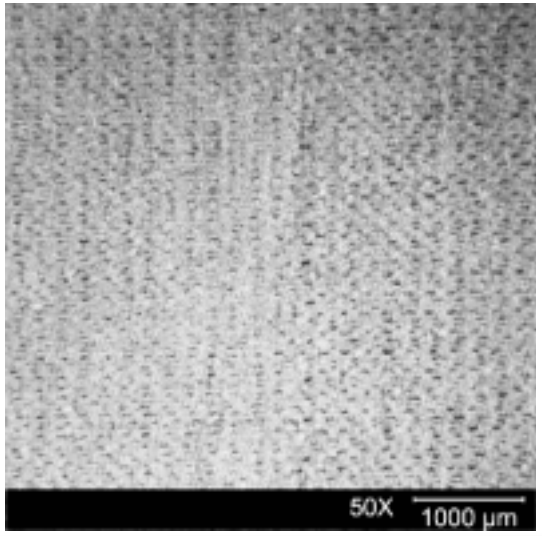
Chemical etch reveals very different solidification landscape between LENS- and PBF- SS316L prototype

3-D LENS print by UCD



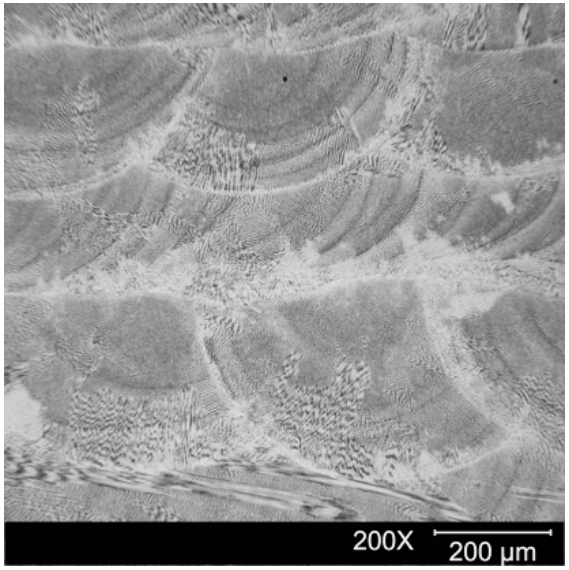


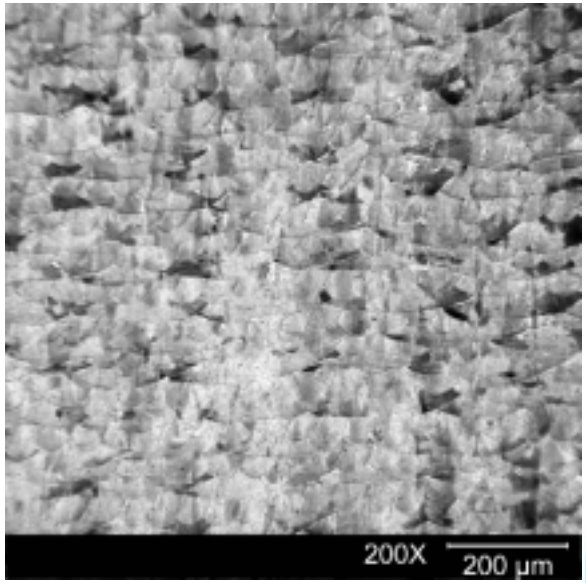
PBF print by GPI



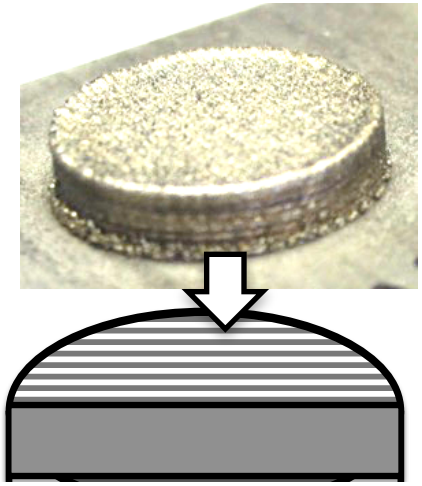
Modulated landscape derived from repetitive metal building →

Much coarser modulation in LENS print →

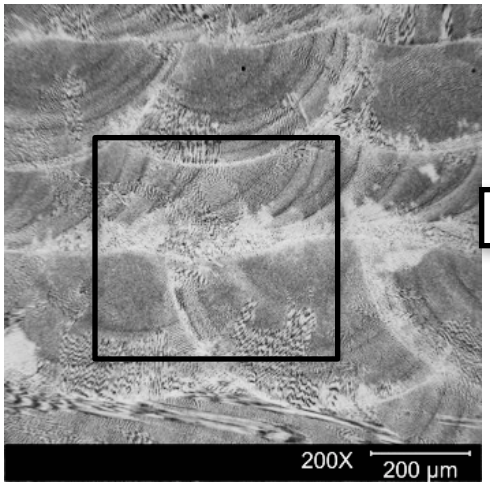


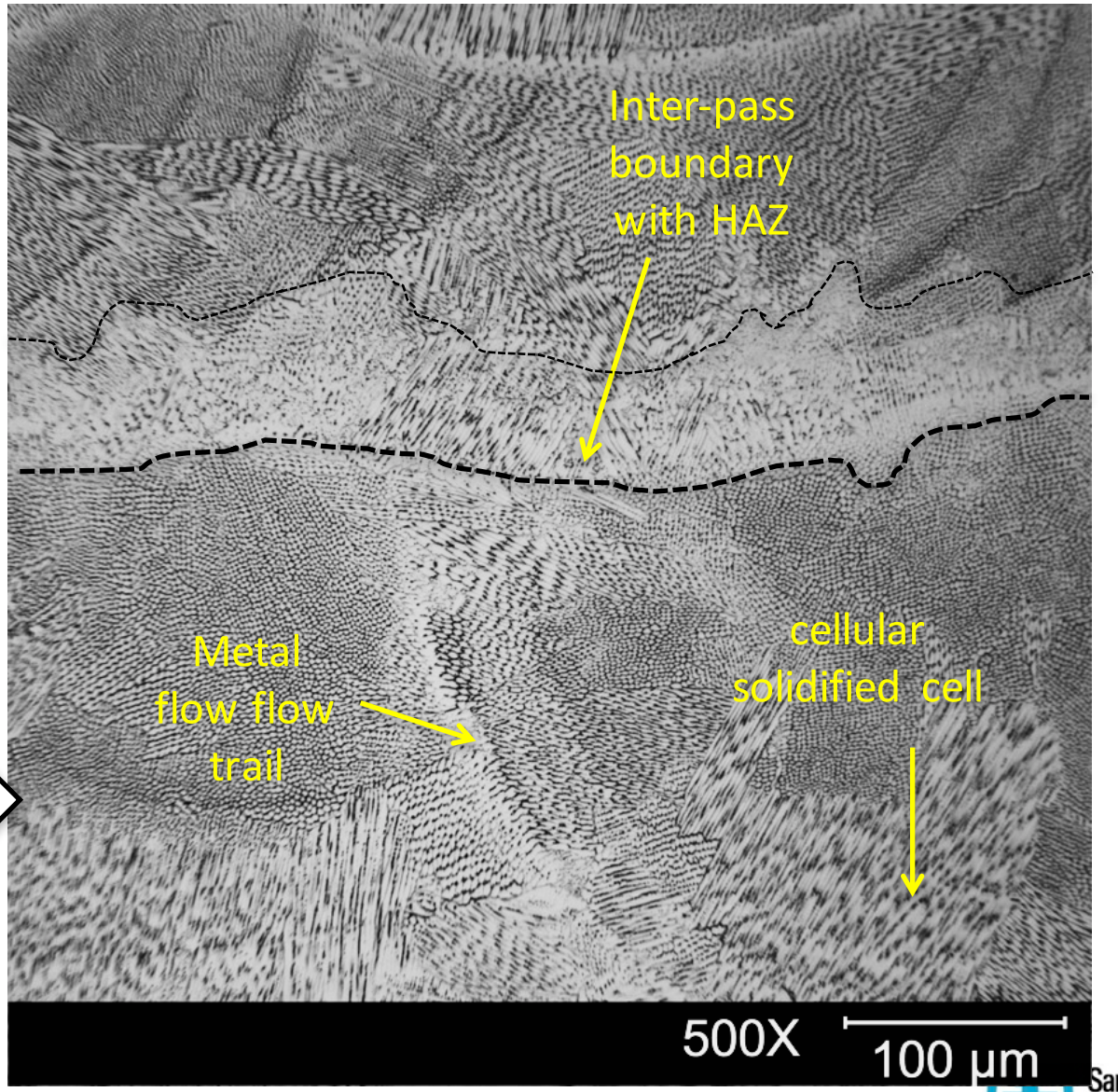


LENS print contains Interpass boundaries with Heat Affect Zone (HAZ), Cellular solidified cells and molten metal flow trails

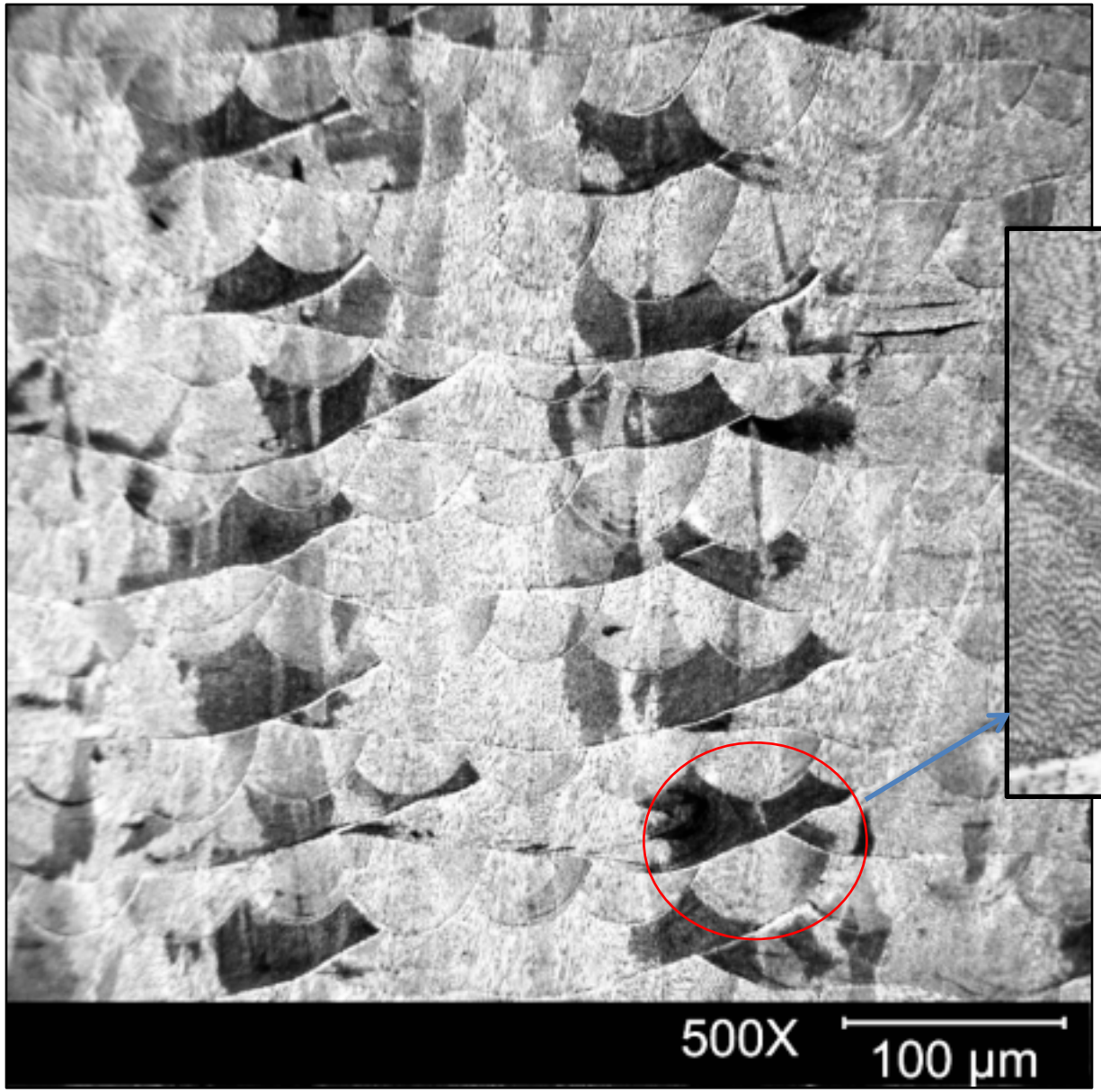


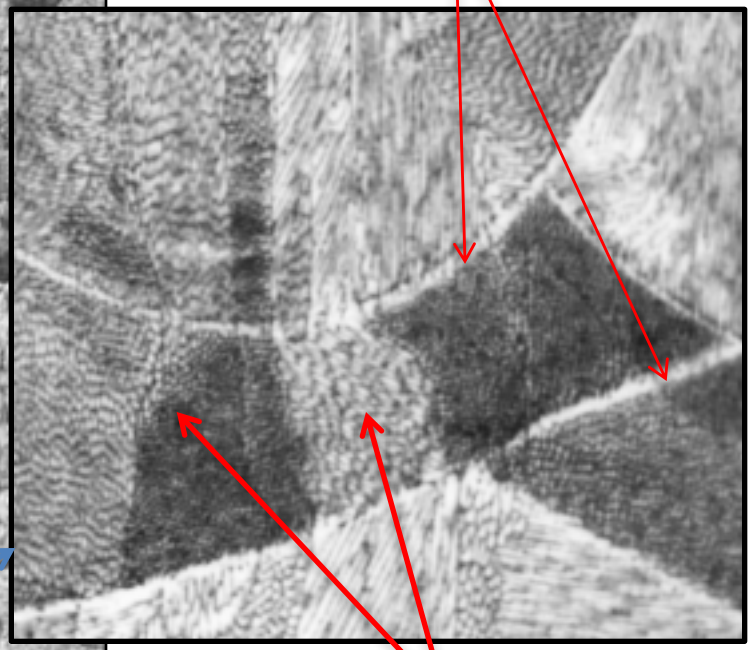
Solidification landscape, revealed by chemical etching





PBF print contains cellular solidified cells, with sharp molten powder fusion interfaces

