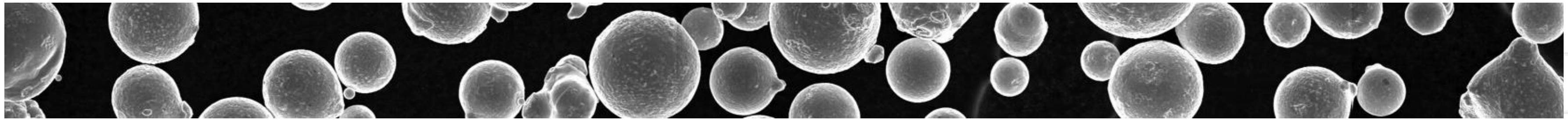
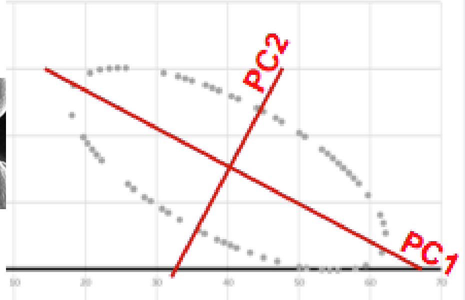
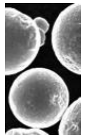
MS&T 2019 - Alloy Design for Additive Manufacturing: Developing New Feedstock Materials Symposium

Discerning the Impact of Powder Feedstock Variability on Structure, Property, and Performance of Selective Laser Melted Alloy 718: A Principal Component Analysis (PCA) of Feedstock Variability







David Ellis and Chantal Sudbrack*

NASA John H. Glenn Research Center at Lewis Field, Cleveland Ohio;

*** Formerly of NASA John H. Glenn Research Center at Lewis Field, Cleveland Ohio**

GLENN RESEARCH CENTER at Lewis Field

October 2, 2019

MARSHALL SPACE FLIGHT CENTER

Funding: NASA HEOMD / SLS Liquid Engine Office / Additive Manufacturing Structural Integrity Initiative Project (FY16-FY18)

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

Project Coordination
- Chantal Sudbrack, Team Lead
- Cheryl Bowman, Team Lead
- Brian West

Powder Characterization
- Richard Boothe
- David Ellis
- Alejandro Hinojos (OSU)
- Chantal Sudbrack

MSFC AM Fabrication
- James Lydon
- Omar Mireles
- Ken Cooper

Analytical Characterization
- Rick Rogers
- Dereck Johnson
- Joy Buehler

Heat Treat & Machining
- Will Tilson
- MSFC Heat Treat Facility
- GRC Specimen Shop

Microstructural Evaluation
- Ivan Locci
- Tim Smith
- Chantal Sudbrack
- Alejandro Hinojos (OSU)
- Michael Kloesel (Cal Poly)
- Bethany Cook (CWRU)
- Jonathan Healy (CWRU)

Mechanical Testing
- Brad Lerch
- Aaron Thompson
- Jonathan Woolley
- GRC Testing Facility

Fractography
- Paul Chao (CMU)
- Ben Richards (NU)
- Ivan Locci

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

Published: Proceedings Superalloy 718 & Derivatives: E. Ott et al (Eds.), TMS (Pittsburgh), 89-113 (2018)

SLM 718 Powder Feedstock Variability Study

A Principal Component Analysis (PCA) of Feedstock Variability

- *Motivation and background*
  - *Overview- 18 powders from 8 suppliers (A-H)*
- *Background into Principal Component Analysis*
- *Experimental Results*
  - Powder characteristics
  - Build Microstructure
  - Mechanical Property Evaluation – Tensile / HCF results
- *PCA Results*
- *Summary and Concluding Remarks*
- *In-Progress Research at NASA GRC*

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

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

Motivation

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

POC: Doug Wells


National Aeronautics and Space Administration

MSFC-STD-xxxx
REVISION: DRAFT 1
EFFECTIVE DATE: Not Released

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

EM20

MSFC TECHNICAL STANDARD

Engineering and Quality Standard for Additively Manufactured Spaceflight Hardware

DRAFT 1 – JULY 7, 2015

This official draft has not been approved and is subject to modification.
DO NOT USE PRIOR TO APPROVAL

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

THIS STANDARD HAS NOT BEEN REVIEWED FOR EXPORT CONTROL RESTRICTIONS
DRAFT VERSIONS DISTRIBUTED FOR REVIEW ARE NOT TO BE DISSEMINATED

Objectives

- Use Principal Component Analysis (PCA) to determine the largest contributors of variability in data set of feedstock characteristics, microstructure, key properties and performance
- Apply PCA to subsets of the data set to determine relationships between variables and their effect on variability
- Use PCA to support down selection of 5 powders for expanded property assessment

Supplier-to-supplier comparison 18 powders and 194 variables measured

Majority of powders were gas-atomized in Ar

Suppliers: 1 Reseller , 7 Direct Source Manufacturers

	Gas	ID	Alloy 718 Powders	Powder cut range in µm
GRC 13 virgin; Off-the-shelf small Lots (>20 kg) from “” commercial production	Ar	A1	Supplier 1, Powder 1 (Reseller)	15-45
	Ar	A2	Supplier 1, Powder 2(Reseller)	10-45
	Ar	A3	Supplier 1, Powder 3 (Reseller)	10-45
	Ar	B1	Supplier 2, P1 (Thermal Spray)	15-45
	N	C1	Supplier 3, Powder 1	15-45
	Ar	D1	Supplier 4, Powder 1	16-45
	N	E1	Supplier 5, Powder 1	10-45
	Ar	F1	Supplier 6, Powder 1	15-45
	Ar	G1	Supplier 7, Powder 1 (No build)	0-22
	Ar	G2	Supplier 7, Powder 2	11-45
	Ar	G3	Supplier 7, Powder 3	16-45
	Ar	G4	Supplier 7, Powder 4	45-90
	Ar	H1	Supplier 8, Powder 1	10-45
MSFC 3 virgin; Full Lots (>1000 kg)	Ar	D2 (V1)	Supplier 4, Powder 2	10-45
	Ar	F2 (V2)	Supplier 6, Powder 2	10-45
	N	E2 (V3)	Supplier 5, Powder 2	10-45
	Ar	D2-R1	Supplier 4, Powder 2 (2nd build)	10-45
	Ar	F2-R2	Supplier 6, Powder 2 (2nd build)	10-45
	N	E2-R3	Supplier 5, Powder 2 (2nd build)	10-45

G Series: Size effect with Same chemistry

R “Reuse” Powders: *2nd Build Same Powder Topped Off*

Feedstock
Powder Chemistry
Particle Size Distribution (PSD), e.g. D(50), FWHH
Shape Factors
Packing Density
Flow measurements
Rheological properties

Build Microstructure
Build Chemistry
Green State - Porosity: % and Size
Green State - Large Nitrides: % and Size
Heat Treated - Porosity: % and Size
Heat Treated – Mean Grain Diameter
Heat Treated– Avg. Flam. Burn Length

Not present in most Ar-atomized powders

Processing
Melt pool depth (150 µm-300 µm)
As-fabricated surface roughness

Properties
Heat Treated - Hardness (425- 471 MPa)
Heat Treated – Tensile YS, UTS, Ra, PL, Elong.
Heat Treated– HCF Fatigue Life for LSG, AF

Published: Proceedings Superalloy 718 & Derivatives: E. Ott et al (Eds.), TMS (Pittsburgh), 89-113 (2018)

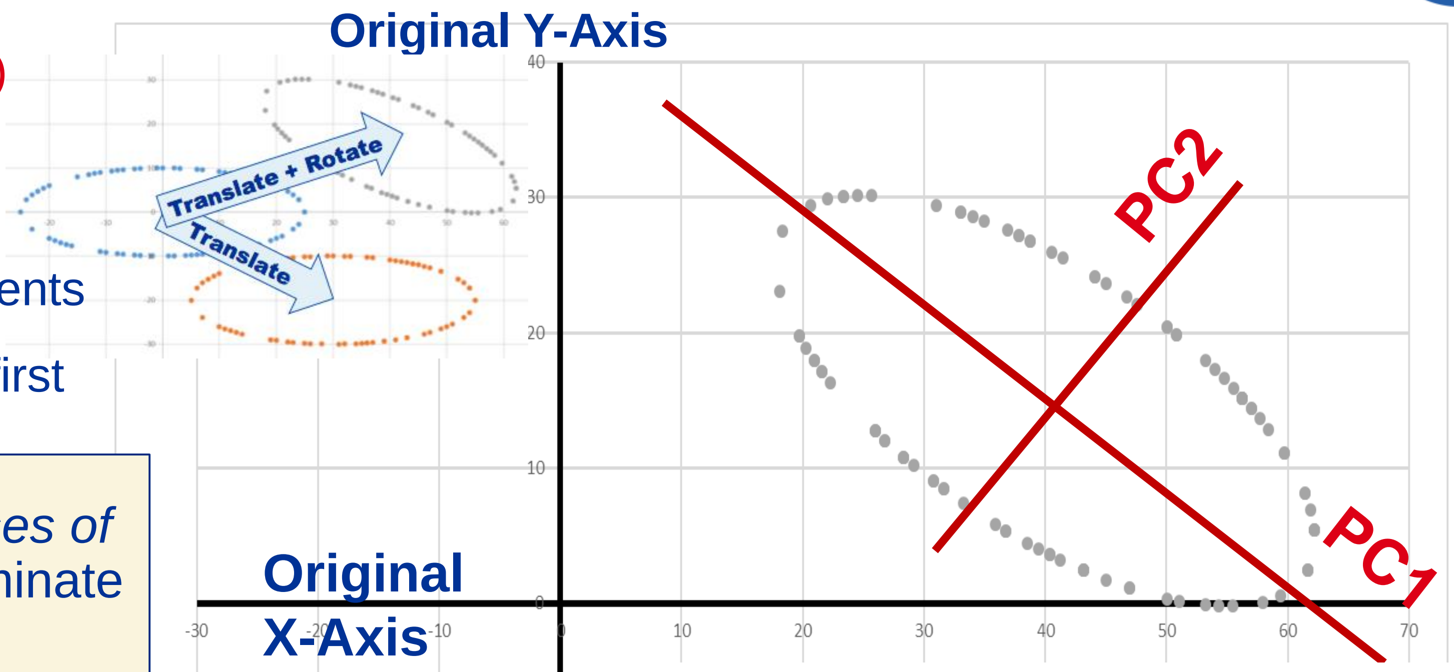
Background into Principal Component Analysis

