Irradiation Performance Testing of Specimens Produced by Commercially Available Additive Manufacturing Techniques

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Advanced Methods for Manufacturing Program Review

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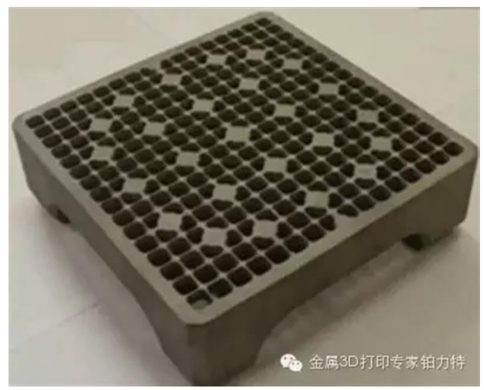
COLORADO SCHOOL OF MINES

EARTH • ENERGY • ENVIRONMENT

Relevance

- The nuclear industry is likely to benefit from additive manufacturing
  - Power conversion (small capillary heat exchangers, pipes, turbine blades)
  - Structural components (core plate assembly, channel grids, spacers, nozzles, plugs)
  - Fuel & Cladding (optimized geometries)
- Circumvents conventional constraints
  - Unique geometries
  - Rapid prototyping
  - Legacy parts
- Several irradiation studies are underway to start the process of qualifying AM metals for reactor use
  - GE Hitachi
  - Westinghouse

AM Heat exchanger (GE)

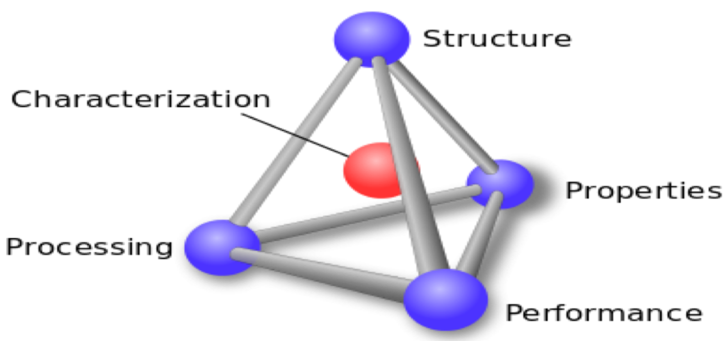


AM Lower tube socket for the CAP1400 nuclear fuel assembly (China National Nuclear Corporation)

World Nuclear Association

Challenges

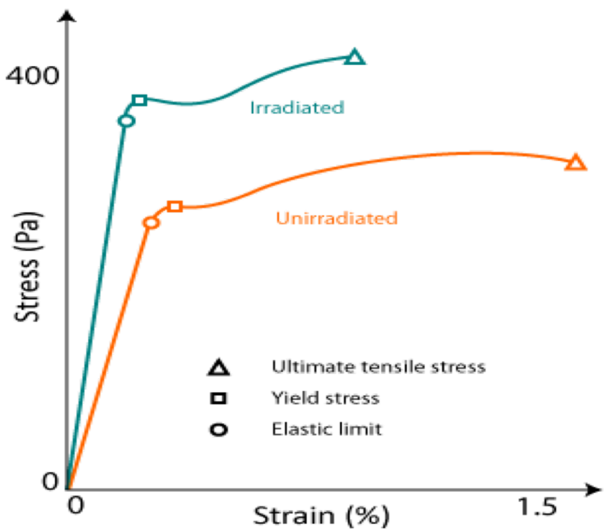
- Nuclear Industry implementation is limited by a lack of understanding regarding AM processing-structure-property relationships
    - Particularly related to irradiation performance



Barakah nuclear power plant (UAE)

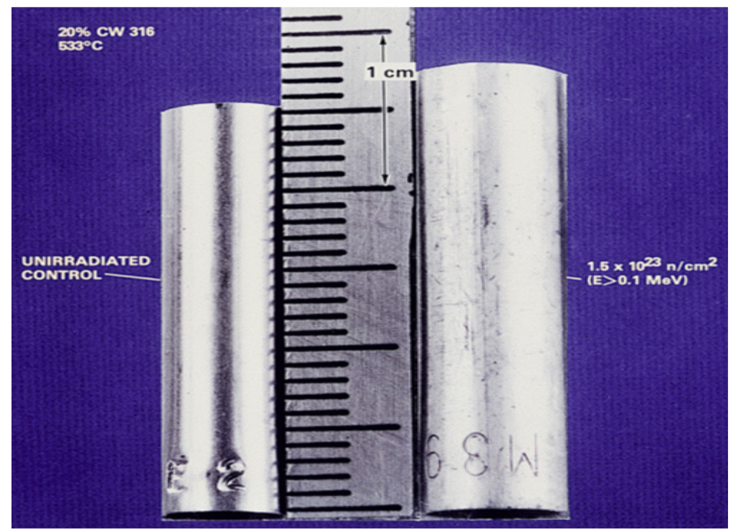
Irradiation Performance

- Build up of defects results in material changes
  - Low-dose - hardening and embrittlement
  - High-dose - phase instabilities, creep, swelling, helium-embrittlement



Hardening/Embrittlement

Creep Rupture



Swelling

Project Overview

- Test the performance of "commercially-available" AM materials in an irradiation environment
- Samples to undergo mechanical (tensile), thermophysical (laser flash/differential scanning calorimetry), and microstructural characterization (x-ray, SEM, TEM, EBSD, serial sectioning)

Specimen ID	Material	Method	Machine	Provider	Heat Treatment
SS P1	SS-316L	Laser Powder Bed	EOS M290	Elementum 3D	SR1
SS P2	SS-316L	Laser Powder Bed	ProX320B	3D Systems	SR1
SS P3	SS-316L	Laser Free Form	Optomec	INL	SR1
SS P4	SS-316L	E-Beam Wire Feed	Sciaky	Naval Reactors	SR1

