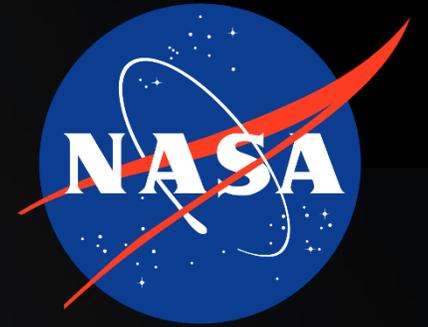


National Aeronautics and Space Administration



Metal Additive Manufacturing for Rocket Engines: Successes and Failures

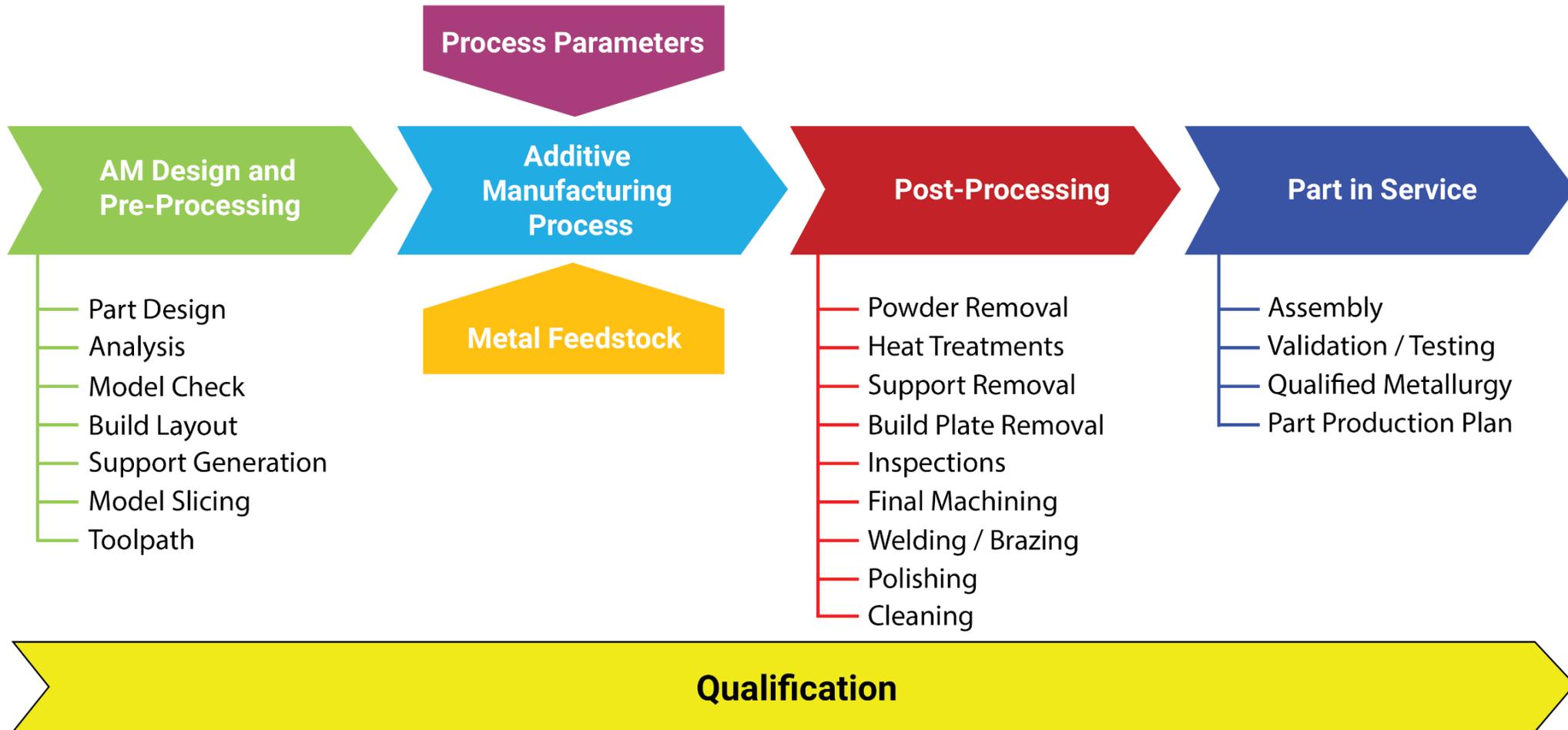
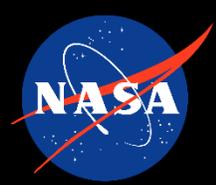
Paul Gradl

National Aeronautics and Space Administration (NASA)

21 March 2023



Additive Manufacturing Typical Process Flow



Proper AM process selection requires an integrated evaluation of all process lifecycle steps



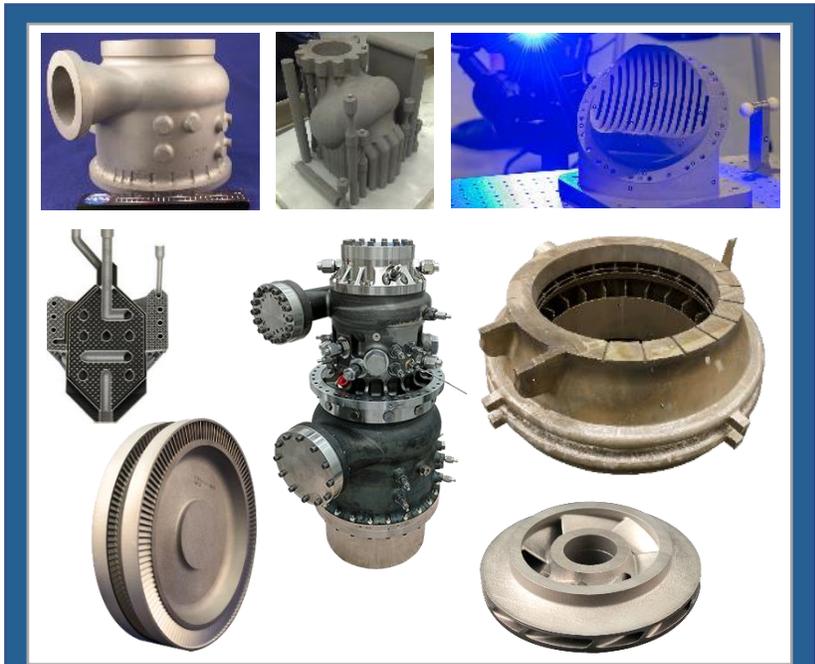
Metal Additive Manufacturing Development for Rocket Engines



Laser Powder Bed Fusion (L-PBF)
Copper Alloys combined with other
AM processes to provide bimetallic



Directed Energy Deposition

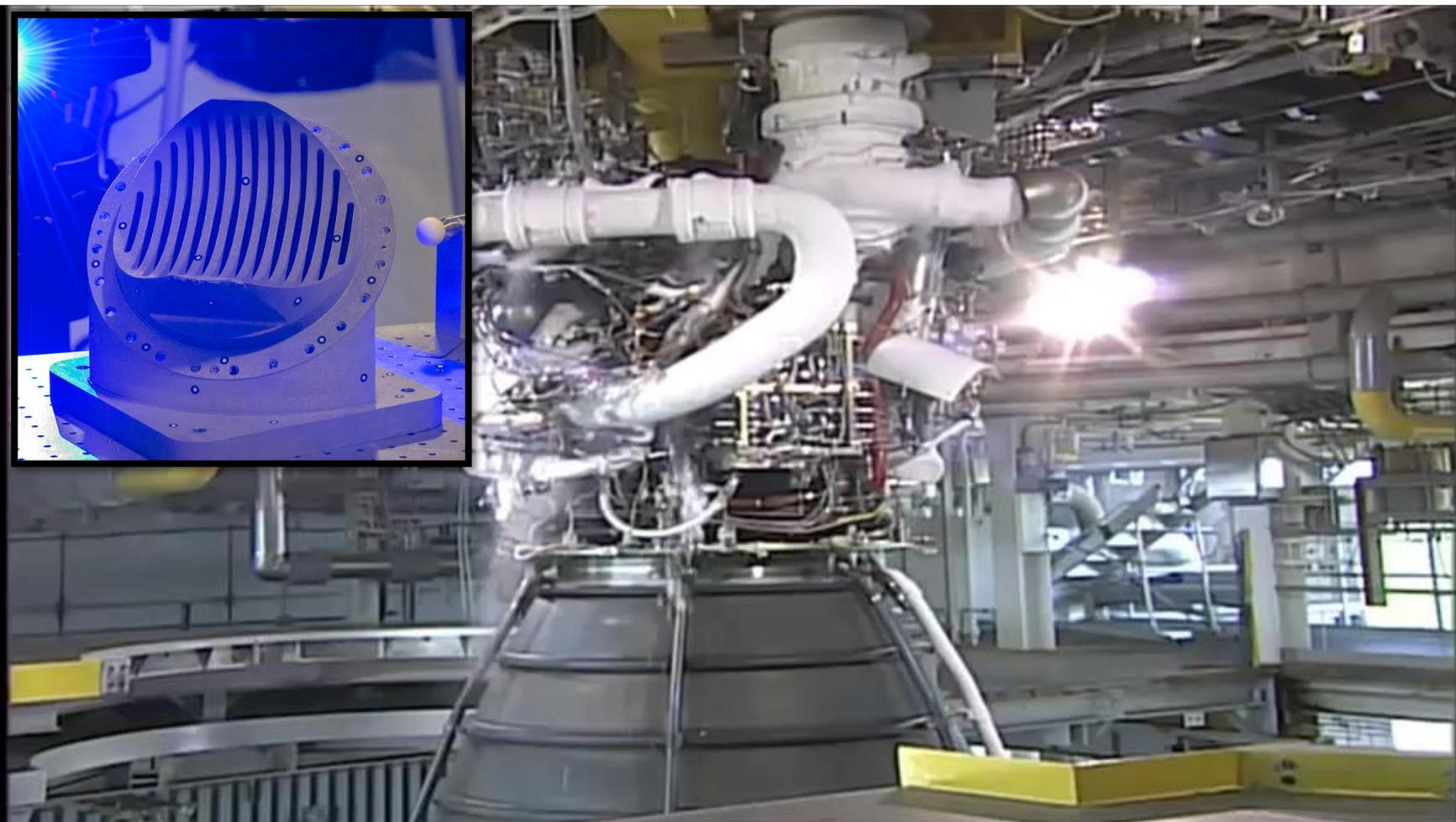


L-PBF of complex components, new
alloy developments for harsh
environment



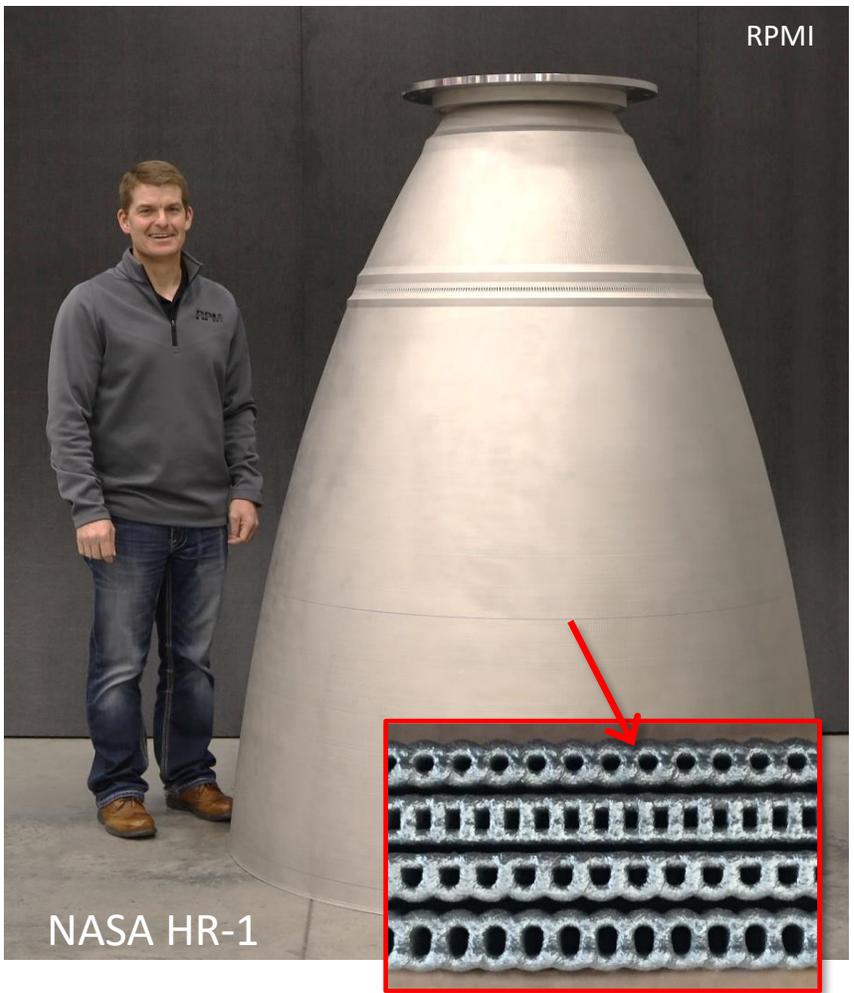


Additive Manufacturing in use on NASA Space Launch System (SLS)



**Successful hot-fire testing of full-scale additive manufacturing (AM) Part to be flown on SLS RS-25
RS-25 Pogo Z-Baffle – Used existing design with AM to reduce complexity from 127 welds to 4 welds**

Large Scale LP-DED Nozzle Development



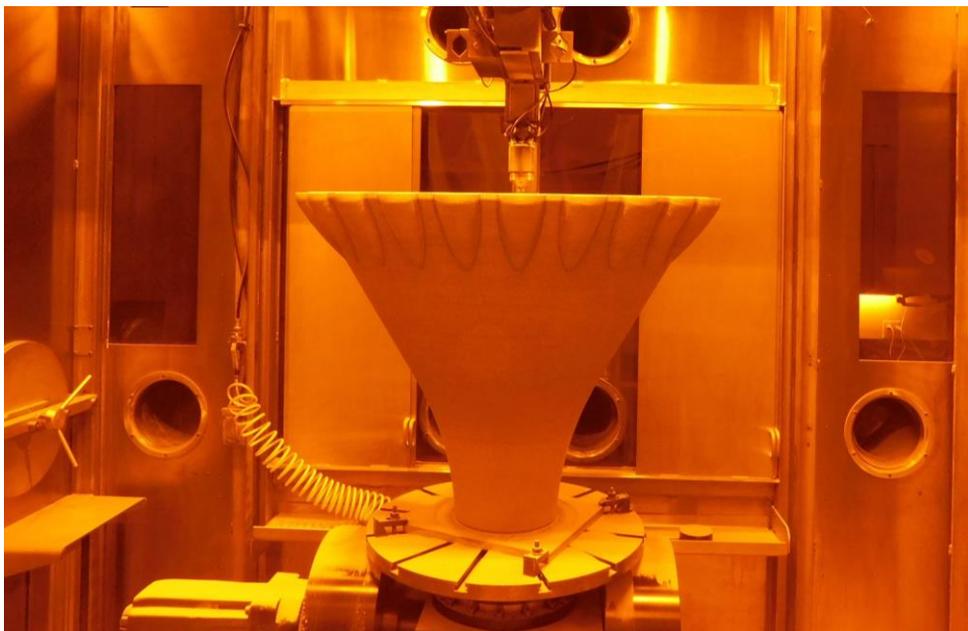
60" (1.52 m) diameter and 70" (1.78 m) height with integral channels
90 day deposition



