Relationship of Powder Feedstock Variability to Microstructure and Defects in Selective Laser Melted Alloy 718

T.M. Smith[1], M.F. Kloesel[2], C.K. Sudbrack[1]

[1] NASA Glenn Research Center, Materials and Structures Division, 21000 Brookpark Road, Cleveland, OH 44135 USA

[2] Universities Space Research Association, NASA Glenn Research Center, 21000 Brookpark Road, Cleveland, OH 44135 USA

Motivation for Study

- Intra-agency effort to develop a **NASA standard** for safety-critical selective laser melting (SLM) parts of Alloy 718 for structural applications in NASA space flight vehicles
- Part of a comprehensive industry **lot-to-lot comparison** to understand and identify the various feedstock controls important to SLM Alloy 718 hardware

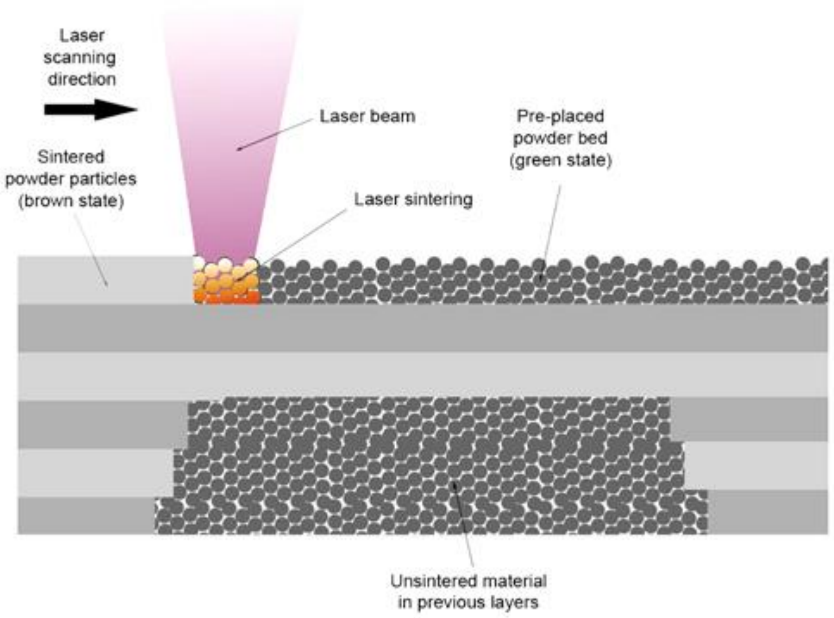
Objectives

- Improve understanding of how powder variability affects microstructure and defects in as-fabricated and heat treated builds.

Background

- The use of powder feedstock is well established within powder metallurgy (PM) industries:
  - Powder feedstock is densely packed into a mold and solidified through sintering and/or hot isostatic pressing (HIPing)
  - Ex. disk superalloys, magnetic materials, high speed steels

- Use of feedstock in metals additive manufacturing (AM) is much less established:
  - Powder feedstock is sintered layer-by-layer by a laser or electron beam.
  - AM is arguably more sensitive to feedstock variability than PM.



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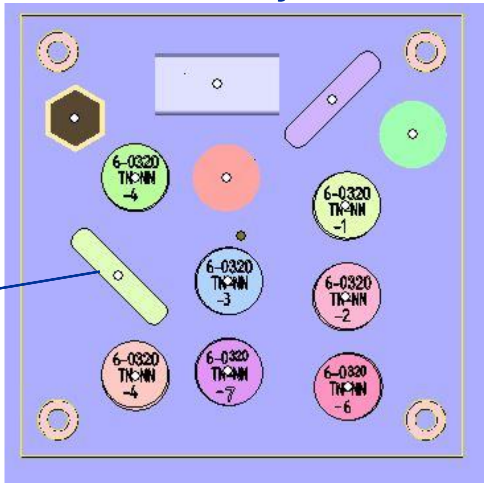
Approach

- Off the shelf industry survey of:
  - 13 powders procured in 50lb lots from 8 vendors (A1-H1)
    - 0-22um / 10-45um / 15-45um / 45-90um*
  - 3 powder lots procured in 1000+lb lots from 3 vendors (V1-V3)
  - Sieve and once recycled 3 powder lots (R1-R3)
- 4 inch builds were produced using the same vendor recommended parameters for 718 using a continuous scan - layer thickness: 30µm
- Stress relieved and heat treated to AMS 5664.
- Characterize melt-pool depth, porosity size and area fraction, hardness, average grain size, and precipitate characterization.

Build layout



Powder Chemistry (wt.%)

Sample	Al	Cr	Fe	Mo	Nb	Ni	Ti	C	N (ppm)	O (ppm)
Specs[1]	0.2-0.8	17-21	Bal.	2.8-3.3	4.8-5.5	50-55	0.7-1.2	.08 max	--------	--------
A1	0.40	18.82	18.25	2.96	5.16	53.1	0.88	0.035	325	181
A2	0.51	18.94	19.06	3.03	4.80	52.7	0.81	0.024	87	240
A3	0.38	18.17	18.19	2.94	5.20	53.6	0.98	0.028	331	182
B1	0.47	19.00	19.03	3.04	5.17	52.4	0.86	0.005	25	158
C1	0.57	17.45	18.76	2.97	4.95	54.3	0.84	0.039	1395	109
D1	0.48	19.02	18.97	3.04	4.91	52.4	0.92	0.033	122	165
E1	0.09	17.72	21.49	2.93	4.86	51.2	0.91	0.096	1220	220
F1	0.35	18.25	18.19	2.97	5.11	53.7	0.94	0.033	607	166
G2	0.46	18.78	18.11	2.99	5.00	53.6	0.94	0.036	207	210
G3	0.49	18.77	18.15	3.02	5.08	53.4	0.92	0.039	176	171
G4*	0.48	18.77	18.03	3.03	5.08	53.7	0.90	0.033	199	143
H1	0.36	18.52	19.02	2.96	4.89	53.1	0.88	0.022	562	331
V1(D)	0.50	19.11	18.54	2.96	4.93	52.9	0.82	0.028	331	182
V2(F)	0.39	18.37	18.46	2.97	4.97	53.6	0.92	0.039	370	109
V3(E)	0.71	18.37	19.18	3.01	5.01	52.0	0.90	0.047	2770	231

- E1 is out of AMS 367 718 chemistry

[1] AMS 367 Specifications

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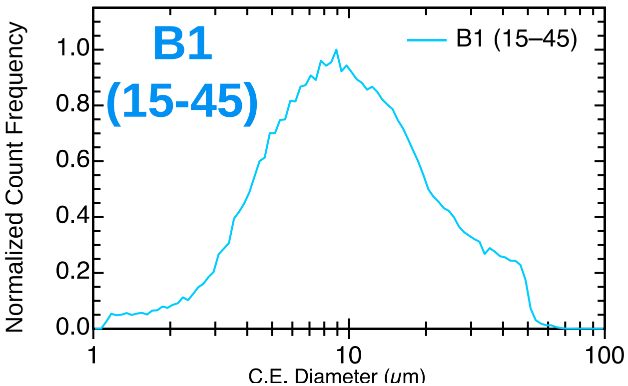
V2-R2(F)	0.39	18.37	18.46	2.97	4.97	53.6	0.92	0.039	370	109
V3-R3(E)	0.71	18.37	19.18	3.01	5.01	52.0	0.90	0.047	2770	231

- E1 is out of AMS 367 718 chemistry
- C1, E1, V3, and R3 were atomized in Nitrogen instead of Argon.

[1] AMS 367 Specifications

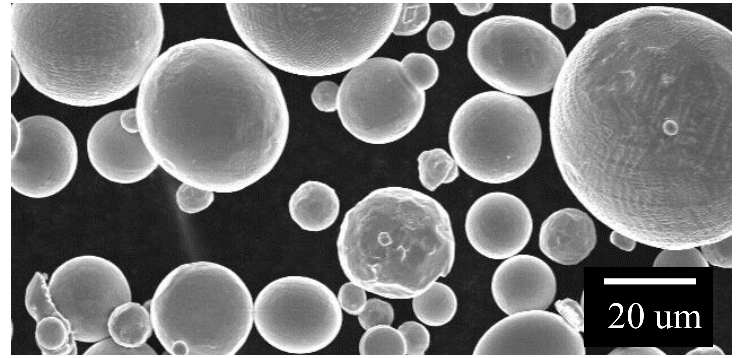
Powder Variability

Particle Size Distributions (PSD)

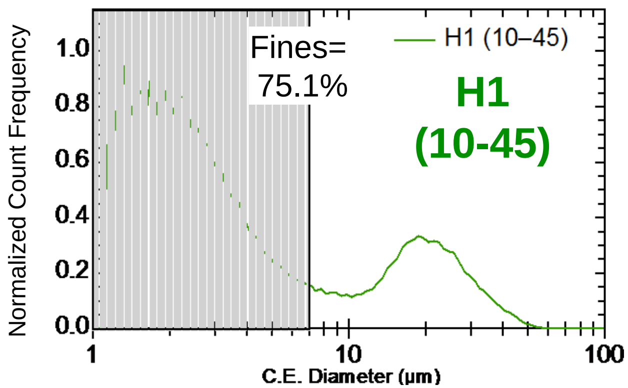


N_{B1}= 170,584

D_{50}	12.8
Mean	12.8
D_{90}-D_{10}	23.1

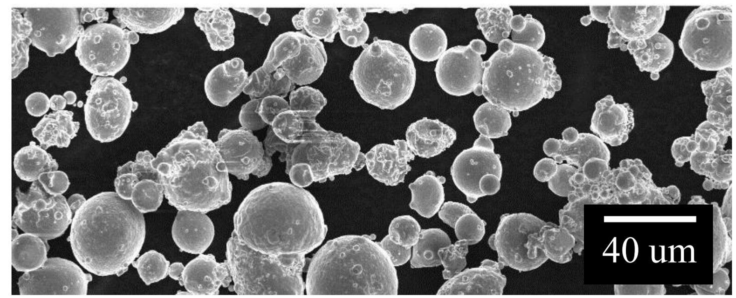


A2

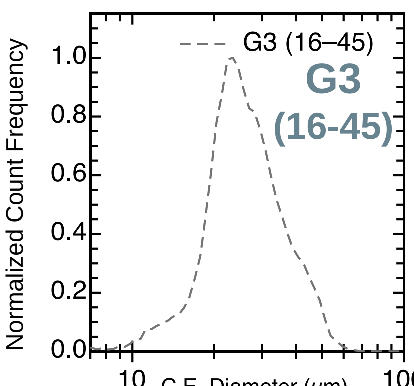


N_{H1}= 63,085

D_{50}	18.7
Mean	20.0
D_{90}-D_{10}	22.9

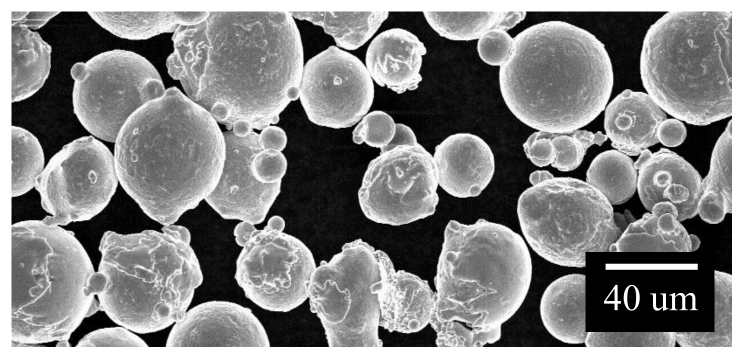


A3
E1
G4



N_{G3}= 34,855

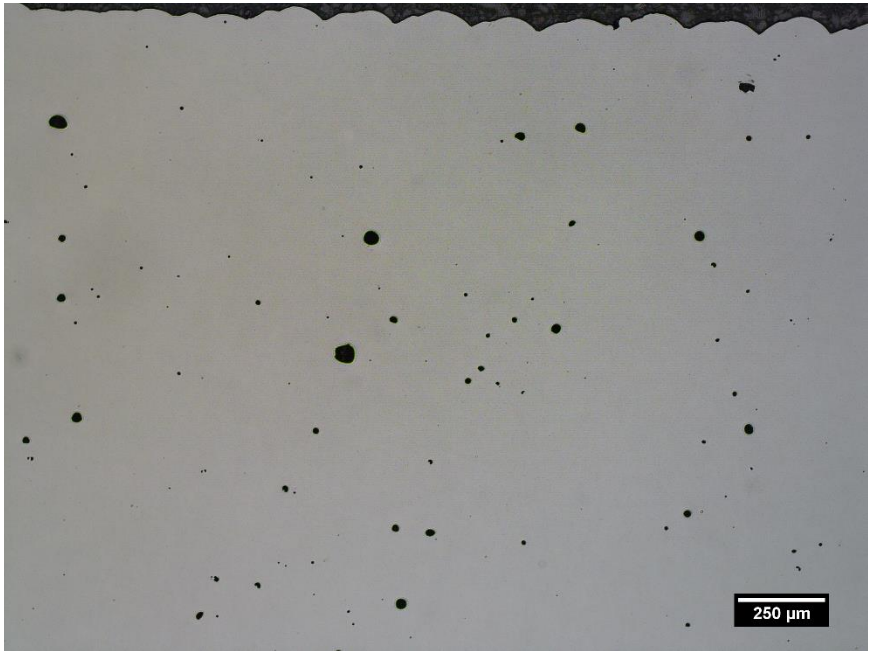
D_{50}	25.4
Mean	27.0
D_{90}-D_{10}	22.1