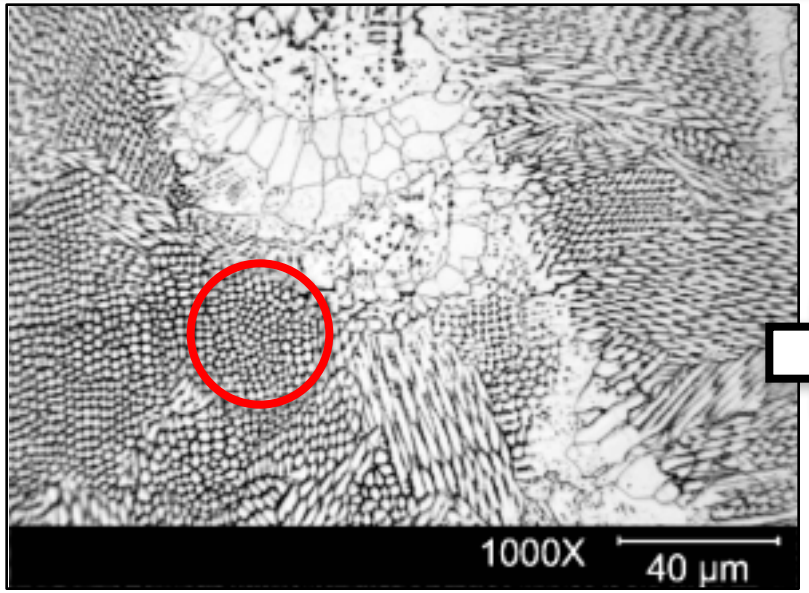


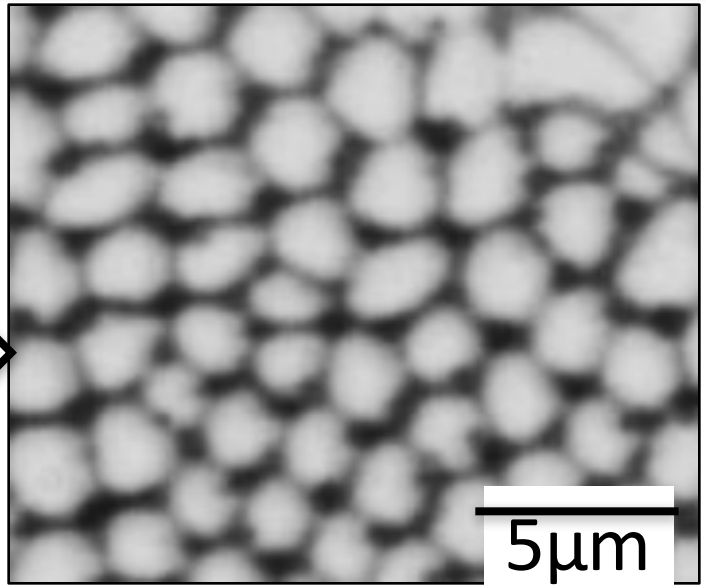


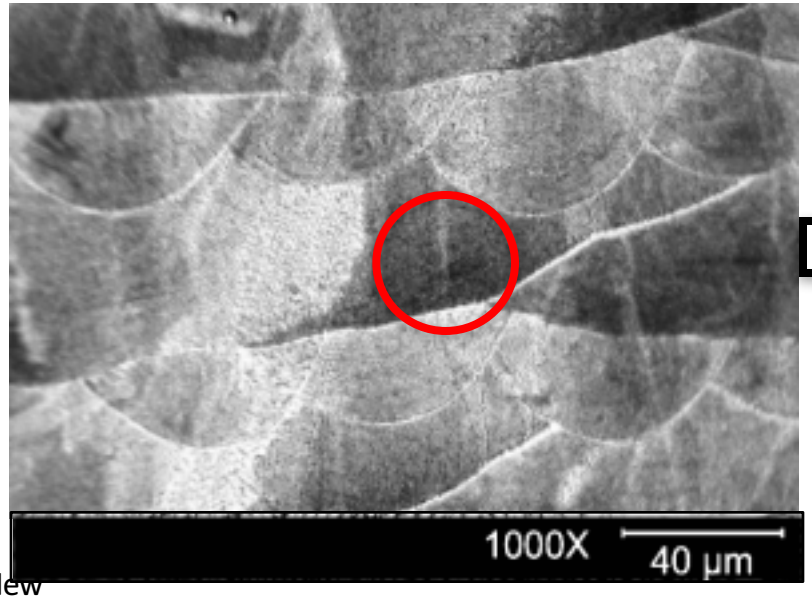
Molten metal fusion interface

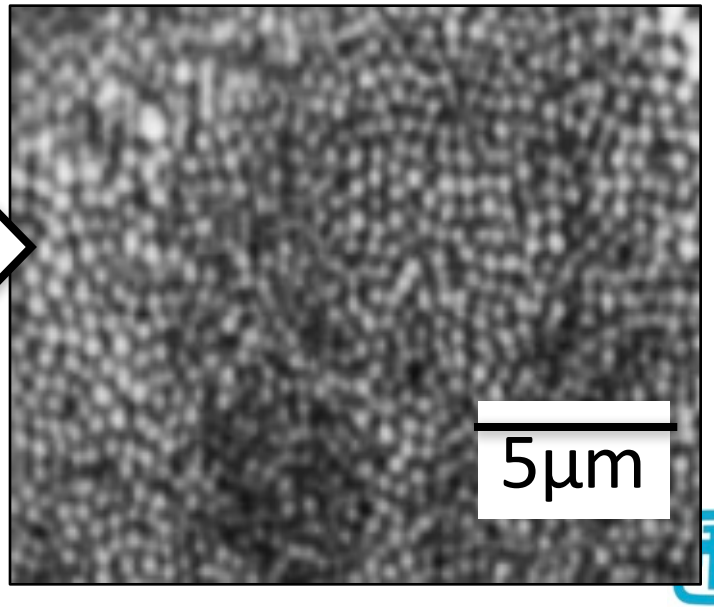
Cellular solidified cell with extremely barely resolved cellular arm spacing, << 5µm

Large SDAS in LENS than in PBF, 3µm vs. ≤1µm, implies much slower cooling rate relative to PBF solidification cooing



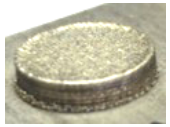




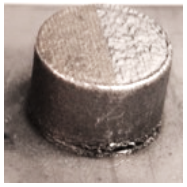


At a given printing condition, 3-D LENS solidification landscape varied with geometry & hatch pattern dependent

Thin disc:
~ 4mm thick

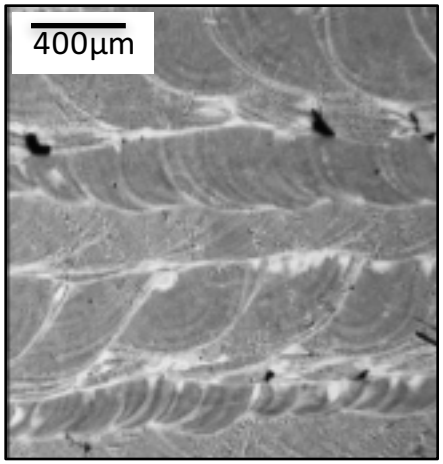


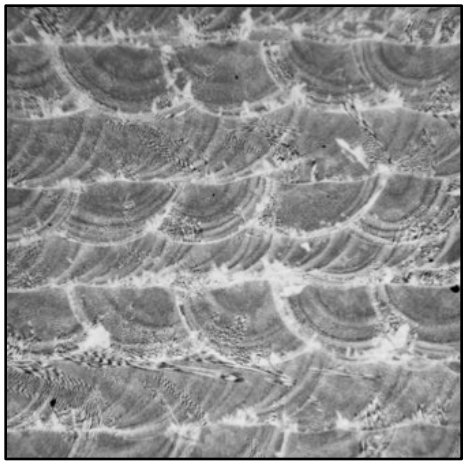
Bulk cylinder
~25mm thick

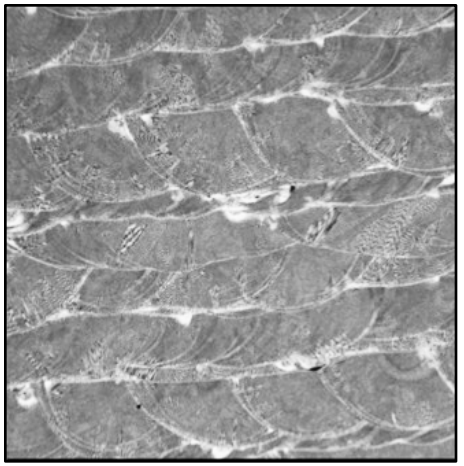


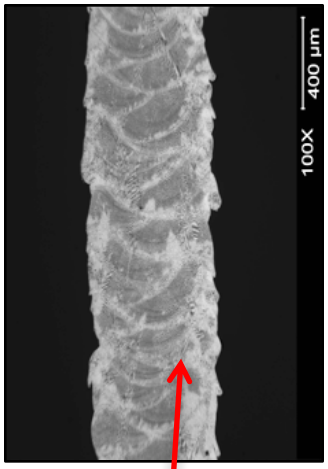
3-tier hexagon

Thin wall funnel





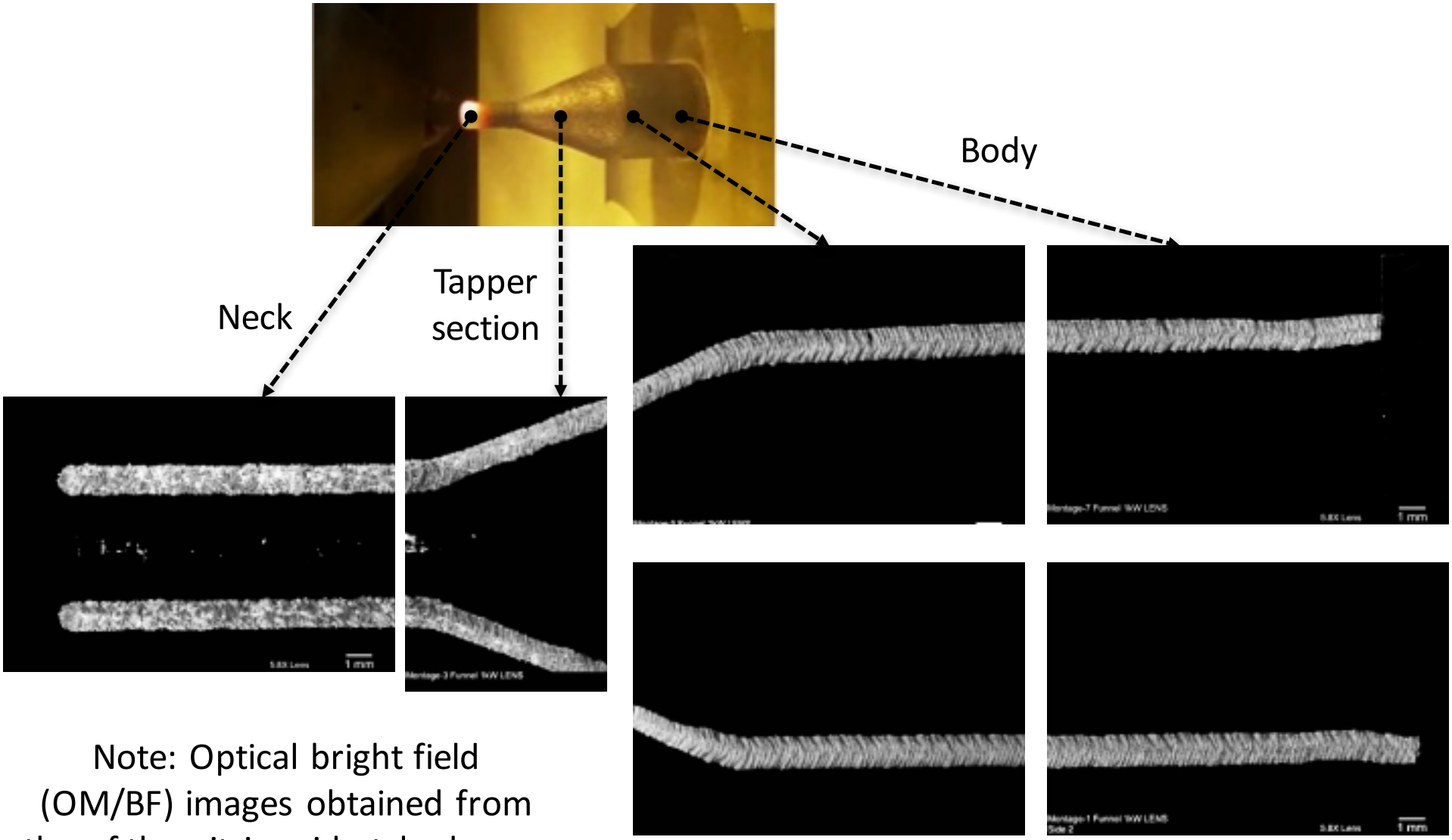




3-D built direction

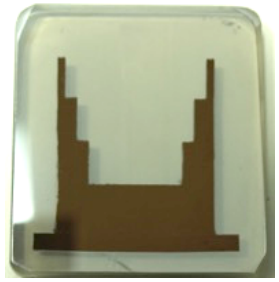
Absence of horizontal straight interpass boundary

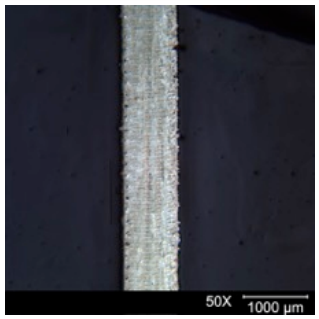
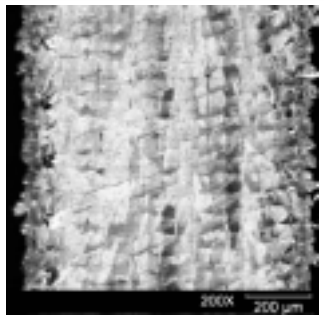
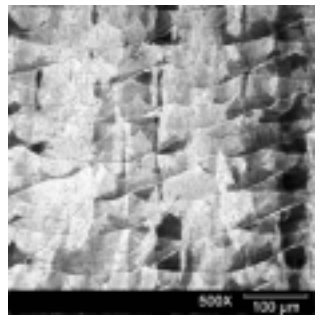
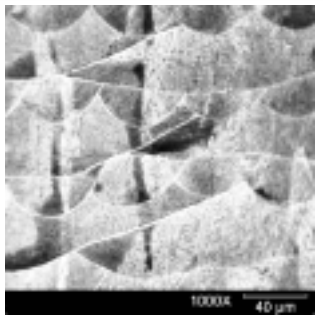
Solidification morphology in the chemical etch UCI funnel, is location dependent, relative to the substrate interface



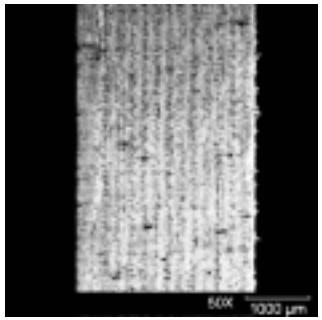
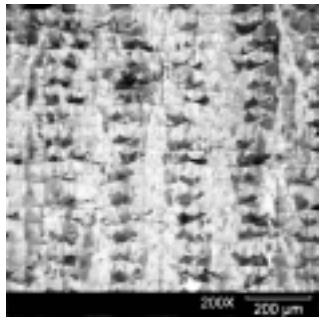
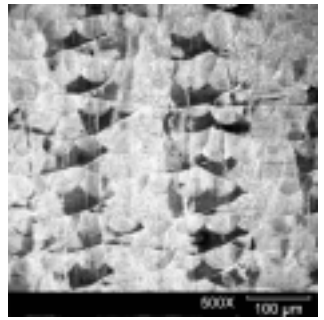
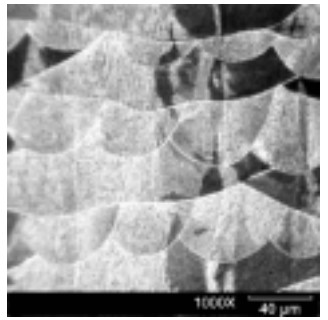
Note: Optical bright field (OM/BF) images obtained from the of the nitric acid etched cross section

Solidification landscape is more consistent, independent of geometry, throughout PBF hexagon



Tier 1

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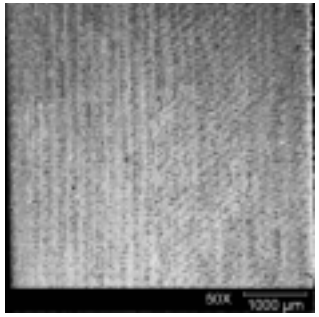
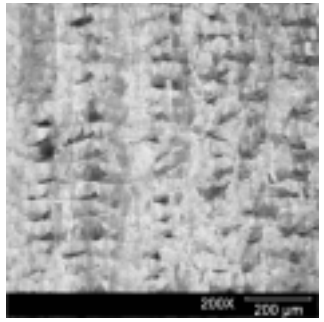
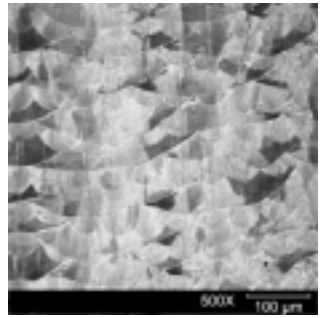
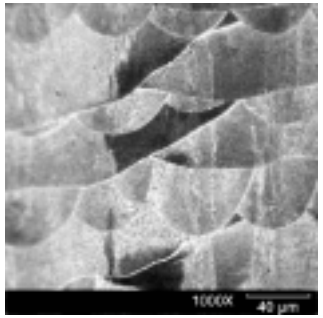

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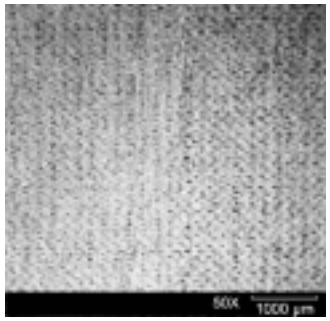
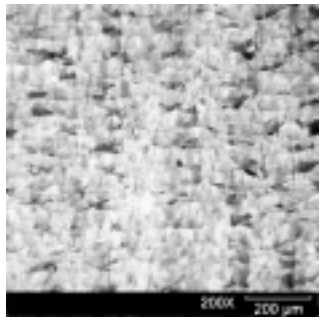
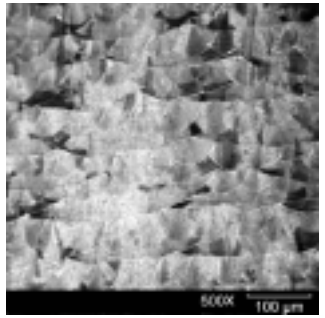
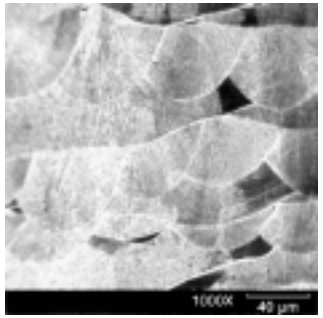
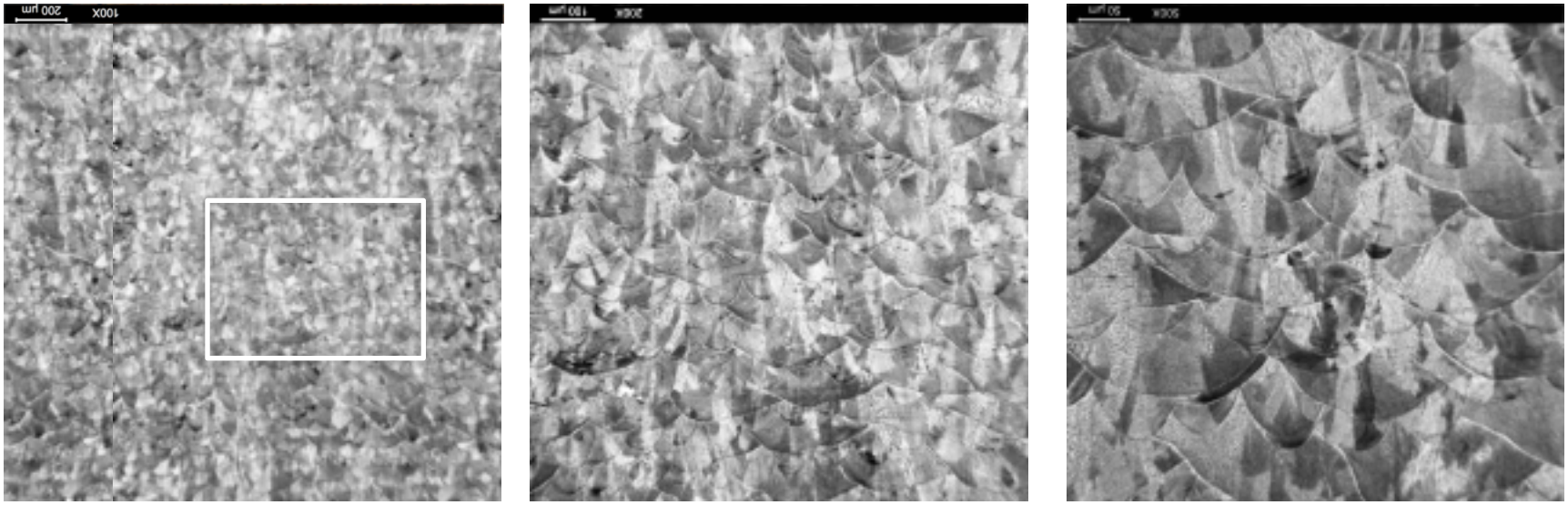