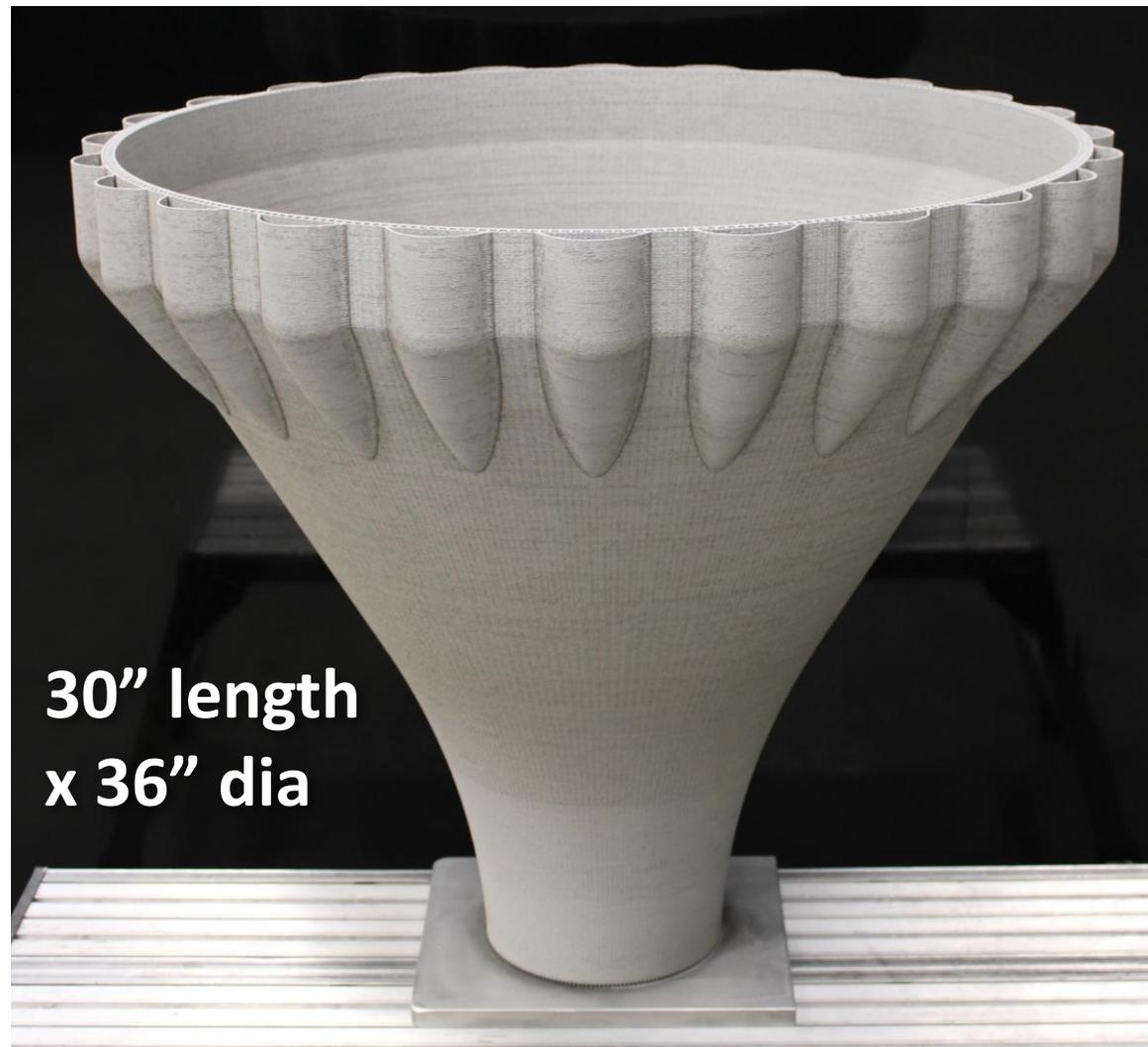
95" (2.41 m) dia and 111" (2.82 m) height
Near Net Shape Forging Replacement

Reference: P.R. Gradl, T.W. Teasley, C.S. Protz, C. Katsarelis, P. Chen, Process Development and Hot-fire Testing of Additively Manufactured NASA HR-1 for Liquid Rocket Engine Applications, in: AIAA Propuls. Energy 2021, 2021: pp. 1–23. <https://doi.org/10.2514/6.2021-3236>.

Demonstrator Aerospike LP-DED Nozzle



LP-DED Aluminum 6061-RAM2

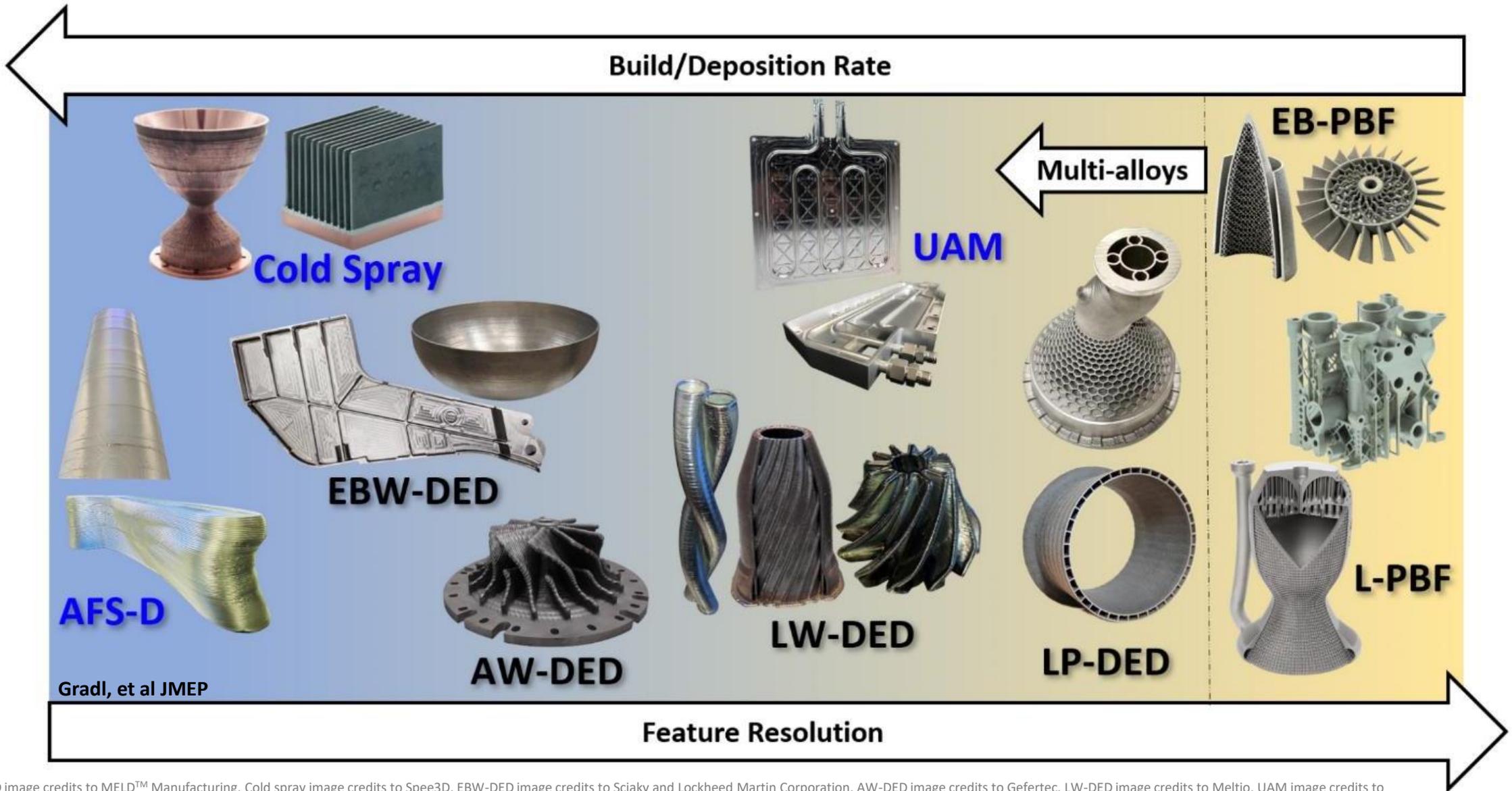


30" length
x 36" dia





Criteria and Comparison Various Metal AM Processes



CREDITS: AFS-D image credits to MELD™ Manufacturing, Cold spray image credits to Spee3D, EBW-DED image credits to Sciaky and Lockheed Martin Corporation, AW-DED image credits to Gefertec, LW-DED image credits to Meltio, UAM image credits to Fabrisonic and NASA JPL, LP-DED image credits to DEPOZ project led by IRT Saint-Exupery and Formally, L-PBF image credits to Renishaw plc and CellCore GmbH/Sol Solutions Group AG, EB-PBF image credits to Wayland and GE Additive/Arcom.

Multi-metallic and multi-process development

Credit: RPMI



L-PBF Liner / LP-DED Jacket



L-PBF Liner / Coldspray Jacket



L-PBF Liner / EBW-DED Jacket



**Direct deposit LP-DED nozzle
(Axial Bimetallic)**



L-PBF GRCop-42 to Inco 625





NASA's Effort on New Alloy Development



Max. Use Temp. (°C)	Alloy Family	Purpose	Novel AM Alloys	Propulsion Use
200	Aluminum	Light weighting	-	Various
750	Copper	High conductivity; strength at temperature	GRCop-42 GRCop-84	Combustion Chambers
800	Iron-Nickel	High strength and hydrogen resistance	NASA HR-1	Nozzles, Powerheads
900	Nickel	High strength to weight	-	Injectors, Turbines
1100	ODS Nickel	High strength at elevated temp; reduced creep	GRX-810 Alloy 718-ODS	Injectors, Turbines
1850	Refractory	Extreme temperature	C-103, C-103-CDS, Mo, W	Uncooled Chambers



GRCop-42 L-PBF



NASA HR-1 LP-DED



GRX-810 L-PBF



C103 L-PBF

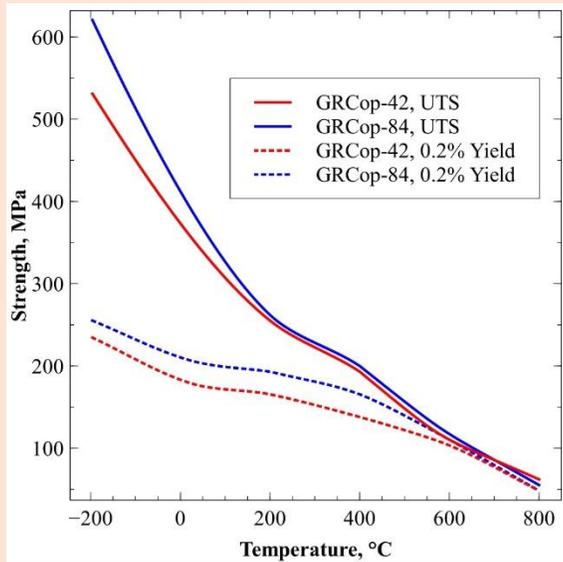
New alloy development using various additive manufacturing processes (PBF and DED) can yield performance improvements over traditional alloys



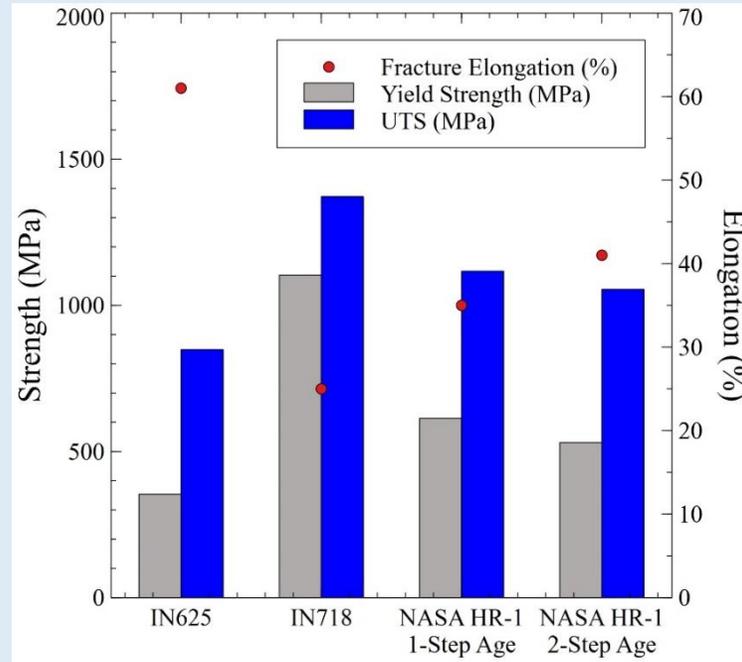
AM Enabling New Alloy Development



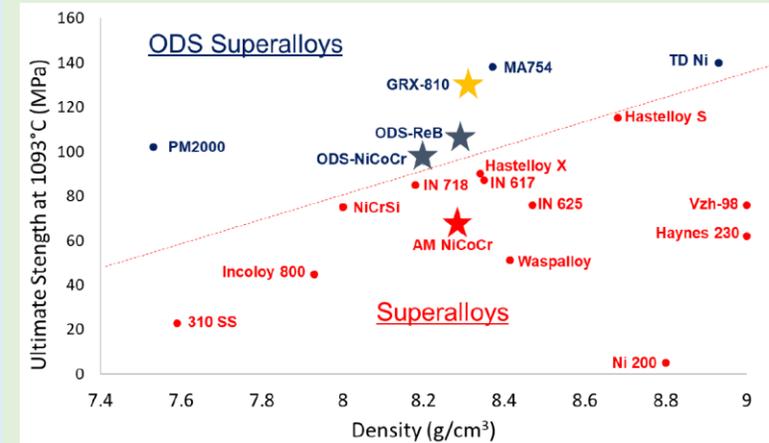
GRCop-42, High conductivity and strength for high heat flux applications



NASA HR-1, high strength superalloy for hydrogen environments



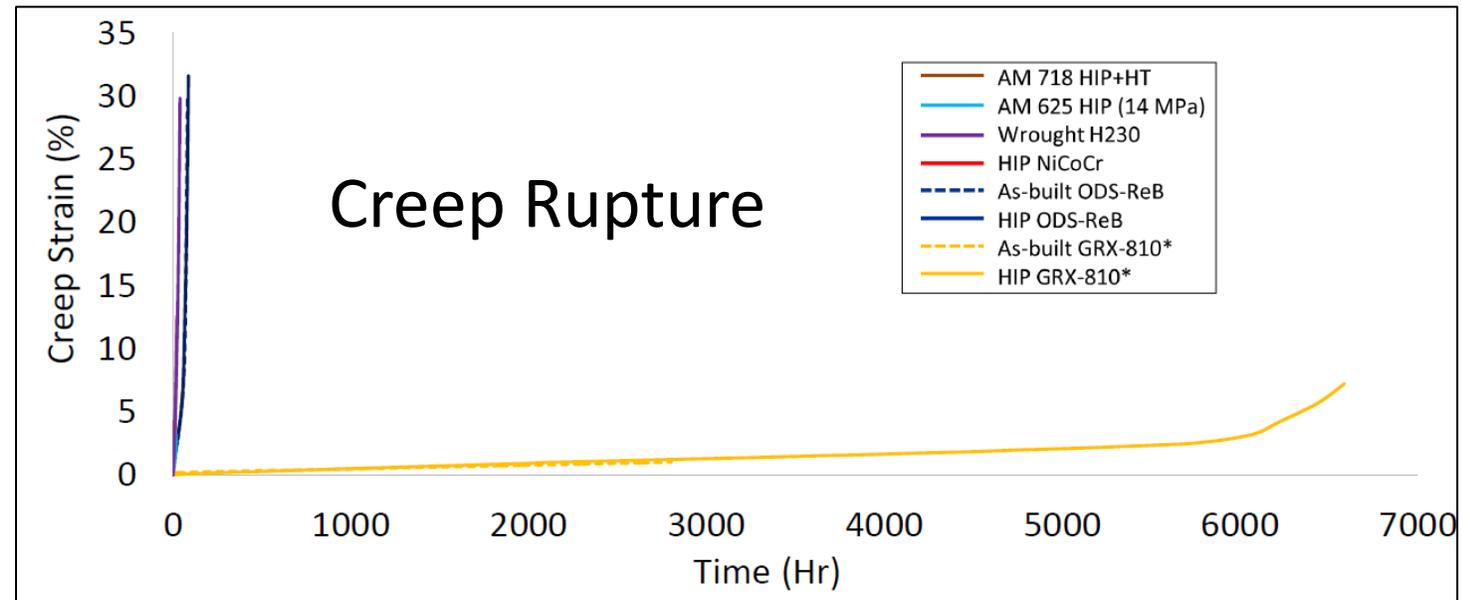
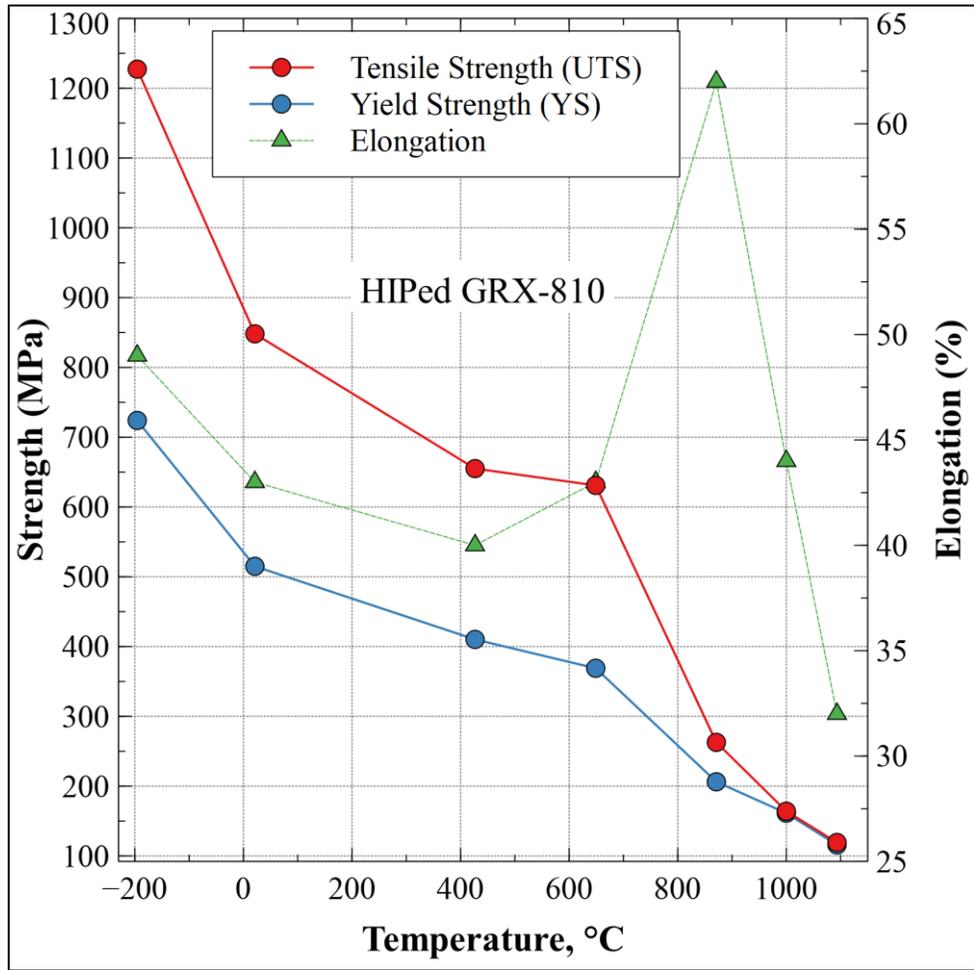
GRX-810, high strength, low creep rupture and oxidation at extreme temperatures