(Transpose)(Original)=Scaling (Covariance)

(Matrices) $X^T X = nV$

Eigenvectors of **V** are the principle components

Eigenvalues give the PC rank, e.g. largest first



Principal Components are axes that represent maximum variability in the data

PC1 – Axis containing the largest variability in the data set

PC2 – Orthogonal axis to PC1 containing the second most variability in the data set

PC*p* – Nth orthogonal axis with the Pth most level of variability in the data set

PCA determines which variables *or classes of variables* have the biggest effect and eliminate variables with minimal contributions

PCA is:

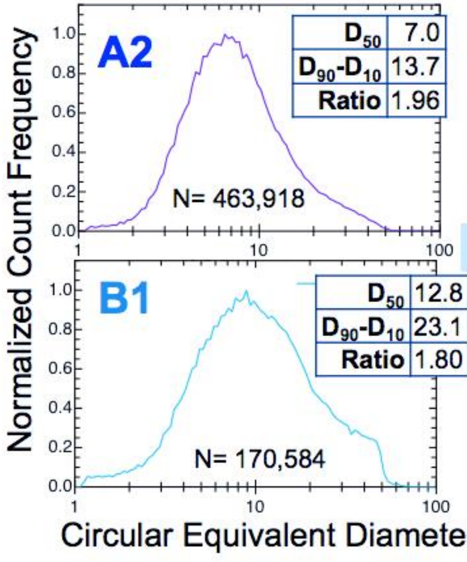
- Not a predictive regression model of the dependent variables
- Used to find the largest contributors to the variability in the data
- Reduces *n* independent variables to *p* principle components

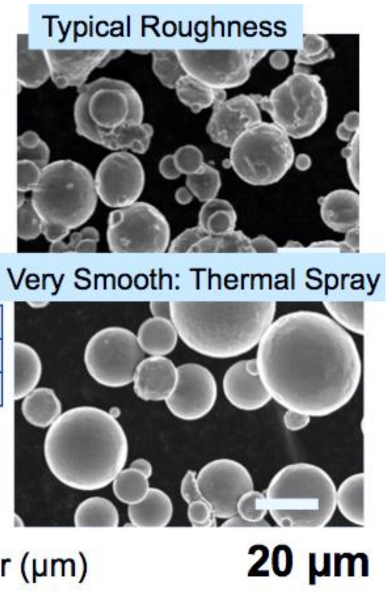
Wide variability in powder characteristics for 18 powders investigated

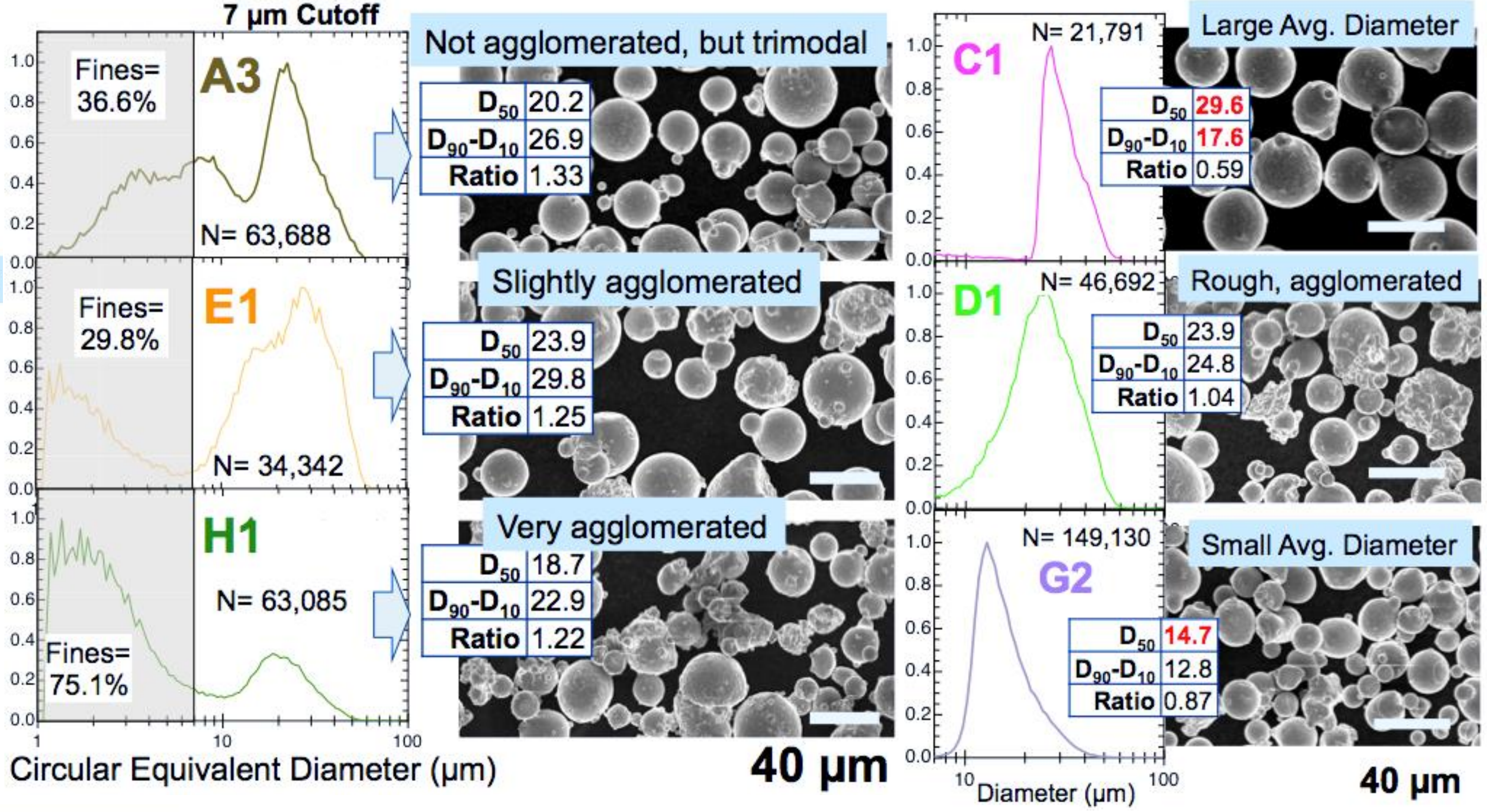
Size PSDs

Undersized

Standard Size: Bimodal vs. Unimodal (few fines)







Number-basis distributions

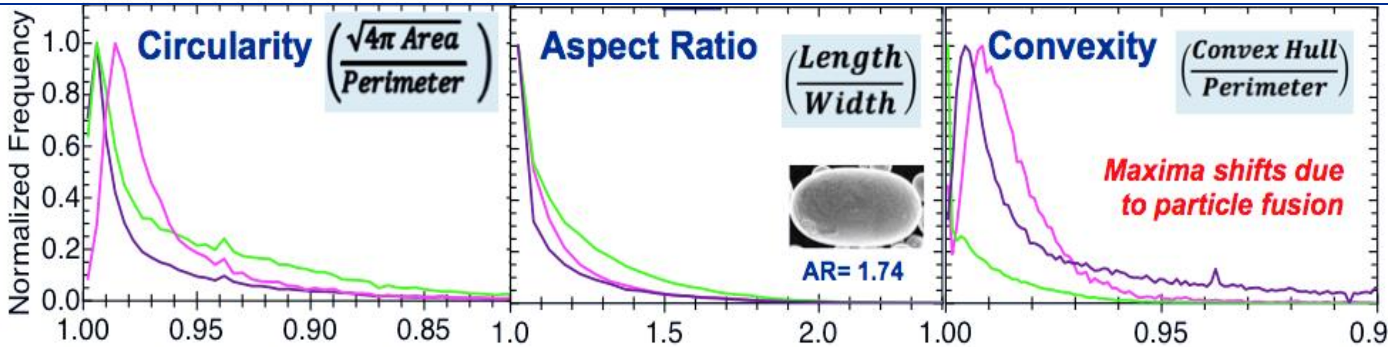
Mean Diameter, D_{50}
Distribution Width, D_{90}-D_{10}
Ratio= $D_{50}/(D_{90}-D_{10})$

