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Laser Powder Bed Fusion of 316H Stainless Steel for High-Temperature Nuclear Applications

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Content

- Background
- Process parameter study
- Variations in powder feedstock
- Thermal aging effect
- Mechanical testing

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- **Background**
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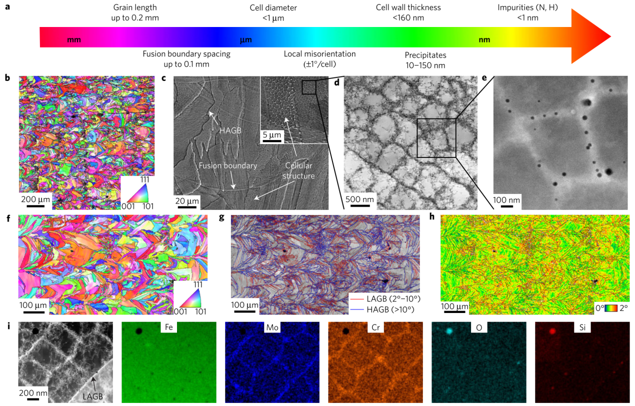
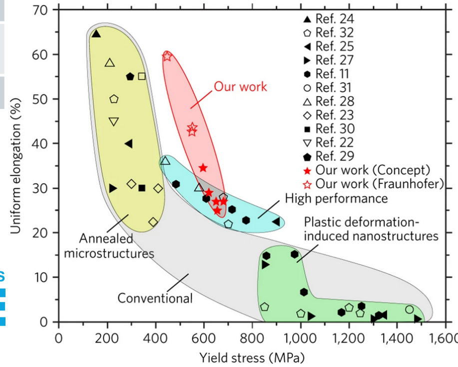
Why qualifying LPBF 316H SS

- **Opportunities for additive manufacturing in nuclear energy:**
    - Innovative component design
    - New materials development
    - Embedding sensors for real-time monitoring
    - Repair/replace obsolete parts
- **Program interest:** DOE-NE's Advanced Materials and Manufacturing Technologies (AMMT) program has an overarching vision of accelerating the development, qualification, demonstration and deployment of advanced materials and manufacturing technologies to enable reliable and economical nuclear energy*.
- **Industry interest and code availability:** The wrought form of 316H SS is one of the six qualified materials in the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code (BPVC) Section III, Division 5 for high temperature reactor construction.
- **Research readiness:** Extensive research has been performed on LPBF 316L SS (L for low carbon) as a reference for LPBF 316H SS. Data show that the LPBF material has a potential for improved performance due to the unique microstructure.
- **LPBF 316H SS** has been chosen by AMMT program* based on the recent material scorecard** work as **the first target material** for design improvements, materials optimization and rapid qualification. It also serves as a test case for qualifying other materials produced by advanced manufacturing.

The laser powder bed fusion (LPBF) process with a Renishaw AM400 machine.

Composition (wt.%)	Type 316H UNS S31609	Type 316L UNS S31603
Fe	Bal.	Bal.
Cr	16.0-18.0	16.0-18.0
Ni	11.0-14.0	10.0-14.0
Mo	2.00-3.00	2.00-3.00
Mn	2.0	2.0
Si	1.00	1.00
C	**0.04-0.10**	**0.035**
O	- -	- -
N	- -	- -
P	0.045	0.045
S	0.030	0.030





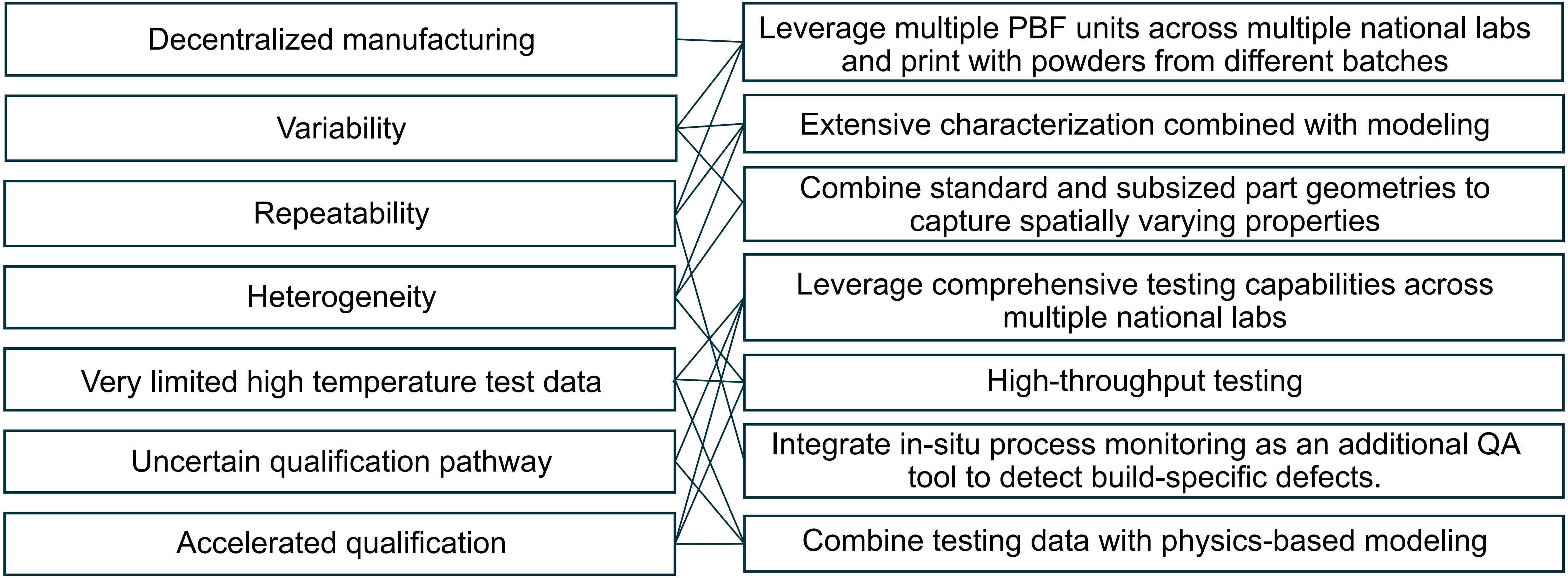
Wang et al. Nature Materials, 17 (2018)

M. Li, et. al., "Advanced Materials and Manufacturing Technologies (AMMT) 2022 Roadmap", ANL-23/12, 2022
**T. Hartmann, et. al., "Materials scorecards, Phase 2," PNNL-32744, PNNL, March 2022.*

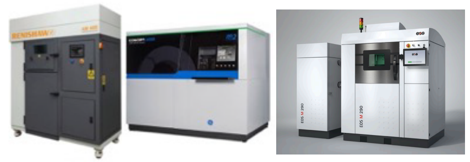
Challenges and approaches in qualifying a LPBF part

Challenges

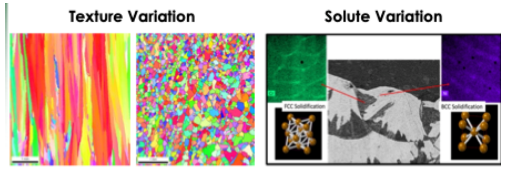
Approaches



Approach to qualification: a multi-lab effort (ANL, INL, LANL, ORNL, PNNL)

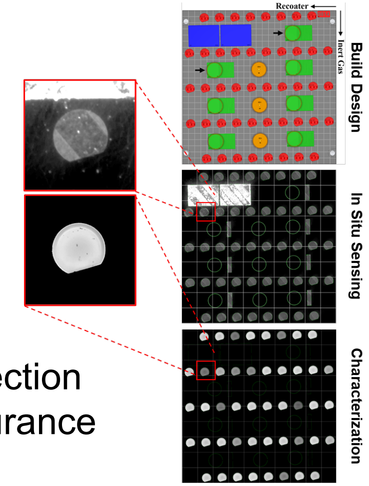


Machine variability
Geographic effects

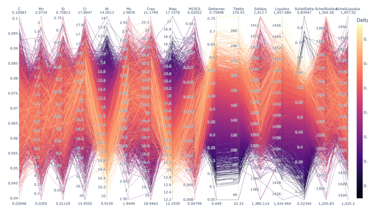


Process parameters
Spatial variation
Geometry effects

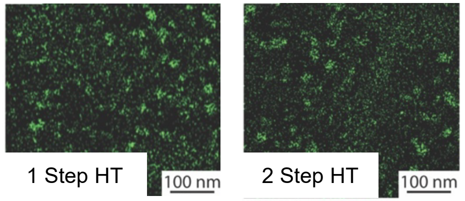
- Process Development & Understanding
- Alloy Chem. Variations
- In-situ Sensing & Implementation
- Post-build Processing
- Round Robin Testing
- Modeling



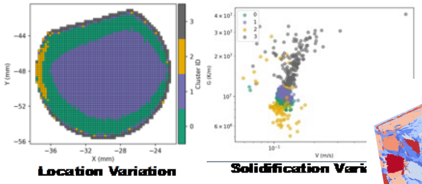
Defect detection
Quality assurance

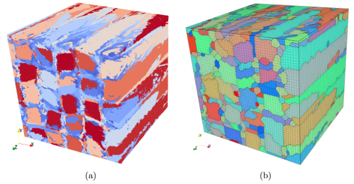


Feedstock variability



Grain structure and precipitation
Heterogeneity control





Process modeling
Microstructure modeling
Thermo-kinetic modeling

Performance evaluation

Laboratory Specific Contributions: Powder and Machine Variation

	ANL	ORNL	LANL
Renishaw	✓ (green)	✓ (green)	
Concept Laser		✓ (green)	
EOS		✓ (black)	✓ (green)

	ANL	ORNL	LANL
0.08 wt% Praxair	R ✓ (green)	R ✓ CL (green)	E ✓ (green)
0.06 wt% Praxiar	R ✓ (green)	✓ CL (black)	
0.05 wt% Praxiar	R ✓ (green)		
0.04 wt% PAC		R ✓ CL (black)	E ✓ (green)

■ Data generated as of 10/19/23

✓ (black) Available to print
✓ (green) Builds complete
R = Renishaw AM400
CL = Concept Laser
E = EOS 290

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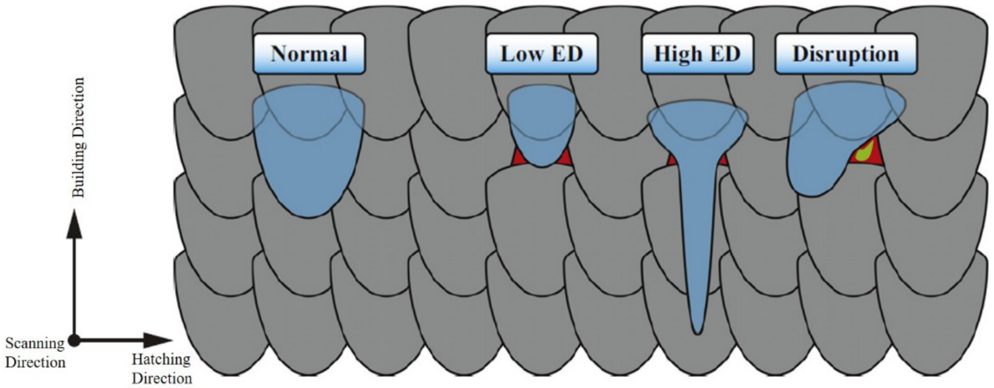
Importance of Processing Parameters

Volumetric Energy Density

$$ED_{vol} = \frac{P}{vhd}$$

P is power (W)
v is velocity (mm/s)
h is hatch spacing
d is layer thickness



- NORMAL "Conduction mode" = adequate penetration and overlap to the previous layers and adjacent melt pools
- LOW ED = low laser power or high lasing speeds that produce a much smaller "convection mode" shaped melt pool. Results in lack of fusion between layers and among adjacent melt pools
- HIGH ED = excessively high laser power and low lasing speeds that concentrates the heat making it penetrate through too many layers. Forms keyholing, and lack of fusion among adjacent melt pools from insufficient overlapping.
- DISRUPTED = poor process parameters, corrupt material properties, contaminants, etc.

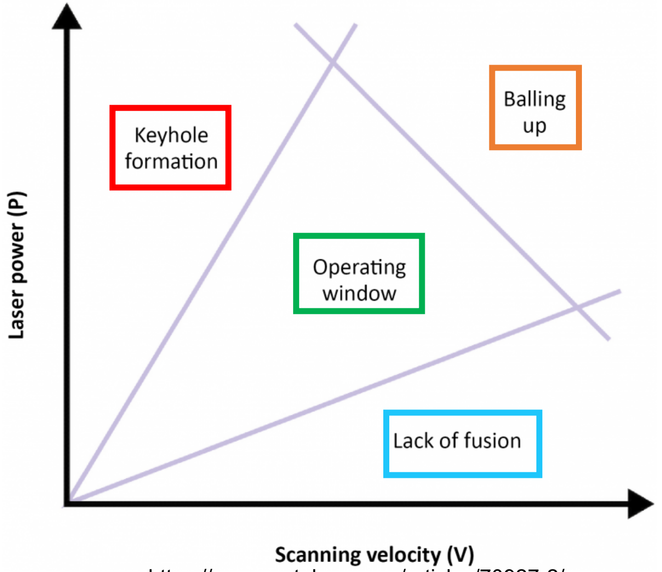
LANL: a single-bead study

Process understanding for qualifying LPBF 316H SS, LA-UR-23-30967, 2023

Objective: Develop process-structure-property data sets linked to in situ monitoring data and detailed feedstock characterization to strengthen process - structure and process -property relationships.