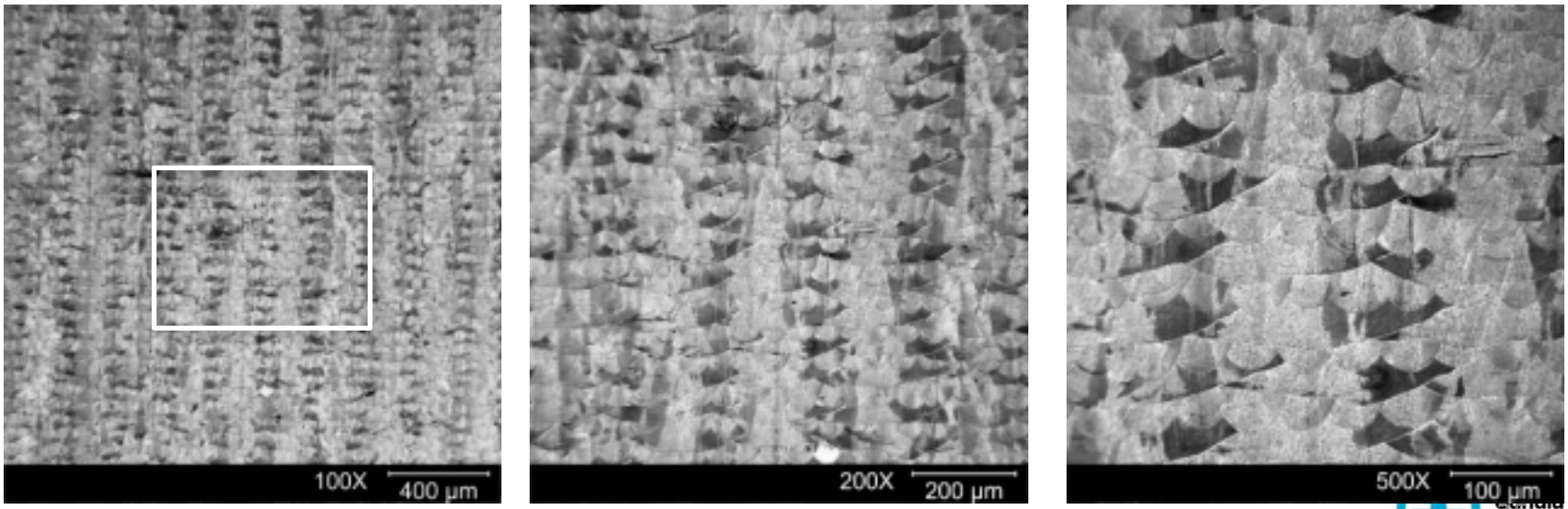
Base

Different solidification structure between the hexagon printed at GPI and SNL,CA

Building direction



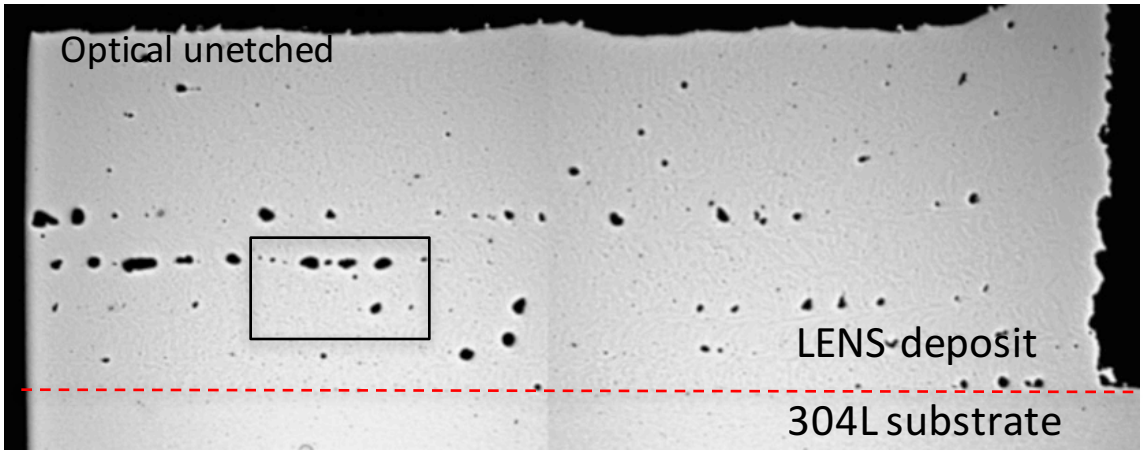
Nitric acid etched PBF hexagon-OM/BF image

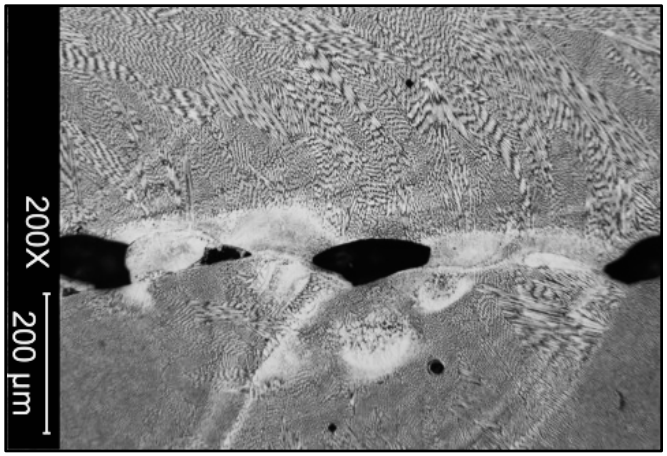


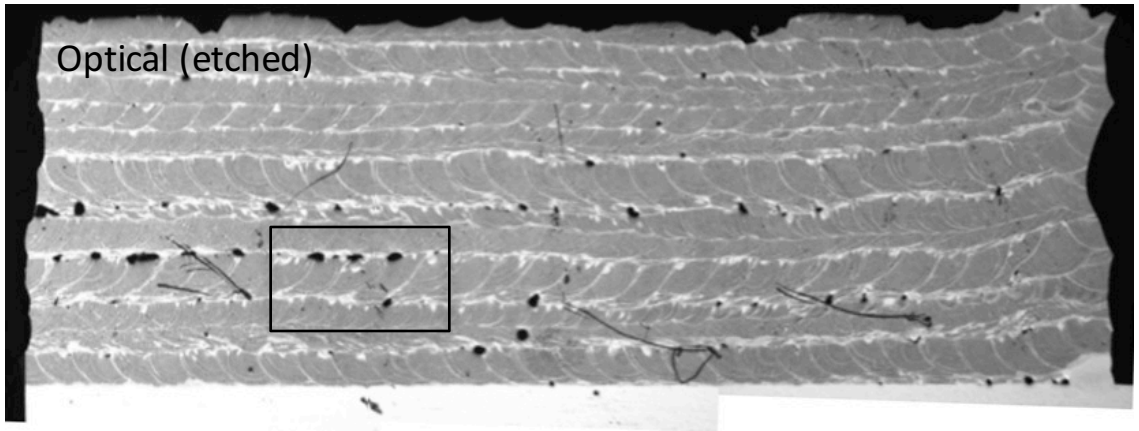
Subjects for discussion

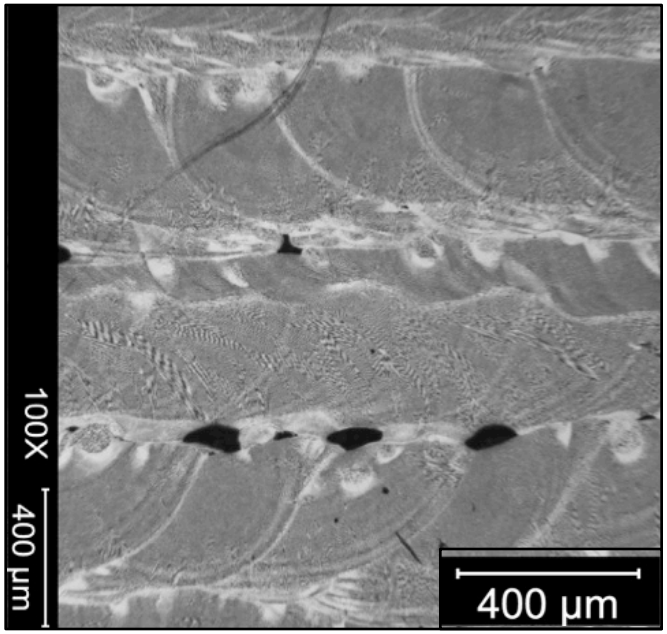
- **3-D LENS and PBF printing induced physical metallurgy & structural irregularities**
  - ✓ *Surface topography and contour*
  - ✓ *Metallurgical characteristics*
  - ✓ Solidification microstructure evolution and geometry factors
  - ✓ Structural irregularity and its origin
  - ✓ Mechanical behavior & engineering properties of AM SS 316L

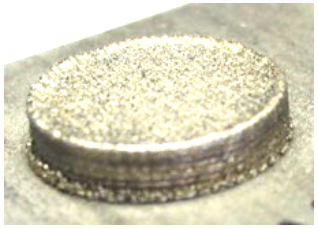
Gross interpass porosity seen of in the LENS thin disc, 4mm thick, short distance from the substrate interface







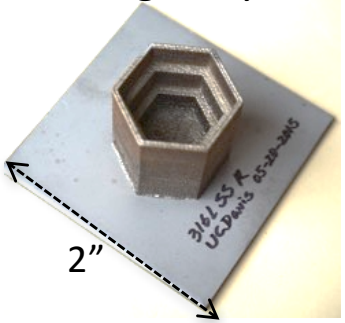


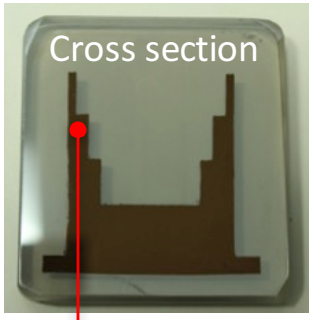


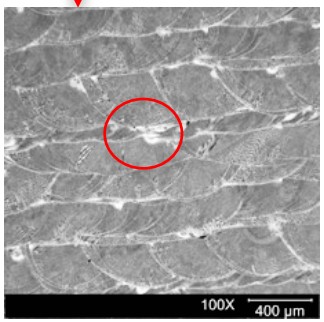
Gross structural defect, i.e., pores surrounded with unmelted powder inclusion

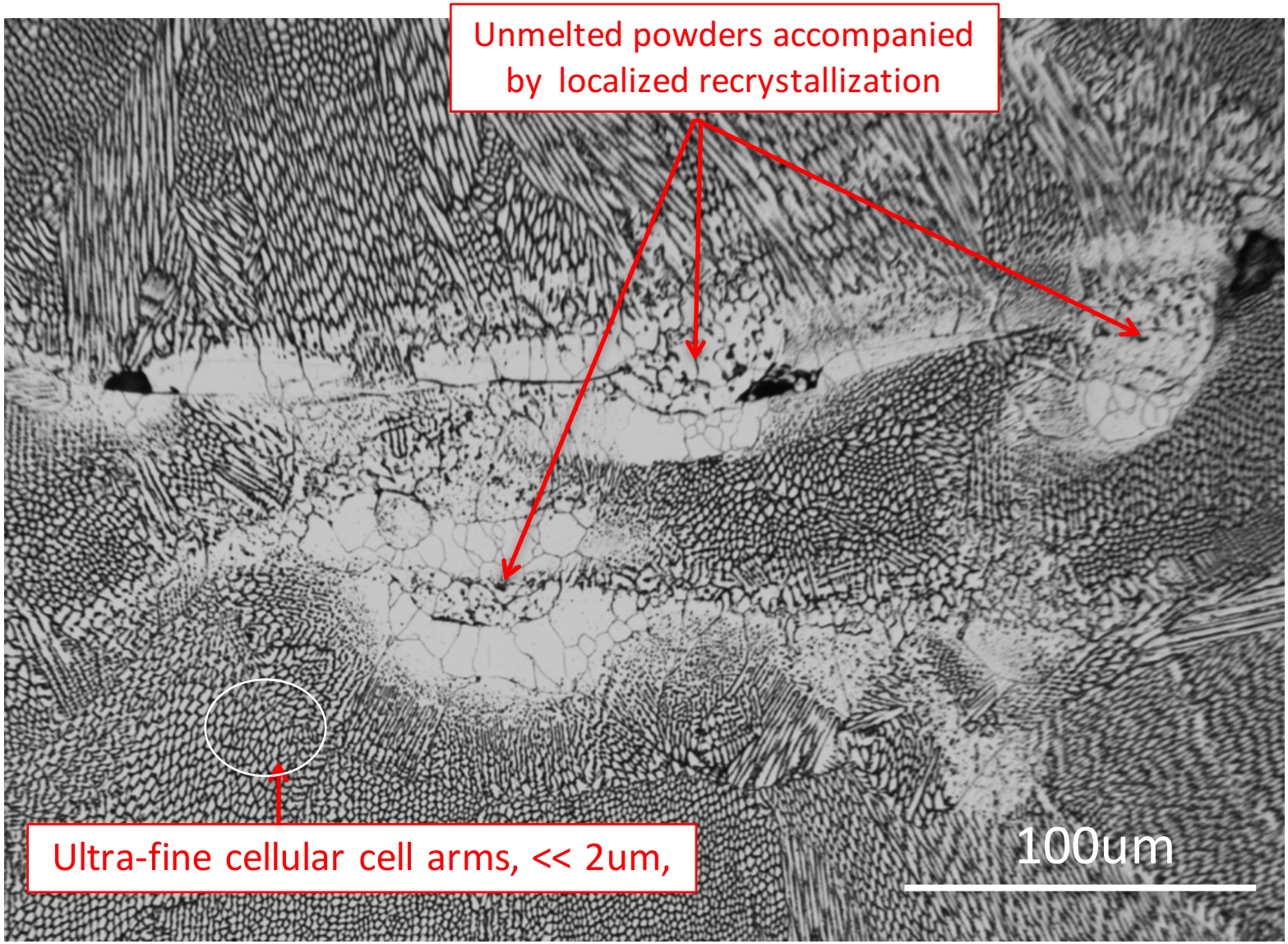
Interpass unmelted powder inclusion impact local microstructure, e.g., recrystallization, and porosity

3-D LENS printed hexagon by UCD



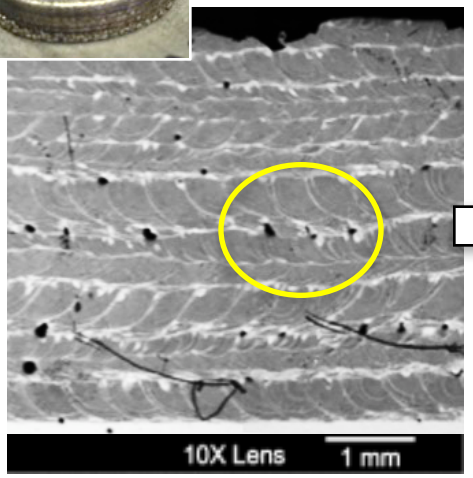


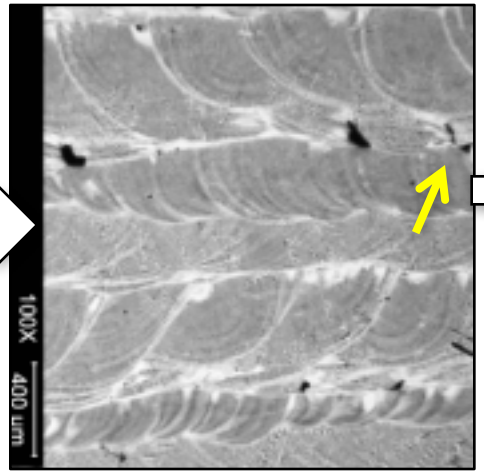


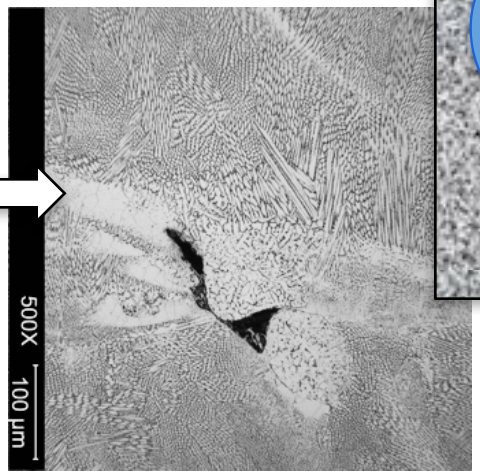


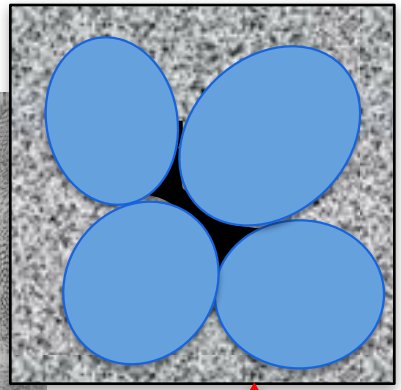
Intrinsic defect in LENS originated from unmelted powder in LENS disc and from foreign contaminant in PBF hexagon

