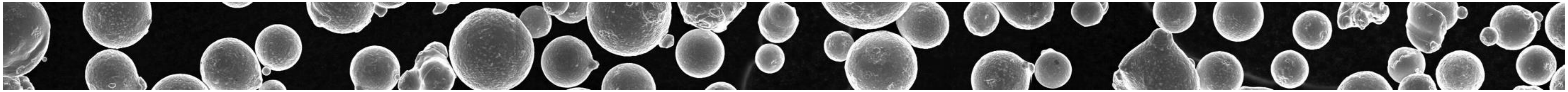
Impact of Powder Supply Variation on Mechanical Properties for Additive Manufacture of Alloy 718



Christopher Kantzos

NASA John H. Glenn Research Center at Lewis Field

Cleveland Ohio

NRC Workshop on Advanced Manufacturing, Dec 2020

SLM 718 Feedstock Variability Project – Intraagency Team: Supplier-to-supplier comparison 18 powders and 194 variables measured

Project Coordination
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- Cheryl Bowman, Team Lead
- Brian West

Powder Characterization
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Analytical Characterization
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- MSFC Heat Treat Facility
- GRC Specimen Shop

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- Tim Smith
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Flammability (Flam.) Analysis
- Jon Tylka (WSTF)
- White Sands Test Facility (WSTF)

Flam. Characterization
- Tim Smith
- Michael Kloesel (Cal Poly)

PCA analysis
- David Ellis

Program Advisors
- Kristin Morgan, Program Manager
- David Ellis
- Doug Wells
- Robert Carter

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

Motivation

- Standardization is needed for consistent evaluation of AM processes and parts in critical applications.
- Powder feedstock variability is a major unknown.
    - Chemistry and Size distribution are essential
    - Atomization Process?
    - Supplier Variation?
    - Variations within AMS Chemistry specification?

NASA Marshall
Standard 3716
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 **supplier-to-supplier comparison** to understand and identify the feedstock controls important to SLM Alloy 718

Approach: Survey wide range of off-the-shelf Alloy 718 powders

16 total powders acquired

- Supplier-to-supplier
- Lot-to-lot
- Gas and rotary atomized
- Ar and N cover gas
- Cut Size
- Once Reuse

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

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

Undersized / oversized cuts

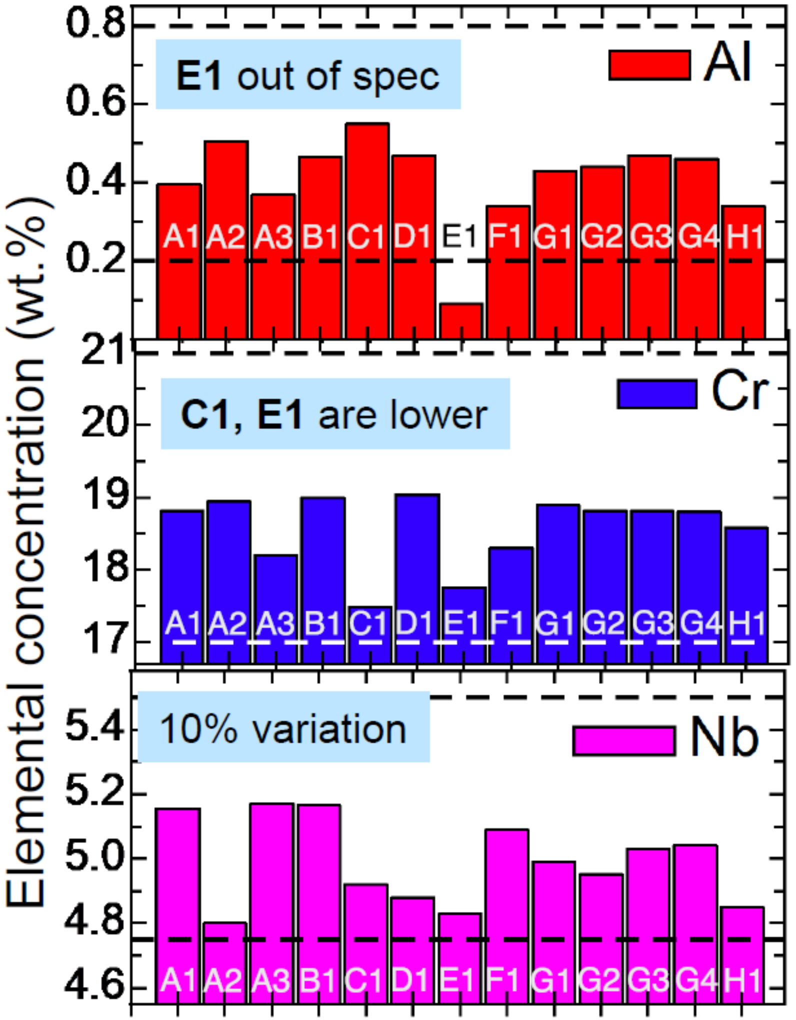
- G1: 0-22 Did not build

- G4: 45-90 Did not build well

ID		Cut	Atomization	Gas
A1	Supplier 1, Powder 1	15-45	Gas	Ar
A2	Supplier 1, Powder 2	10-45	Gas	Ar
A3	Supplier 1, Powder 3	10-45	Gas	Ar
B1	Supplier 2, Powder 1	15-45	**Rotary**	Ar
C1	Supplier 3, Powder 1	15-45	Gas	**N**
D1	Supplier 4, Powder 1	16-45	Gas	Ar
D2	Supplier 4, Powder 2	11-45	Gas	Ar
E1	Supplier 5, Powder 1	10-45	Gas	**N**
E2	Supplier 5, Powder 2	10-45	Gas	**N**
F1	Supplier 6, Powder 1	15-45	Gas	Ar
F2	Supplier 6, Powder 2	10-45	Gas	Ar
G	Supplier 7:	**G2:11-45** **G3: 16-45**	Gas	Ar
H1	Supplier 8, Powder 1	10-45	Gas	Ar

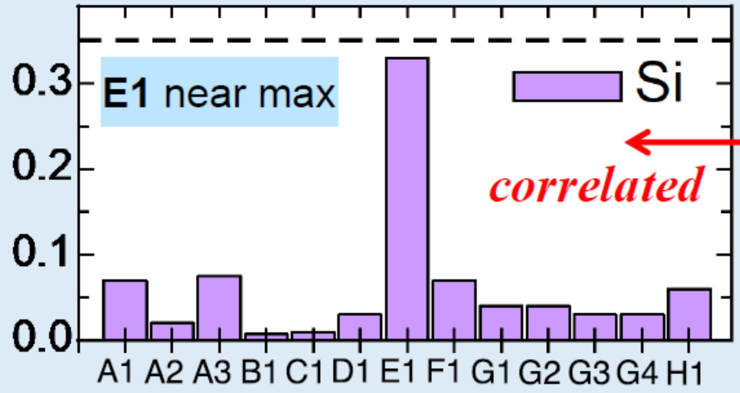
Majority of powder compositions within AMS 5664 chemistry specification
B1 low C, E1 high C, low Al

Precipitate strengtheners

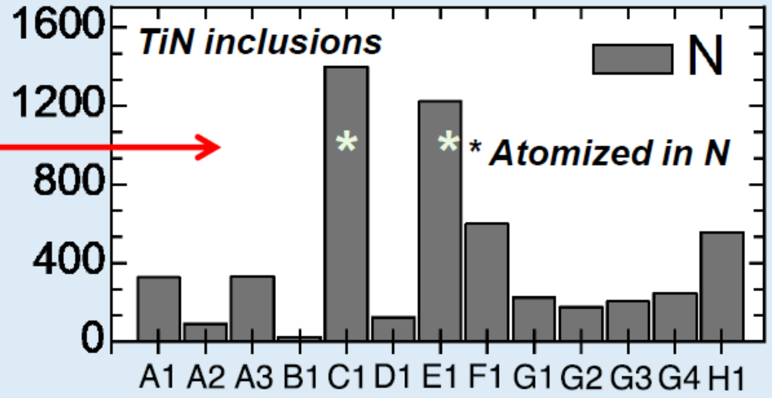


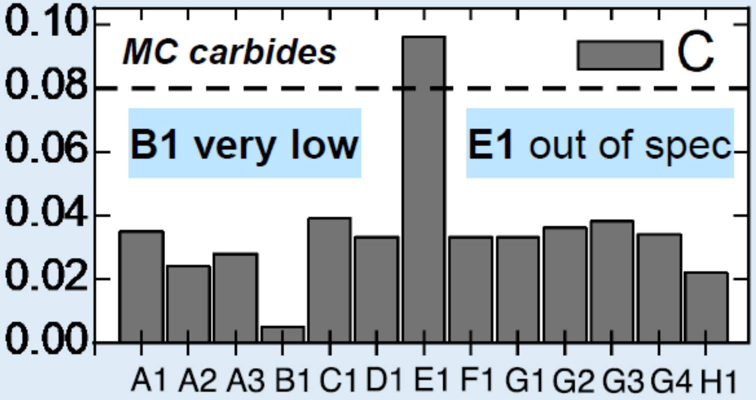
High trace impurity could lead to segregation, inclusions, & weldability issues

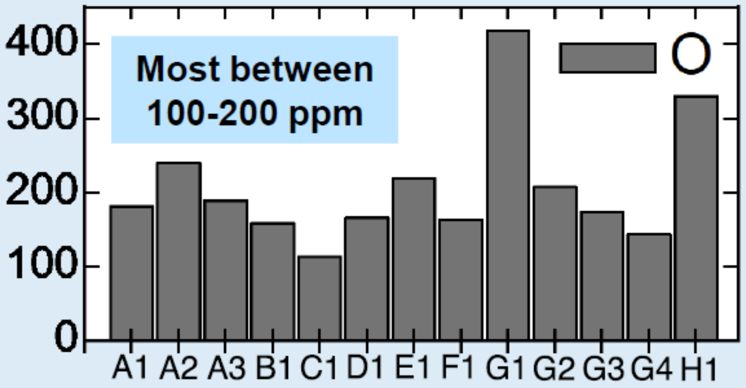
Elemental concentration (wt.%)



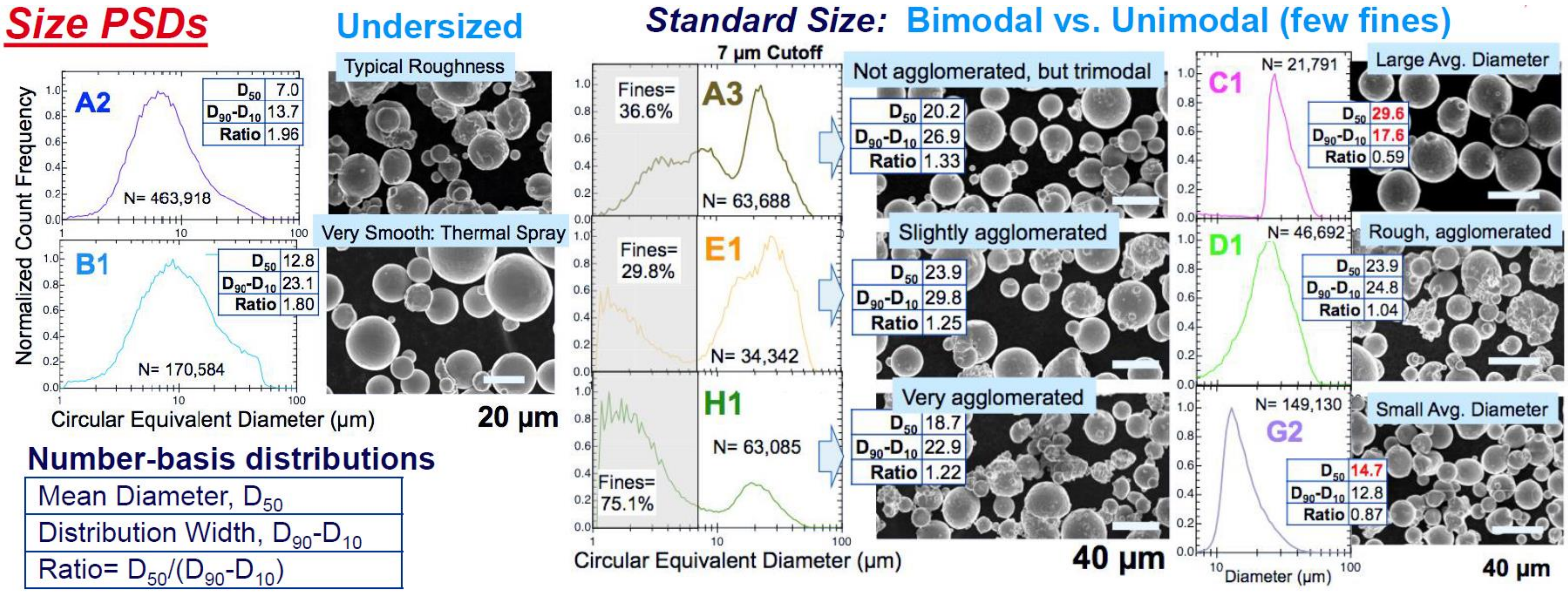
Elemental concentration (ppm wt.%)







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



Number-basis distributions

Mean Diameter, D_{50}
Distribution Width, D_{90}-D_{10}
Ratio= $D_{50}/(D_{90}-D_{10})$

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

Processing Details

NASA MSFC Concept Laser M1 machine:

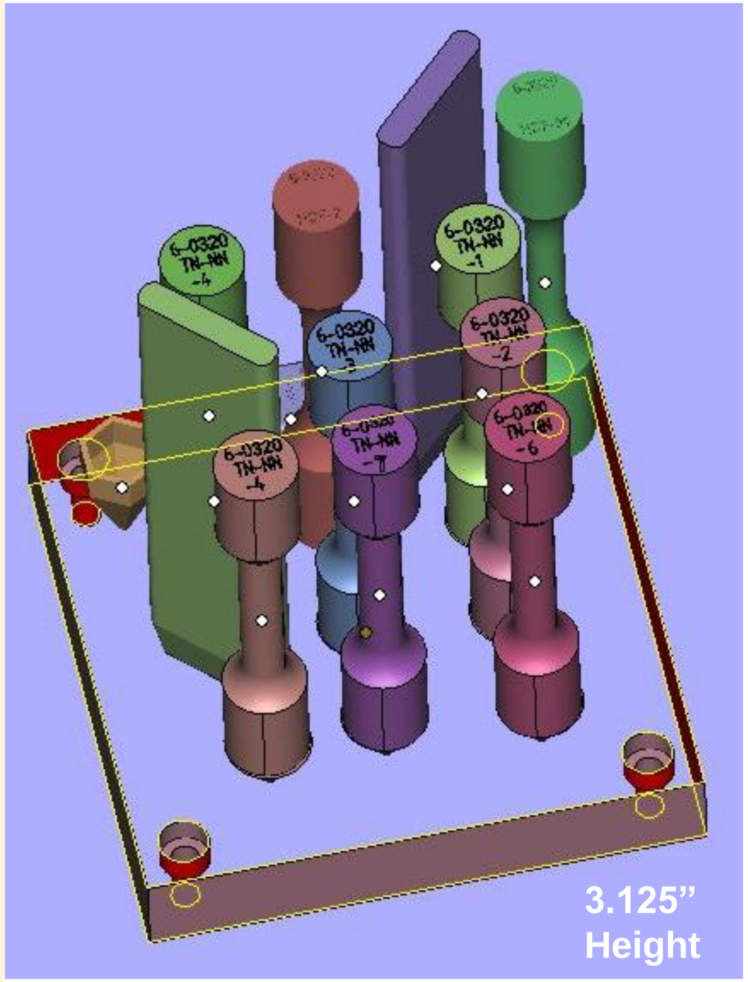
- Customized SLM 718 parameters for MSFC RS-25 projects
- Layer thickness: 30 µm
- Continuous scan strategy plus contours