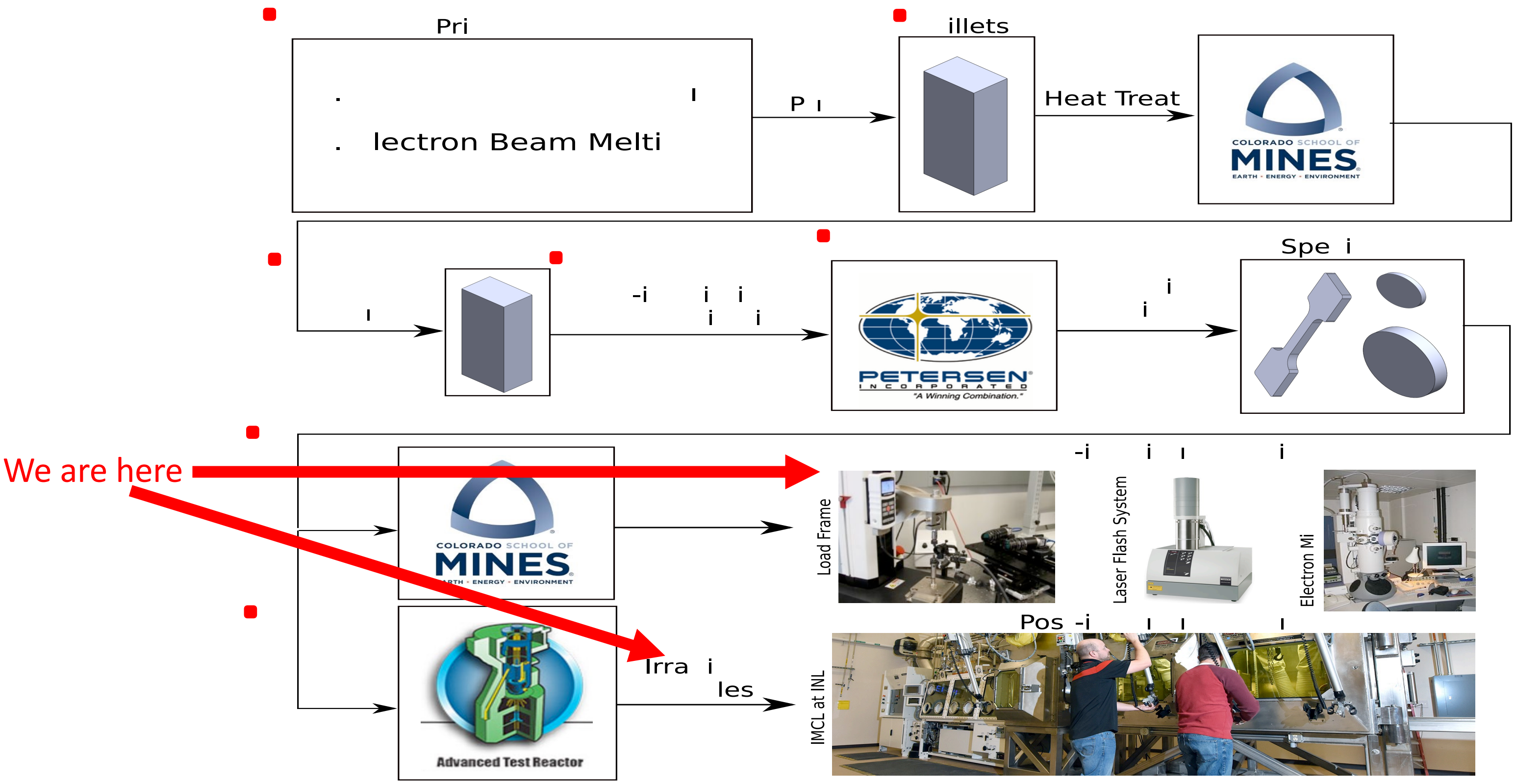
SR1 - Stress relieved @ 1650 F for 3.25 hours, air cool

Specimen ID	Material	Method	Machine	Provider	Heat Treatment
IN P1	IN-718	Laser Powder Bed	EOS M290	Elementum 3D	AMS 5664
IN P2	IN-718	Laser Powder Bed	ProX320B	3D Systems	AMS 5664
IN P3	IN-625	Laser Powder Bed	EOS M290	Elementum 3D	AN1
IN P4	IN-718	E-Beam Wire Feed	Modified Sciaky	Lockheed Martin	AMS 5664

AMS 5664 - Heat treat per AMS 5664 (solution anneal and aging)
AN1- Anneal at 870 °C for 1-hour, rapid cool

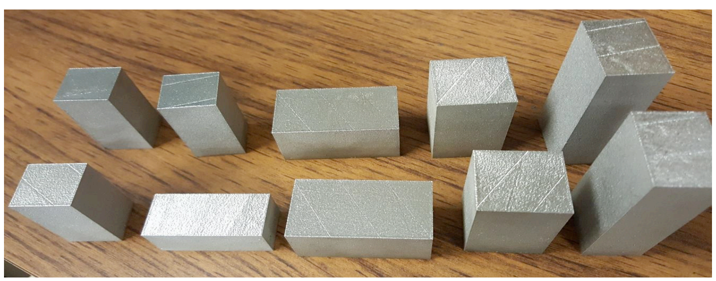
Project Plan

- Test the performance of “commercially-available” AM materials in an irradiation environment

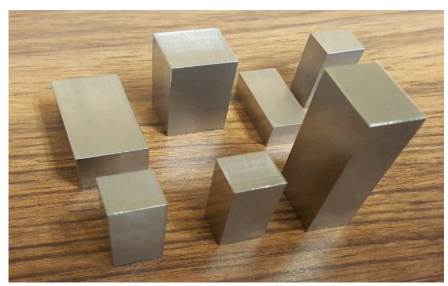


Laser Powder Bed	Elementum 3D	SS-316L	SS-P1	15-45 µm LPW	40 µm	Proprietary	Proprietary	Argon
		IN-718	IN-P1	15-45 µm Truform (Praxair)	40 µm	Proprietary	Proprietary	Argon
		IN-625	IN-P3	15-45 µm Truform (Praxair)	40 µm	Proprietary	Proprietary	Argon
Laser Powder Bed	3D Systems	SS-316L	SS-P2	Oerlikon Metco	30 µm	85, 215 W	450, 900 mm/s	Vacuum
		IN-718	IN-P2	15 µm LaserForm	30 µm	115, 220 W	625, 1180 mm/s	Vacuum
Laser Free Form	INL	SS-316L	SS-P3	-	-	400 W	150 g/hr	Argon
E-Beam Wire Feed	Naval Reactors	SS-316L	SS-P4	-	-	N/A	20 lb/hr	Vacuum
	Lockheed Martin	IN-718	IN-P4	-	-	N/A	-	Vacuum

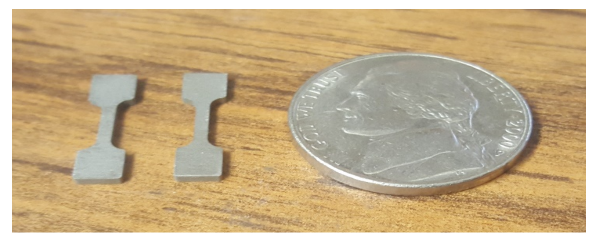
SS-316L: Stress relieved @ 1650 °F (899 °C) for 3.25 hours, air cool
IN-718: Heat treat per AMS 5664 (solution anneal @ 1065 °C for 1 hour and aging @ 760 °C for 10 hours and 650 °C for 8 hours)
IN-625: Anneal at 870 °C for 1-hour, rapid cool