LENS printed 4mm disc



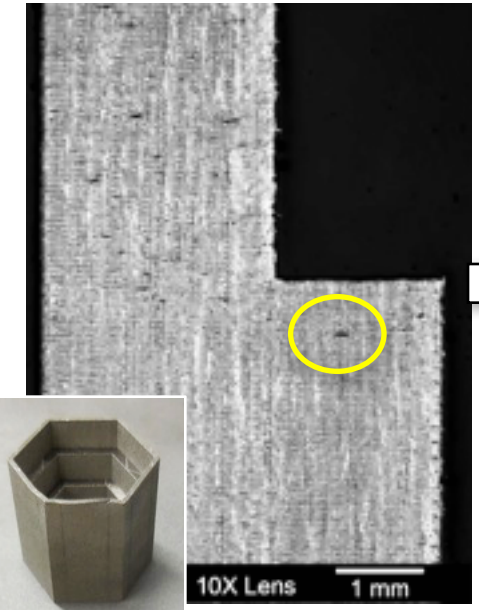


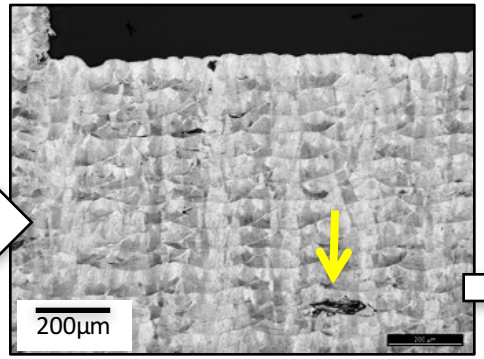


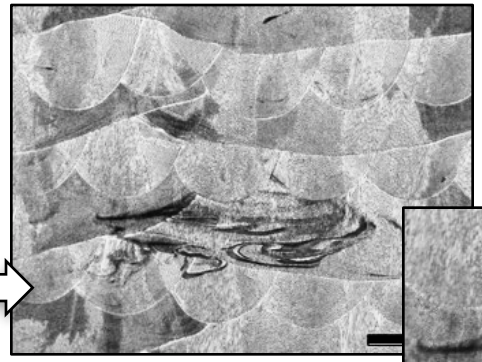


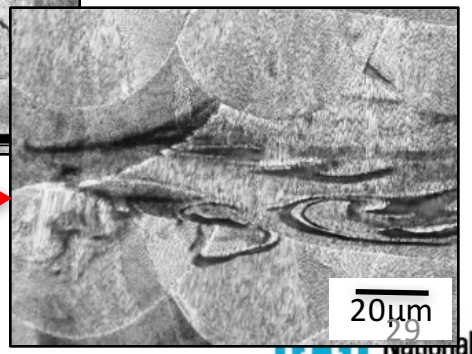
Schematic for pore formation

PBF printed multi-tier hexagon









Defect yields from foreign inclusion interrupt the metal fusion

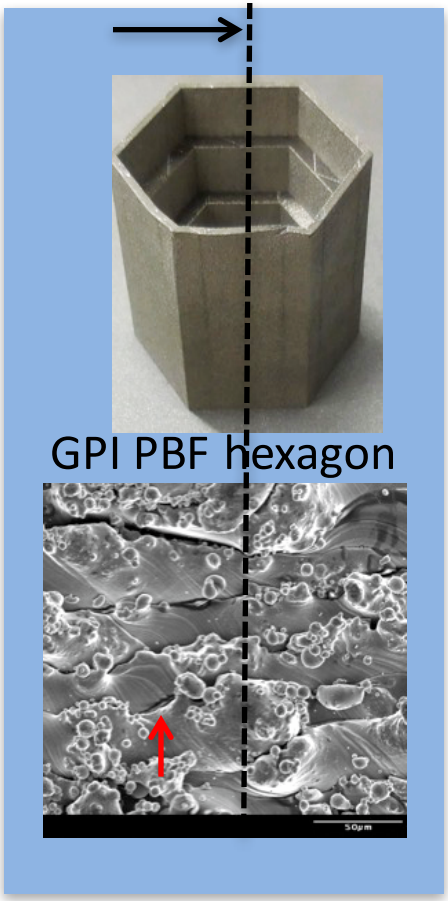
Thin surface openings and sub-surface porosity follow inter-particle boundaries and/or at trip point

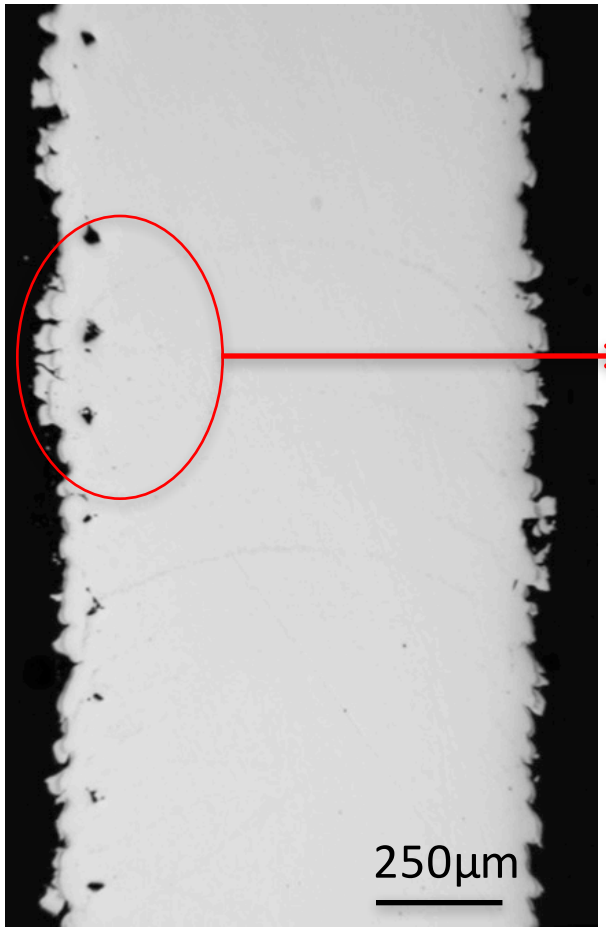
Polished cross section

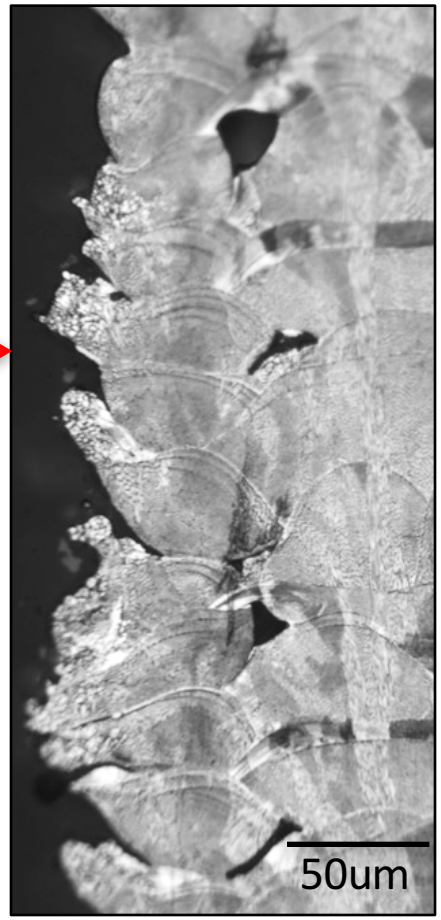
Polishing direction

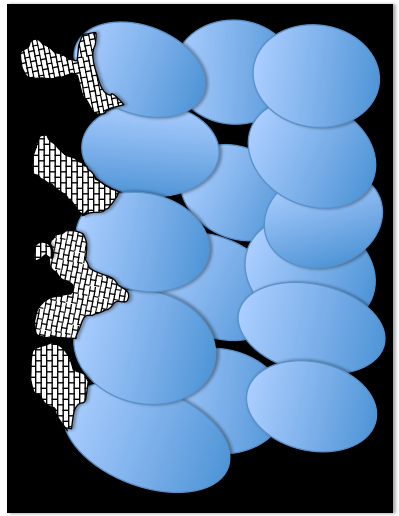
AS polished

Chemically etched







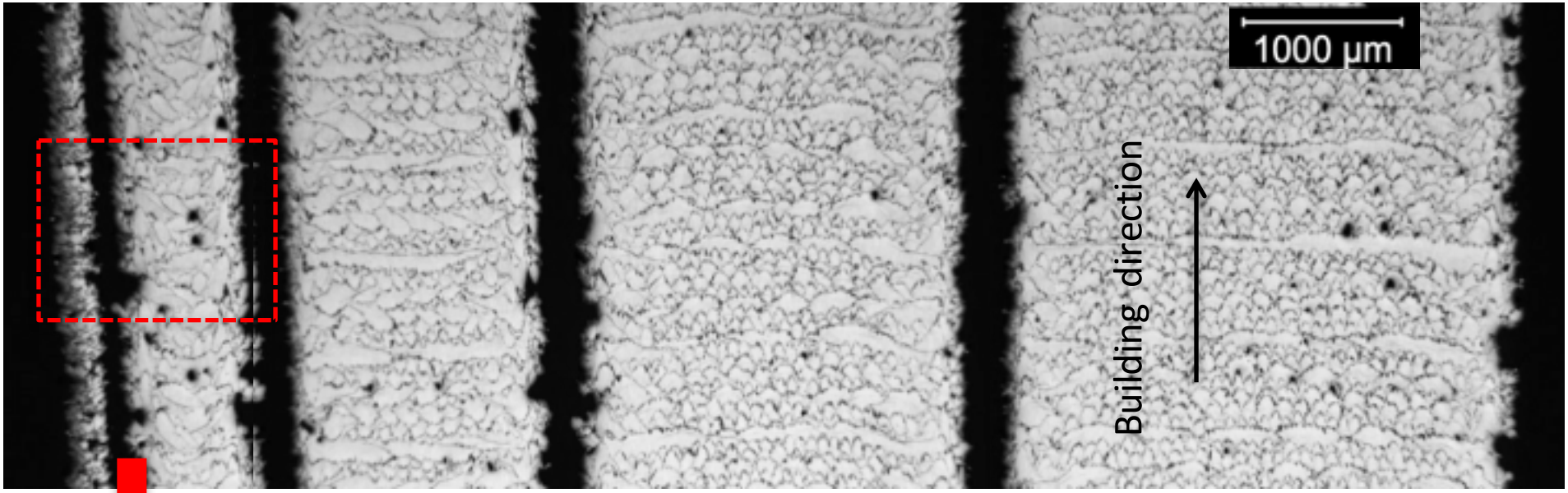


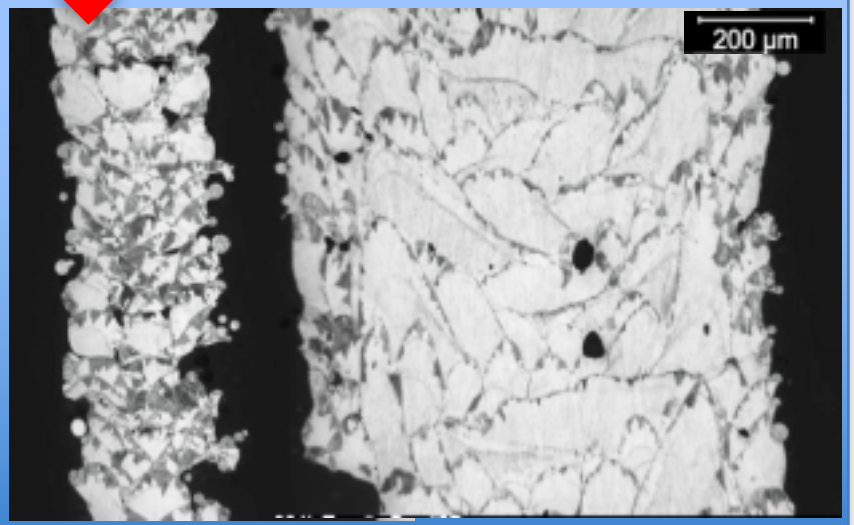
built direction

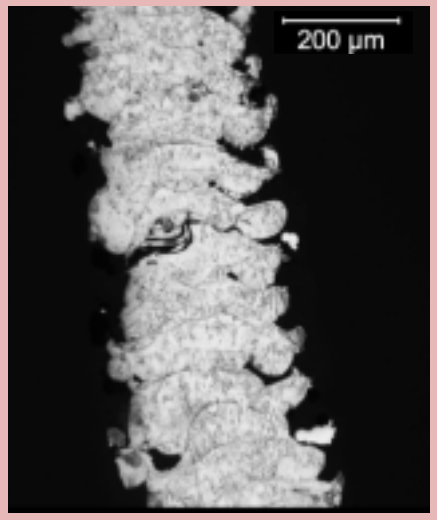
- Red arrows indicate the origin of the thin surface openings.

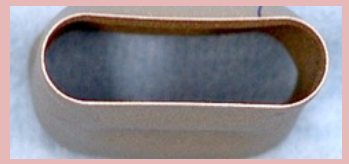
Porosity & surface roughness impact rises as the sidewall thickness decreases.

Decreasing pore density →







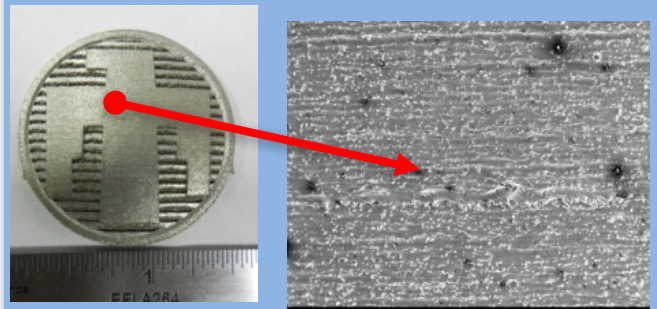


Minimum achievable dimension limit?

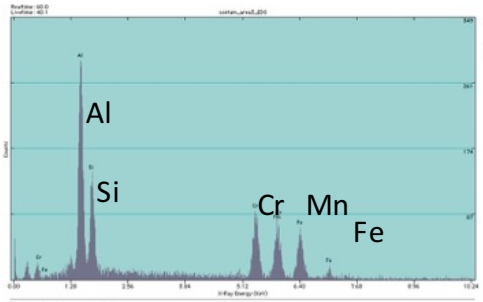
Analytical approach to determine the origin of foreign contaminant

Non-metal foreign contaminant

SNL,C PBF coin

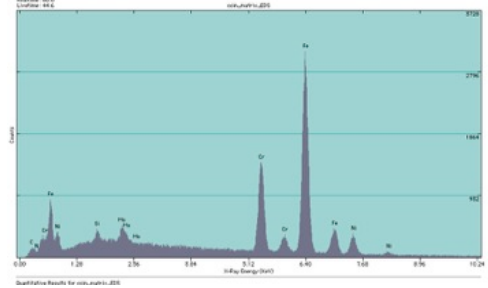


Surface contaminant



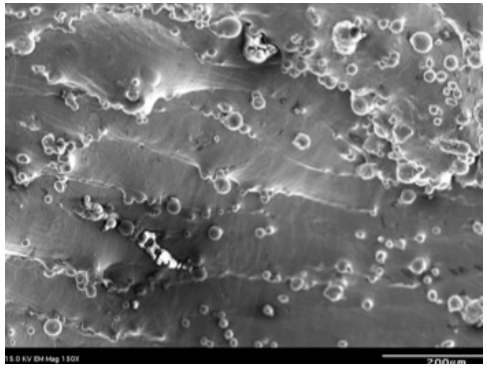
Element	Weight %
Al	24.36
Cr	21.49
Fe	21.40
Mn	20.90
Si	11.85
Total	100.00

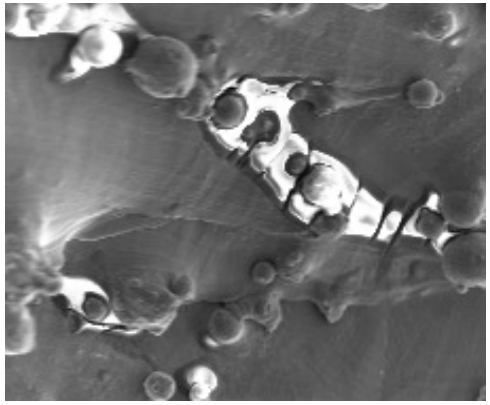
Intrinsic contaminant

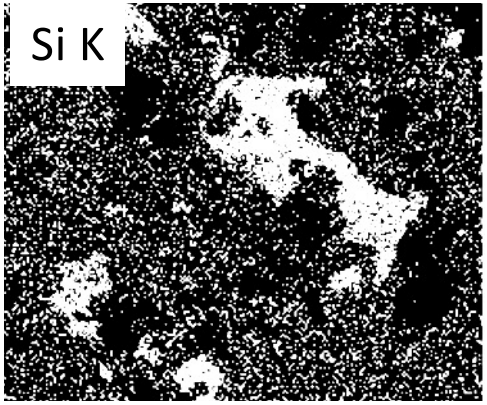


Element	Weight %
C ?	0.00
Cr	18.19
Fe	67.86
Mo ?	1.31
N ?	0.00
Ni	11.57
Si ?	1.07
Total	100.00

