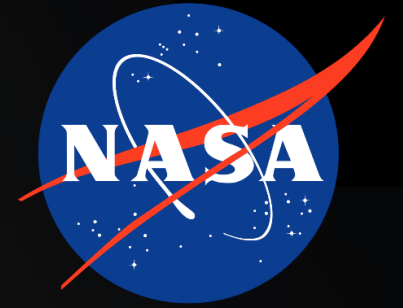




Maturation and Hot-Fire Testing of Extreme Environment Additively Manufactured Alloys for Rocket Engine Applications

Paul Gradl, Colton Katsarelis, Darren Tinker

NASA Marshall Space Flight Center

Timothy Smith, Christopher Kantzos

NASA Glenn Research Center

30 October 2024



The need for novel extreme environment alloys

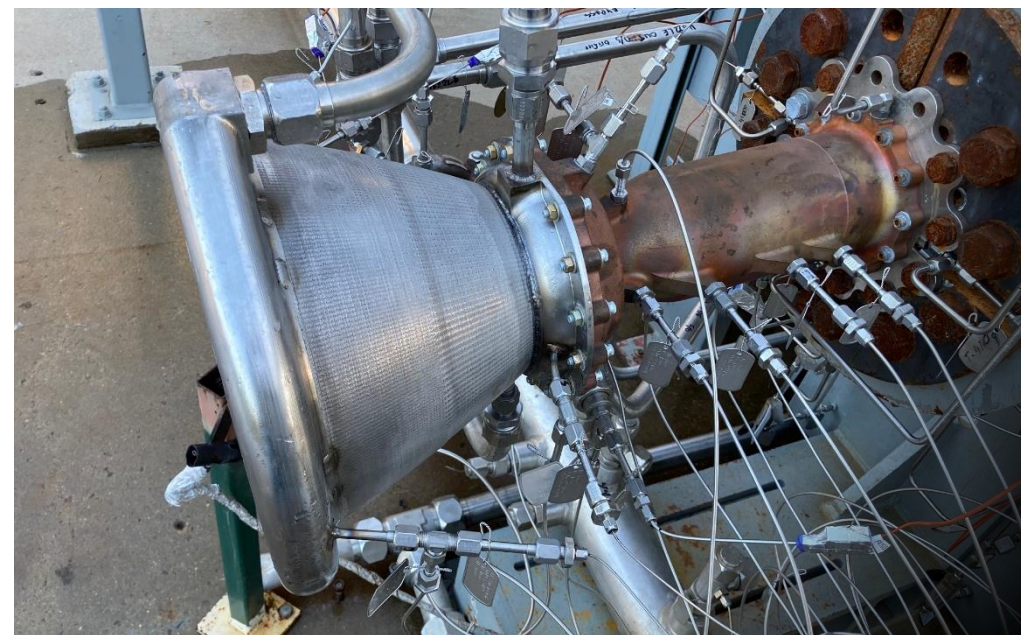
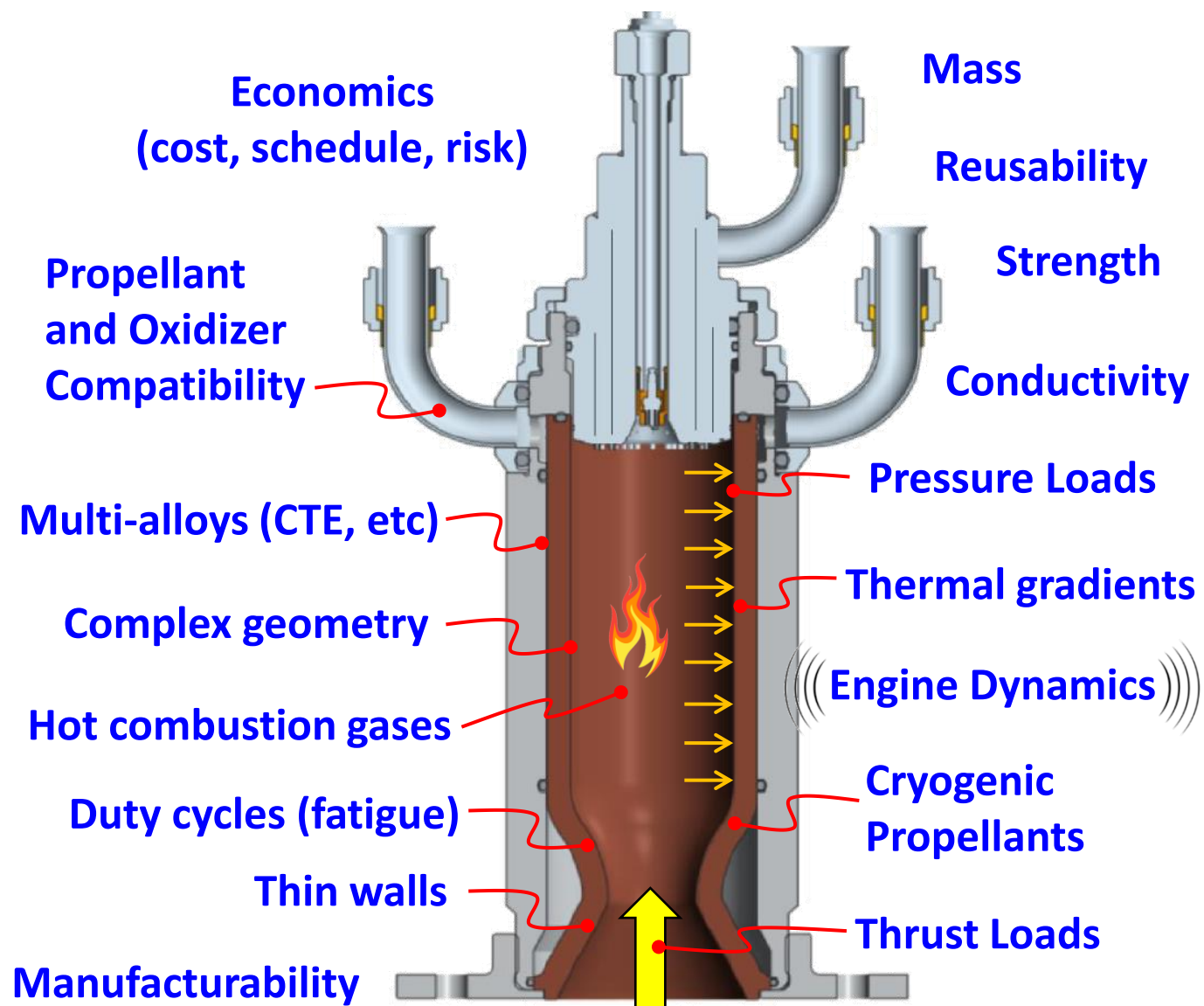