Shape Factors

Larger diameter *C1*

Agglomerated D1

Typical



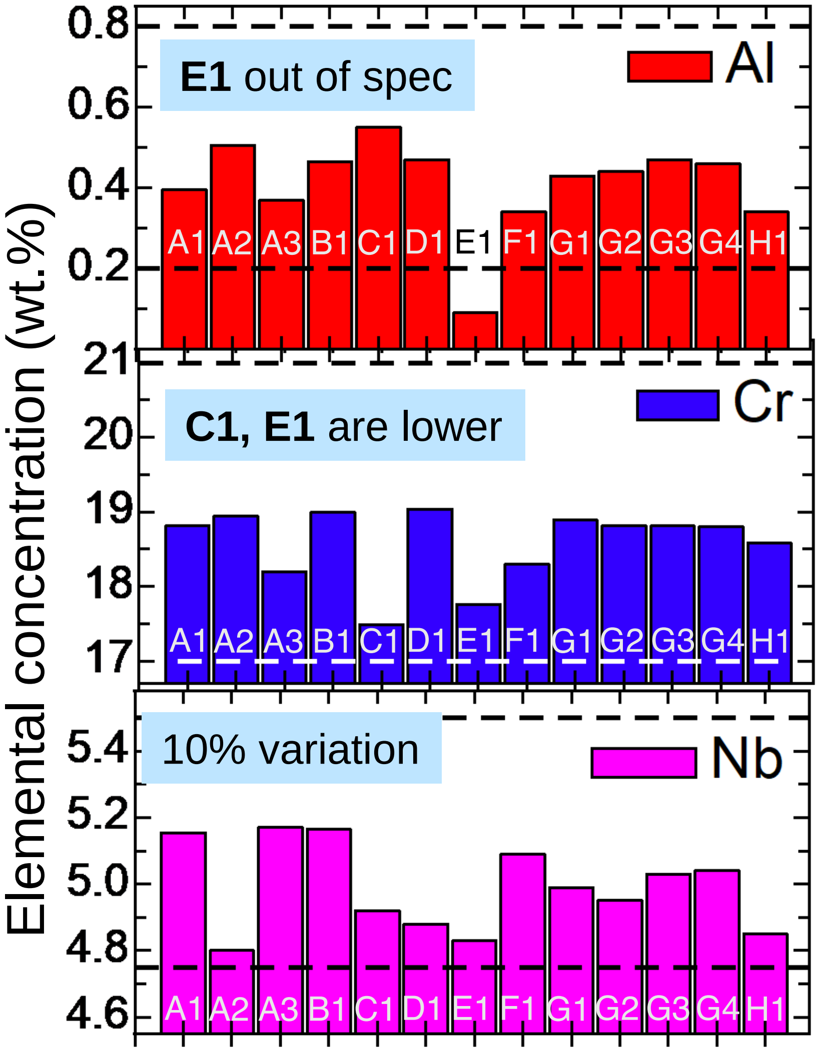
Circularity = Aspect Ratio = Convexity = 1

For a perfect sphere silhouette

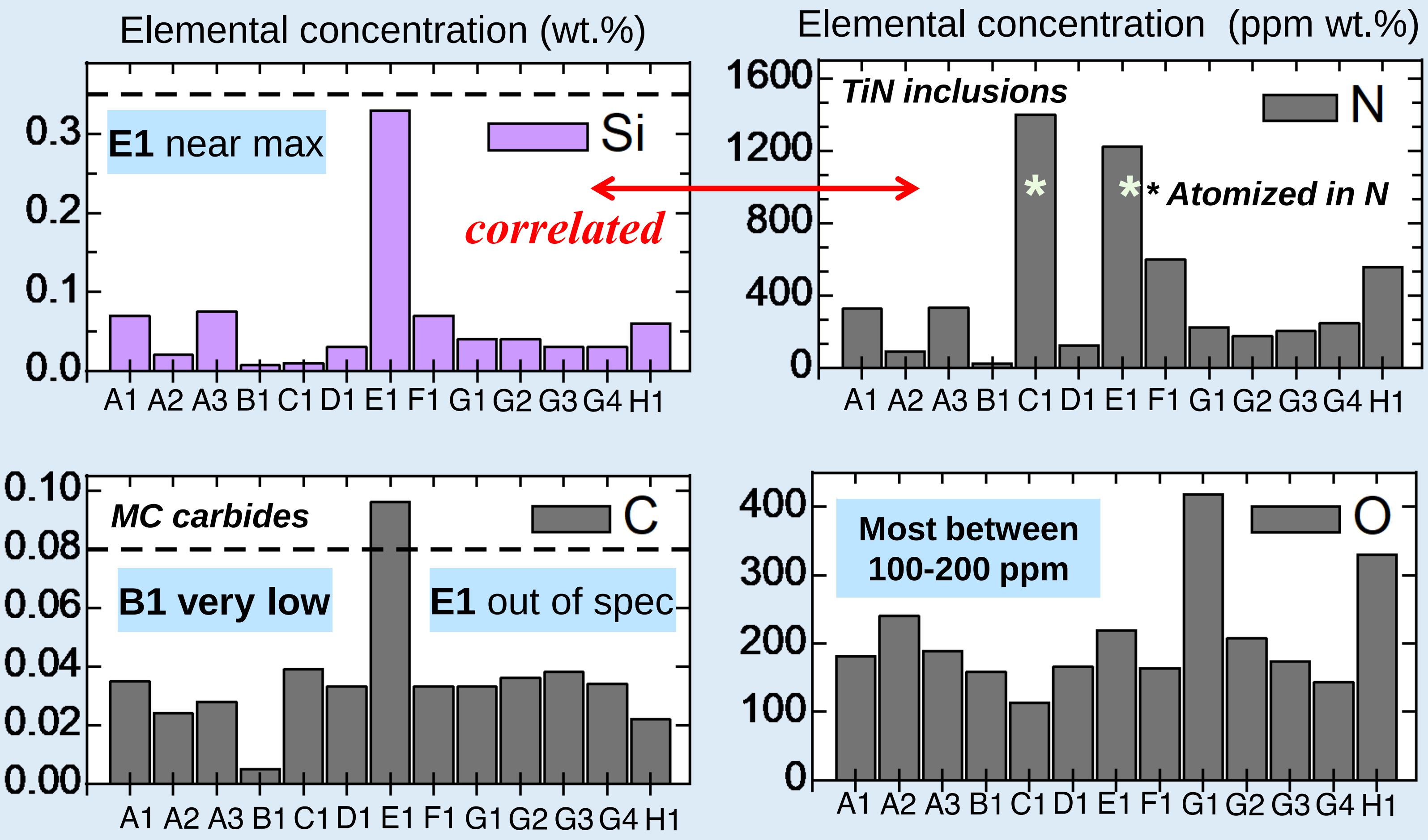
Wide variability in nitrogen content

Two main outliers: B1 low in C; E1 powder is out of spec-low Al, high C & Si

Precipitate strengtheners



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



2 ICP-AES run average compared to ASTM 367

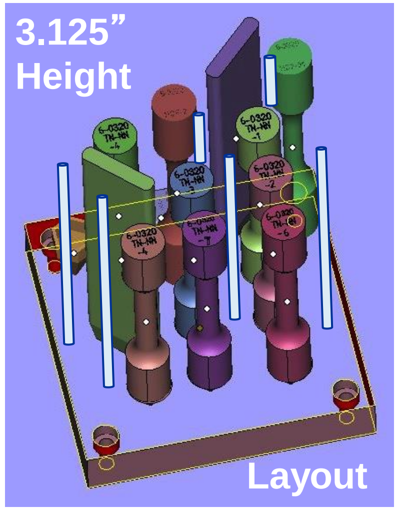
Some Experimental Details

Selective laser melting fabrication using Concept Laser M1

MSFC Build → *10 – 16 weeks* → *To GRC*

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

18 builds over 3 months



Taper Ends for Easy Snap Off

Screen room temperature mechanical behavior of fully heat treated test bars

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 $R_\sigma = -1$
  - 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

Builds from N_2-atomized powders retain the fine SLM grains after heat treat

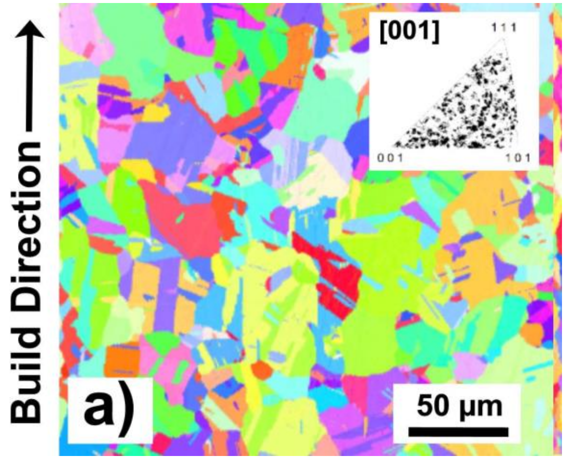
ID	Gas	D(50)	Avg Grain	All builds have fine nitrides in bulk
A1	Ar	25.1	70.0 ± 5.5	Recrystallized
A2	Ar	**7.0**	57.3 ± 3.6	Recrystallized
A3	Ar	20.1	74.4 ± 12.2	Recrystallized
B1	Ar	9.5	67.9 ± 8.6	Recrystallized
C1	**N**	**29.1**	35.9 ± 4.5	Anisotropic
D1	Ar	23.7	52.5 ± 3.6	Recrystallized
D2	Ar	17.9	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