Visible refill lines

Green-state “met” bar

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

Planned restarts



- **Custom Build Parameters**
- **10 cm height**
- **Snap off construction; no stress relief**
- **HIP: >1100 C hot isostatic press**
- **AMS 5664 heat treat schedule**

- **Two microstructure bars**
  - **Green-state bar** → inherent to the process
  - **HIP + heat treated bar** → post process response

- **Eight Mechanical Test Specimens**
  - **Two Tensile specimen**
  - **Six High Cycle Fatigue specimens**
- **Six Flammability specimens**

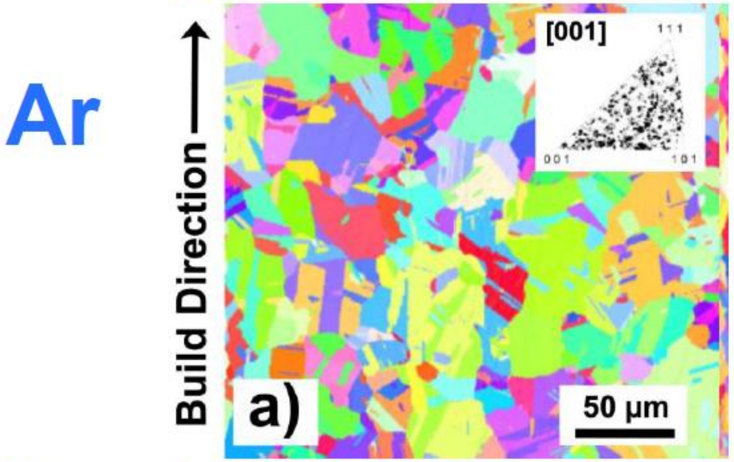
Microstructure and Grain Size

FG

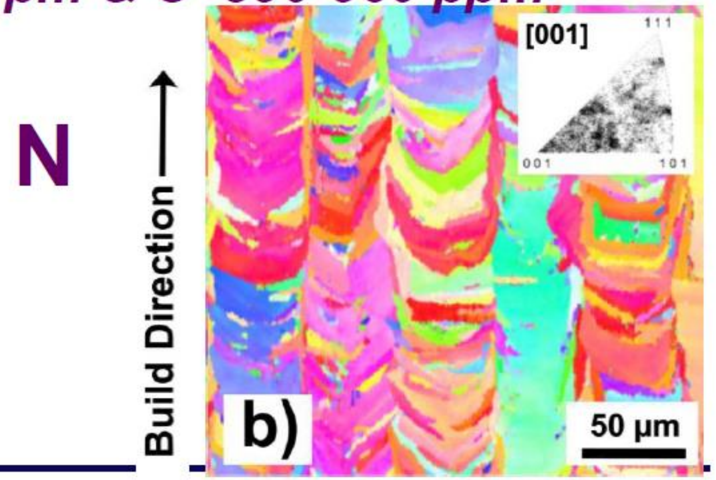
Fine grain

ID	Gas	D(50)	Avg Grain	All builds have fine nitrides in bulk
A1	Ar	25.1	70.0 ± 5.5	Recrystallized
A2	Ar	7.0	57.3 ± 3.6	Recrystallized
A3	Ar	20.1	74.4 ± 12.2	Recrystallized
B1	Ar	9.5	67.9 ± 8.6	Recrystallized
C1	N	29.1	35.9 ± 4.5	Anisotropic
D1	Ar	23.7	52.5 ± 3.6	Recrystallized
D2	Ar	17.9	51 ± 10	Recrystallized
D2-R	Ar	17.9	62.7 ± 8.6	Recrystallized
E1	N	23.8	21.5 ± 1.3	Anisotropic
E2	N	19.1	31.6 ± 5.0	Anisotropic
E2-R	N	19.1	19.5 ± 5.6	Anisotropic
F1	Ar	23.0	88.8 ± 12.3	Recrystallized
F2	Ar	17.7	64 ± 18	Recrystallized
F2-R	Ar	17.7	70 ± 14	Recrystallized
G2	Ar	14.6	63.2 ± 6.0	Recrystallized
G3	Ar	25.3	71.2 ± 6.4	Recrystallized
H1	Ar	18.7	40.9 ± 2.3	Partially Recryst'd

Few minor phases at GBs: N<600 ppm & C=50-390 ppm



Minor phases at GBs: N>1000 ppm & C=390-960 ppm



Mechanical Property Evaluation

Screen room temperature mechanical behavior

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

- **One tensile test per surface condition**
  - **Strain control up to 2% then stroke control at equivalent strain rate**

- **Three 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**

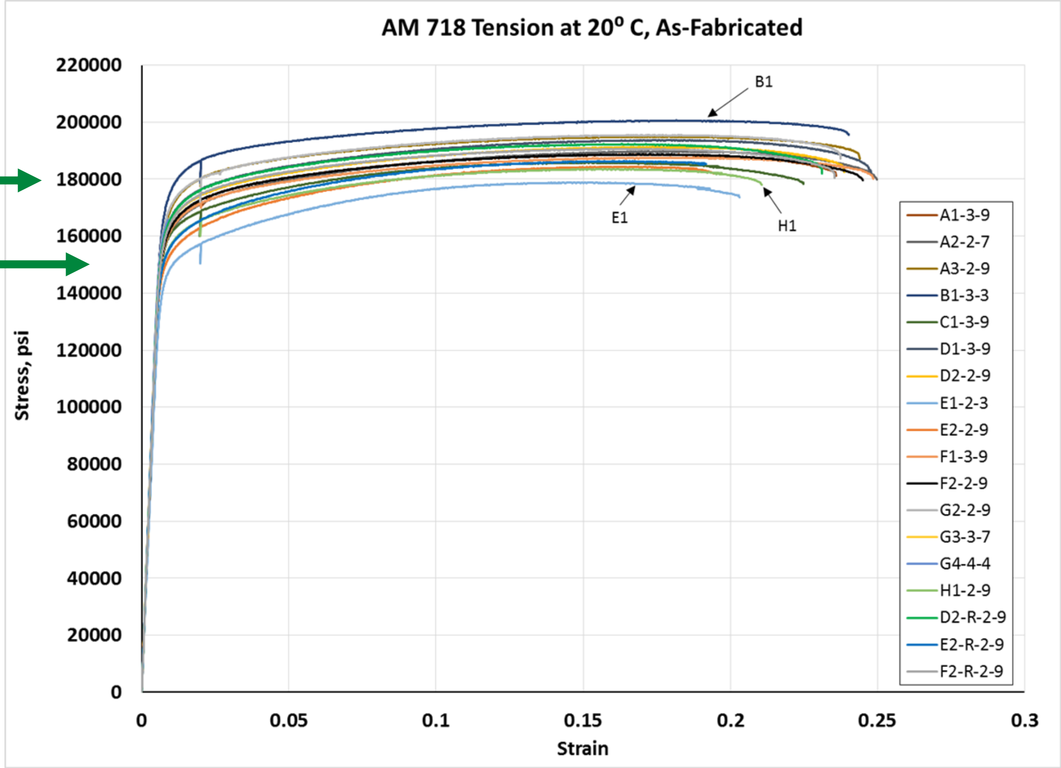
All mechanical testing performed after HIP (1160 C) + Soln (1065 C) + Precipitation Aging (760 C, 650 C)

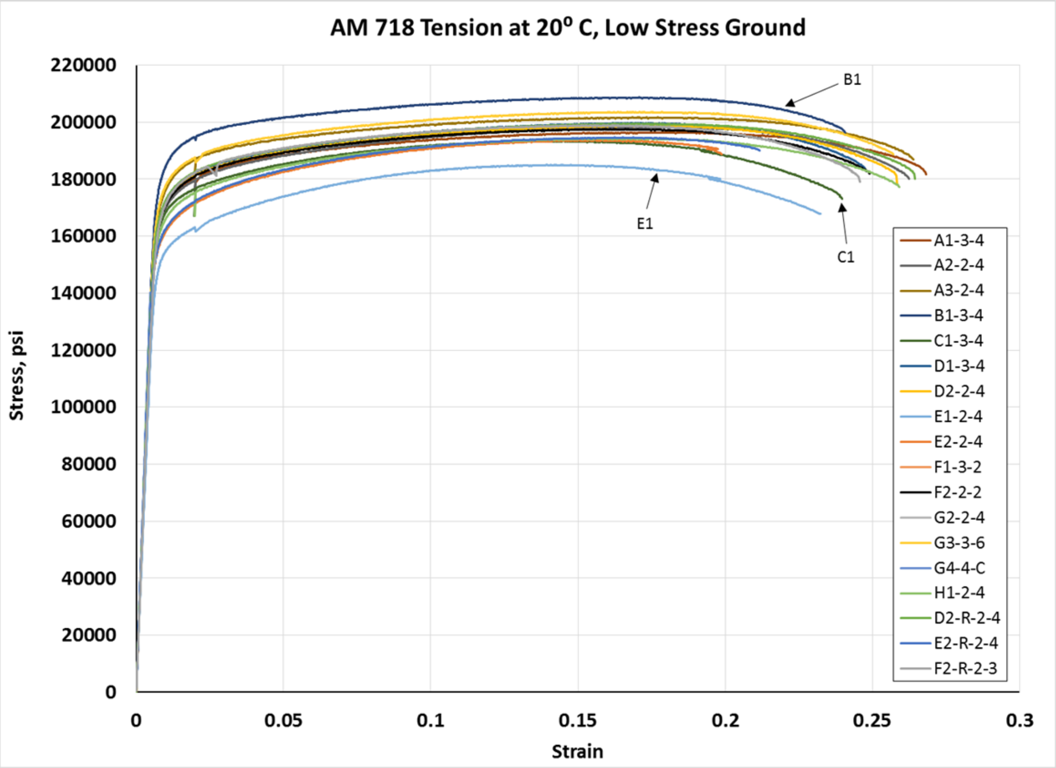
Room Temperature Tensile Meets Minimum Standard

AMS 5664E spec.

UTS

YS

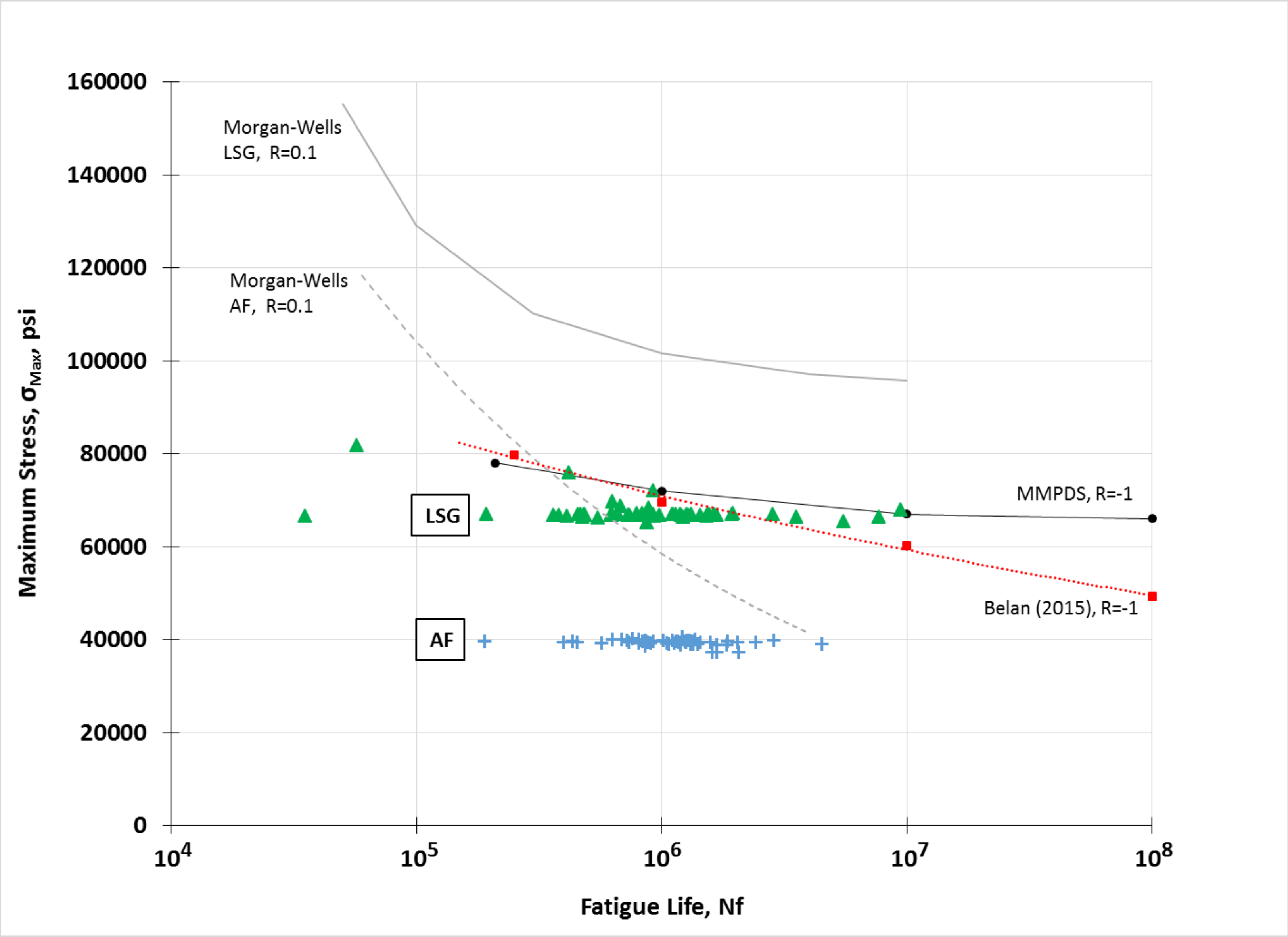




As-fabricated	UTS (ksi)	0.2% YS Offset (ksi)
B1	200.5	171.1
Avg	183.5-195.5	151.6-165.4
E1 (Low Al, Hi C)	178.8	144.9

Low Stress Ground	UTS (ksi)	0.2% YS Offset (ksi)
B1	208.8	179.3
Avg	193.4-203.6	160.8-165.4
E1 (Low Al, Hi C)	185.0	150.6