As-built billets

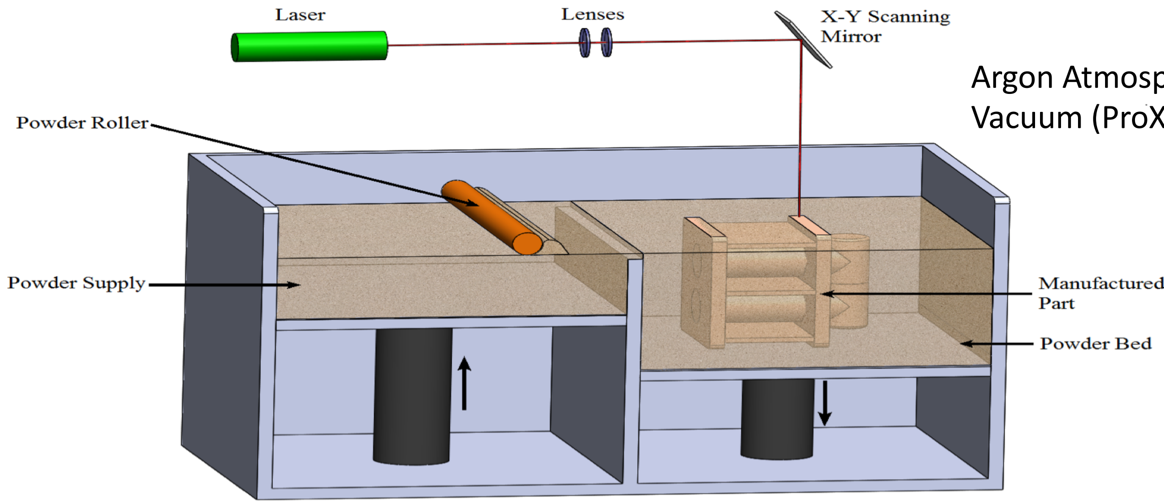


Skinned billets &



Tensile specimens &

Laser Powder Bed Fusion

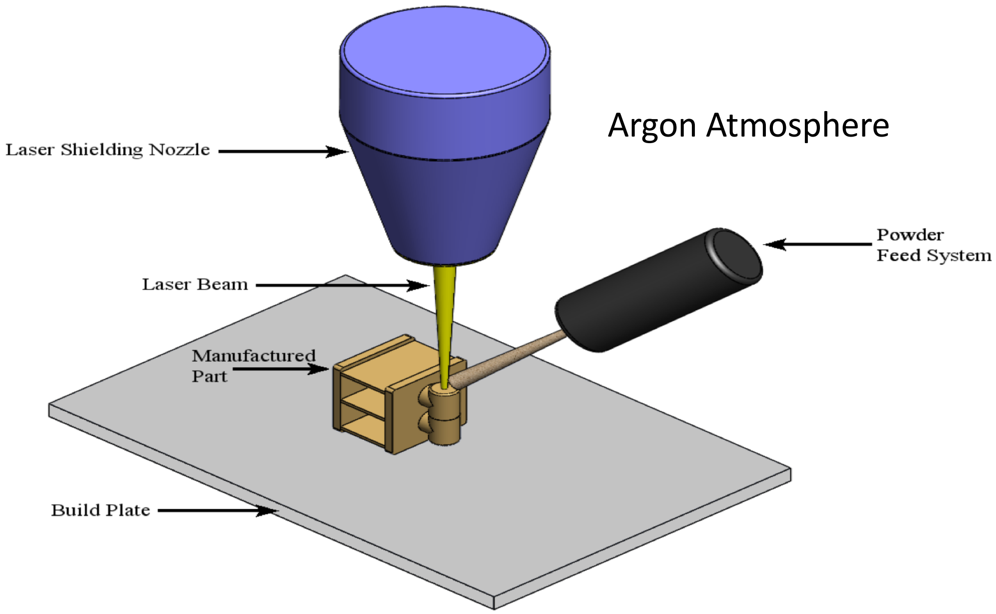


Argon Atmosphere (EOS)
Vacuum (ProX)

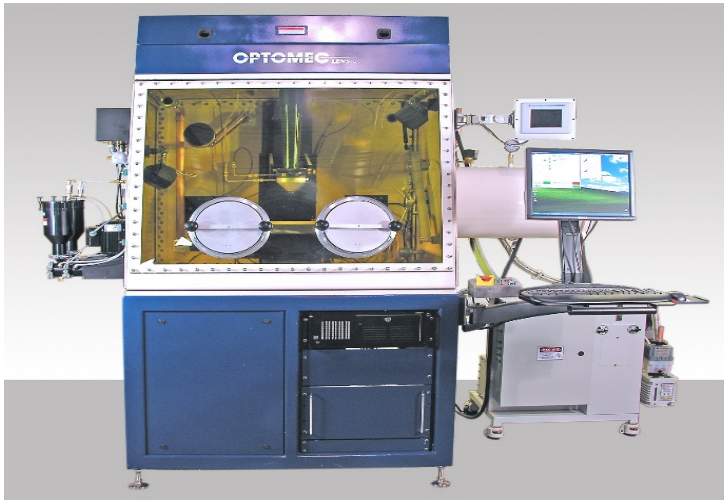
EOS M 290

ProX320B

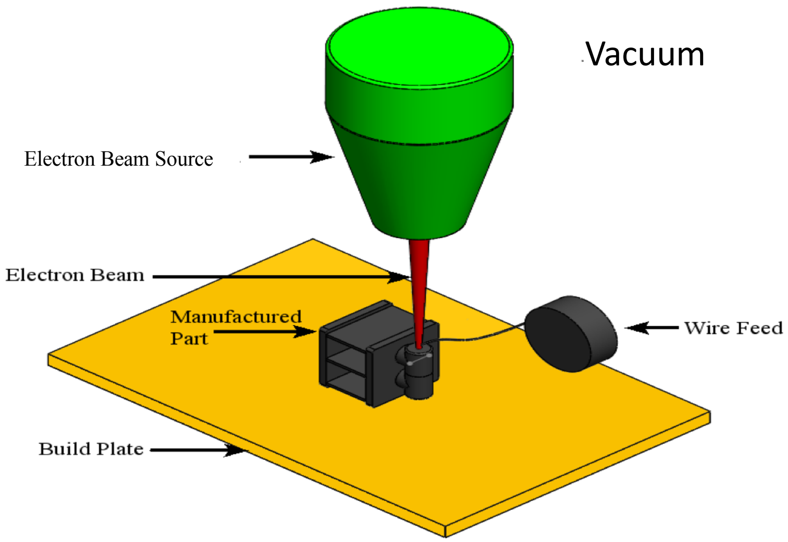
Laser Powder Free Form (Directed Energy Deposition)



Optomec MR7



Electron Beam Wire Feed (Directed Energy Deposition)