FG

Fine grain

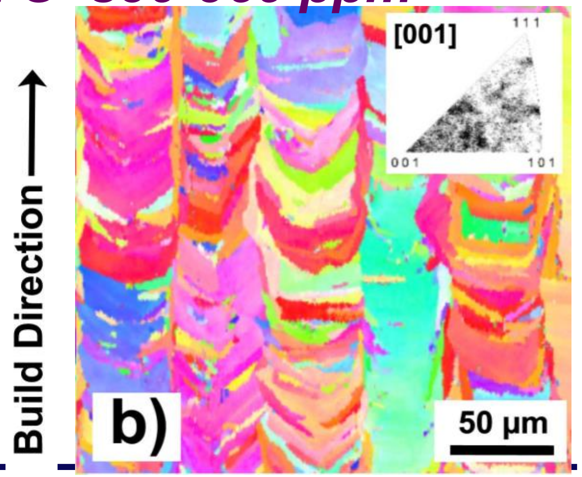
Few minor phases at GBs: N<600 ppm & C=50-390 ppm

Ar



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

N



Builds from N_2-atomized powders retain the fine SLM grains after heat treat

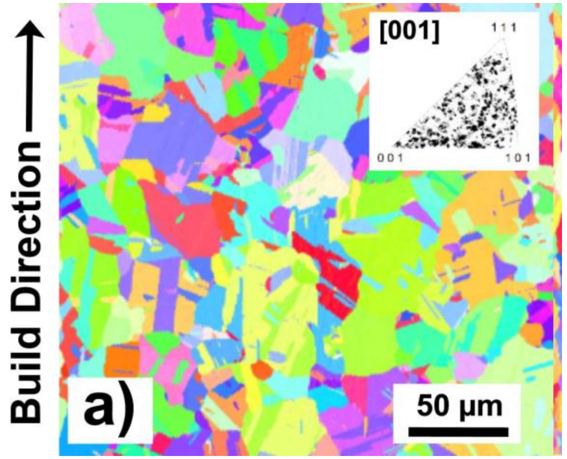
Best RT HCF (FG) — C1

Fine grain

ID	Gas	Many fine GB Nitride/Carbides	Large Bulk Nitrides	Large GB Carbides	All builds have fine nitrides in bulk
A1	Ar				Recrystallized
A2	Ar				Recrystallized
A3	Ar				Recrystallized
B1	Ar				Recrystallized
C1	N	★			Anisotropic
D1	Ar				Recrystallized
D2	Ar				Recrystallized
D2-R	Ar				Recrystallized
E1	N		★	★	Anisotropic
E2	N		★		Anisotropic
E2-R	N		★		Anisotropic
F1	Ar				Recrystallized
F2	Ar				Recrystallized
F2-R	Ar				Recrystallized
G2	Ar				Recrystallized
G3	Ar				Recrystallized
H1	Ar		★		Partially Recryst'd

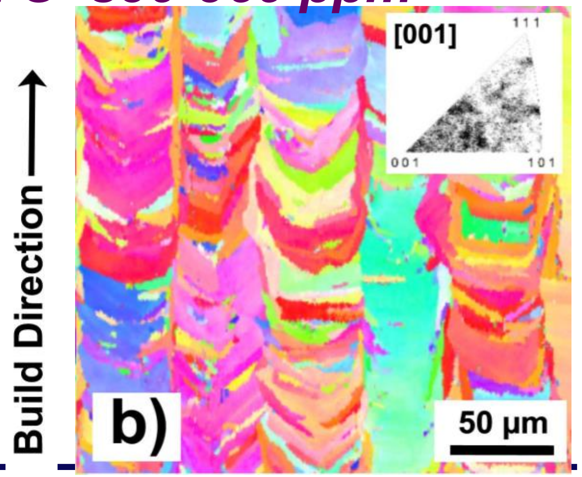
Few minor phases at GBs: N<600 ppm & C=50-390 ppm

Ar



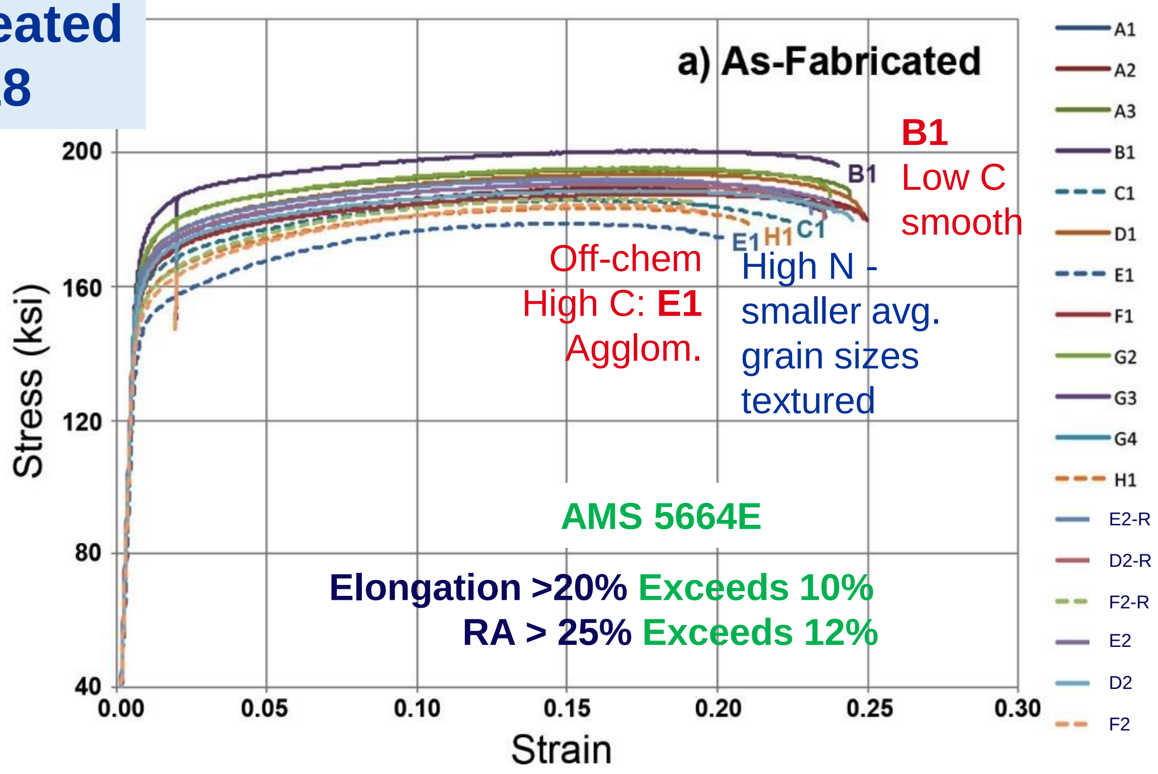
Minor phases at GBs: N>1000 ppm & C=390-960 ppm

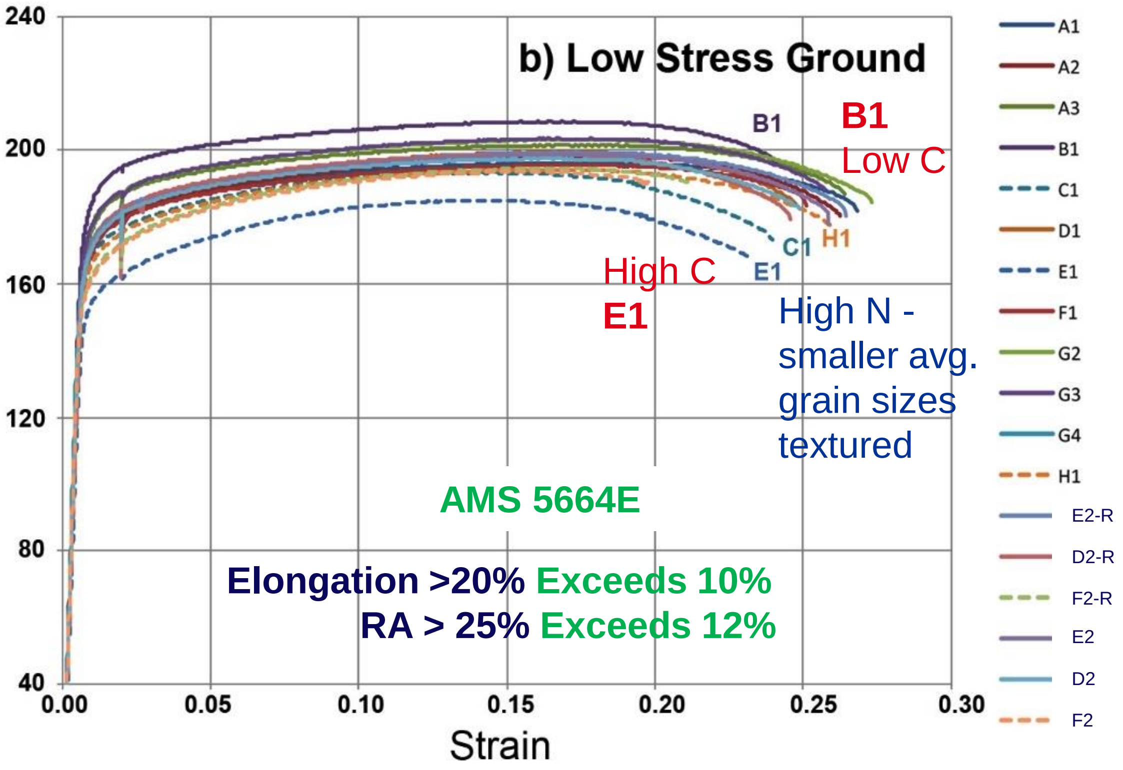
N



Tensile properties meet/exceed AMS5664, show comparable response with surface condition; relate to chemistry and microstructure