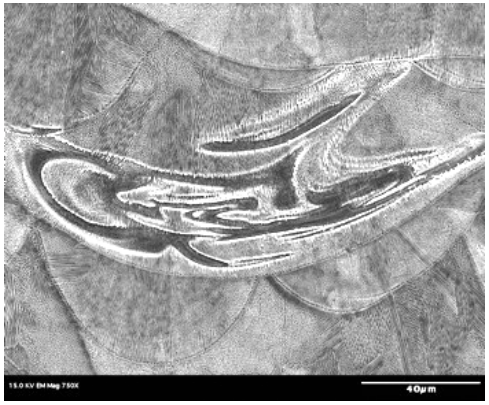
Sidewall surface

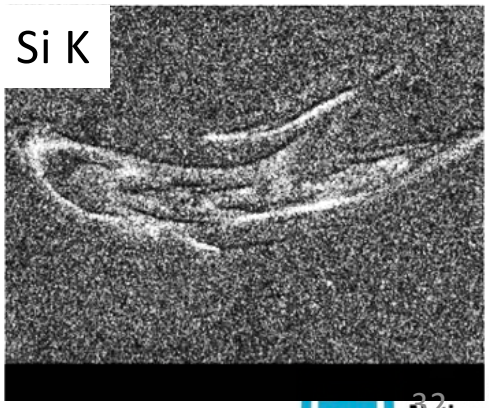






Cross section





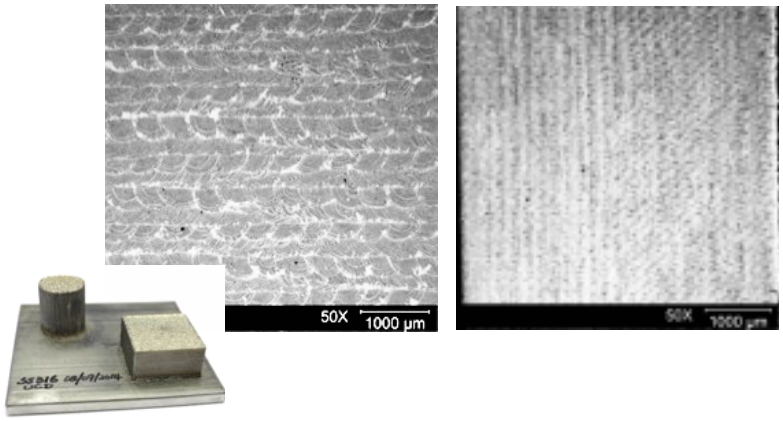
Subjects for discussion

- **Material characteristics & LENS/PBF induced structural irregularities**
  - ✓ *Surface topography and contour*
  - ✓ *Metallurgical characteristics*
  - ✓ Solidification microstructure evolution and geometry factors
  - ✓ Structural irregularity and their root cause or origin
  - ✓ Mechanical behavior & engineering properties of AM SS 316L

- **Summary Conclusion**

High strength, low ferrite and fine cellular arm spacing are common feature for LENS- and PBF- prints

Modulated solidification structure



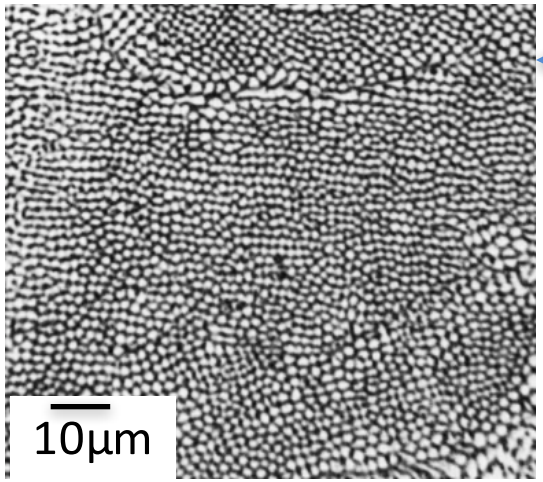
High tensile strength

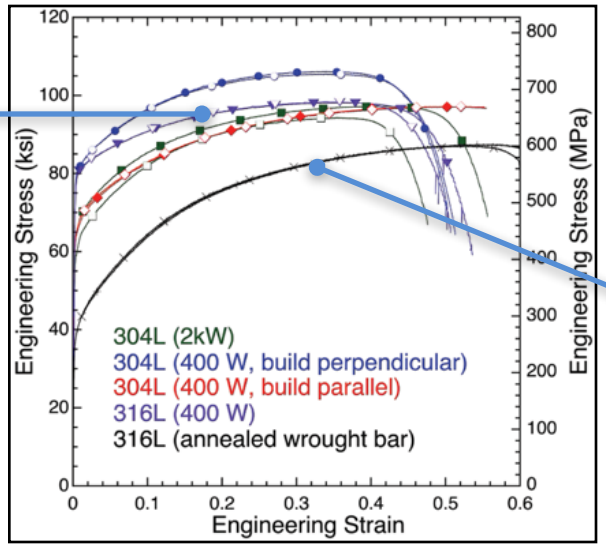
Sample ID	YTS	UTS	Elongation	Vickers HV
	(Mpa)	(Mpa)	(%)	(VHN)
316L wrought	170	450	40	222
LENS hexagon T	538	690	35	220 (tier1)
LENS hexagon T	552	703	38	260 (base)
GPI PBF hexagon	TBD	TBD	TBD	247 (Base)

Low Ferrite:

~0.2-0.4 %, for LENS; <<02% for PBF VS; 1-3% for annealed wrought 304L substrate

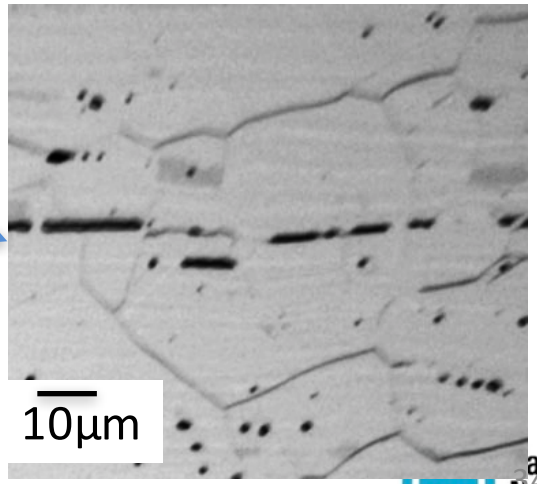
Cellular solidified cell with fine arm spacing



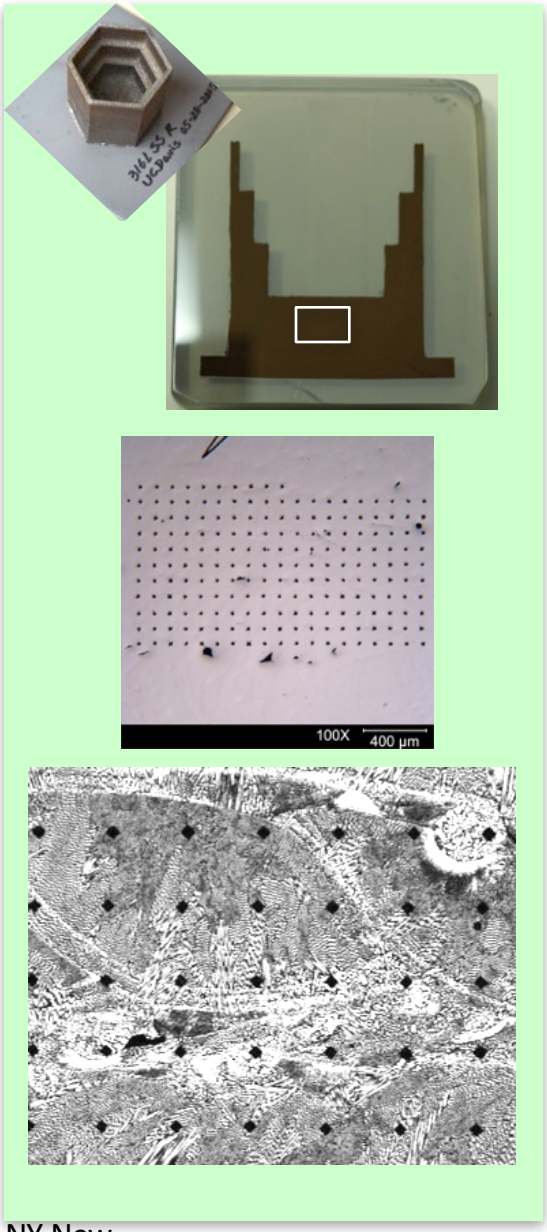


Courtesy of M. Maguire

Coarse-grained wrought 316L

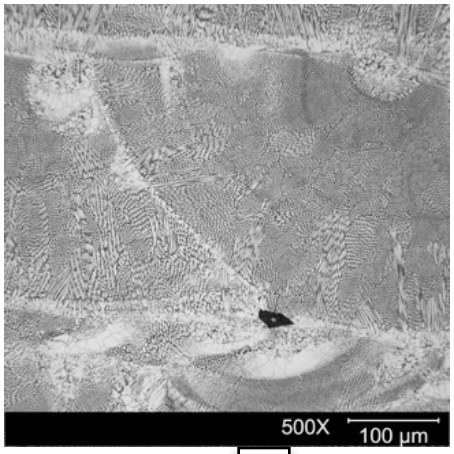


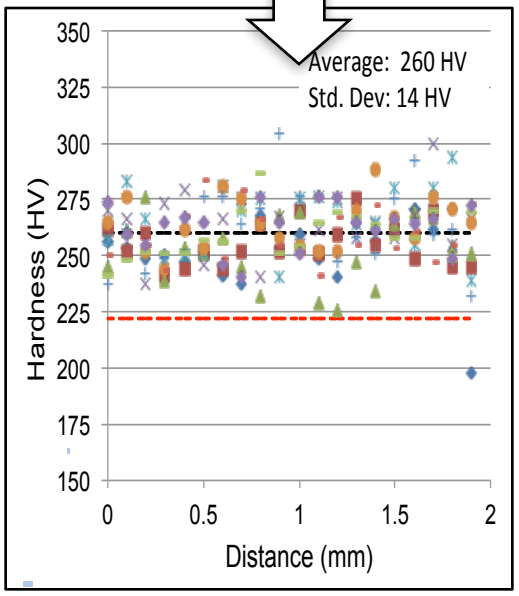
200-point Vickers indents show greater microhardness scattered in LENS print



3-D LENS print

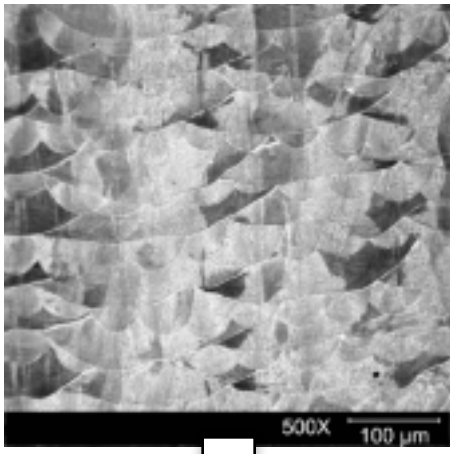
Large hardness scattered due to localized HAZ

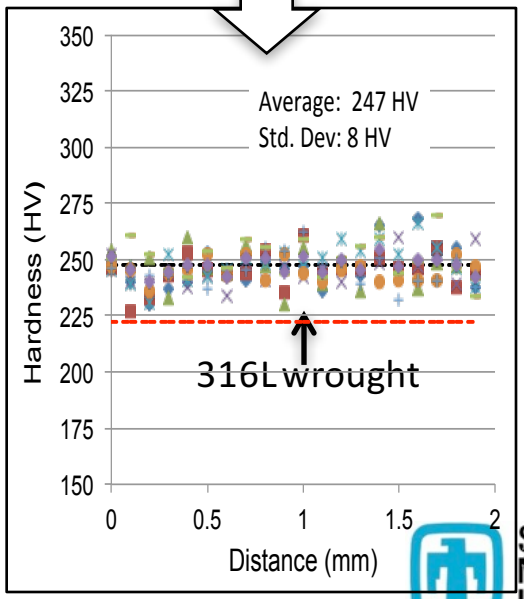




3-D PBF-print

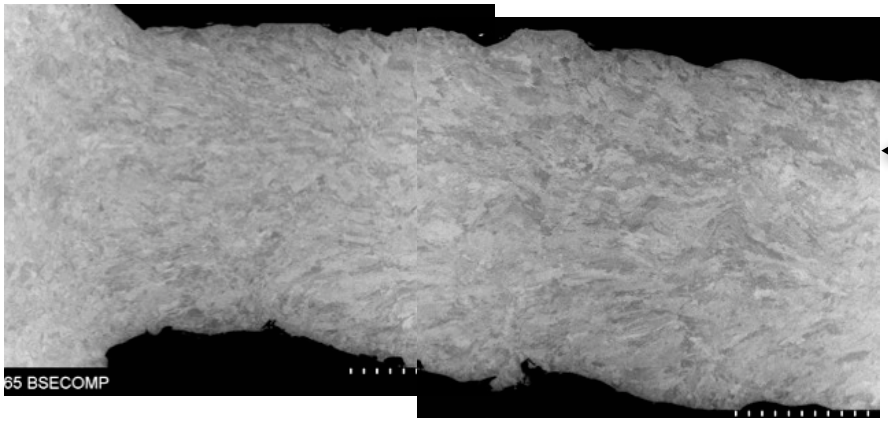
Strong and consistent



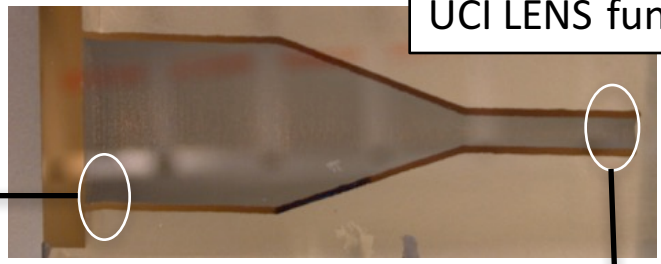


High heat intensity at the small-diameter neck coarsens the solidification cells leading to lower strength

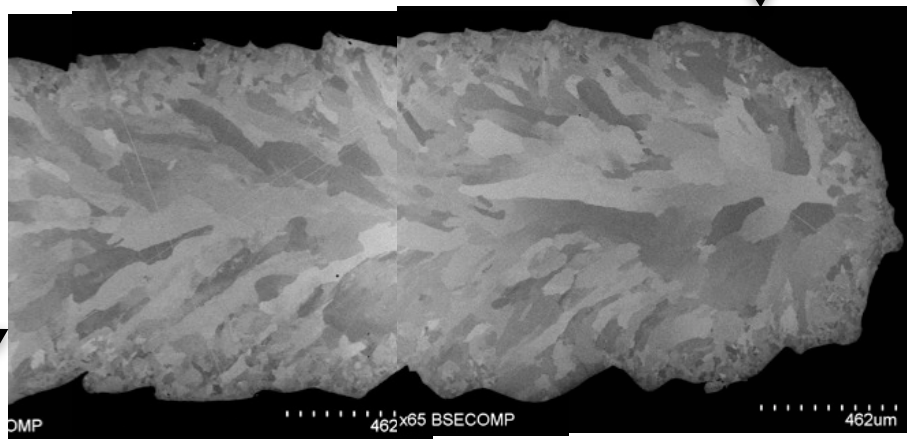
- Fine-celled harder body



UCI LENS funnel

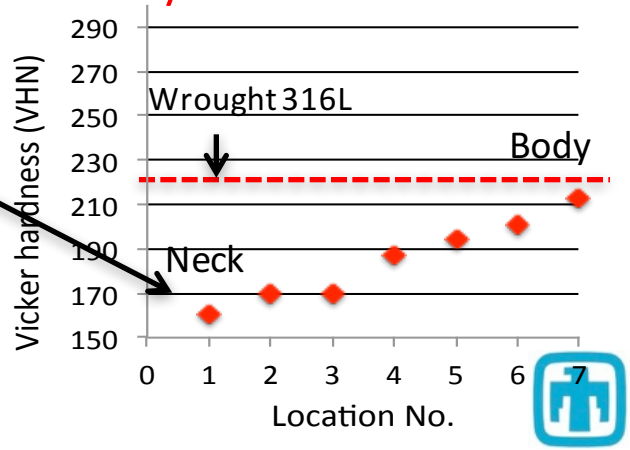


- Coarse-celled neck



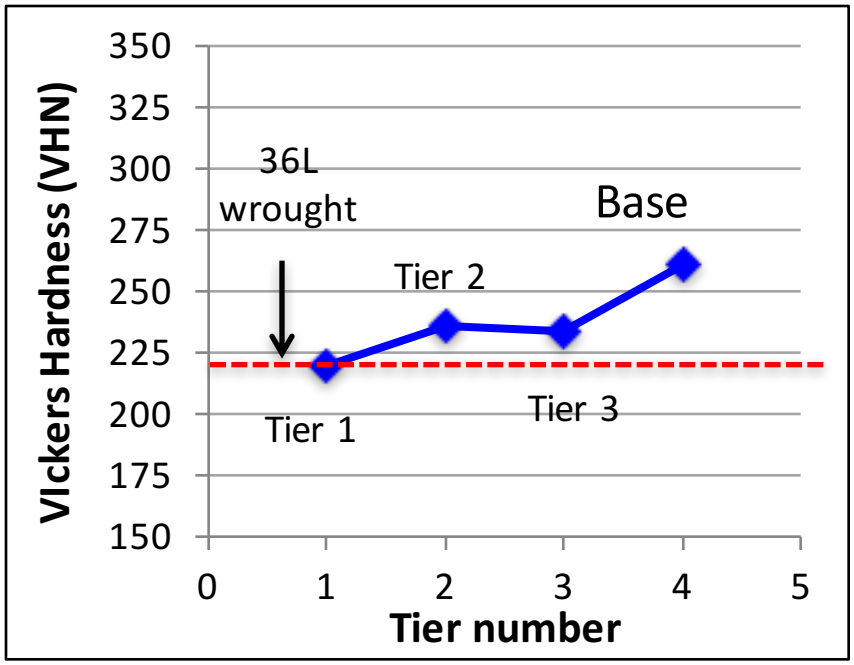
- The glowing neck

- Unusually soft UCI LENS funnel



Sandia National Laboratories

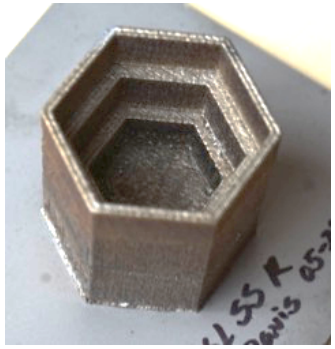
Vickers hardness increases with tier width attributed to solidified cell coarsening