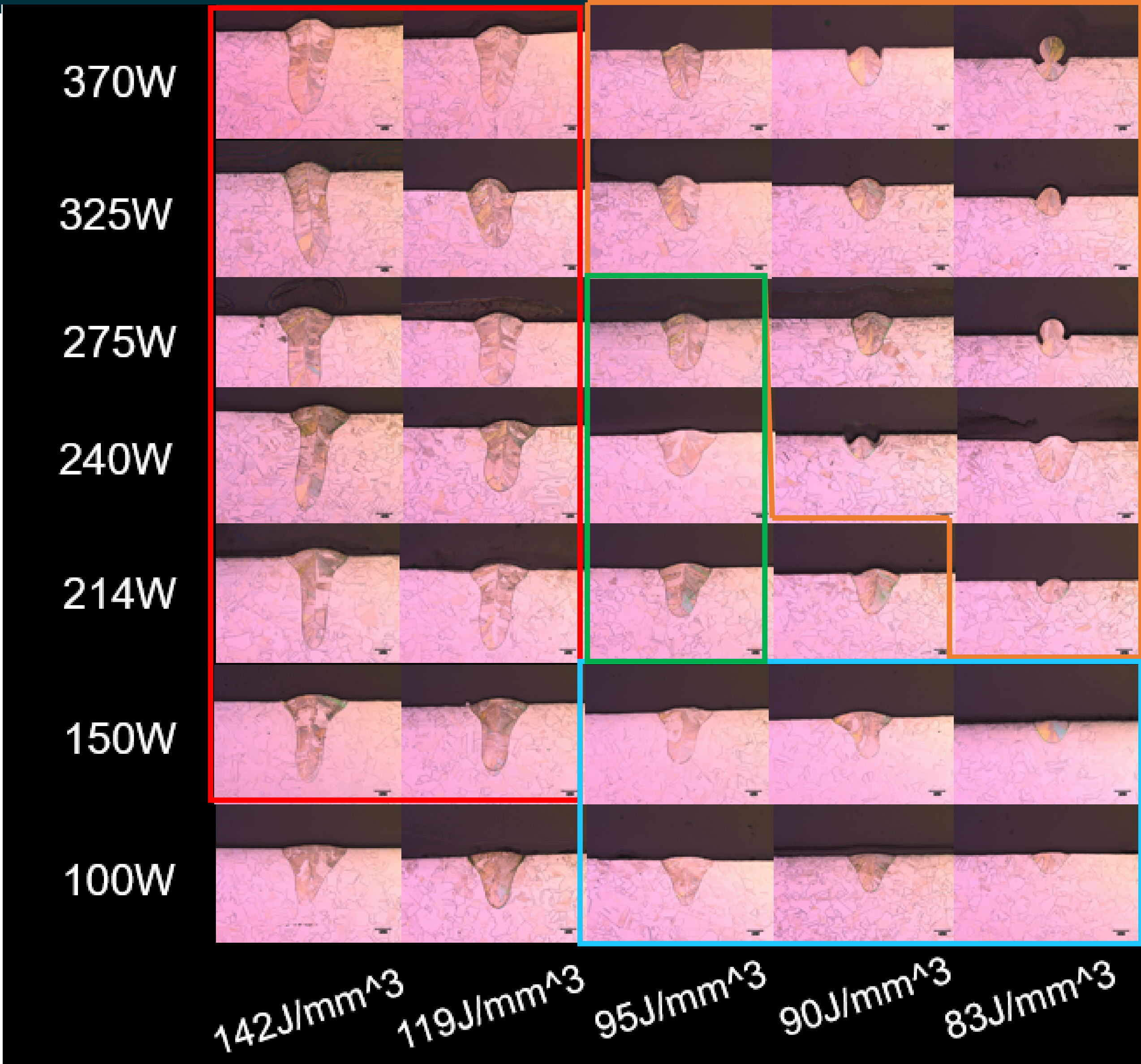
Initial Development

- Optimizing process parameters using single bead welds to find the acceptable operating window (OW) that provides full density parts.
- With layer thickness held constant, the P vs. V chart indicates the behavior for single bead trials and LPBF parameters
- Parameter used for 316H builds were based on 3 sets from the OW and three variation sets



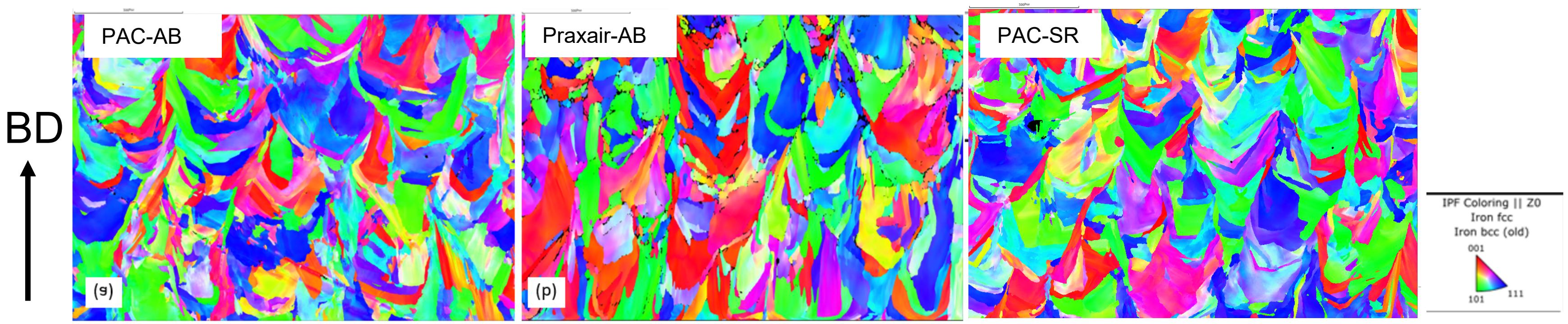
https://www.metal-am.com/articles/70927-2/

Set	Power (W)	Speed (mm/s)	ED (J/mm^3)
13 OW	275	688	95
18 OW	240	600	95
23 OW	214	525	95
Var. 1	257	644	95
Var. 2	227	567	95
Var. 3	200	521	95



LANL: 316H Characterization

EBSD



All as-built and stress-relieved materials showed grain morphology typical to the laser printing process. Additionally, the microstructures are non-equilibrium and are weakly textured.

Surface Roughness of As Built Parts

Set	Surface Roughness				
	Area1				
	Sa	Sz	Str	Spc	Sdr
	µm	µm		1/mm	
13 - OW	8.073	106.257	0.745	120.235	0.1696
18 – OW	8.540	133.659	0.628	201.005	0.2078
23 – OW	7.980	116.047	0.566	136.678	0.2067
Variation #1	8.311	125.209	0.480	165.832	0.1753
Variation #2	7.181	111.503	0.634	111.176	0.1360
Variation #3	7.061	106.431	0.597	161.975	0.1558

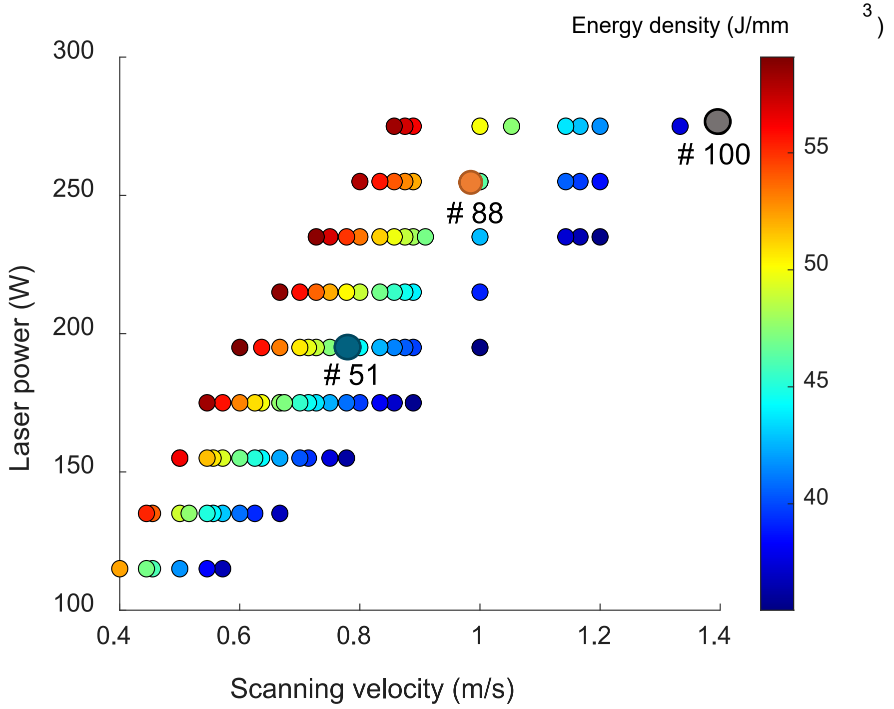
Porosity

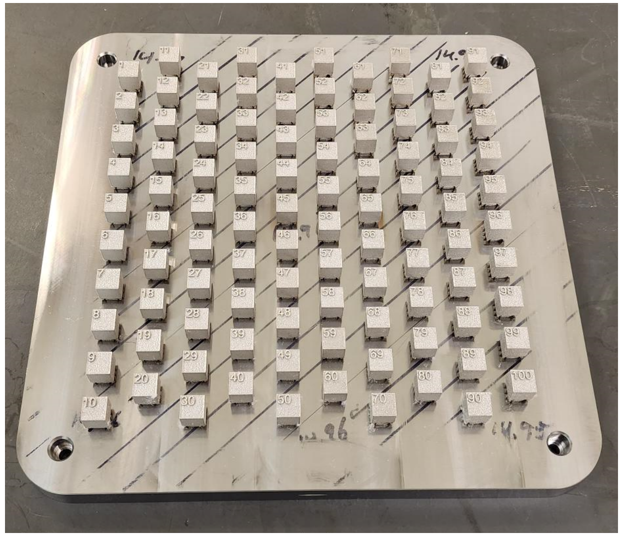
Set	As-Built		Stress-relieved
	Praxair Porosity %	PAC Porosity %	PAC Porosity %
13 – OW	0.049	0.069	0.046
18 – OW	0.054	0.051	0.057
23 – OW	0.176	0.236	0.159
Variation #1	0.069	0.139	0.017
Variation #2	0.107	0.257	0.02
Variation #3	0.085	0.058	0.029

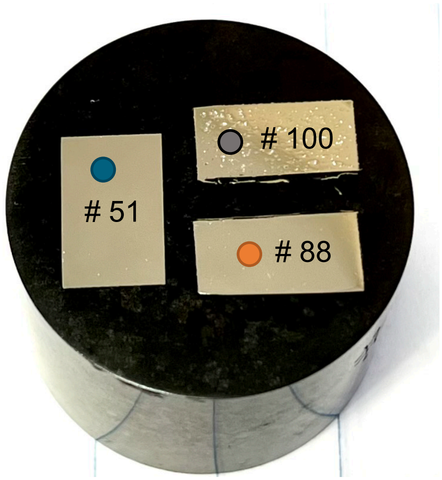
ANL: Building upon LANL single track experiments, exploring high-throughput printing

Development of process parameters and post-build conditions for qualification of LPBF 316 SS, ANL-AMMT-004, 2023

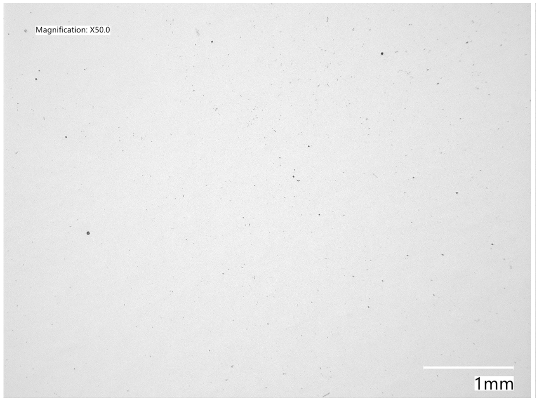
- Build 20230303 has 100 sets of laser parameters for 100 cubes
  - 0.06% carbon, 0.03% oxygen in powder
  - Varying **laser power, point exposure time, point distance**