A1
C1
D1
F1
G2
V1
V2
V3

New As-Fabricated Melt Pool Depth Characterization

R2



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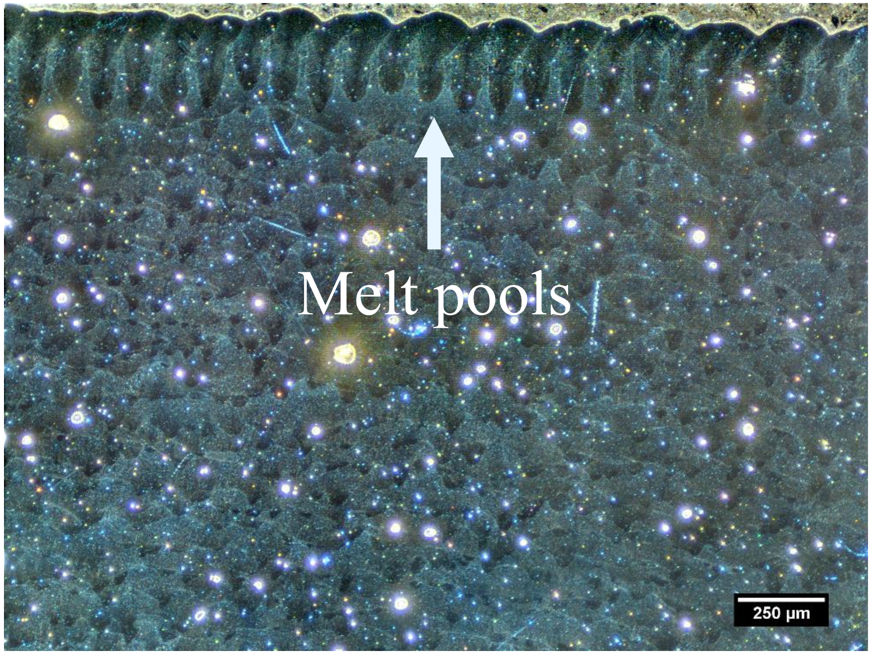
R2

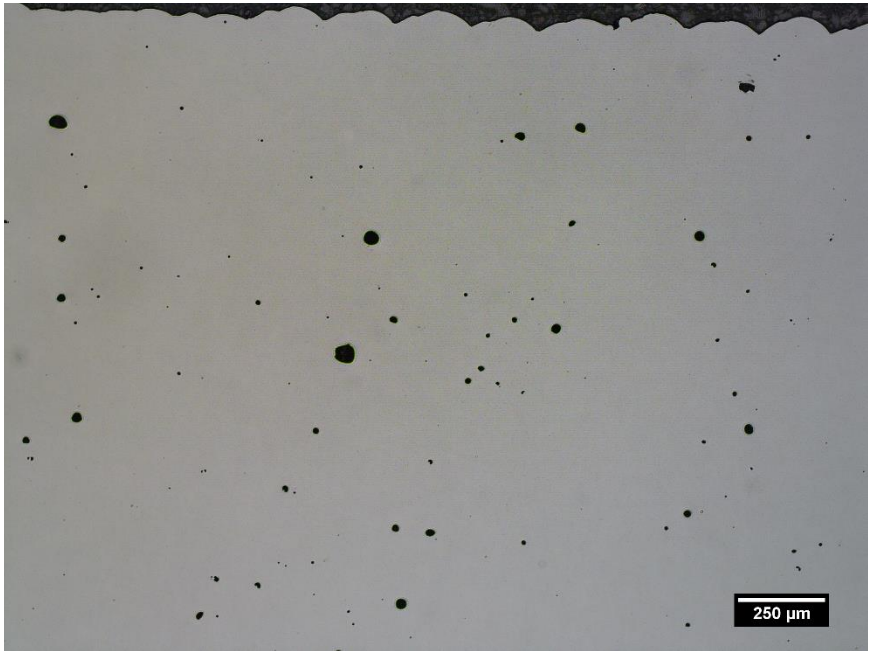


unetched

High dynamic range imaging in DF mode allows for melt pool characterization without any etching step!

New As-Fabricated Melt Pool Depth Characterization

R2



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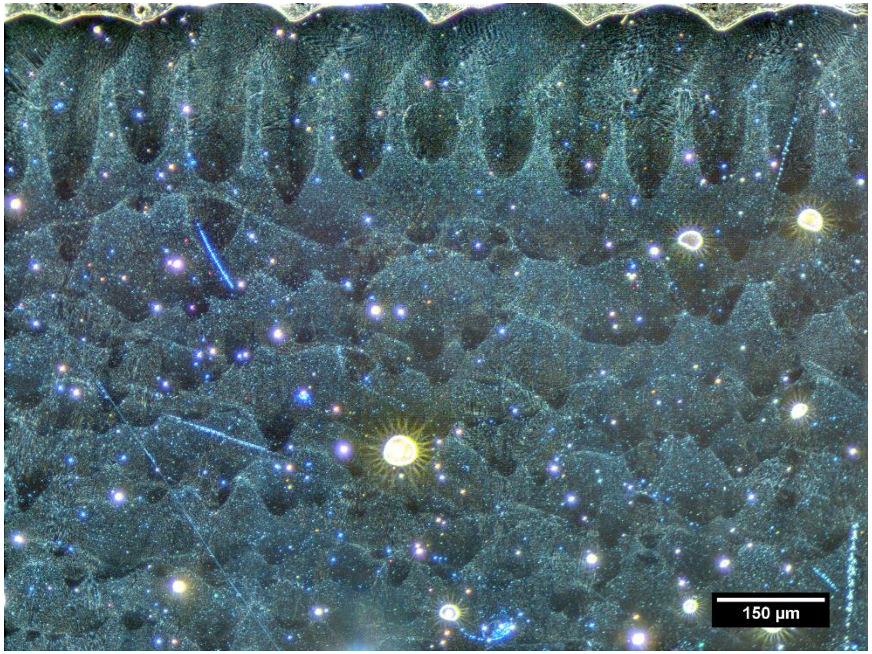
R2



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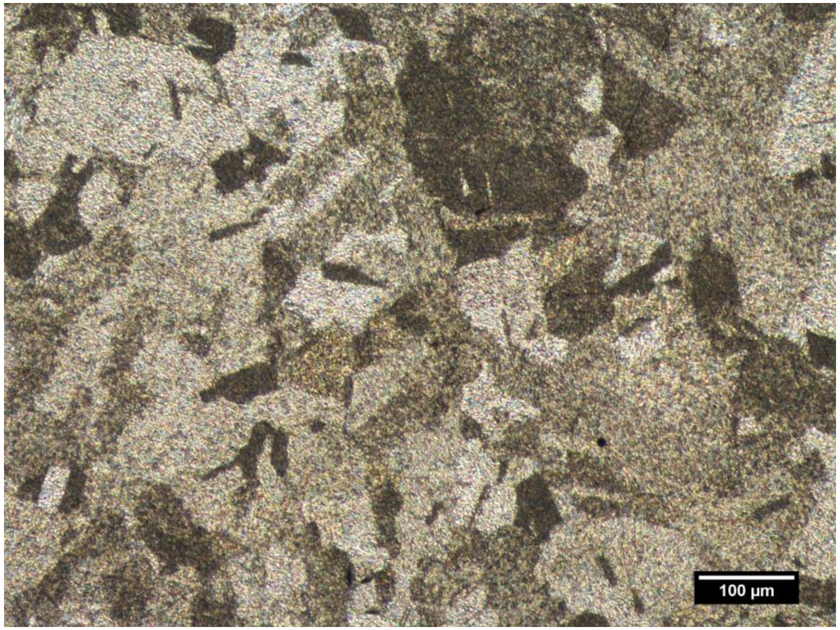
Measure depth of melt pool across surface. Avoid melt pools near edge or markings.

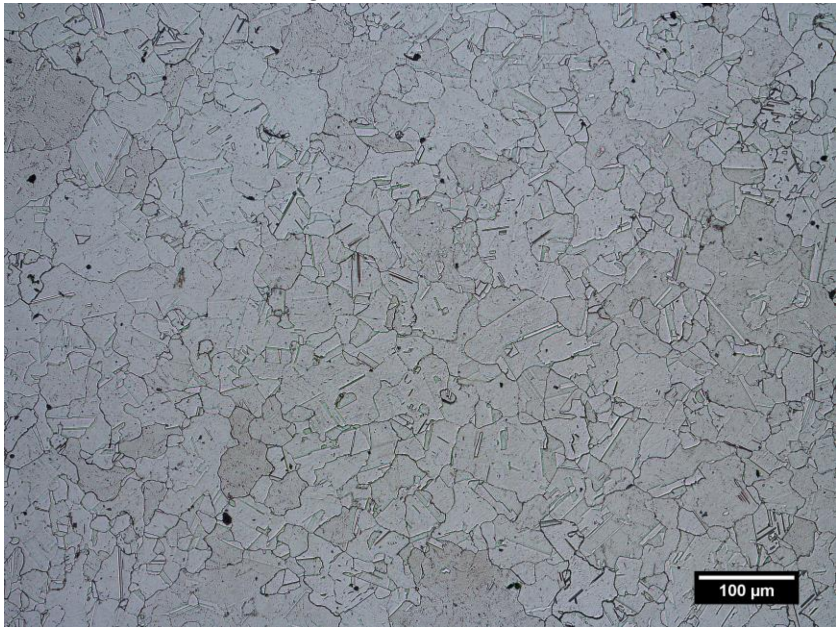
Microstructural Characterization – Grain Size

Kallings #2 etch



- Caused significant intragranular attack / corrosion
- Grain boundaries not highlighted

85mL H_2O/ 45mL HCl / 15mL HNO_3 / 15mL HF etch

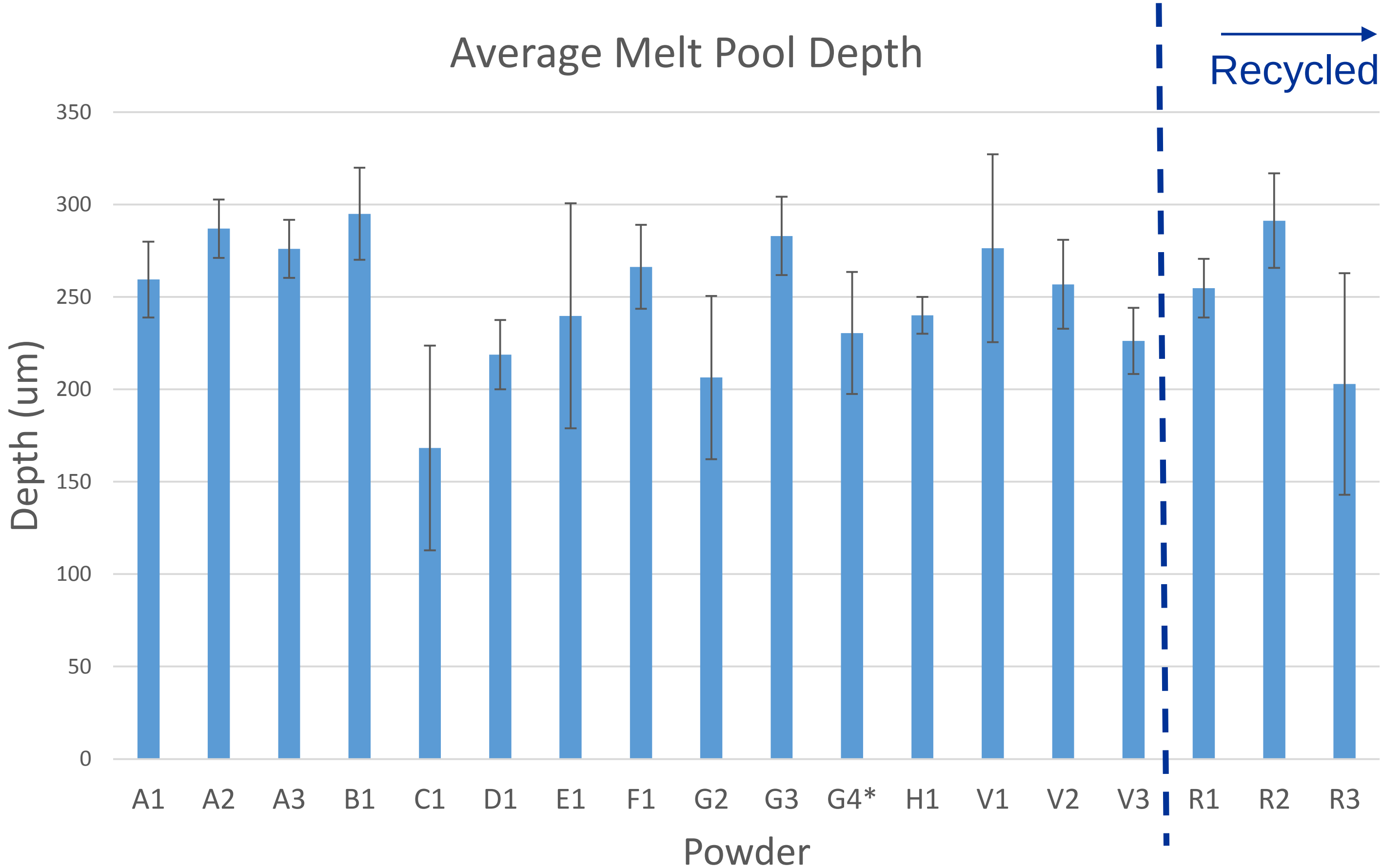


- Minimal intragranular attack / corrosion
- Grain boundaries are highlighted

ASTM circular intercept procedure[1] was used for grain size measurements

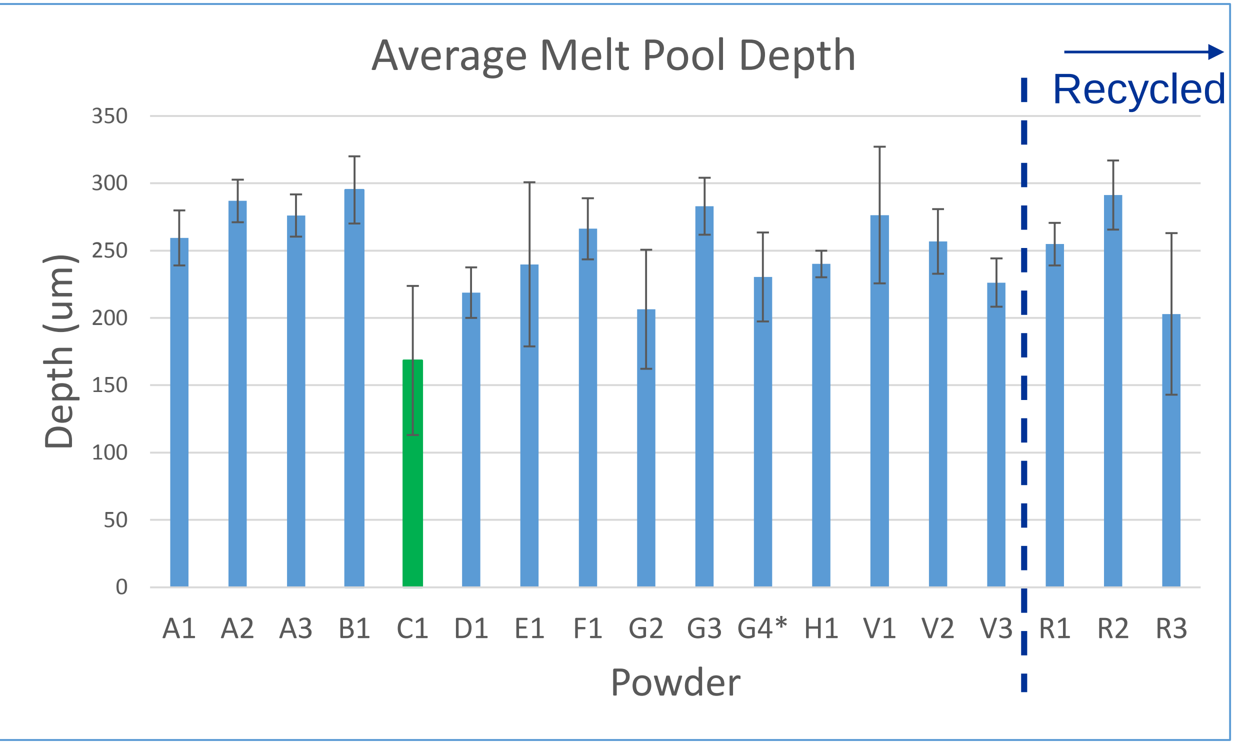
[1] ASTM E112-3

As-Fabricated Melt Pool Depth Characterization



Small differences in melt pool depths between builds.

