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Maturation of Additive Manufactured Aerospace Alloys and Development of Mechanical and Thermophysical Properties for Space Applications

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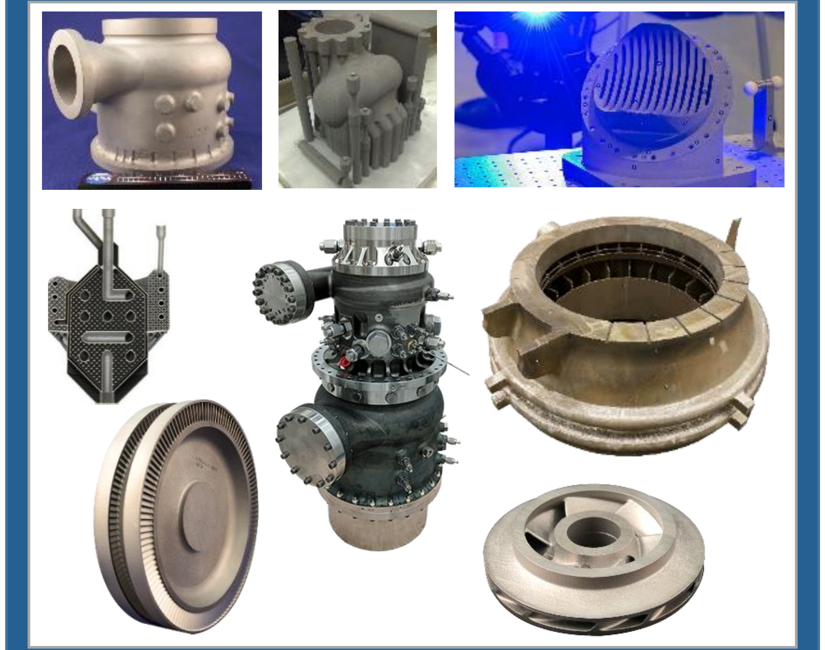
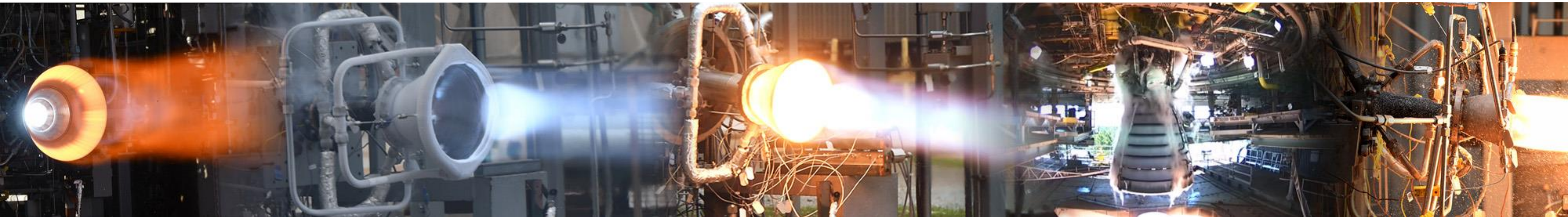
AM at NASA for Rocket Engine Applications

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



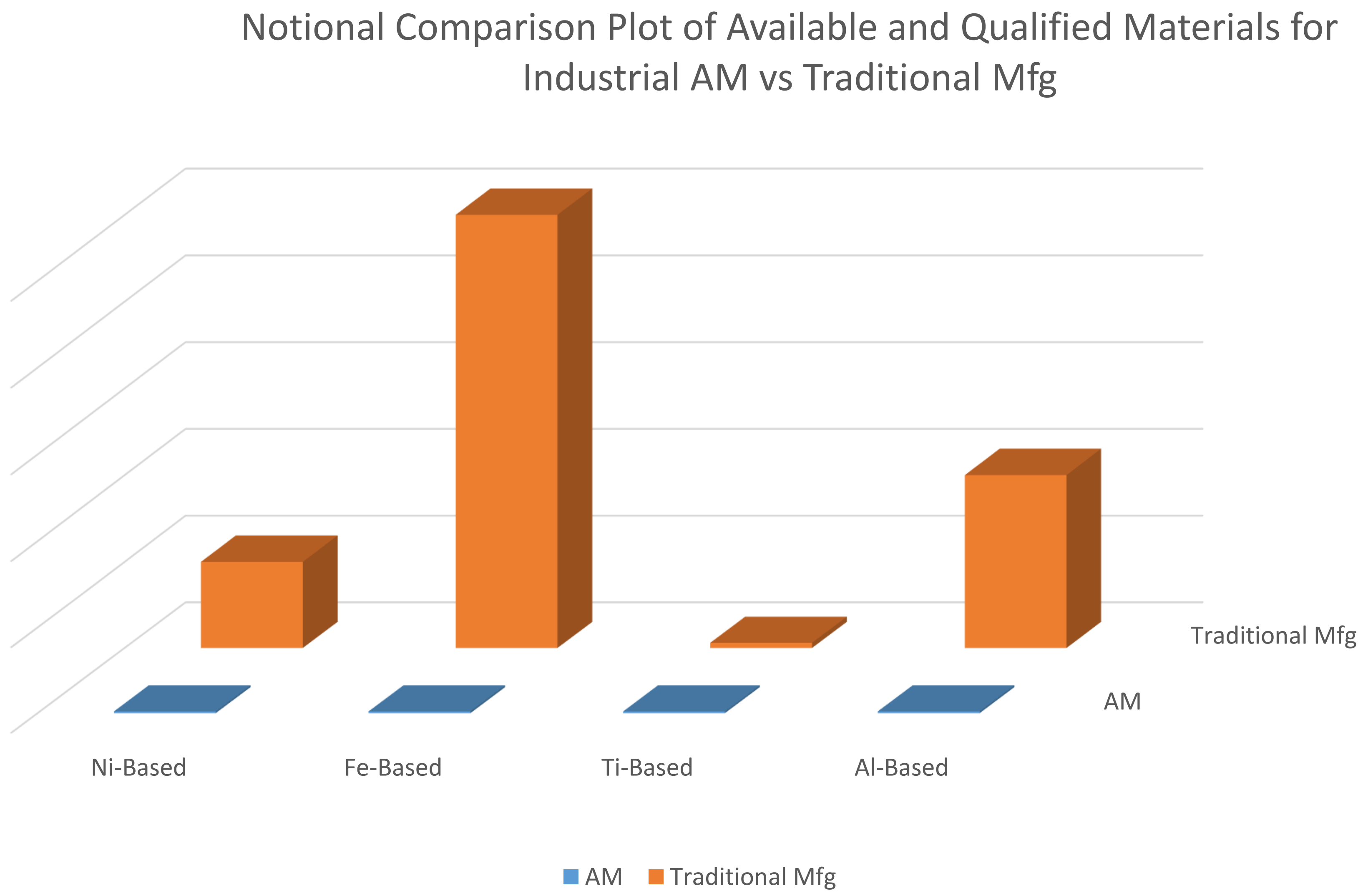
Possible Conversations heard around Engineers Cubes

- *Design Engineer* : I have a great idea of making this rocket thrust chamber using AM! I need to size this design and finish my trades; If I want to use this new AM alloy, are there the basic tensile properties I can use for my ***conceptual*** design?

- *Materials Engineer* : Hmm. The data is very limited. We may have to start $$$ testing campaign to get you the basic properties

- *Design Engineer* : (disappointed) I will just make it up… (and ceases the communication with his buddy)