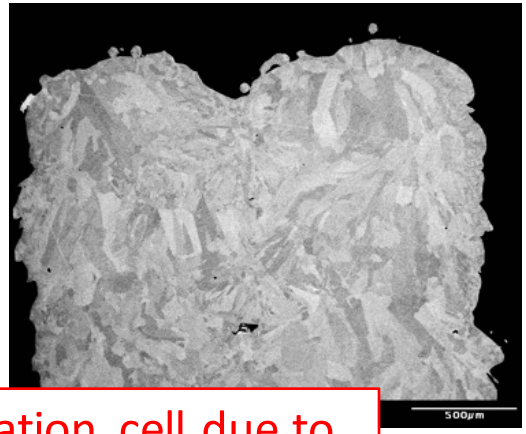
Hexagon base

Tier 2

3-tier LENS Hexagon



Tier 1

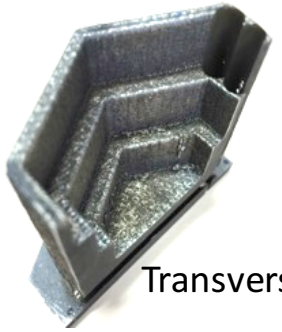


Coarse solidification cell due to absence of interpass boundaries

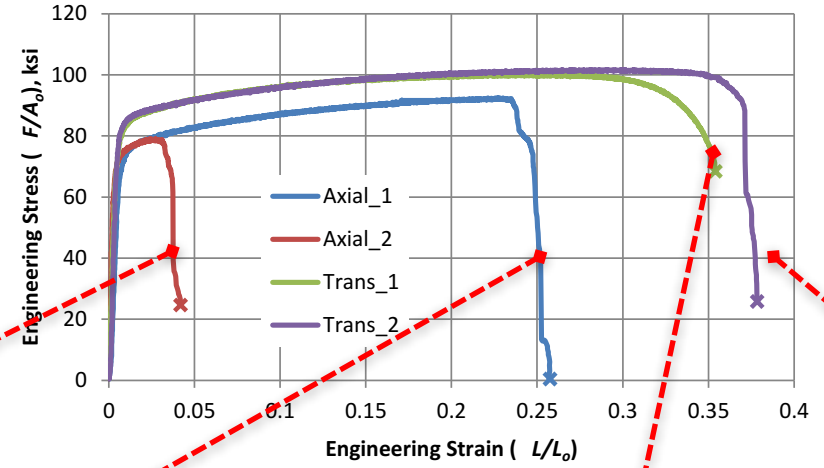
Fine solidification cell due to interpass boundary pinning

Presence of unmelted powder inclusions disrupts metal fusion, therefore, lowers ductility

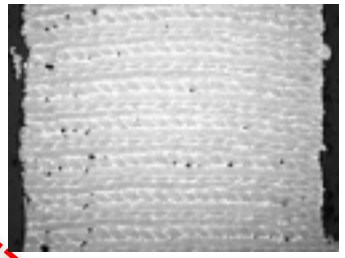
Axial 1 & 2

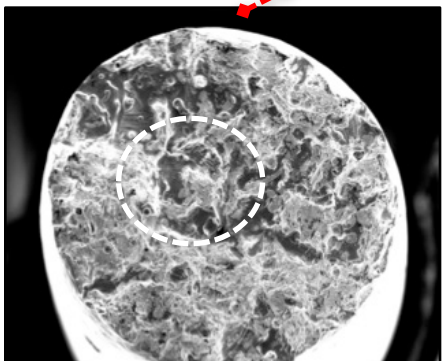


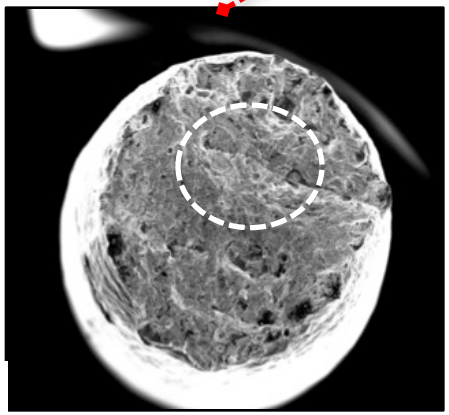
Transverse 1 & 2

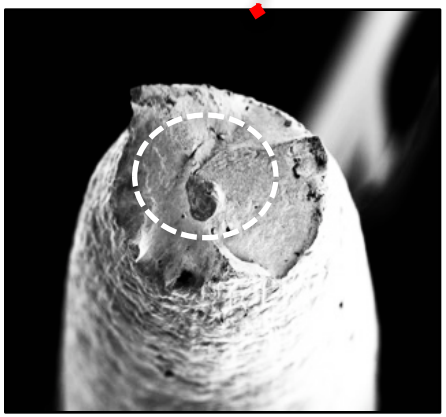


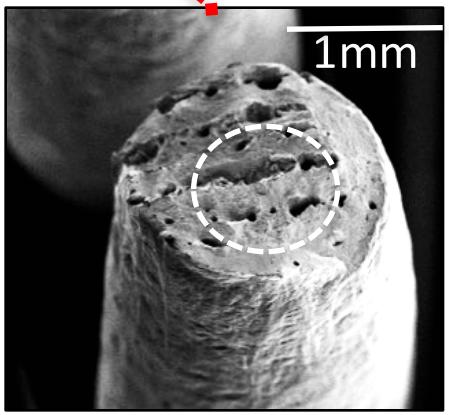
Randomly distributed interpass pores at the failure



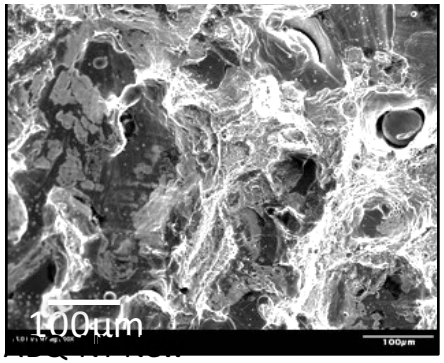


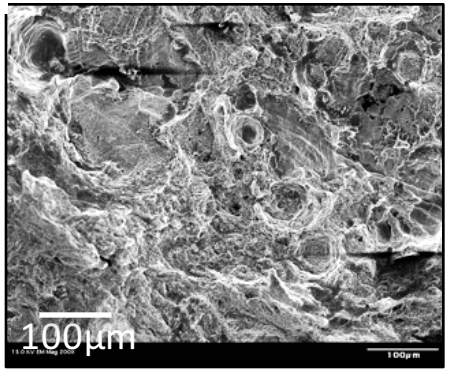


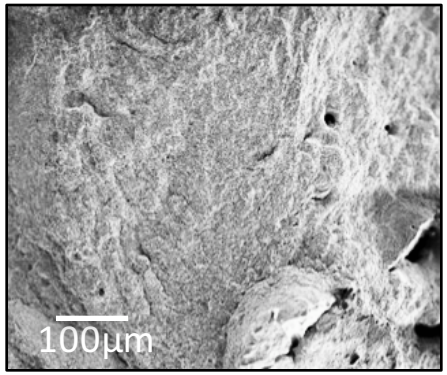


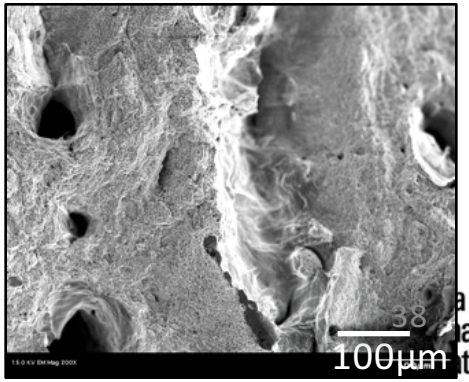


Lack of metal fusion









Summary

- We perform a extensive characterization on 3-D LENS- & PBF- induced physical metallurgy, i.e., surface morphology /contour, solidification structure, mechanical property, and structural integrity.
- The origin of structural defects seen in the LENS- & PBF- prototypes and their subsequent impact on engineering property has been determined .
- The study also concluded the followings:
  - ✓ LENS & PBF printed SS316L prototypes exhibit relatively high engineering strength due to the fine solidified microstructure and others(?).
  - ✓ Solidification structure using AM printing is dictated by laser-material interaction and/or solidification cooling mechanism, which are printing technique and its cooling medium, dependent.
  - ✓ Microhardness of 3-D LENS SS316L prototype is geometry dependent, derives from localized solidification cooling.
- Process control/optimization to yield a consistent solidification structure and mitigate structural defect are the key to achieve ultimate material assurance or product qualification,

On-going activities and future plan

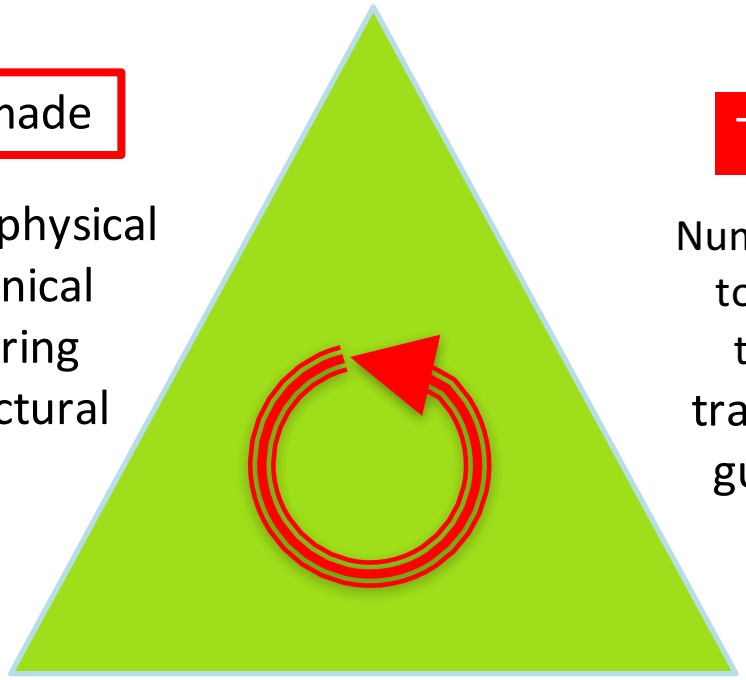
Process control/optimization

Good progress made

Identify AM induced physical metallurgy, mechanical behavior, engineering property, and structural integrity.

To be developed

Numerical simulation/modeling to validate/predict thermal transport and local heat transport & distribution to guide process control & optimization strategy



Material property

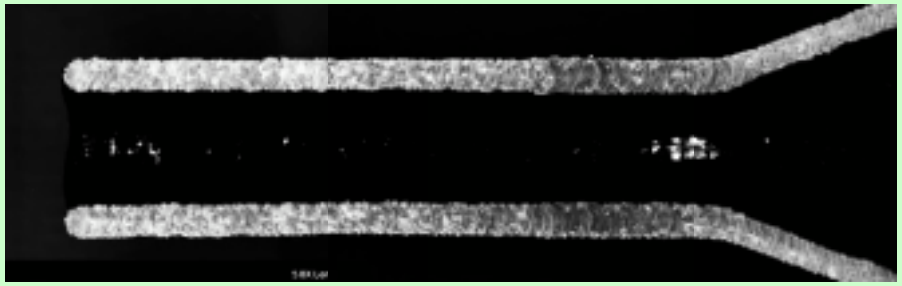
Numerical simulation

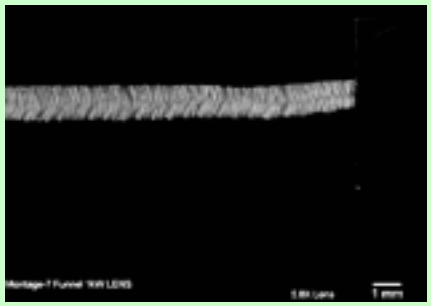
On-going effort

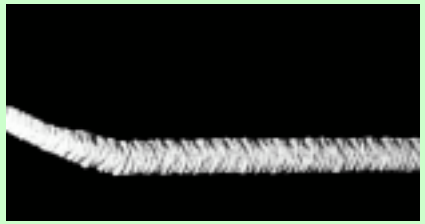
Iednetify origin of metallurgical characteristics and structural defect for enabling technical path to control material property and mitigate structure defect

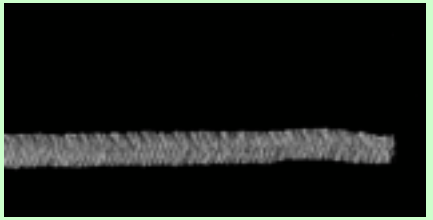
Extra slides

400W Fiber Optics thick UCI funnel



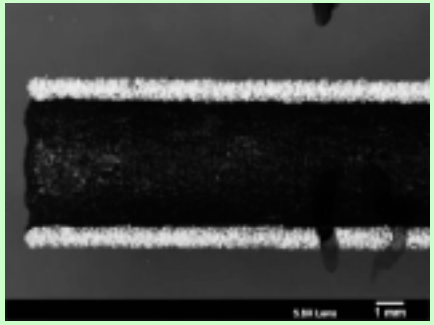


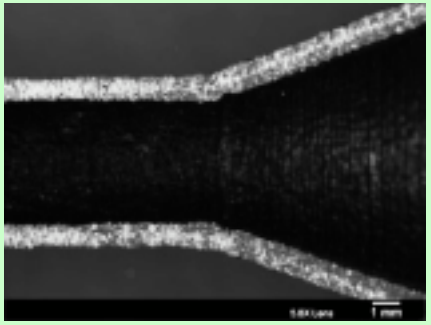


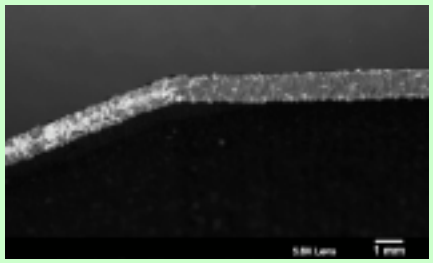


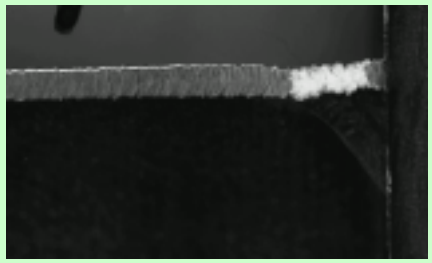
OM/DF

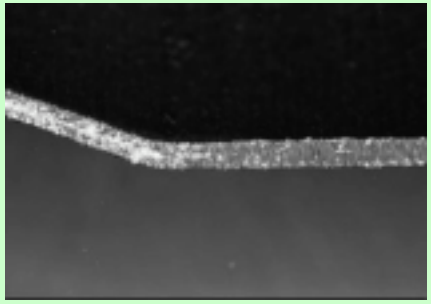
360W YAG thin UCD funnel

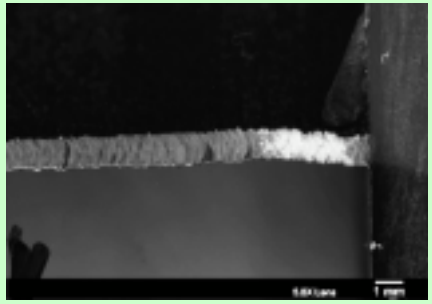






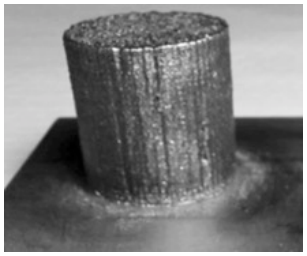


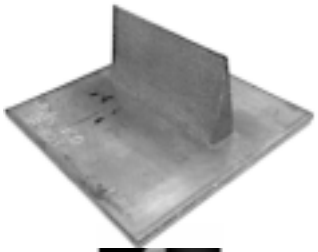


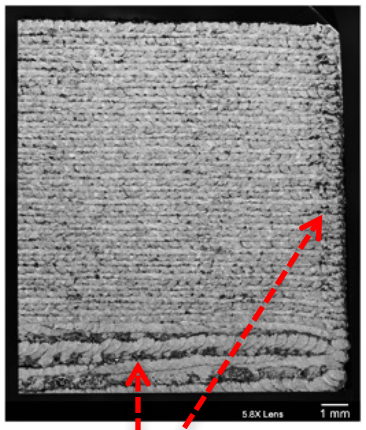


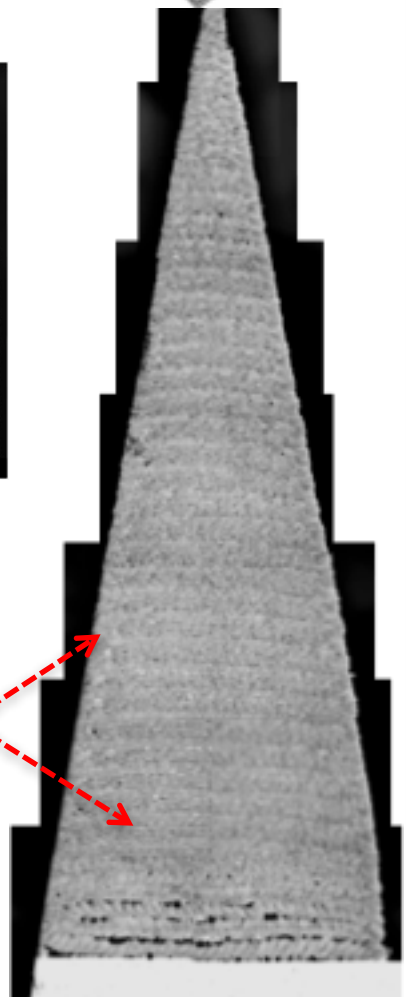
OM/DF

Structural defect caused by the localized reheating or ΔT at the substrate interface that results in localized HAZ