Ref: Tim Smith, Christopher Kantzos / NASA GRC 12



GRX-810 Oxide Dispersion Strengthened (ODS) Alloy



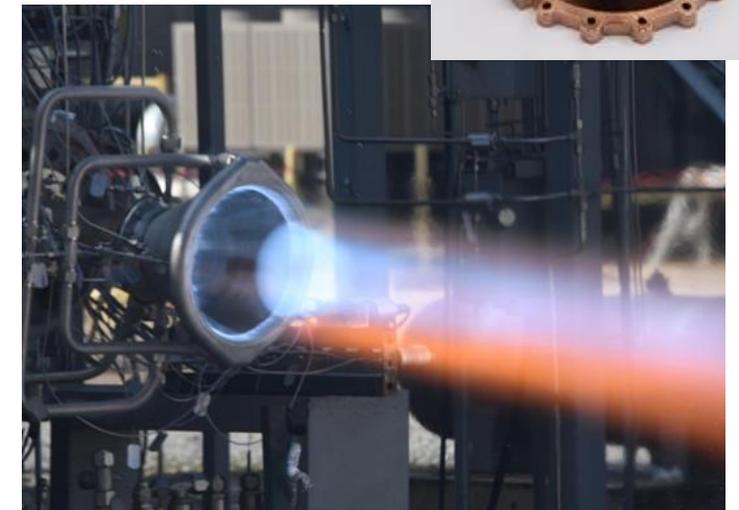




LLAMA Project Overview



- **Long Life Additive Manufacturing Assembly (LLAMA) project goals**
 - Fabrication and testing of additively manufactured high duty cycle (>50 starts) lander engine hardware (highly instrumented).
 - Determine performance of GRCop-42 alloy used for the thrust chamber.
 - Disseminate data to industry partners.
- **GRCop-42**
 - Copper-based (Cu-Cr-Nb) alloy w/ Cr₂Nb precipitates for dispersion strengthening.
 - Developed for high heat flux environments such as combustion chambers.
- **LLAMA hardware and test program**
 - Laser Powder Bed Fusion (L-PBF) GRCop-42 chambers.
 - Carbon-Carbon (C-C) and Direct Energy Deposition (DED) NASA HR-1 nozzles.
 - Additively manufactured L-PBF injectors (Alloy 625 and 718).
 - Two separate test phases completed in February 2021.





LLAMA Hardware Overview



L-PBF GRCop-42 Chambers



DED of Integral Channels

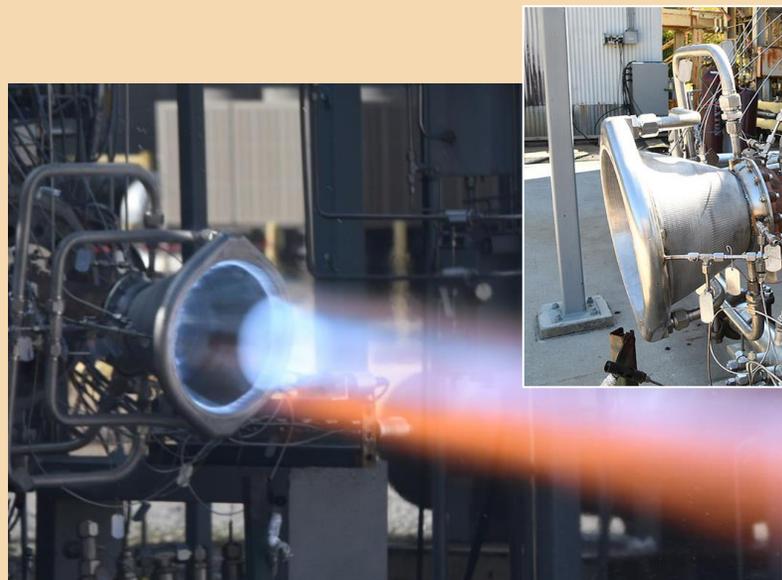
Final DED Regen Nozzle



Carbon-Carbon Nozzles



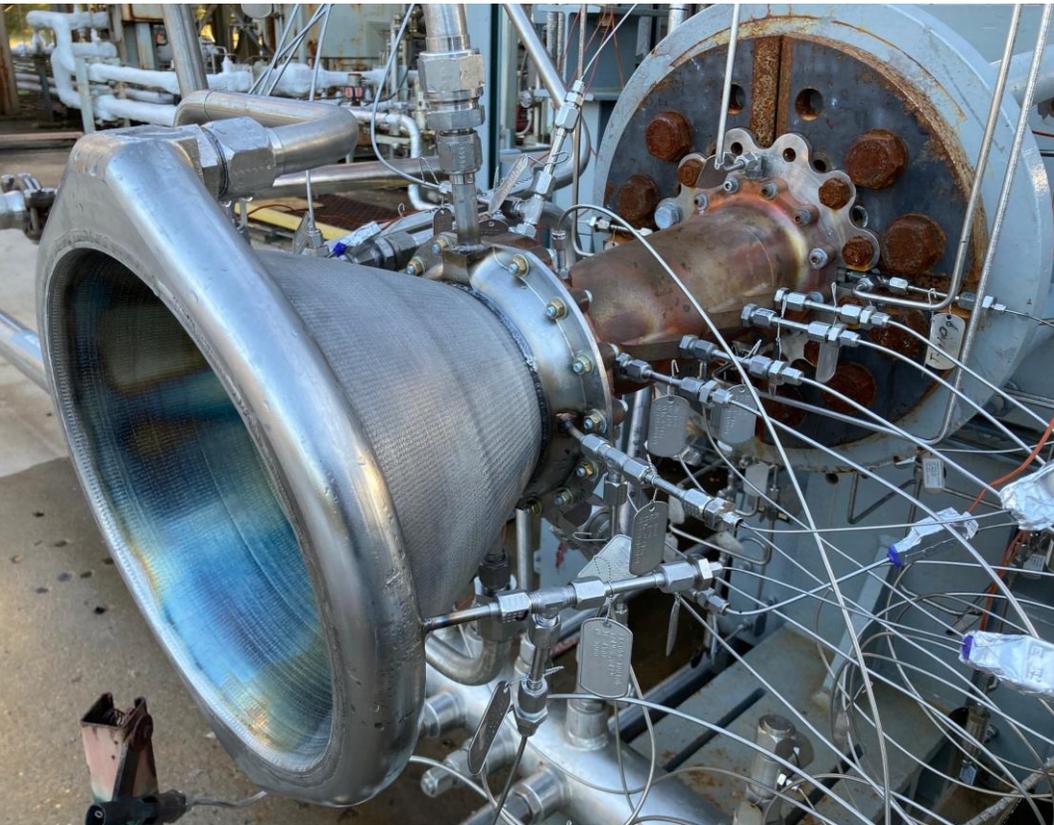
Additive Injectors



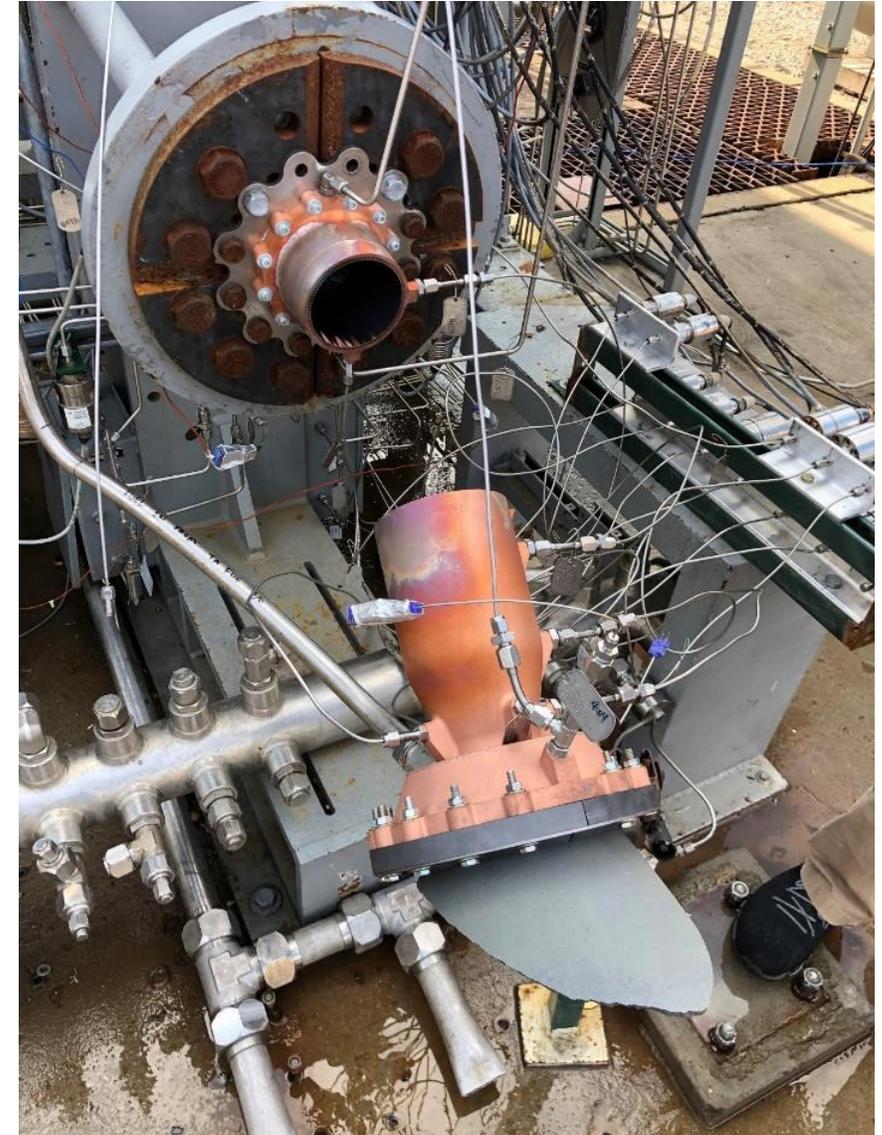
7k-lb_f GRCop-42 chamber and Composite Nozzle

Successful Test of “Sister” Chamber – 51 starts

L-PBF GRCop-42 chamber from same build plate and identical processing successfully tested 51 starts and 1,000 seconds



- Tested in early 2021
- Location: MSFC Test Stand 115
- L-PBF GRCop-42 chamber
 - 8 starts and 83.3 seconds total before separation.
 - No issues observed in prior chamber test data.
- Carbon-Composite experimental nozzle
 - Untested and possessed a noticeable crack.
 - Deemed an acceptable risk for test.

