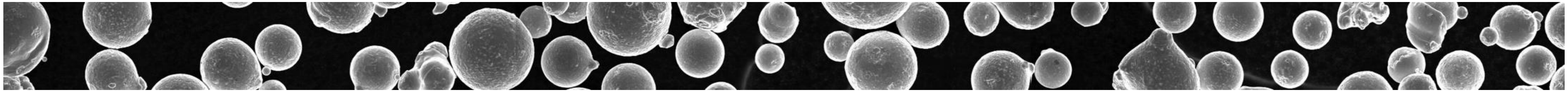
9th International Symposium on Superalloy 718 and Derivatives

Impact of Powder Variability on the Microstructure and Mechanical Behavior of Selective Laser Melted (SLM) Alloy 718



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GRC
GLENN RESEARCH CENTER
at Lewis Field

June 6, 2018

MARSHALL SPACE FLIGHT CENTER

Acknowledgements

NASA HEOMD / Space Launch System Liquid Engine Office / Additive Manufacturing Structural Integrity Initiative Project (FY16-FY18)

Powder Task:

NASA MSFC

- Kristin Morgan
- William Tilson
- Richard Boothe
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- AM Fabrication Facility
- Heat Treatment Facility

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Space Launch System – Heavy Lift Launch Vehicle – Requires four RS-25 engines to lift core stage

RS-25 Affordability Initiative

33% Reduction in Cost

> 700 Welds Eliminated
> 700 Parts Eliminated

35 AM Opportunities

718 Powder Feedstock Variability Study

- *Powders evaluated – 18 powders from 8 suppliers (A-H)*
  - ICP / LECO bulk powder chemistry measurements
  - Count basis particle size distributions (optical silhouettes)
  - Visual comparison of powders
- *Processing and Testing Details*
- *Properties evaluated*
  - Build quality and microstructure
  - Tensile behavior
  - High Cycle Fatigue (HCF) results
    - Crack initiation and failure mechanisms
- *Summary and Concluding Remarks*

Motivation

- Standardization is needed for consistent evaluation of AM processes and parts in critical applications.
- Data on powder feedstock variability in open literature are limited & inadequate
- Supported **MSFC technical standard** for <u>SLM 718 hardware</u> by examining feedstock relationships to processing, homogeneity, durability & performance

POC: Doug Wells

MEASUREMENT SYSTEM IDENTIFICATION METRIC/SI (ENGLISH) UNITS

National Aeronautics and Space Administration

MSFC-STD-3716
BASELINE
EFFECTIVE DATE: October 18, 2017

George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama 35812

EM20

MSFC TECHNICAL STANDARD

STANDARD FOR ADDITIVELY MANUFACTURED SPACEFLIGHT HARDWARE BY LASER POWDER BED FUSION IN METALS

Approved for Public Release; Distribution is Unlimited

CHECK THE MASTER LIST — VERIFY THAT THIS IS THE CORRECT VERSION BEFORE USE

Objectives

- Obtain comprehensive industry **<u>supplier-to-supplier comparison</u>** to understand and identify the feedstock controls important to SLM Alloy 718
- 5 unique powder lots *(B1, C1, G2, G3, H1)* have been down-selected for a larger-scale (300 lbs each) investigation underway to include reuse / recyclability study and more expansive mechanical testing

Approach: Procure as many off-the-shelf Alloy 718 powders as possible for a comprehensive supplier-to-supplier comparison

- Compare powder characteristics
- Screen mechanical behavior

- Lot-to-lot variability
- N_2-atomized: 3 of 16
- 4 cuts same G supplier (separate out size effects)

- (*) 2nd builds allowed once reuse comparisons (SEE PAPER)

Unable to build G1, poor G4 builds

	GRC ID	Alloy 718 Powders	Powder Cut (µm)	Process	Gas
Reseller Vendor A	A1	Supplier 1, Powder 1	15-45	GA	Ar
	A2	Supplier 1, Powder 2	10-45	GA	Ar
	A3	Supplier 1, Powder 3	10-45	GA	Ar
Direct Suppliers	B1	Supplier 2, Powder 1	15-45	Rotary	Ar
	C1	Supplier 3, Powder 1	15-45	**GA**	**N**
	D1	Supplier 4, Powder 1	16-45	GA	Ar
*	D2	Supplier 4, Powder 2	11-45	GA	Ar
	E1	Supplier 5, Powder 1	10-45	**GA**	**N**
*	E2	Supplier 5, Powder 2	10-45	**GA**	**N**
	F1	Supplier 6, Powder 1	15-45	GA	Ar
*	F2	Supplier 6, Powder 2	10-45	GA	Ar
	G1	Supplier 7, Powder 1	0-22	GA	Ar
	G2	Supplier 7, Powder 2	11-45	GA	Ar
	G3	Supplier 7, Powder 3	16-45	GA	Ar
	G4	Supplier 7, Powder 4	45-90	GA	Ar
	H1	Supplier 8, Powder 1	10-45	GA	Ar

Standard ~15-45 µm SLM cuts (6 powders)

Standard ~10-45 µm SLM cuts (8 powders)

Undersized / oversized cuts (2 powders)

Approach: Procure as many off-the-shelf Alloy 718 powders as possible for a comprehensive supplier-to-supplier comparison

Majority of powder compositions fall within a narrow range than AMS 5664 specification

Ni-0.35-0.51 Al, 0-0.039 C, 18.1-19.2 Cr, 18.0-19.2 Fe, 2.9-3.1 Mo, 4.8-5.2 Nb, 0.8-1.0 Ti wt.% + trace impurities

Tight Nb range for primary strengthening by γ”-precipitates

Variation in Al, Cr for secondary strengthening by γ’-precipitates

Will discuss impact of N

MC carbides (Nb, Ti)

***E1** did not meet AMS 5664 ranges*

***E2** higher in Al & C but within spec*

***C1** high in Al, low in Cr but within spec*

Reseller Vendor A: A1–A3

Direct Suppliers: B1–H1

GRC ID	Alloy 718 Powders	Powder Cut (µm)	Process	Gas	Al (wt.%)	Cr (wt.%)	C (wt.% ppm)	N (wt.% ppm)
A1	Supplier 1, Powder 1	15-45	GA	Ar	0.395	18.82	350	325
A2	Supplier 1, Powder 2	10-45	GA	Ar	0.505	18.94	240	90
A3	Supplier 1, Powder 3	10-45	GA	Ar	0.380	18.17	280	331
B1	Supplier 2, Powder 1	15-45	Rotary	Ar	0.465	19.00	50	25
C1	Supplier 3, Powder 1	15-45	**GA**	**N**	**0.565**	**17.45**	**390**	**1395**
D1	Supplier 4, Powder 1	16-45	GA	Ar	0.480	19.02	330	122
* D2	Supplier 4, Powder 2	11-45	GA	Ar	0.495	19.11	305	115
E1	Supplier 5, Powder 1	10-45	**GA**	**N**	**0.090**	**17.71**	**960**	**1220**
* E2	Supplier 5, Powder 2	10-45	**GA**	**N**	**0.705**	**19.11**	**470**	**2770**
F1	Supplier 6, Powder 1	15-45	GA	Ar	0.345	18.25	330	607
* F2	Supplier 6, Powder 2	10-45	GA	Ar	0.390	18.37	340	370
G1	Supplier 7, Powder 1	0-22	GA	Ar	0.440	18.82	330	207
G2	Supplier 7, Powder 2	11-45	GA	Ar	0.455	18.77	360	176
G3	Supplier 7, Powder 3	16-45	GA	Ar	0.485	18.77	390	199
G4	Supplier 7, Powder 4	45-90	GA	Ar	0.475	18.77	330	246
H1	Supplier 8, Powder 1	10-45	GA	Ar	0.355	18.52	215	562