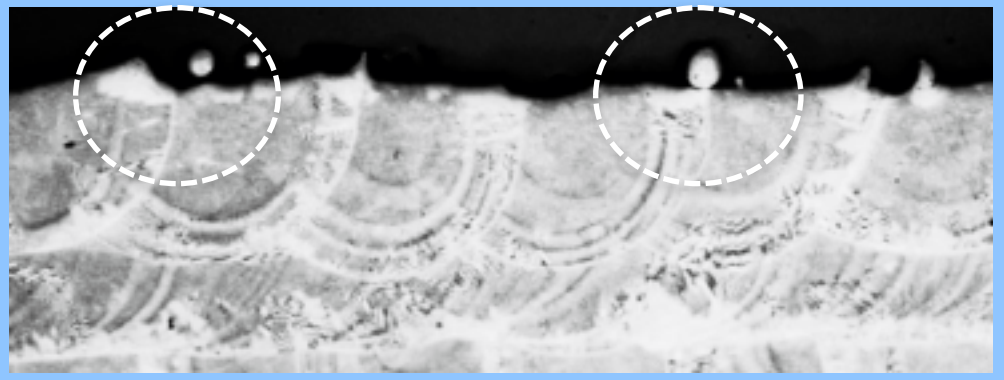


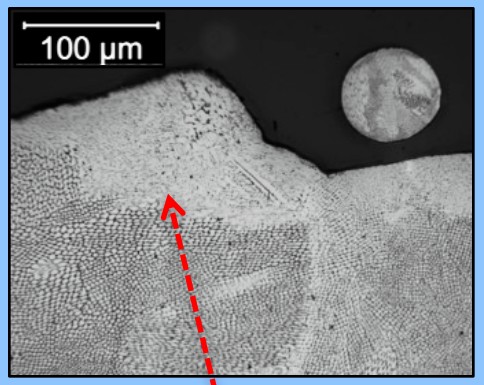


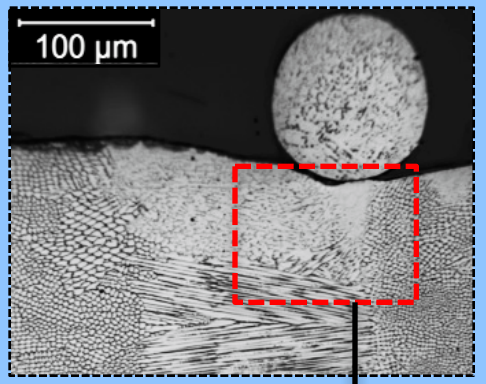




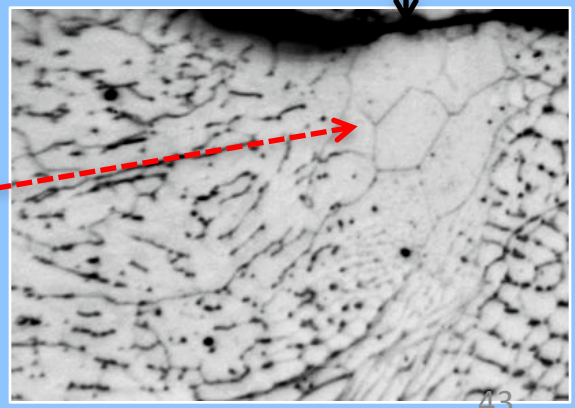
Gross porosity due to high ΔT, at substrate interface and/or free surface



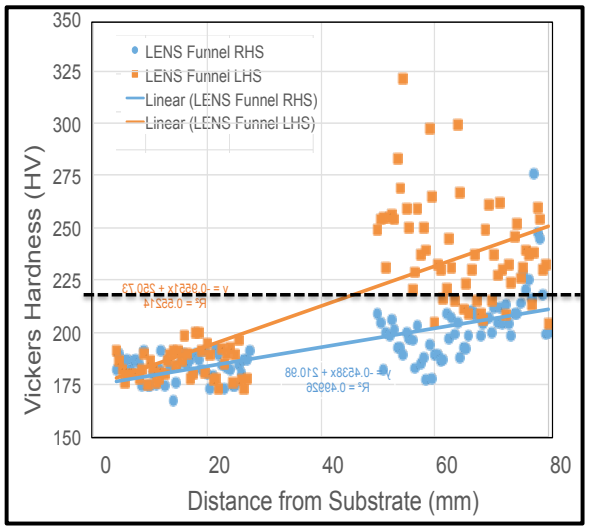


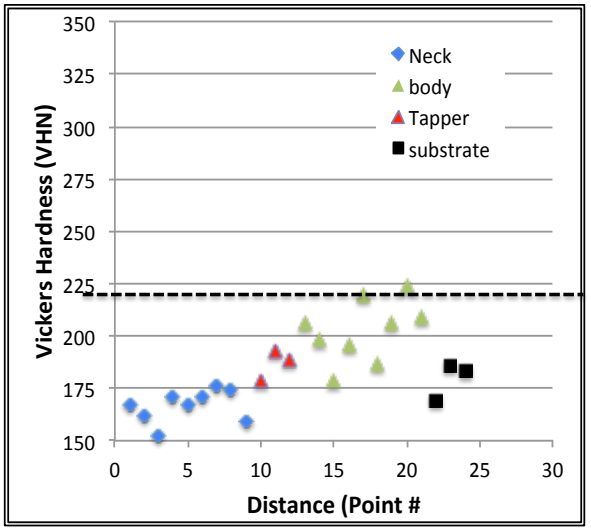


HAZ with recrystallization induced by unmelted surface powders

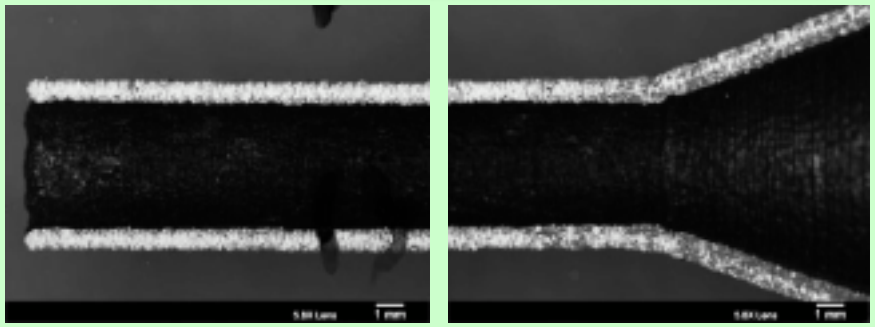


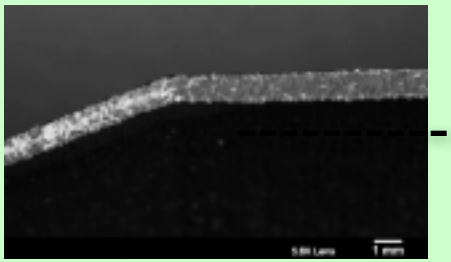
Vickers hardness profile and uniformity varied between two funnels, printed at UCD and UCI respectively

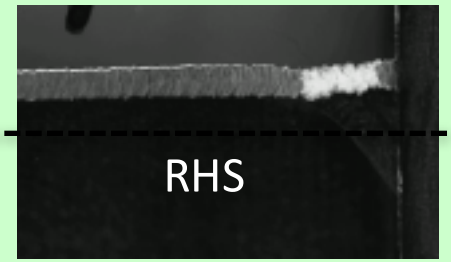


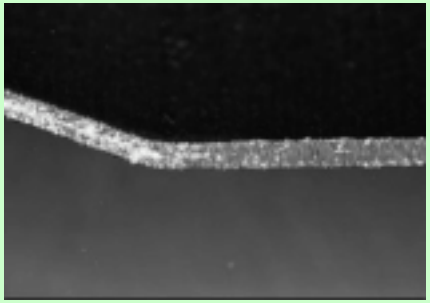


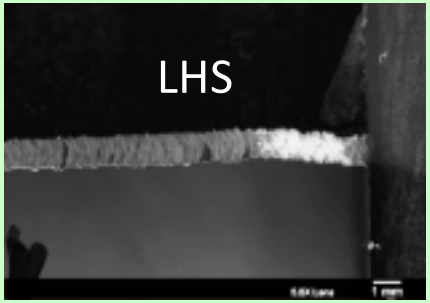
360W YAG UCD funnel DF





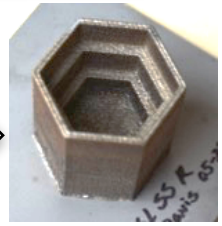


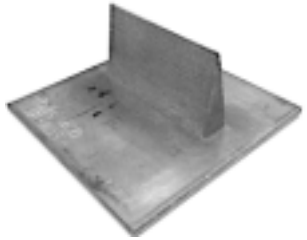




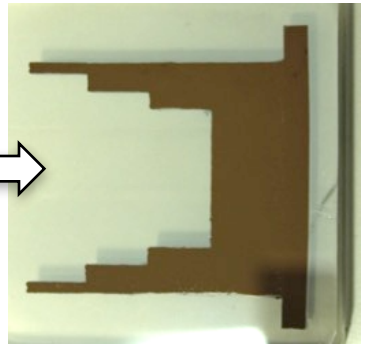
The overall Vickers microhardness varies among the prototypes, printed with the same LENS parameter at the same condition

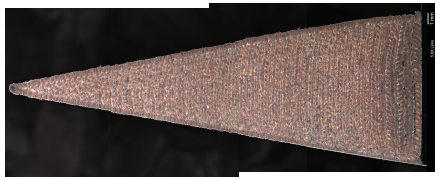
As-printed geometry

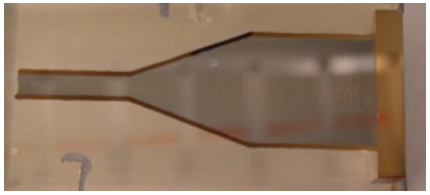




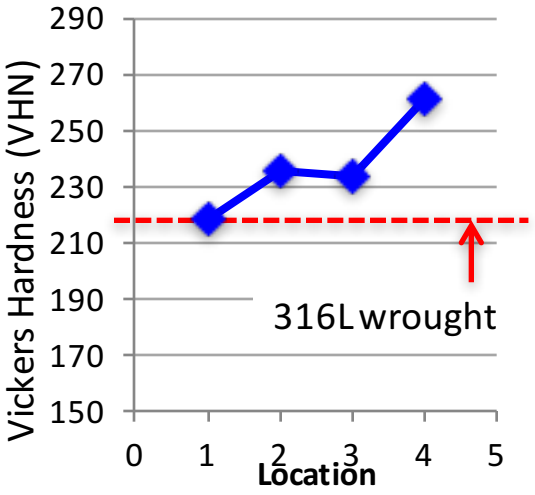
Polished cross sections



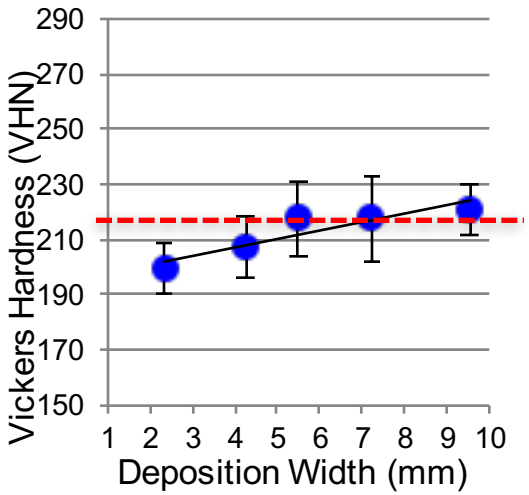


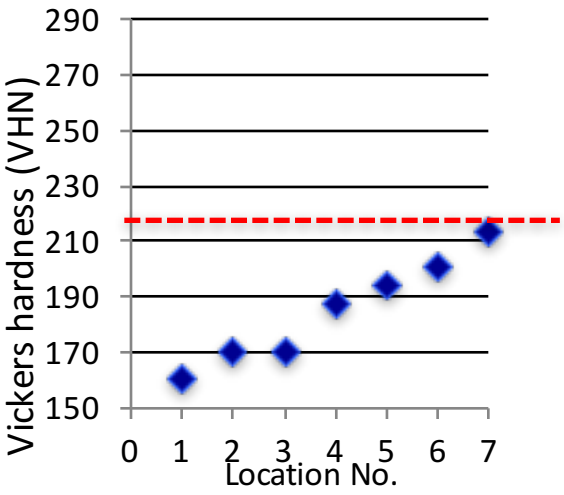


- Harder than wrought 316Lt



- Softer than wrought 316L

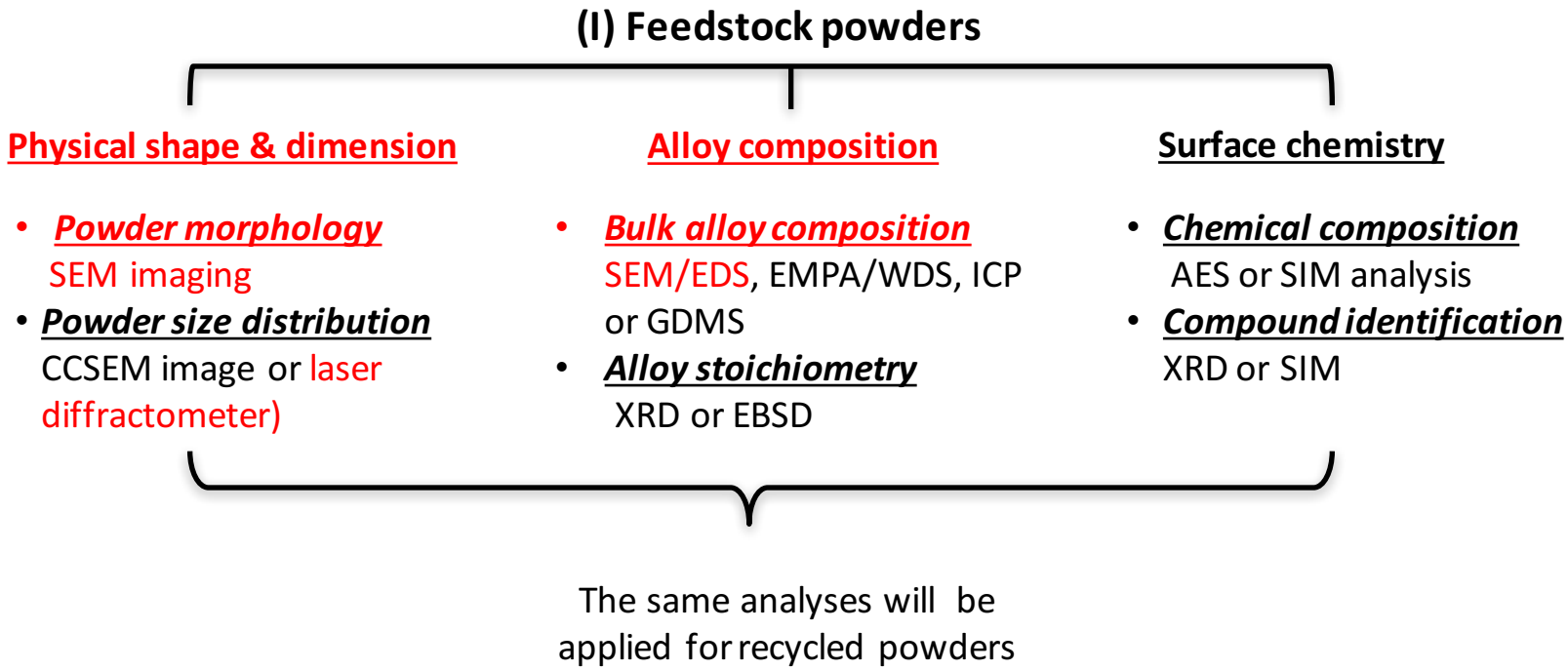




The Vickers hardness increases toward the substrate interface in all cases

Sandia National Laboratories

Material properties of interest for feedstock powder and their measurement techniques