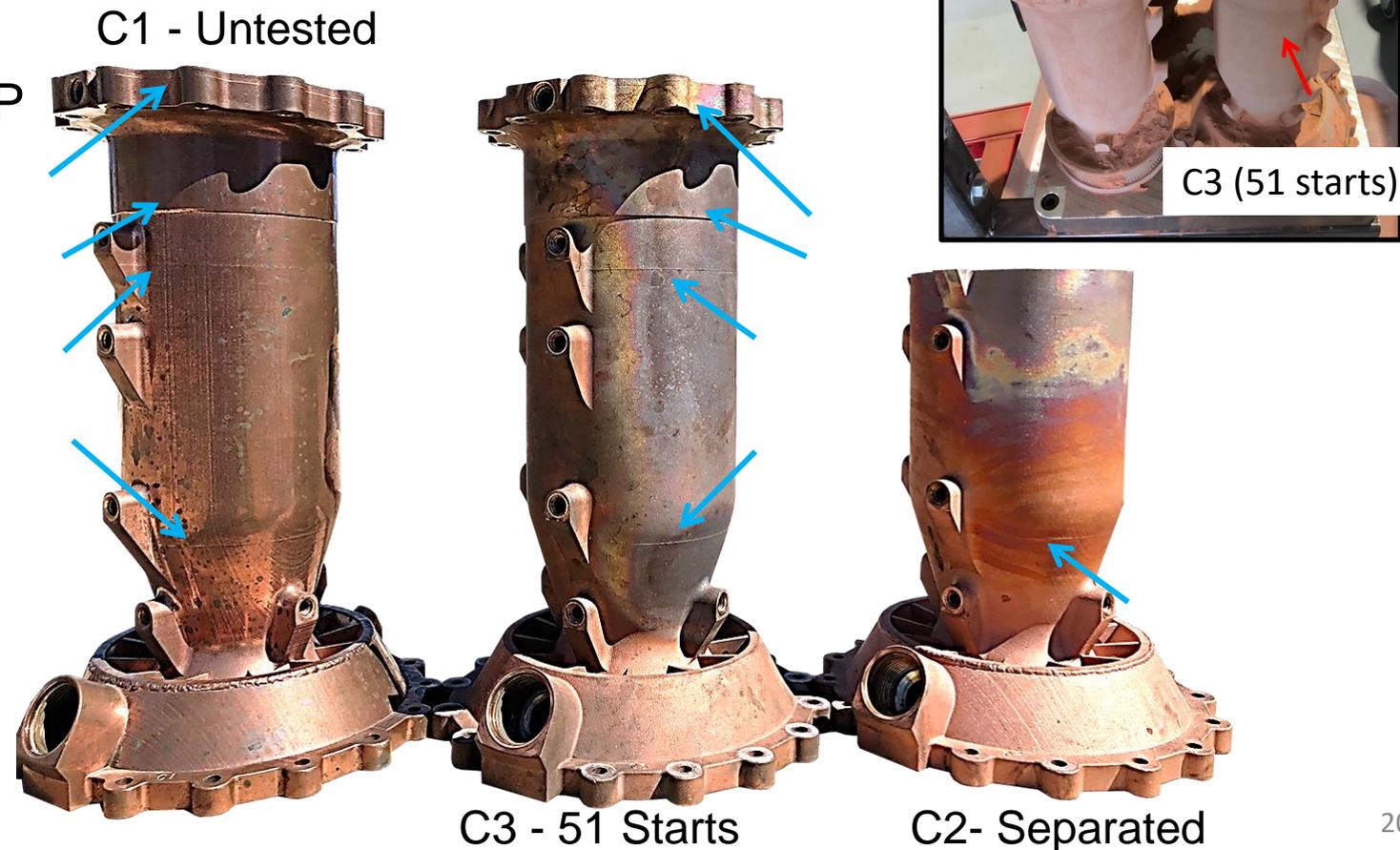




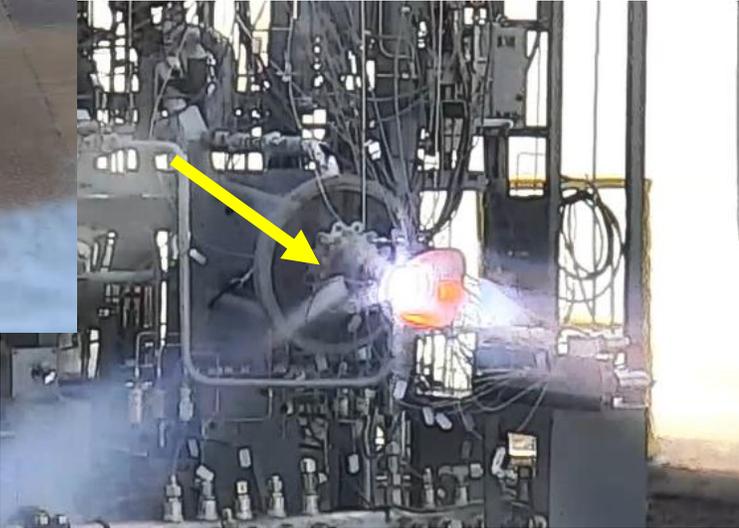
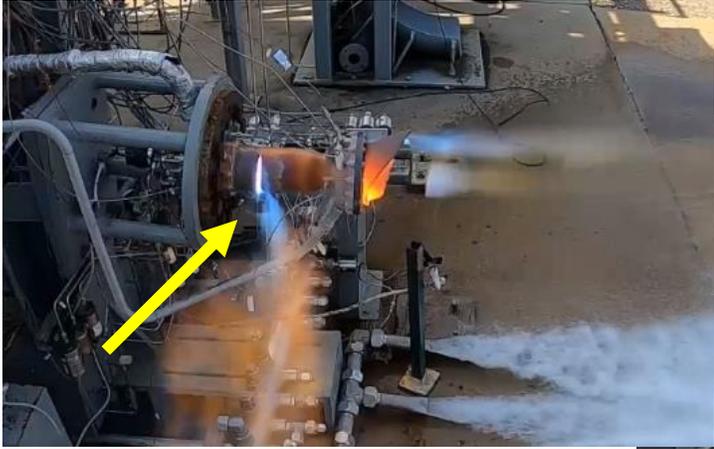
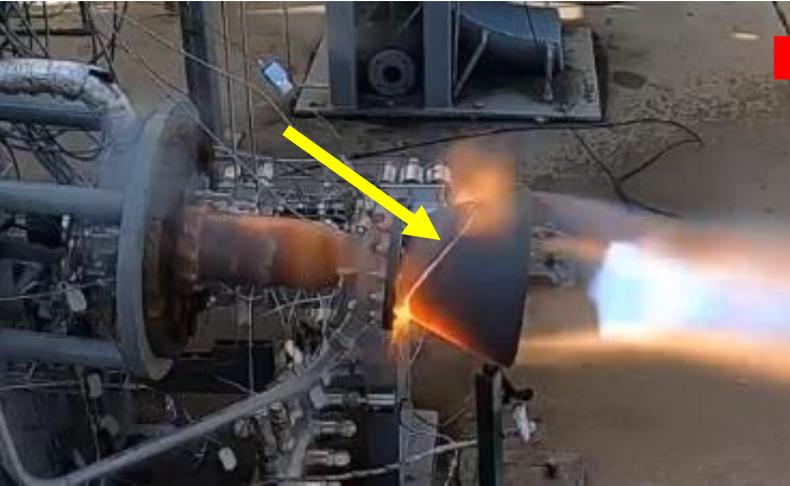
Multiple L-PBF Chambers Built and Tested



- EOS M400 L-PBF printer
 - Certified GRCop-42 powder lot
 - 4 chambers on the build
 - 3 w/ identical designs for LLAMA
- Computed Tomography Scans
 - No observations from data prior to HIP
 - Did not specifically look for witness lines – focused on powder removal verification
- Post-processing
 - C1 – HIP, EB weld manifold, exterior polishing
 - C2 – HIP, EB weld manifold
 - C3 – HIP, EB weld manifold, chemically milled



Anomaly Timeline

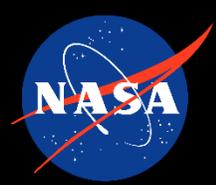


Separated Chamber





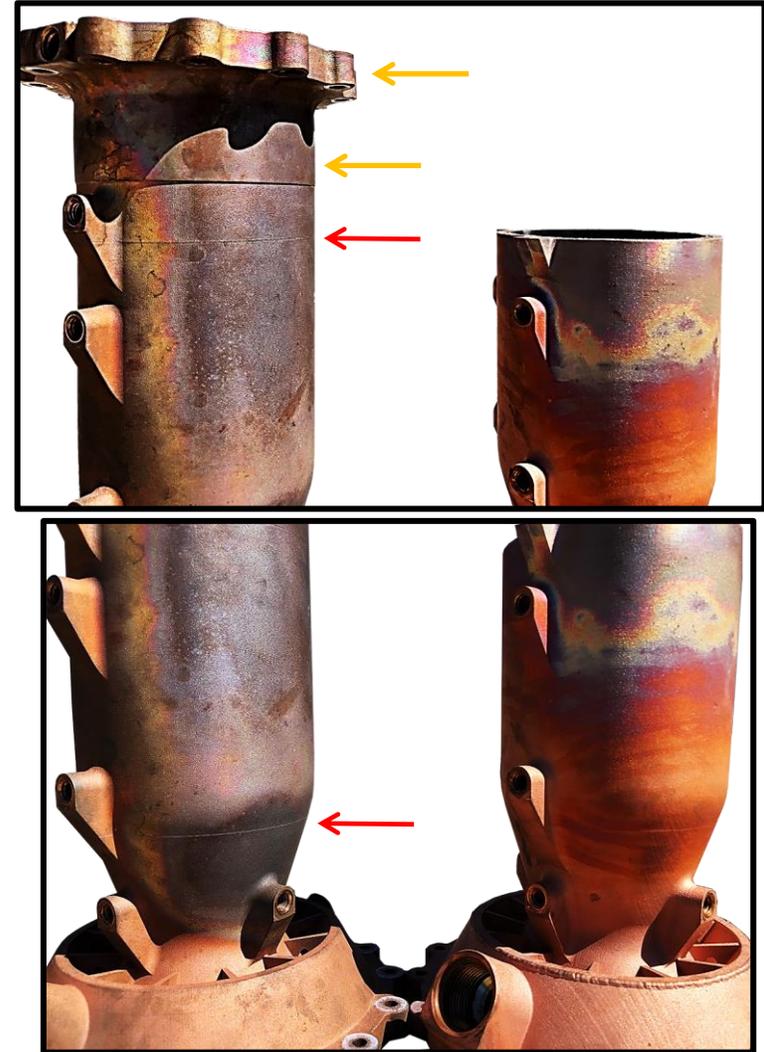
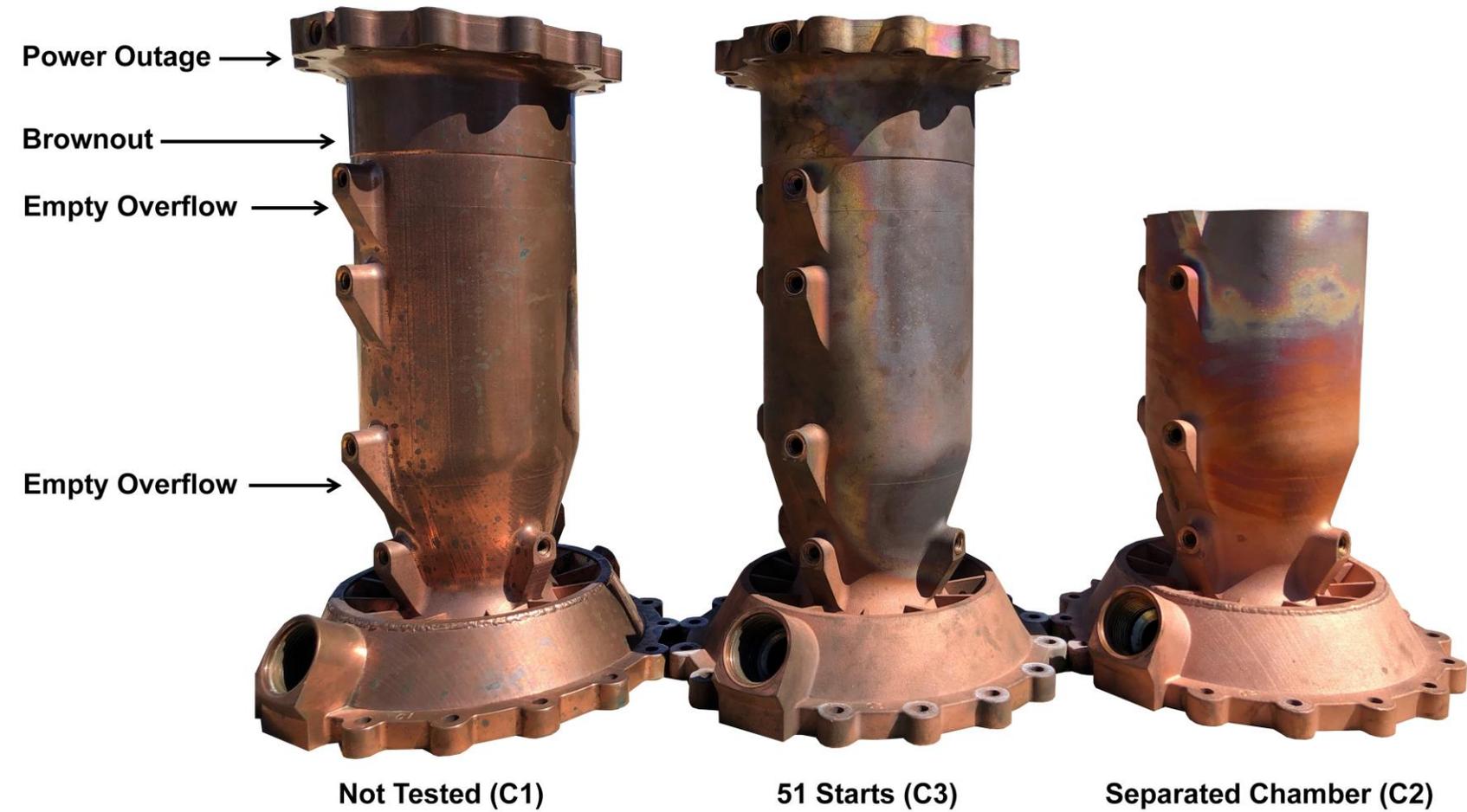
Chamber Process Evaluation



Goal: Determine the root cause and impacts of build interruptions that occur following stop and start during the chamber build process. Areas of concern were at witness lines (chamber with 51 cycles) and separation (back-up chamber) during testing.

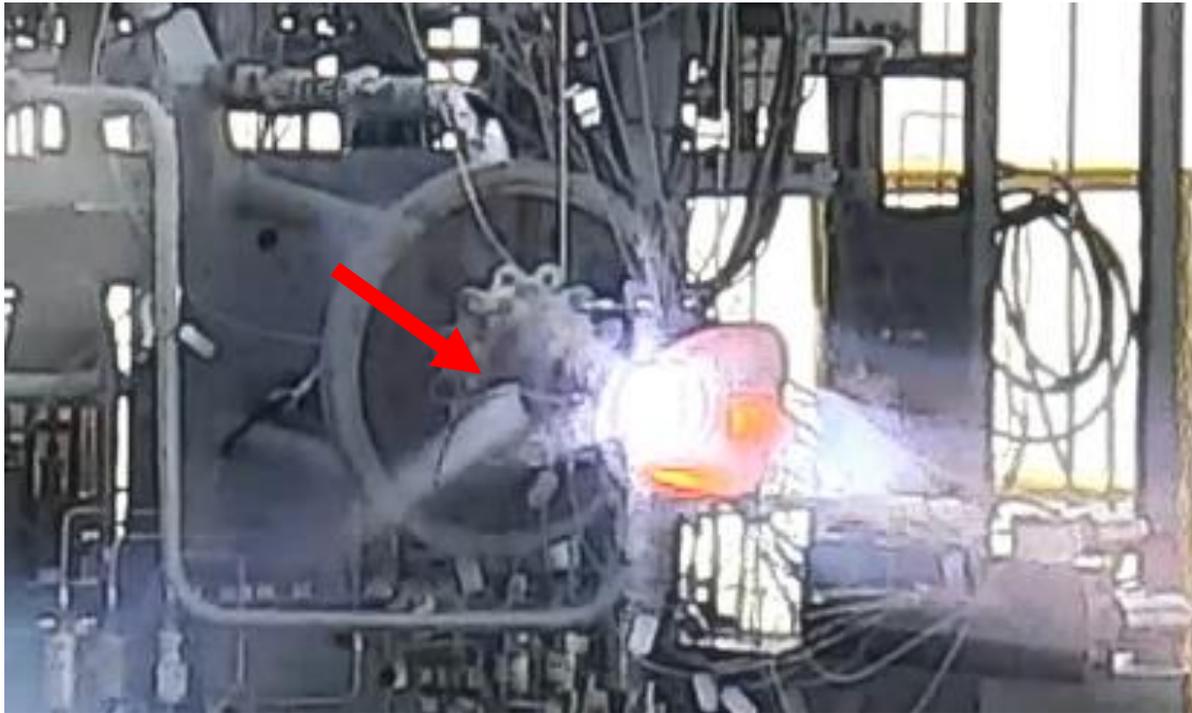
1. Created a plan to evaluate this process issue.
2. Evaluated fabrication process and build records of the chambers.
3. Completed NDE and evaluations of chambers.
 - Digital images, microscopic images, and measurements for **witness line** reference locations.
 - Fracture surface of chamber C2 examined for any noticeable defects or fracture points.
 - MSFC NDE team completed CT scan of all three chambers.
4. Sectioned chambers for metallography.
5. Completed metallography to understand region of concern and “good” regions.
 - Fabricated tensile and fatigue specimens with **representative witness lines**. (*full length specimens from original build not available*)
6. Completed tensile and fatigue testing of material from chambers (micro) and witness specimens.
7. Completed fractography and microstructural characterization of (sectioned) chambers and samples.
8. Reporting of lessons learned and recommendations.