Heat treated SLM 718





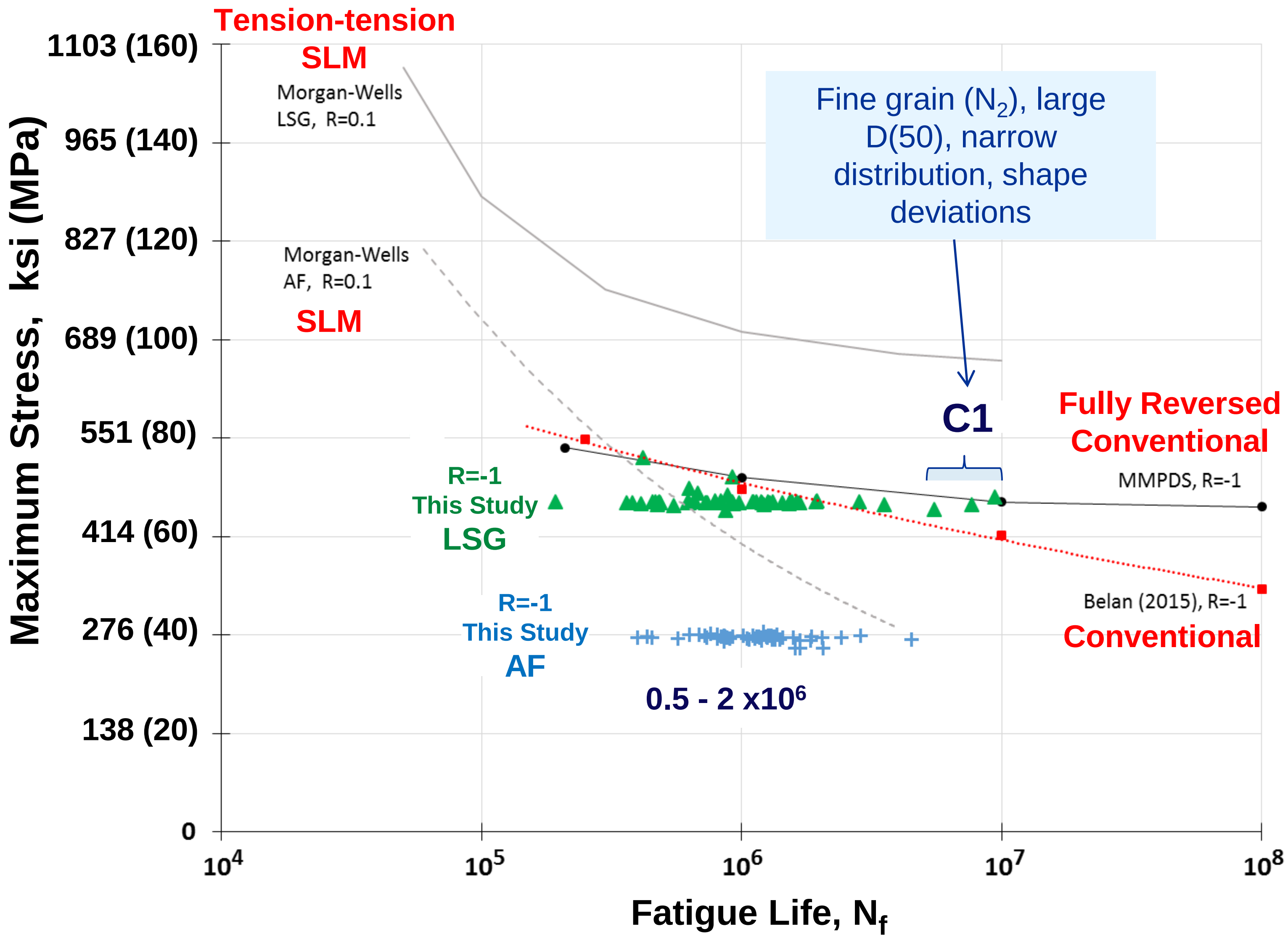
Room Temperature Tensile Testing

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

Room Temperature High Cycle Fatigue



<u>Low stress ground</u> compares well to wrought data

Statistical analysis shows that C1 is different, with single run outliers for B1 (high) and E2 (low)

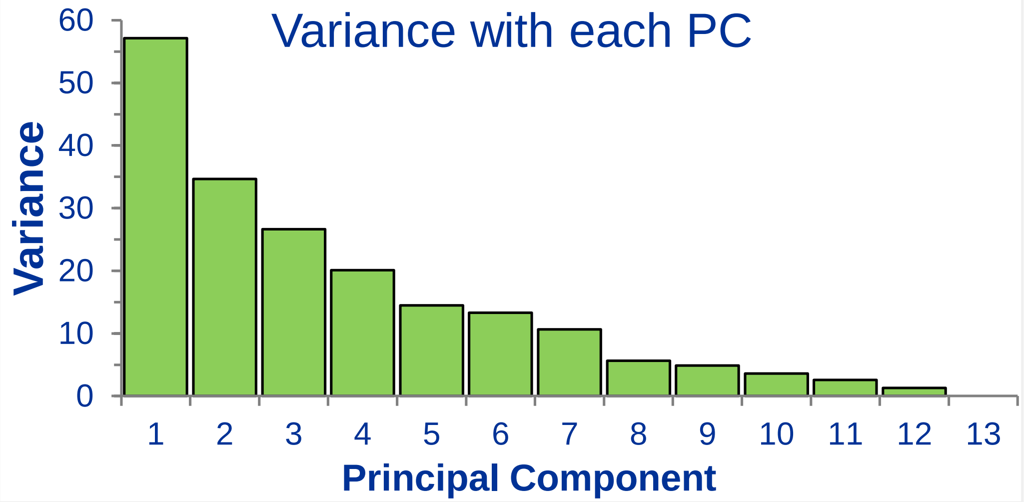
As Fabricated has less scatter, but 40% lower stress for comparable life

- **Crack initiation driven by rough surface due to SLM processing**

Full PCA Analysis

Full PCA Analysis of 194 Variables reduces to 12 Principal Components

Scree plot



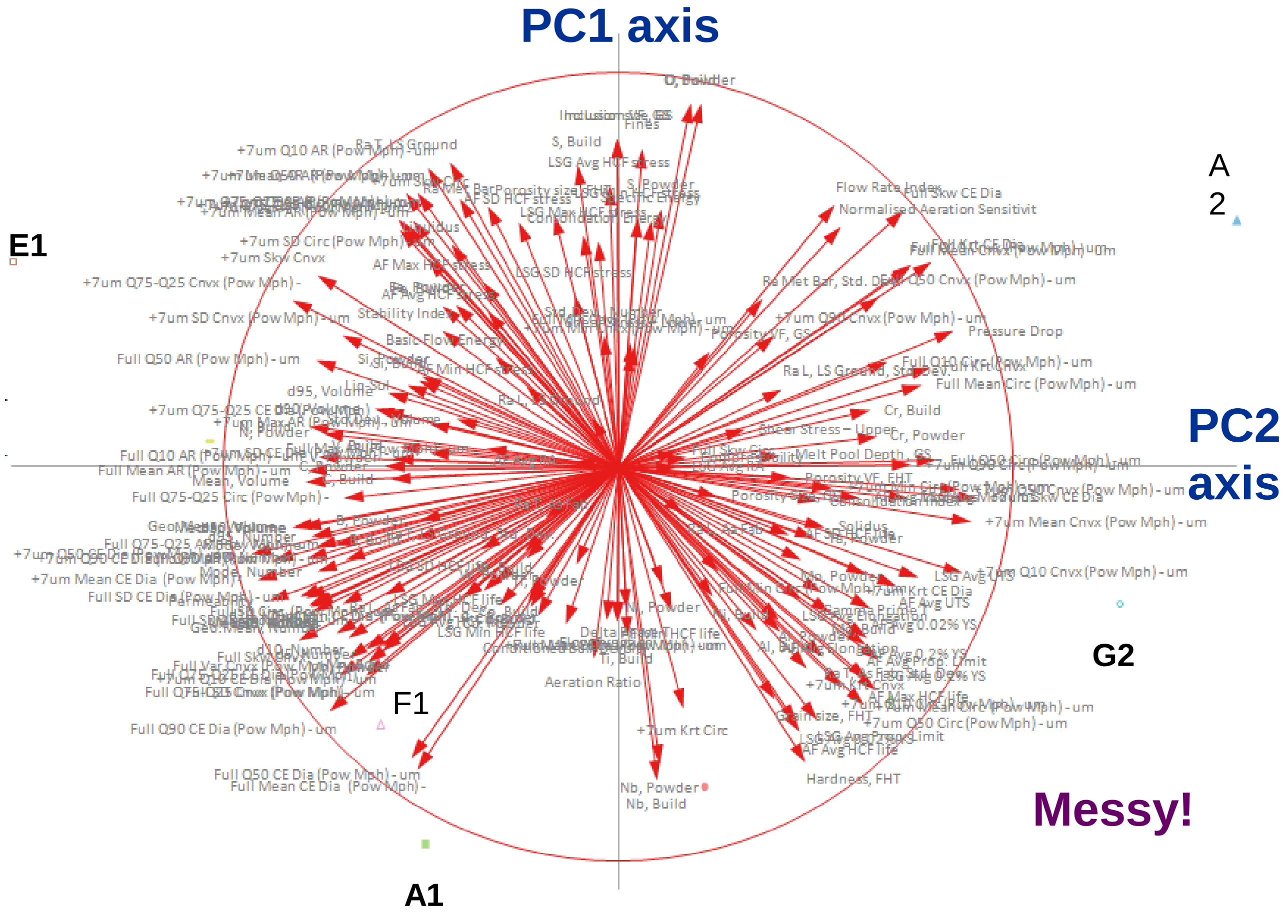
Component	Variance	Proportion	Cumulative proportion
1	57.140	0.293	0.293
2	34.648	0.178	0.471
3	26.658	0.137	0.607
4	20.121	0.103	0.711
5	14.472	0.074	0.785
6	13.325	0.068	0.853
7	10.661	0.055	0.908
8	5.645	0.029	0.937
9	4.880	0.025	0.962
10	3.584	0.018	0.980
11	2.575	0.013	0.993
12	1.291	0.007	1.000