Note: The items been performed are highlighted in red

Relevant material properties and their measurement techniques for the finished prints

(II) Finished 3-D LENS or PBF print

Physical Metallurgy

- ***Alloy composition***
  SEM/EDS, EMPA/WDS, ICP, GDMS
- ***Chemical segregation***
  SEM/EDS, EMPA/WDS profiling and mapping
- ***Solidification microstructure***
  OM/BF, SEM/BEI, SEM/EBSD, TEM imaging
- ***Localized heat affected zone (HAZ)***
  OM/BF imaging, EMPA/WDS
- ***Anisotropy or texture***
  XRD or EBSD
- ***Localized recrystallization & grain growth***
  OM/BF imaging
- ***Thermal stability or aging***
  In situ DSC or DTA, OM/BF and TEM imaging
- ***Mechanical property and fractography***
  UTS by tensile testing, fracture toughness by Sharpy testing or 3-point bend, and fractography by SEM/SEI/BEI, EDS
- ***Microhardness***
  Vickers and/or Rockwell indentation.
- **Ferrite content:** for SS316L or SS304L
  Eddy current ferrite scope

Surface property

- ***Surface morphology***
  SEM/SEI/BEI imaging
- ***Surface topography & contour***
  Profilometer or others (TBD)
- ***Shape & Dimension variation***
  SEM/SEI imaging, CMM
- ***Surface chemistry***
  AES, SIM SEM/EDS

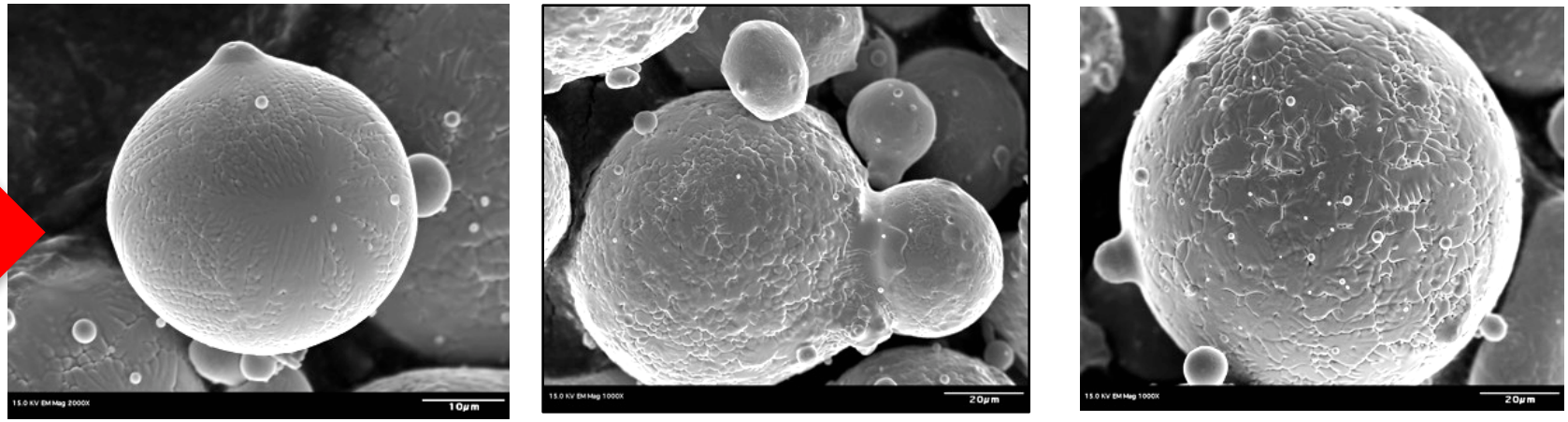
Structural integrity

- ***Interfacial fusion***
  OM/SEM imaging Radiology, CT scan
- ***Foreign contaminant & inclusion***
  SEM/SEI/BEI imaging, EDS , radiography, CT scan
- ***Porosity***
  OM imaging, radiography, CT scan

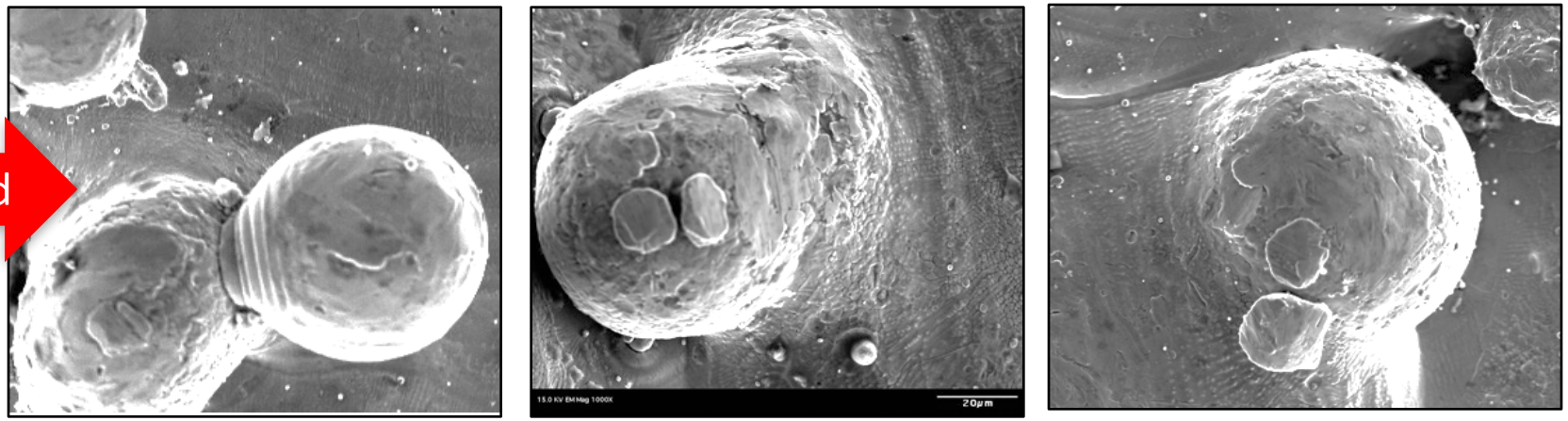
Note: The items been performed are highlighted in red

Powders on the LENS print are mechanically smeared and deformed, implies powder life limit?

Feed stock



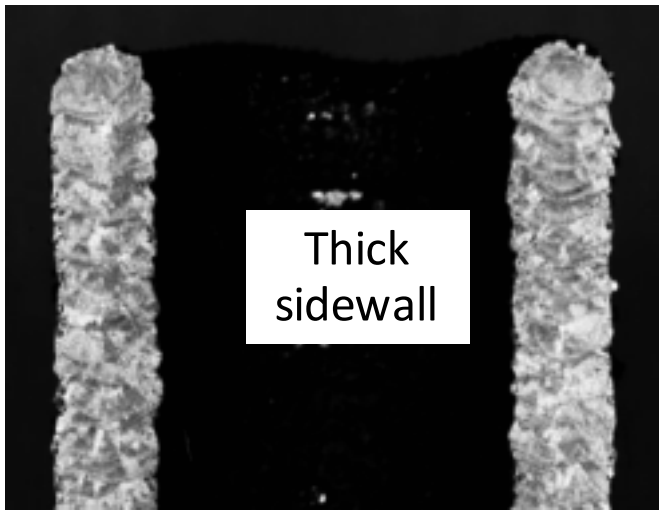
LENS Printed

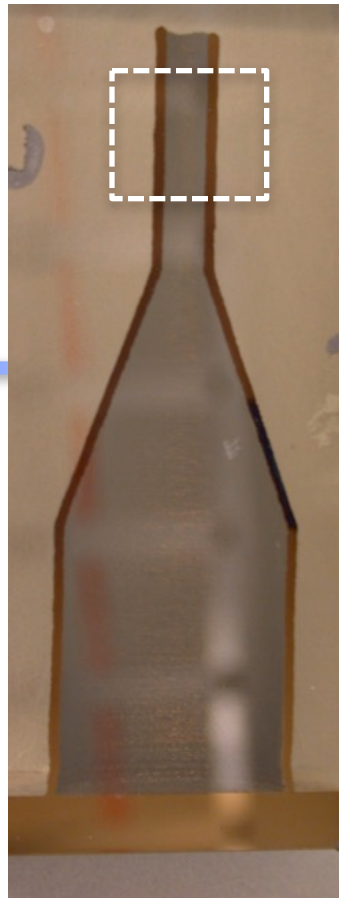


For LENS and PBF printing, powder morphology and size impact powder flow during powder feeding and packing density, respectively.

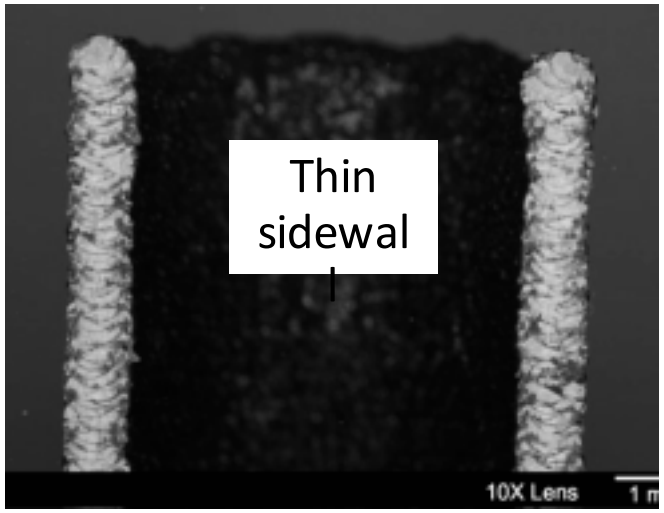
Different sidewall thickness observed between the funnel printed at UCI and UCD using the same STL design

400W using fiber optic laser at UCI

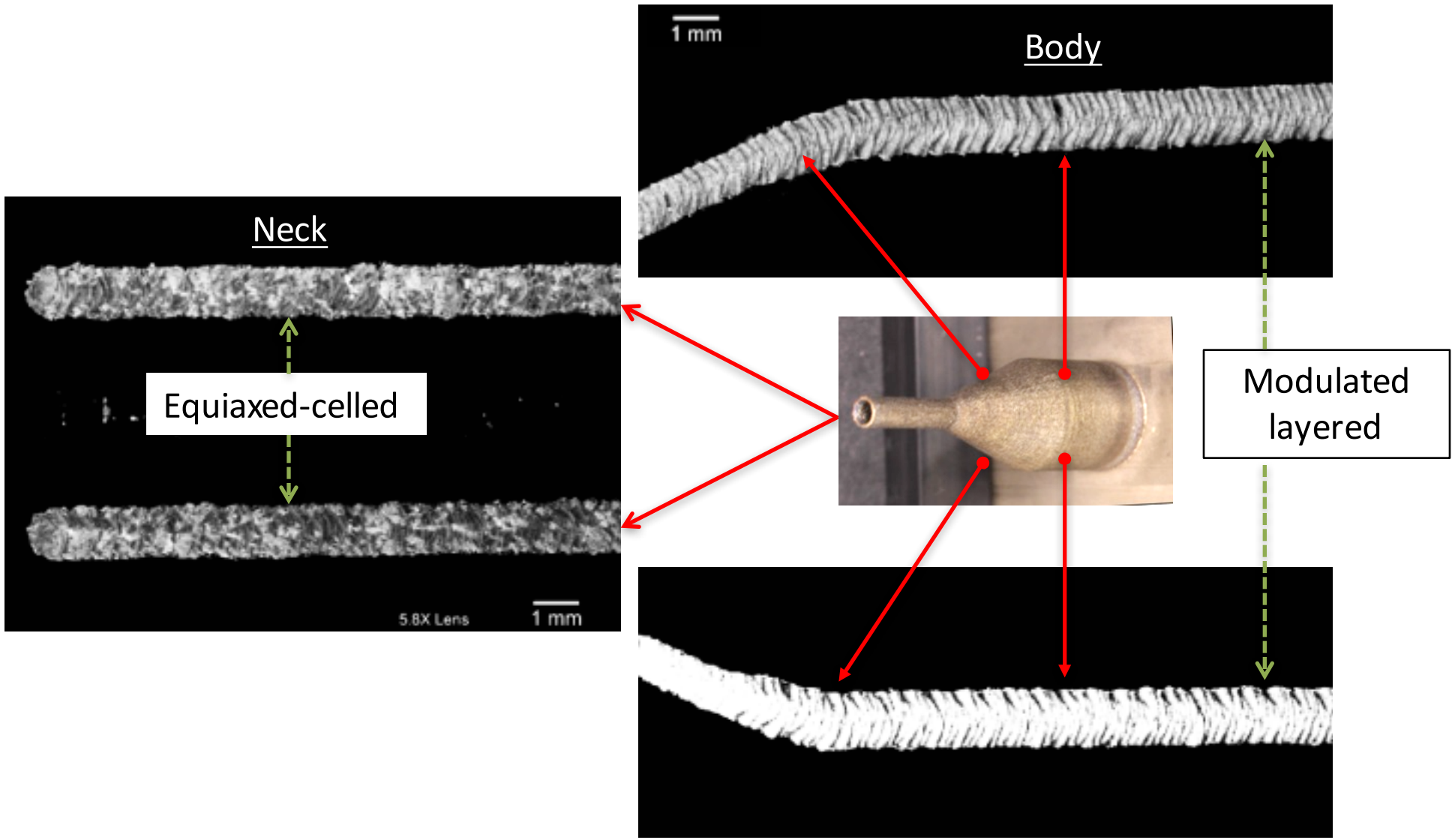




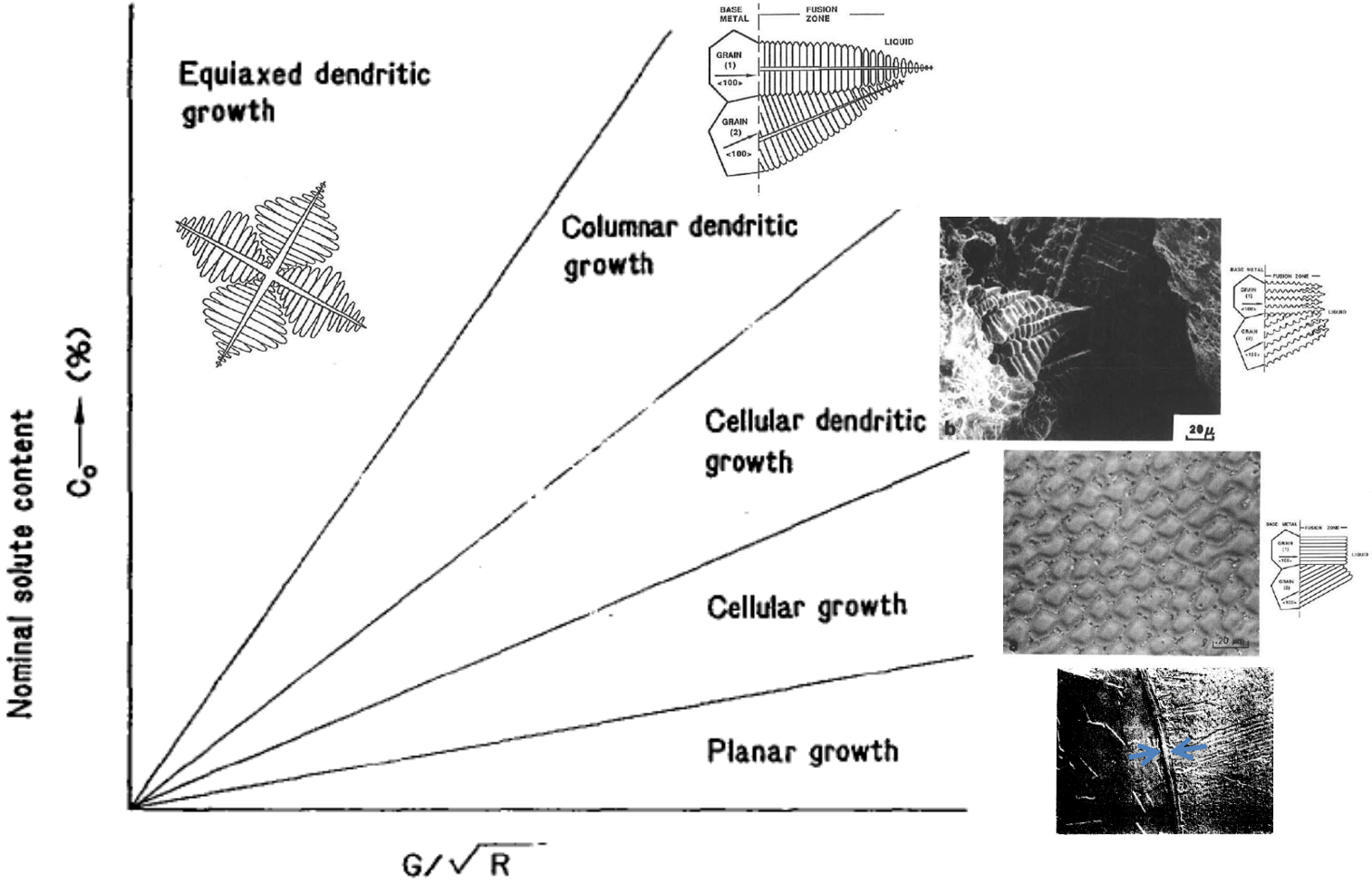
360W system using YAG laser at UCD



3-D LENS solidification landscape varied from the neck to body toward the substrate interface



Resulting solidification structures as a function of conditions



From Brooks and Mahin, SAND88-8210