In Ar N=25 to 607 ppm

C=50 to 960 ppm

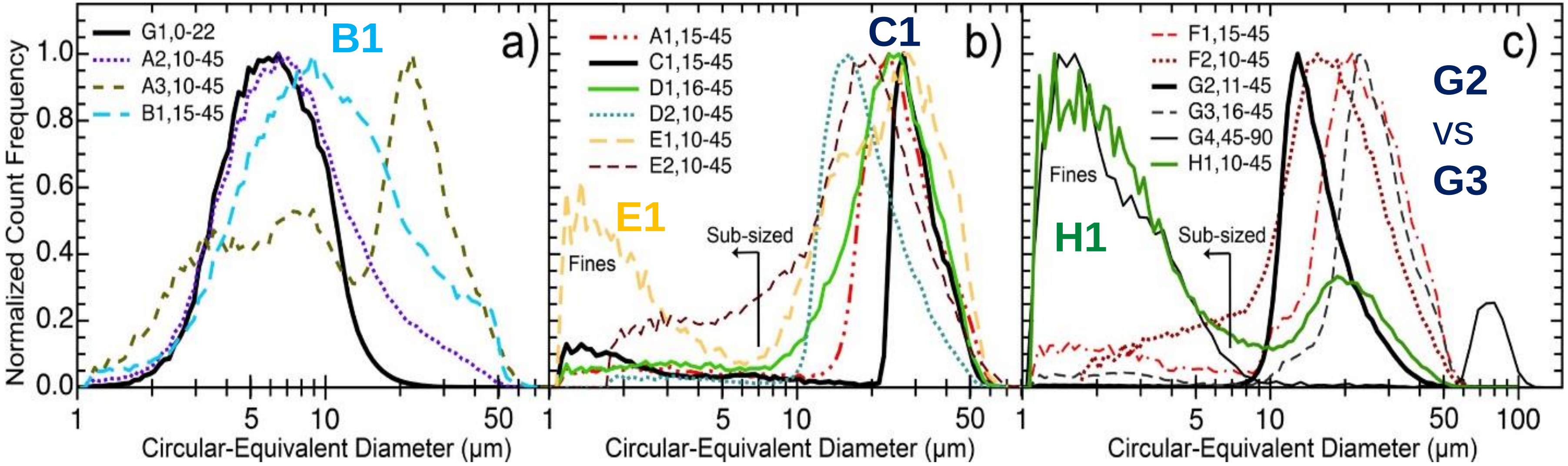
Powders exhibit distinct particle size distributions

There is variation in average diameters, particle size distribution widths and modalities

Malvern Morphologi G3SE
Silhouettes of a minimum 20,000 individual powder particles per scan

Avg. particle diameter for primary peak for most powders is between 23-26 μm for most powders

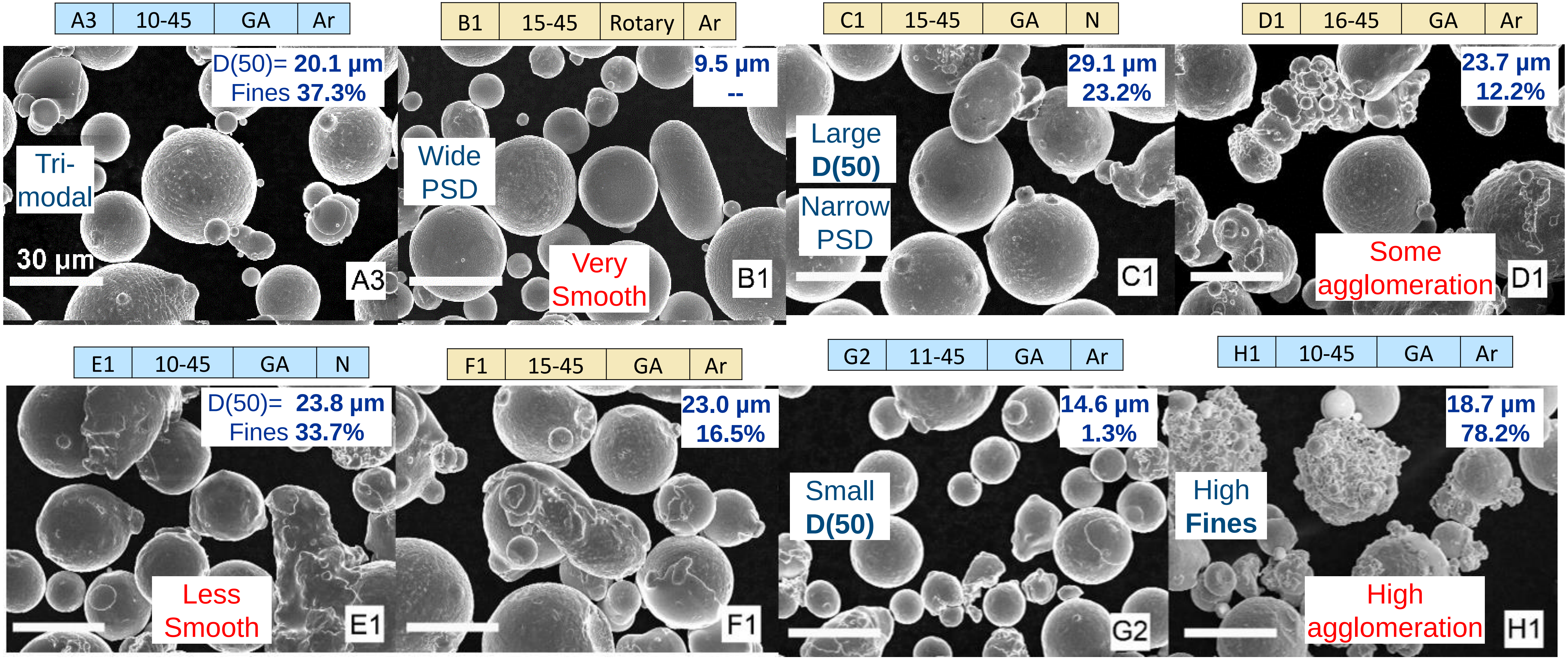
Undersized / Trimodal **Regular-sized powders mixture of unimodal and bimodal**