Witness Lines Matched to Build Timeline



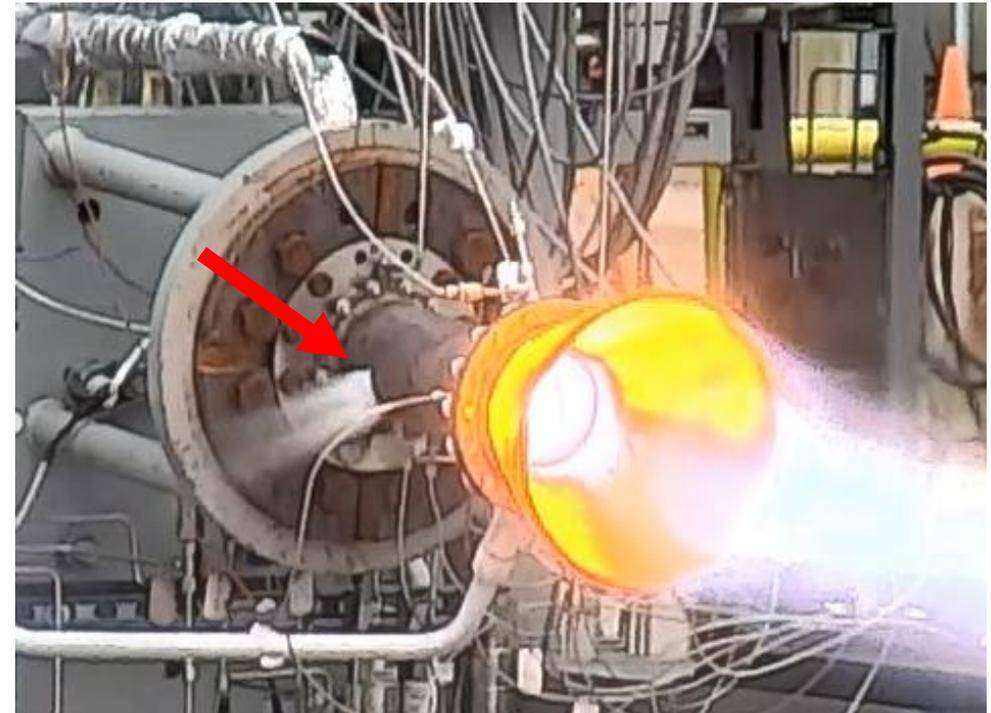
C3 & C2 Separation Comparison

PK129 Test 18



C2 - separated chamber (9 starts)

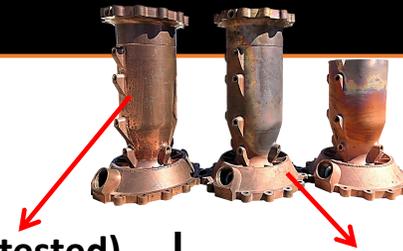
PK129 Test 13



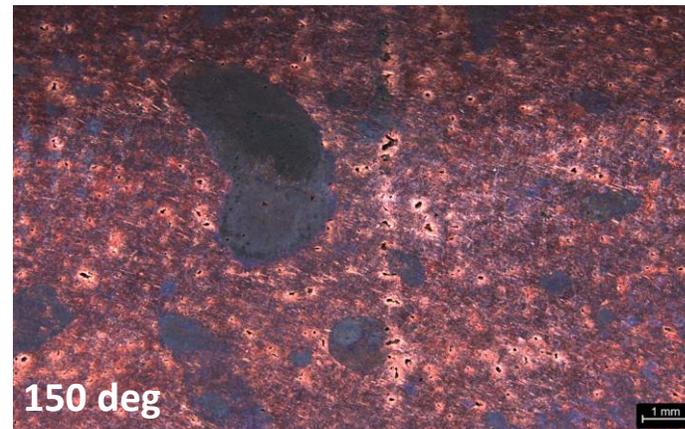
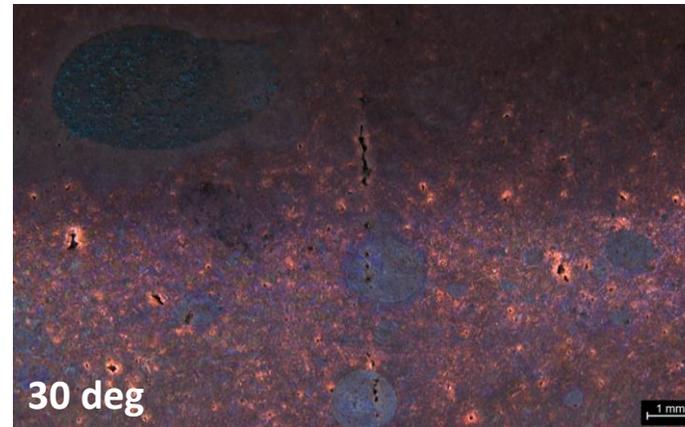
C3 - 51 start chamber (full life cycle)

Optical Images of Chambers Post-Test

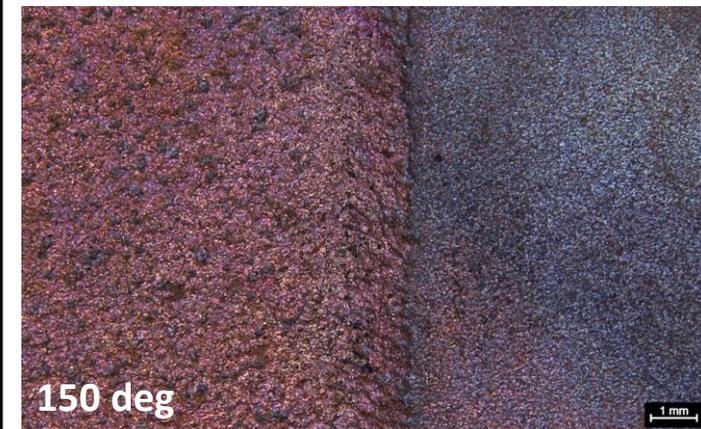
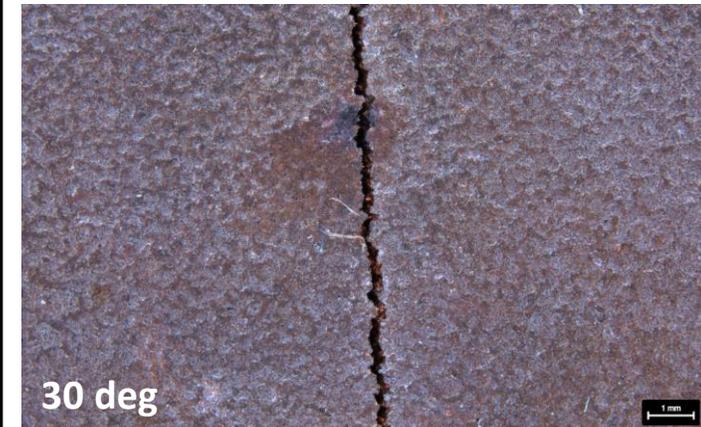
- Unpolished external surfaces.
- Top (power outage) and bottom (powder overflow) witness lines did not appear to have many detrimental defects.
- Middle witness line on chamber 1, there were some large lack of fusion defects that appeared to line up with the restart line.
- Chamber 3 – no defects visible at the surface beyond the crack that had already developed after test.



Middle witness line: C1 (untested)
Surface was polished using CMP

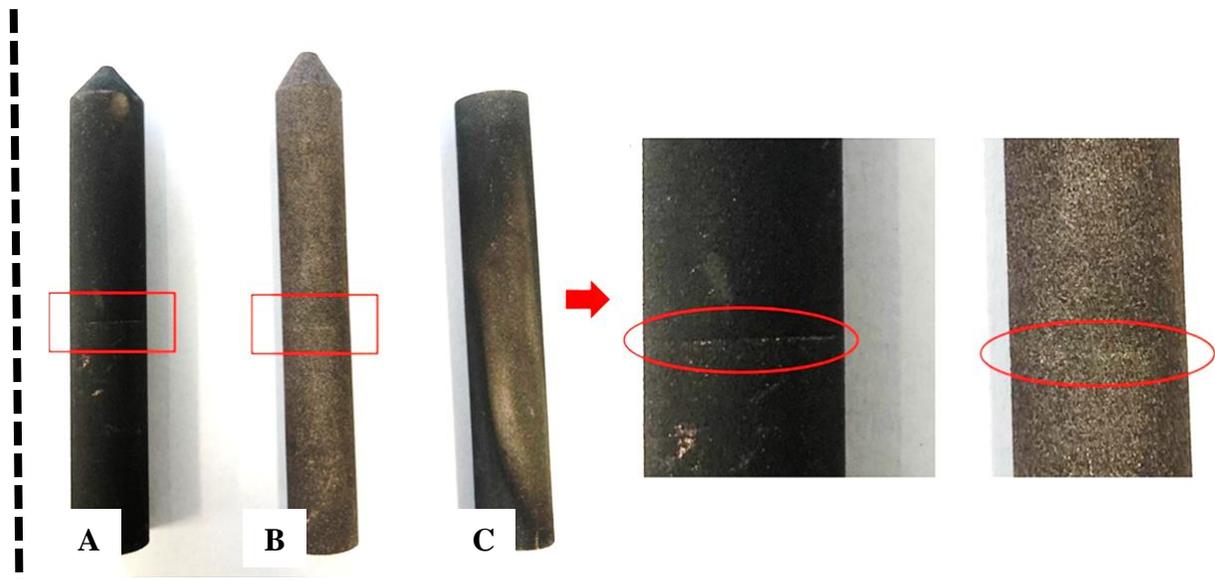
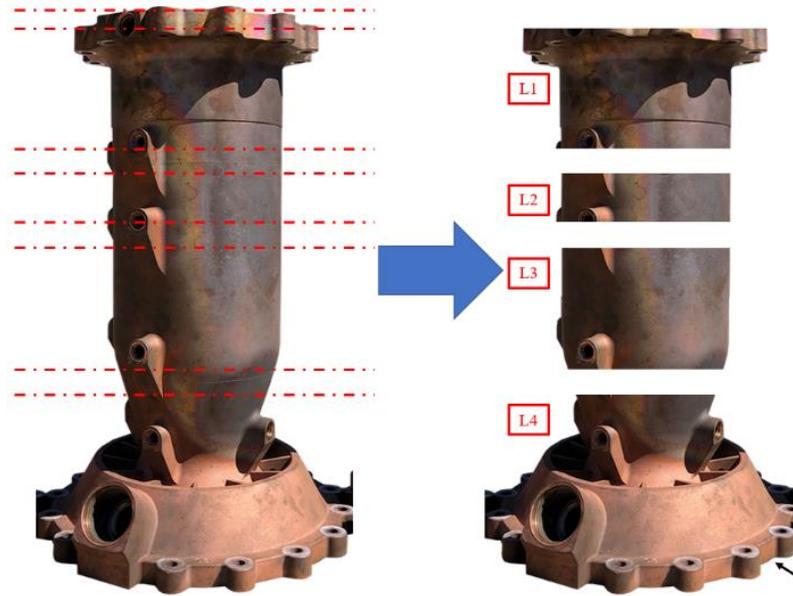


Middle witness line: C3 (51 starts)



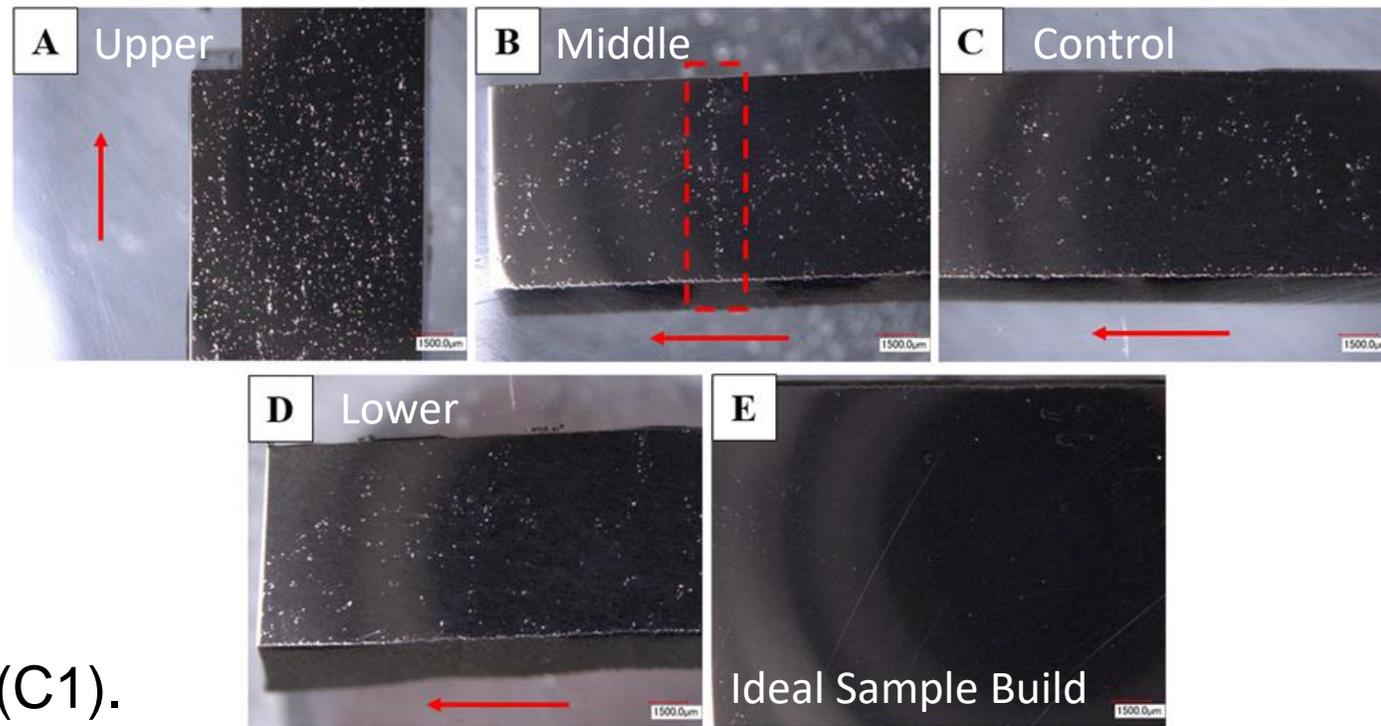


Test Specimens: Chamber Sectioning, Test Bars



Bar	Chamber Restart Replicated	Witness Line Replicated	Restart
A	None	Control Section	None
B	Empty Overflow	Middle and Lower	Chamber Open
C	Power Outage	Upper	Chamber Closed

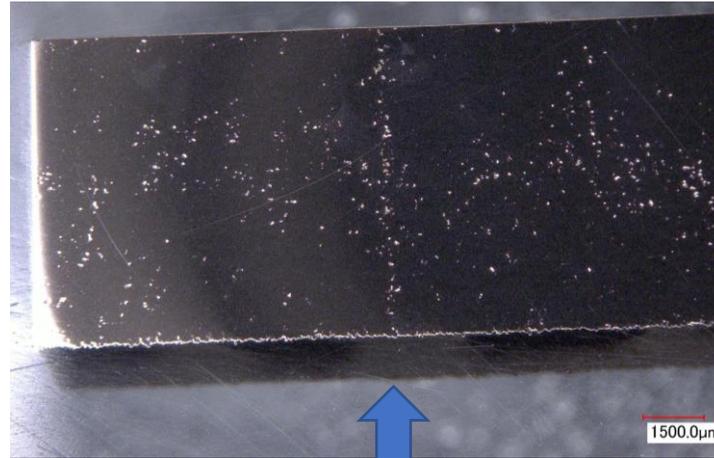
Label	Section	Porosity
A	Upper Witness Line	0.748%
B	Middle Witness Line	1.906%
C	Control Section	0.511%
D	Lower Witness Line	1.743%
E	Tensile Bar	0.006%



- Samples taken from un-tested chamber (C1).
- Tensile bar built separately as part of investigation.
- Proper HIP of chambers was confirmed.
- Porosity is evident throughout samples.
- Clear congregation of porosity around witness lines.
- Porosity reduces load bearing capacity (reduced area) and can act as stress concentrators/crack initiators.

Optical Images of Section

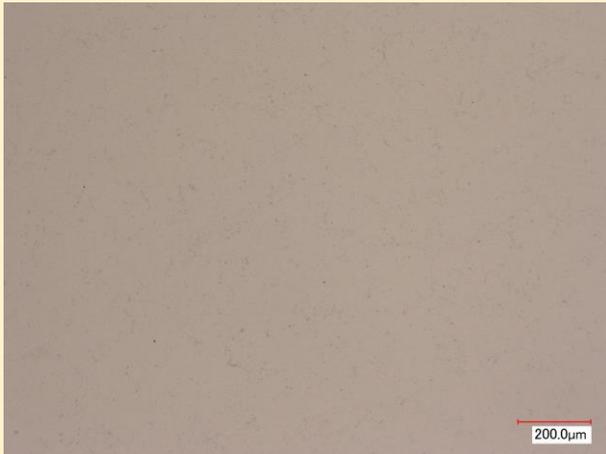
C1 (Leaked), Middle Witness Line



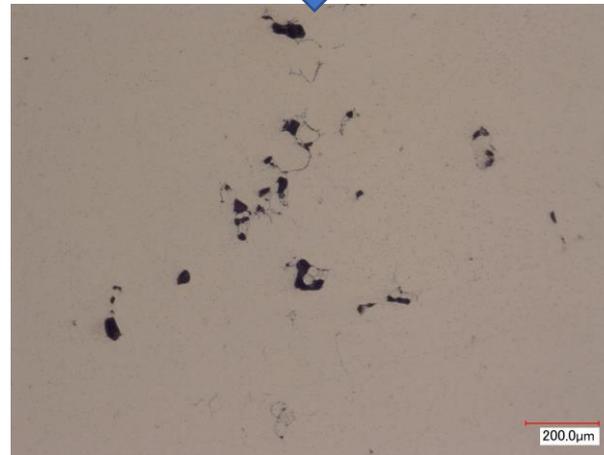
C1 (Leaked), Chamber Control



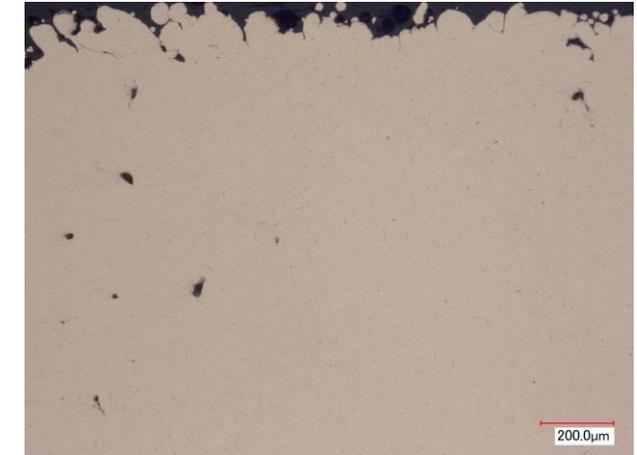
Witness Bar Control
with identical restart