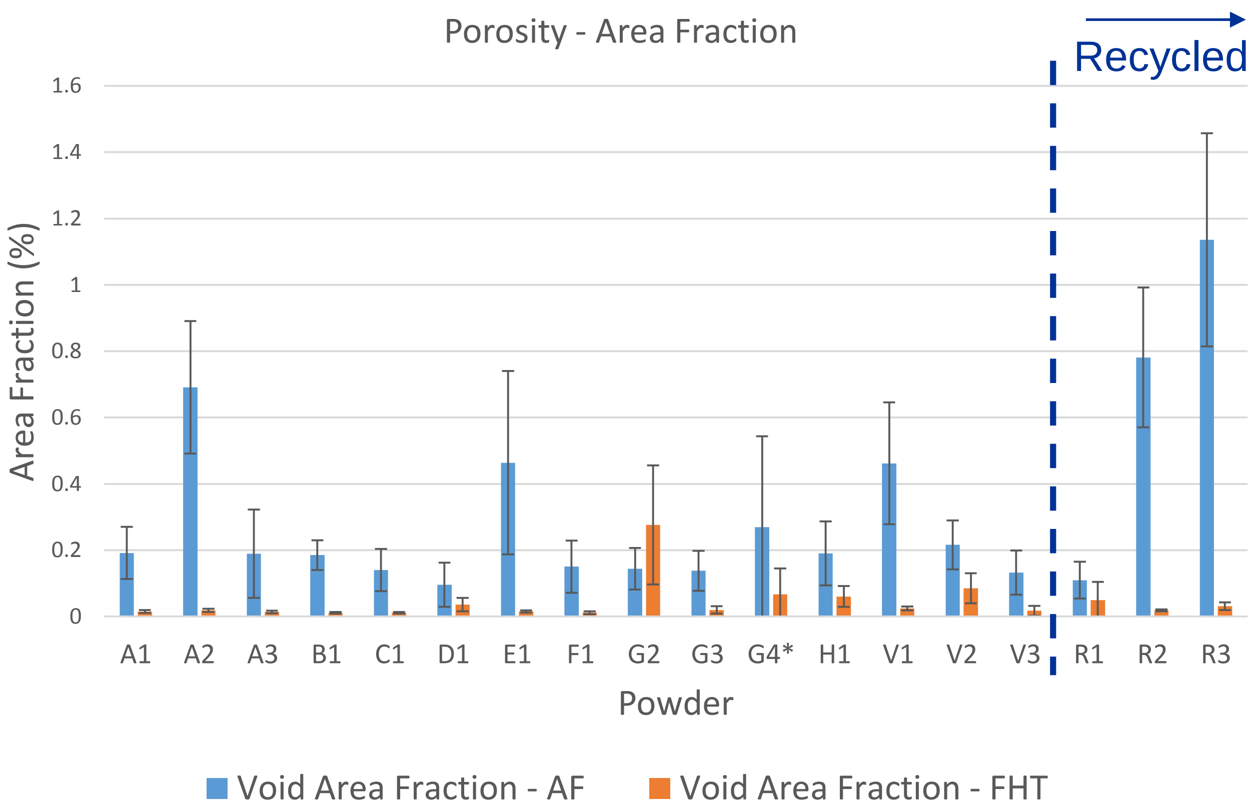
As-Fabricated Melt Pool Depth Characterization



Sample	Rank (Shallow to Deep)
A1	11
A2	16
A3	13
B1	18
C1	1
D1	4
E1	7
F1	12
G2	3
G3	15
G4*	6
H1	8
V1	14
V2	10
V3	5
R1	9
R2	17
R3	2

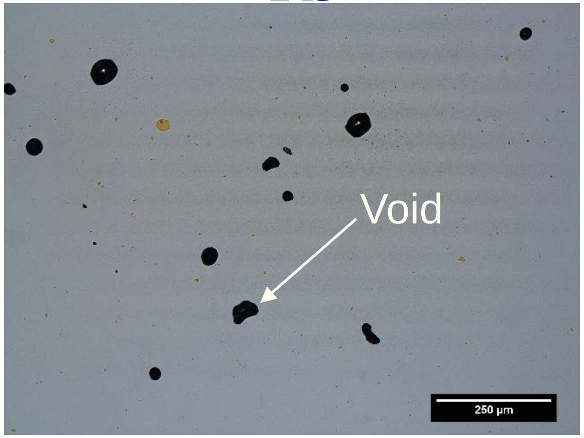
Small differences in melt pool depths between builds.

Porosity Characterization – Area Fraction



5 representative images in the XY plane used for analysis

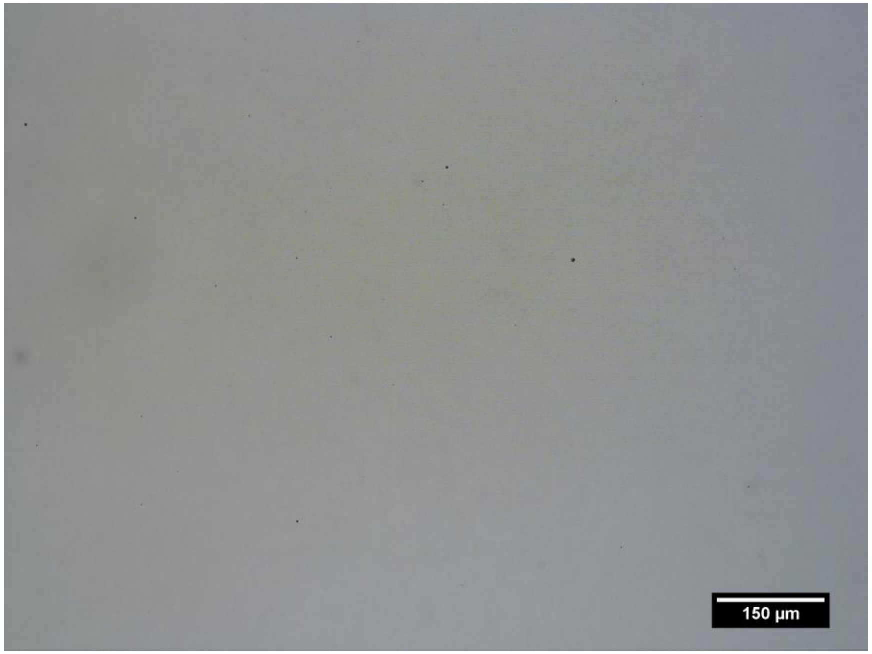
R3

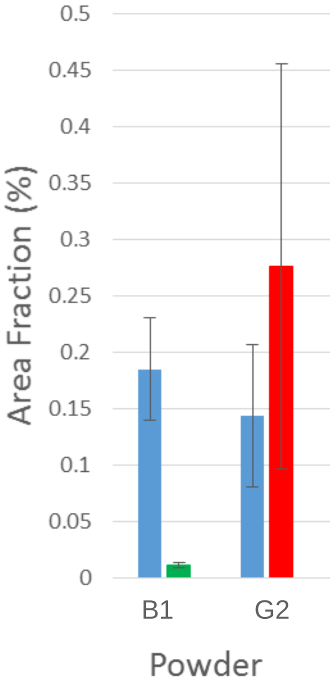


Vendor recommended parameters produced builds with below 1% porosity (exception as-fabricated R3)

Porosity Characterization – Area Fraction

B1

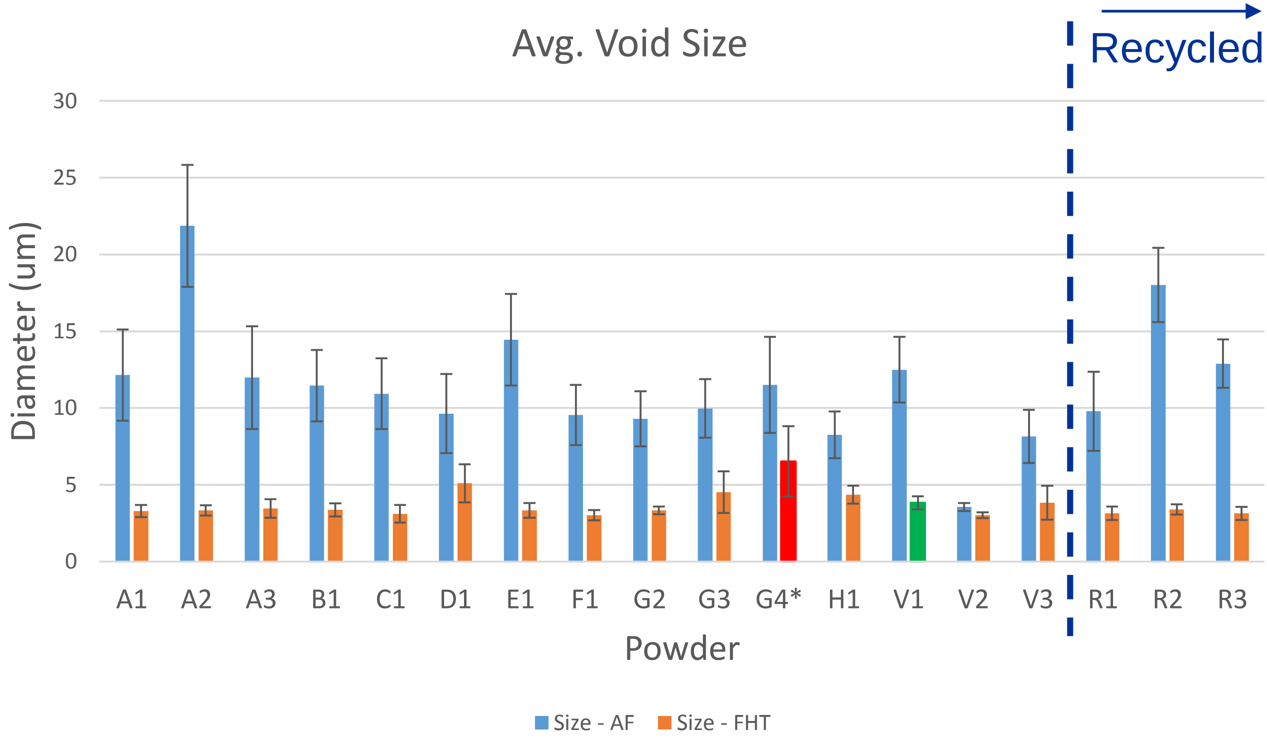




Sample	FHT Void Area Fraction - %	Ranking FHT
A1	0.015	5
A2	0.019	9
A3	0.014	4
B1	0.011	1
C1	0.012	2
D1	0.036	13
E1	0.015	6
F1	0.012	3
G2	0.276	18
G3	0.020	10
G4*	0.067	16
H1	0.060	15
V1	0.024	11
V2	0.085	17
V3	0.017	7
R1	0.049	14
R2	0.019	8
R3	0.031	12

Significant defect improvements between as-fabricated and post-HIP builds (exception G2).