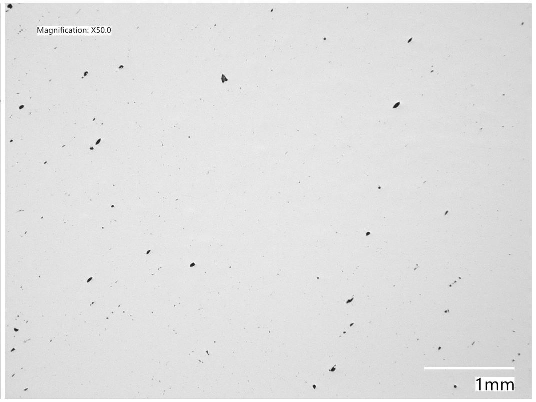




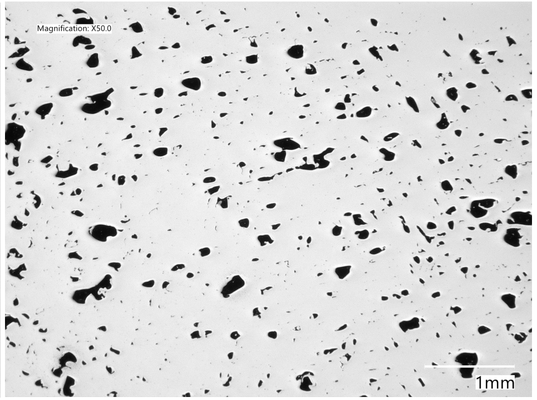
51: 0.05% areal porosity



88: 0.23% areal porosity



100: 8.82% areal porosity



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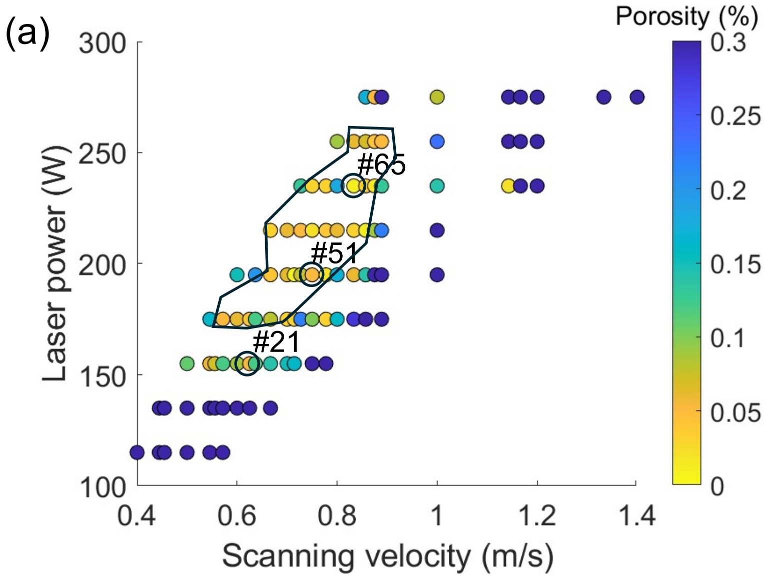
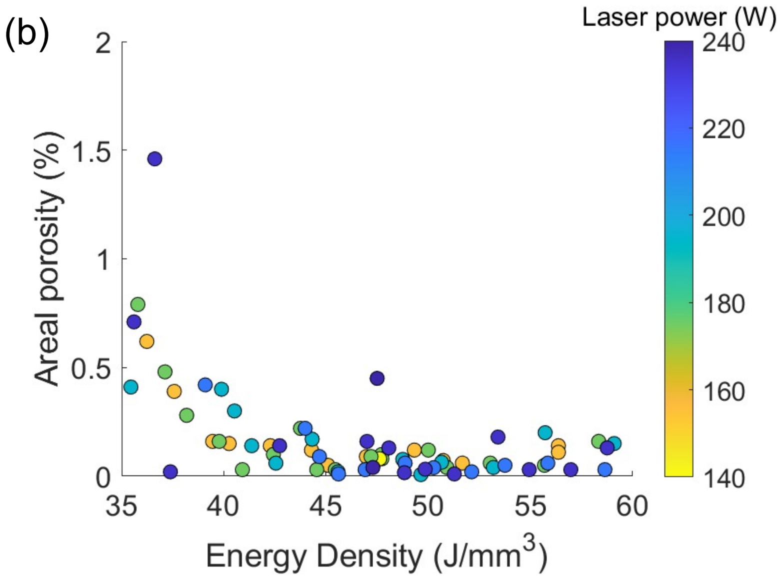
ANL: An optimum process window identified

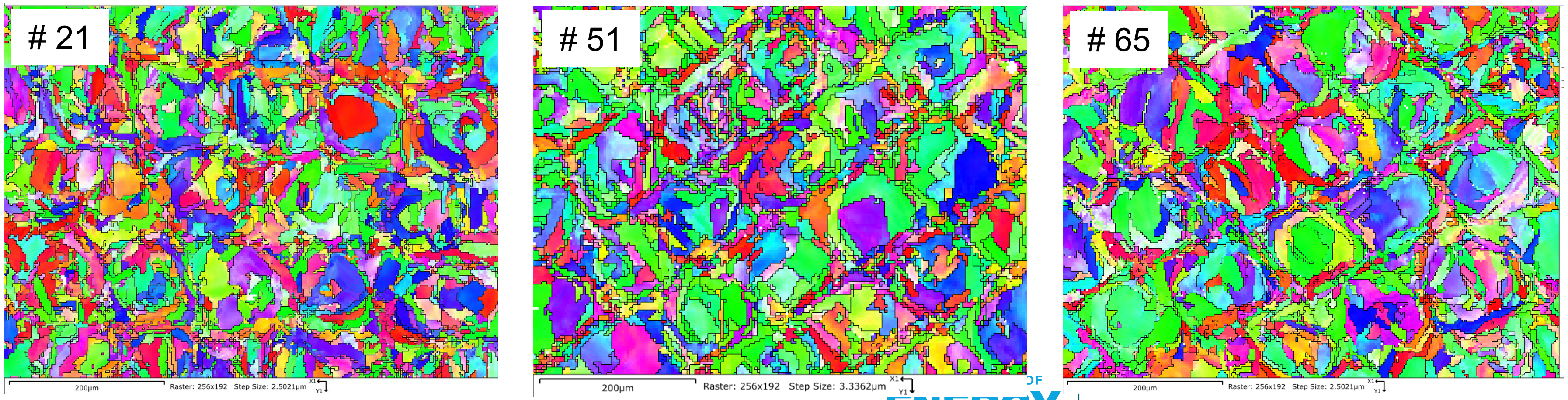
Development of process parameters and post-build conditions for qualification of LPBF 316 SS, ANL-AMMT-004, 2023

Observations:

- Through optical examination of interior surfaces of the cubes, an optimum operating window is identified.
- This window corresponds to an optimum energy density range with ~ 40 J/mm^3 as the minimum.
- Grain structures of materials printed with similar energy density (47 J/mm^3) are similar, displaying random textures.
- The grain structures reflects the rapid solidification process.







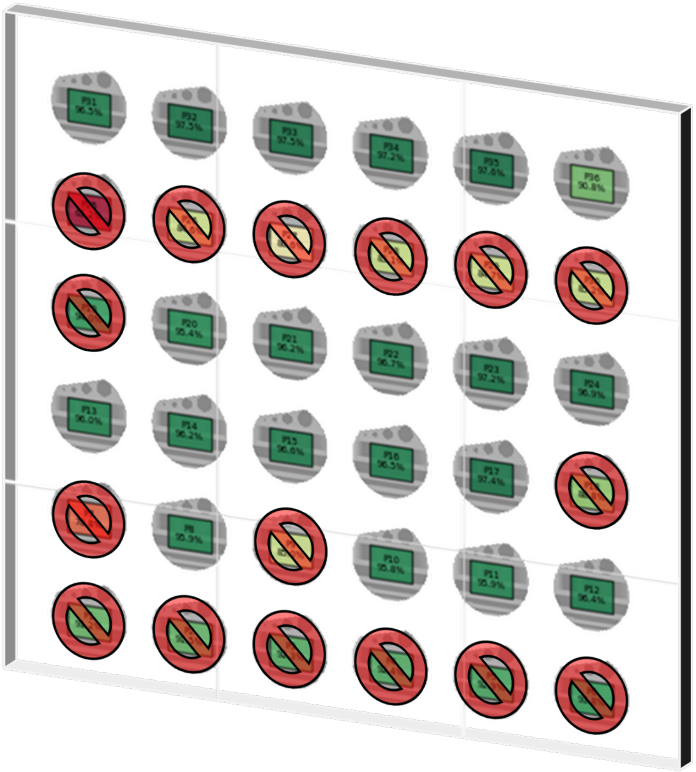
Images taken in normal to build directions

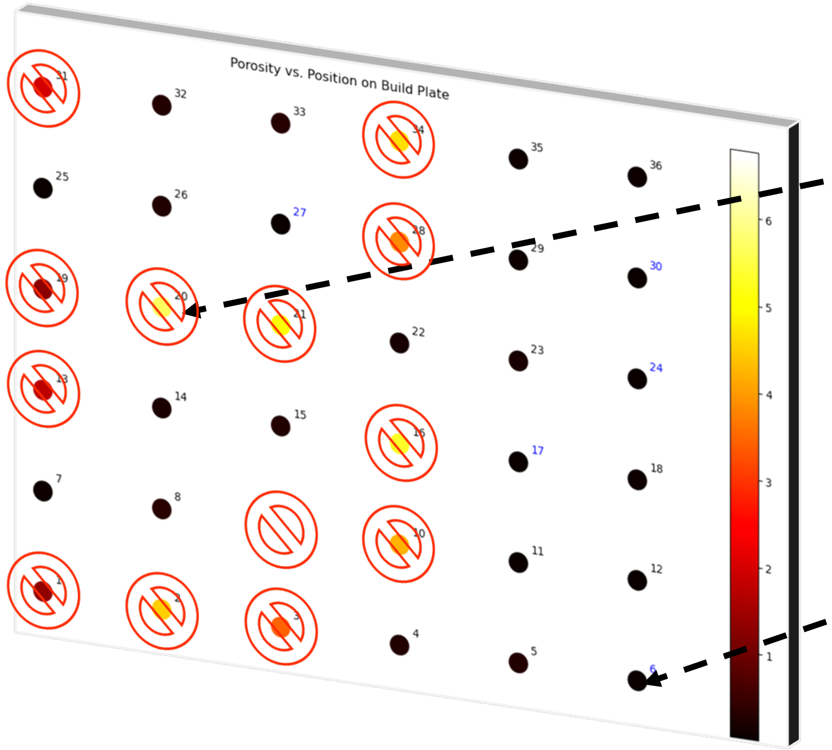
ORNL: Combining Process Monitoring and X-ray Tomography for high-fidelity screening

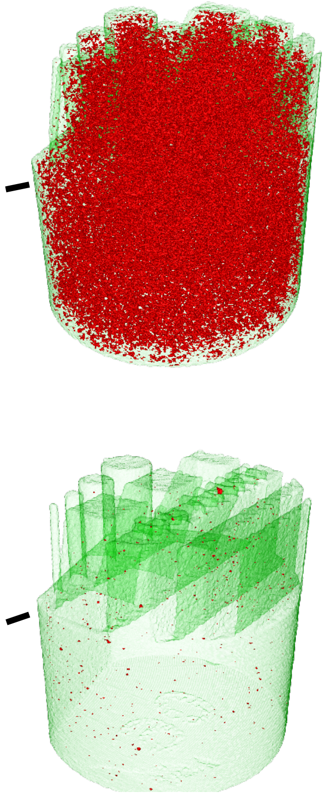
Use in-situ data as a QA tool to assess general part quality (Filter 1)

Use high-throughput XCT data to identify specimens of desired heterogeneity (Filter 2)

Extract specimens to evaluate microstructure variation in high-density parts (Filter 3)