***Number basis distributions** are more sensitive to fines; Volume basis often reported.*

Some suppliers are more successful at reducing fine content

Particles are all highly regular spheroids from all suppliers; Show distinct differences in roughness, fines, & agglomeration



Powders with higher percentage of fines and agglomeration more prone to unplanned stops

Standard ~15-45 µm SLM cuts, Standard ~10-45 µm SLM cuts

Processing and Testing Details

NASA MSFC Concept Laser M1 machine:

- Customized SLM 718 parameters for MSFC RS-25 projects
- Layer thickness: 30 µm
- Continuous scan strategy plus contours

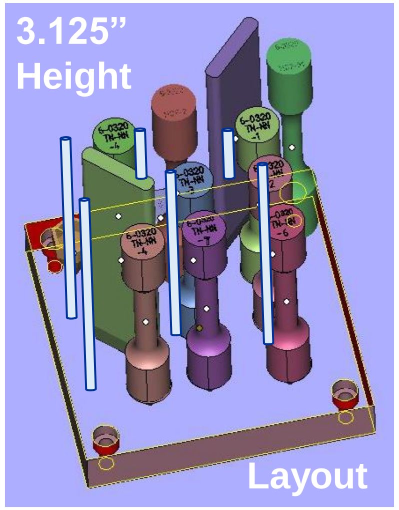
Visible refill lines

Green-state “met” bar

Small box configuration requires start /stop to refill piston with powder

Planned restarts

18 builds over 3 months at NASA MSFC



Taper Ends for Easy Snap Off

- **50 lbs of 718 powder procured from most suppliers**
- **Two microstructure bars**
  - **Green-state bar** → inherent to the process
  - **Fully heat treated (FHT) bar** → post process response
- *MSFC Build* —— *10 – 16 weeks* ——→ *To GRC*

  Stress Relief at MSFC → Hot Isostatic Pressing in batches at Outside Vendor → Solution and Aged to AMS 5664E in batches at MSFC → Machine

Reduce porosity, homogenize and remove as-built texture

Screen room temperature mechanical behavior

As-Fabricated (AF) vs. Low Stress-Ground (LSG) Surface Conditions

- **A tensile test per surface condition**
  - **Strain control up to 2% then stroke control at equivalent strain rate**
- **3 HCF tests per surface condition at 20 Hz and R(σ)= -1**
  - **Targeted 1 million cycle averages, Runouts above 10 million**
  - **Stress amplitudes of 271 MPa (40 ksi) for AF and 464 MPa (67 ksi) for LSG**

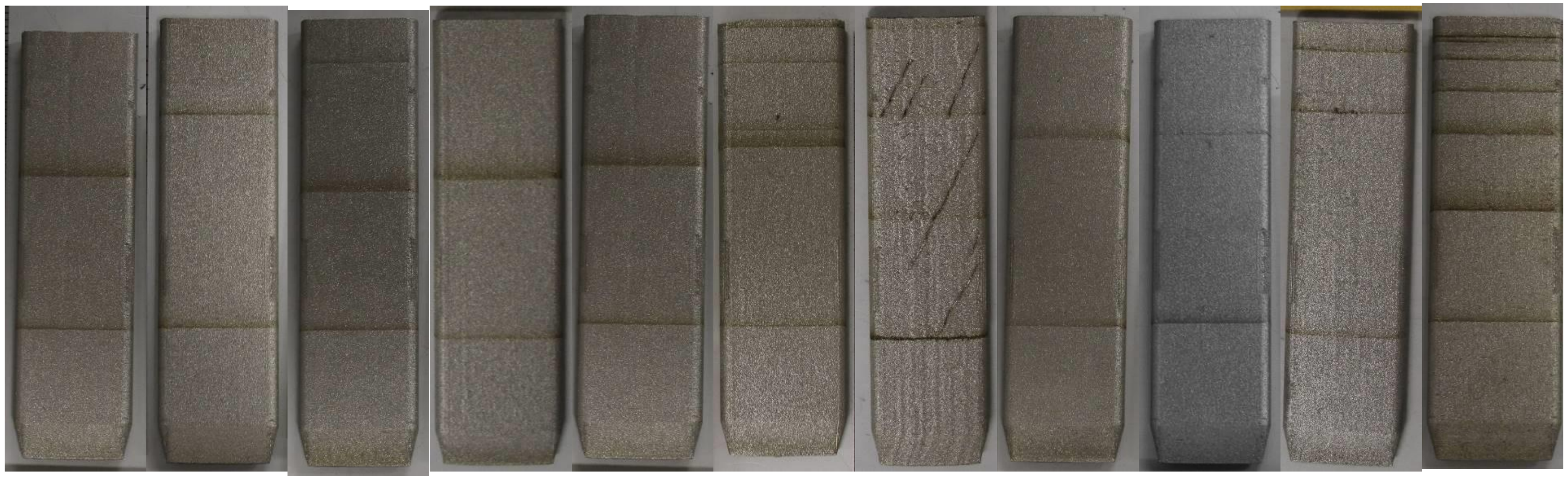
Impact of Feedstock Variability on Build Quality

Green State Met Bars *Threshold image analysis of 5 areas in 1 cm x 1 cm XZ piece from mid-section*

A1 A2 A3 B1 C1 D1 E1 F1 G2 G3 H1

1” wide bars:

Build Direction (Z)