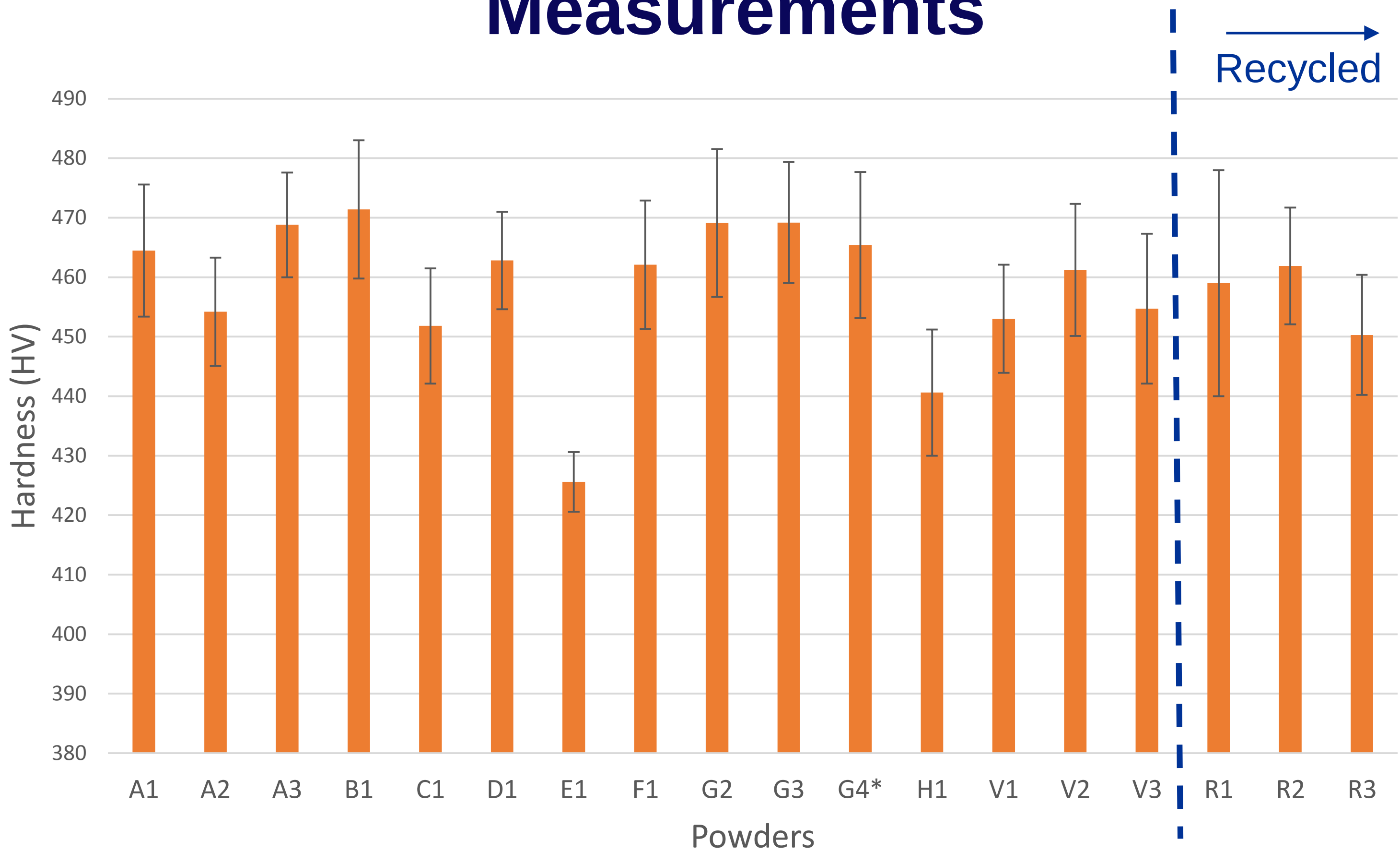
Porosity Characterization - Size



Sample	Avg. Diameter - FHT	Rank
A1	3.29	6
A2	3.33	7
A3	3.46	12
B1	3.37	10
C1	3.11	3
D1	5.09	17
E1	3.33	9
F1	3.02	2
G2	3.33	8
G3	4.52	16
G4*	6.52	18
H1	4.35	15
V1	3.82	13
V2	3.01	1
V3	3.84	14
R1	3.14	5
R2	3.40	11
R3	3.13	4

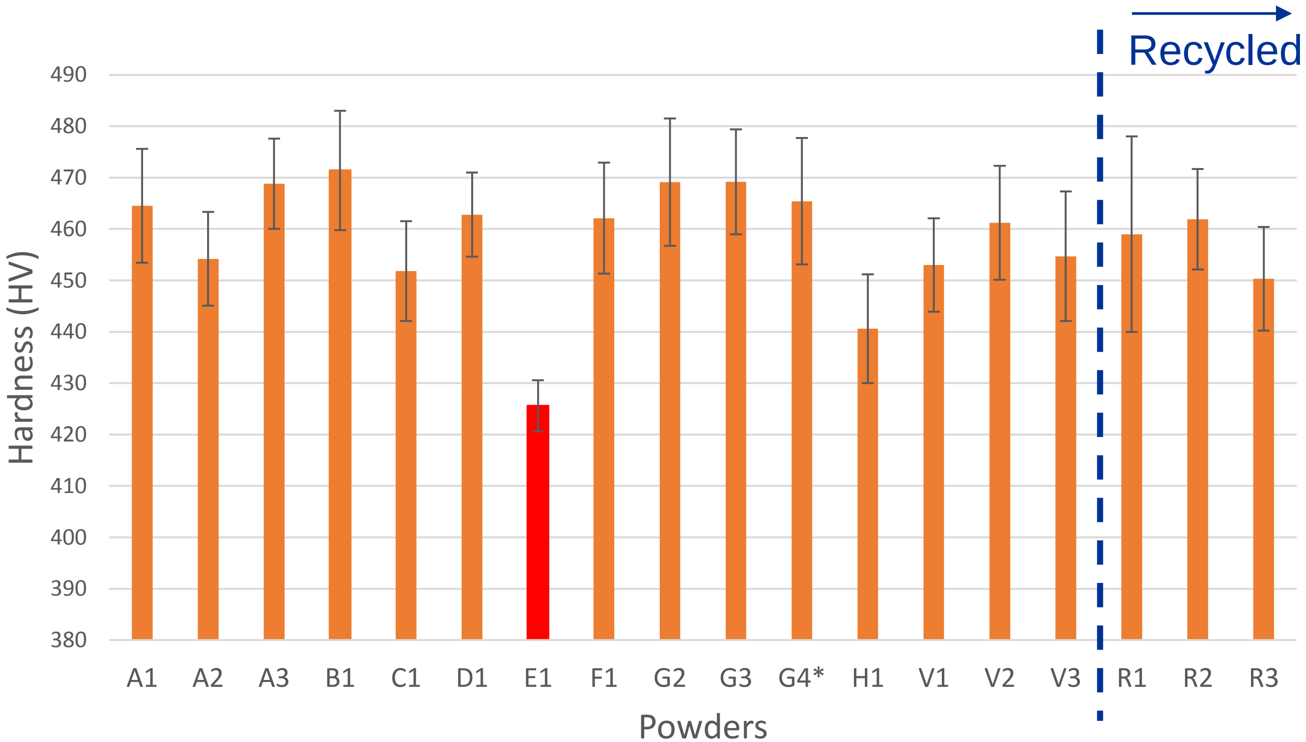
Average void diameters significantly reduced after HIP and heat treatment

Fully Heat Treated Vickers Hardness Measurements



Average of 20 Vickers hardness measurements across polished heat treated surface

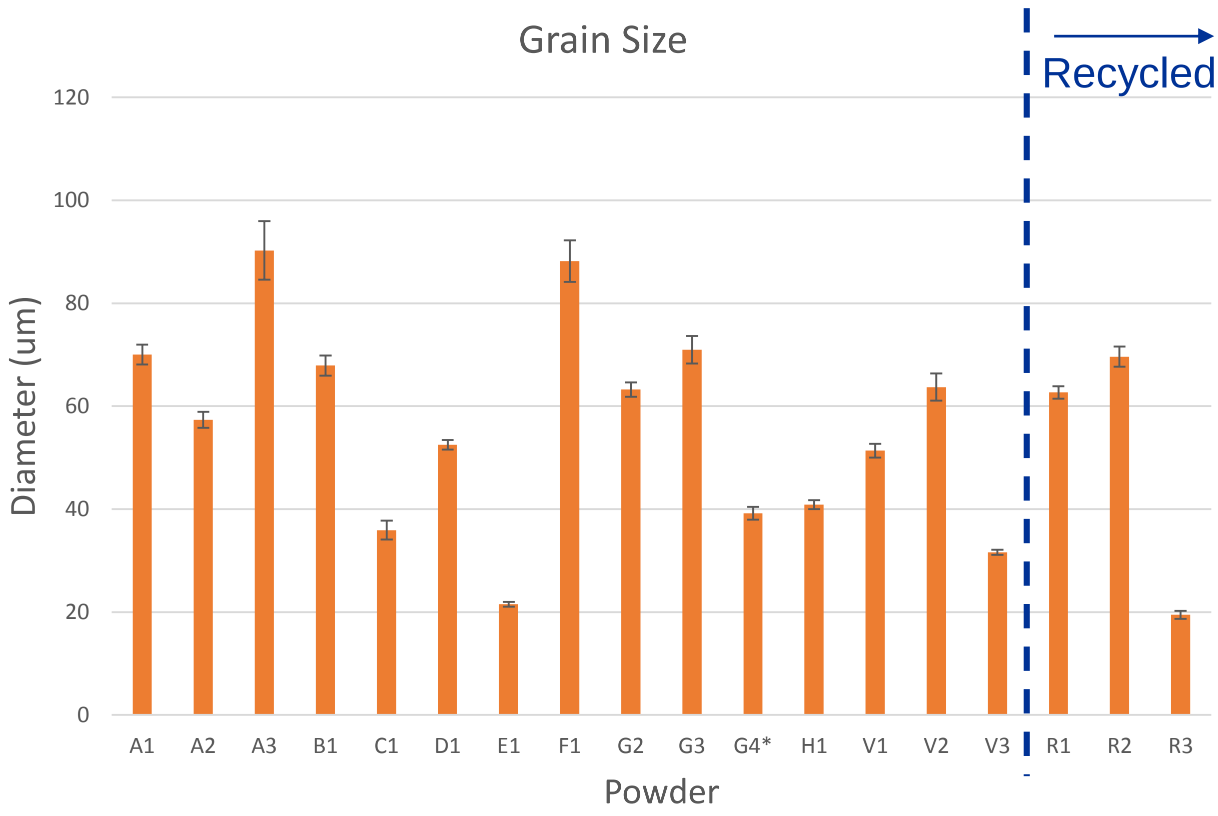
Fully Heat Treated Vickers Hardness Measurements



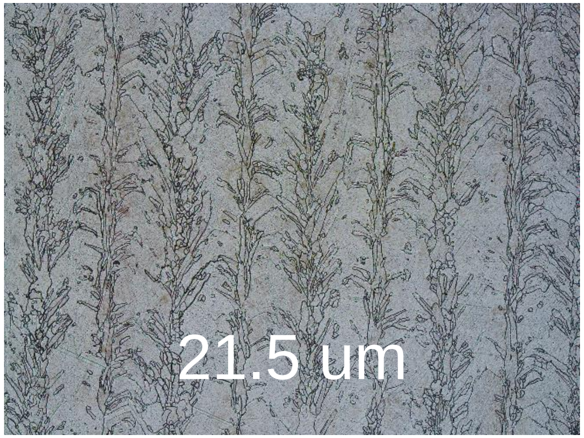
Powder lot	Hardness (HV)	Rank
A1	465	6
A2	454	13
A3	469	4
B1	471	1
C1	452	15
D1	463	7
E1	426	18
F1	462	8
G2	469	3
G3	469	2
G4*	465	5
H1	441	17
V1	453	14
V2	461	10
V3	455	12
R1	459	11
R2	462	9
R3	450	16

-E1 (composition out of AMS 718 specifications) had lowest average hardness.
-Hardness measurements consistent across heat treated builds (between 43-46 Rc hardness – comparable to wrought IN718).

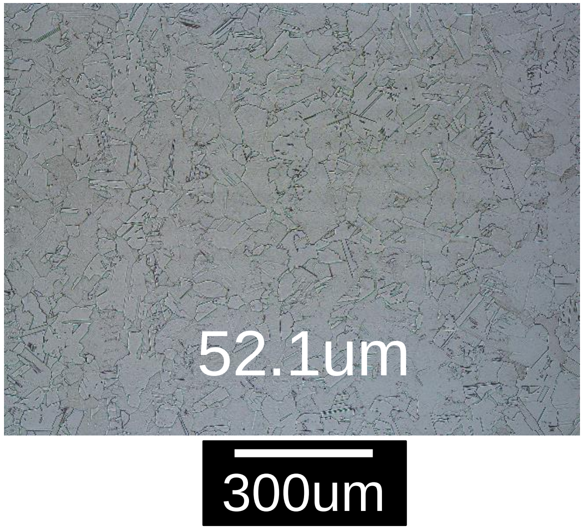
Fully Heat Treated Grain Size Measurements