200x



200x

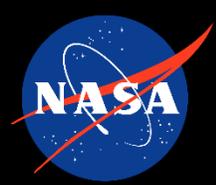


200x

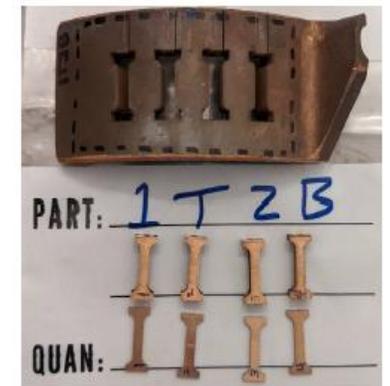
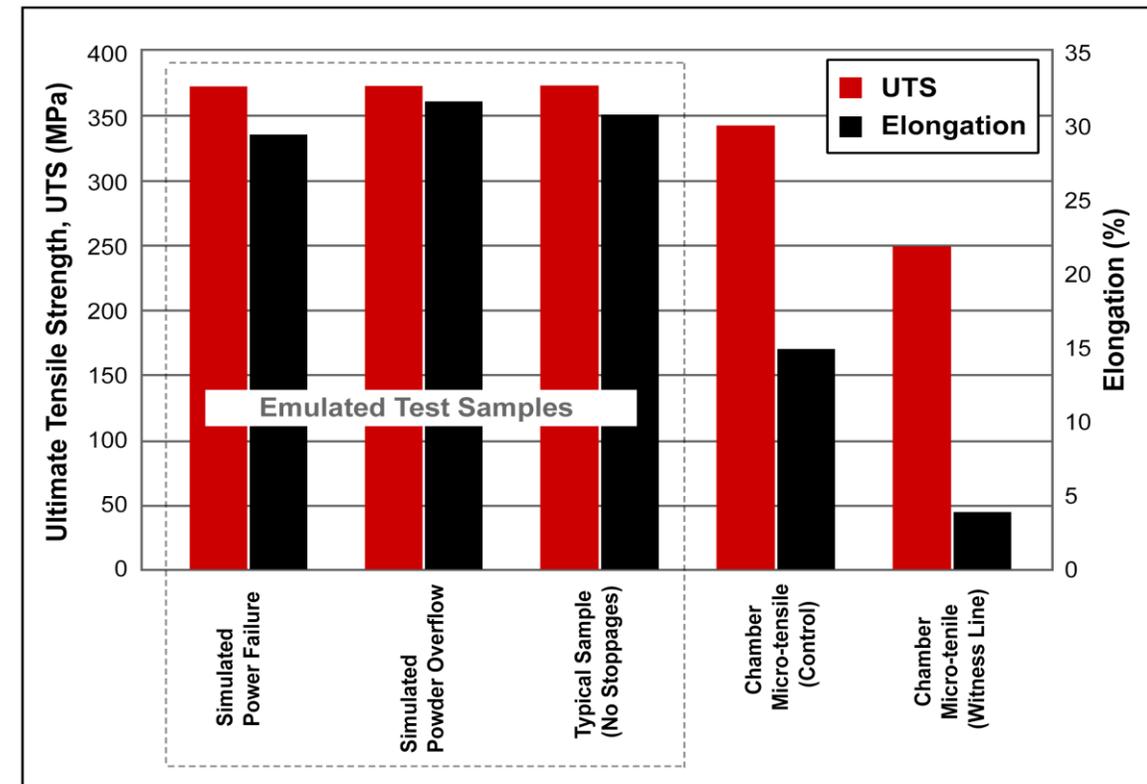
← Build Direction



Combined Microtensile & Tensile Results



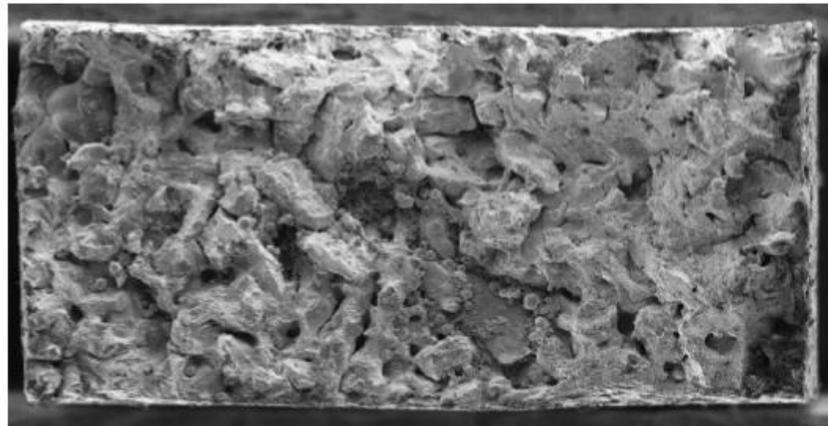
- Room temp tensile testing conducted on ASTM E8 specimens (0.25" dia gage) from witness bars with various restarts
 - Testing at 1200F for ASTM E8 round bars showed similar trends
 - Fracture surfaces appeared similar
- Microtensile testing conducted at room temp on section from chamber (C1 and C3) at witness line and non-witness



Fractography of Samples after Mechanical Testing

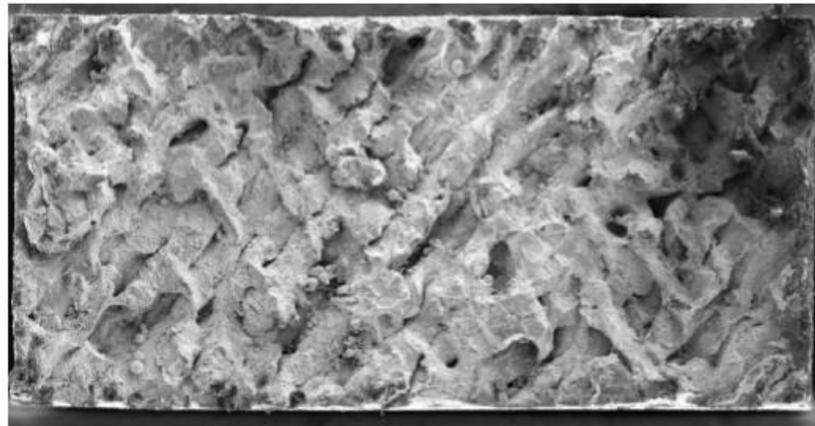
Microtensile

1T1B



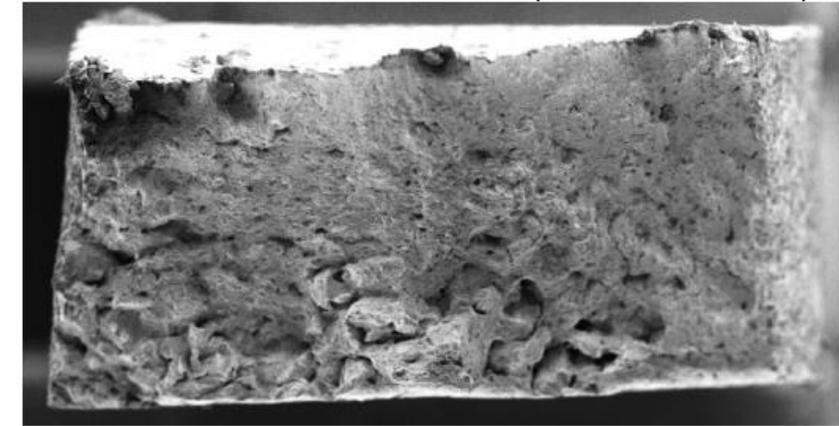
- Granular fracture surface, indicating a brittle fracture
- Irregular shape porosity, indicating lack of fusion

1T2B



- Granular fracture surface, indicating a brittle fracture
- Laser scan pattern clearly visible

1T3B (Chamber Control)



- Overloaded fracture surface and necking, indicating a more ductile fracture
- Less porosity compared to witness lines

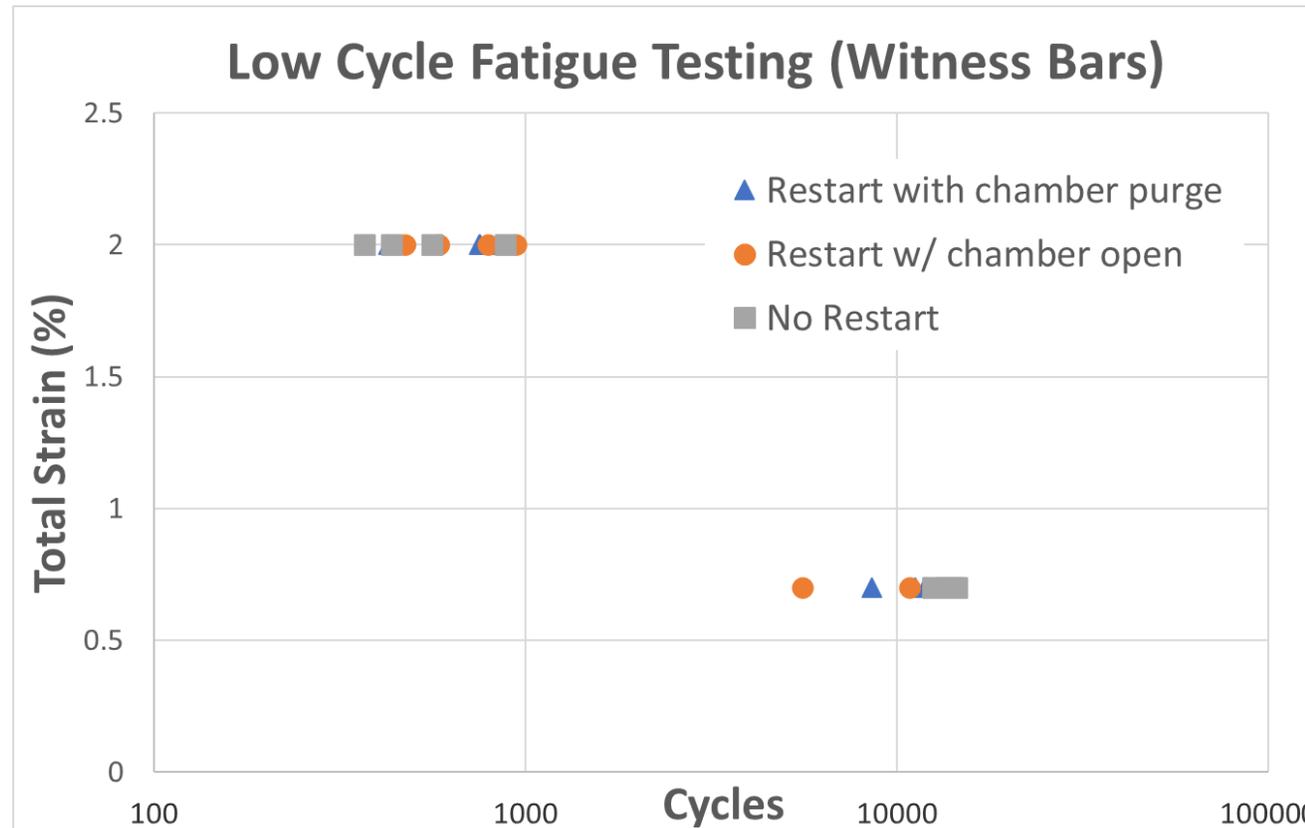




Low Cycle Fatigue of Post-build Witness Specimens



- LCF conducted at room temperature, total strain of 0.7% and 2%
- R = -1, triangular



Strain, %	Cycles, Nf
2	924
2	840
2	529
2	846
0.7	9,760
0.7	12,597
0.7	13,531
0.7	12,796
2	668
2	593
2	986
2	878
0.7	5,579
0.7	10,805
0.7	13,426
0.7	12,899
2	369
2	437
2	559
2	882
0.7	14,038
0.7	12,514
0.7	14,499
0.7	13,624

	Restart w/purge	Restart, open	No restart
2%, Avg	785	781	562
St Dev	175	182	228
0.7%, Avg	12171	10677	13669
St Dev	1657	3582	849

*4 samples per test case



Failure Conclusions



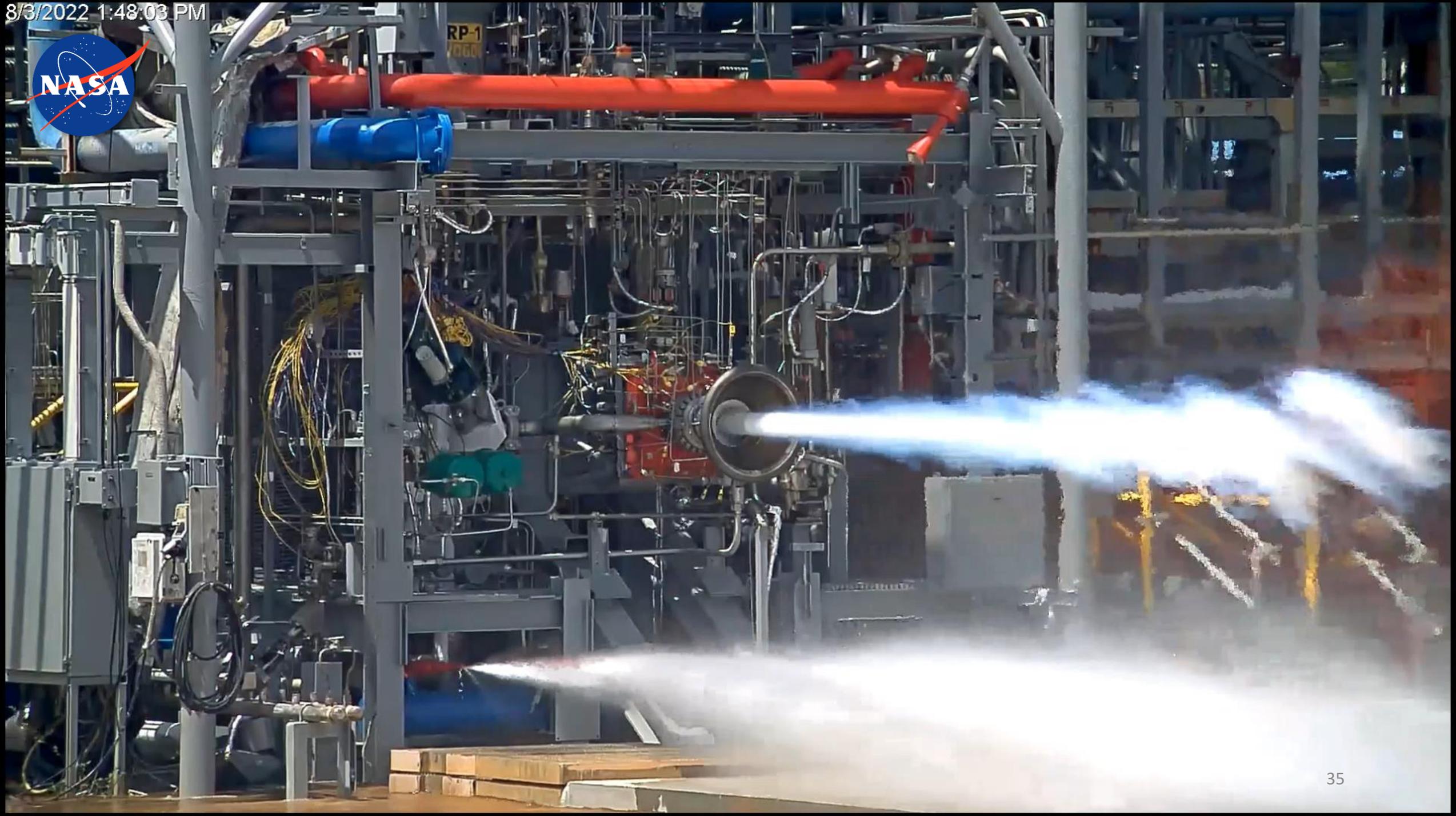
- Chamber experienced tensile overload during hot-fire at the witness line that had a higher degree of voids.
- The L-PBF GRCop-42 chambers built under LLAMA had higher porosity (1-2%) that congregated more at witness lines causing lack of fusion.
- Granular surfaces, unmelted particles, and irregular pores were observed in microtensile specimens (sectioned) from chambers.
- Areas affected by build interruptions must be properly evaluated and dispositioned. AM machine restarts represent a risk, and appropriate restart procedures should be developed and followed to maintain material quality.
 - Witness specimens using different types of restarts showed similar tensile strengths and LCF results.
- Build log indicated no issues with parameters, but *an issue* (parameters, lens, etc) caused the porosity and HIP did not fully close these voids.
- Demonstrates the process sensitive nature of AM parts and build interruptions need to be properly documented, fully evaluated, and properly dispositioned.