Sciaky

Pre-irradiation Characterization

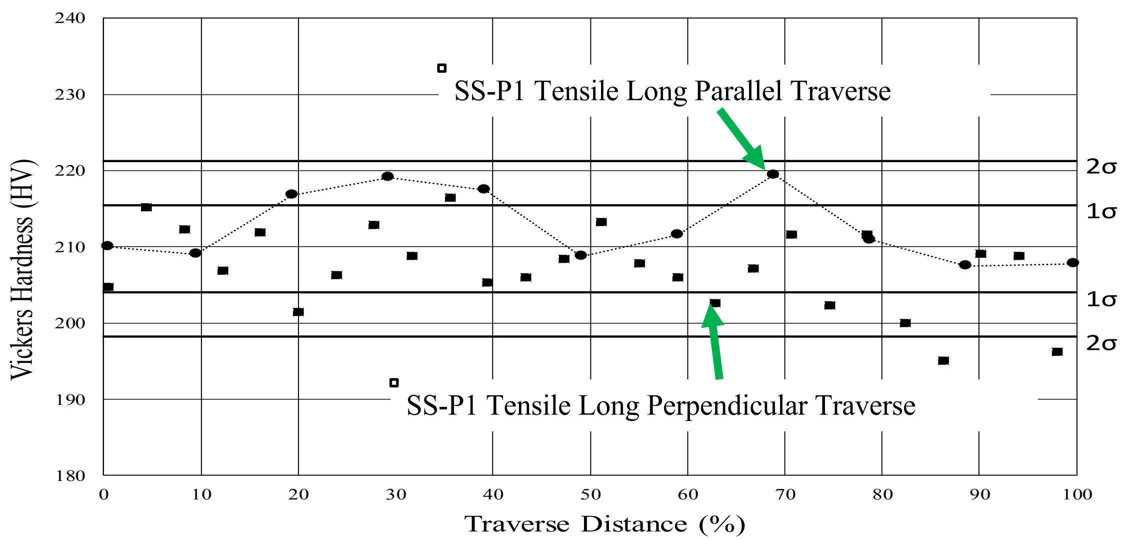
- A pre-irradiation characterization study verified that the billets met the minimum requirements necessary to produce specimens suitable for irradiation in the ATR
- *Density*
  - All theoretical densities must be above 95%
- *Microhardness*
  - No significant hardness gradients or inconsistencies
- *SEM/EDS*
  - Elemental composition is as expected
- *Optical Microscopy*
  - Microstructure is consistent across each sample
  - The microstructure is free of large voids or unreacted material that could cause specimen failure

Density

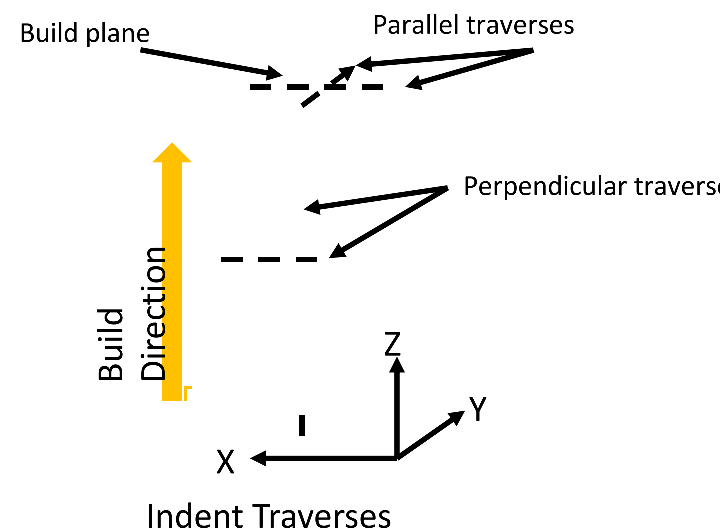
- The potential for AM samples to have excessive porosity was a major concern prior to reactor insertion
- Irradiated parts should have > 95% density
- Simple density calculations demonstrated that this is not a concern for these billets

PASS

Sample ID	[illegible]	[illegible]	[illegible]	% Theoretical
SS P1	[illegible]	[illegible]	[illegible]	97.93
SS P2	[illegible]	[illegible]	[illegible]	98.93
SS P3	[illegible]	[illegible]	[illegible]	99.38
SS P4	[illegible]	[illegible]	7.95	99.35
IN P1	[illegible]	[illegible]	8.18	99.58
IN P2	[illegible]	[illegible]	8.19	99.61
IN P3	[illegible]	8.44	8.39	99.36
IN P4	[illegible]	8.22	8.07	98.21



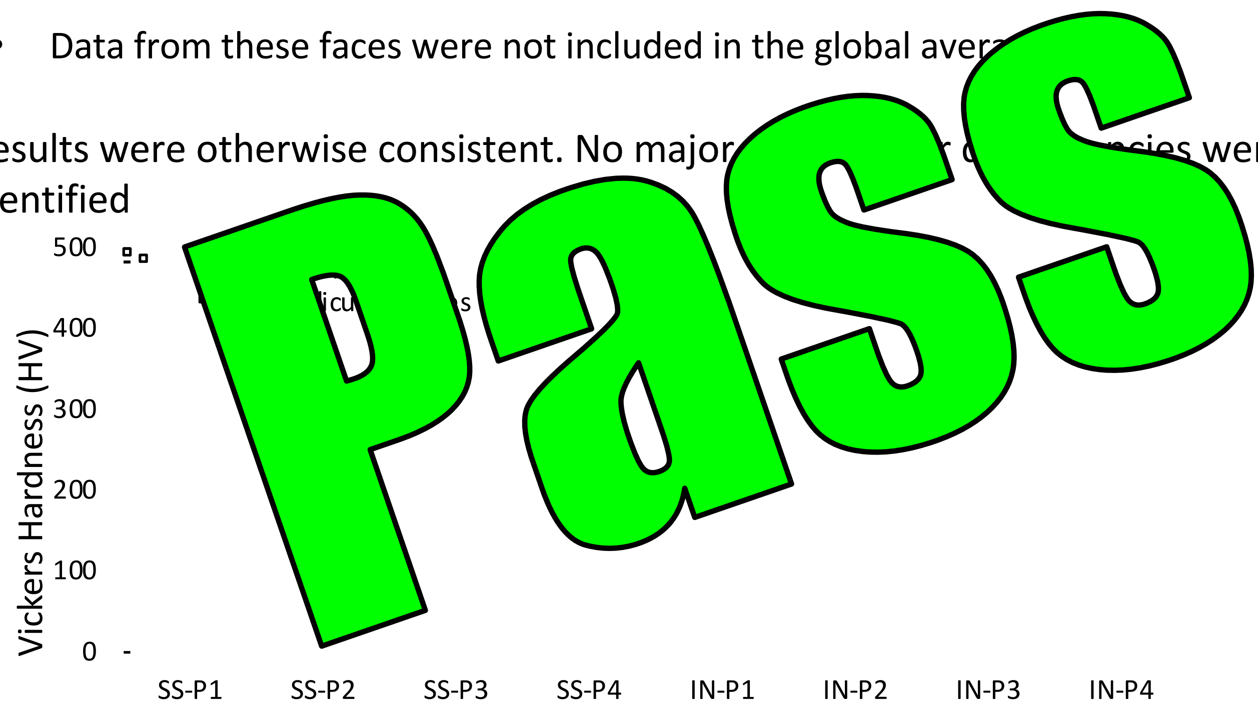
Vickers Hardness (HV)
240
230
220
210
200
190
180
0
10
20
30
40
50
60
70
80
90
100
Traverse Distance (%)
SS-P1 Tensile Long Parallel Traverse
SS-P1 Tensile Long Perpendicular Traverse
2σ
1σ
1σ
2σ



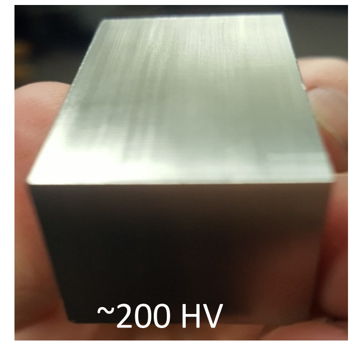
Build plane
Parallel traverses
Perpendicular traverse
Build Direction
Z
Y
X
Indent Traverses

Microhardness (cont.)