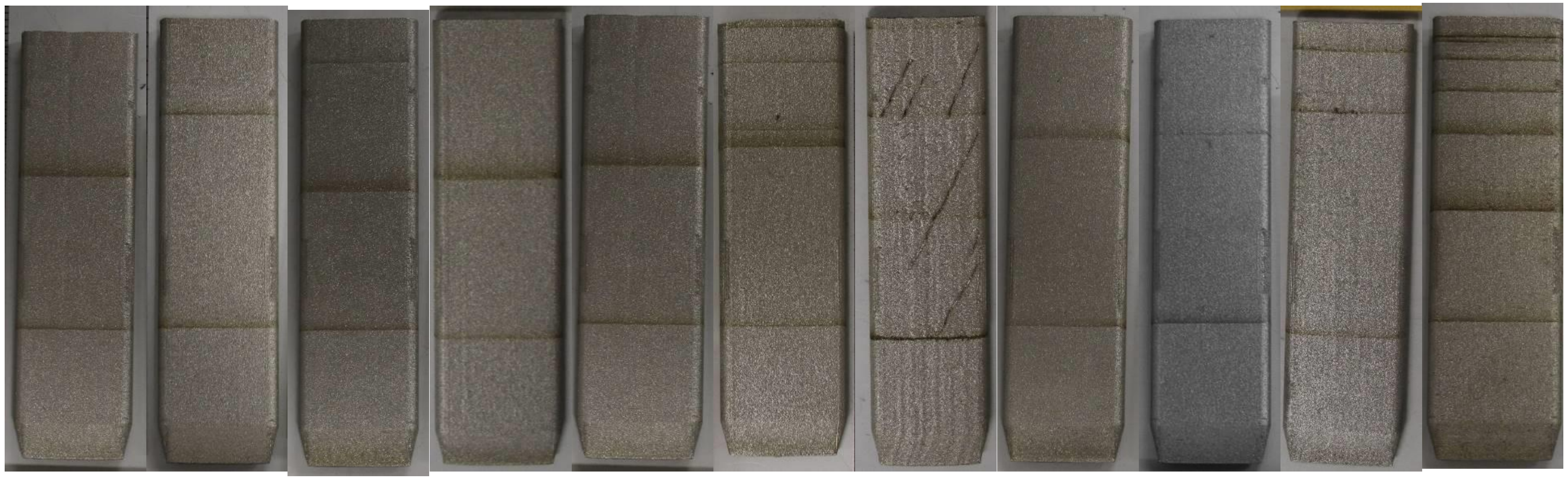


Optimized SLM parameters produces low porosity →

	A1	A2	A3	B1	C1	D1	E1	F1	G2	G3	H1
Green Porosity	0.19 ± 0.09 %	0.69 ± 0.23 %	0.19 ± 0.15 %	0.18 ± 0.09 %	0.14 ± 0.07 %	0.10 ± 0.07 %	0.46 ± 0.32 %	0.15 ± 0.09 %	0.14 ± 0.09 %	0.14 ± 0.07 %	0.19 ± 0.11 %
Green Pore Size	12.2 ± 3.0 µm	22 ± 4 µm	12 ± 3 µm	11.5 ± 2.3 µm	10.9 ± 2.3 µm	9.6 ± 2.6 µm	14.4 ± 3.0 µm	9.5 ± 2.0 µm	9.3 ± 1.8 µm	10.0 ± 1.9 µm	8.3 ± 1.5 µm
FHT Porosity											
FHT Pore Size											

Impact of Feedstock Variability on Build Quality

Green State Met Bars *Threshold image analysis of 5 areas in 1 cm x 1 cm XZ piece from mid-section*



Optimized SLM parameters produces low porosity → excellent build quality that is further improved with HIP

	A1	A2	A3	B1	C1	D1	E1	F1	G2	G3	H1
Green Porosity	0.19 ± 0.09 %	0.69 ± 0.23 %	0.19 ± 0.15 %	0.18 ± 0.09 %	0.14 ± 0.07 %	0.10 ± 0.07 %	0.46 ± 0.32 %	0.15 ± 0.09 %	0.14 ± 0.09 %	0.14 ± 0.07 %	0.19 ± 0.11 %
Green Pore Size	12.2 ± 3.0 μm	22 ± 4 μm	12 ± 3 μm	11.5 ± 2.3 μm	10.9 ± 2.3 μm	9.6 ± 2.6 μm	14.4 ± 3.0 μm	9.5 ± 2.0 μm	9.3 ± 1.8 μm	10.0 ± 1.9 μm	8.3 ± 1.5 μm
FHT Porosity	< 0.02 %	< 0.02 %	< 0.02 %	< 0.02 %	0.04 ± 0.02 %	< 0.02 %	< 0.02 %	< 0.02 %	< 0.02 %	< 0.02 %	0.06 ± 0.04 %
FHT Pore Size	3.3 ± 0.4 μm	3.3 ± 0.3 μm	3.5 ± 0.6 μm	3.4 ± 0.4 μm	3.1 ± 0.6 μm	5.1 ± 1.2 μm	3.3 ± 0.4 μm	3.3 ± 0.5 μm	5.0 ± 0.6 μm	4.5 ± 1.4 μm	4.3 ± 0.6 μm

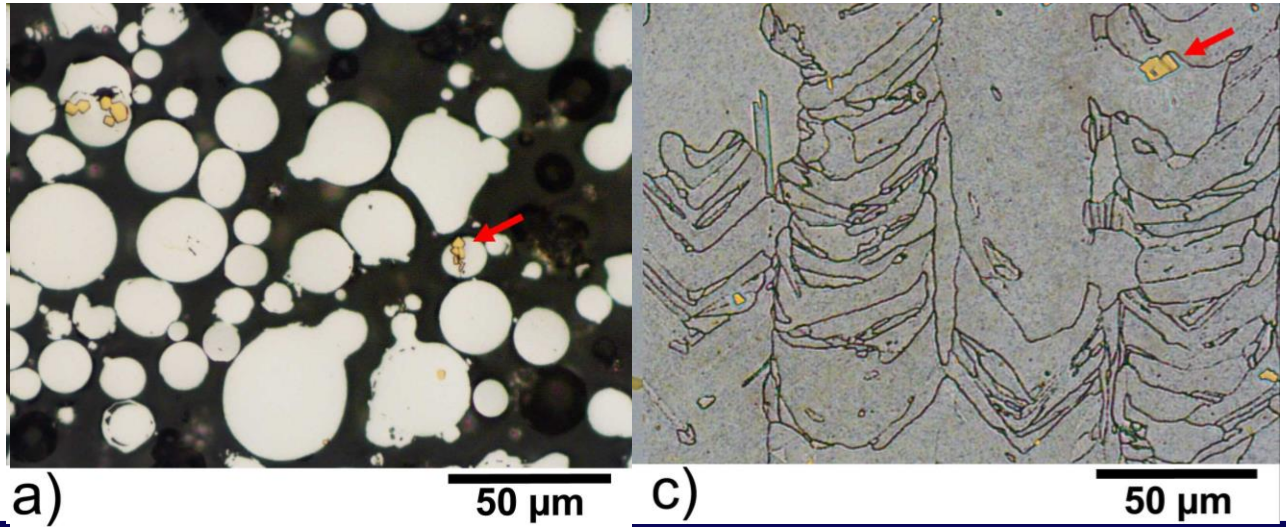
Fine ~100 nm nitrides present in all builds where volume fraction is linked to N content. Select builds have large nitrides

ID (Gas)	N (wt.% ppm)	Nitride V_f ** (%)	Nitride Mean Diam. ** (nm)	Carbide V_f (%)	Carbide Mean Diam. (µm)
A1	325	0.32	122 ± 18	0.038	0.56 ± 0.18
A2	90	0.23	106 ± 9	0.10	0.56 ± 0.09
A3	331	0.38	104 ± 12	0.023	0.47 ± 0.19
B1	25	0.17	97 ± 6	0.002	0.24 ± 0.14
C1 (N)	1395	0.54	127 ± 26	0.021	0.37 ± 0.11
D1	122	0.22	90 ± 3	0.09	0.56 ± 0.13
D2	115	0.26	94 ± 8	0.07	0.59 ± 0.17
E1 (N)	1220	0.49	80 ± 16	0.25	0.47 ± 0.05
E2 (N)	2770	0.87 (0.13)	141 ± 11 (7.8±1.0µm)	0.039	0.43 ± 0.05
F1	607	0.47	92 ± 9	0.012	0.40 ± 0.10
F2	370	0.35	110 ± 11	0.054	0.49 ± 0.10
G2	176	0.27	90 ± 5	0.058	0.59 ± 0.18
G3	199	0.34	105 ± 4	0.110	0.49 ± 0.08
G4	246	0.29	114 ± 14	0.058	0.59 ± 0.17
H1	562	0.42 (0.013)	112 ± 11 (6.9±0.4µm)	0.009	0.33 ± 0.10