Failure Conclusions and Recommendations



- Build interruptions in GRCop-42 components do not inherently possess weakened material properties if a restart procedure is properly executed.
- Full height specimens should be built with all components to characterize the material.
- While not subject to NASA-STD-6030, this chamber provides a good case study on why it is important that AM materials used in critical applications adhere to NASA-STD-6030 standards and the need for robust process development, in-depth material evaluation, and process controls.



- The proper planning...
 - High quality ingredients...
 - Controlled environments and parameters...
 - Properly planned post-processing...
 - Repeatable and controlled process...
 - Verified process...
- = Successful Component (Happy Customers!)



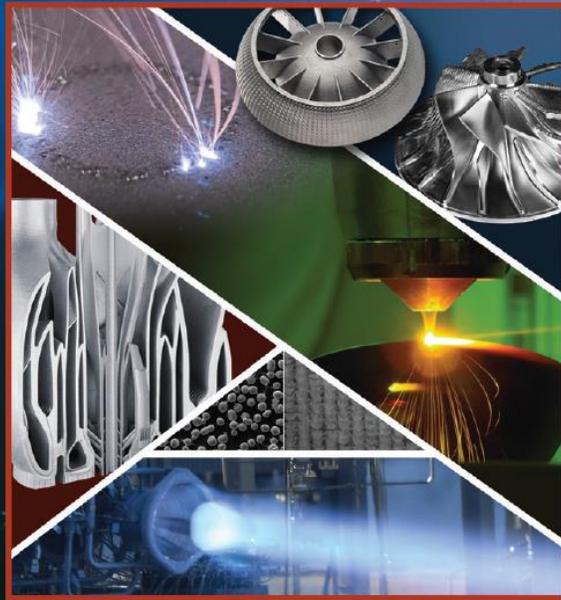


NASA led book on Metal Additive Manufacturing



Metal Additive Manufacturing for Propulsion Applications

Edited by
Paul R. Gradl, Omar R. Mireles,
Christopher S. Protz, and Chance P. Garcia



PROGRESS IN ASTRONAUTICS AND AERONAUTICS

Timothy C. Liewen, Editor-in-Chief
Volume 263

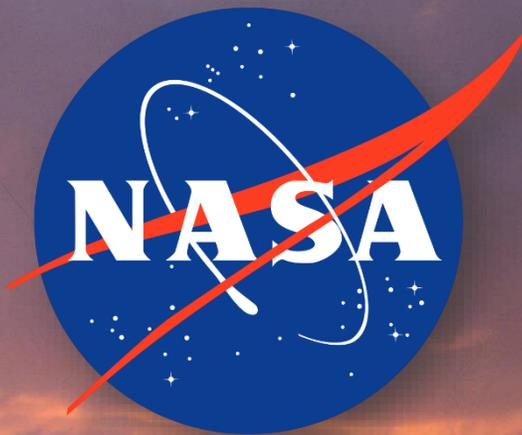
<https://arc.aiaa.org/doi/book/10.2514/4.106279>

Online version and hardcopy available

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<https://arc.aiaa.org/doi/book/10.2514/4.106279>

Additive manufacturing (AM) processes are proving to be a disruptive technology and are grabbing the attention of the propulsion industry. AM-related advancements in new industries, supply chains, design opportunities, and novel materials are increasing at a rapid pace. The goal of this text is to provide an overview of the practical concept-to-utilization lifecycle in AM for propulsion applications.



Contact:

Paul Gradl

NASA MSFC

Paul.R.Gradl@nasa.gov



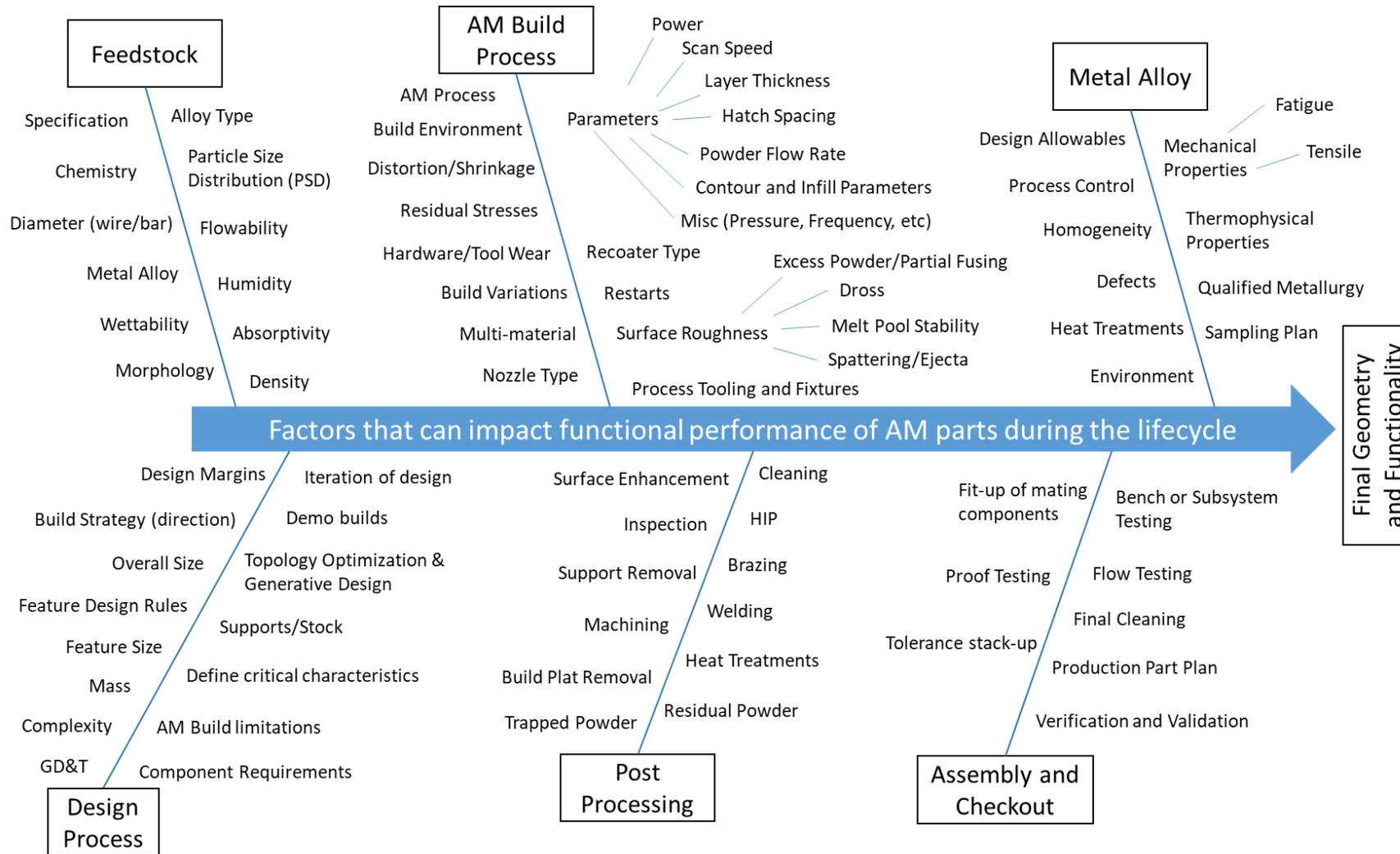
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- Lynn Machamer
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- Tyler Blumenthal
- DM3D
- GE Research
- Bhaskar Dutta
- REM Surface Engineering
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- AP&C
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- Marissa Garcia
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- Kevin Baker
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- IMR Test Labs
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- Ron Beshears
- James Walker
- Steve Wofford
- Johnny Heflin
- Mike Shadoan
- Keegan Jackson
- Many others in Industry, commercial space and academia



The Challenges with AM Processes



There are a lot of inputs and steps in the AM lifecycle that must go right to meet the expected geometry

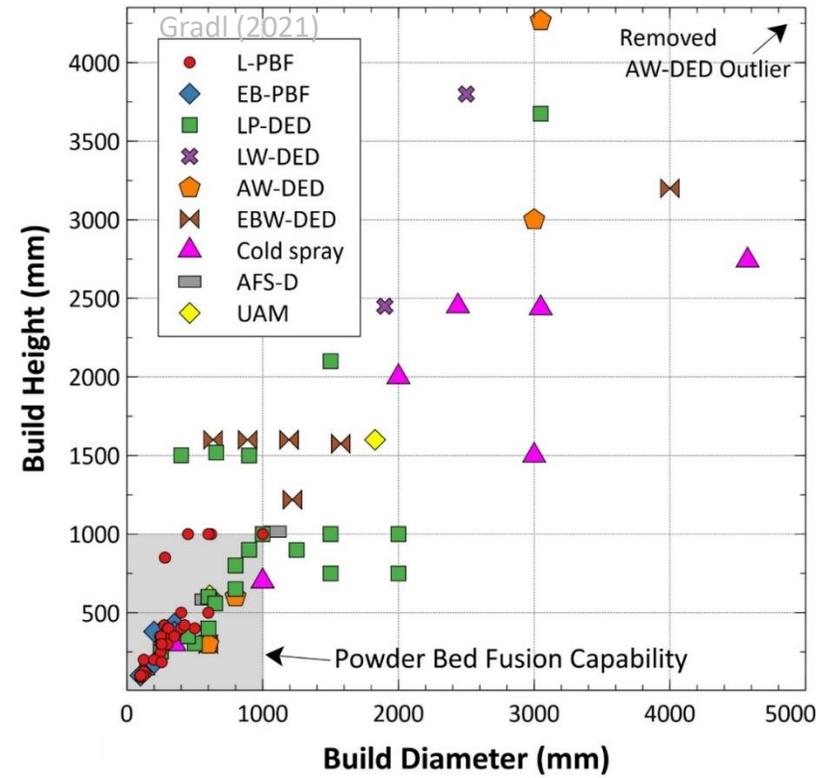
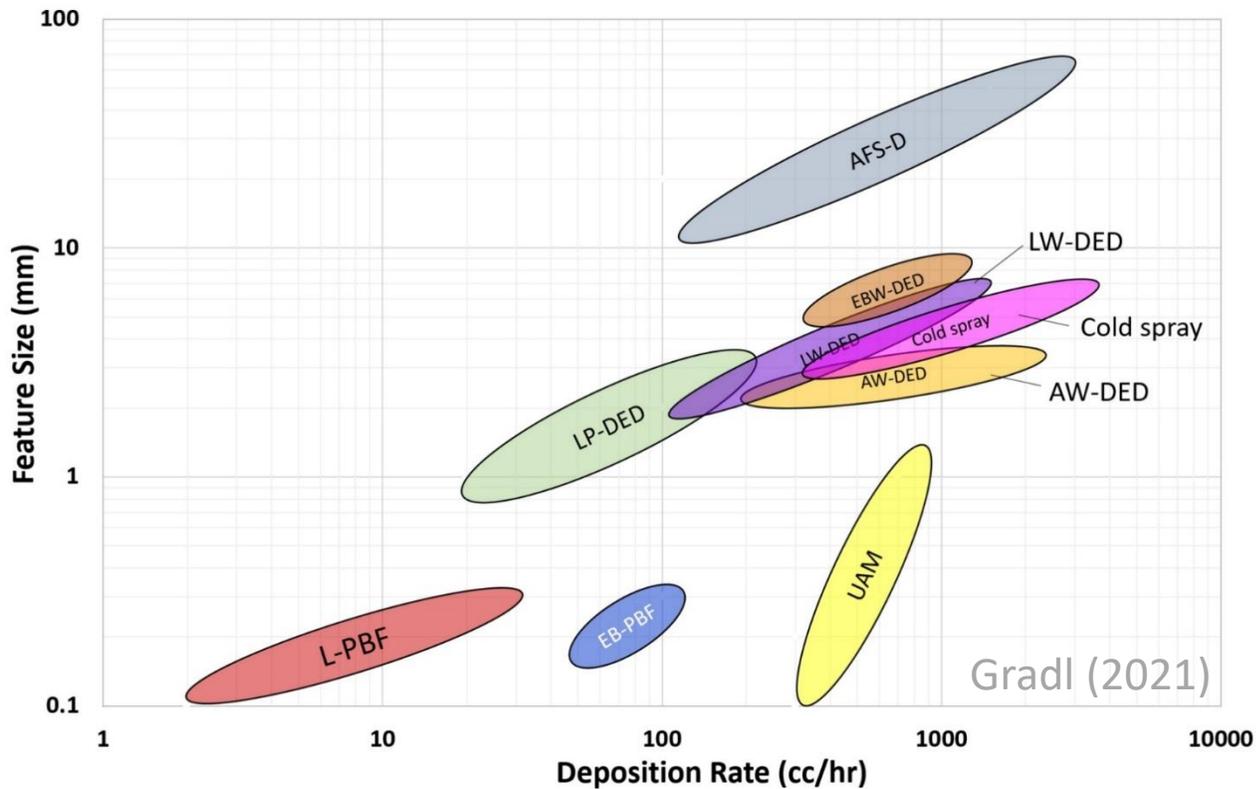
Methodical AM Process Selection



- What is the **alloy** required for the application?
- What is the **overall part size**?
- What is the **feature resolution** and internal **complexities**?
- Is it a **single alloy or multiple**?
- What are **programmatic requirements** such as cost, schedule, risk tolerance?
- What are the end-use environments and **properties required**?
- What is the **qualification/certification** path for the application/process?



Various criteria for selecting AM techniques



Complexity of Features

Scale of Hardware

Material Physics

Cost

Material Efficiency

Speed of Process

Material Properties

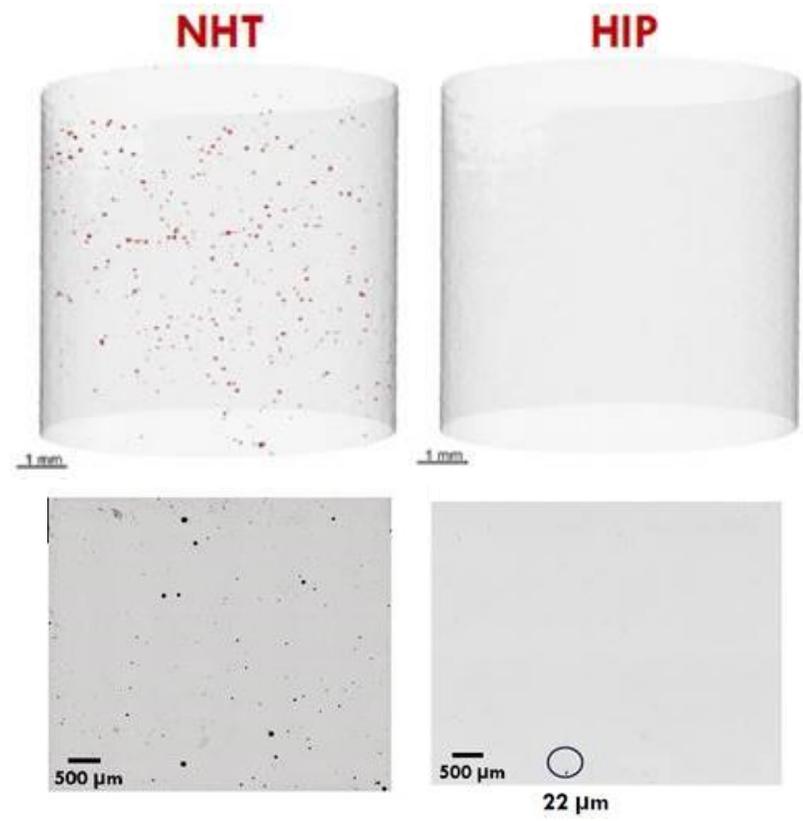
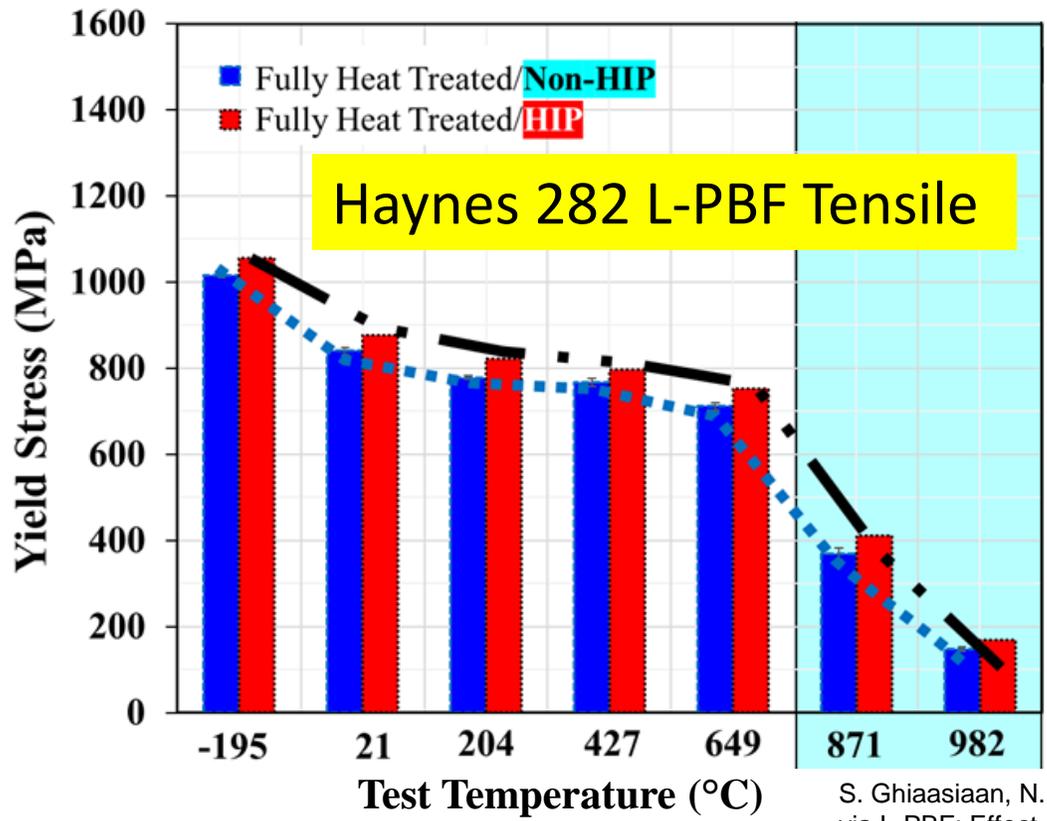
Internal Geometry

Availability

Post Processing

Why baseline HIP?