Room Temperature High Cycle Fatigue



Low stress ground compares well to literature

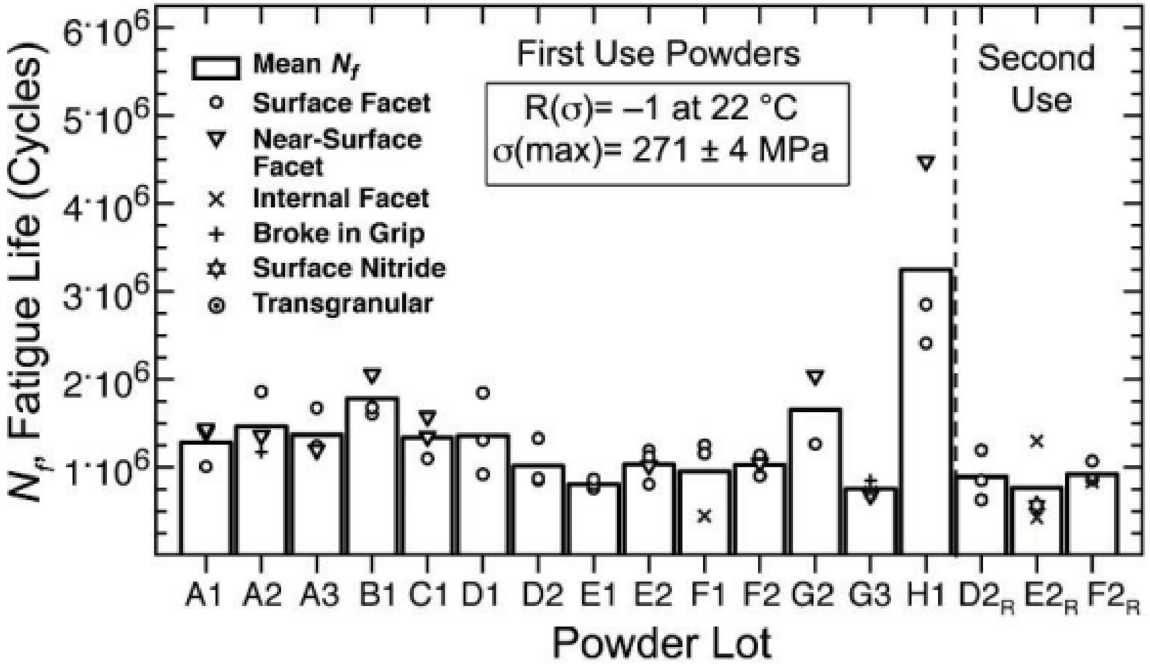
Statistical analysis shows two populations: C1 & B1 had highest lives, G4 and E2 the lowest

As Fabricated has less scatter, but 40% lower stress for comparable life

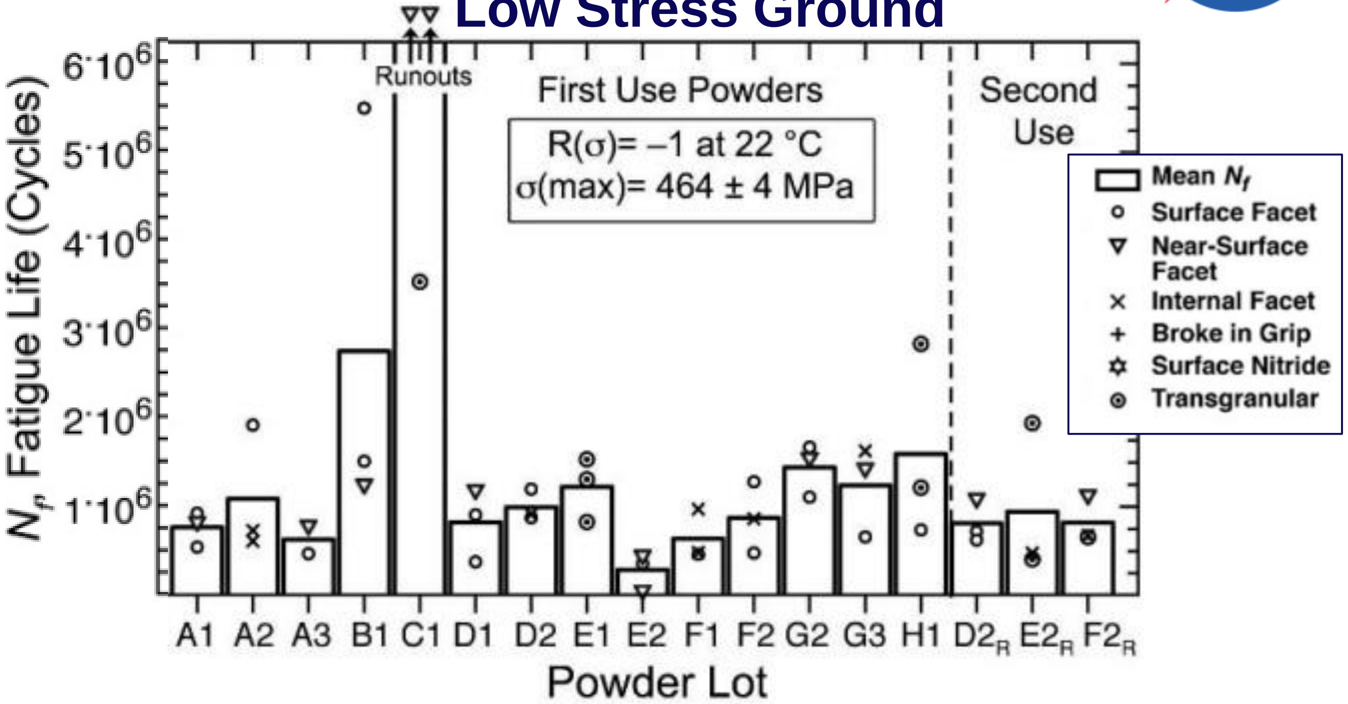
Only H1 lot was significantly different, with higher lives

Room Temperature High Cycle Fatigue

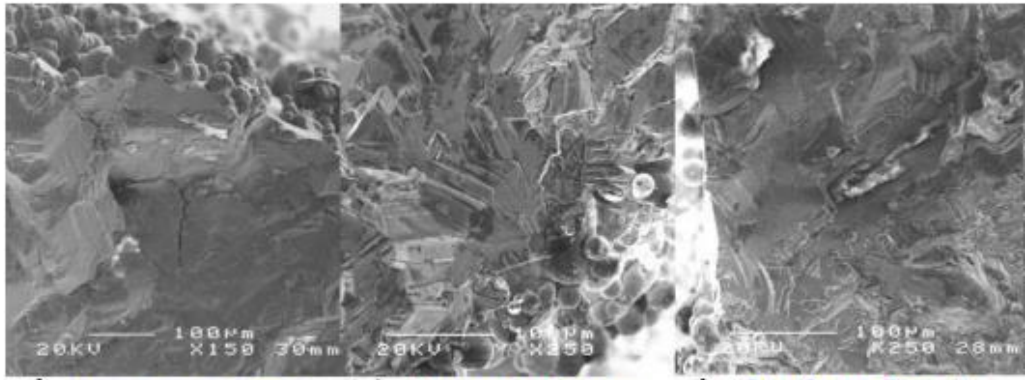
As-built Surface



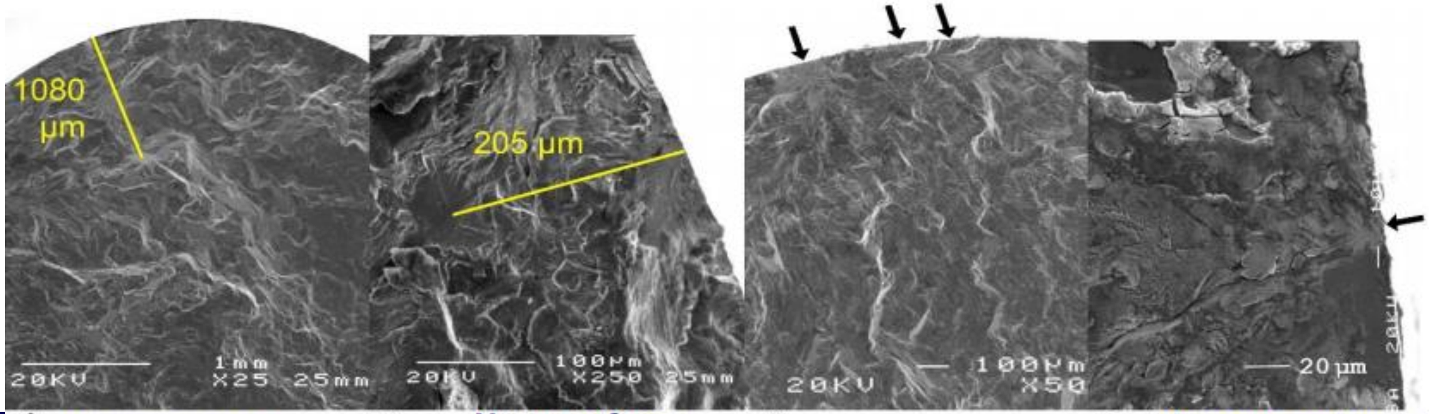
Low Stress Ground



Fine grained specimens often had improved fatigue performance



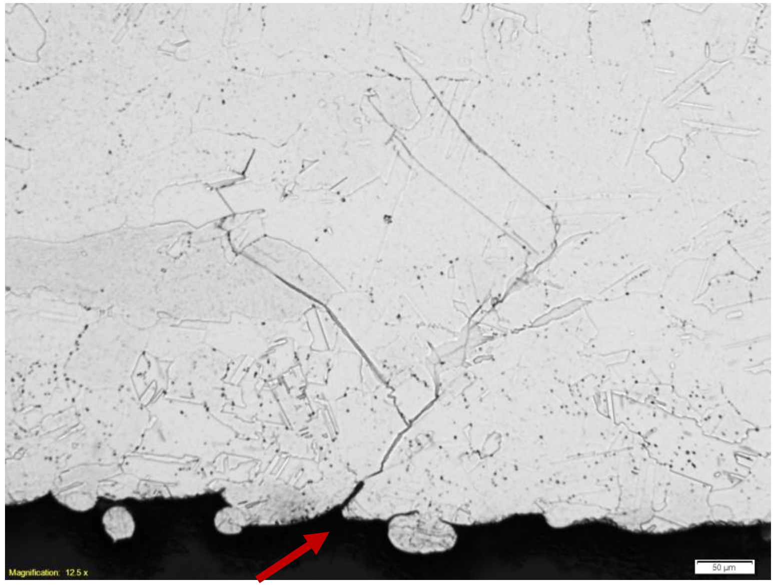
a) Surface facet b) Near-surface facet (within ~200 µm) c) Surface facet with steps

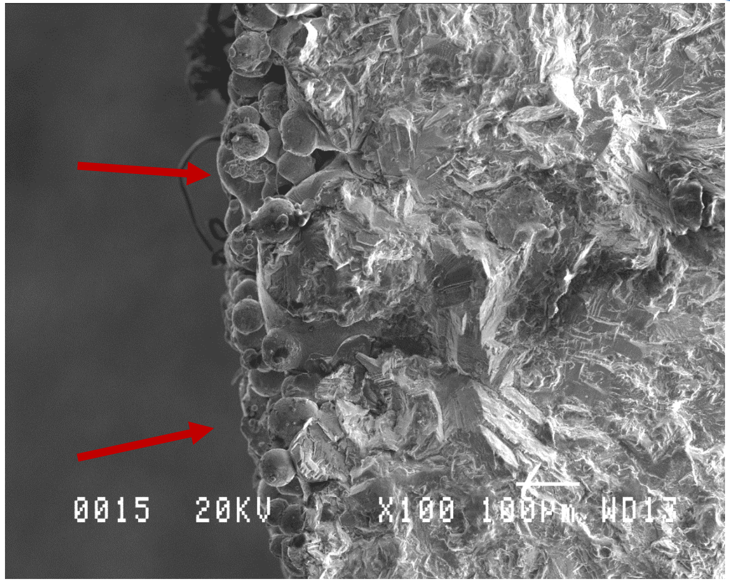


a) Internal facet b) Near-surface facet (within ~200 µm) c) d) Transgranular initiation

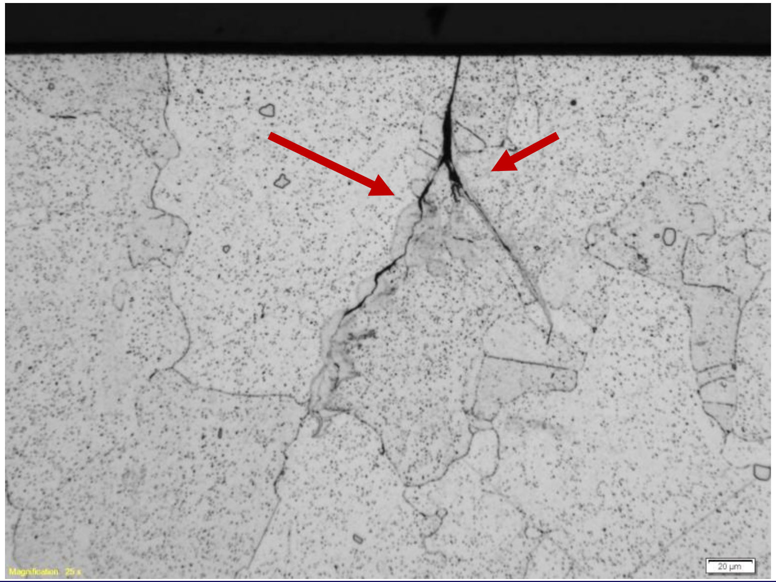
Fatigue Crack Initiation Sites

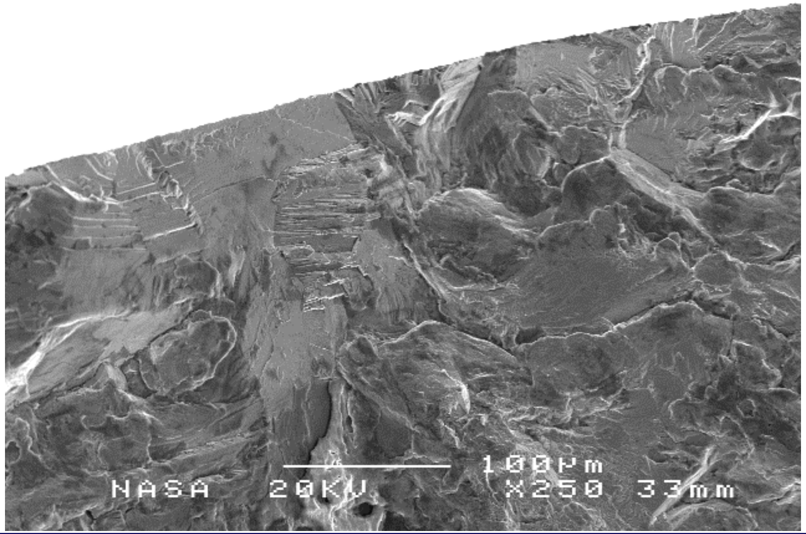
As fabricated: Incidence of surface failures was significantly higher for AF surfaces due to stress concentrators associated with SLM surface asperities