E1

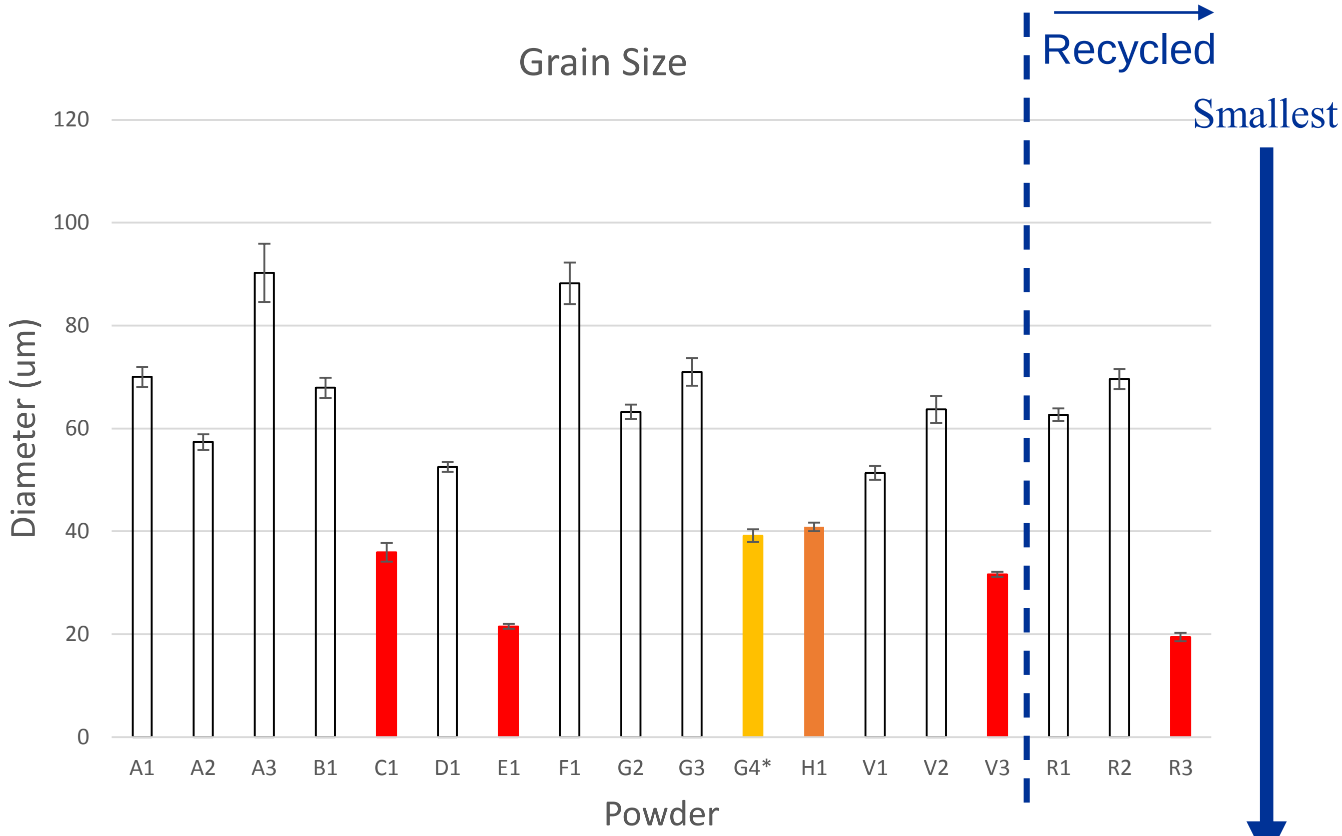


D1



Significant variation among grain size after HIP and heat treatment

Fully Heat Treated Grain Size Measurements



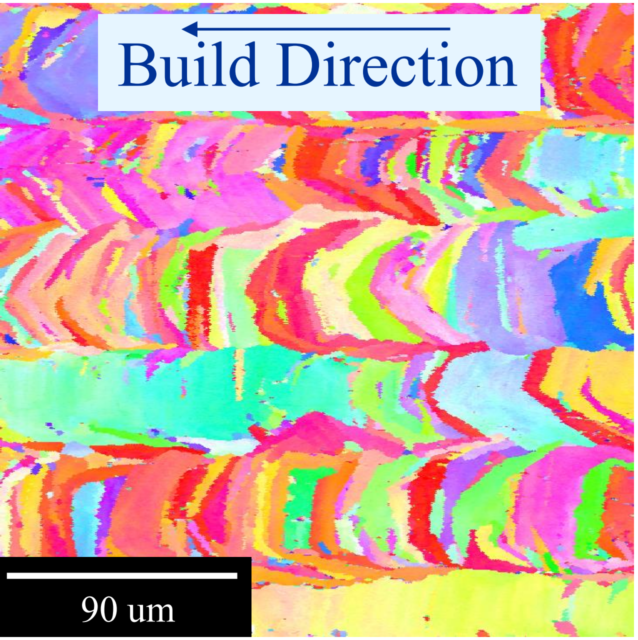
Powder	Grain Dia. (um)
R3	19.5
E1	21.5
V3	31.6
C1	35.9
G4*	39.2
H1	40.9
V1	51.4
D1	52.5
A2	57.3
R1	62.7
G2	63.2
V2	63.7
B1	67.9
R2	69.6
A1	70.0
G3	70.1
F1	88.2
A3	90.3

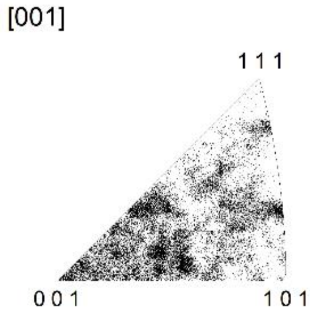
Red: Anisotropic
Orange: Partial recrystallization
Black: Full recrystallization

Other than G4-4, smallest average grain builds did not recrystallize during HIP and heat treatment

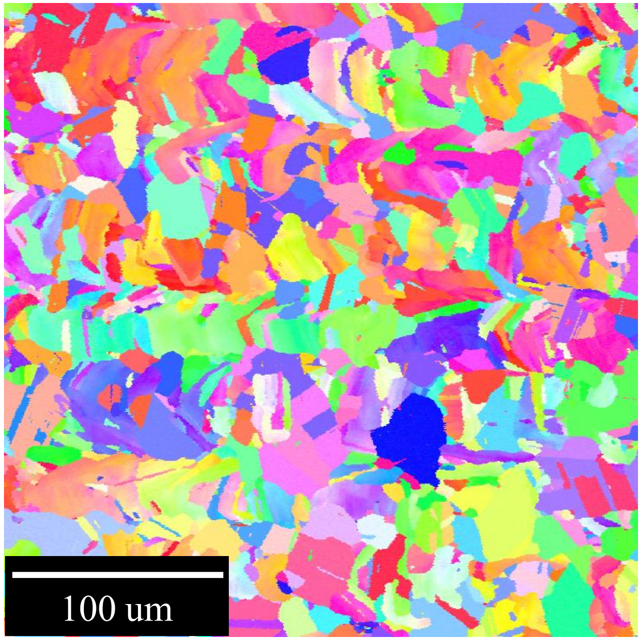
EBSD Maps of Different Grain Structures Observed – XZ Face

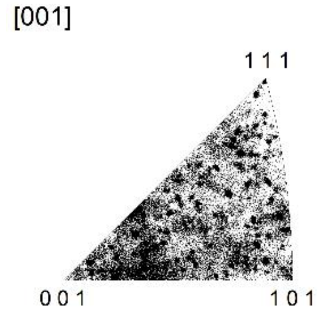
C1 – Retained As-Fab MX



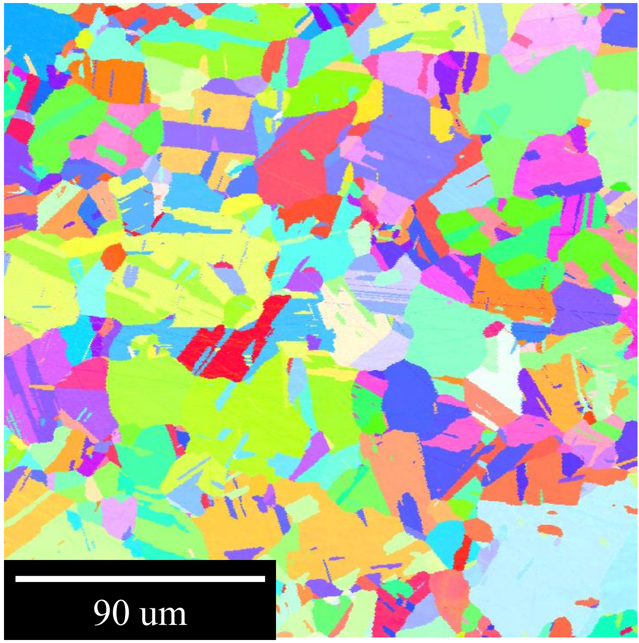


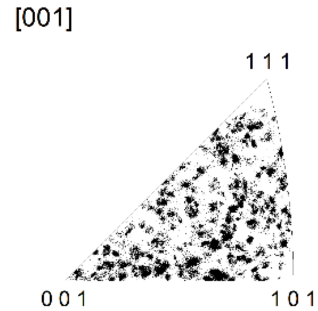
H1 – Partially Recrystallized





B1 – Fully Recrystallized





Strong Correlation Between Grain Structure and N Content

Powder	Grain Dia (um)	N Content (wt%)
R3	19.5	2770
E1	21.5	1220
V3	31.6	2770
C1	35.9	1395
G4*	39.2	199
H1	40.9	562
V1	51.4	331
D1	52.5	122
A2	57.3	87
R1	62.7	331
G2	63.2	207
V2	63.7	370
B1	67.9	25
R2	69.6	0370
A1	70.0	325
G3	70.1	176
F1	88.2	607
A3	90.3	331

The four powders with the highest N content didn't recrystallize during HIP and heat treatment process.

High N content is associated with decrease in rupture life and a reduction in creep properties in superalloys[2]

2 Singh *et al. Engineering Failure Analysis (2013)*

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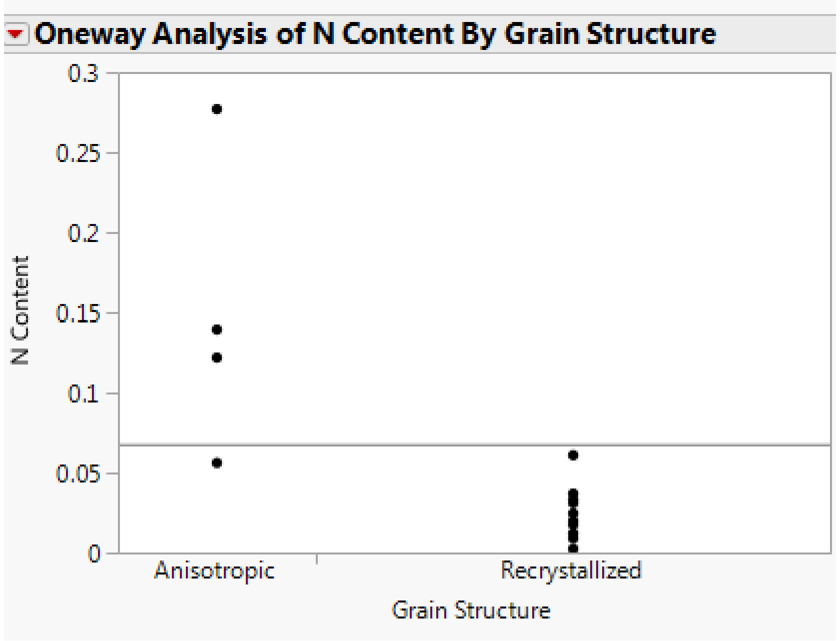

Significant correlation between N content and grain structure[3]: $p=.0143$

[3]JMP 2017

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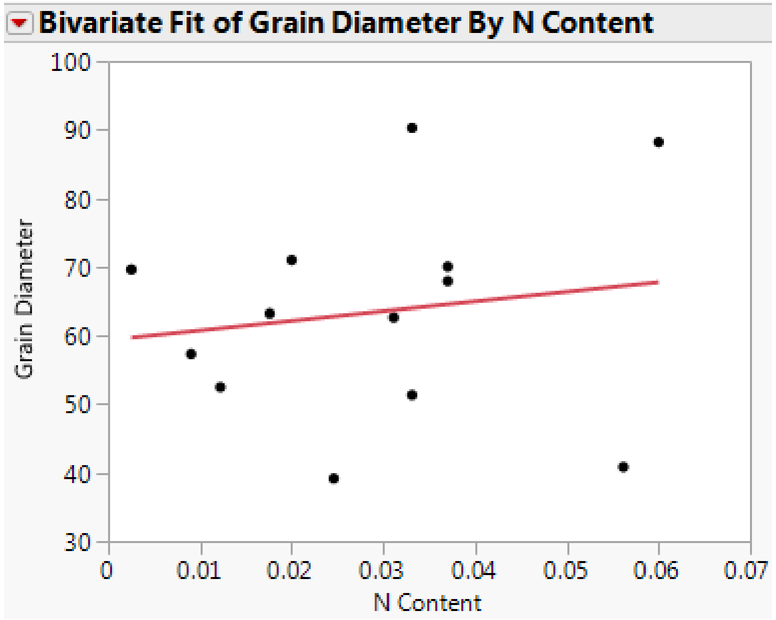
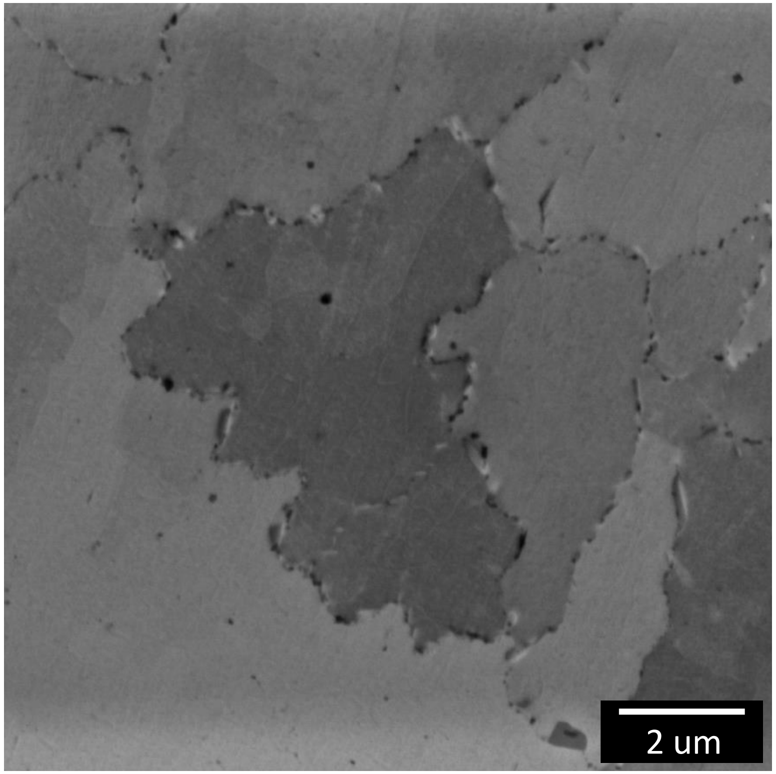


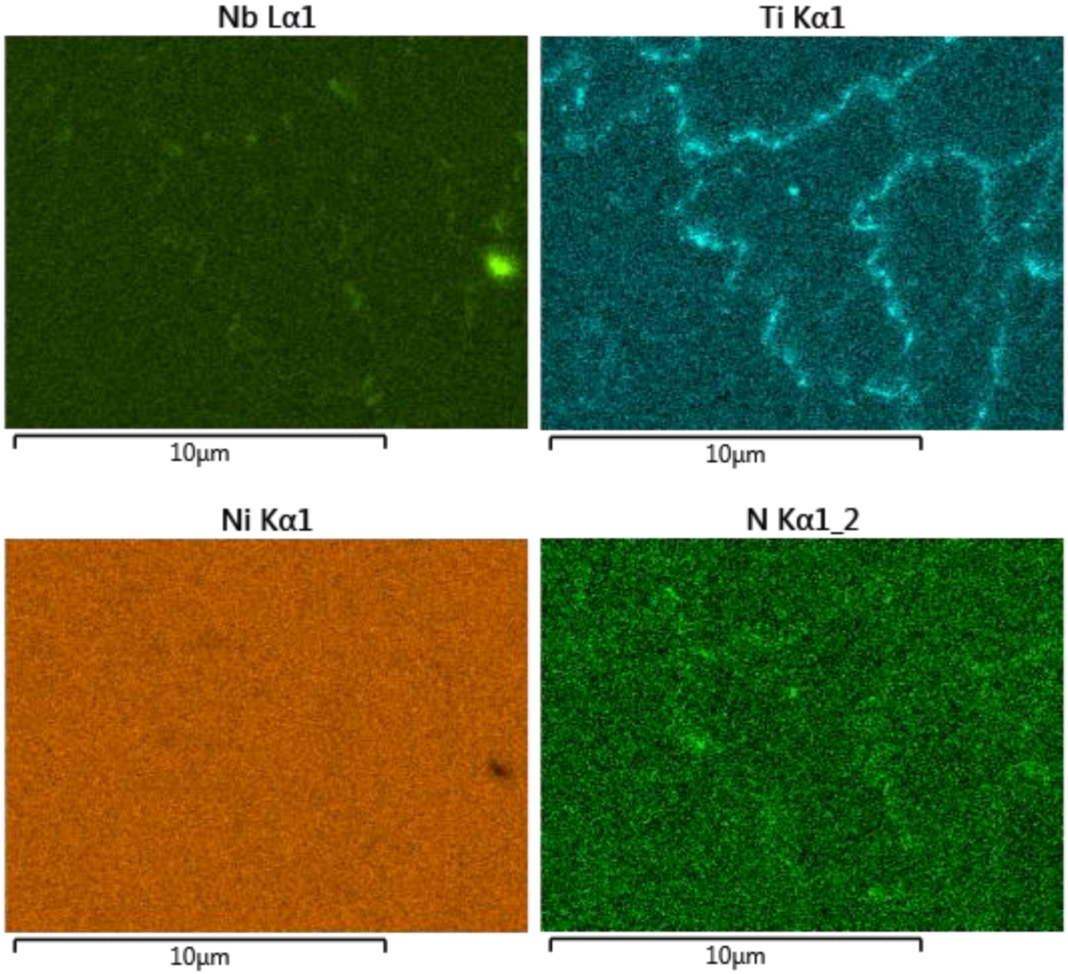
Below .07wt% N - no correlation between N content and average grain diameter[3]: p=.6189

Other secondary phases contributing to grain size?