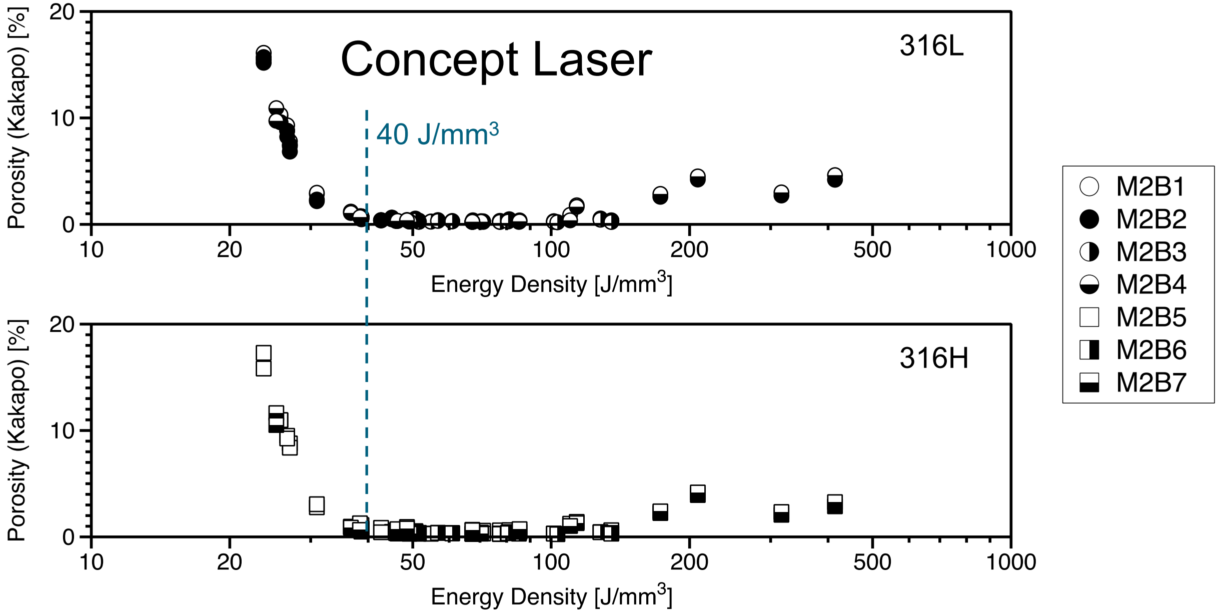


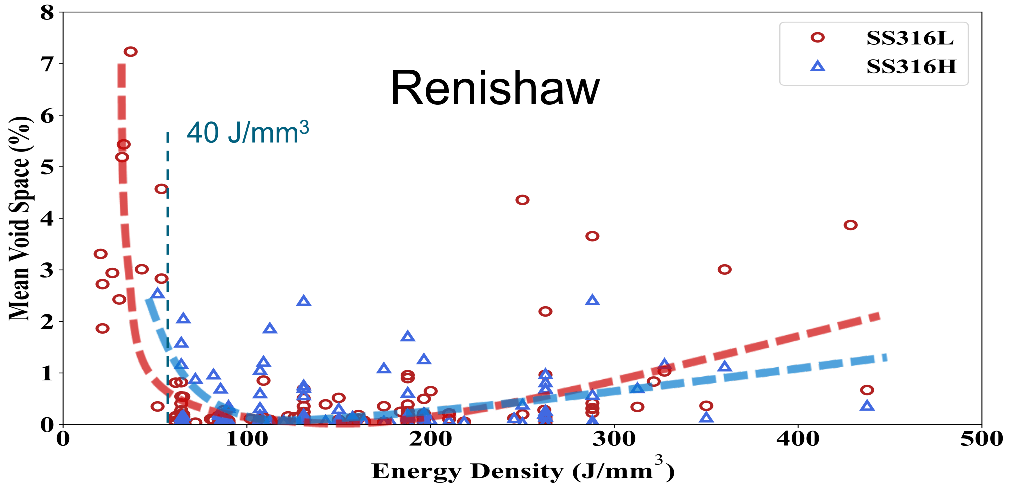




ORNL: High-Throughput Porosity Measurements (XCT + Pycnometry)

Data-Driven Optimization of the Processing Window for 316H Components Fabricated Using Laser Powder Bed Fusion, ORNL/TM-2023/3115, 2023





To Date, 288 printed samples from the Concept Laser and 390 printed samples from the Renishaw have been analyzed using high-throughput XCT.

Both Renishaw and Concept Laser builds have identified energy density windows of minimal porosity.

The existence of potentially different "windows" of optimal parameters between machines is being investigated, using similar agnostic down-selection criteria depending on available data.

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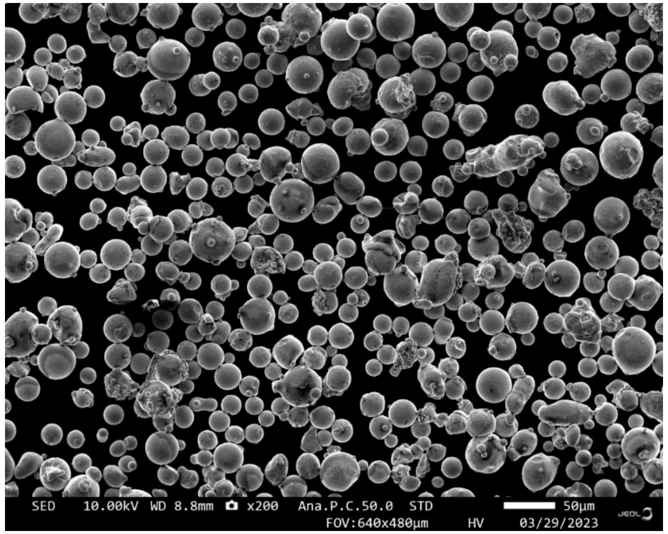
Variability in powder feedstocks

The ASTM specifications for 316H SS and the measured feedstock powder chemistry

		ASTM UNS S31609	316H powder	316H powder	316H powder	316H powder
Manufacturer			Praxair	Praxair	Praxair	PAC
Designation		--	Prax-**AM316H-1**	Prax-**AM316H-2**	Prax-**AM316H-3**	PAC-**AM316H-4**
Order quantity (kg)		- -	50	200	500	200
Composition (wt.%)	Fe	Bal.	Bal.	Bal.	Bal.	Bal.
	Cr	16.0-18.0	17.6	16.8	17.0	16.94
	Ni	11.0-14.0	12.3	12.1	12.3	10.88
	Mo	2.00-3.00	2.6	2.5	2.3	2.23
	Mn	2.0*	1.03	1.13	1.05	1.02
	Si	**1.00***	**0.41**	**0.48**	**0.07**	**0.37**
	C	**0.04-0.10**	**0.05**	**0.06**	**0.08**	**0.043**
	O	**- -**	**0.05**	**0.03**	**0.03**	**0.048**
	N	- -	0.01	0.01	0.01	**0.05**
	P	0.045*	<0.005	<0.005	<0.005	0.031
	S	0.030*	0.00	0.00	0.00	0.001

* Maximum.



SEM image of the powders from the Prax-AM316H-1 batch

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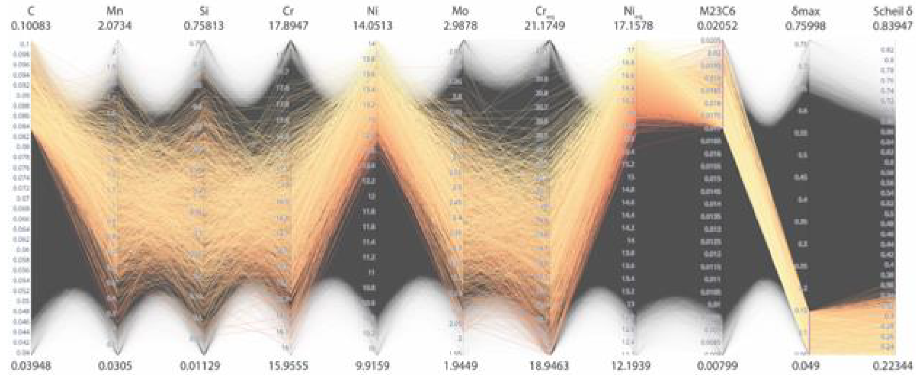
ORNL: informed printing with synthetic data simulations

Preliminary Report on Compositional Specifications for Printed 316SS, ORNL/TM-2023/3031, 2023

1 million synthetic compositions of 316H (within spec) were analyzed to determine what expected variations in carbide and delta-ferrite content would occur based on powder variability



Comparison of the ASTM specifications with the proposed compositions for 316SS-H. Compositions in wt. %

	Fe	C	Mn	Si	Cr	Ni	Mo
ASTM	Bal.	0.04-0.1	2.0	1.00	16-18	11-14	2-3
Proposed	Bal.	0.08-0.1	2.0	0.75	16-18	12-14	2-3

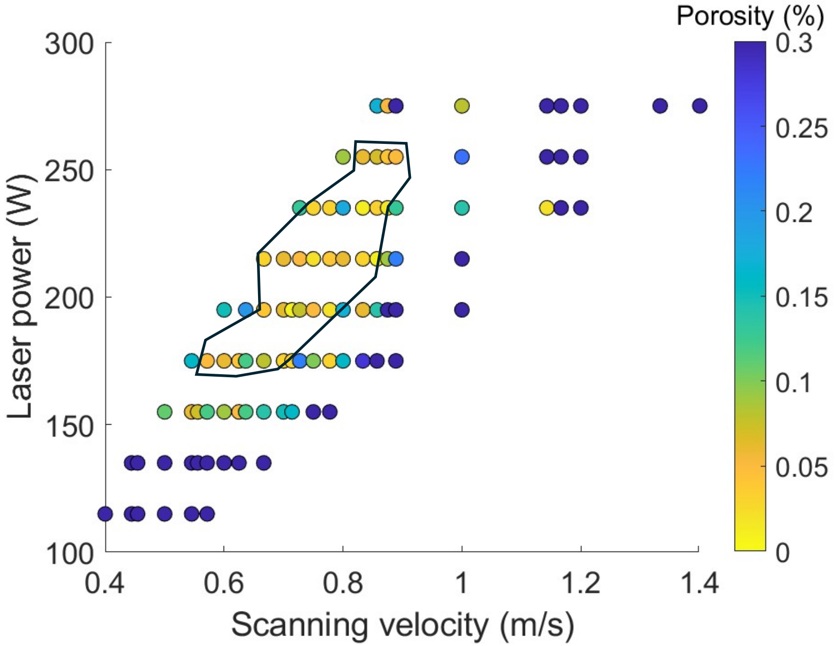
 Approach based on Kannan & Nandwana, Scripta Mat. 2023

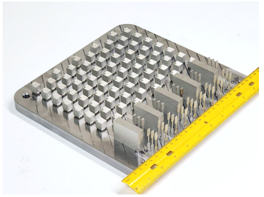
ANL: exploring compositional effect on printability

Unpublished data

Build 20230303

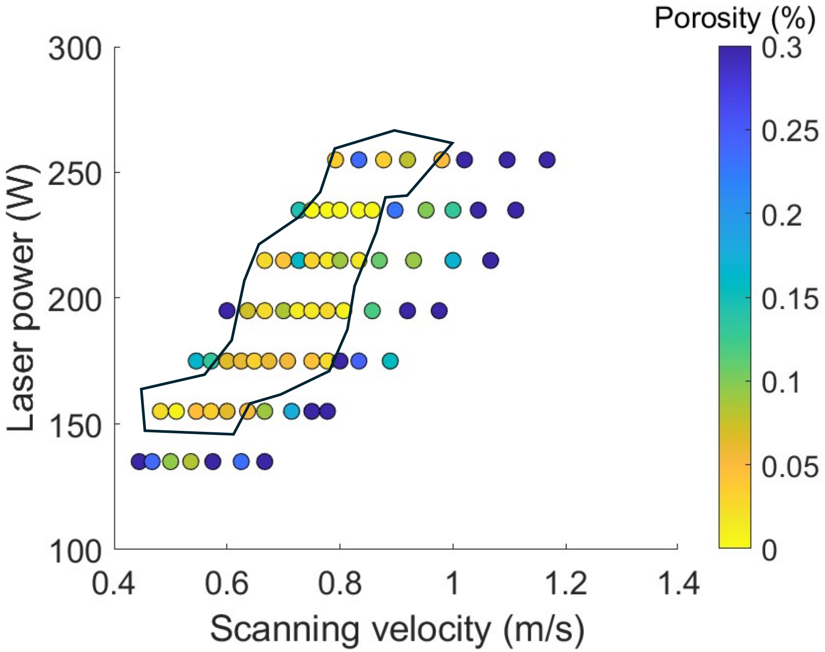
(Powder: Prax-AM316H-2, 0.06% C, 0.48% Si)