Low Stress Ground: More internal initiation sites





Powder and Build Quality Summary

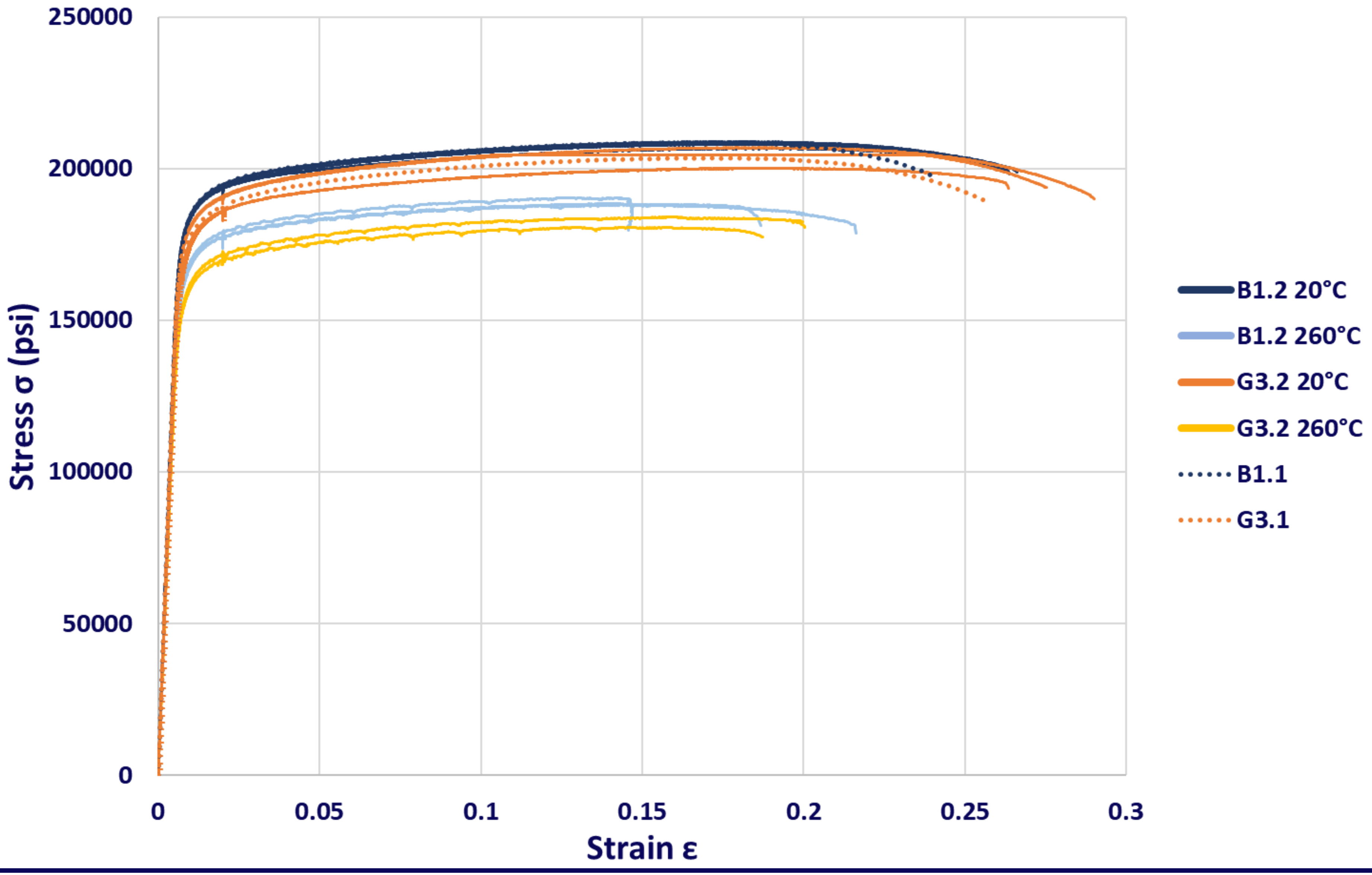
- **Majority of powder compositions within AMS 5664 chemistry specification** (E1 out)
- **Powders evaluated are distinct** – similar in that particles are highly regular spheroids; differences in N; Particle Size Distributions; degree of agglomeration and surface roughness
- **Optimized SL M parameters for 718 yielded high quality builds** with low porosity and full recrystallization across many distinct powder lots
- **Compositional differences had 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
- **As-Fabricated surfaces met minimum tensile strength** except for E1 which was chemically out-of-spec
- **Low stress ground surface produced high cycle fatigue lives comparable with literature**
- **Fatigue strength reduced 40 percent for as-fabricated surface**

(In-Progress) Phase 2: Downselection

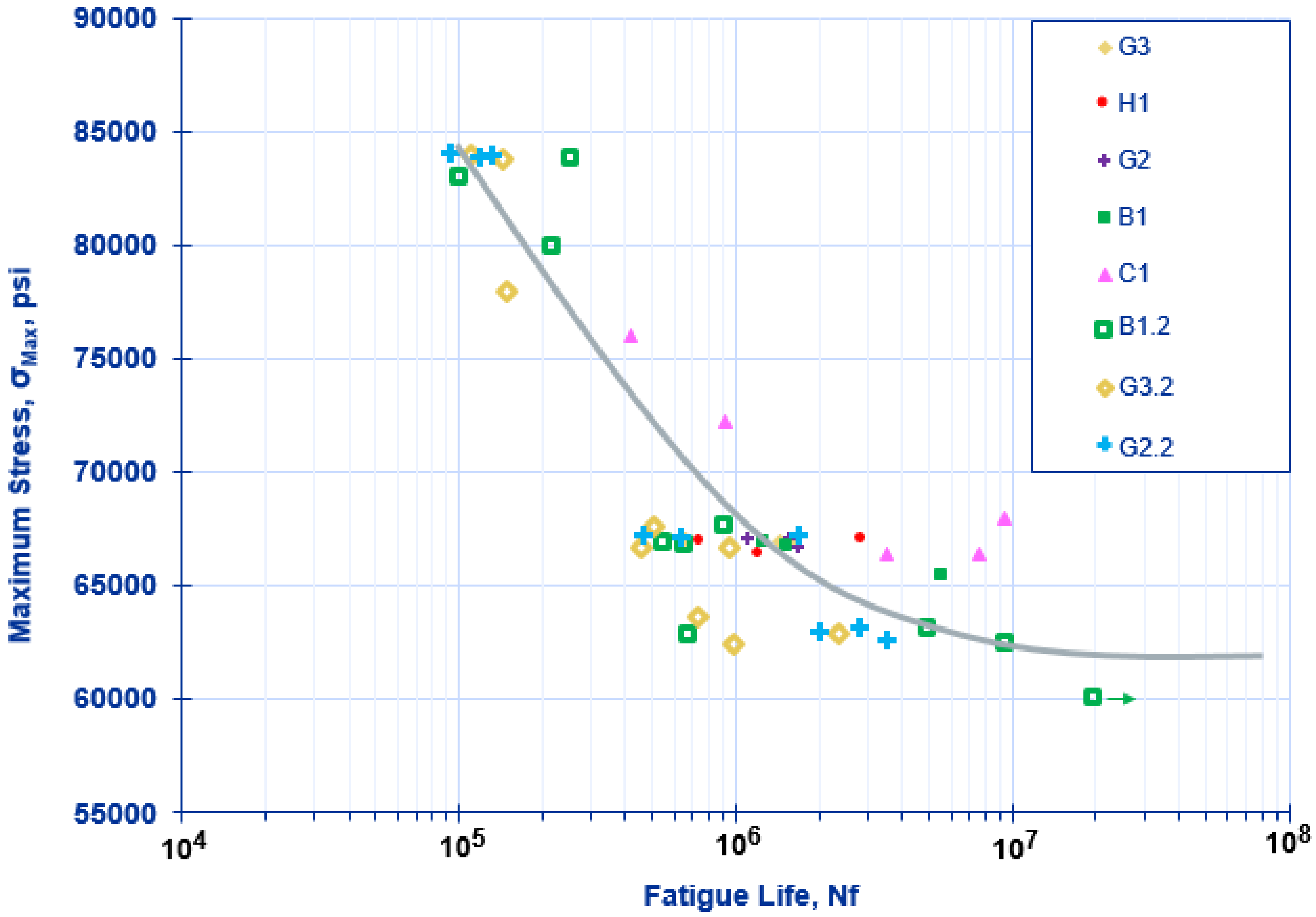
- **Five powder lots selected for a further investigation: *B1, C1, G2, G3, H1***
- **Powder, chemistry, and microstructure analysis**
- **Expanded Mechanical Testing**
  - **Cryogenic and Elevated Temperature Tensile**
  - **Room and Elevated Temperature High Cycle Fatigue**
  - **Creep**
  - **Crack Growth and Fracture Toughness**
  - **Broader As-built and Ground Surface Flammability**

ID	Cut	Atomization	Gas	Note
B1	15-45	Rotary	Ar	Low C/N, V. Smooth
C1	15-45	Gas	N	High N, Narrow PSD
G2	11-45	Gas	Ar	Good PSD
G3	16-45	Gas	Ar	Good PSD
H1	10-45	Gas	Ar	Moderate N, High Fines

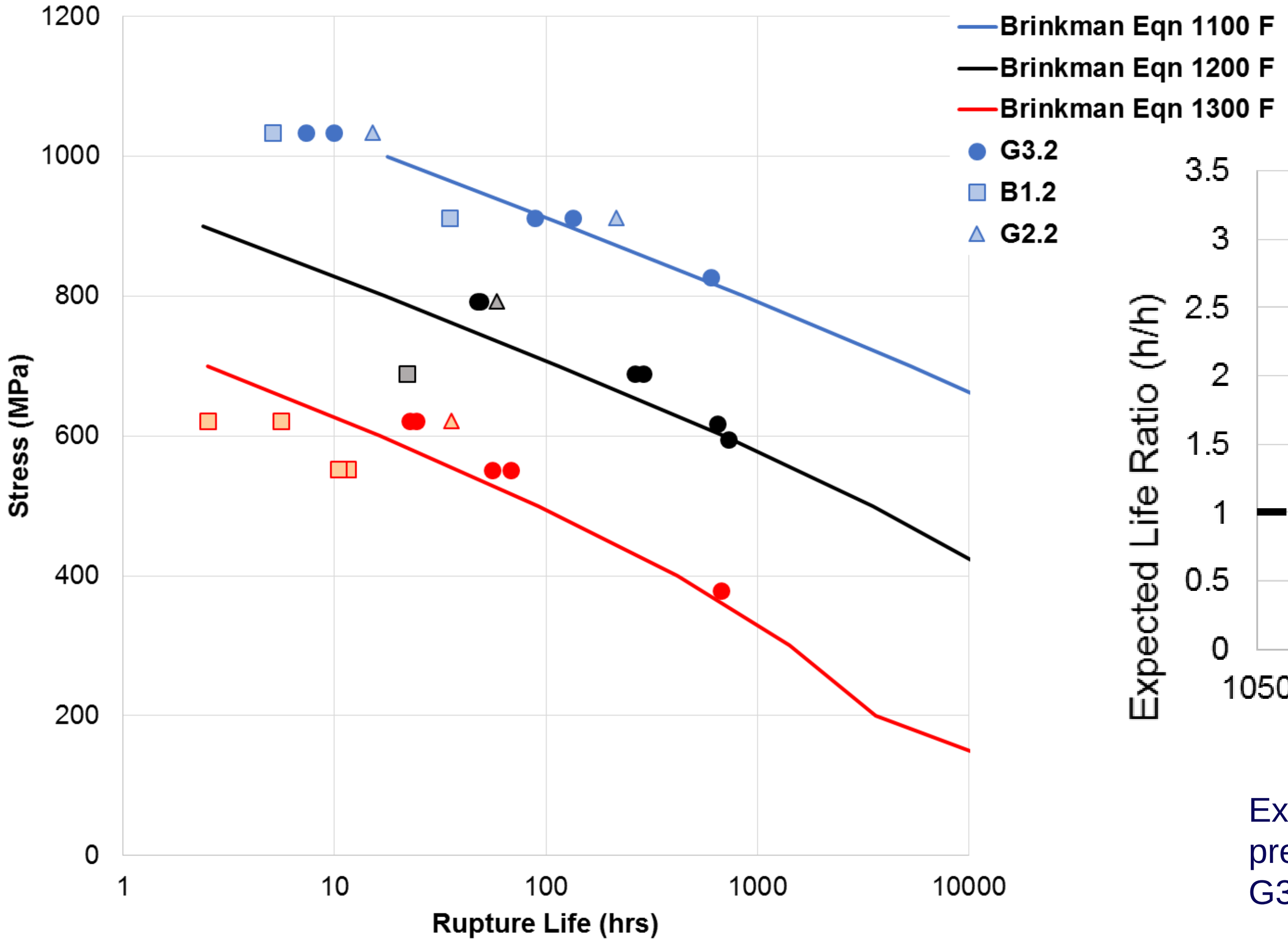
Round 2 Mechanical Testing (On-Going) - Tensile

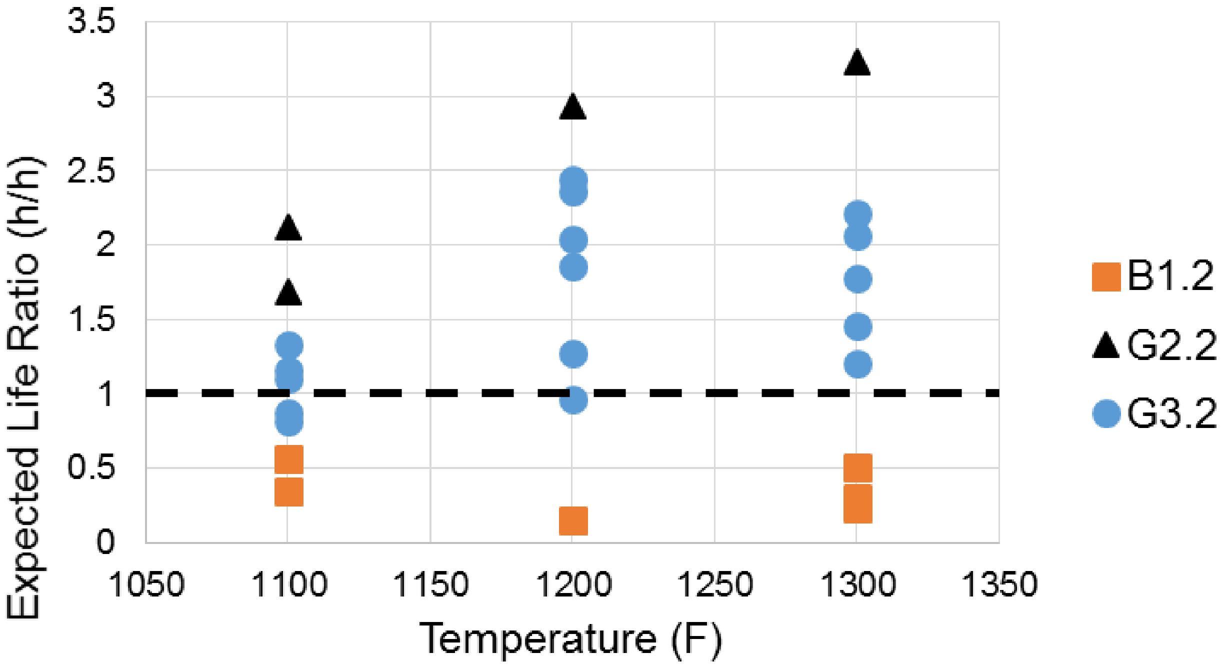


Round 2 Mechanical Testing (On-Going) - Fatigue



Round 2 Mechanical Testing (On-Going) - Creep

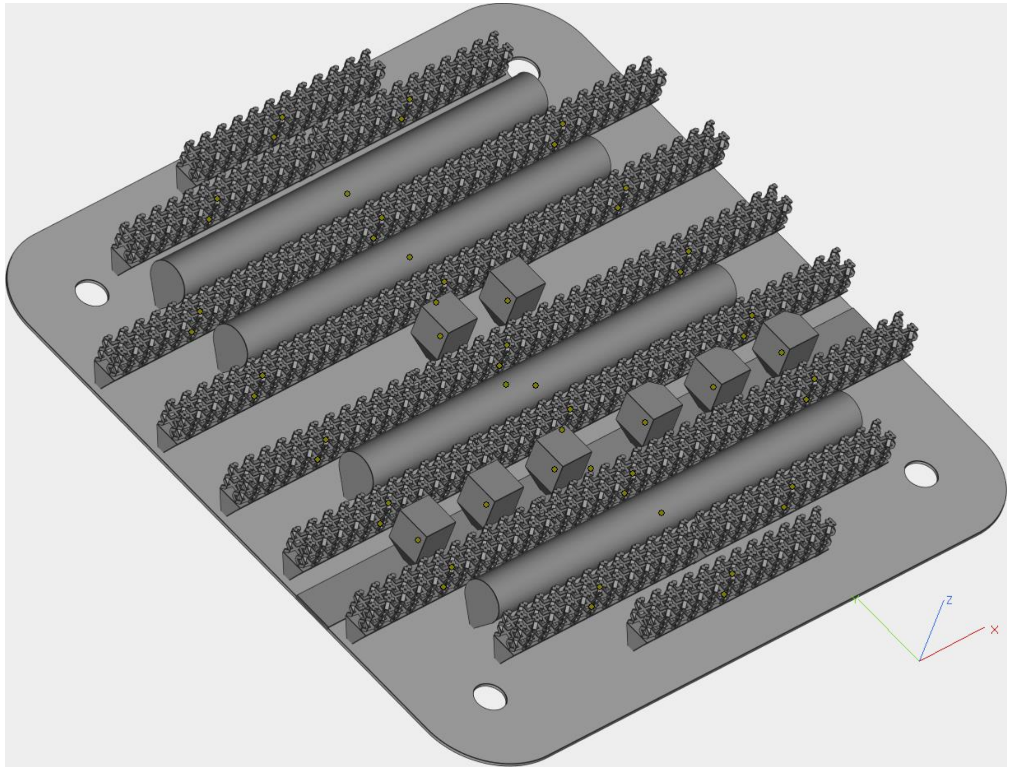




Expected Life Ratio of 1 = Life matches Brinkman prediction. B1.2 lives are falling below while G2.2 and G3.3 are meeting or exceeding.