Nearly half

98%

Higher order PCs comparable to a rounding error

Full PCA Plot



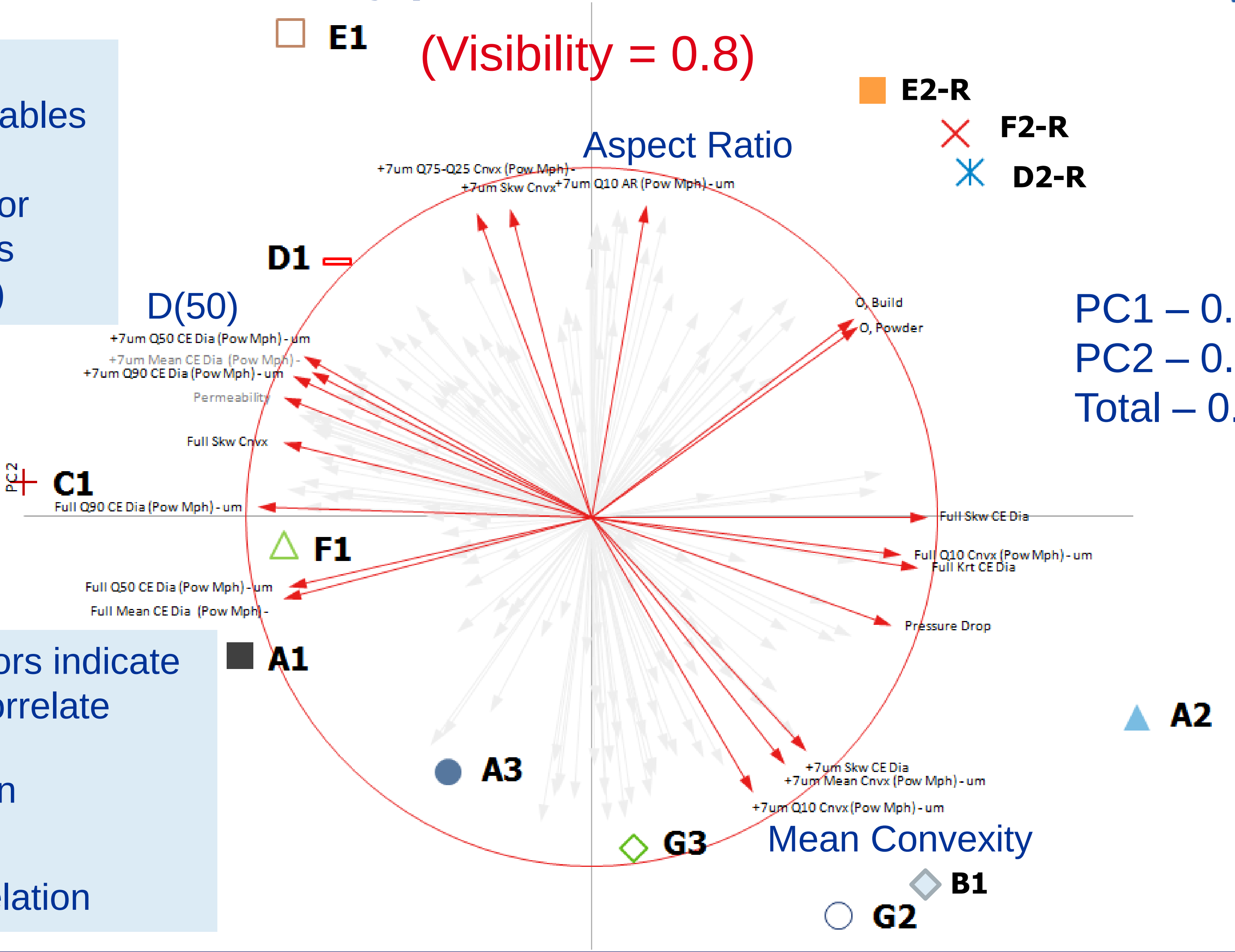
- Plot independent variables as (PC2, PC1) vectors
- Powders reduce to a single point as a vector summation
- Variables with high PC1 or PC2 character along the axes

Full PCA Analysis

Full PCA plot dominated by powder characteristics

1. **Set visibility**: a vector cutoff length to show significant variables

1. **Plots Rotated:** Longest vector points to the right at 0 degrees (**Here:** Skewness of Full PSD)



PC1 – 0.296
PC2 – 0.175
Total – 0.468

Orientation relationship of vectors indicate how the independent variables correlate

- Small angle – Strong correlation
- Perpendicular – No correlation
- Large angle – Strong anti-correlation

Full PCA Analysis

PC1 & PC2 character shows consistency with metallurgical experience

Principal Component 1

	Variable	PC1		Variable	PC1
	+7um Skw CE Dia	+ 0.120		d95, Number	− -0.103
	+7um Mean Cnvx (Pow Mph) - um	0.118		d5, Volume	-0.103
	+7um Q10 Cnvx (Pow Mph) - um	0.115		Mean, Number	-0.104
	+7um Q50 Cnvx (Pow Mph) - um	0.114	Nitrogen	N, Build	-0.104
Ease of air escape	Pressure Drop	0.112		d90, Number	-0.104
	Full Q50 Circ (Pow Mph) - um	0.106	Shape	Full Mean AR (Pow Mph) - um	-0.104
	Full Krt CE Dia	0.105		Mean, Volume	-0.105
	Full Krt Cnvx	0.104		d10, Volume	-0.105
	Full Mean Circ (Pow Mph) - um	0.102		Median, Volume	-0.107
Tensile	LSG Avg UTS	0.100		d50, Volume	-0.107
	Full Q10 AR (Pow Mph) - um	− -0.100		Full SD CE Dia (Pow Mph) - um	-0.107
	+7um Skw Cnvx	-0.101		Full Skw Cnvx	-0.107
	Mode, Number	-0.101		+7um Q75-Q25 Cnvx (Pow Mph) - um	-0.109
	Full Q50 AR (Pow Mph) - um	-0.101		Geo.Mean, Volume	-0.109
	Mode, Volume	-0.101		+7um Q90 CE Dia (Pow Mph) - um	-0.116
	Geo.Mean, Number	-0.102	Air flow resistance	Permeability	-0.117
	Median, Number	-0.103		+7um Mean CE Dia (Pow Mph) - um	-0.121
	d50, Number	-0.103		+7um Q50 CE Dia (Pow Mph) - um	-0.123

Principal Component 2

	Variable	PC2		Variable	PC2
Oxygen	O, Powder	+ 0.157	Dynamic flow	Specific Energy	+ 0.106
	O, Build	0.157		AF SD HCF stress	0.106
Nitrides	Inclusion VF, GS	0.141		+7um Q75-Q25 Circ (Pow Mph) - um	0.105
	Inclusion size, GS	0.141		+7um Var AR (Pow Mph) - um2	0.104
Fines	Fines	0.137		Normalised Aeration Sensitivity	0.104
Tensile	Ra T, LS Ground	0.131		+7um Mean AR (Pow Mph) - um	0.102
Sulfur	S, Build	0.130		LSG Max HCF stress	0.100
	+7um Q10 AR (Pow Mph) - um	0.129	Grain size	Grain size, FHT	− -0.100
	LSG Avg HCF stress	0.121		+7um Q50 Circ (Pow Mph) - um	-0.103
	+7um Mean AR (Pow Mph) - um	0.118		+7um Krt Circ	-0.104
	+7um Q50 AR (Pow Mph) - um	0.118		Full Q90 CE Dia (Pow Mph) - um	-0.106
	+7um Skw Circ	0.115	Tensile	LSG Avg Prop. Limit	-0.109
Dynamic flow	Flow Rate Index	0.113	Tensile	LSG Avg 0.02% YS	-0.110
Roughness	Ra Met Bar	0.112	Fatigue life	AF Avg HCF life	-0.114
	S, Powder	0.111		Full Q50 CE Dia (Pow Mph) - um	-0.125
	Full Skw CE Dia	0.110	Hardness	Hardness, FHT	-0.127
Porosity	Porosity size, FHT	0.109	Strengtheners	Nb, Powder	-0.128
	LSG Min HCF stress	0.108		Full Mean CE Dia (Pow Mph) - um	-0.131
Shape	+7um Q75-Q25 AR (Pow Mph) - um	0.107		Nb, Build	-0.135

Powder characteristics dominate PC1

- Size and shape distributions show largest variability, as does resistance to air flow that relates packing density
- Captures anti-correlation with UTS and N content observed