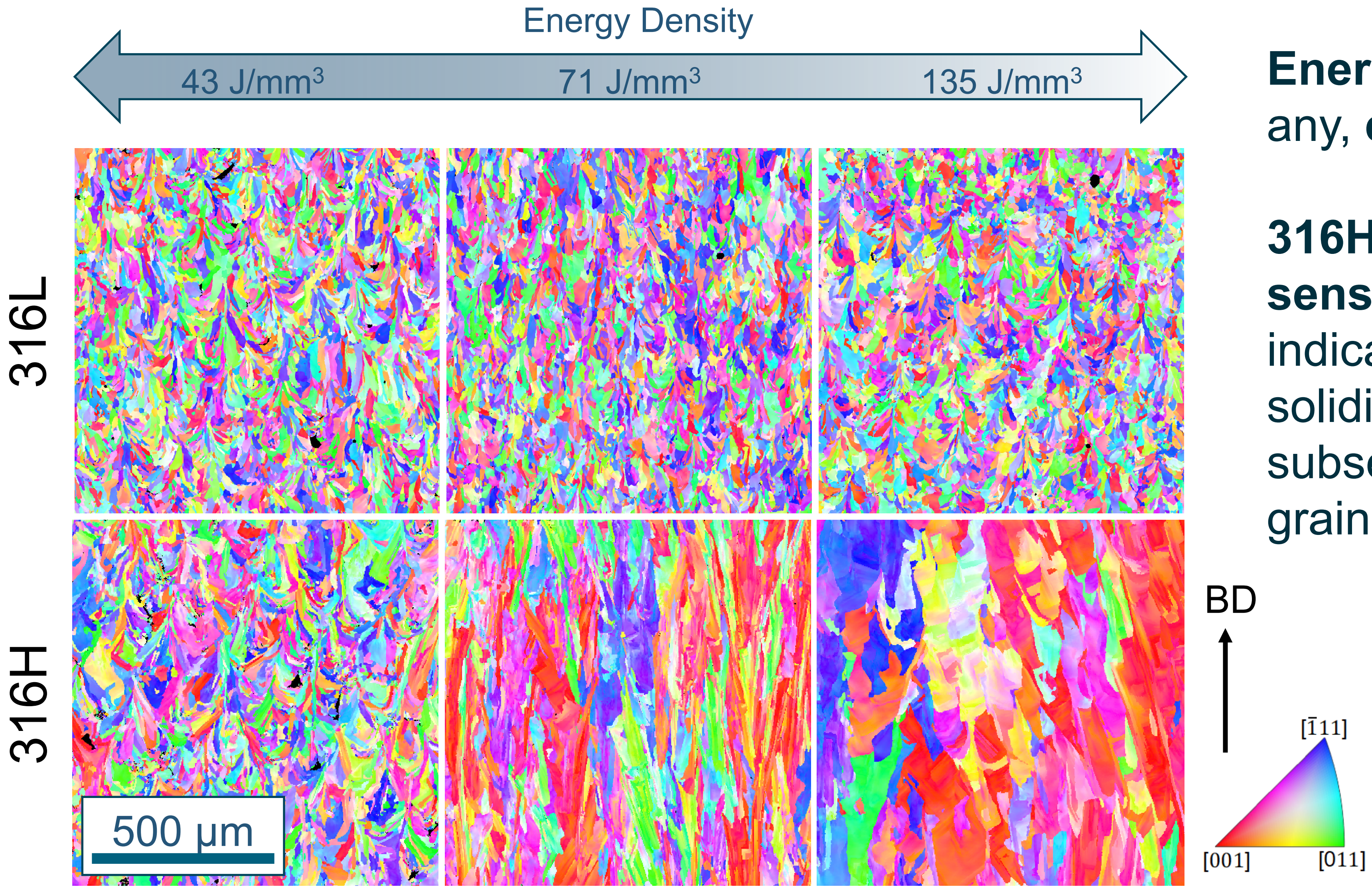
Build 20230811

(Powder: Prax-AM316H-3, 0.08% C, 0.07% Si)



ORNL: 316L vs. 316H exhibit differences in grain structures within the same low-porosity region

Data-Driven Optimization of the Processing Window for 316H Components Fabricated Using Laser Powder Bed Fusion, ORNL/TM-2023/3115, 2023



Energy density has a weak, if any, **effect on SS316L**

316H, on the other hand is **more sensitive to the heat input**, indicating a difference in solidification behavior and subsequent texture evolution and grain morphology.

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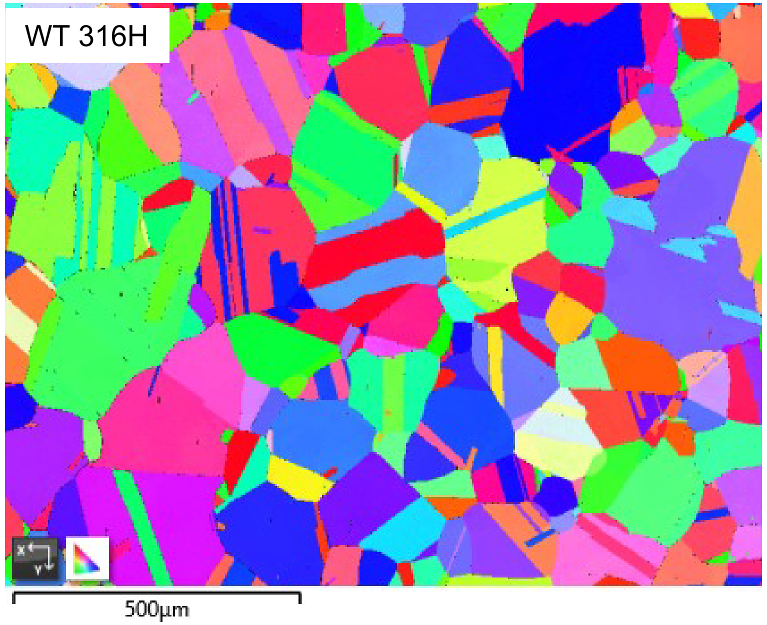
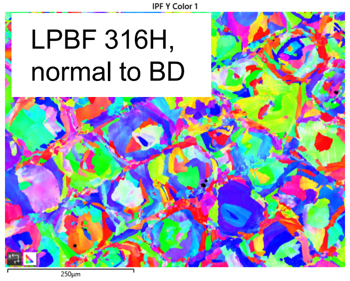
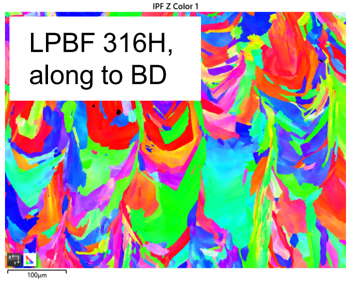
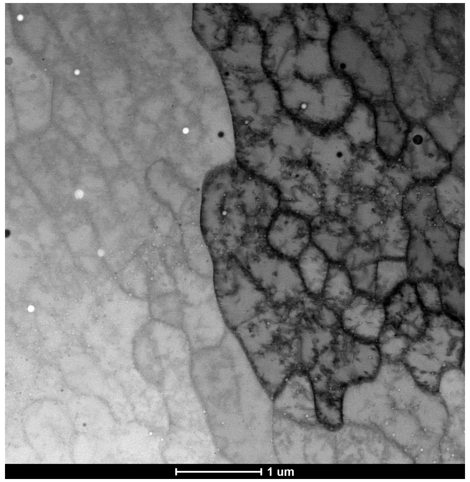
Long term behavior of LPBF 316H under advanced reactor service conditions needs to be addressed

- LPBF 316H has very different microstructure from the wrought.
  - Far from thermal equilibrium
  - Fine grain structure
  - High density dislocations and dislocation networks ➔ internal stresses
  - Chemical heterogeneity
  - Porosity
- Thermal aging is an important factor in assessing materials in-service performance.
- Experimental data is also needed for developing physics-based mechanistic models to predict long-term creep behavior.





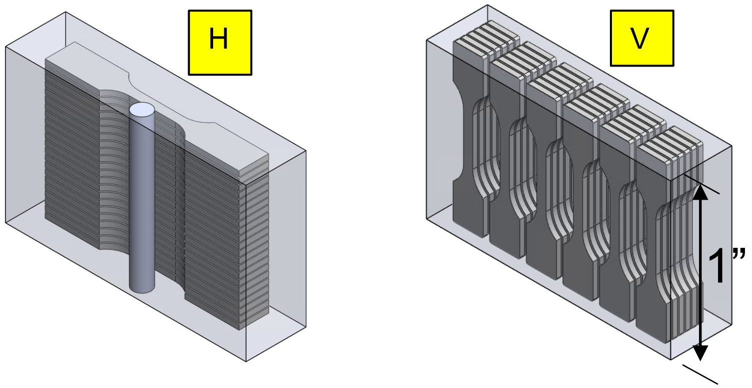




ANL: Thermal aging experiment

Development of process parameters and post-build conditions for qualification of LPBF 316 SS, ANL-AMMT-004, 2023

- **LPBF 316H aging experiment is on going.**



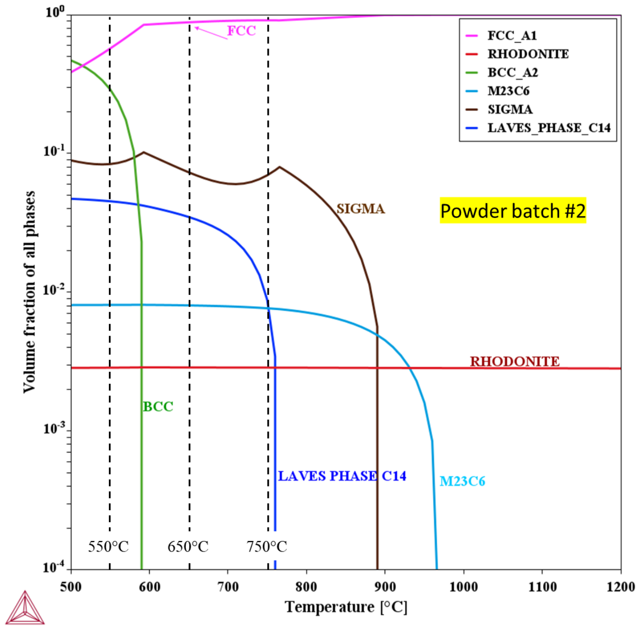
- **Three temperatures are picked to expedite kinetics (trade temperature for time)**

T (°C)	t0	t1 (h)	t2 (h)	t3 (h)	t4 (h)	t5 (h)	t6 (h)
550	AR	5	25	100	500	2500	10000
650	AR	5	25	100	500	2500	10000
750	AR	5	25	100	500	2500	

Black conditions are done.
Red conditions are on-going.

- **Encapsulated specimens are placed in furnaces.**

- **ThermoCalc simulation predicts equilibrium phases.**

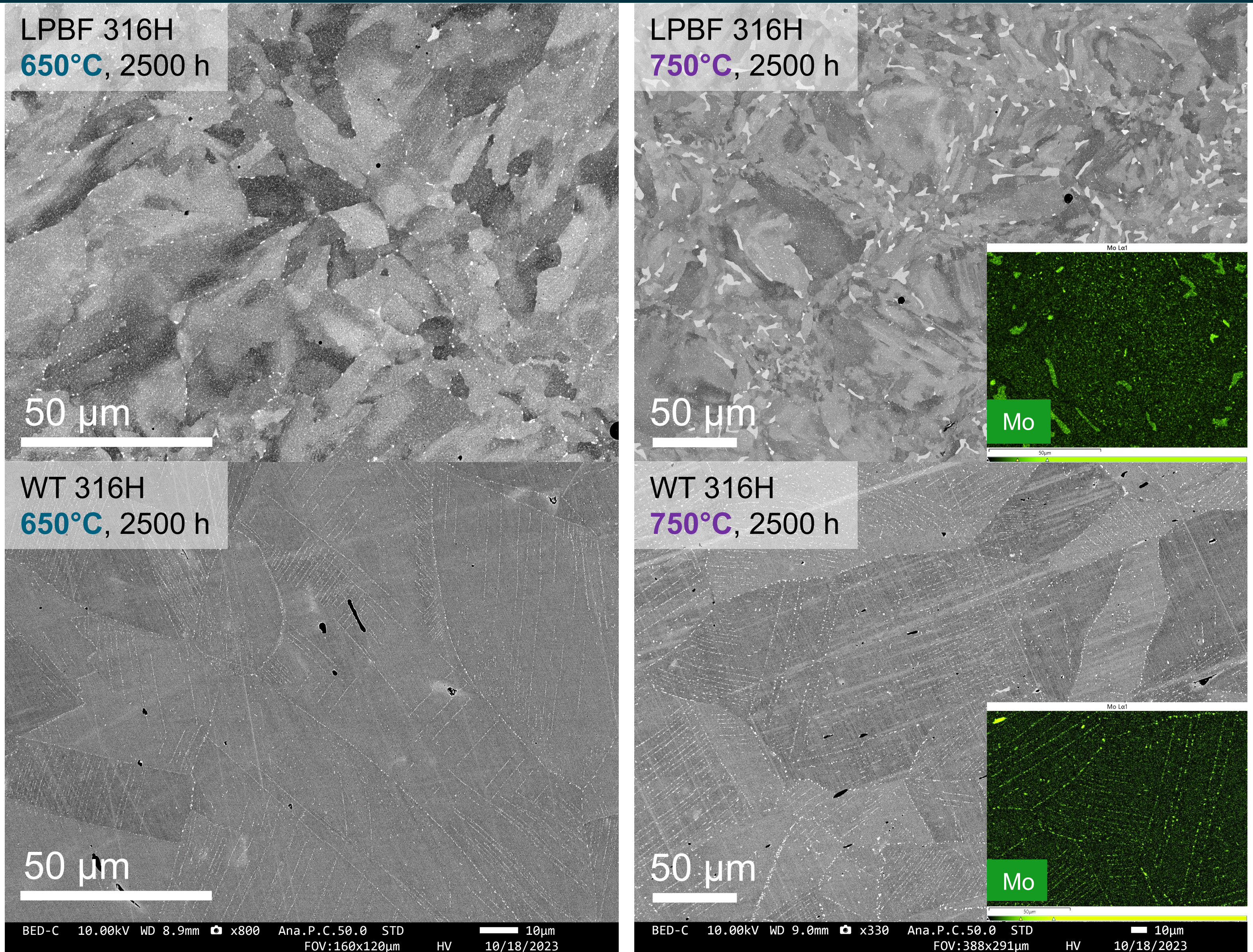


Distinct precipitation behavior compared to wrought material

Compared to WT 316H SS, LPBF 316H SS has:

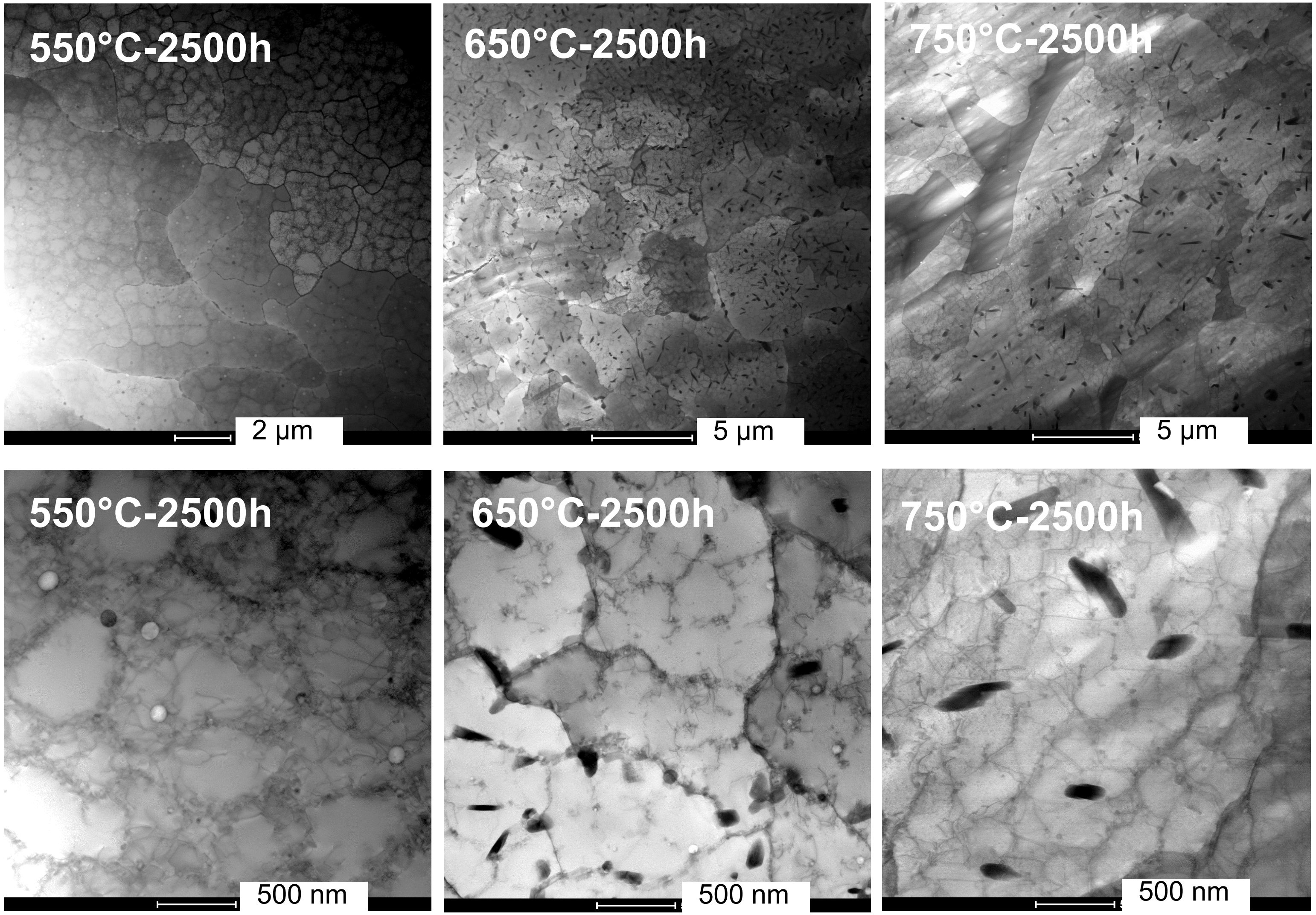
- Smaller grain size and higher dislocation density → fast diffusion channels for heterogenous precipitation
- Chemical segregation at grain boundaries and dislocation cell walls → low nucleation barriers

The result is very different precipitation behaviors.



Complicated precipitation in LPBF 316H SS

Development of process parameters and post-build conditions for qualification of LPBF 316 SS, ANL-AMMT-004, 2023

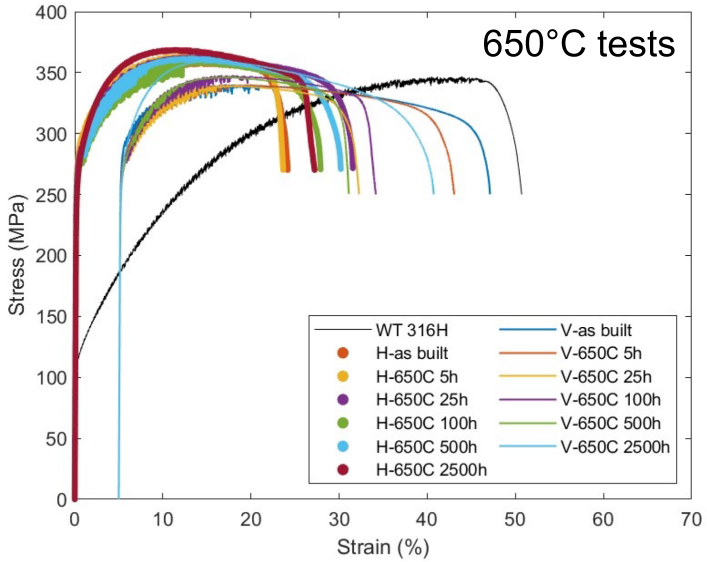


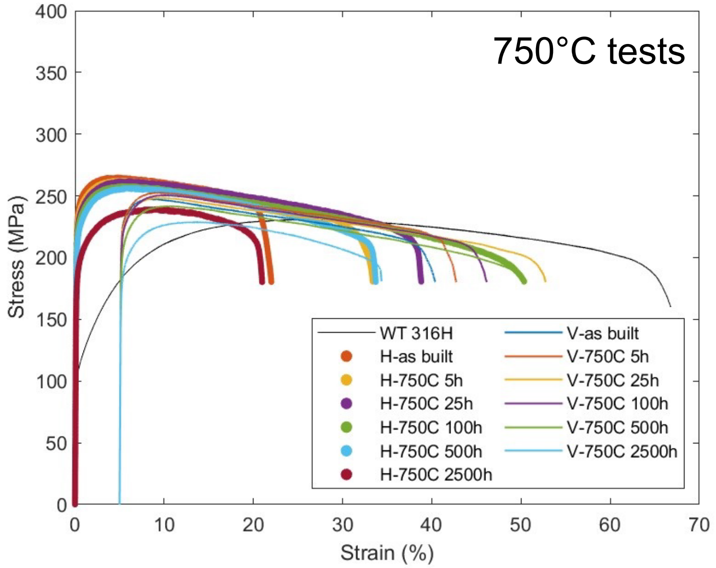
Material condition	Secondary phase description	Location	Shape	Size
As built	Dislocation cells: thick dislocation tangle walls, Cr and Mo segregation	Entire material	Cellular	Average 531 nm [14]
	$MnSiO_3$ Rhodonite	Cell walls	Round	Average 110 nm
550°C-2500h	Dislocation cells: thick dislocation tangle walls	Entire material	Cellular	Average 530 nm
	$MnSiO_3$ Rhodonite	Cell walls	Round	Average 110 nm
	Cr-C-O enriched	Cell walls and junctions	Cuboidal	5-20 nm
	Mo-Si enriched	On cell walls and junctions	Elongated	50 nm
650°C-2500h	Dislocation cells: dislocation tangle walls	Entire material	Cellular	Average 310 nm
	$MnSiO_3$ Rhodonite	Cell walls	Round	Average 50 nm
	Cr-C-O enriched	Grain boundaries, cell walls and junctions	Cuboidal	Average 20 nm
	Mo-Si enriched	Cell walls and junctions	Elongated	Hundreds of nm to 1 μm in length
750°C-2500h	Dislocation cells: thin walls	Entire material	Cellular	Average 320 nm
	$MnSiO_3$ Rhodonite	Cell walls	Round	Average 55 nm
	Cr-C-O enriched	Cell walls and junctions	Cuboidal	Average 30 nm
	Mo-Si enriched	Cell walls and junctions	Elongated	Hundreds of nm to 1 μm in length
	Mo-Cr-O enriched	Grain boundaries	Irregular	5-20 μm

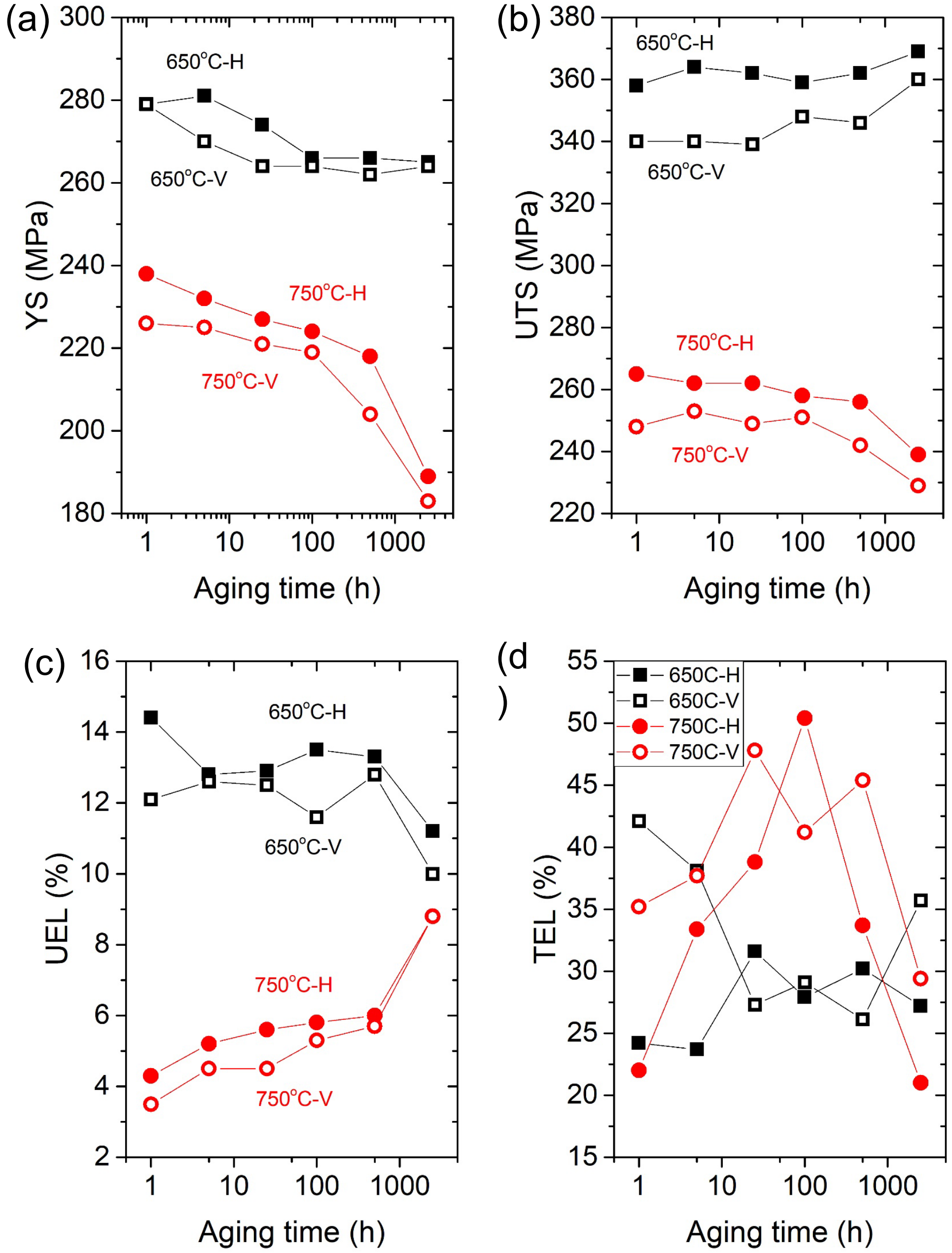
Baseline tensile property evaluation

Development of process parameters and post-build conditions for qualification of LPBF 316 SS, ANL-AMMT-004, 2023

- Compared to WT 316H SS, the LPBF material exhibited much higher yield stresses (YS) and comparable or higher ultimate tensile stress (UTS), but much lower uniform elongation (UEL). The total elongation (TEL) of the LPBF material has a relatively large scatter.
- During the 650° C aging, the YS decreases, the UTS increases and the UEL decreases with aging time. Specimens exhibit dynamic strain aging except the two 2500-h aged specimens.
- During the 750° C aging, the YS decreases, the UTS decreases and the UEL increases with aging time.