- NASA has baselined HIP for data generation (per NASA 6030).
- HIP does add process time and cost but can improve performance of alloys for tensile and fatigue based on comparison studies.



S. Ghiaasiaan, N. Ahmad, P. Gradl, S. Shao, N. Shamsaei: Additively Manufactured Haynes 282 via L-PBF: Effect of Post-processing on Mechanical Properties at Different Temperatures



AM Alloys and Processes In-work



Material	Process
Haynes 282	L-PBF
Haynes 282	LP-DED
Hastelloy X	L-PBF
Hastelloy X	LP-DED
Inconel 625	L-PBF
Inconel 625	LP-DED
Inconel 625	LW-DED
Inconel 625	AW-DED
Inconel 718	L-PBF
Inconel 718	LP-DED
Inconel 718	AW-DED
Inconel 939	L-PBF
Haynes 230	L-PBF
Haynes 230	LP-DED
Haynes 214	L-PBF
Haynes 233	L-PBF
Haynes 233	LP-DED

Material	Process
NASA HR-1	L-PBF
NASA HR-1	LP-DED
JBK-75	L-PBF
JBK-75	LP-DED
CoCr	L-PBF
CoCr	LP-DED
Invar 36	LP-DED
Stellite 21	LP-DED
316L	LP-DED
15-5	LP-DED
17-4	L-PBF
17-4	LP-DED
Scalmalloy	L-PBF
6061-RAM2	L-PBF
6061-RAM2	LP-DED
F357	L-PBF
F357	LP-DED
1000-RAM10	L-PBF
AlSi10Mg	L-PBF
AlSi10Mg	LP-DED
7A77	L-PBF

Material	Process
Monel K500	LP-DED
Monel K500	L-PBF
GRCop-42	L-PBF
GRCop-42	LP-DED
GRCop-84	L-PBF
C-18150	L-PBF
Ti6Al-4V	L-PBF
Ti6Al-4V	LP-DED
Ti6Al-4V	LW-DED
Ti6Al-4V	EBW-DED
Ti6242	L-PBF
Ti6242	LP-DED
GRX-810	L-PBF
GRX-810	LP-DED
Haynes 214-ODS	L-PBF
C-103	LP-DED

55+ Alloys in characterization

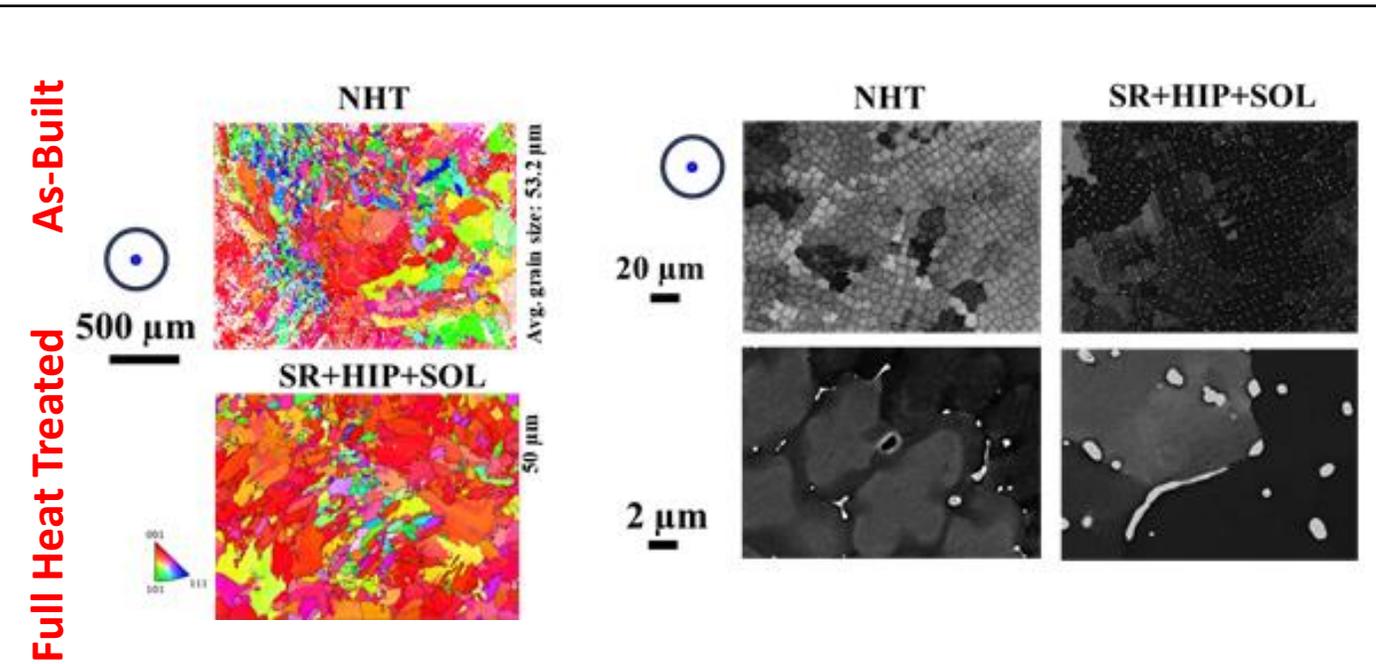


Data example of Haynes 230 LP-DED



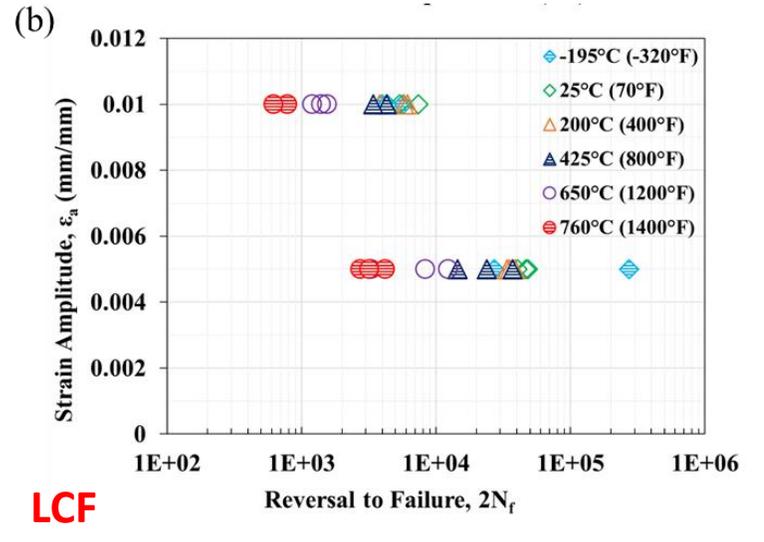
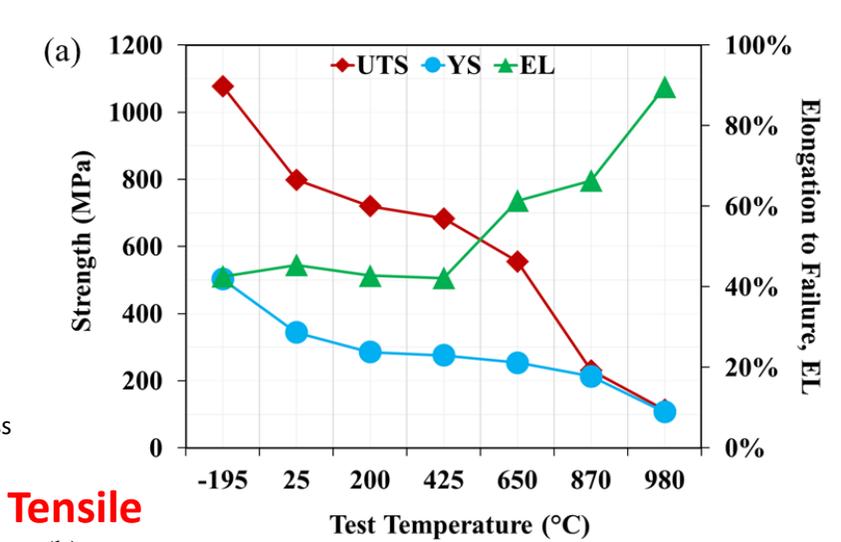
Power (W)	Layer height (μm)	Travel speed (mm/min)	Powder feed rate (g/min)
1070	381	1016	19.10

Procedure (Designation)	Temperature (°C)	Time (hrs)	Cooling
Stress Relief (SR)	1066	1.5	Furnace cool
HIP [2]	1163/103 MPa	3	Furnace cool
Solution Annealing (SOL)	1177	3	Argon quench



[2] HIP per ASTM F3301

Data from Gradl, Mireles, Protz, Garcia. "Metal Additive Manufacturing for Propulsion Applications", AIAA Progress Series. (2022). Appendix A.





ICP & IGF Chemical Analysis

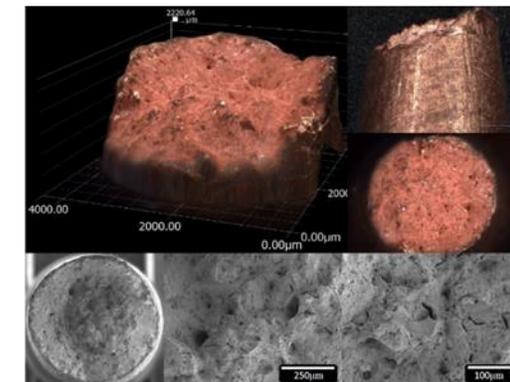
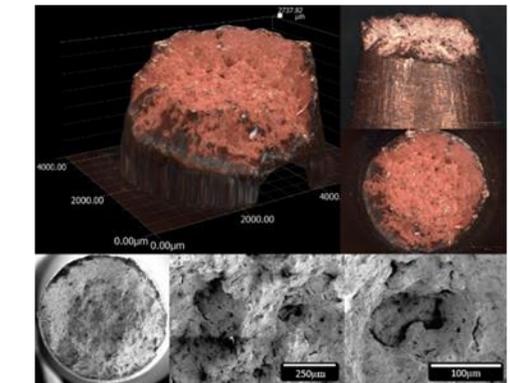
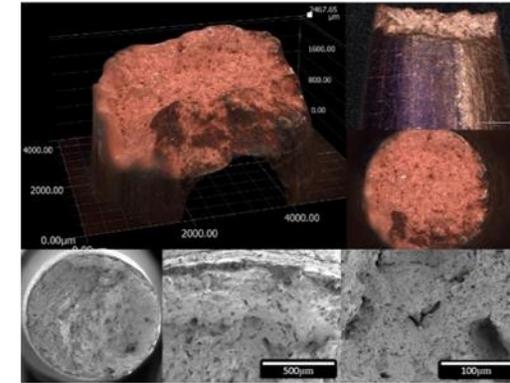
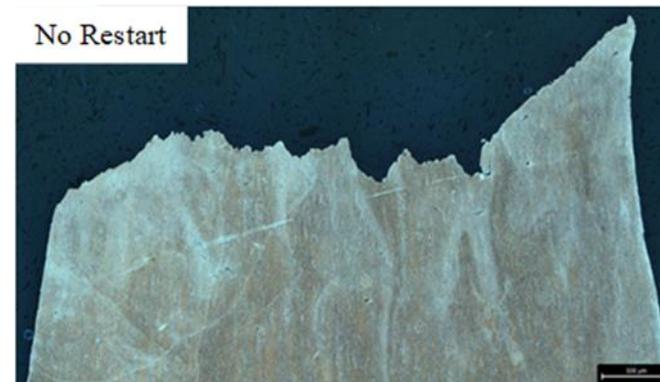
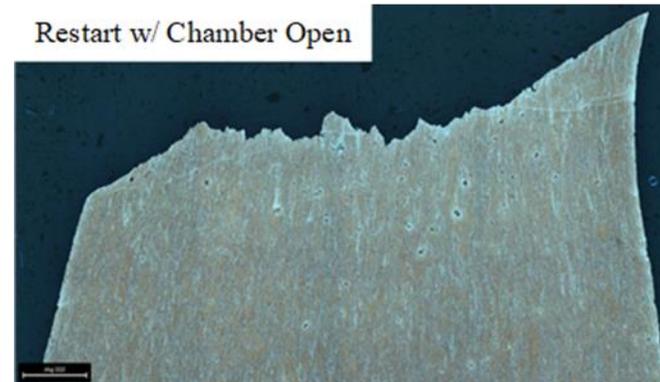
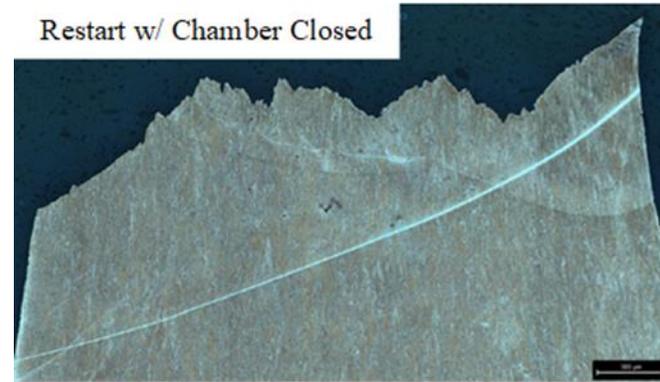


- 7 pieces from entire length of chamber C1
- Observations:
 - Composition did not vary throughout length of chamber
 - Al, Si higher than expected – crucible fluxing potentially
 - Ni, Co, Fe within detection limits
 - O notably high – can reduce conductivity and produce Al-Nb-O particles that create fatigue cracking
- Cr/Nb ratio is strong indicator of GRCop’s effectiveness (precipitates for dispersion strengthening)
 - High ratio in chamber pieces
 - Results in excess chromium precipitates
 - Reduces high temp strength and creep resistance
- Important for AM GRCop parts to have consistent compositions

Element	Chamber Avg.	Spec Target	PC Results
Copper	93.88	-	-
Chromium	3.37	3.27	3.10
Niobium	2.57	2.92	2.70
Oxygen	0.06	0.025	0.033
Iron	0.02	0.005	0.007
Aluminum	0.05	0.04	-
Silicon	0.02	0.01	-
Nickel	0.02	-	-
Cobalt	0.01	-	-
Phosphorus	0.01	-	-
Silver	0.01	-	-

Documentation	Cr/NB Ratio
Chamber Average	1.31
Specification Target	1.13 - 1.18
PC Results	1.15

- 3 witness test bars from tensile simulating build stoppages
- Observations:
 - No major differences between fractures
 - Typical cup-cone fracture surfaces common for ductile metals
 - Fracture surfaces had elongated grains
- Conclusions:
 - Similar fractures track with similar properties observed in bars previously



- 3 test bars from LCF
 - Fracture surfaces smeared b/c LCF had fully reversible cycles
 - Closed chamber restart bar never fully fractured
- Observations:
 - Open restart: flat surface before overload failure transition, secondary crack below primary crack
 - No restart: three separate cracks jogged together