Larger nitrides that are 6-8 µm in diameter may act as crack initiators

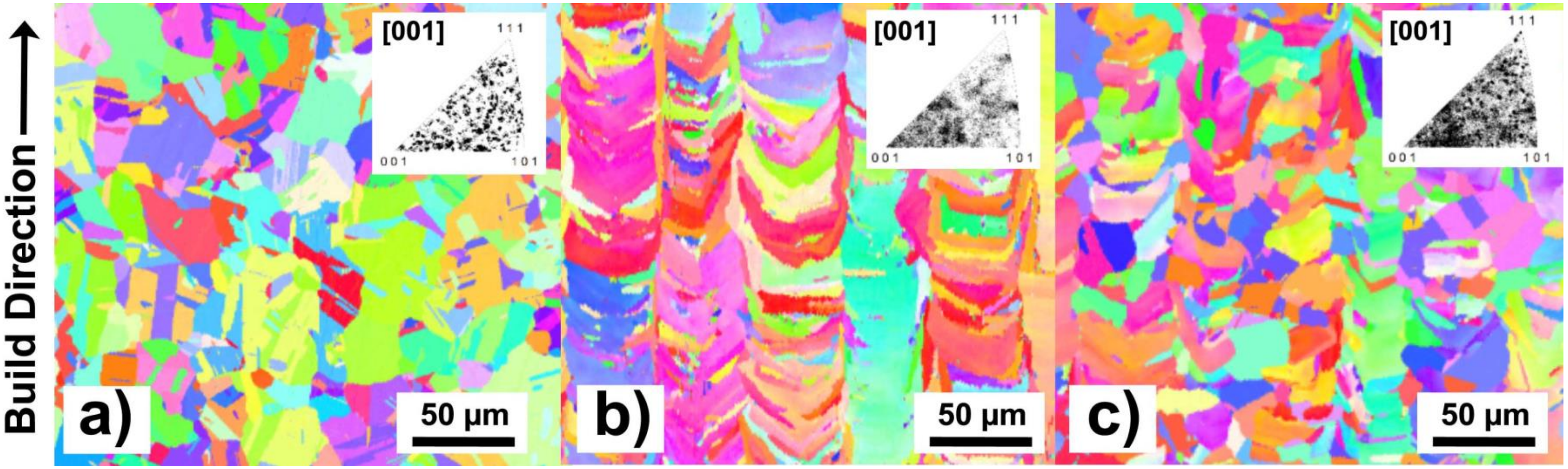
These large nitrides form during powder production

MC carbides are sub-micron in diameter and mostly uniformly distributed



Three grain structure regimes observed after heat treat

Fully Recrystallized Grain sizes 50 -90 µm | **Retained SLM structure for lots atomized in nitrogen** | **Partially Recrystallized for H1 lot**