- Some work hardening from sample skinning was observed on individual faces
  - Hardening depth was small (< 1 mm) and did not impact specimens
  - Data from these faces were not included in the global aver
- Results were otherwise consistent. No major r d ncies were identified



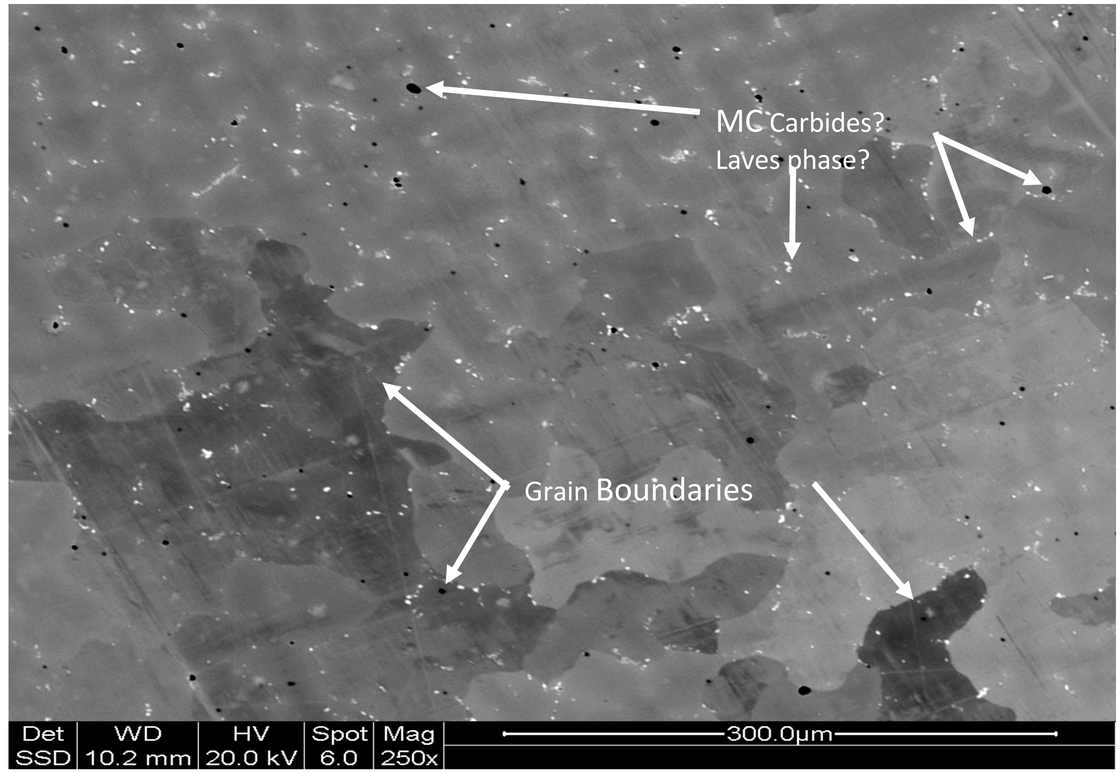
Face Mill



End Mill

Electron Microscopy

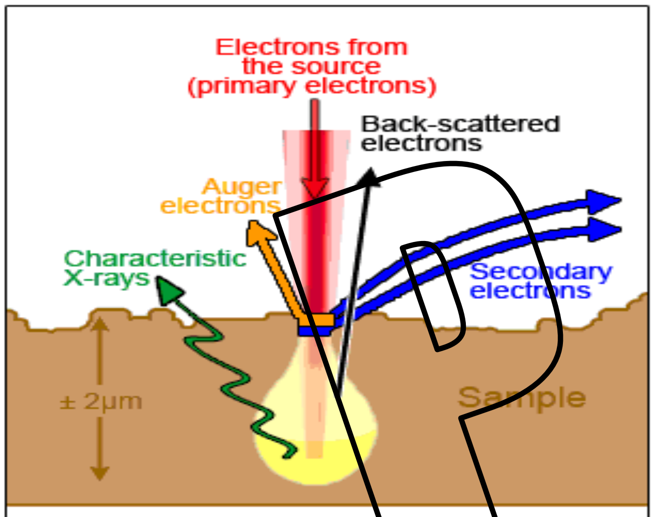
- One sample of each material was examined in three locations across each polished face to verify sample composition and uniformity
- SEM imaging provided expected results

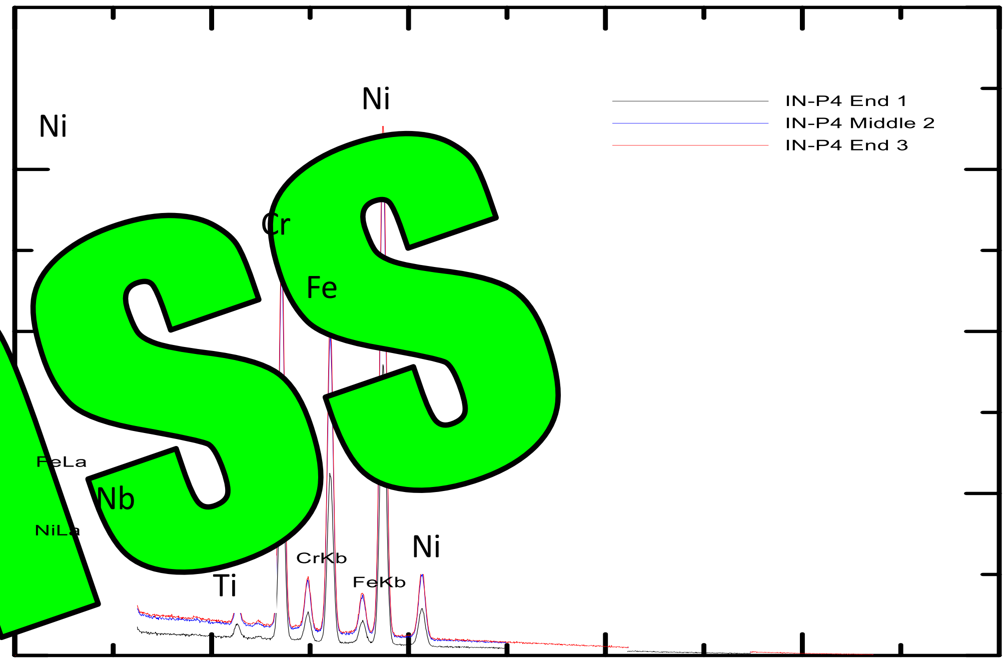


IN-P4 backscatter SEM image

Energy-dispersive X-ray Spectroscopy (EDS)