AM Alloys have limited data for design purposes



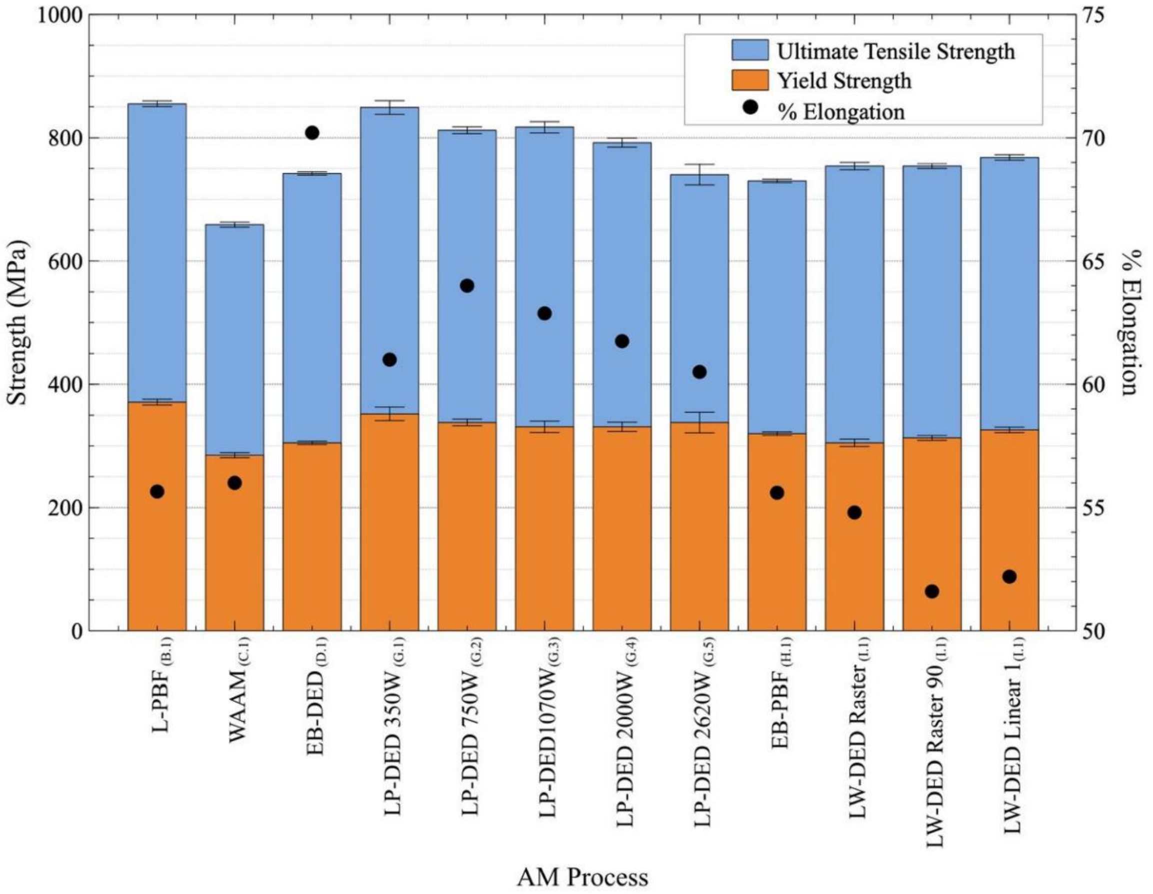
Motivations of this project

- Only a few alloys have been matured with significant property development and reached qualified state at NASA (Alloy 718, Alloy 625, GRCop-42/84) for Rocket Propulsion
- Other alloys are of interest to include in component trades for development as well as AM modeling efforts, <u>but limited data is available</u>
  - While many of the alloys of interest and have been characterized by various companies, a majority of the data is proprietary and not accessible
- Under various NASA projects, well-established alloys using various AM processes are being characterized and mechanical testing completed.
- A current task under this development is to complete **an initial database of key properties** to help with the **initial trades and modeling** efforts.

Fundamentals are necessary for Component Applications

- Material properties are highly dependent on the type of process (L-PBF, DED, UAM, Cold spray….), the starting feedstock chemistry, the parameters used in the process, and the heat treatment processes used post-build.
- Each AM process results in different grain structure, precipitates, and porosity, all of which influence final properties.
- Heat treatments should be developed based on the requirements and environment of the end component use.
- Process, parameters, and feedstock should all be stable before property development.

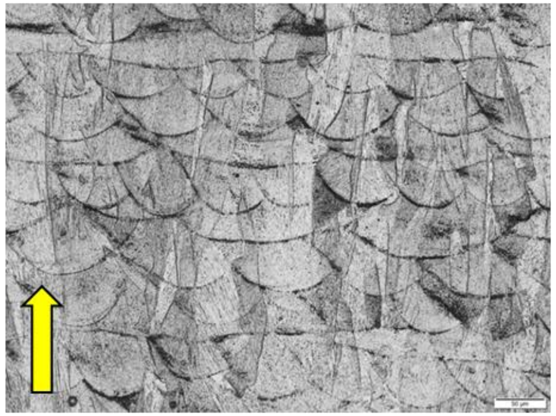
Alloy 625, Heat Treated per AMS 7000
Room Temperature UTS



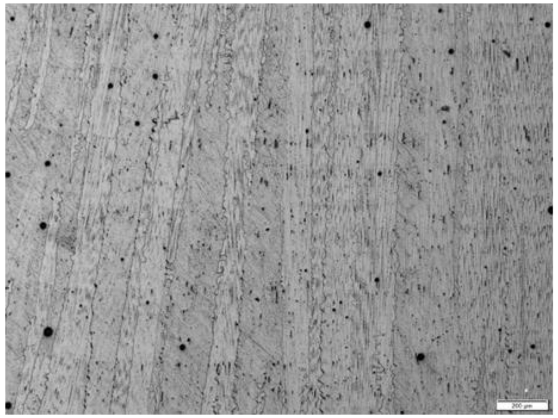
*Not design data and provided as an example only

Luna, V.; Trujillo, L.; Gamon, A.; Arrieta, E.; Murr, L.E.; Wicker, R.B.; Katsarelis, C.; Gradl, P.R.; Medina, F. Comprehensive and Comparative Heat Treatment of Additively Manufactured Inconel 625 Alloy and Corresponding Microstructures and Mechanical Properties. *J. Manuf. Mater. Process.* **2022**, *6*, 107. https://doi.org/10.3390/jmmp6050107

Microstructure of Various AM Processes
Alloy 625 – As-Built

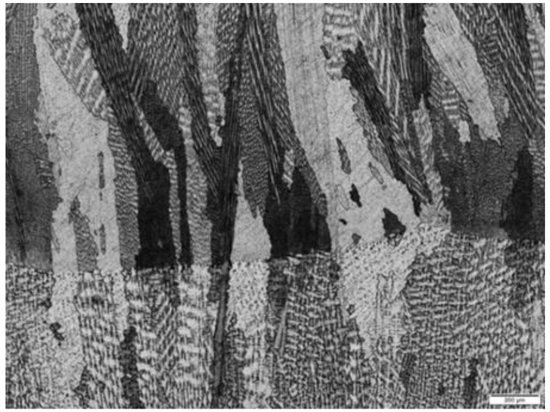


Laser Powder Bed Fusion

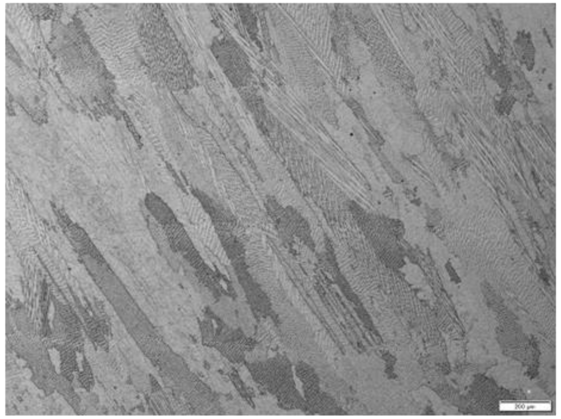


Electron Beam Powder Bed Fusion

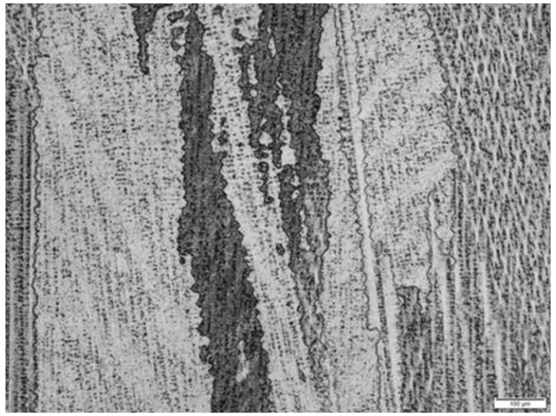
Laser Powder DED (1070 W)



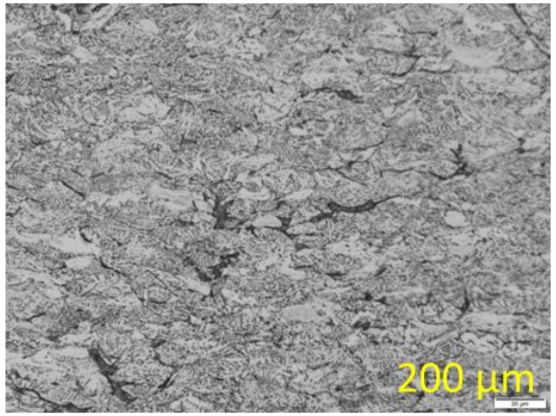
Electron Beam Wire DED



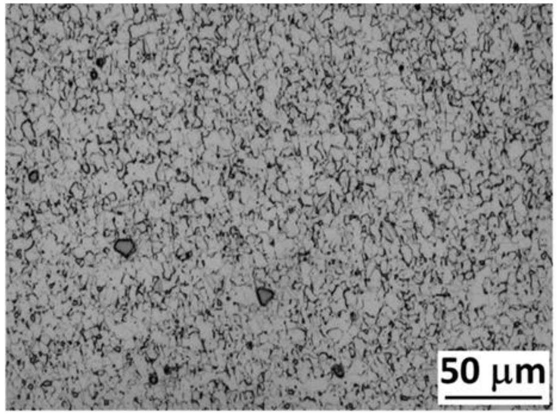
Laser Wire DED



Arc Wire DED



Cold Spray



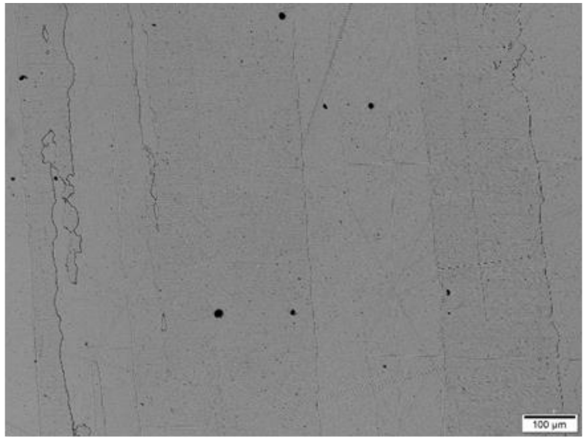
Additive Friction Stir Deposition

Each AM process results in different grain structures, which ultimately influence properties