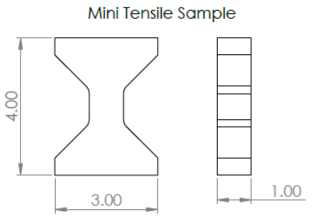
Content

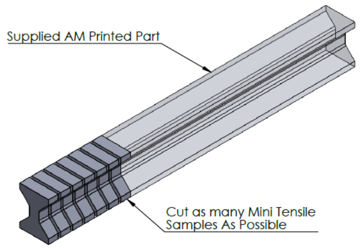
- Background
- Process parameter study
- Variations in powder feedstock
- Thermal aging effect
- **Mechanical testing**

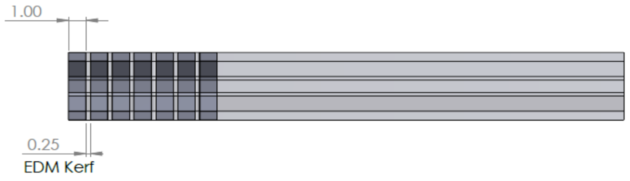
LANL: tension tests with miniature specimens addressing data scatter

Unpublished data

Micro-Tension Geometry

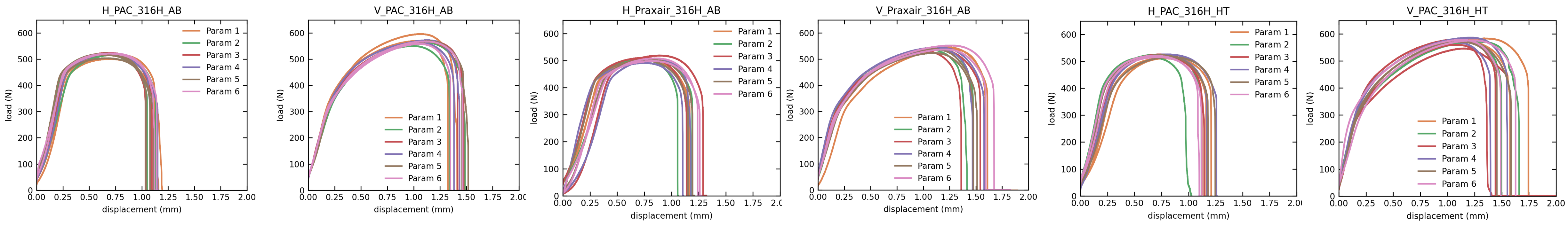




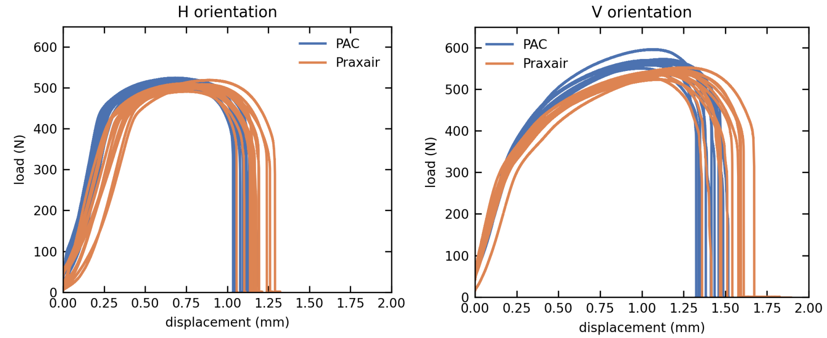


To be compared to full size tensiles in FY24

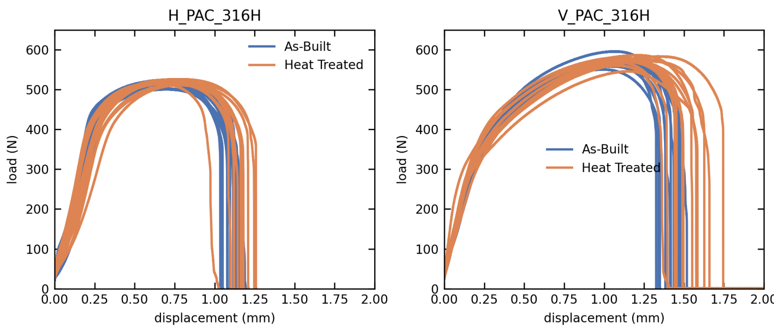
Build Parameters Results (AB and HT)

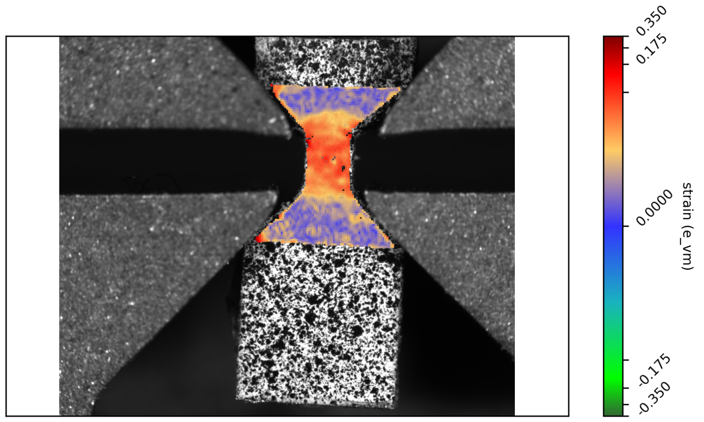


Powder Results (PAC)



Stress-relief Results (PAC)





ORNL: tension tests with miniature specimens addressing anisotropy

Data-Driven Optimization of the Processing Window for 316H Components Fabricated Using Laser Powder Bed Fusion, ORNL/TM-2023/3115, 2023

Micro-Tension Geometry:

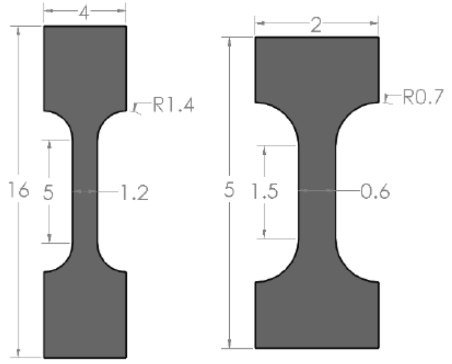
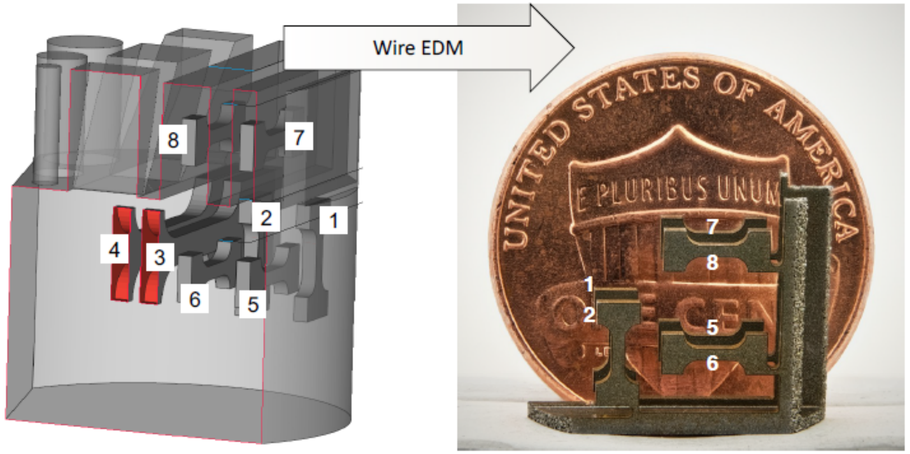
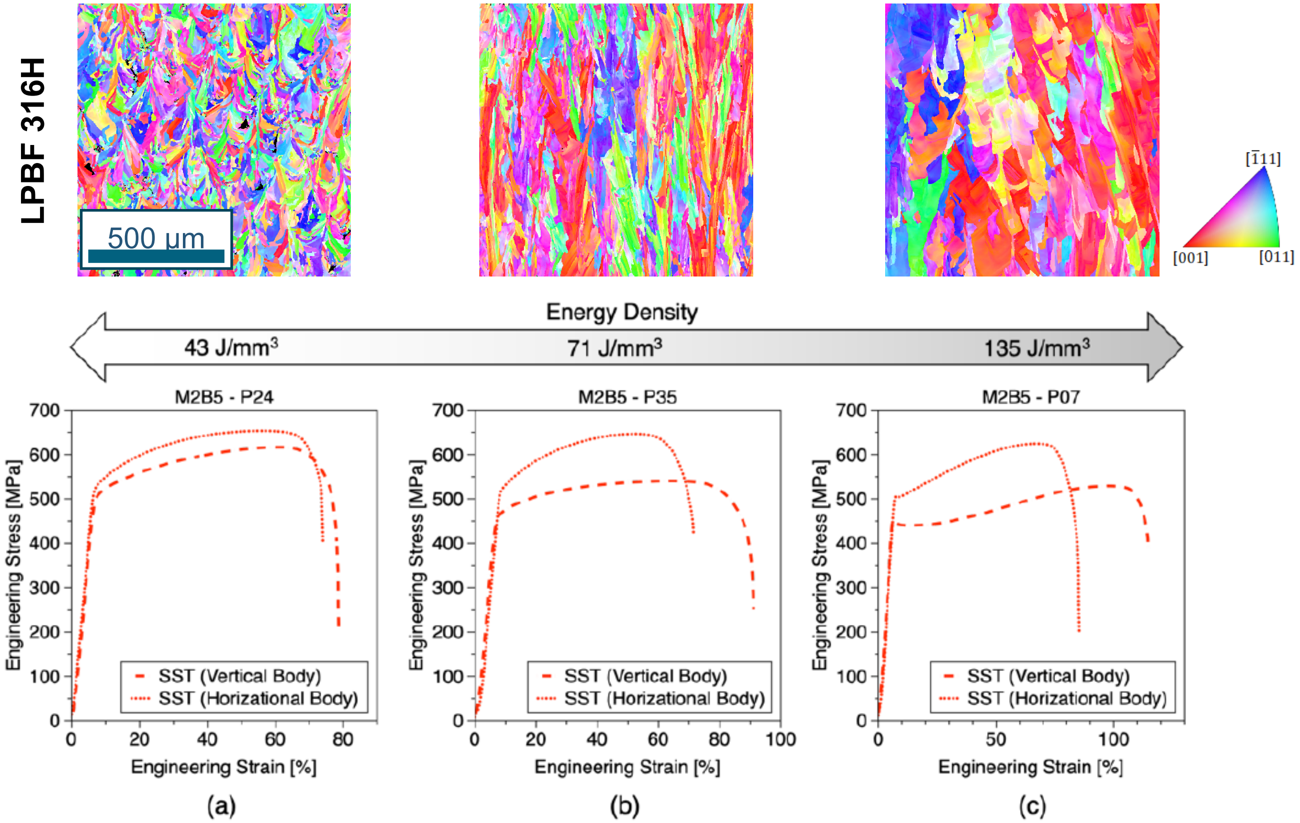


Figure 11. SSJ3 (left) and SST (right) nominal geometries; both are flat dog bone–type specimens. The nominal sample thicknesses are 0.75 mm and 0.6 mm, respectively.

Tensile specimen harvesting strategy for SSJ3 and SST specimens:



(a) (b)