Colors correspond to test temperature; Symbol shapes represent build batches

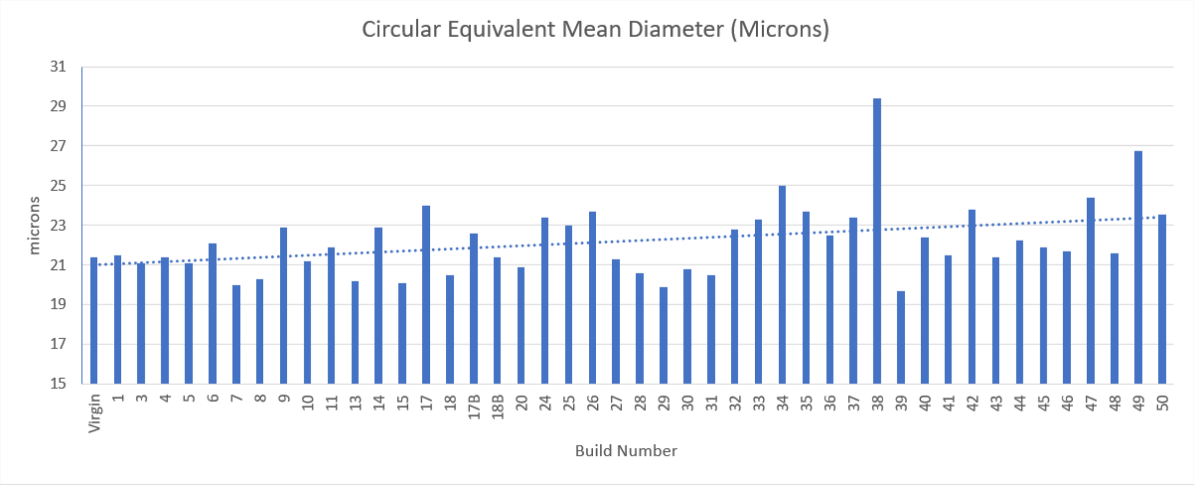
Phase 3: Powder Recyclability

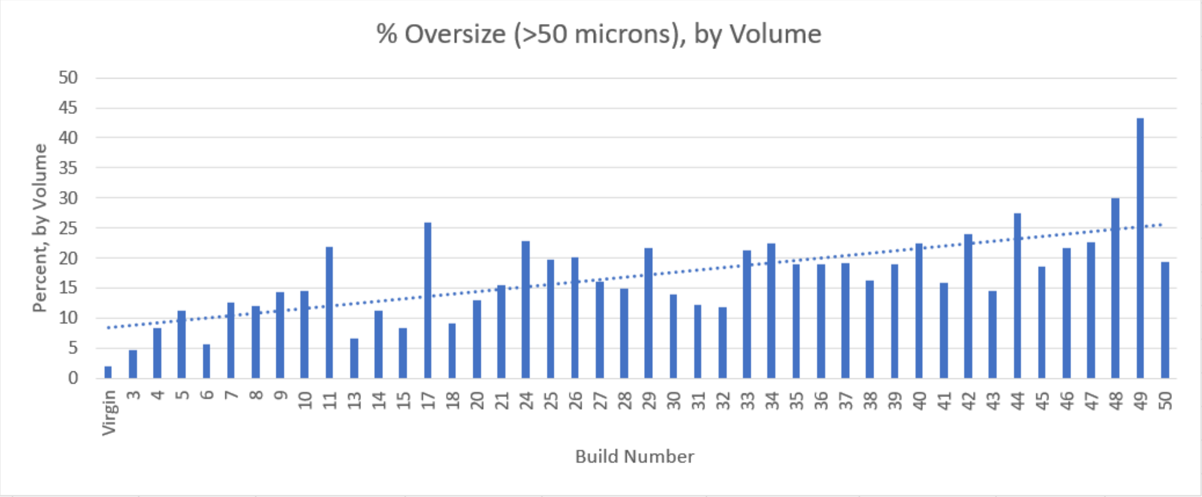


- One powder lot selected for a further investigation: *G2*
- Recycling Study: 50 builds reusing powder
    1. Virgin powder sieved -270/+500
    2. Complete build
    3. Leftover powder sieved again to -270/+500
    4. Recycled powder is blended with as much virgin powder necessary to complete next build
    5. Repeat steps 2-4 49 times for a total of 50 builds
- Builds included
    - 8 cubes for microstructural/defect analysis
    - 4 bars for mechanical testing.
- Horizontal test bars to keep build short
- Lattice “Fences” to increase laser-powder interaction
- Everything HIPed and heat treated

Powder Recyclability: Powder

- **Powder showed increase in dark particles suggesting oxidation**
- **Particle size did not change significantly, though percentage of oversized powder increased**



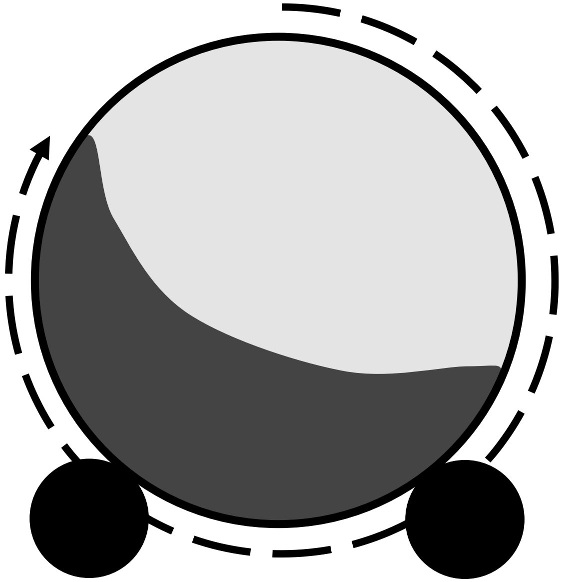


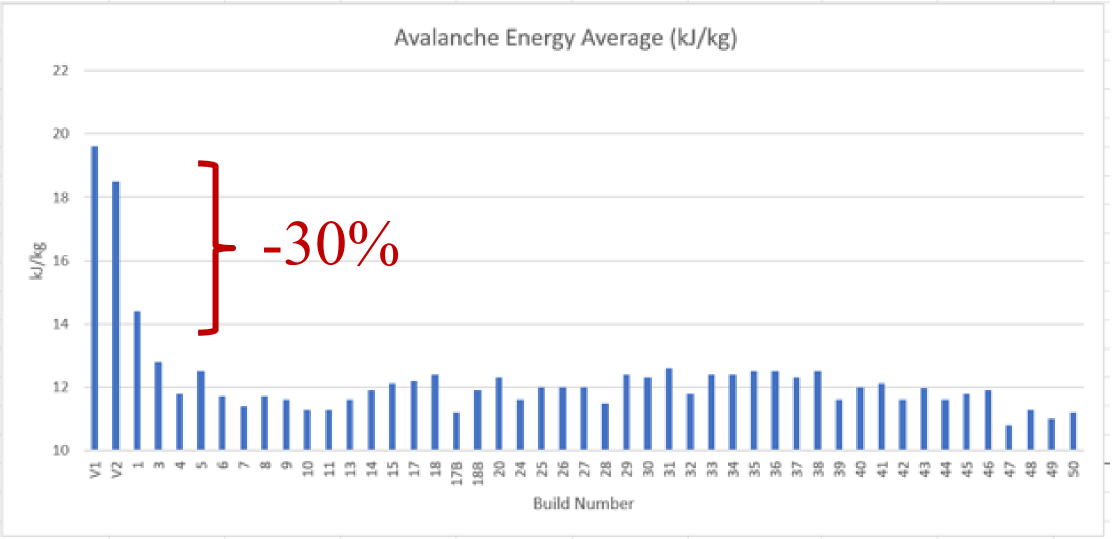
Build 3

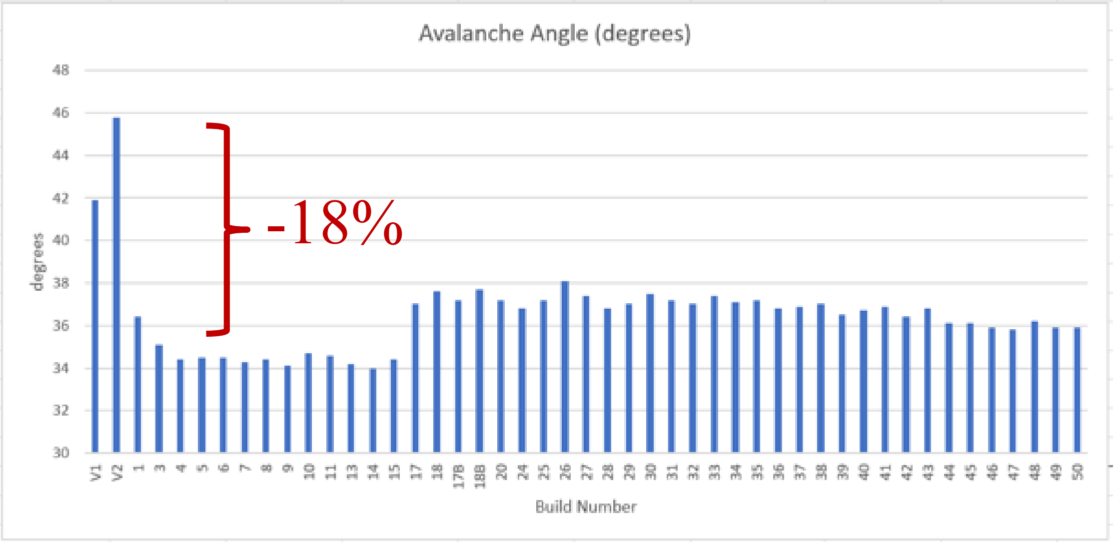
Build 50

Powder Recyclability: Powder

- **Flowability significantly improved after build 1**
- **Measured using Revolution rotating drum technique**

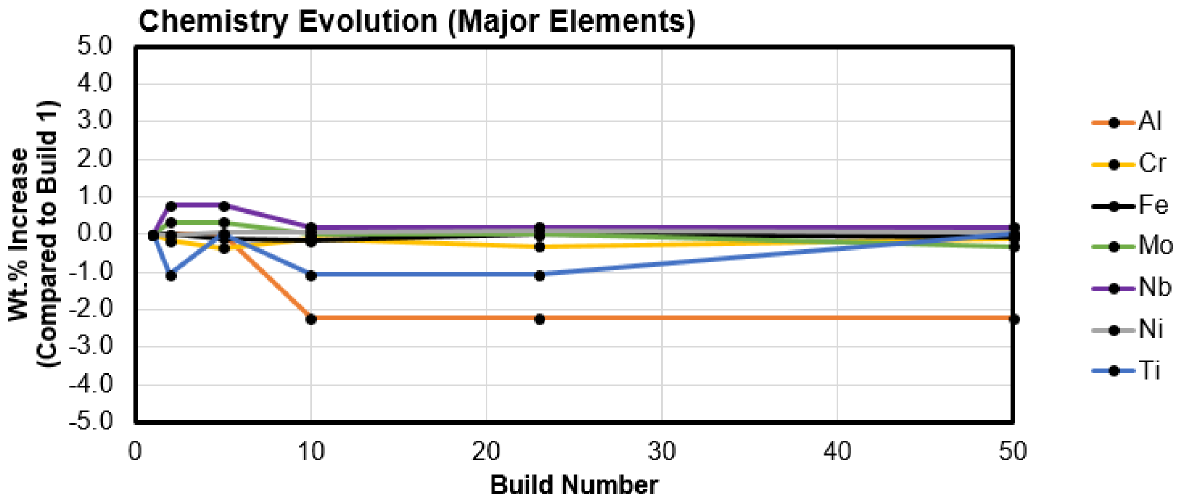


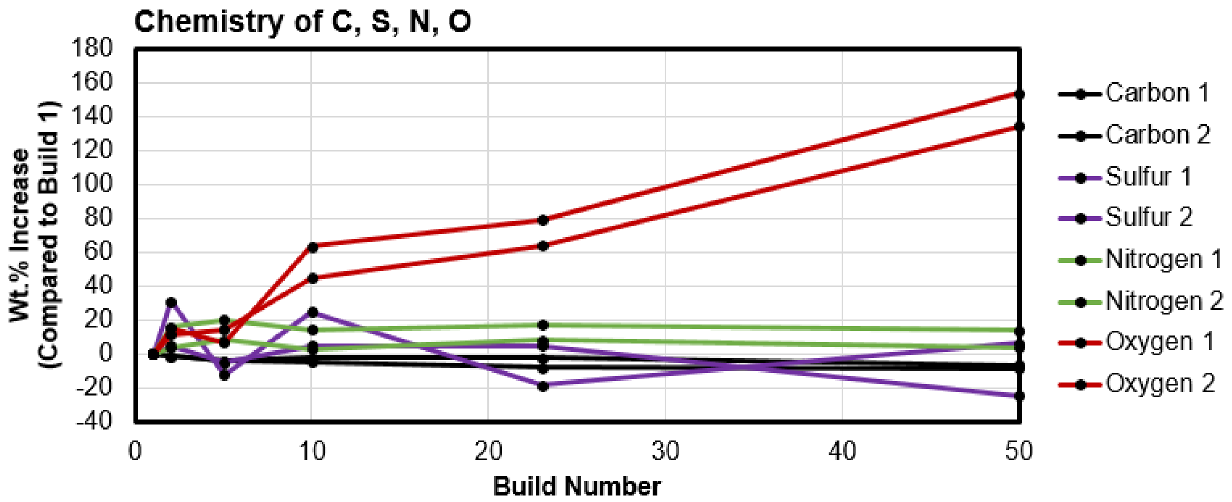




Powder Recyclability: Chemistry

- **Chemistry measured using ICP-AES and combustion based techniques**
- **Significant increase in oxygen from build 1 (virgin powder) to build 50 (220 ppm to 530 ppm)**
- **Other elements quite stable**