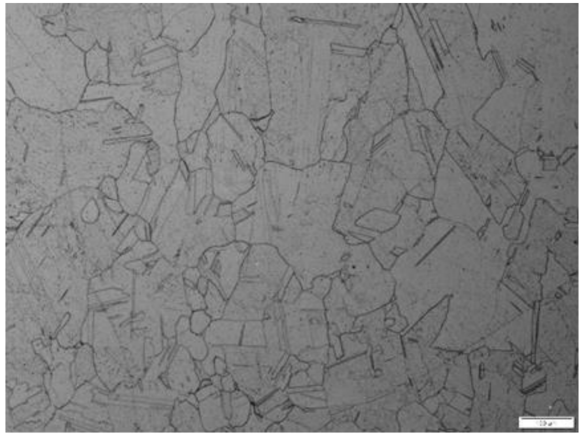
- Gamon, A., Arrieta, E., Gradl, P.R., Katsarelis, C., Murr, L.E., Wicker, R.B., Medina, F., 2021. Microstructure and hardness comparison of as-built Inconel 625 alloy following various additive manufacturing processes. Results in Materials 12. https://doi.org/10.1016/j.rinma.2021.100239
- Gradl, P., Tinker, D., Park, A., Mireles, O., Garcia, M., Wilkerson, R., Mckinney, C., 2021. Robust Metal Additive Manufacturing Process Selection and Development for Aerospace Components. Journal of Materials Engineering and Performance, Springer. https://doi.org/10.1007/s11665-022-06850-0
- Rivera, O. G., Allison, P. G., Jordon, J. B., Rodriguez, O. L., Brewer, L. N., McClelland, Z., ... & Hardwick, N. (2017). Microstructures and mechanical behavior of Inconel 625 fabricated by solid-state additive manufacturing. Materials Science and Engineering: A, 694, 1-9.
- Image from Mark Norfolk, Fabrisonic

Microstructure of Various AM Processes
Alloy 625 – Stress Relief, HIP, Solution per AMS 7000

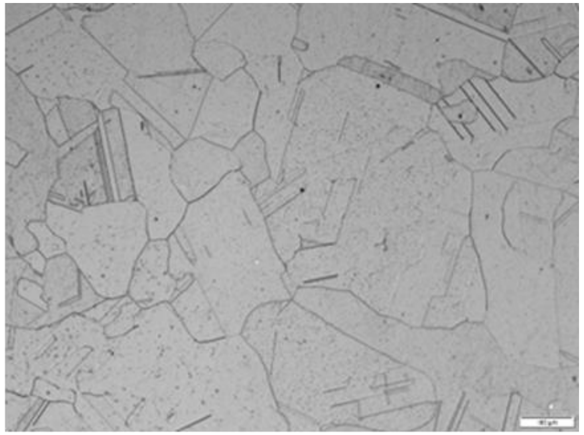
Laser Powder Bed Fusion



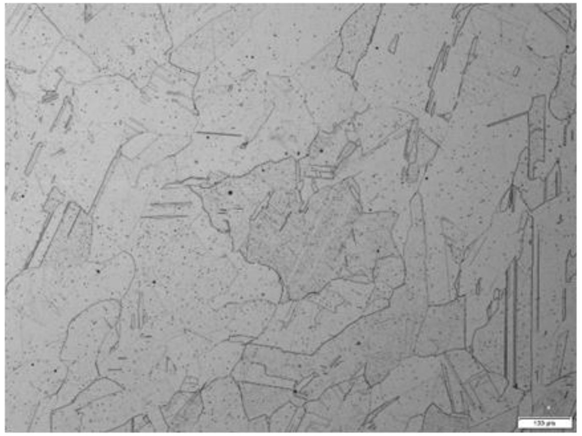
Electron Beam PBF



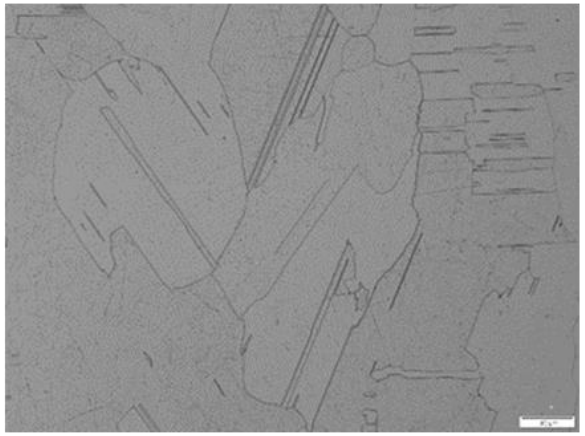
Laser Powder DED (1070 W)



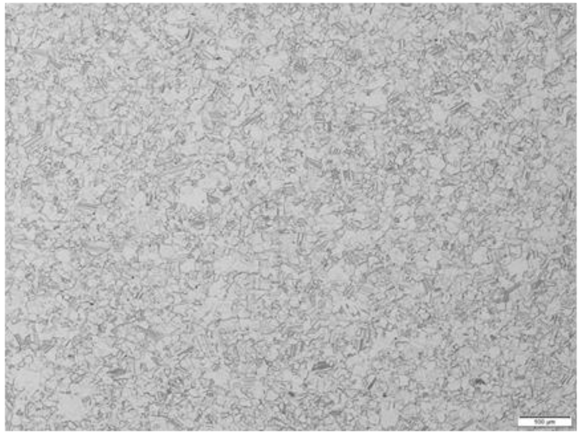
Electron Beam Wire DED



Laser Wire DED



Arc Wire DED



Cold Spray

- Gamon, A., Arrieta, E., Gradl, P.R., Katsarelis, C., Murr, L.E., Wicker, R.B., Medina, F., 2021. Microstructure and hardness comparison of as-built Inconel 625 alloy following various additive manufacturing processes. Results in Materials 12. https://doi.org/10.1016/j.rinma.2021.100239
- Gradl, P., Tinker, D., Park, A., Mireles, O., Garcia, M., Wilkerson, R., Mckinney, C., 2021. Robust Metal Additive Manufacturing Process Selection and Development for Aerospace Components. Journal of Materials Engineering and Performance, Springer. https://doi.org/10.1007/s11665-022-06850-0

Project Approach

- While we are not specifically limiting materials, we are trying to focus on many of the current alloys that have been matured across industry and of interest across various components at NASA.
  - A fundamental understanding of the build process and material using some best practices from MSFC-SPEC-3717 (along with MSFC-STD-3716).
  - Characterization and evolution of the alloys through build and heat treatments.
  - Recommending proper heat treatment schedules.
  - Complete basic mechanical and thermophysical property testing.
    - Tensile from -320F through 1800F+
    - Low Cycle Fatigue (LCF) at various strains from -320F through 1600F+
    - High cycle fatigue (HCF) testing as allows at various peak stresses.
    - Thermal conductivity, CTE
  - Document results to make available for component trades and modeling efforts at NASA
  - Publish open reports/journals/articles on properties, heat treatments, and make data available to industry and research community to build upon

***This is not design data and feasibility properties for conceptual designs. Data is provided as typical averages.**

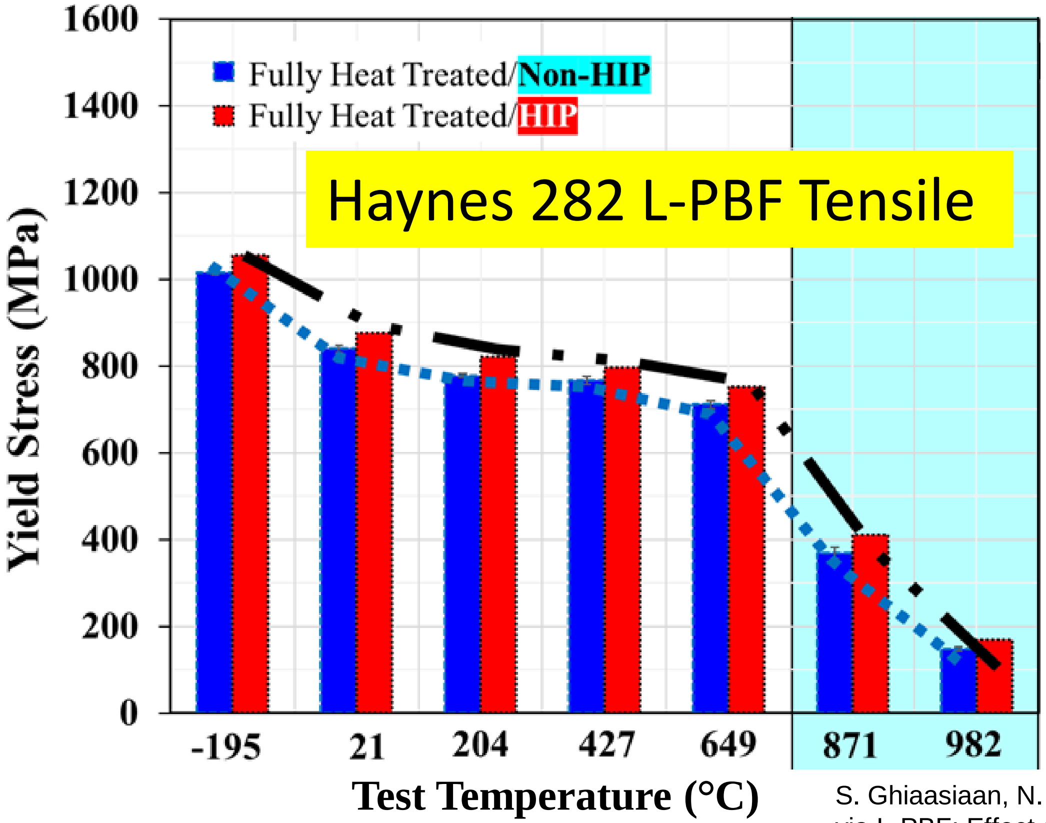
***The data is funded and developed by NASA. It is available, but at users' discretion.**

Project Approach - continues

- Obtained material samples from L-PBF and DED vendors with full traceability.
- Characterization of as-built samples and evolution through heat treatments to select optimal heat treatment cycle (HIP baselined).
  - Partnership with Auburn NCAME for alloy characterization.
  - LSU responsible for thermophysical properties.
  - UTEP supporting AM process studies.
- After appropriate heat treatment cycle, complete temperature dependent tensile and fatigue testing.
- Partial data set is published as Appendix in AIAA book
  - P. R. Gradl, O. Mireles, C.S. Protz, C. Garcia. (2022). *Metal Additive Manufacturing for Propulsion Applications*. AIAA Progress in Astronautics and Aeronautics Book Series. https://arc.aiaa.org/doi/book/10.2514/4.106279
- Future goal to publish all data and characterization in a handbook and raw data uploaded to selected database (potentially MAPTIS).