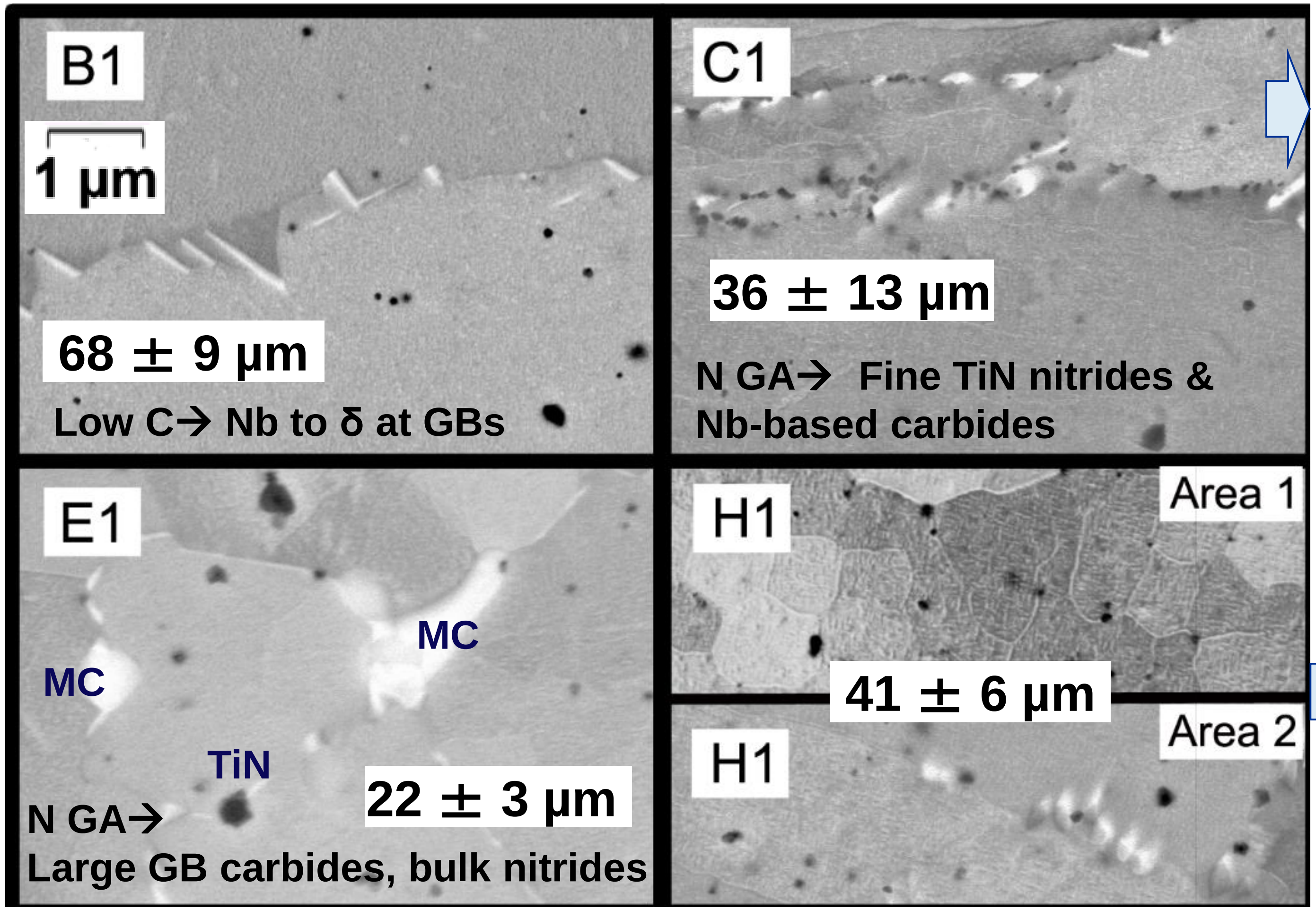


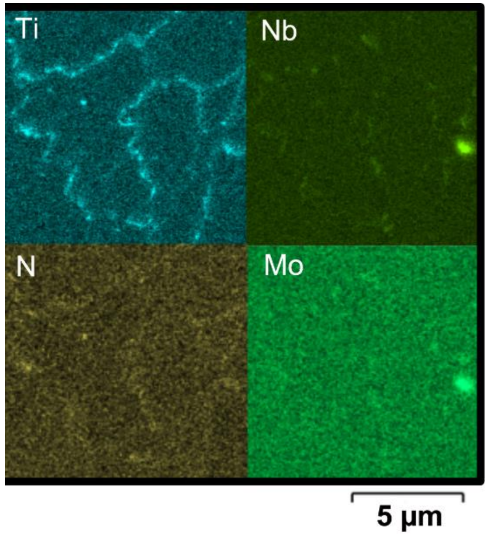
Recommend Ar-atomization and N content < 400 ppm for homogeneous grain distribution

Linear intercept	A1	A2	A3	B1	C1 (N GA)	D1	D2	E1 (N GA)	E2 (N GA)	F1	F2	G2	G3	H1
Mean grain diameter (± 95% CI)	70 ± 5 µm	57 ± 4 µm	74 ± 12 µm	68 ± 9 µm	36 ± 5 µm	53 ± 4 µm	51 ± 10 µm	21.5 ± 1.3 µm	32 ± 3 µm	89 ± 12 µm	64 ± 18 µm	63 ± 6 µm	71 ± 6 µm	40.9 ± 2.3 µm
N content ppm	325	90	331	25	1395	122	115	1220	2770	607	370	176	199	562

Nitrides and carbides pin grain boundaries in N-atomized powders (C1, E1, E2), retains smaller (001)-oriented grain sizes from SLM fabrication post HIP.

Select builds show distinct minor phase distributions at GBs



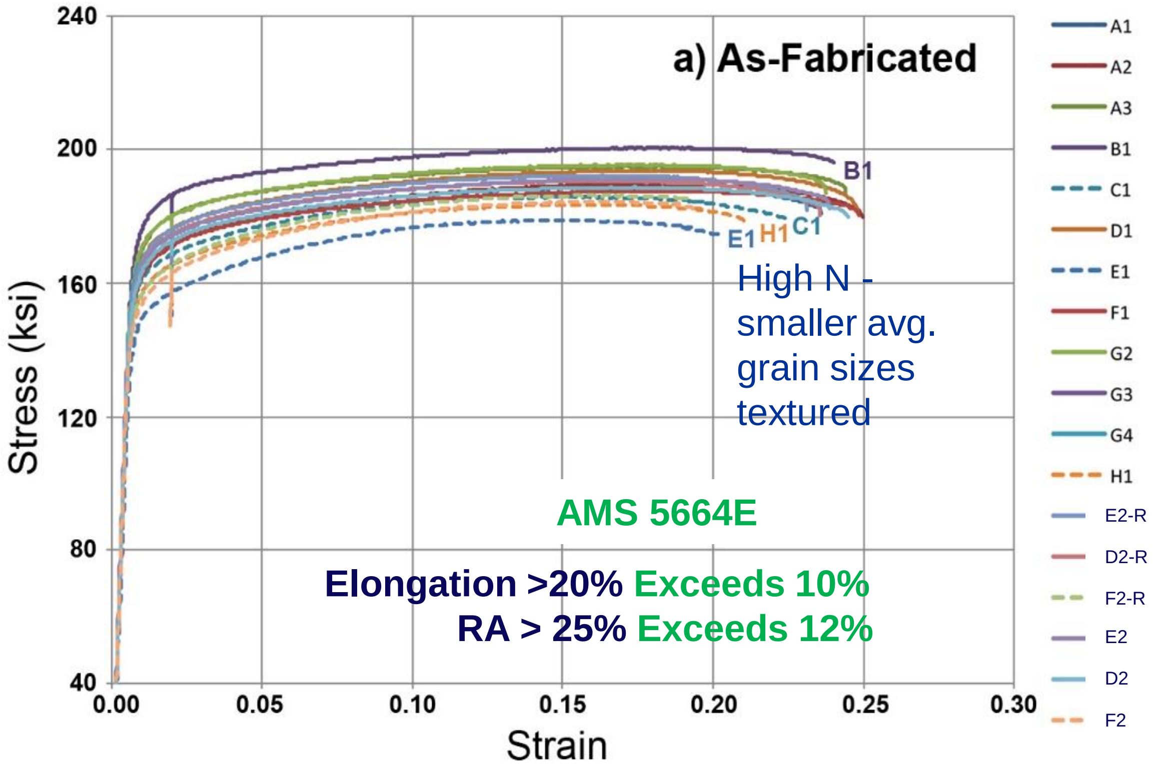


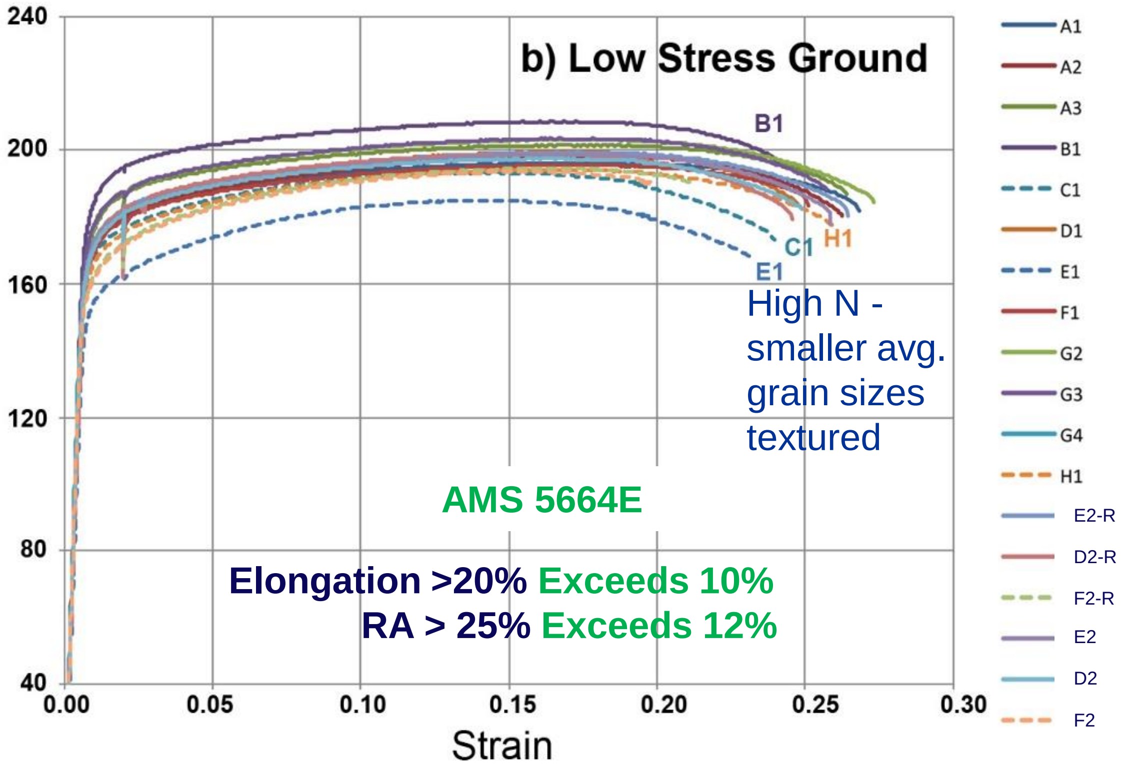
Partial recrystallization may be related to high fine content.