- Elemental compositions were as expected

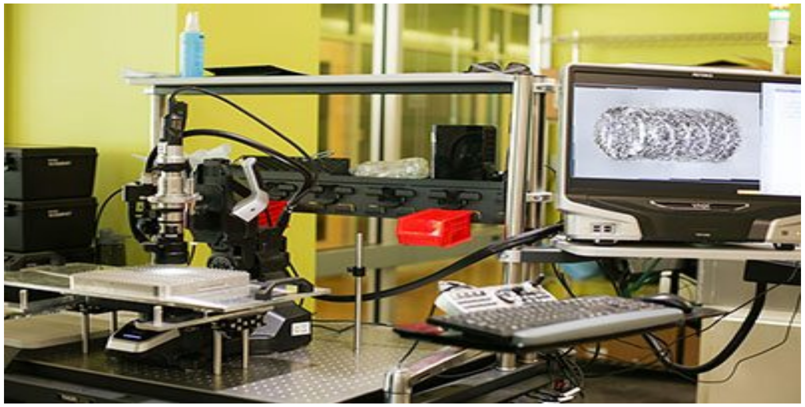




Optical Microscopy

- Polished samples etched using Kalling’s no. 2 reagent
- Images taken on a Keyence VHX-5000
- Full surface scans looked for significant porosity and/or anisotropy in crystallographic size or orientation
- Results highlight the microstructures associated with the different AM methods

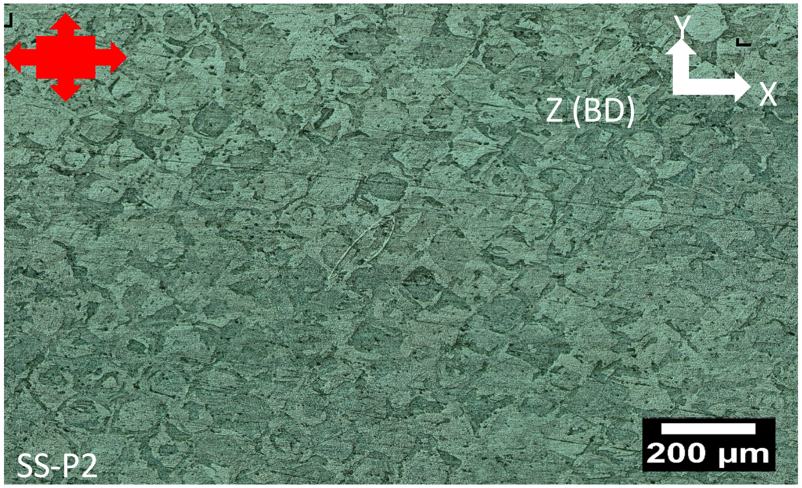
CSM Physical Metallurgy Lab

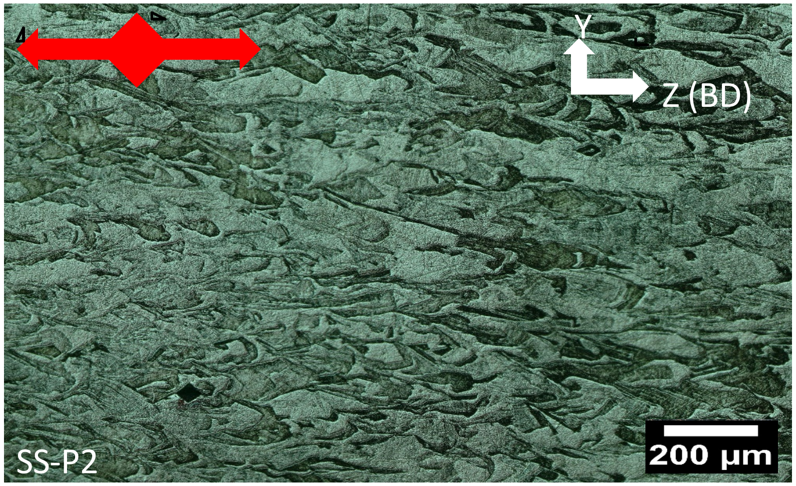


ADAPT’s Keyence Microscope

Laser Powder Bed SS-316L (ProX)

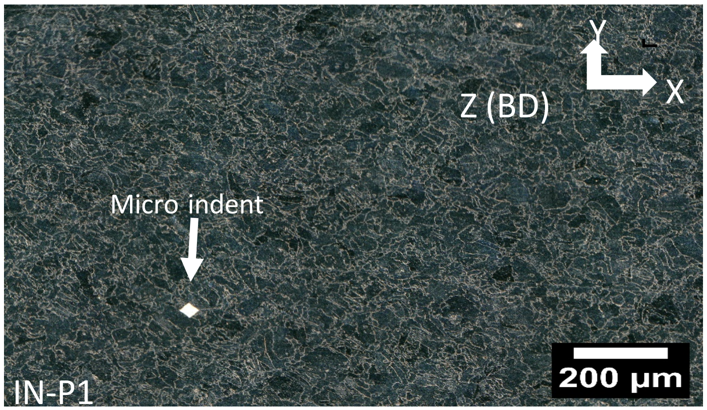
- Clear dependence on build orientation
  - Chessboard-type grain pattern normal to build direction
  - Elongation along the build direction
  - Strongly textured