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.



Ti6Al4V LP-DED HIP Study

	As-built	#01	#02	#03	#04
Number of defects	167	119	28	28	25
Maximum size of defects (μm)	124	61	61	59	71
Porosity (%)	0.0027	0.0006	0.0004	0.0004	0.0006

Nano-CT Scanning following each HIP Cycle

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

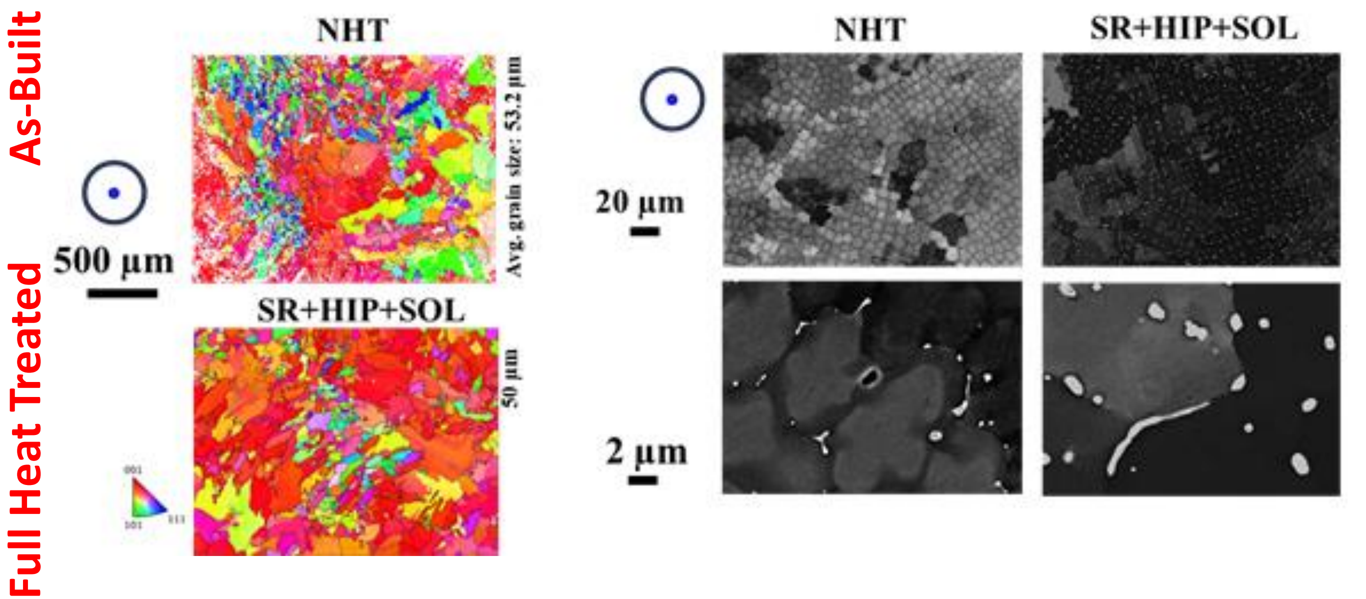
Material	Process
Monel K500	LP-DED
Monel K500	L-PBF
GRCop-42	L-PBF
GRCop-42	LP-DED
GRCop-84	L-PBF
C-18150	L-PBF
Ti6Al-4V	L-PBF
Ti6Al-4V	LP-DED
Ti6Al-4V	LW-DED
Ti6Al-4V	EBW-DED
Ti6242	L-PBF
Ti6242	LP-DED
GRX-810	L-PBF
GRX-810	LP-DED
Haynes 214-ODS	L-PBF
C-103	LP-DED

55+ Alloys in characterization

Data example of Haynes 230 LP-DED

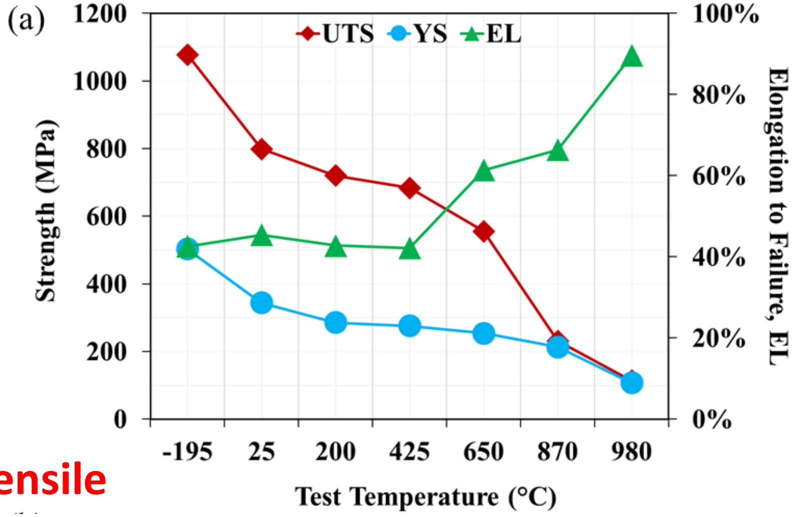
Power (W)	Layer height (µm)	Travel speed (mm/min)	Powder feed rate (g/min)
1070	381	1016	19.10

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

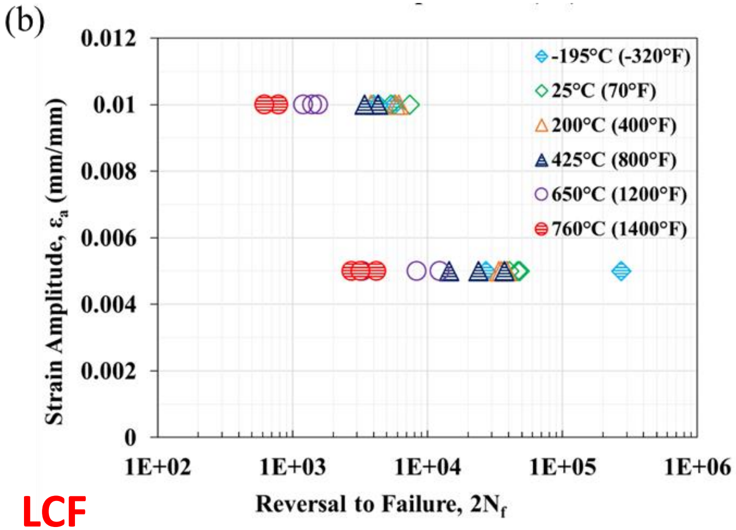


[2] HIP per ASTM F3301

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



Tensile



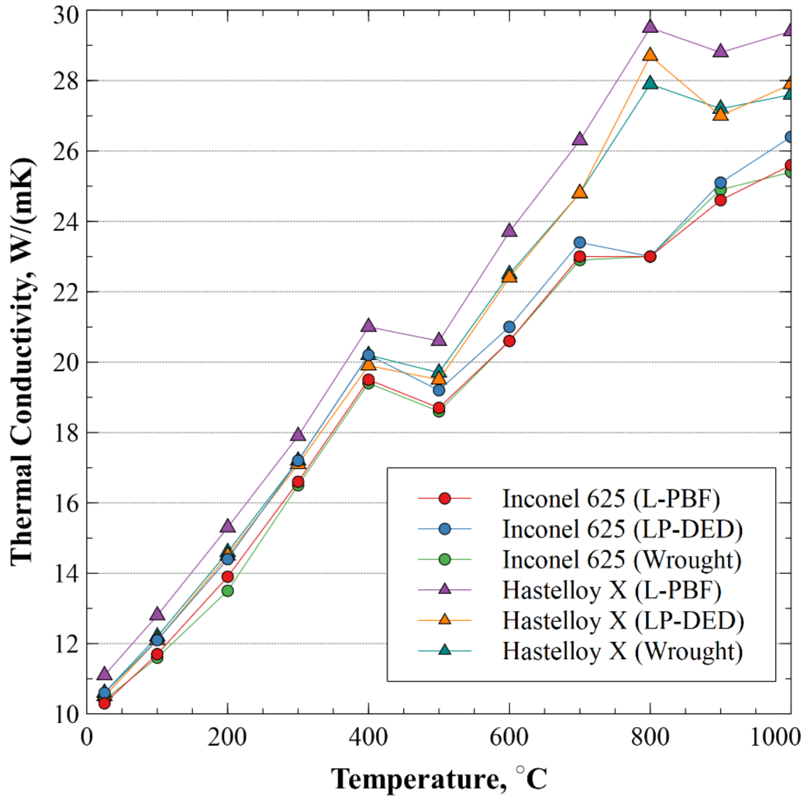
LCF

AM Thermophysical Property Database

- Identical samples and heat treatments determined for mechanical testing also complete thermophysical testing
  - CTE, thermal diffusivity, specific heat, thermal conductivity measurements
  - Comparison of data to select alloys wrought form
  - Data tested in partnership with LSU

TABLE 1-1 COEFFICIENTS OF THERMAL EXPANSION (CTE) OF THE ALLOYS (10^{-6}/K)