Majority builds show few minor phases at GBs: (N<500 ppm) & modest C

Room Temperature Tensile Testing

Heat Treated SLM 718 meets or exceeds minimum requirements for lots within chemistry specification



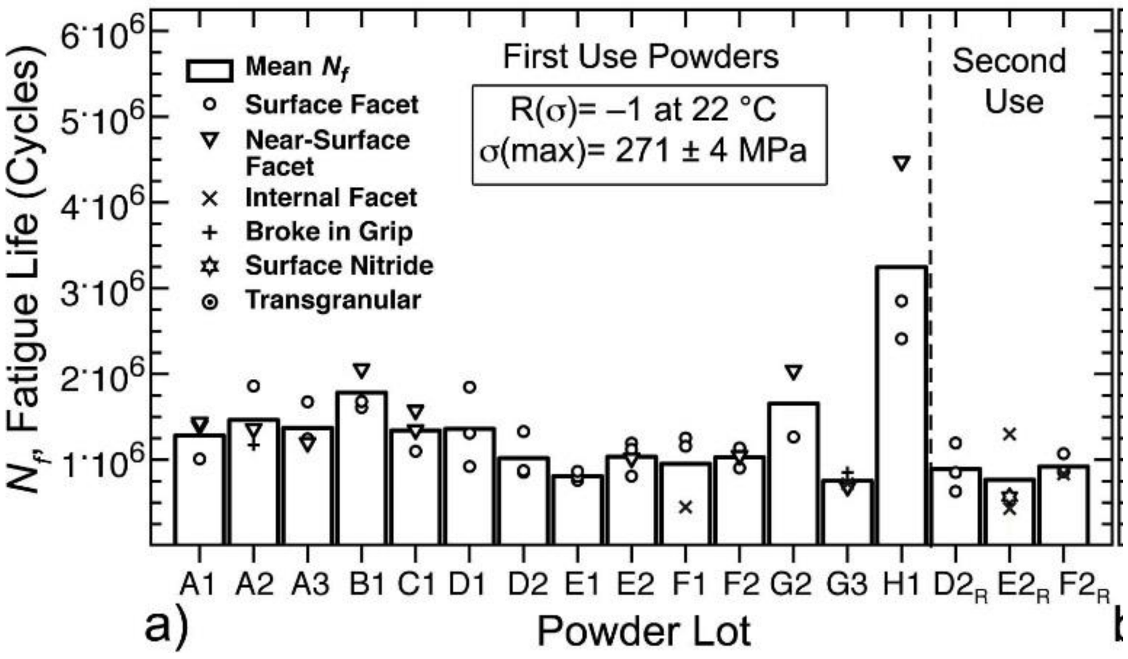


Solution and aged bars

As-fabricated	UTS (ksi)	0.2% YS Offset (ksi)
AMS 5664E	180.0	150.0
B1 (Low C)	200.5	171.1
Rest (H1 >>G2)	183.5-195.5	151.6-165.4
E1 (Off Spec)	178.8	144.9

Low Stress Ground	UTS (ksi)	0.2% YS Offset (ksi)
AMS 5664E	180.0	150.0
B1 (Low C)	208.8	179.3
Rest (H1 >>G3)	193.4-203.6	160.8-165.4
E1 (Off Spec)	185.0	150.6

HCF Response for As-fabricated surface condition

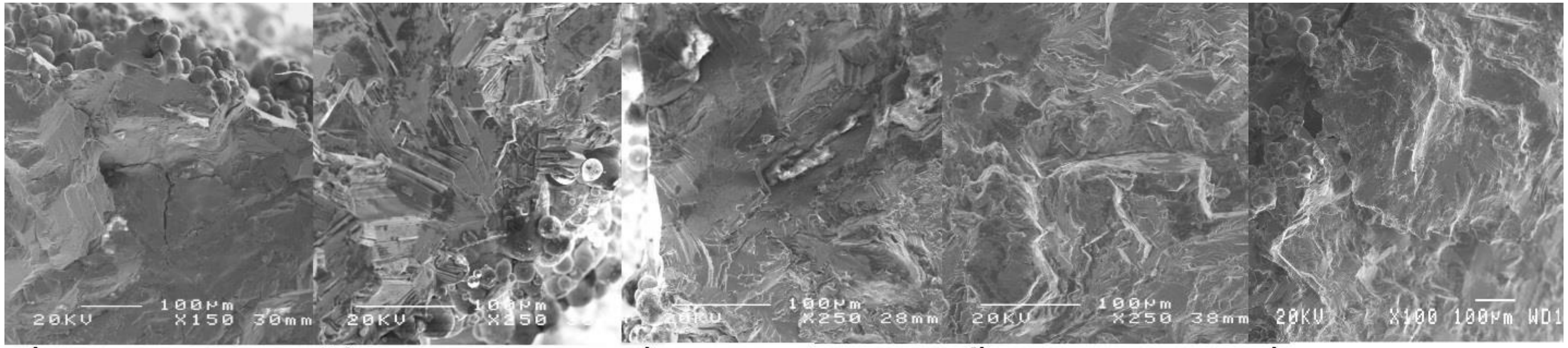


The surfaces of H1 test bars were more oxidized (SEE PAPER)