[3]JMP 2017

SEM-EDS of TiN Inclusions

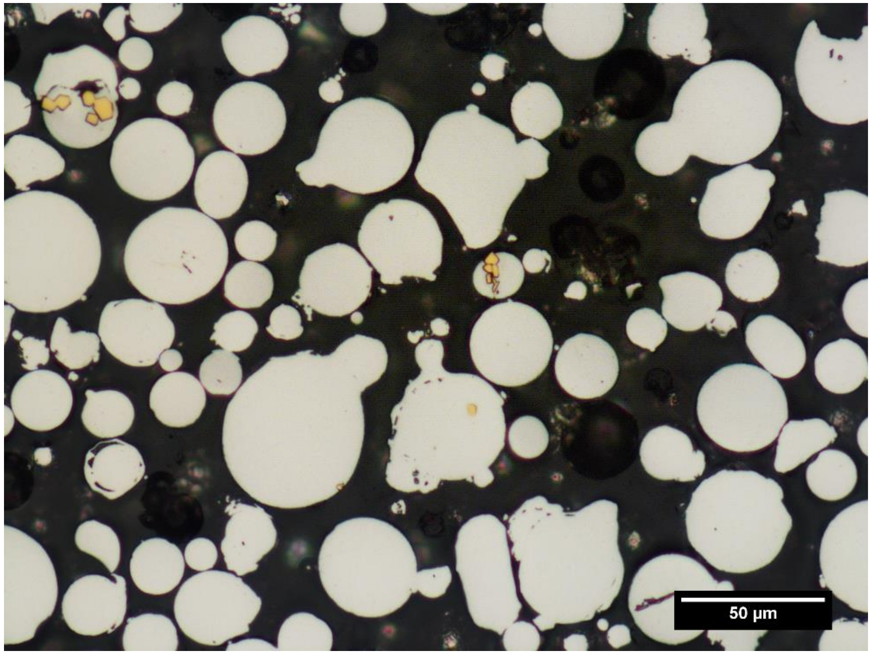




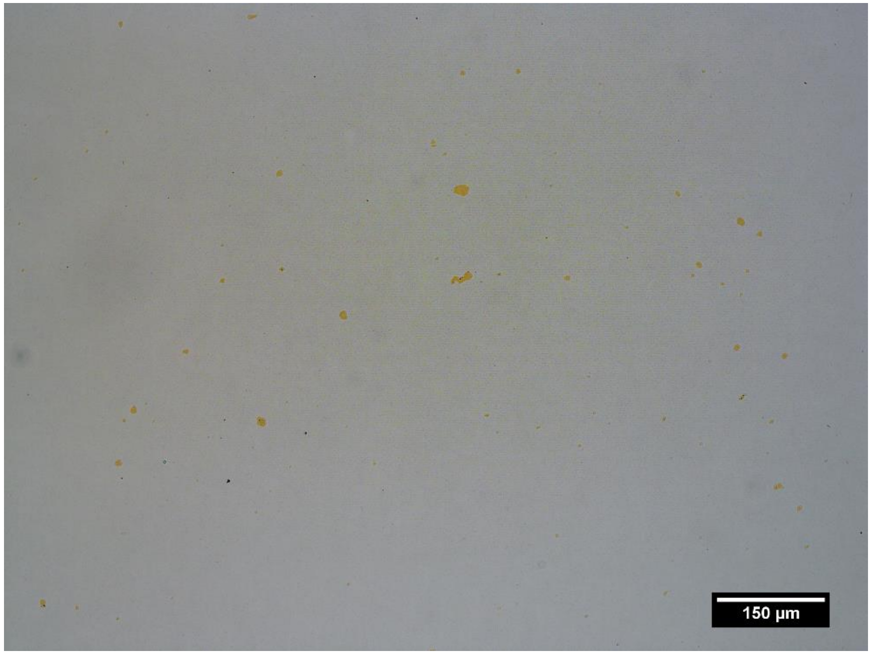
- Evidence of (Ti,Nb)N inclusions pinning grain boundaries in the C1 build.
- Fine (Ti,Nb)N inclusions were also observed in E1 and F1

(Ti,Nb)N Optical Microscopy Observations

V3 Powder



V3 – HIP + Heat Treat



Large (>10um) (Ti,Nb)N inclusions were also observed in V3, R3 and H1 heat treated builds/powders. All three did not recrystallize

Summary and Conclusions

- Overall, significant microstructural improvements, including grain structure and density, were obtained through HIP and heat treatment.
- Different powder characteristics produced notable differences of porosity and grain size in as-fabricated/heat treated microstructures.
- Powders with higher N content (700ppm), produced higher number (Ti,Nb)N inclusion, and consequentially retained the <001> textured build microstructure after post-processing.
- Sub-micron (Ti,Nb)N inclusions were found to pin grain boundaries during post-processing for certain powders with high N content.

Future Work

- Determine the population density of nitrides and other minor phases on grain boundaries and within the samples.
- Closer examination of relationships between defects and microstructure to powder characteristics and mechanical behavior.
- Measure γ'-Ni_3Al, size and area fractions for all FHT builds.
- Further examine the micro-porosity observed in G2.

***Acknowledgments*:** Relationship of powder feedstock variability to microstructure and defects in selective laser melted alloy 718

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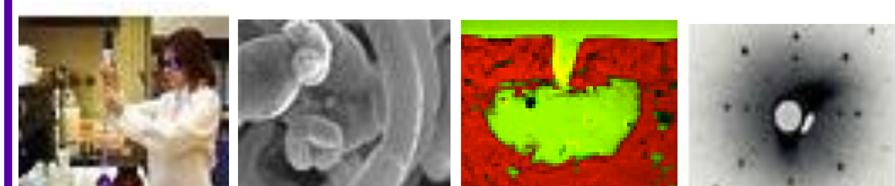
- **Metallography**
- **X-ray Diffraction**
- **Computed Tomography**

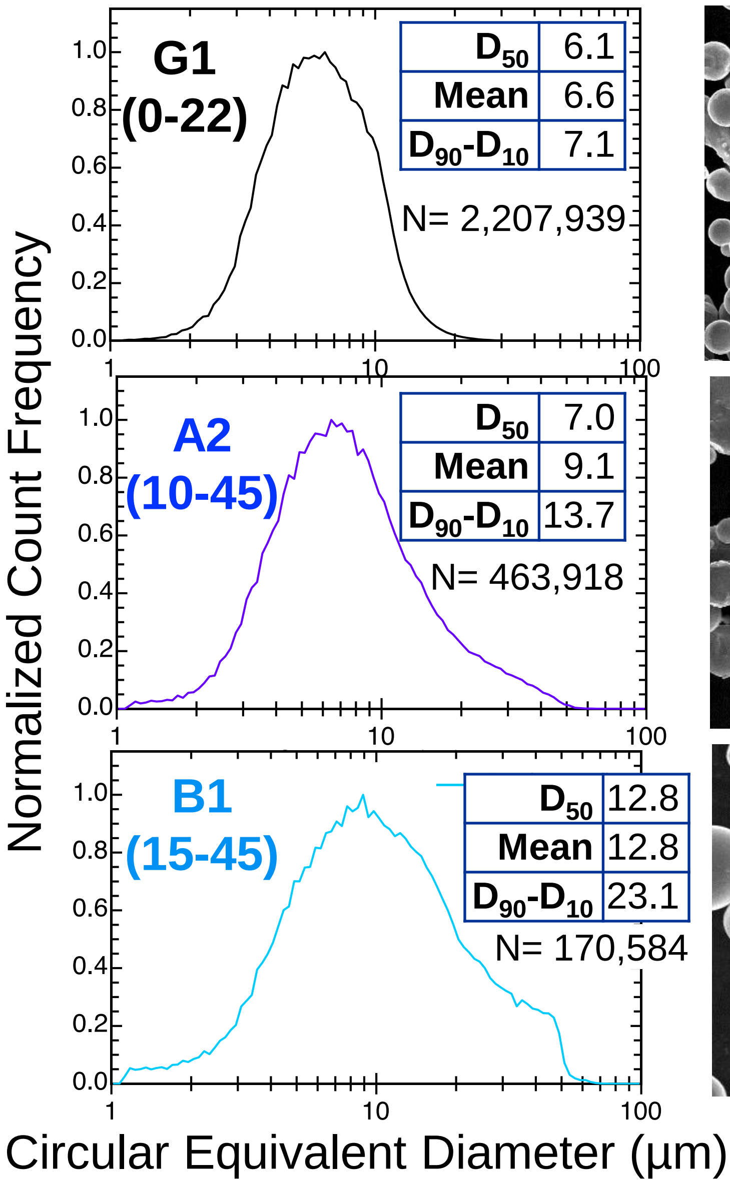


Questions?

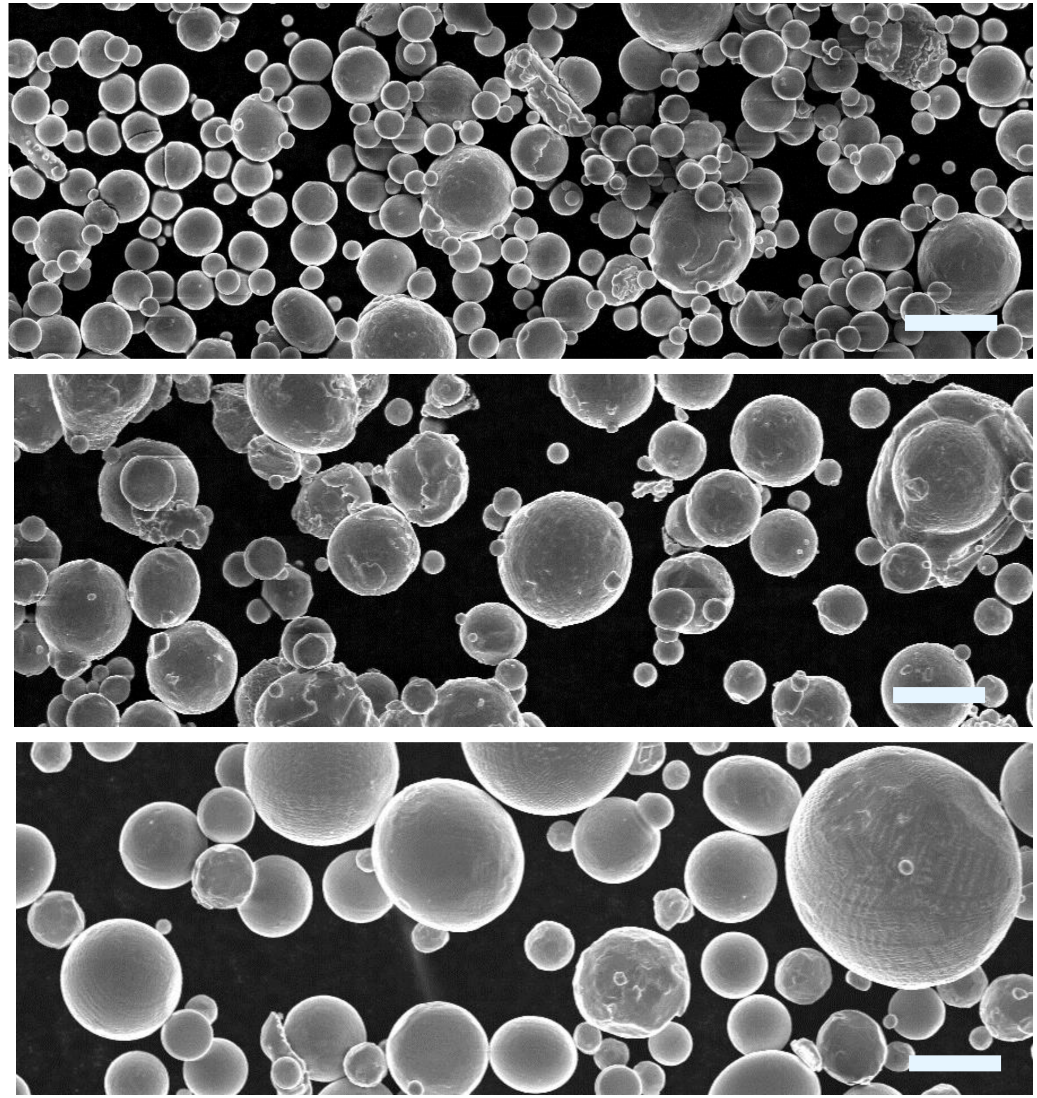
Extra Slides

“Undersized” Group 1: PSDs are unimodal & vary in width

Particle Size Distributions (PSD)