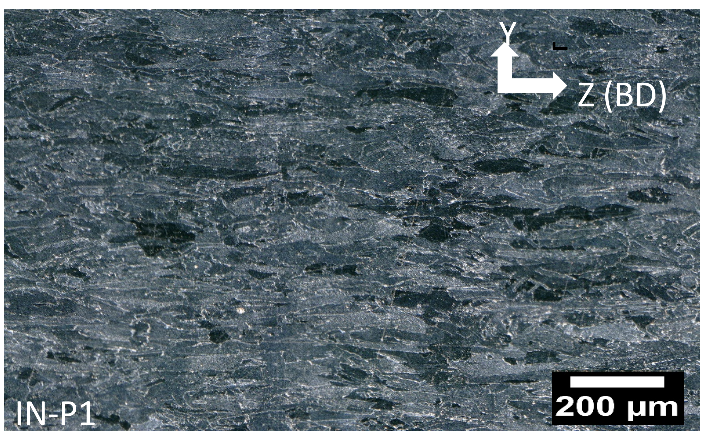




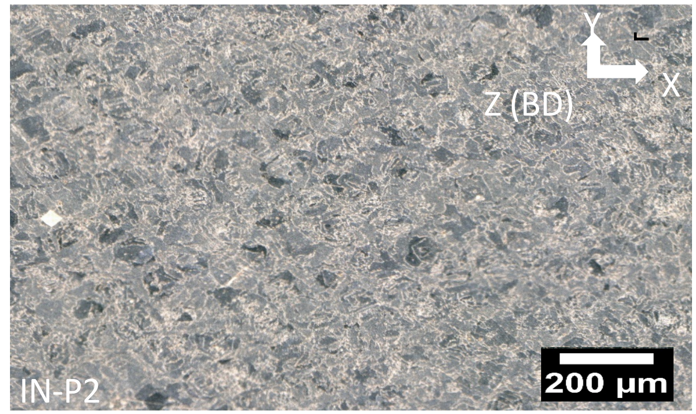
Laser Powder Bed Inconel-718

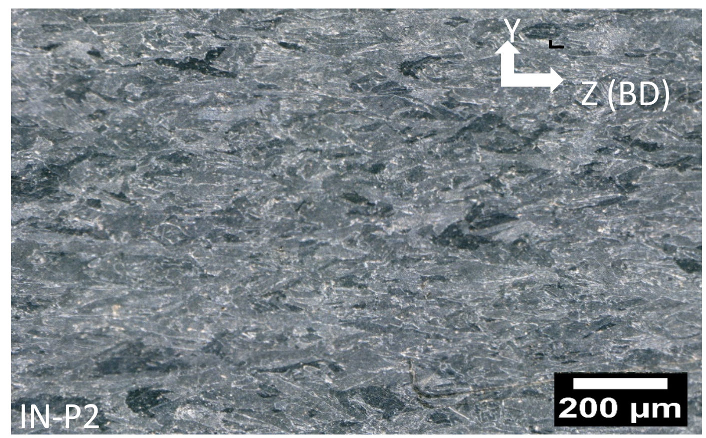
EOS





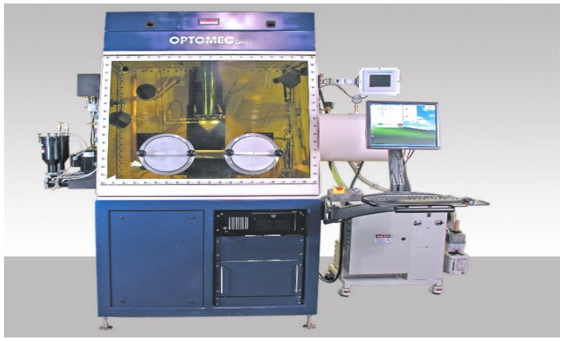
ProX





Laser Powder Free Form SS-316L

- Imbricated (fish-scale) features perpendicular to the build path
  - Present in most samples prior to heat treating
- Grains appear to form independently of the melt path, spanning across boundaries of the raster pattern
- INL had some concern about pauses during printing, but microstructural evidence was not obvious
  - Possibly eliminated by heat treatment

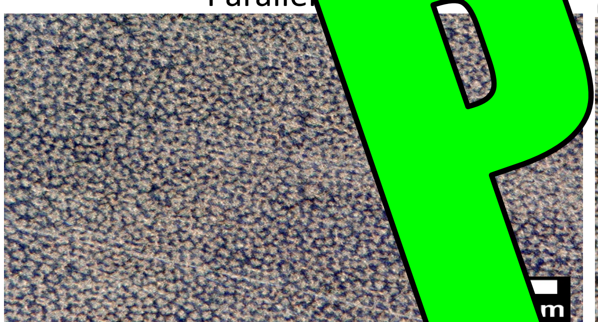


E-beam Wire Feed SS-316L

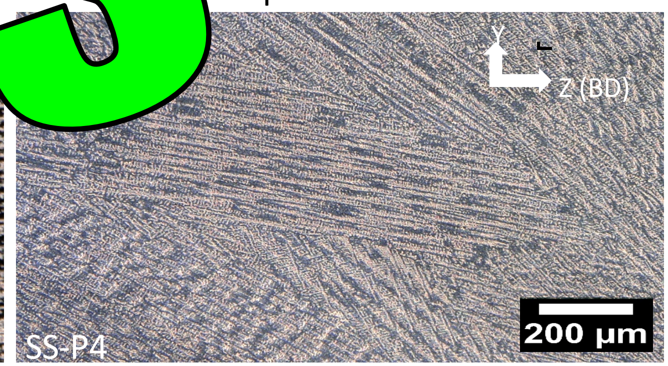
- Feature size is significantly larger compared to powder fusion machines
  - Imbricated pattern still visible without magnification after heat treatment
- Dendritic features orient themselves relative to the build direction
- Growth pattern is interrupted and … at … boundaries

Dendrites

Parallel



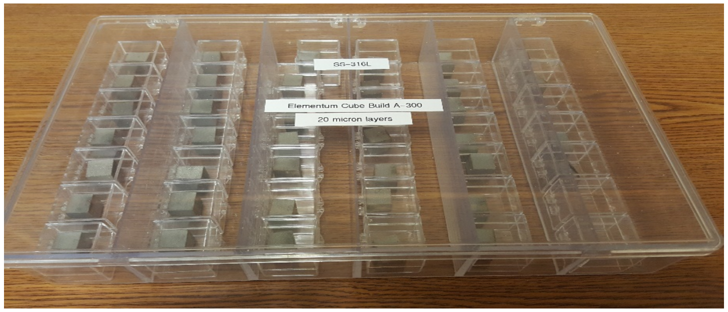
Perpendicular



Next Steps

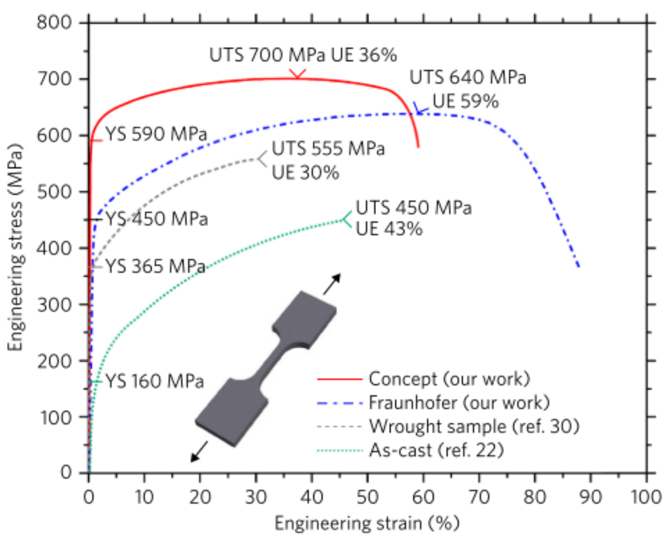
- Irradiation is in progress (specimens to be shipped to HFEF in April)
- Mechanical and thermophysical testing of control (non-irradiated) specimens is in progress at Mines
- Post-irradiation testing and examination to be conducted by the NSUF
- Compare data and draw conclusions that could aid in the qualification of AM processes for nuclear applications

- Additionally, consider how the sub-grain cellular defect structures resulting from many AM methods might impact irradiation performance

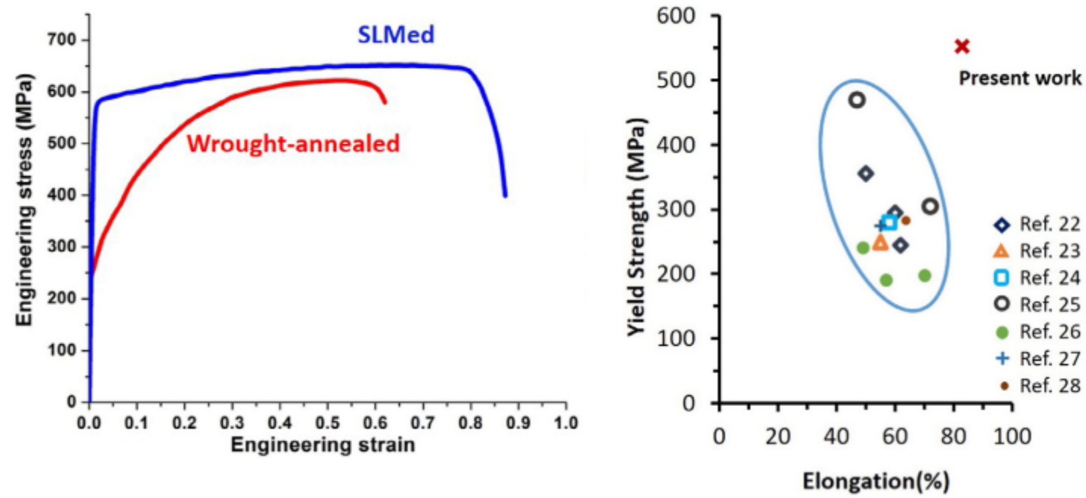


Sub-Grain Cellular Structures - Motivation

- Recent papers in the AM literature have demonstrated improved mechanical properties compared to wrought metals
  - Simultaneously increased strength and ductility