Temp. (°C)		100	200	300	400	500	600	700	800	900	1000
Material	Process										
GRCop-42	L-PBF	14.7	16.2	16.7	17.1	17.7	18.2	18.7	19.4	20.2	/
GRCop-84	L-PBF	/	/	/	/	/	/	/	/	/	/
C-18150	L-PBF	/	/	/	/	/	/	/	/	/	/
Inconel 625	L-PBF	11.5	12.8	13.3	13.6	13.9	14.3	14.9	15.5	16	16.4
Inconel 625	LP-DED	12.1	12.6	13.1	13.5	13.8	14	14.6	15.2	15.6	16.1
Inconel 718	L-PBF	12.6	13.9	14.2	14.5	14.8	15.1	15.6	16.4	17.5	17.8
Inconel 718	LP-DED	12.7	13.9	14.4	14.7	15	15.3	15.7	16.7	17.9	18.2
Inconel 939	L-PBF	11.4	12.4	12.9	13.3	13.7	14.0	14.6	15.1	16.0	11.4
HastelloyX	L-PBF	13	14	14.3	14.7	14.9	15.3	15.8	16.1	16.4	16.8
HastelloyX	LP-DED	12.6	14	14.5	14.8	15	15.4	15.9	16.2	16.5	16.8
Haynes 214	L-PBF	12.4	13.7	14.1	14.4	14.7	15.0	15.5	16.4	17.5	/
Haynes 230	L-PBF	11.1	12.4	12.9	13.3	13.7	13.9	14.6	15.2	15.7	11.1
Haynes 230	LP-DED	11.6	12.6	13.1	13.4	13.7	13.9	14.5	15	15.5	16.1
Haynes 282	L-PBF	/	11.5	12.3	12.9	13.3	13.6	14.3	14.9	16	17.3
SS 316L	LP-DED	14.4	16.1	16.7	17.1	17.4	17.8	18.1	18.5	18.8	19.1
15-5, H900	LP-DED	9.7	10.5	10.9	11.3	11.6	11.9	9.7	9.8	11.1	/
15-5, H1150	LP-DED	10.9	11.9	12.5	12.9	13.2	13.2	12.6	12.5	13.3	14.4



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

Key Properties – Typical Testing

Tensile - # specimens at each						
-320 F	70 F	400 F	800 F	1200 F	1600 F	1800 F
6	6	6	6	6	6	6
	Total	42				

Example Only / conditions based on alloy family

LCF - # specimens at each ; Various Strain Ranges						Varies for each alloy	
-320 F	70 F	400 F	800 F	1200 F	1600 F	Strain Amplitude	Strain Rate
3	3	3	3	3	3	1.00%	1.00%
3	3	3	3	3	3	0.70%	1.00%
	3					0.50%	1.00%
	3					0.30%	1.00%
	3					0.20%	1.00%
	Total	45					
						R=-1	
						Triangular	

Test conditions depend on alloy, trying to keep general comparisons as much as feasible

- Additional samples used for heat treatment evolution and characterization, metallography, and SEM
- Min 2 samples for conductivity and CTE
- Reserve samples also used for non-conformance testing (fractures outside gages, etc)
- Additional samples used for more testing of Tensile, LCF, HCF, other properties

New Alloy Development to Improve Performance

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

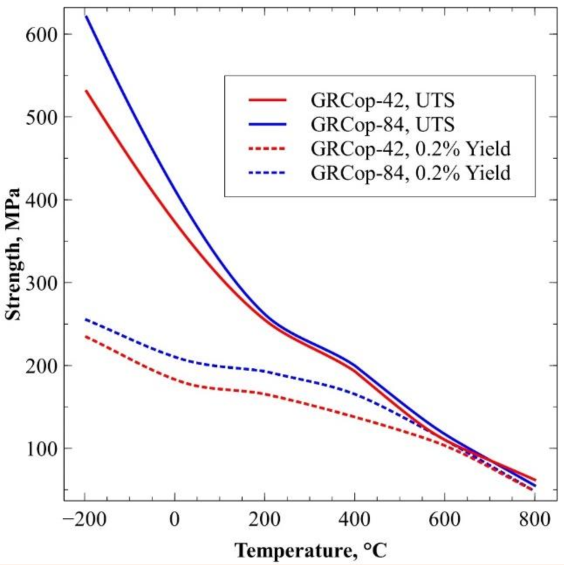

GRX-810 L-PBF

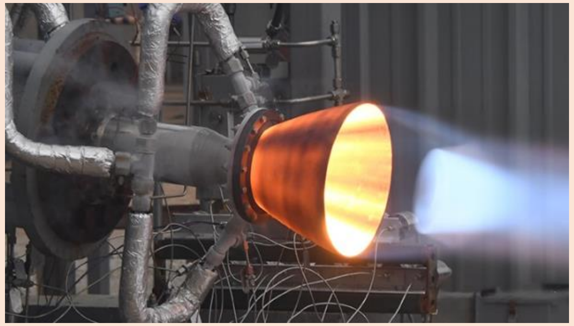

C103 L-PBF

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

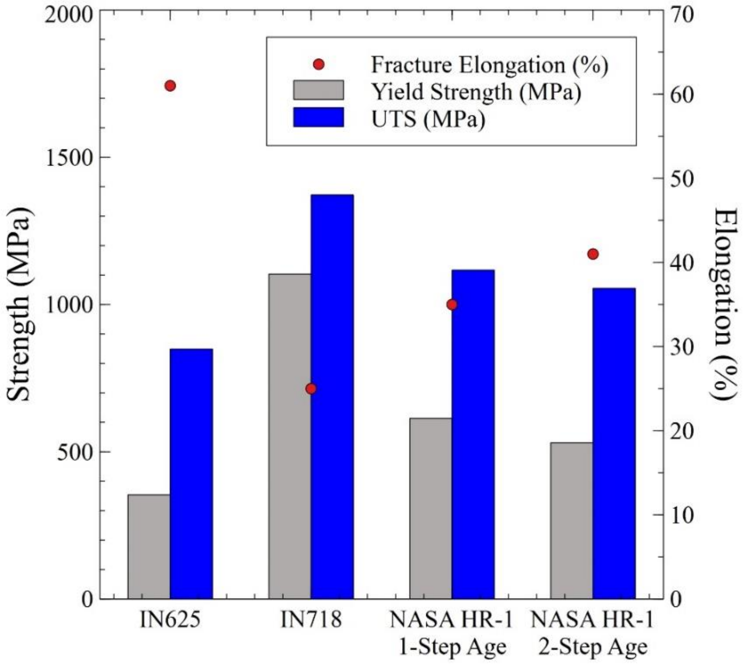
Enabling New Alloy Development using Additive Manufacturing

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

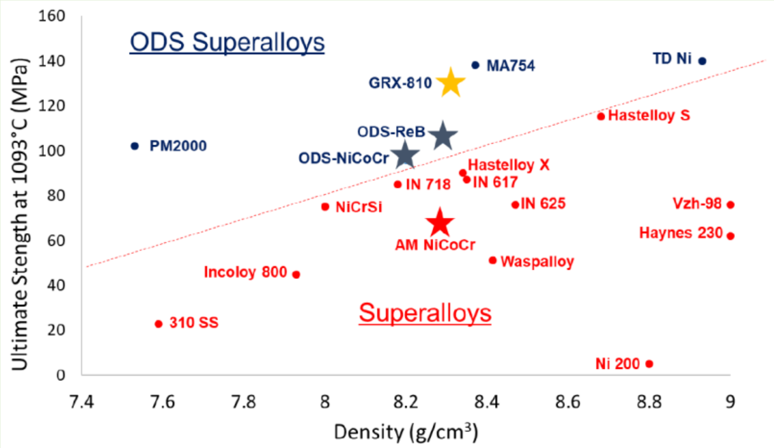




NASA HR-1, high strength superalloy for hydrogen environments



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



Ref: Tim Smith, Christopher Kantzos / NASA GRC

Conclusions

- NASA is systematically maturing the initial AM material property database for over 55+ alloys to accelerate the initial trades and preliminary design.
  - Characterization and evolution of microstructure through various heat treatments.
  - Selection of proper heat treatment including HIP.
  - Mechanical and thermophysical testing after heat treatment determined.
  - Optical, SEM, EBSD, CT, fractography characterization of samples.
  - Summary of all properties and publications for each alloy.
- NASA is investing in new alloy development using various additive manufacturing processes (PBF and DED) that can yield performance improvements over traditional alloys.
- As AM parts increase complexity, process modeling is being evolved to help successful builds, but detailed data required.