1 of 6 larger & 1 of 5 smaller standard SLM cuts



20 µm

“Mixed” Group 2: PSDs are bimodal or trimodal

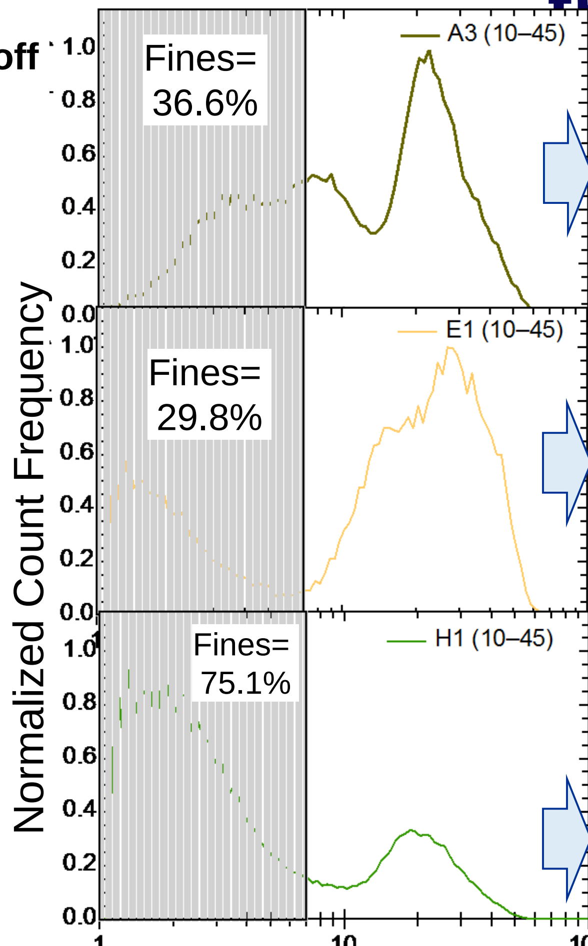
3 of 5 smaller standard SLM cut

7 µm Cutoff

A3 (10-45)

E1 (10-45)

H1 (10-45)



N_c= 63,688

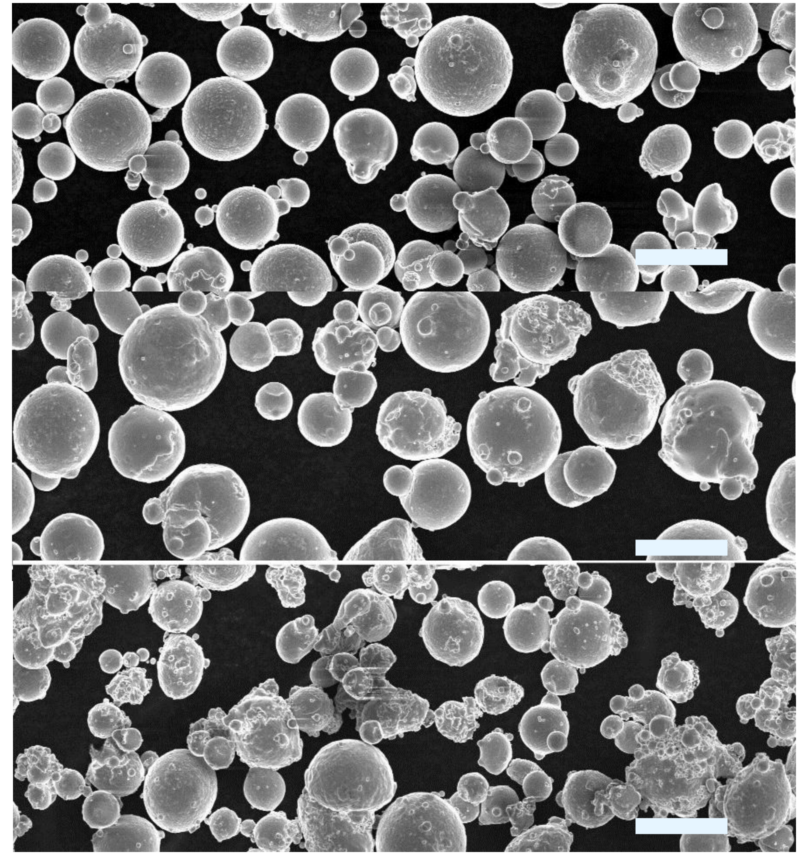
D_{50}	20.2
Mean	21.1
D_{90}-D_{10}	26.9

N_c= 34,342

D_{50}	23.9
Mean	25.3
D_{90}-D_{10}	29.8

N_c= 63,085

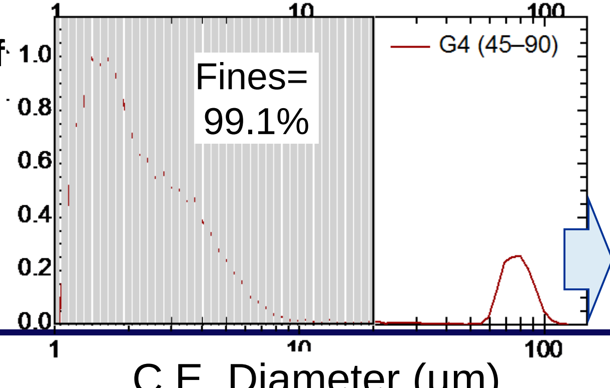
D_{50}	18.7
Mean	20.0
D_{90}-D_{10}	22.9



40 µm

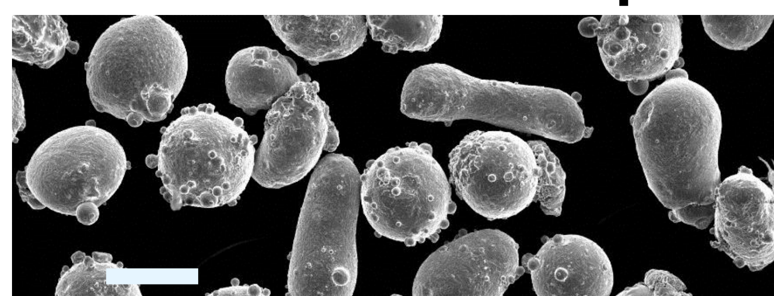
20 µm Cutoff

G4 (45-90)