Overall low scatter in HCF response compared to the low stress ground

Predominant failure sites for was grain facets at or near the surface

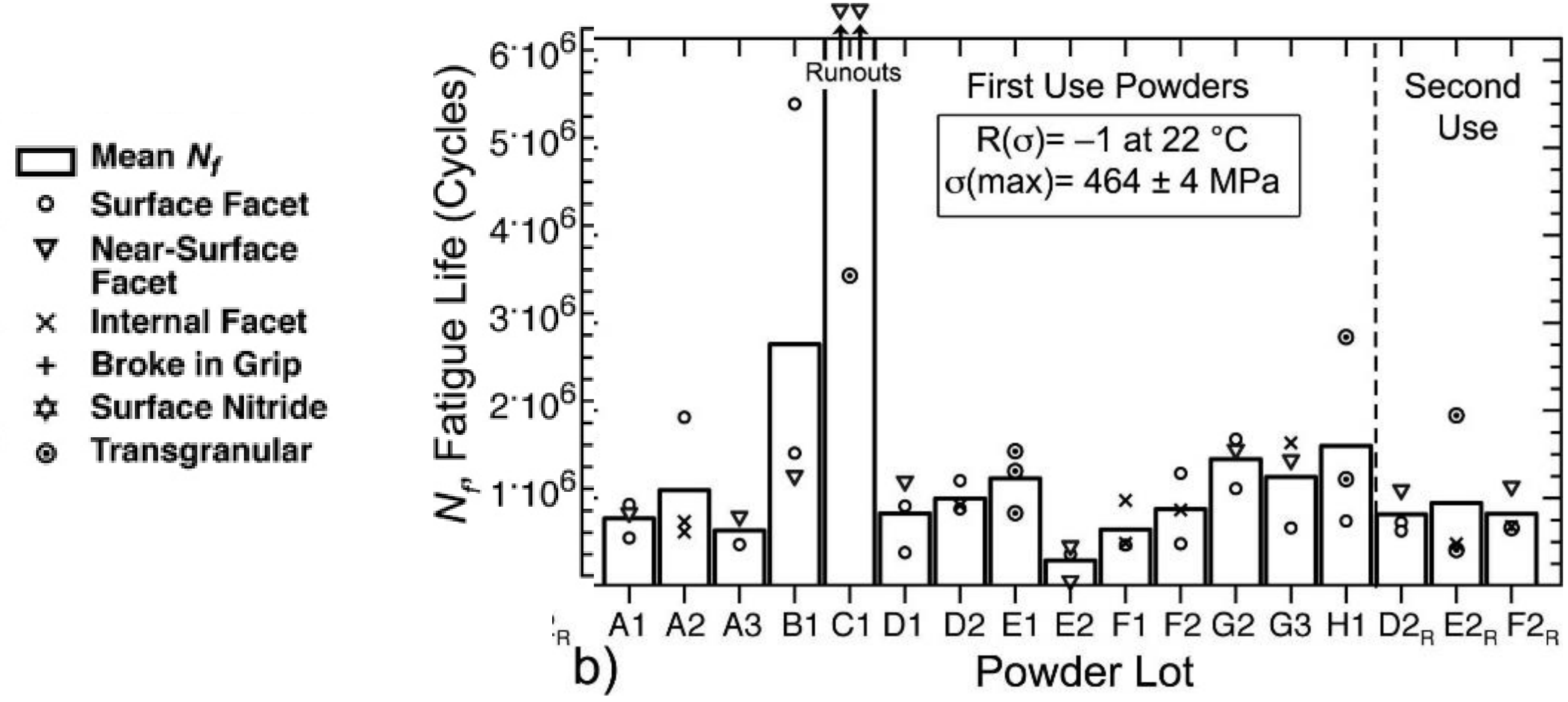
Very few internal initiations



a) Surface facet b) Near-surface facet (within ~200 µm) c) Surface facet with steps d) Internal facet e) Large 50 µm nitride (dark contrast)

Incidence of surface failures was significantly higher for AF surfaces due to stress concentrators associated with SLM surface asperities

HCF Response for low-stress ground surface condition



Overall more scatter in HCF

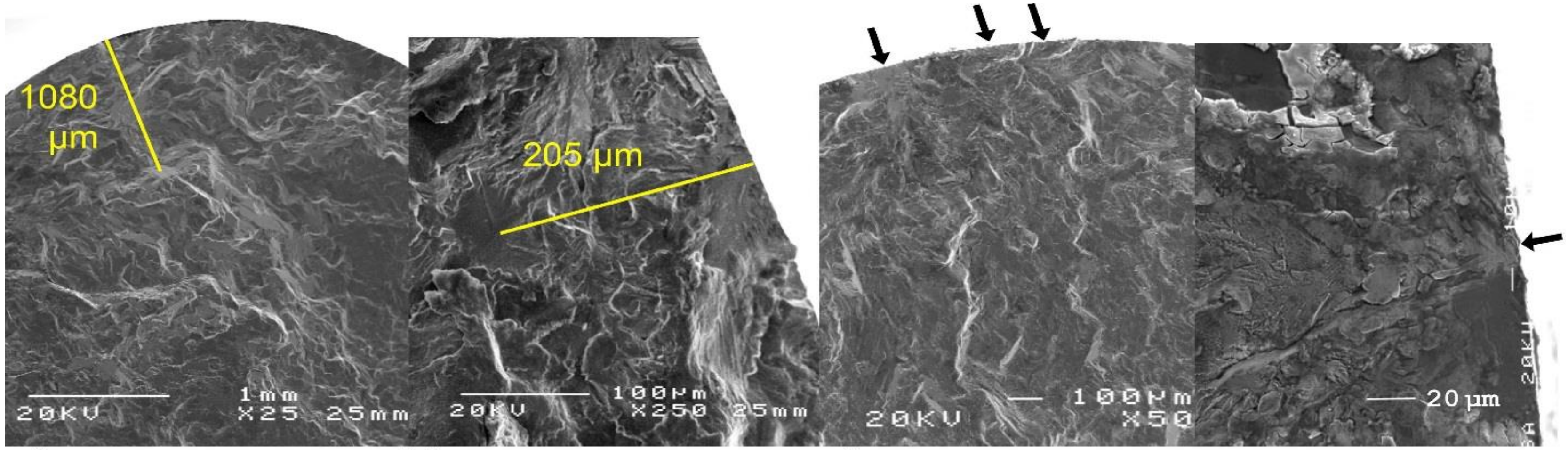
C1: N-atomized with refined grain size from pinned GBs

B1: highest strength, some GB pinning from delta

Predominant failure sites for was grain facets at or near the surface

More internal initiations

Transgranular crack initiation also observed



a) Internal facet b) Near-surface facet (within ~200 µm) c) d) Transgranular initiation

Summary and concluding remarks

- **Powders evaluated are distinct** – similar in that particles are highly regular spheroids- show differences in Al, C, N; PSDs, degree of agglomeration and surface roughness
- **Optimized SL M parameters for 718 yielded high quality builds** with low porosity and acceptable tensile properties across many distinct powder lots
- **Compositional differences has strongest impact on SLM 718 microstructure**
  - High N and C contents form TiN-nitrides and MC carbides on GBs that suppresses recrystallization during HT → 400 ppm N content a good rule of thumb cutoff to ensure equiaxed grain distribution
  - The **B1** alloy with very low in C led to **higher delta content leading to highest UTS**, while the **E1** alloy with very low in Al and high in C exhibited the lowest UTS
- **Significant knock-down in room temp HCF response for as-built SLM surface condition**; Stress concentrators at surface lead to higher incidence of surface crack initiation than observed in low stress ground condition
- For LSG surface condition, **the best room temperature HCF was for N-atomized C1** with prior GB particles (TiN, Nb-based carbides) that persist through heat treatment

Acknowledgements

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