Other NASA presentations at ICAM

- Doug Wells* – Monday AM “Fatigue Behavior and Modeling of AM 718”
- William Tilson/Doug Wells – Monday PM “Developing Approaches for Certification of Un-Inspectable Fracture Critical AM Components”
- Paul Gradl* – Monday PM “Impact of Surface Finishing/Texture on Fatigue Performance of L-PBF GRCop-42”
- Edward Glaessgen* – Monday PM “Development of a Roadmap for Computational Materials-informed Qualification and Certification of Process Intensive Metallic Materials”
- Doug Wells* – Monday PM “PBF-LB Process Qualification”
- Mallory James* – Monday PM “Std Guide for Density Measurement of AM Parts”
- Fiske/Clinton/Effinger* – Application of AM in Construction on Earth and Beyond
- Paul Gradl* - Weds PM “Thickness Size Effects on Laser PBF and LDED GRCop-42 Copper Alloys”
- Paul Gradl* - Weds PM “A Comprehensive Study on Tensile Properties of AM Ni-Based Superalloys from Cryogenic to Elevated Temperatures”
- Paul Gradl* - Thurs AM “Internal Channel Polishing and Controlled Orifice Geometry Modification via Chemical Polishing for Liquid Rocket Engine Fuel Injector Optimization”
- Paul Gradl – Thurs AM “Having a Come-Apart; Lessons Learned from AM Hardware Failures
- Erin Lanigan – Thurs AM “Role of NDE and In Situ Process Monitoring in Managing Risk of AM Space Hardware”
- Paul Gradl* - Thurs PM “The Effect of Post-Processing on Tensile and Fatigue Behaviors of AM Haynes 282 Superalloy at Cryogenic and Elevated Temperatures
- Erin Lanigan/Doug Wells* – Thurs AM “In Situ Monitoring for AM Qualification and Certification”
- Paul Gradl* – Fri AM “Making and Breaking the Rules for DED Design”
- Paul Gradl and Alison Park – Fri AM Panel “The Path to Production for Space Exploration for AM”

*Partnered with external partners

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Photo: NASA/Ben Smegelsky

Acknowledgements

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Bhaskar Dutta / DM3D
Rem Surface Engineering
Launcher Space for C-18150 L-PBF
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AME
Elementum 3D
Castheon (ADDMan)
Powder Alloy Corp
Praxair
ATI

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Test Stand 115 crew
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Matt Medders
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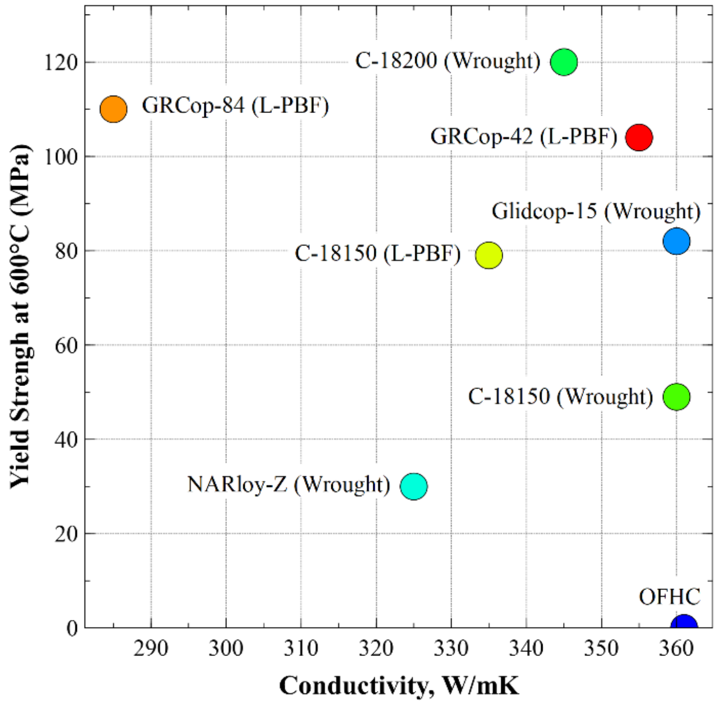
Dwight Goodman
Will Brandsmeier
Jonathan Nelson
Bob Witbrodt
Shawn Skinner
Will Evans
John Ivester
John Bili
Will Tilson
Zach Jones
Dave Ellis
Jim Lydon

Judy Schneider / UAH
PTR-Precision Technologies
Westmoreland Mechanical Testing
David Myers
Ron Beshears
James Walker
Steve Wofford
Johnny Heflin
Mike Shadoan
Keegan Jackson
Many others in Industry, commercial space and others

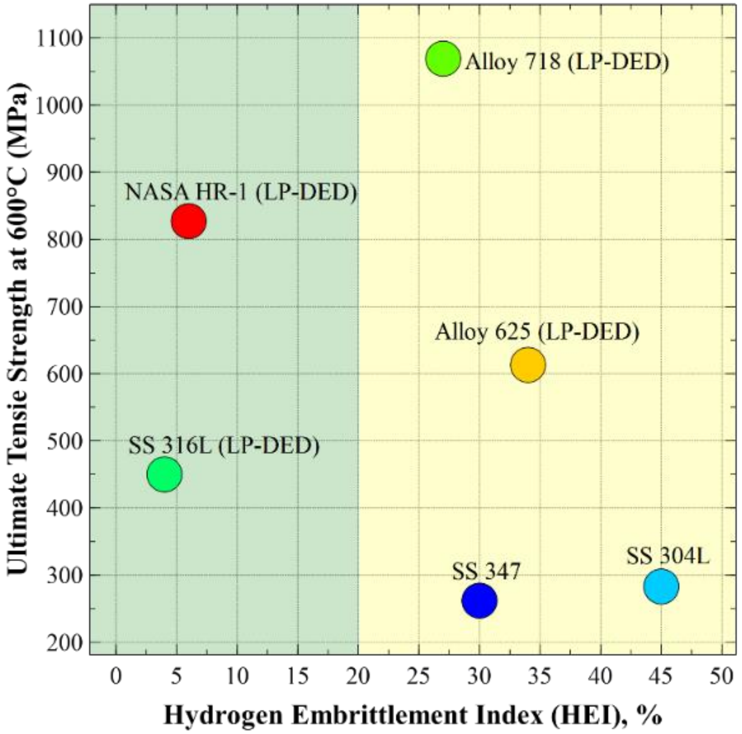
GRCop-42 and GRCop-84 for High Conductivity

- GRCop-42 and GRCop-84 (Cu-Cr-Nb) offer high conductivity (>350 W/mK) and high strength at elevated temperatures (up to 800 °C).
- Oxidation and blanching resistance during thermal and oxidation-reduction cycling.
- Established powder supply chain and commercial supply chain.
- Significant maturity in characterization and hot-fire testing (high TRL).
- Over 41,000 seconds of hot-fire time and 1,100 starts on >30 chambers

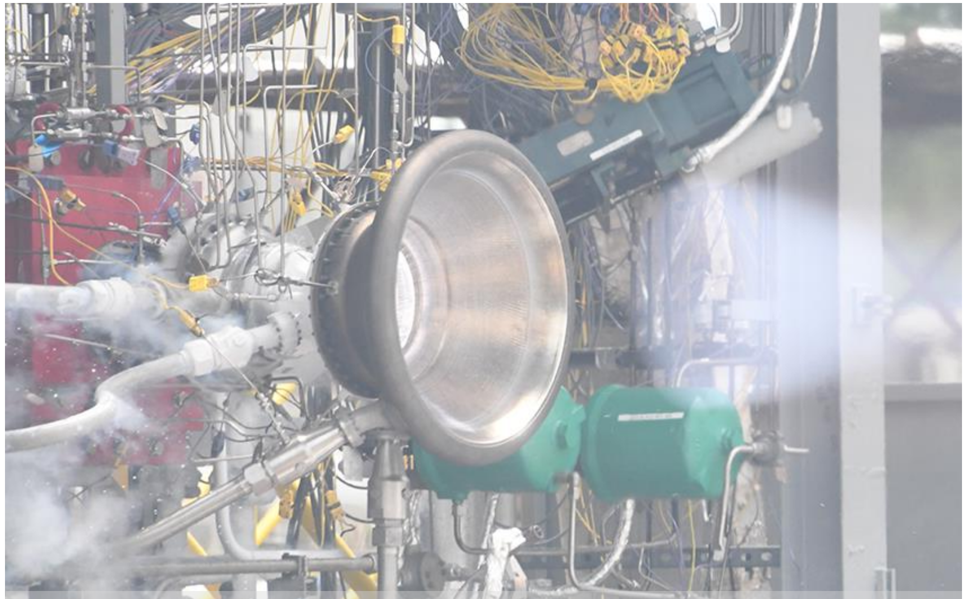


NASA HR-1 Hydrogen Resistant Alloy

- NASA HR-1 (Fe-Ni-Cr) is a hydrogen resistant high strength superalloy.
- Formulated for AM processes for low cycle fatigue, ductility, and H2 resistance properties.
- Targeted use is Laser Powder Directed Energy Deposition (LP-DED) for large scale nozzles.
- Supply chain maturity for powder feedstock, build parameters, and demonstrator builds.
- Single NASA HR-1 LP-DED nozzle accumulated 207 starts and >6,800 secs.