More variables / relationships influence PC2

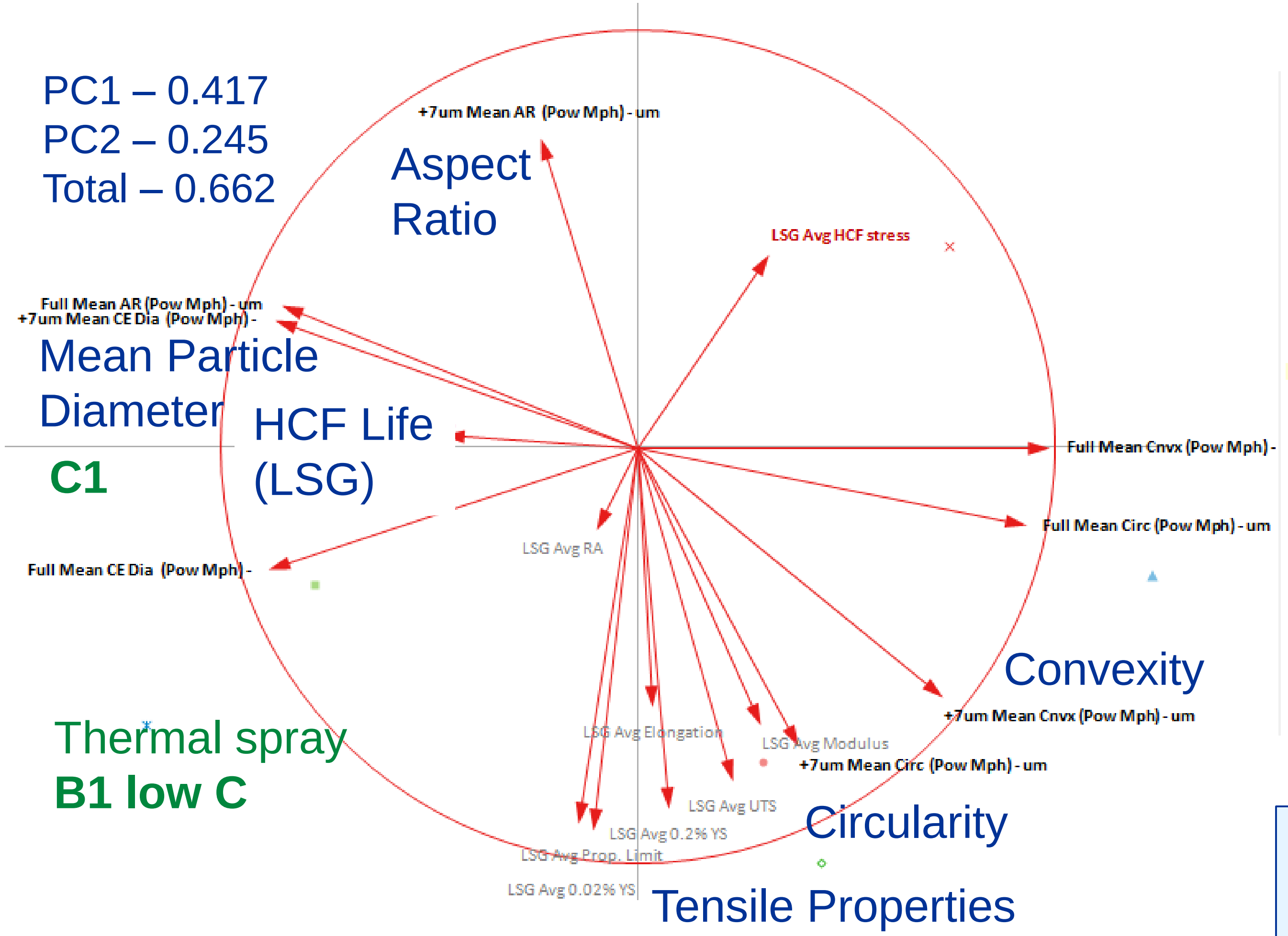
- Microstructure (grain size, nitrides, Nb-rich γ'') vs Tensile
- Roughess ↑, Fatigue life ↓ in bars with AF surfaces
- Powder Production: O, S, AR, fines ↑, HIP/HT pore size ↑

National Aeronautics and Space Administration

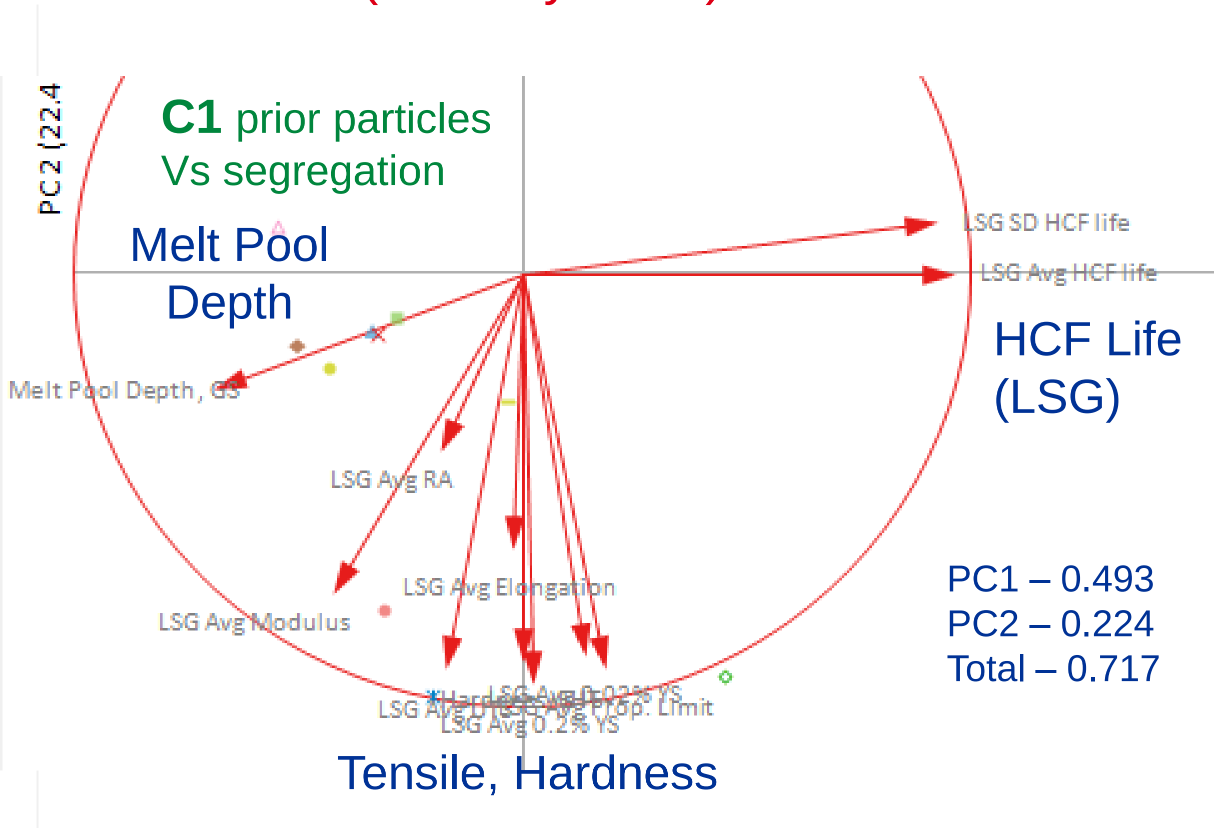
Subset PCA Analyses

Powder Characteristics - Mechanical Properties
(Visibility = 0.6)