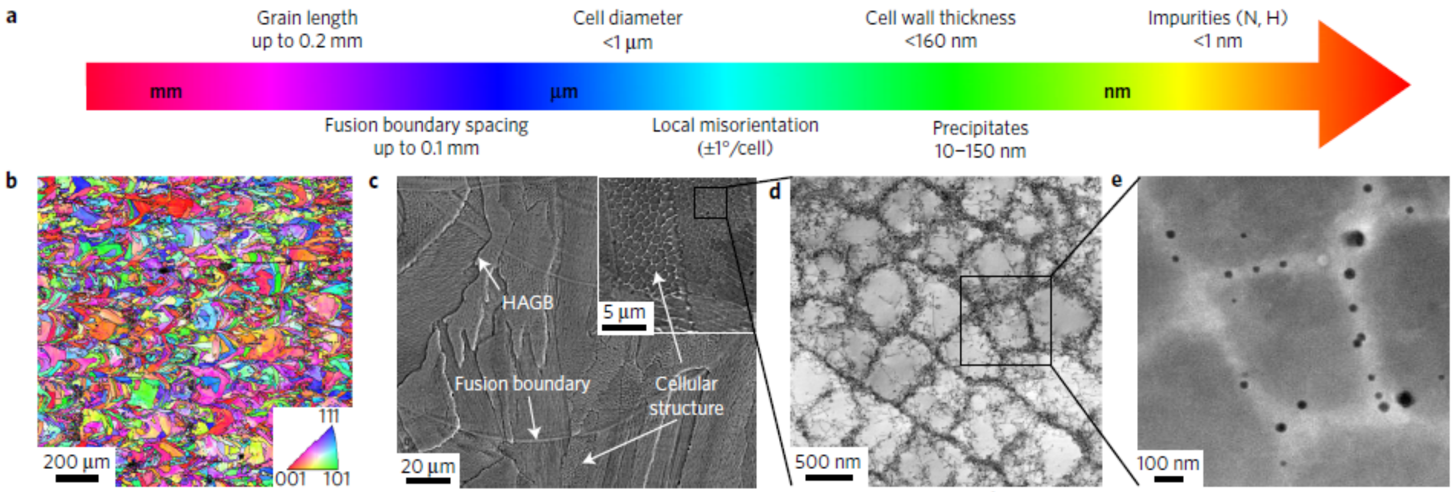
Wang et al., *Nature Materials*, October 2017



Liu et al., *Materials Today*, May 2018

Sub-Grain Cellular Structures - Motivation (cont.)

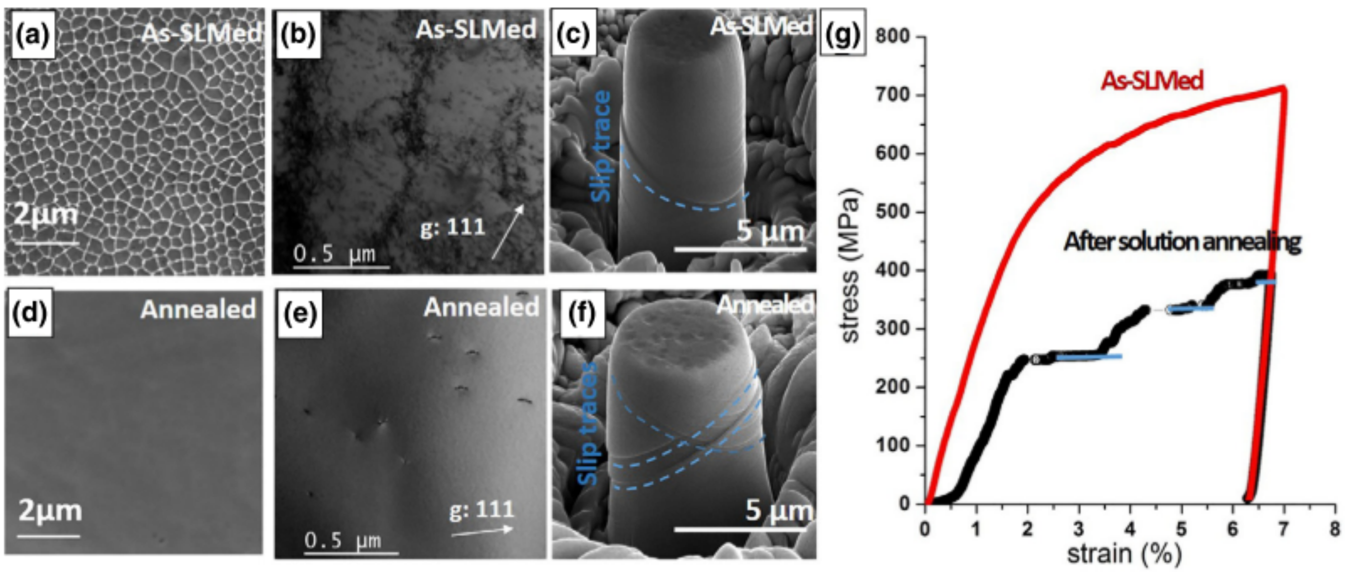
- These results are attributed to cellular structures formed during rapid solidification
  - Cell walls consist of a network of dislocations and segregated alloying elements
  - These walls limit dislocation motion (strengthening) but also allow for continuous plastic flow by transmitting the impeded dislocations through them
  - Segregated elements pin the dislocation network and maintain its size



Wang et al., *Nature Materials*, October 2017

Consequences of Heat Treatment

- Annealing SS-316L at 1050 °C for 2 hours wiped out the cellular dislocation network
  - Current irradiation studies may not capture the impact of these structures



Liu et al., *Materials Today*, May 2018

The Problem

- The impact of this effect is two-fold
  - Better than wrought
  - Highlights the potential to customize the microstructure to the application
- Advanced structures like this one could be a boon for nuclear
- However, nuclear changes the equation
  - Corrosive environment
  - Thermal flux
  - Radiation flux
- **Will the cellular structures survive?**
  - Do they break down?
  - At what point?
  - Even if they are annihilated, what impact do the structures have on long-term irradiation performance?

Acknowledgements

- Nuclear Science User Facilities
- Elementum 3D, 3D Systems, Idaho National Laboratory
  - Sample development and printing
- Lockheed-Martin, Naval Reactors
  - Less common source material
- Alliance for the Development of Additive Processing Technologies (ADAPT)

Questions/Comments?