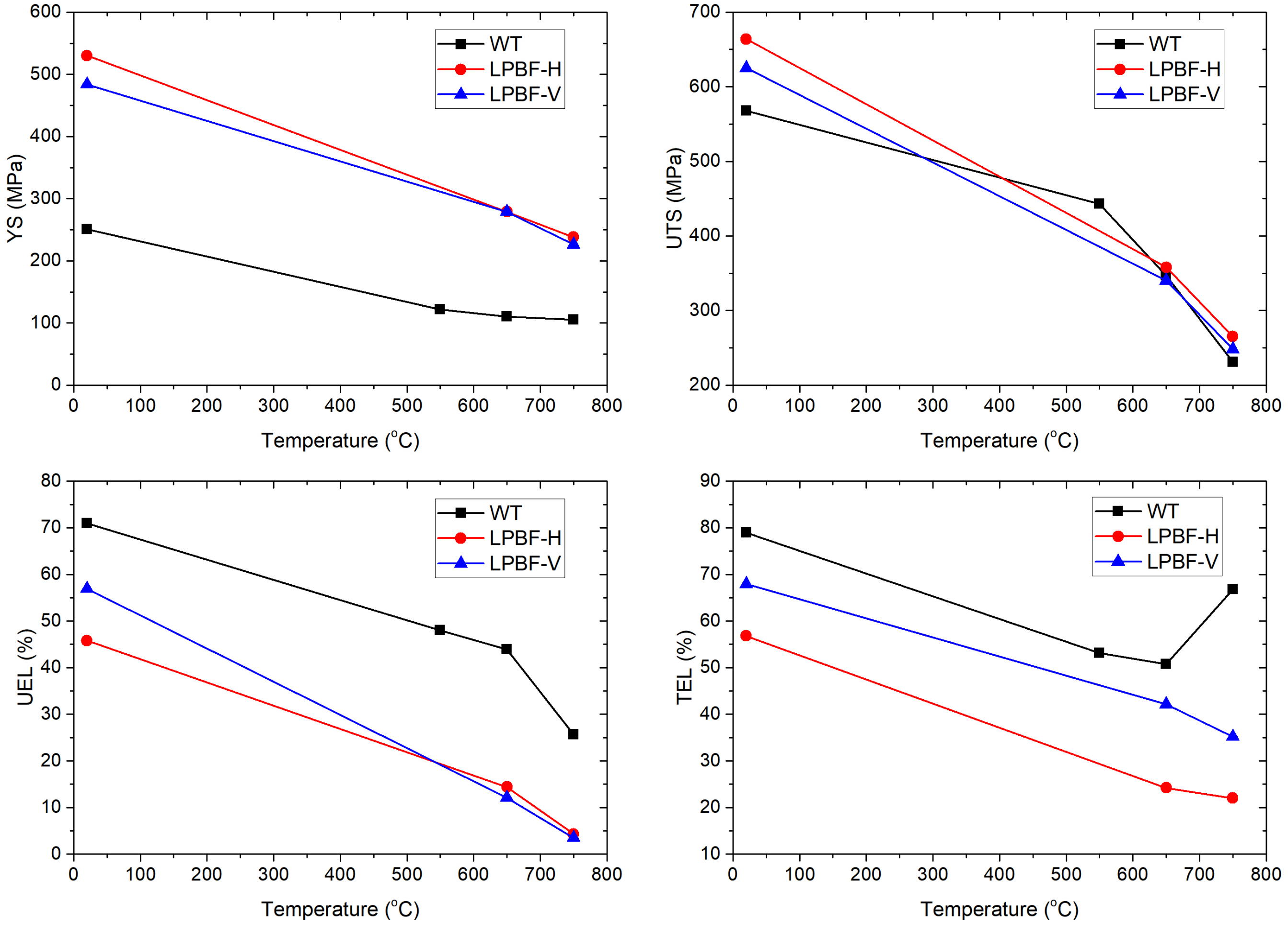
- This comparative analysis reveals the degree of anisotropy in each microstructure at RT.
- The larger the microstructural anisotropy, the larger the differences in yield stresses and work hardening rates at RT.

ANL: Baseline tensile property evaluation at elevated temperatures

Development of process parameters and post-build conditions for qualification of LPBF 316 SS, ANL-AMMT-004, 2023

Observations:

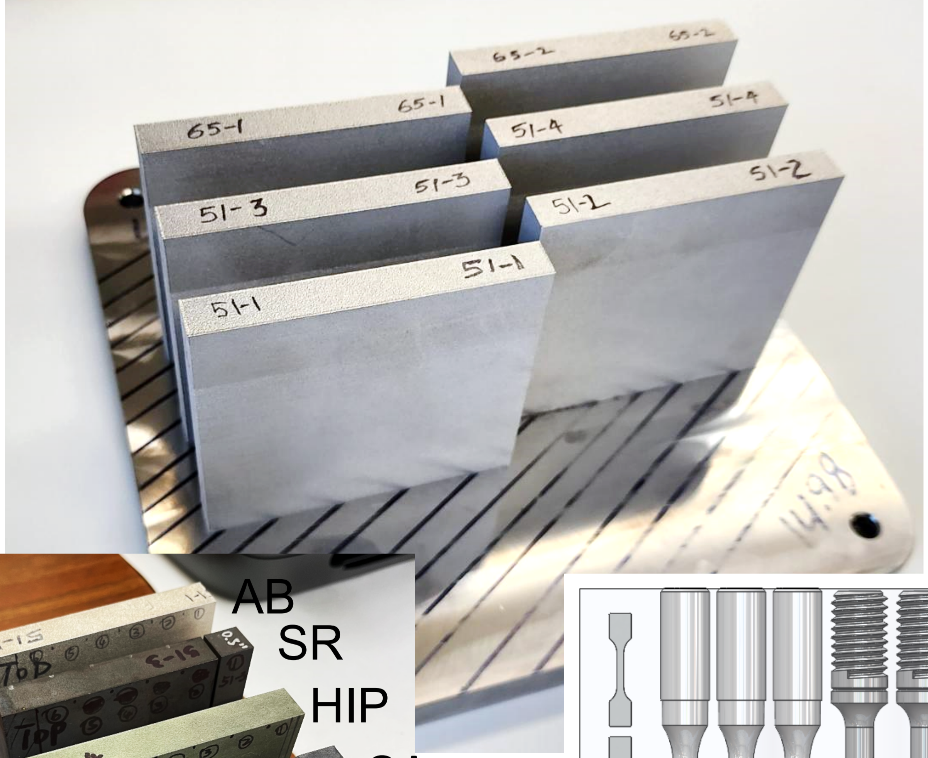
- At all temperatures, the as-built LPBF material has higher yield stress (YS), comparable ultimate tensile stress (UTS), lower uniform elongation (UEL) and lower total elongation (TEL) compared to the WT material.
- All materials show decreasing YS, UTS, UEL and TEL as the test temperature increases, except for the WT material that has an increase in TEL from 650°C to 750°C.

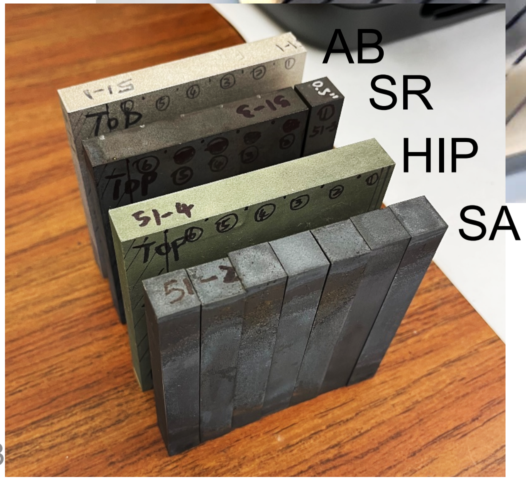


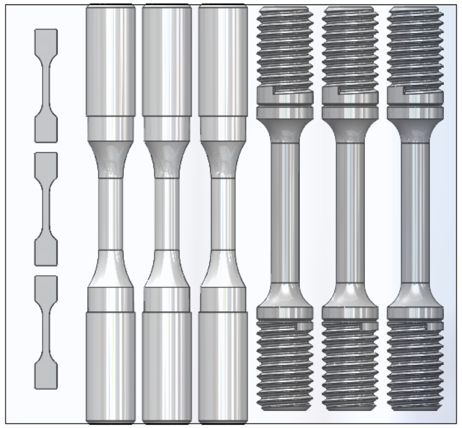
ANL: Larger builds for heat-treatment down-selection

Development of process parameters and post-build conditions for qualification of LPBF 316 SS, ANL-AMMT-004, 2023

- Build 20230410 was completed with 0.06%-C powder.





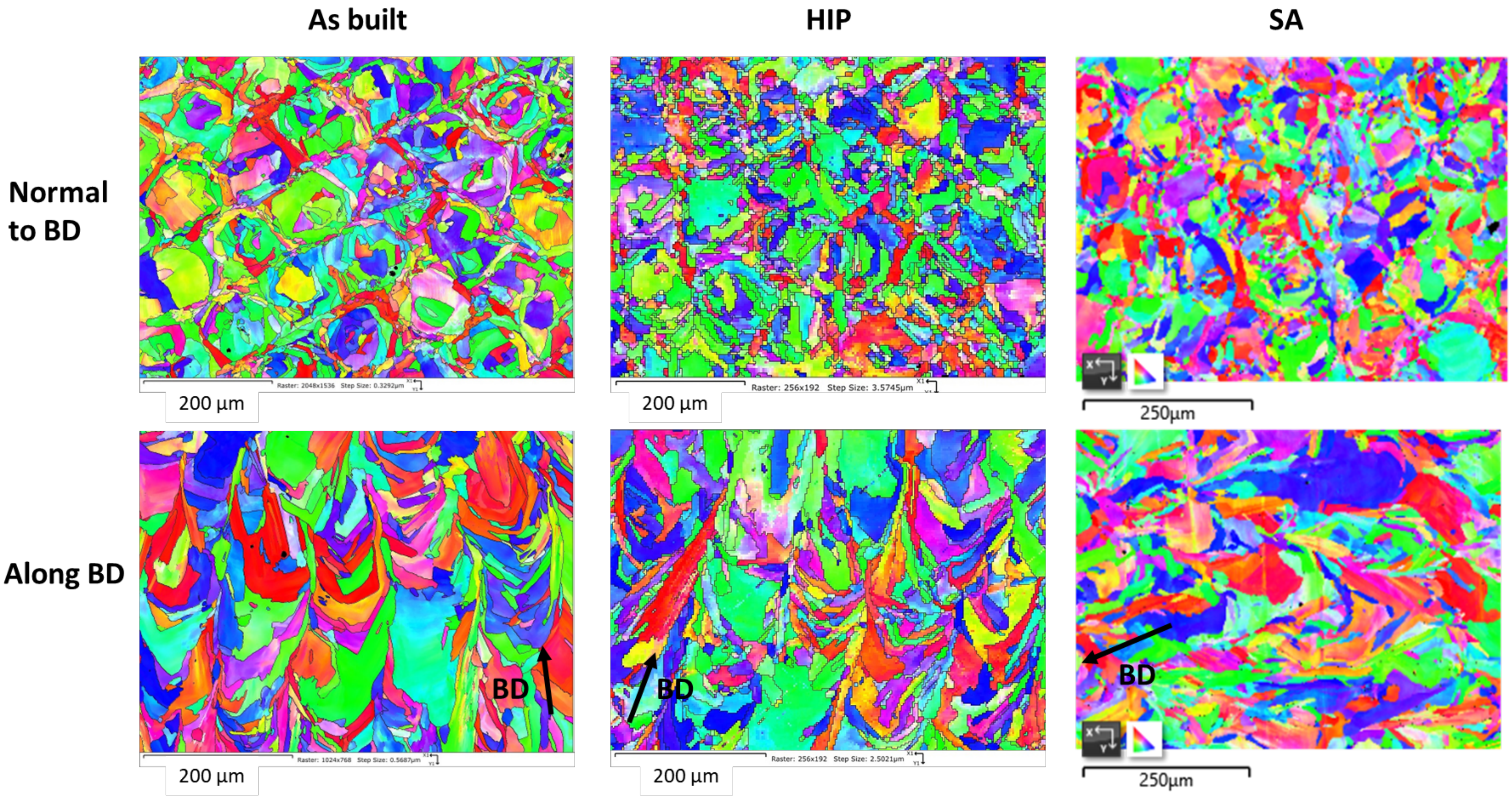


Heat treatment	Temperature	Time	Quench	Pressure
As-built	n/a	n/a	n/a	n/a
Stress relieved	650° C	24 hour	Furnace	n/a
Solution annealed	1100° C	1 hour	Water	n/a
HIP	1120° to 1163° C	4 hours	Cool in inert atmosphere	>100 MPa

Test type	Temp.	Other test conditions	Sample type	ASTM standard	Repeats	Total tests
Tension	20° C		Subsized	None	2	8
Tension	600° C		Subsized	None	2	8
Creep	600° C	248 MPa	Standard	E139	1	4
Creep	600° C	248 MPa	Subsized	None	2	8
Fatigue	550° C	0.3% strain range, R = -1	Standard	E606	1	4
Fatigue	550° C	0.5% strain range, R = -1	Standard	E606	1	4
Creep-fatigue	550° C	0.5% strain range, R = -1 6 min tensile hold	Standard	E2714	1	4
					Total	40

Post-treatment grain structures

Development of process parameters and post-build conditions for qualification of LPBF 316 SS, ANL-AMMT-004, 2023



High-temperature fatigue, creep and creep-fatigue tests are initiated at ANL and INL in FY24.

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Summary

- Qualification of LPBF 316H SS will require comprehensive phenomena identification/ranking, enhanced QA tools, extensive testing and close collaboration between experimental and modeling teams.
- ORNL, LANL, and ANL teams are leveraging resources to provide a process understanding of LPBF 316H SS and provide recommendations on materials optimization.
- Initial builds have been completed on three different laser powder bed systems using three different 316H powder compositions.
- Microstructural variation (porosity and grain structure) has been captured as a function of process variables, enabling future studies on post-build anisotropy.
- Initial thermal ageing has identified unique second phase particles requiring additional consideration on compositional specifications and long-term performance.
- Larger-scale builds are underway to provide performance metrics (tensile, creep, fatigue, creep-fatigue) as a function of microstructure and post-process heat treatment.

References

- Zhang, X., *et. al.*, **Development of process parameters and post-build conditions for qualification of LPBF 316 SS,** ANL-AMMT-004, 2023
- Massey, C., *et. al.*, **Data-Driven Optimization of the Processing Window for 316H Components Fabricated Using Laser Powder Bed Fusion,** ORNL/TM-2023/3115, 2023
- Montoya, R., *et. al.*, **Process understanding for qualifying LPBF 316H SS,** LA-UR-23-30967, 2023
- Nandwana, P., *et. al.*, **Preliminary Report on Compositional Specifications for Printed 316SS,** ORNL/TM-2023/3031, 2023

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