PC1 – 0.417
PC2 – 0.245
Total – 0.662



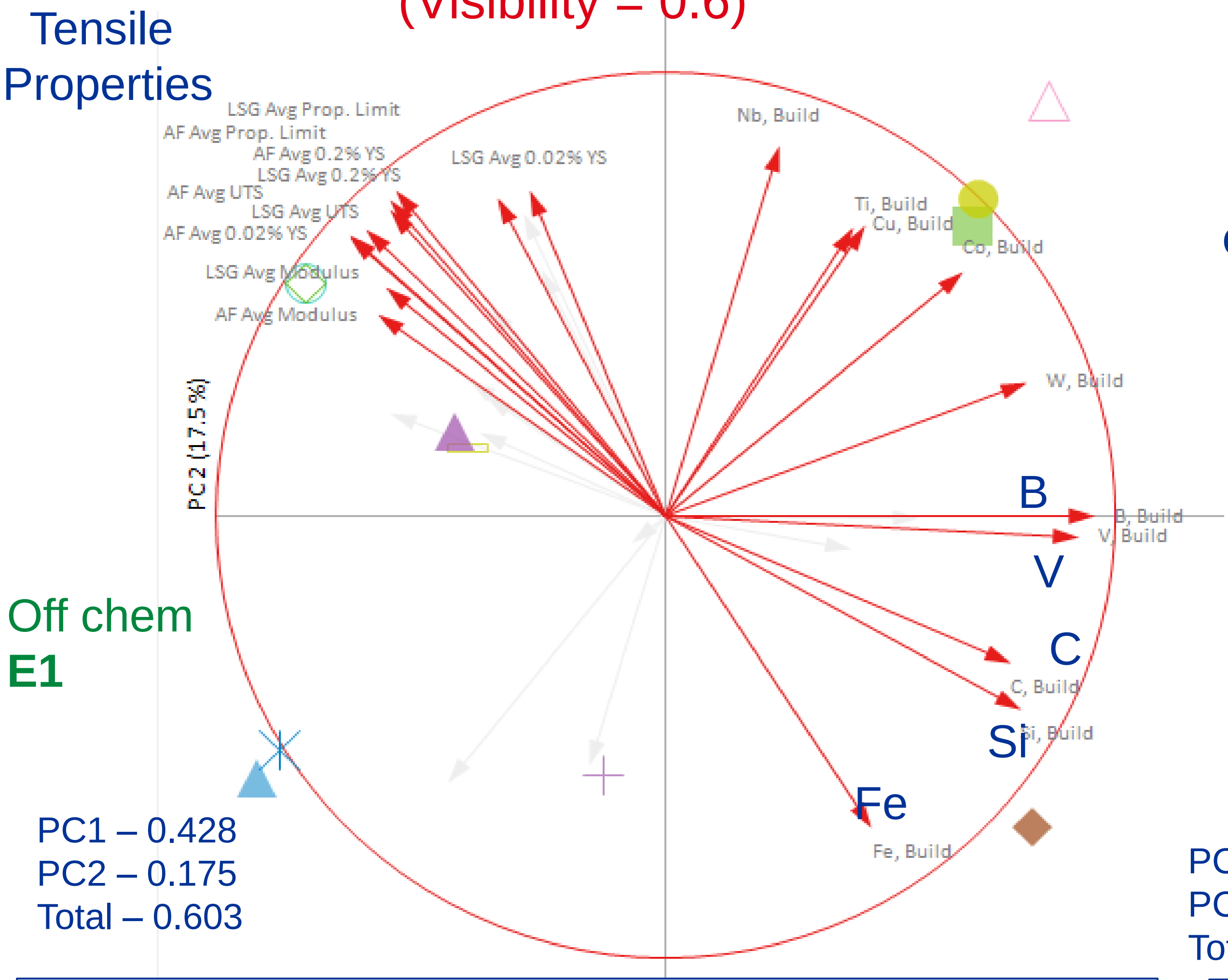
Melt Pool Depth - Mechanical Properties
(Visibility = 0.6)



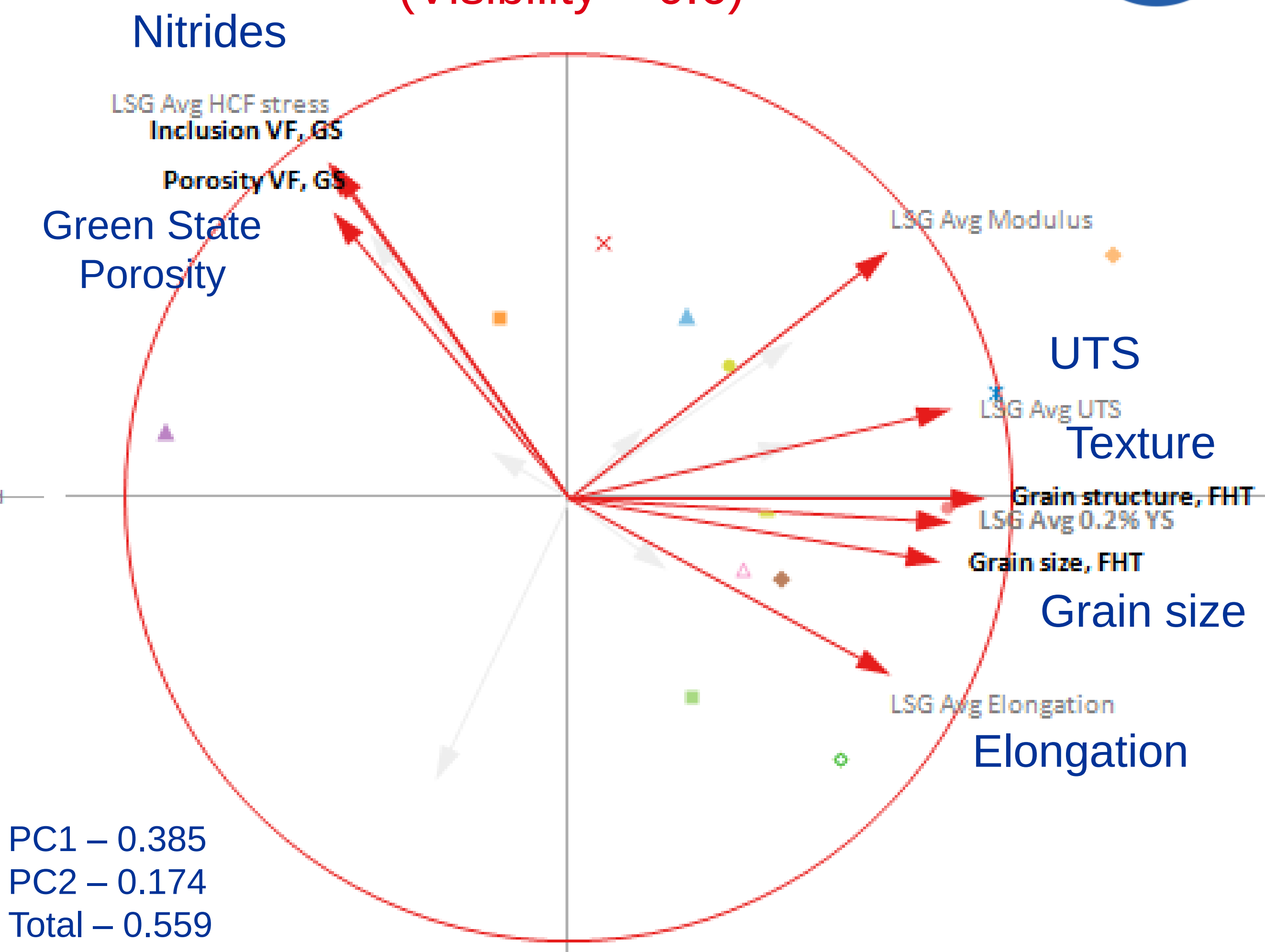
- Poor explanation of HCF properties by PC1 & PC2
- Strong correlation of tensile properties to circularity and convexity; anti-correlation to particle aspect ratio
- Correction of HCF Life (LSG) to D(50)

- Melt pool depth (MPD) is our only direct measurement of the SLM process
- **Strong anti-correlation of HCF Life to MPD**
- Weak correlation of tensile and hardness to MPD: stronger with modulus and reduction in area

Build Chemistry - Tensile Properties
(Visibility = 0.6)



Microstructure - Tensile Properties
(Visibility = 0.6)



- Strong anti-correlation of tensile with Fe, Si, C
- Weaker anti-correlation of tensile to V, B
- No to little correlation of tensile to Nb, Cu, Co, Ti, W

- Strong correlation of Tensile to Grain Size and Texture
- Strong anti-correlation of Elongation with Nitrides and As-built Porosity

Summary and concluding remarks

- **Powders characteristics (size, shape, rheology) show wide variability that dominated full PCA analysis of 194 variables that reduced to 12 principal components, where PC1 and PC2 capture nearly half the variability in the data set**
- **Supplied powders have a wide variety of characteristics that led to reasonable properties**
- **Compositional differences have the strongest impact on SLM 718 microstructure and mechanical properties**
  - Fully heat-treated builds from **N_2-atomized powders have fine SLM structure that is highly textured**. TiN-nitrides and MC carbides present on the GBs that suppresses recrystallization during HT
  - The **B1 alloy with higher delta-phase had the highest UTS** due to a very low C content, while the **E1 alloy exhibited the lowest UTS and was off chemistry** with very low in Al and high in C
  - For LSG surface condition, **the best room temperature HCF was for N_2-atomized C1** with prior GB particles (TiN, Nb-based carbides) that persist through heat treatment
- **PCA analysis of the entire data set was able to highlight groups of important variables that provided general guidance consistent with metallurgical experience**
- **PCA subset analysis showed some interesting relationships between mechanical properties and processing, microstructure, chemistry, and powder characteristics**

In-Progress Research at NASA GRC

- **Five powder lots selected for a further investigation:** ***B1, C1 (N), G2, G3, H1***

- **Comparable 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**

- **Recycling Study: 40+ powder reuse builds**
  - **Limited powder characterization between each build**
  - **Control and defect-seeded samples with *in situ* monitoring by AMSense profilometer**
  - **Tensile**

Information about the current program can be directed to Dr. Cheryl Bowman at Cheryl.L.Bowman@nasa.gov

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

Project Coordination
- Chantal Sudbrack, Team Lead
- Cheryl Bowman, Team Lead
- Brian West

Powder Characterization
- Richard Boothe
- David Ellis
- Alejandro Hinojos (OSU)
- Chantal Sudbrack

MSFC AM Fabrication
- James Lydon
- Omar Mireles
- Ken Cooper

Analytical Characterization
- Rick Rogers
- Dereck Johnson
- Joy Buehler

Heat Treat & Machining
- Will Tilson
- MSFC Heat Treat Facility
- GRC Specimen Shop

Microstructural Evaluation
- Ivan Locci
- Tim Smith
- Chantal Sudbrack
- Alejandro Hinojos (OSU)
- Michael Kloesel (Cal Poly)
- Bethany Cook (CWRU)
- Jonathan Healy (CWRU)

Mechanical Testing
- Brad Lerch
- Aaron Thompson
- Jonathan Woolley
- GRC Testing Facility

Fractography
- Paul Chao (CMU)
- Ben Richards (NU)
- Ivan Locci

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

Published: Proceedings Superalloy 718 & Derivatives: E. Ott et al (Eds.), TMS (Pittsburgh), 89-113 (2018)