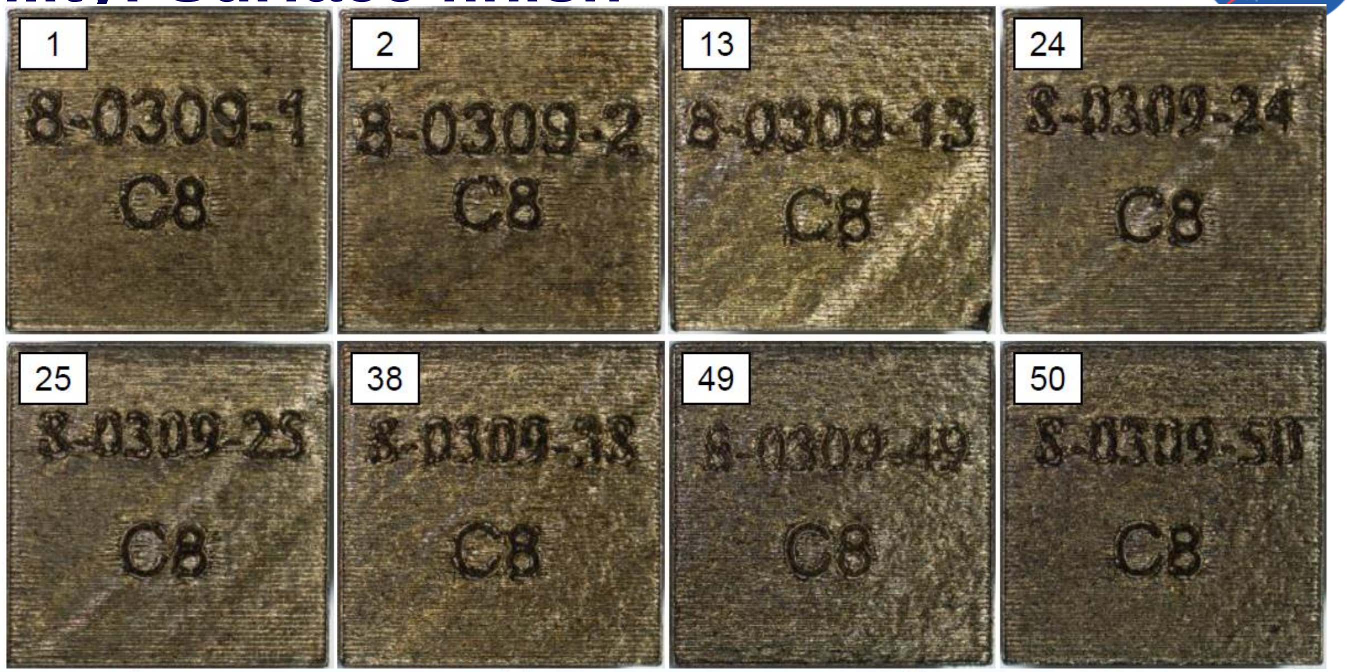


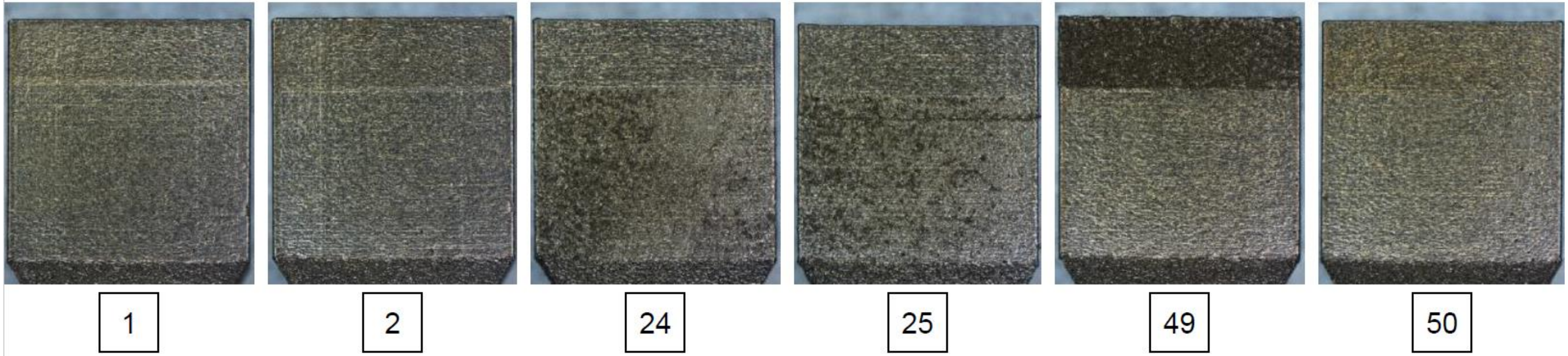


Build	Al	Cr	Fe	Mo	Nb	Ni	Ti	Si	C1	C2	S1	S2	N1	N2	**O1**	**O2**
1	0.45	18.85	18.06	3.09	5.11	53.38	0.94	0.0240	0.0237	0.0020	0.0016	0.0263	0.0263	0.0285	**0.0215**	**0.0220**
2	0.45	18.82	18.06	3.10	5.15	53.37	0.93	0.0237	0.0234	0.0021	0.0021	0.0305	0.0305	0.0296	**0.0248**	**0.0245**
5	0.45	18.78	18.04	3.10	5.15	53.41	0.94	0.0230	0.0227	0.0019	0.0014	0.0316	0.0316	0.0308	**0.0229**	**0.0252**
10	0.44	18.83	18.03	3.09	5.12	53.42	0.93	0.0229	0.0231	0.0021	0.0020	0.0301	0.0301	0.0293	**0.0351**	**0.0319**
23	0.44	18.79	18.06	3.09	5.12	53.44	0.93	0.0221	0.0231	0.0021	0.0013	0.0308	0.0308	0.0308	**0.0385**	**0.0361**
50	0.44	18.83	18.05	3.08	5.12	53.40	0.94	0.0220	0.0221	0.0015	0.0017	0.0299	0.0299	0.0296	**0.0546**	**0.0516**

Powder Recyclability: Surface finish

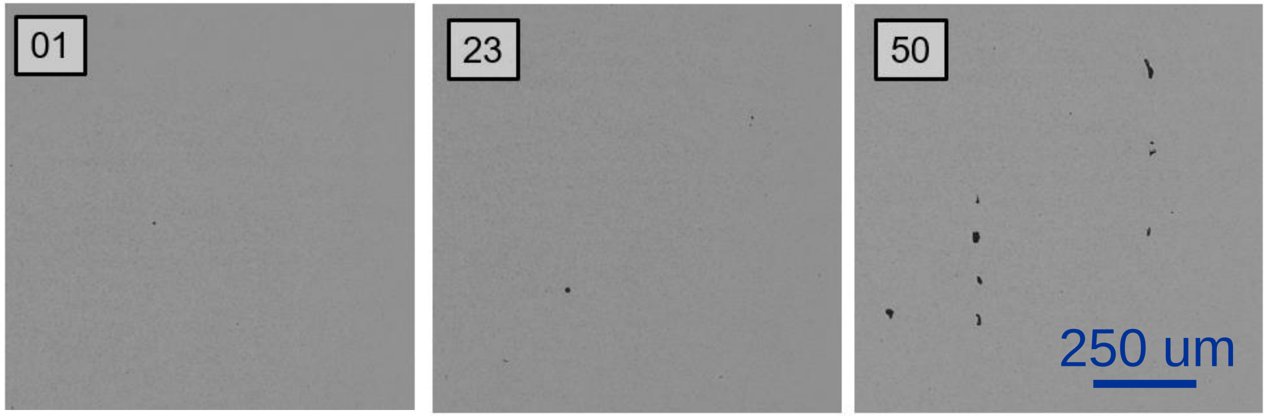
- **Surface finish somewhat worse with notable increase in oxidation**
- **Surface roughness anomalies seem unrelated to extent of recycling**

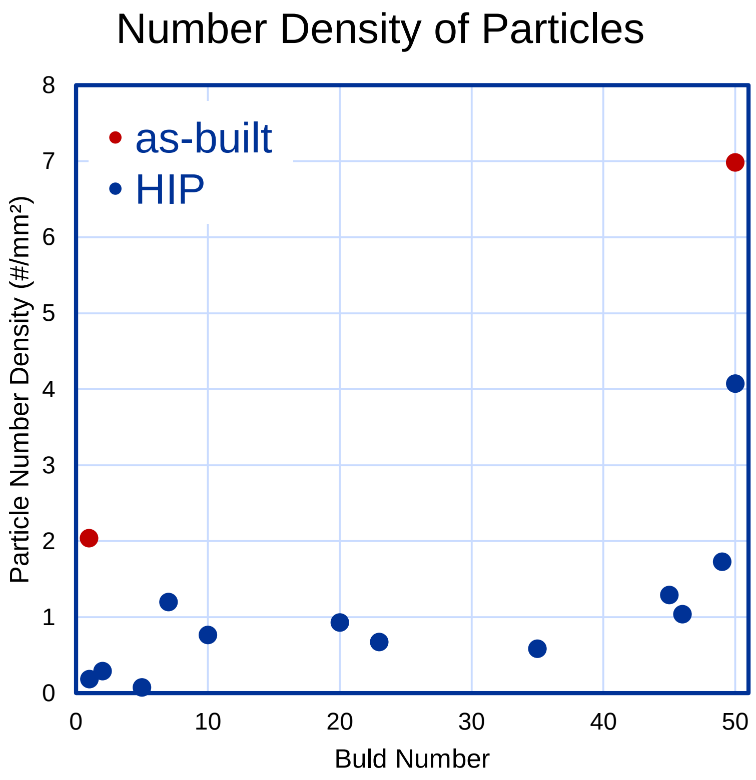


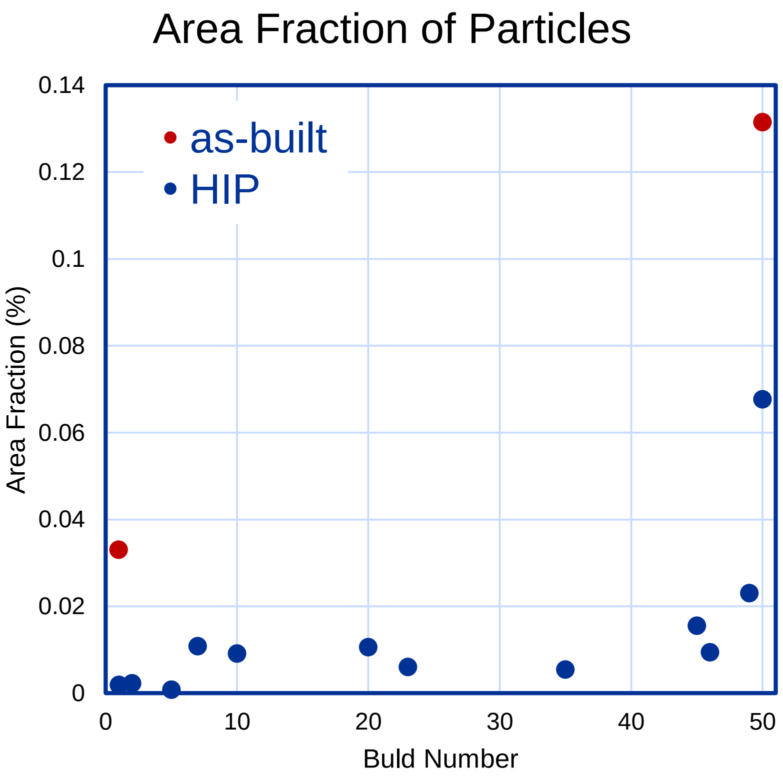


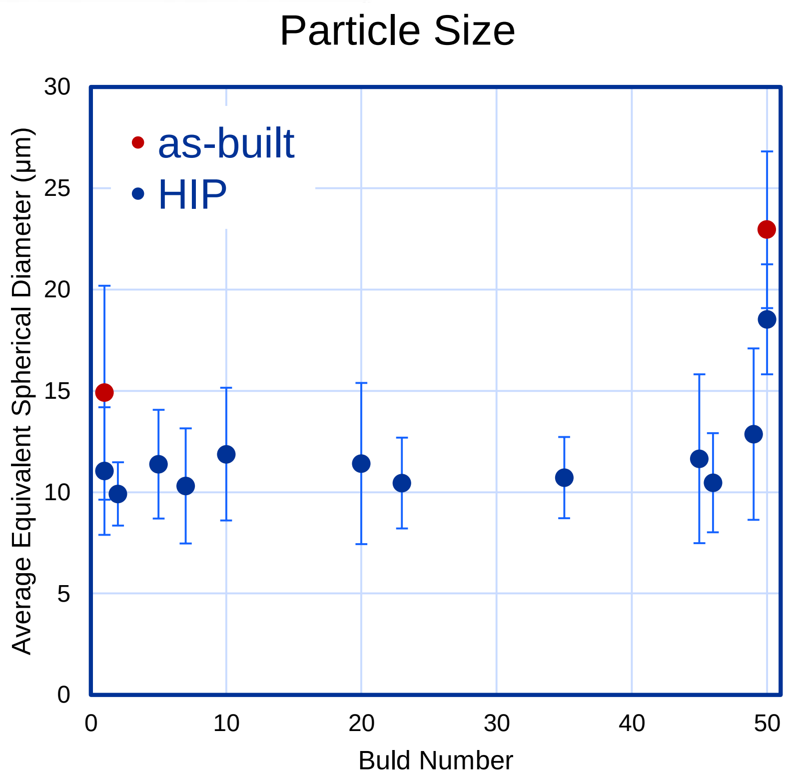
Powder Recyclability: Defects

- **Increase in defects found internally, though a lot of scatter**
- **For quantitative analysis particles with area<50μm2 (diameter<8μm) ignored**