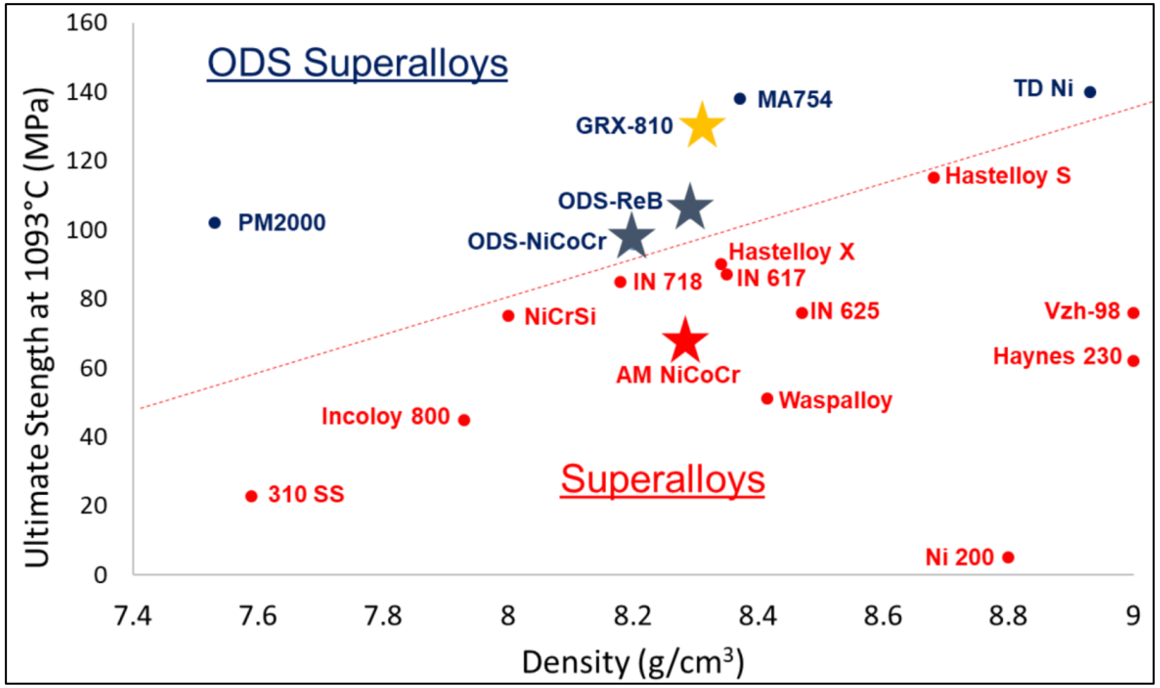
1.52 m dia. x 1.78 m height

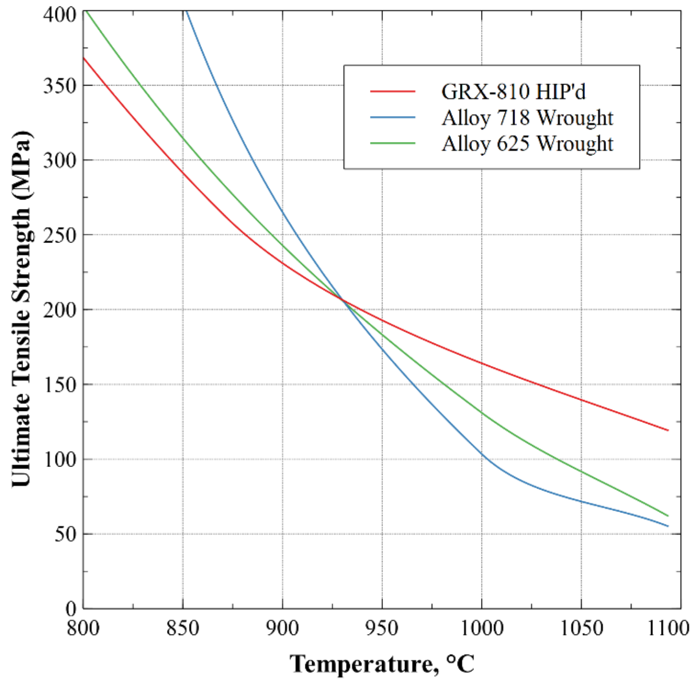


Hot-fire Testing of 156 KN LP-DED Cooled Nozzle

GRX-810 for Extreme Temperature and High Strength

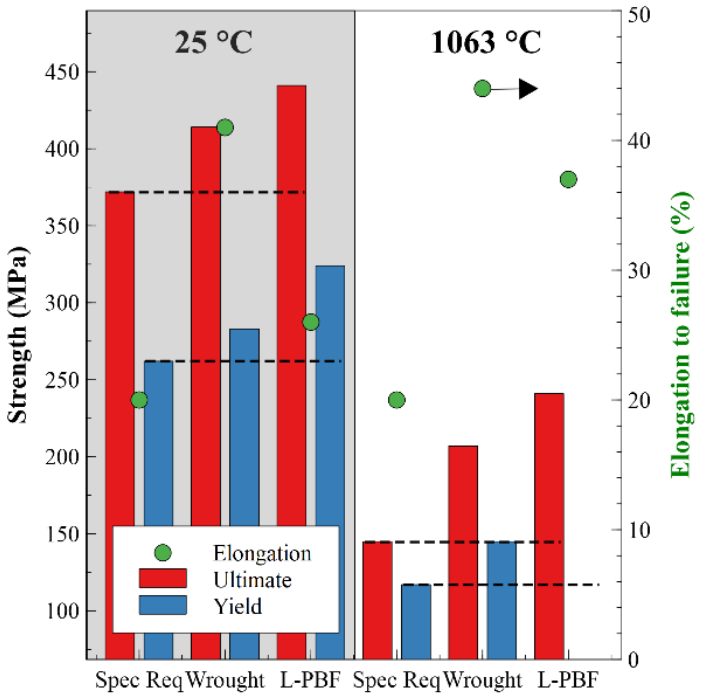
- GRX-810 (**G**lenn **R**esearch Center E**X**treme -810) is an oxide dispersion strengthened (ODS) Ni-Co-Cr alloy specifically formulated for AM using Y_2O_3 nanoparticles.
- 2x strength of standard superalloys approaching 1100 °C.
- Orders of magnitude better oxidation resistance compared to superalloys.
- Demonstrated process parameters and feasibility of powder feedstock.



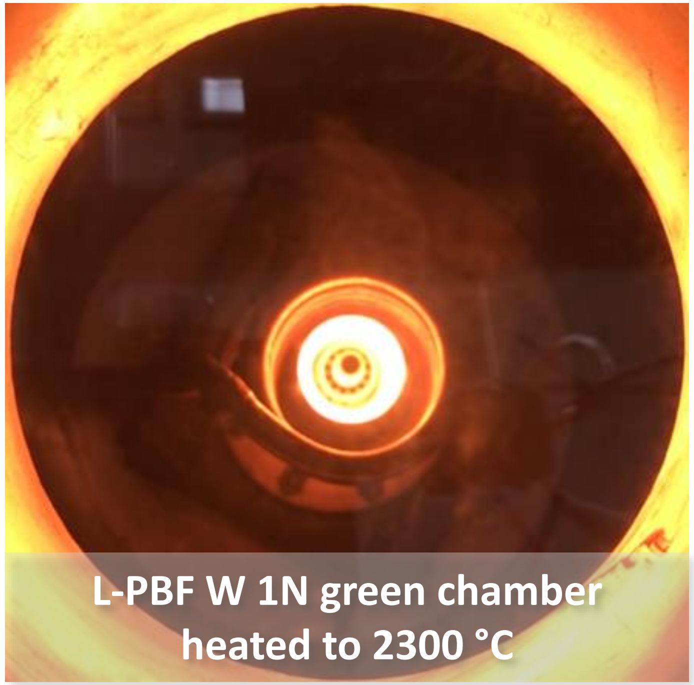


Refractory Alloys for Ultra High Temperature

- Refractory alloy development for AM allows for significant reduction in feedstock cost.
- Tungsten, C-103 has been matured with L-PBF and LP-DED processes along with feedstock.
- Mechanical properties shown to exceed specification minimums and density >99.98%.
- W, Mo, Ta, Re, and Nb alloys being developed under Refractory Alloy Additive Manufacturing Build Optimization (RAAMBO) project.



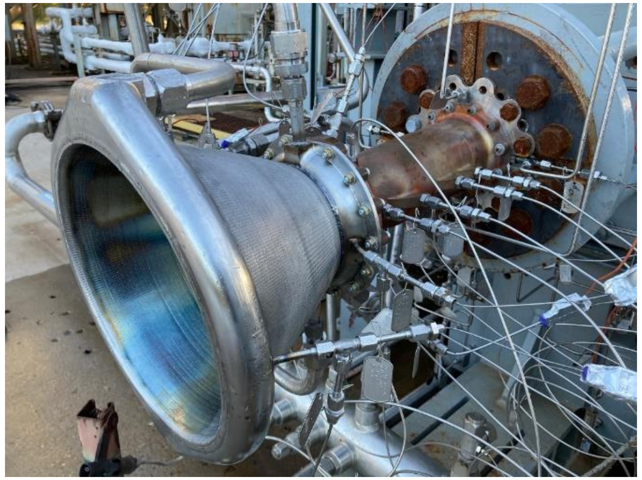
L-PBF C-103 Demonstrator Parts



L-PBF W 1N green chamber heated to 2300 °C

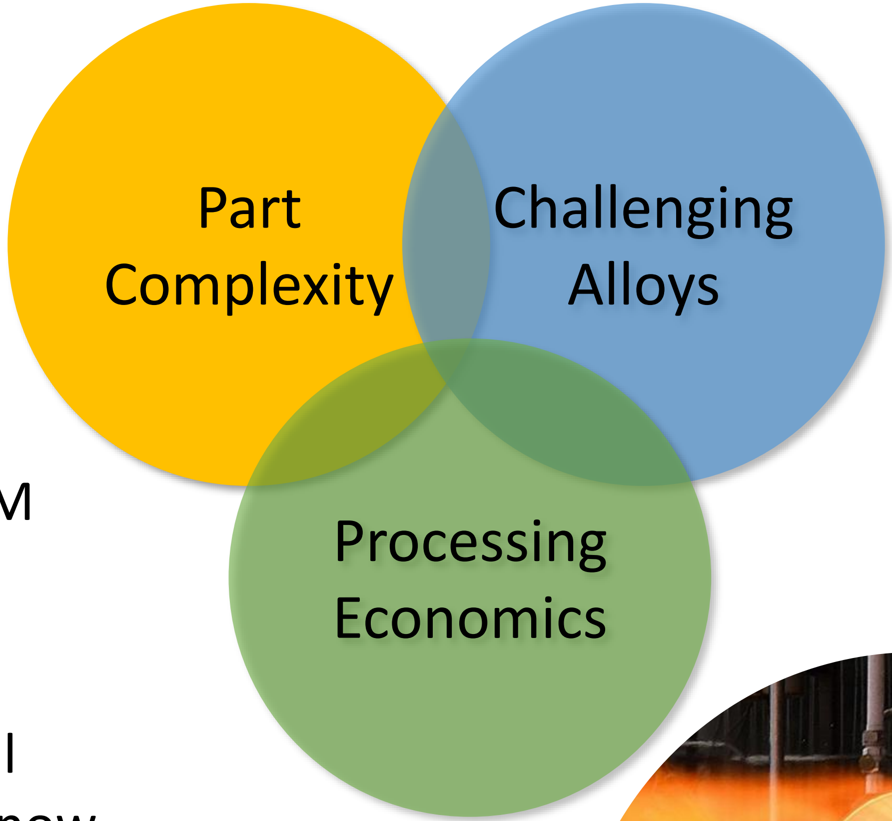
Summary

- NASA has formulated and matured novel alloys specifically intended for use with additive manufacturing for high temperature and harsh environments.
- Alloys include GRCop-42, GRCop-84, NASA HR-1, GRX-810, Refractory-based (C103).
- AM processes to manufacture components and material properties required have matured.
- NASA has accumulated over 50,000 secs and 1400 starts of hot-fire testing on these alloys.
- Commercial space is actively using these alloys for development and flight infusion.
- Data and properties available to commercial and government partners.