N_c= 2,379

D_{50}	77.4
Mean	26.8
D_{90}-D_{10}	28.0

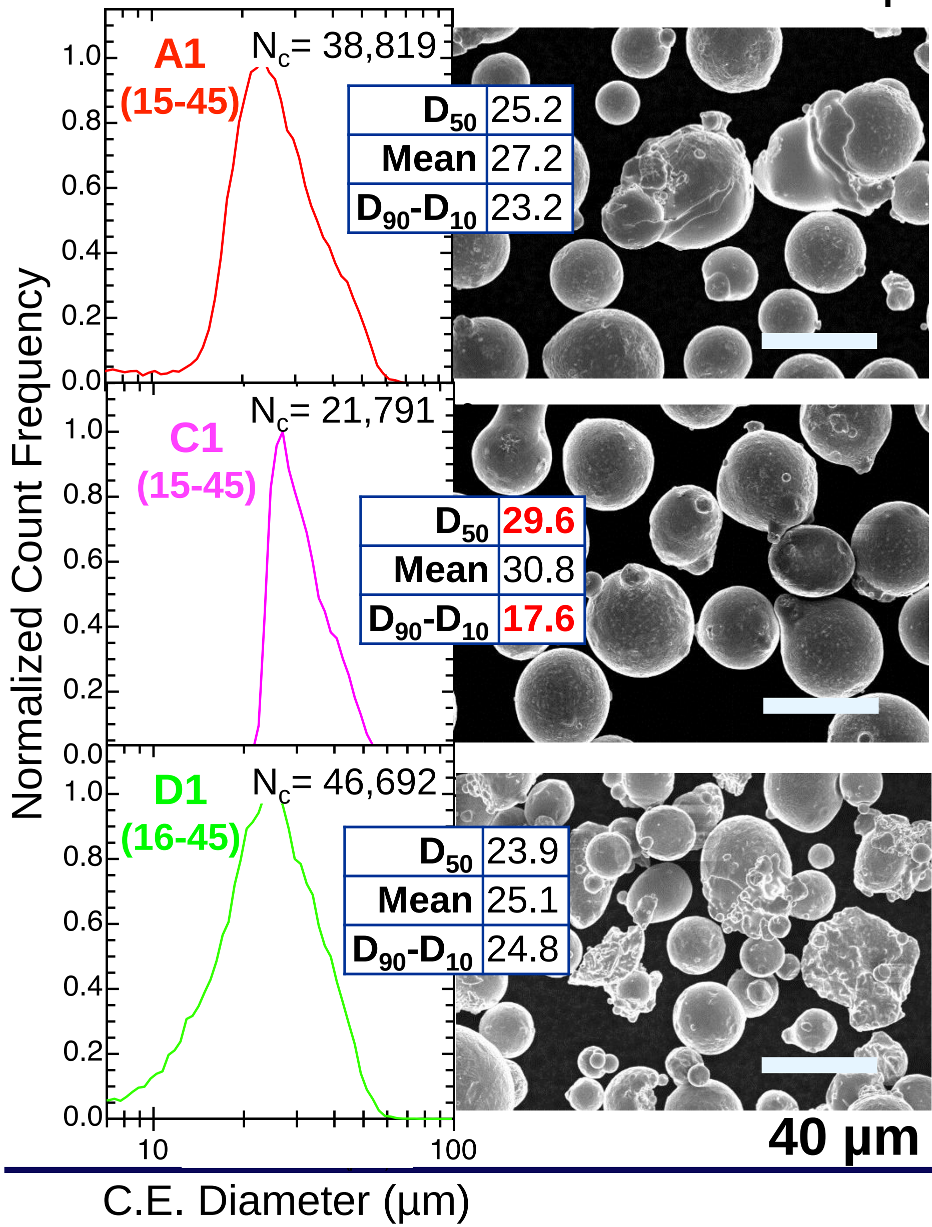


80 µm

“Normal” Group 3: Unimodal PSDs with few satellites

5 of 6 larger and 1 of 5 smaller standard SLM cuts

7 µm Cutoff

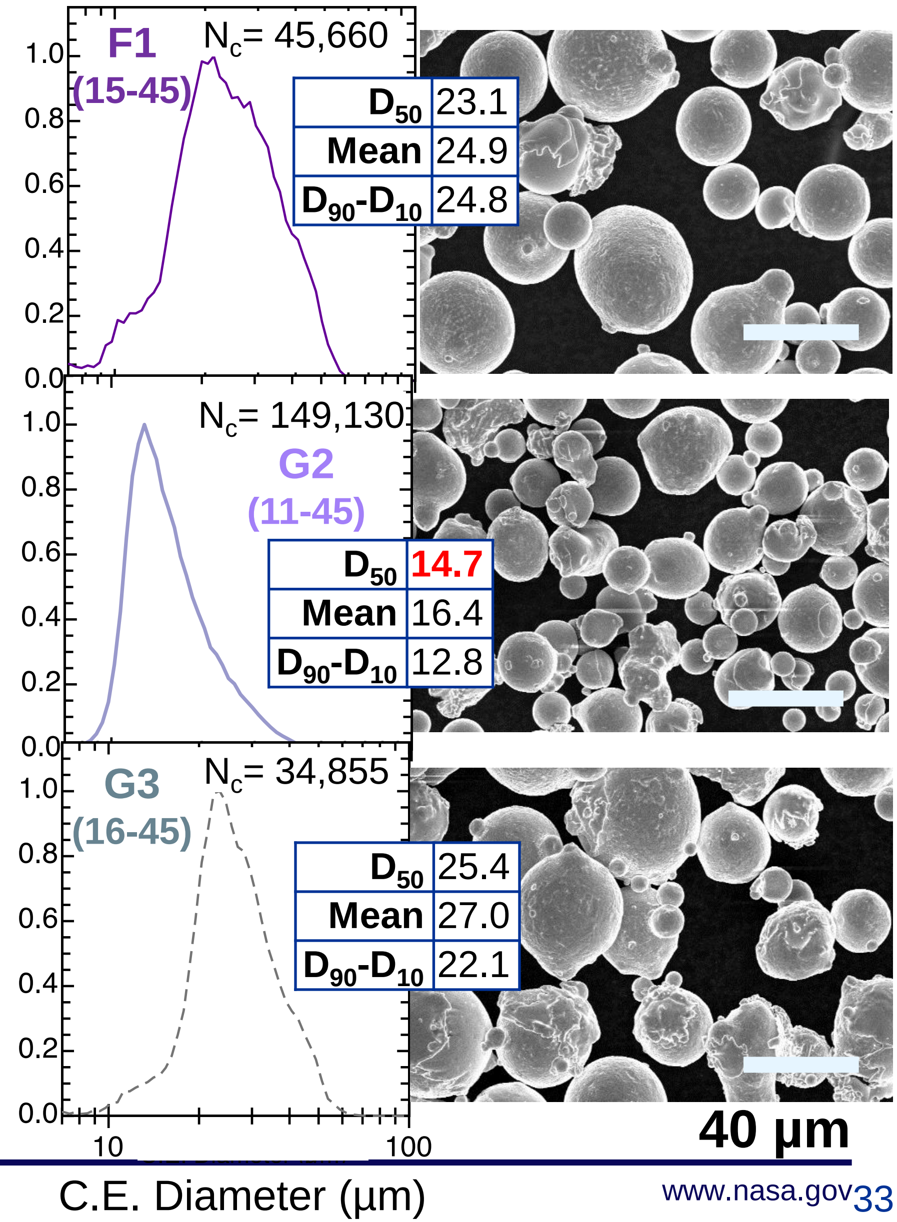


D_{50}	25.2
Mean	27.2
D_{90}-D_{10}	23.2

D_{50}	**29.6**
Mean	30.8
D_{90}-D_{10}	**17.6**

D_{50}	23.9
Mean	25.1
D_{90}-D_{10}	24.8

7 µm Cutoff



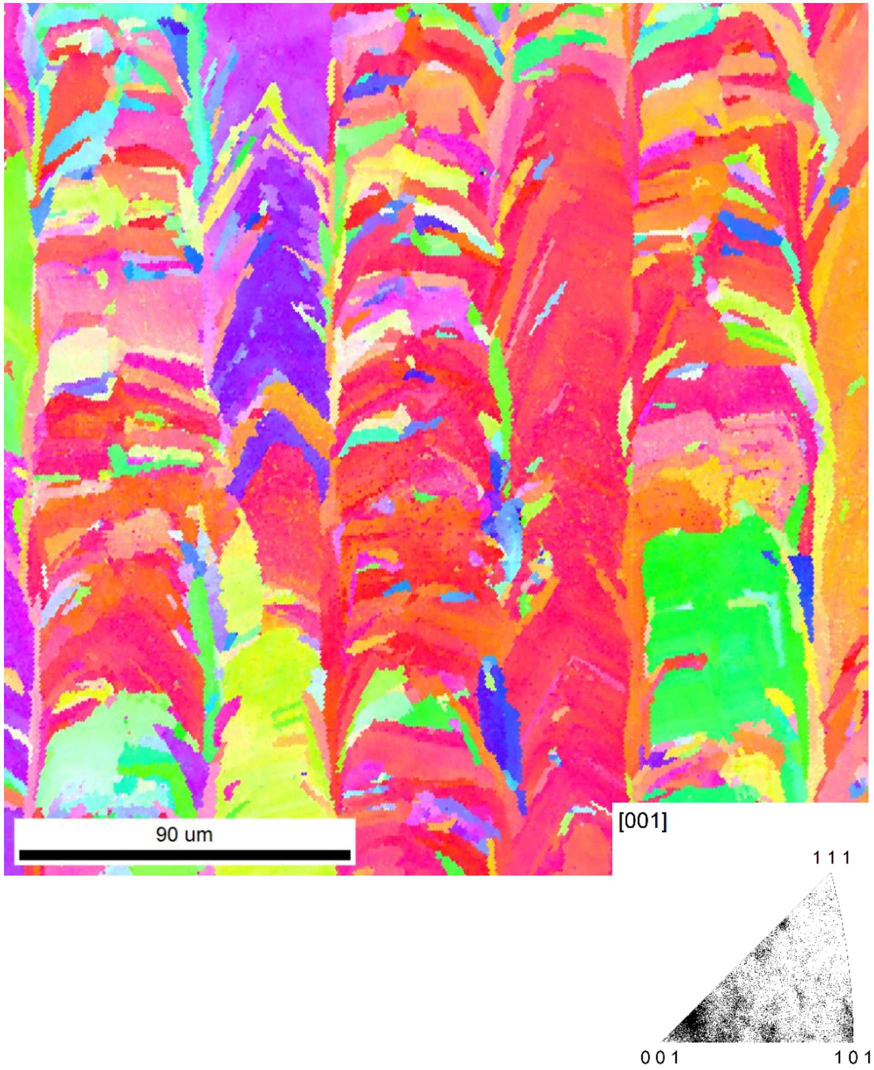
D_{50}	23.1
Mean	24.9
D_{90}-D_{10}	24.8

D_{50}	**14.7**
Mean	16.4
D_{90}-D_{10}	12.8

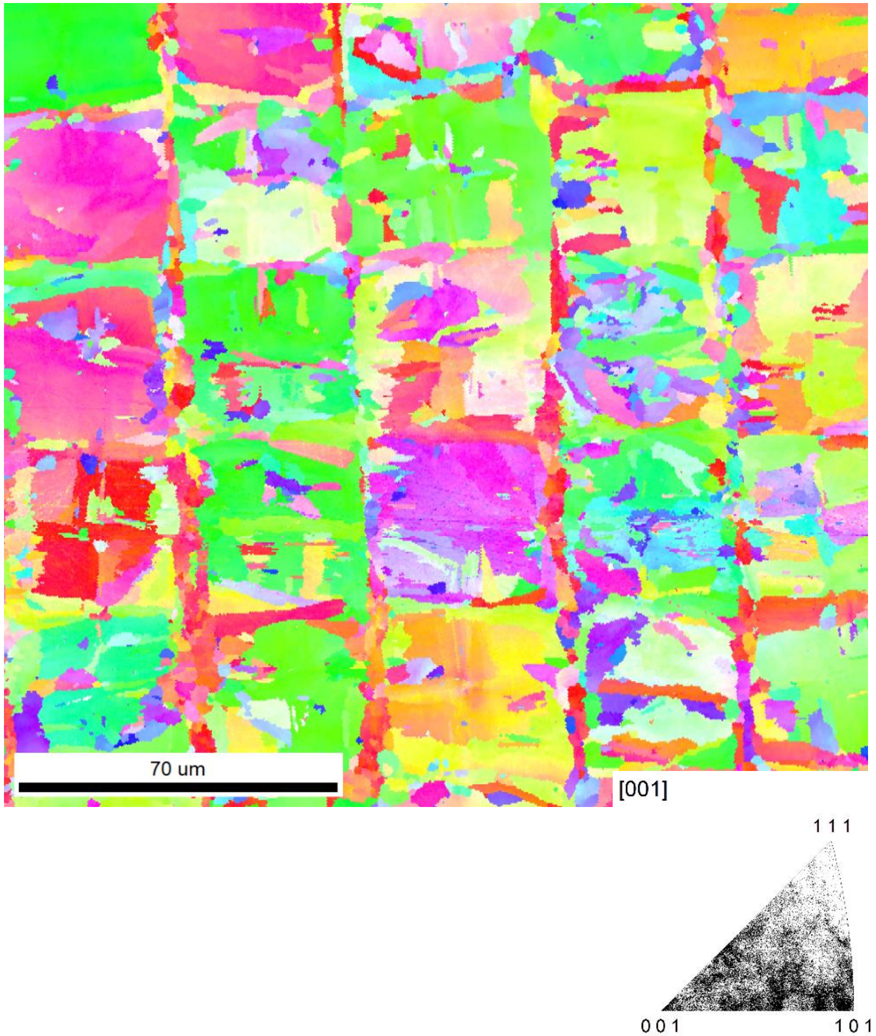
D_{50}	25.4
Mean	27.0
D_{90}-D_{10}	22.1

EBSD of As-Fabricated Microstructure

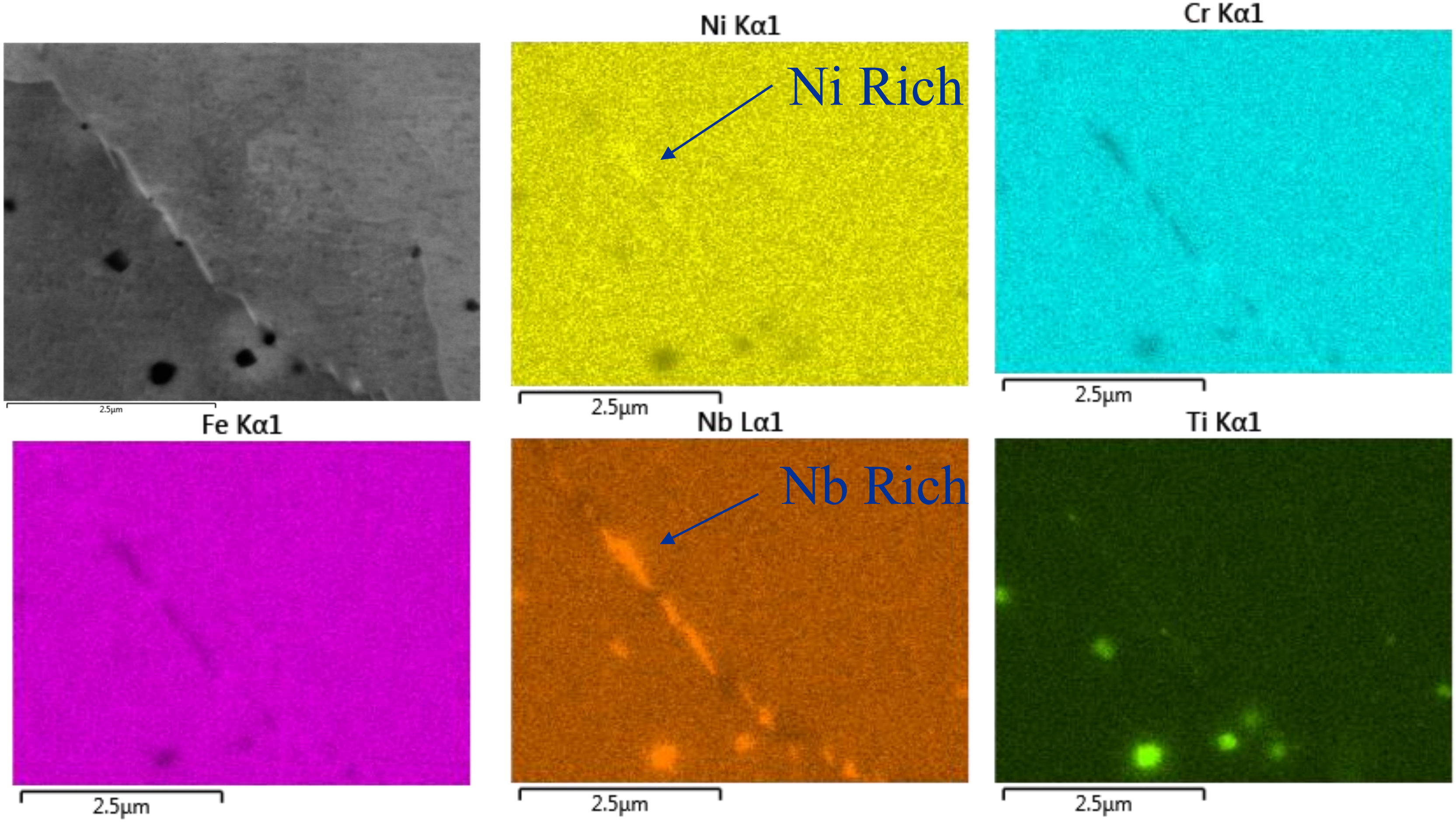
Long – XZ face



Trans – XY face

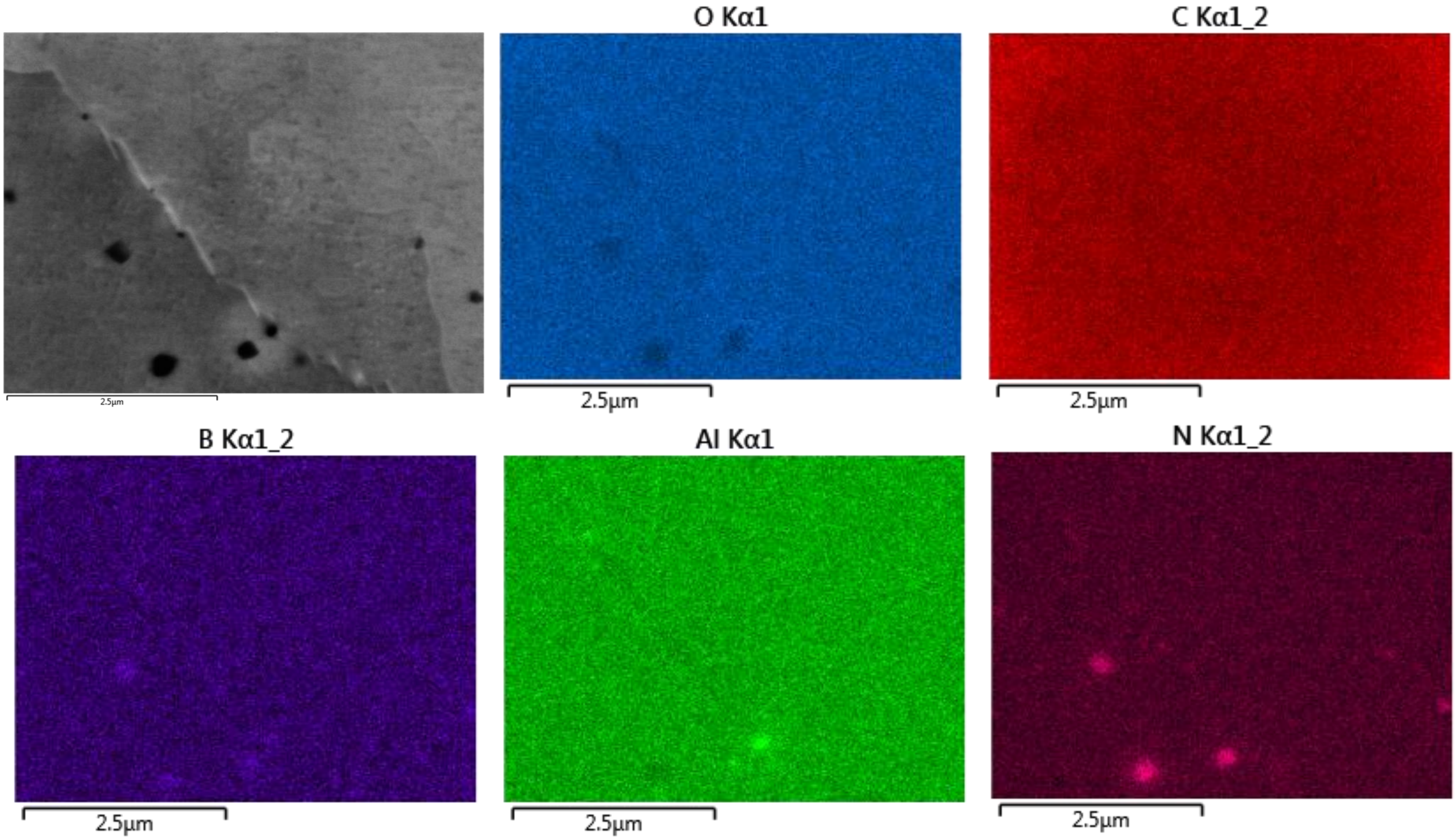


C1 New High Resolution EDS



The bright phases near the grain boundary are rich in Ni and Nb. If this was a secondary phase the Ni map should appear depleted like the Cr and Fe maps.

C1 New High resolution EDS



No evidence that these phases are N, O, C, or B rich. In conclusion, the bright GB phases appear to be the Ni_3Nb delta phase.