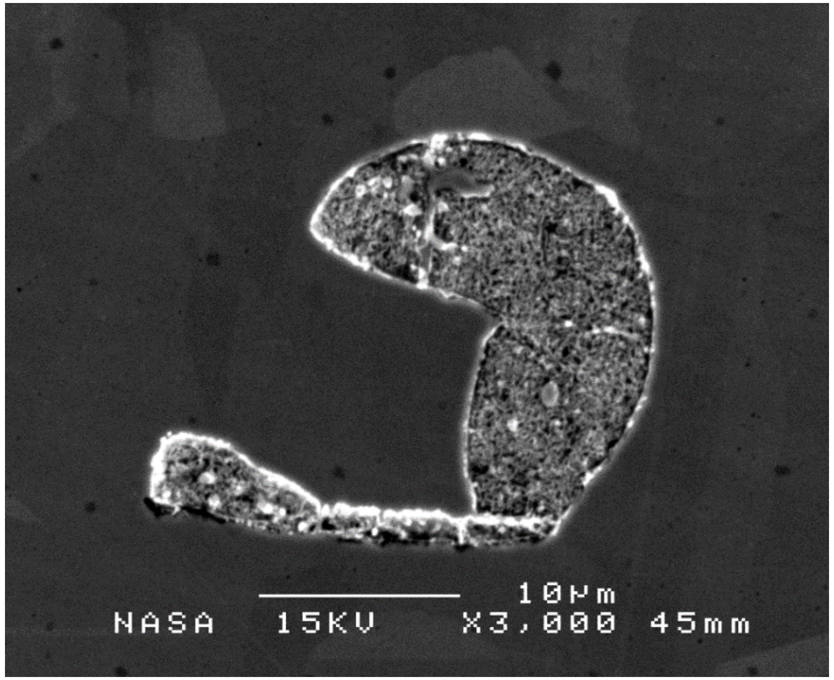


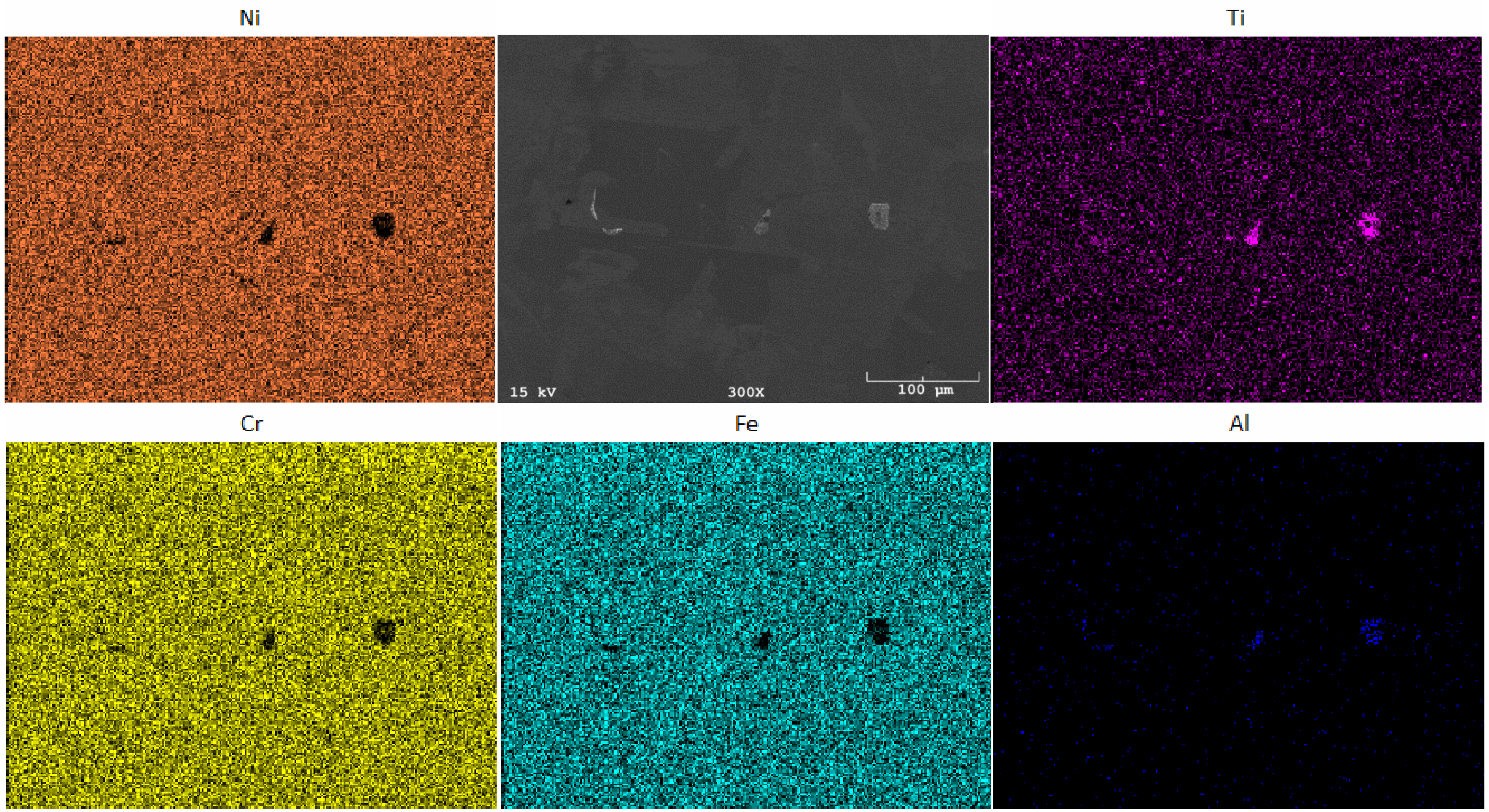




Powder Recyclability: Defects

- **A lot of defects confirmed to be Al/Ti oxides**

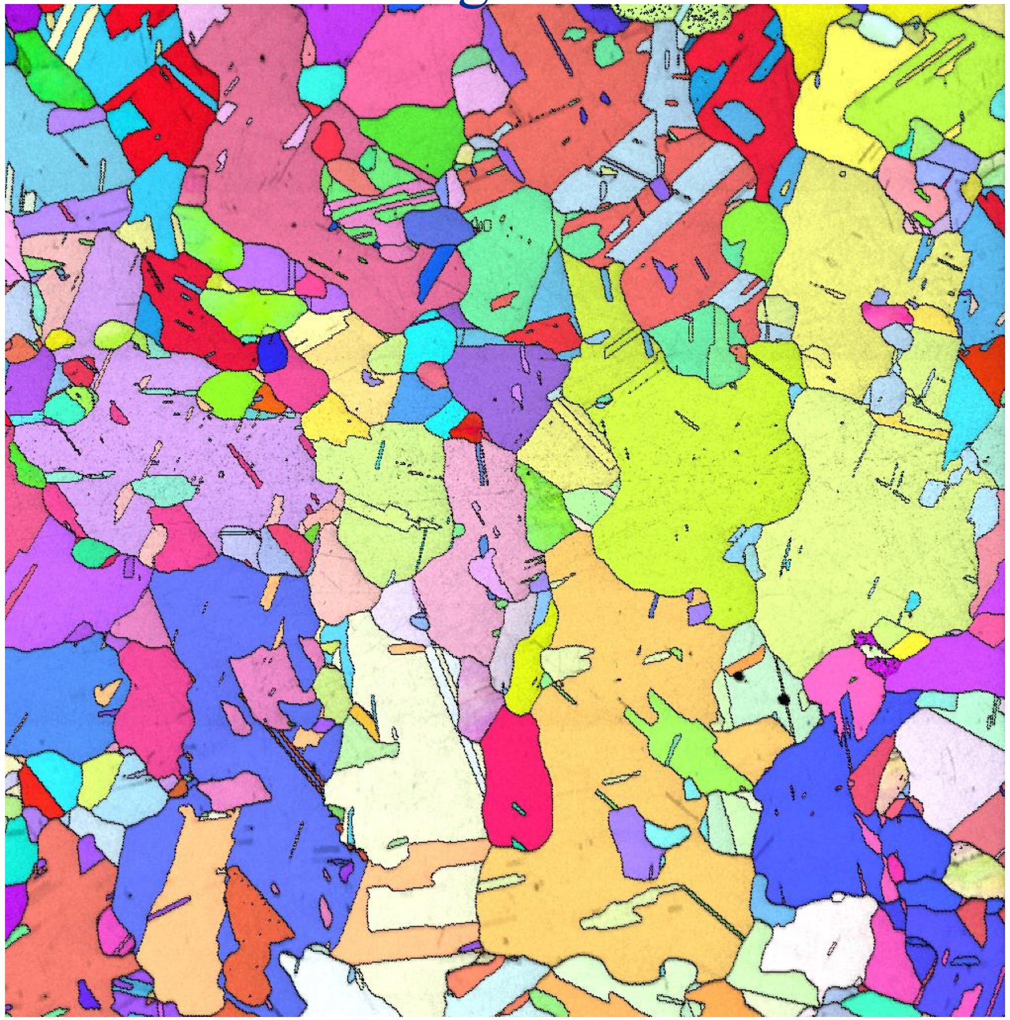




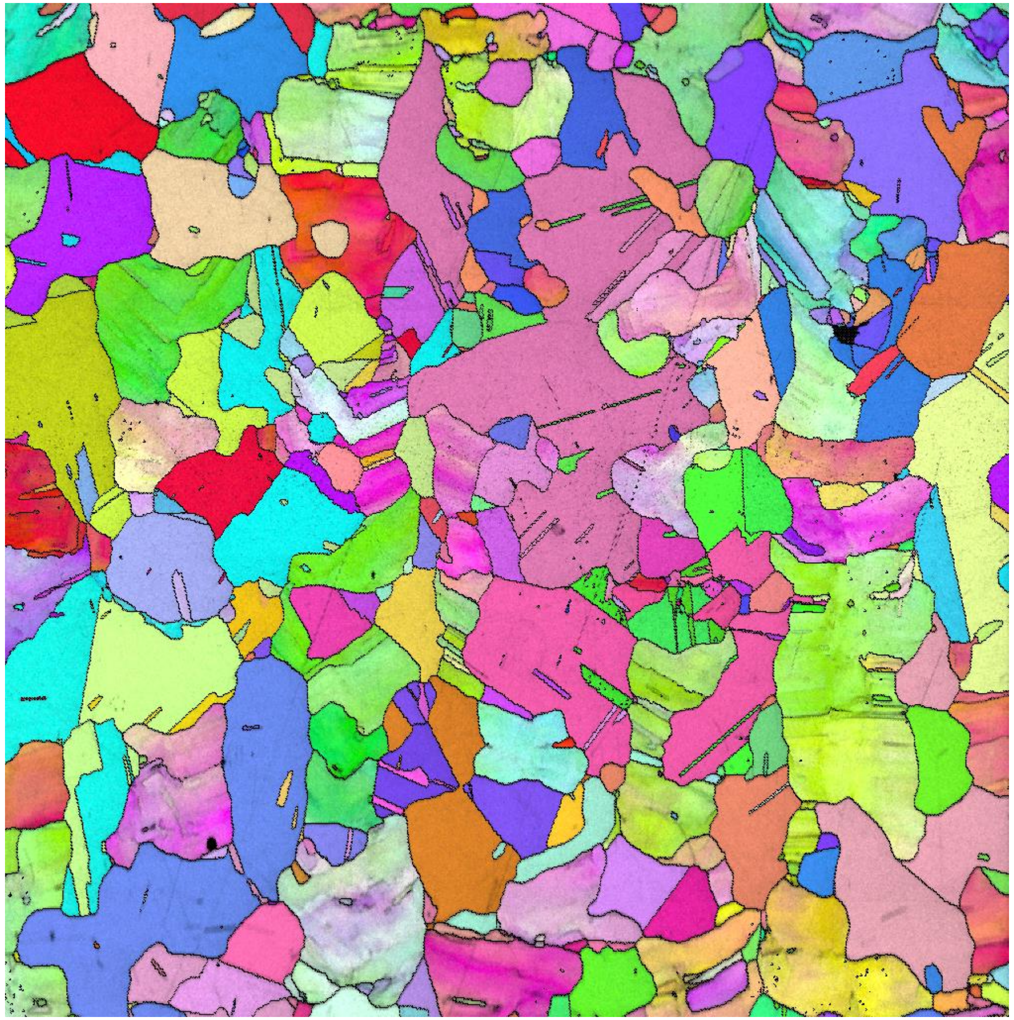
Powder Recyclability: Microstructure

Build direction

Virgin Powder



50th build



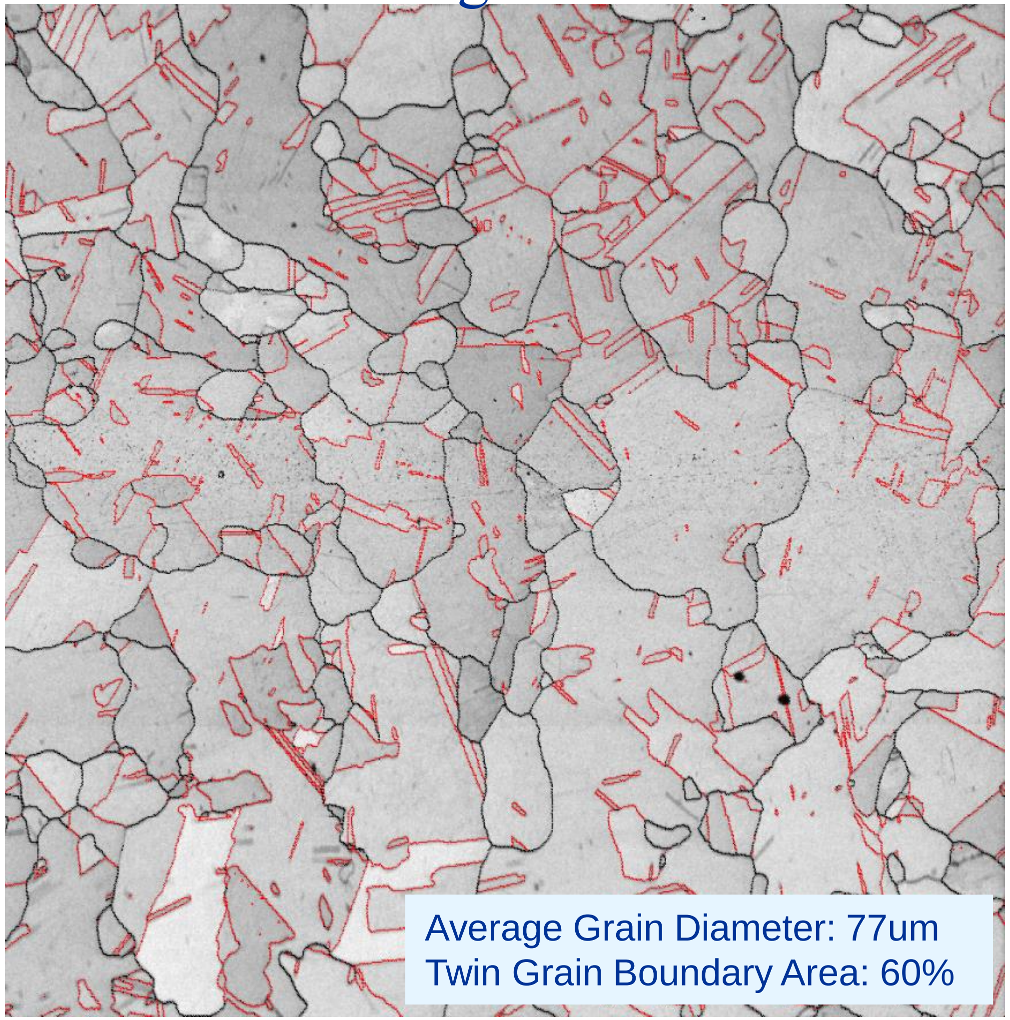
100 um

Overlaying Image Quality map, along with grain boundaries in black

Powder Recyclability: Microstructure

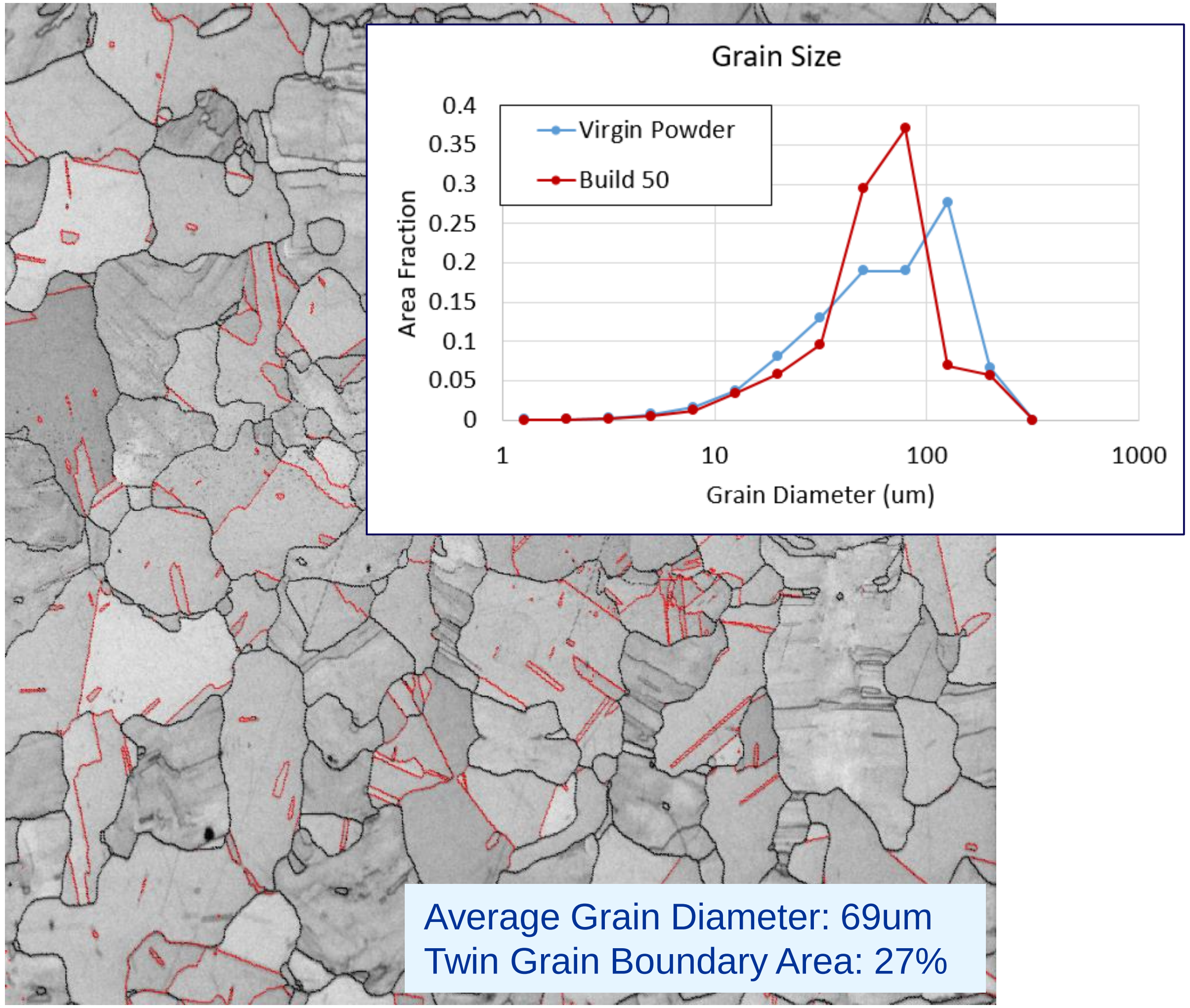
Build direction

Virgin Powder



Average Grain Diameter: 77um
Twin Grain Boundary Area: 60%

50^{th} build



Average Grain Diameter: 69um
Twin Grain Boundary Area: 27%

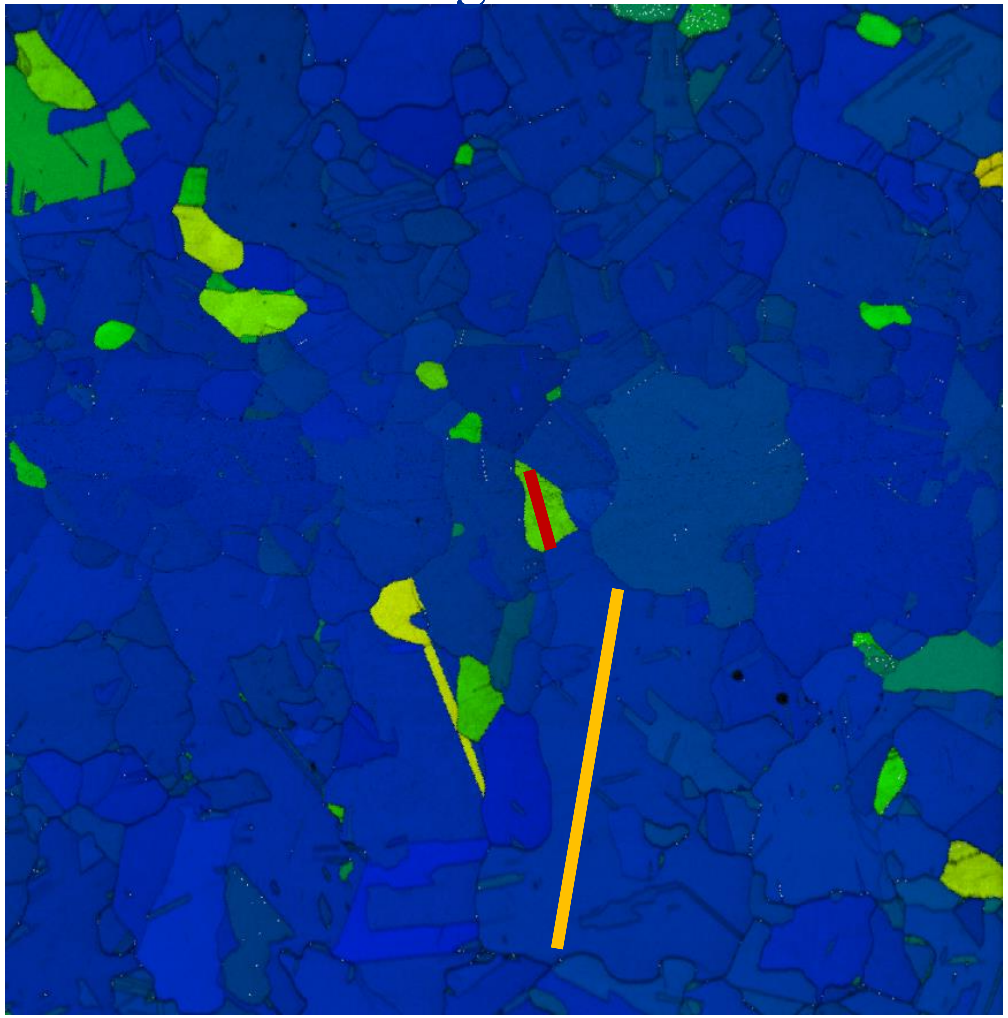
100 um

Grain sizes are similar. Virgin Powder slightly larger, but significantly more twins

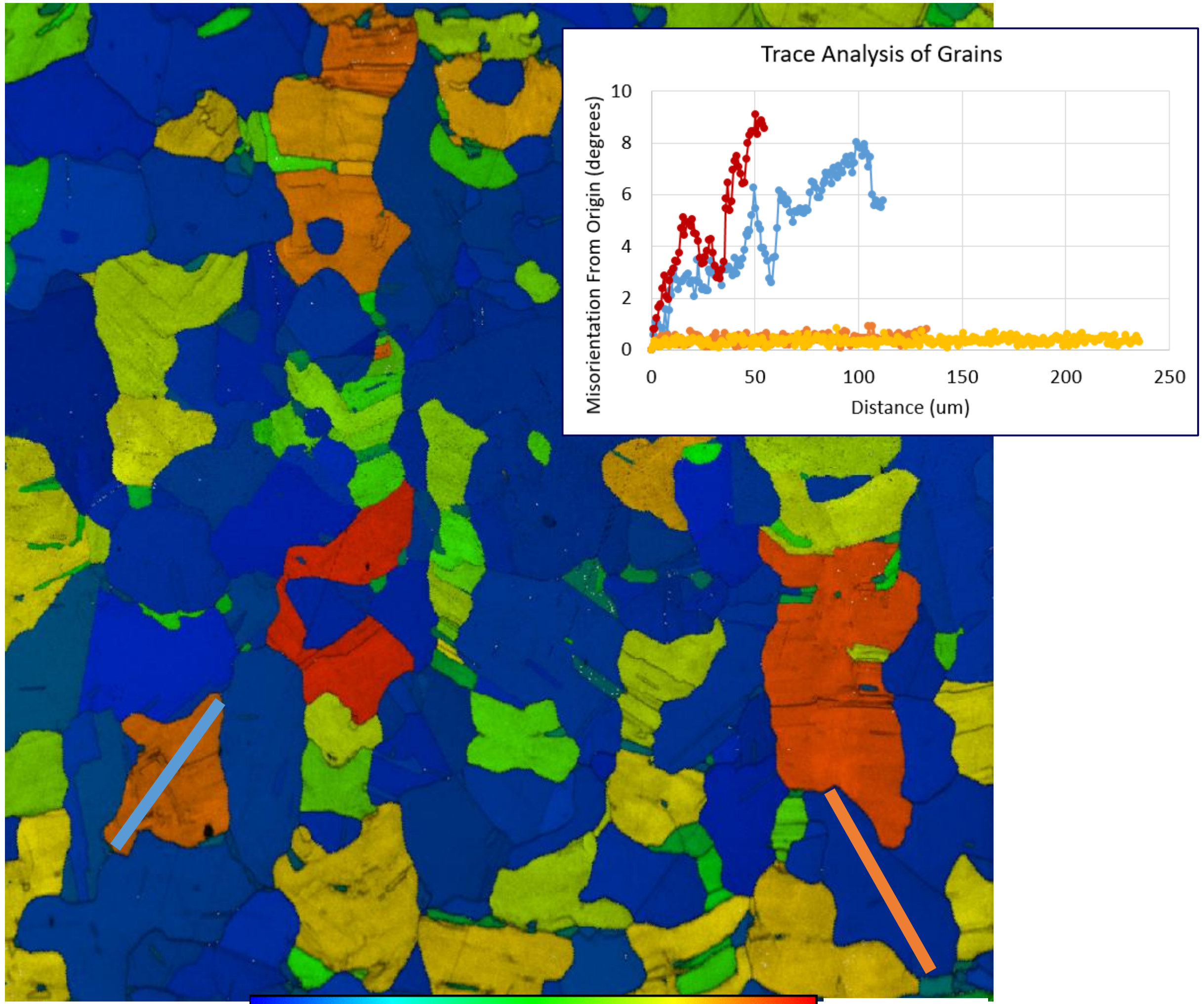
Powder Recyclability: Microstructure

Build direction

Virgin Powder



50th build