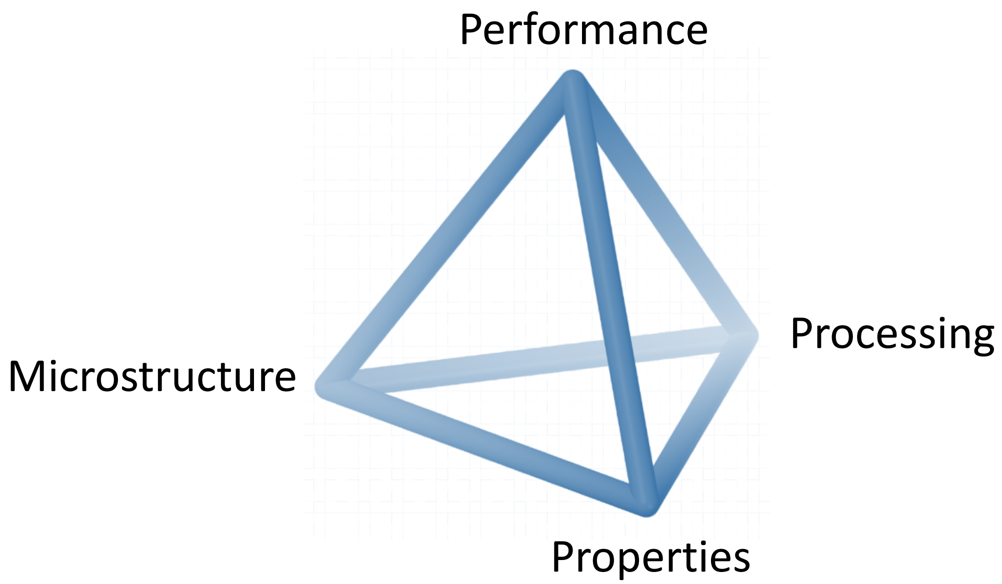

The Case for Additive Manufacturing in Propulsion

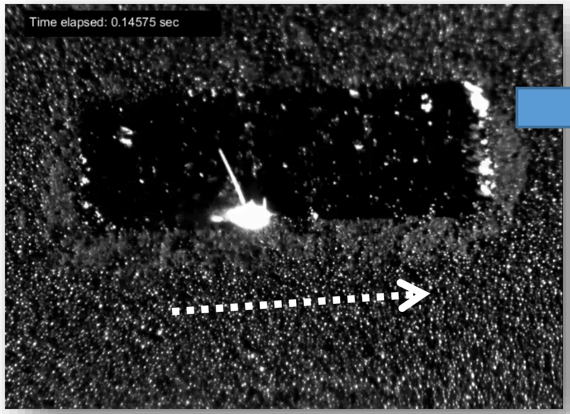
- Metal Additive Manufacturing (AM) can provide significant advantages for lead time and cost over traditional manufacturing for rocket engines.
  - Lead times reduced by 2-10x
  - Cost reduced by more than 50%
- Complexity is inherent in liquid rocket engines and AM provides new designs, part consolidation, and performance opportunities.
- Materials that are difficult to process using traditional techniques, long-lead, or not previously possible are now accessible using metal additive manufacturing.

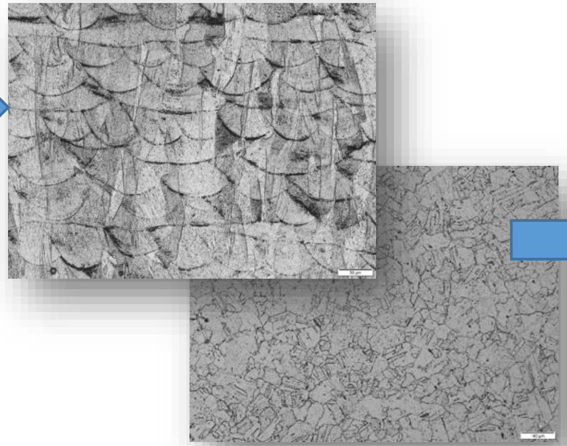


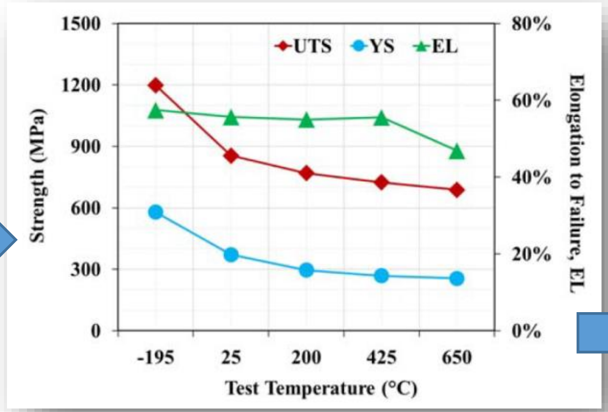
While AM has programmatic and technical advantages, material characteristics must be understood to properly apply in designs

Fundamentals are necessary for Component Applications





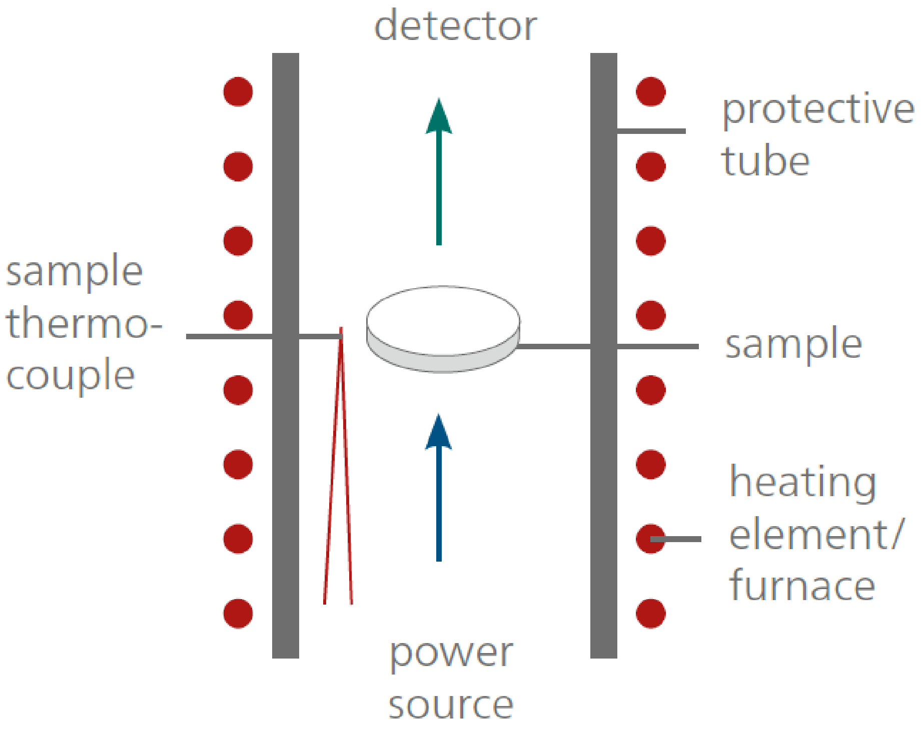


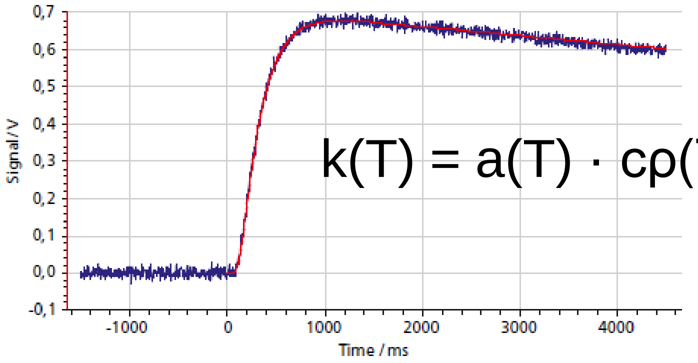


Thermal Diffusivity / Conductivity Measurement

Netzsch LFA467

Temperature range	RT - 1250°C
Thermal diffusivity	0.01 mm²/s - 2000 mm²/s
Thermal conductivity	0.1 W/(m·K) - 4000 W/(m·K)
Accuracy	Thermal diffusivity1: ± 3% Specific heat2: ± 5%
Data acquisition	2 MHz
Integrated automatic sample changer	4 insets for 1 sample each
Heating rate (max.)	50 K/min





$$k(T) = a(T) \cdot c_p(T) \cdot \rho(T)$$