Grain Orientation Spread shows the orientation variation within a grain. Un-recrystallized (as-built) grains show high GOS. The recycled powder (with high oxide volume fraction) recrystallizes poorly.

Powder Recyclability: Summary

- Mechanical testing results soon to come (tensile and fatigue)
- Both the powder and printed parts pick up Oxygen with increased reuse.
- This manifests in the surface finish, and in ~10 um oxide particles in the bulk.
- Significant impacts on microstructure and extent of recrystallization during HIP + HT
- Reused powder leads to less recrystallized microstructures with fewer twin boundaries.

SLM 718 Feedstock Variability Project – Intraagency Team: Supplier-to-supplier comparison 18 powders and 194 variables measured

GRC
GLENN RESEARCH CENTER
at Lewis Field

MARSHALL SPACE FLIGHT CENTER

Johnson Space Center
White Sands Test Facility

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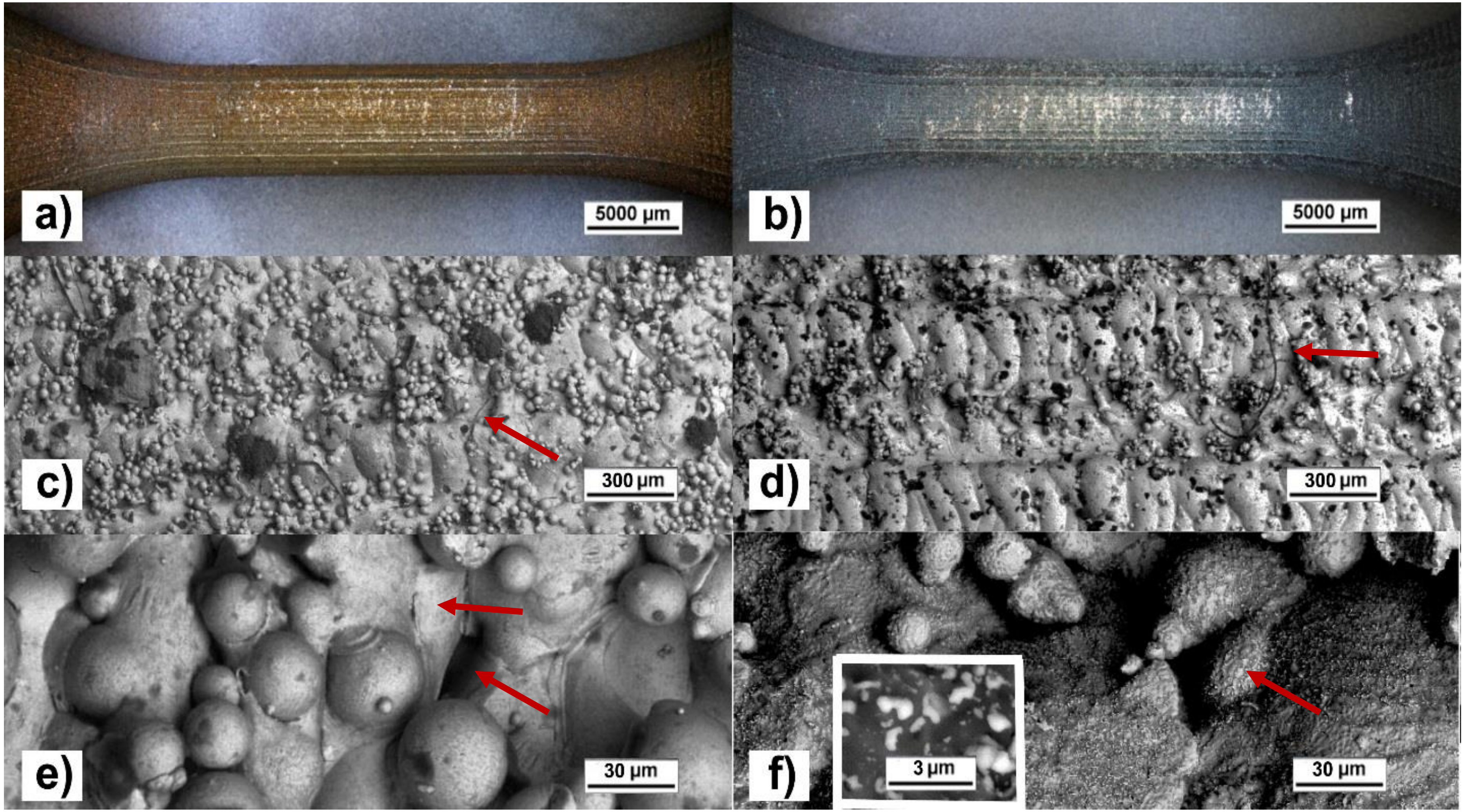
As Fabricated Surface Finish



Evidence of pre-existing flaws, surface cracking