- Rocket engine components (injectors, nozzles, turbine static and rotating components, chambers) operate in extreme environments.
 - High pressure, high heat flux, high thermal gradients, long duty cycles, stress rupture (creep), oxidation
- Additive manufacturing (AM) has played a pivotal role in component development and production, but materials are still based on heritage metal alloys.
- New alloys using unique advantages of AM processing can provide a step change in properties to allow for higher performance.
- Rocket engine development and vehicle architectures are pushing for high performance resulting in higher heat loads, longer durations, reusability.
- Novel developments in materials have been advanced in the last 5 years.

There is a need to advance and infuse metal alloys using additive manufacturing processes to enable higher performing components.



Example: Combustion Chamber Material Considerations





Material Selection – General Requirements



- 1) Operate under static and dynamic loads in both tension and compression.
- 2) Compatible with fuels and oxidizers (hydrogen, oxygen, hydrocarbons).
- 3) Integrate multiple subsystems (possibly different metal alloys).
- 4) Optimized for strength-to-weight ratio (design- and metal-based).
- 5) Operate in extreme environments (cryogenic and high temperatures, high pressure, combustion, oxidizing or reducing condition).
- 6) Withstand high thermal gradients and high heat transfer in a high-heat-flux environment.
- 7) Exhibit high fracture toughness at temperature for potential impacts by domestic object debris (DOD) or foreign object debris (FOD).
- 8) Exhibit corrosion resistance when in contact with other alloys and operating fluids or contaminants.
- 9) High wear resistance for contact or running surfaces between components.



Unique alloy requirements for harsh rocket environments

High Conductivity, High Temperature, Oxidation Resistance



High Temperature, Hydrogen Resistant, High Strength-Weight Ratio



Extreme Temperature, Creep and Oxidation Resistant, High Strength-Weight



Extreme Temperature, Burn Resistant in Oxygen





NASA 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
700	Iron-Nickel	High strength and hydrogen resistance	NASA HR-1 NASA HR-2 JBK-75	Nozzles, Powerheads
800-1100	Nickel	High strength to weight Burn-Resistance	ORCAIloy-21 Alloy 754-ODS OR-F1	Preburners, 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



Maturity of Metal AM for NASA Applications

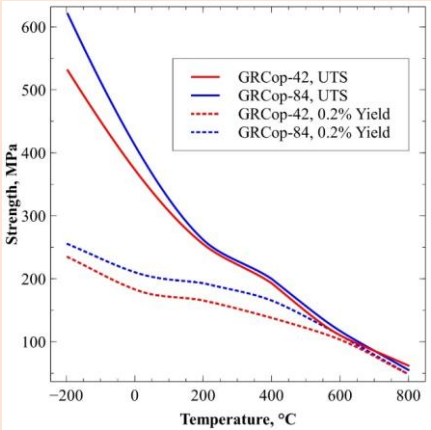
LASER POWDER BED FUSION



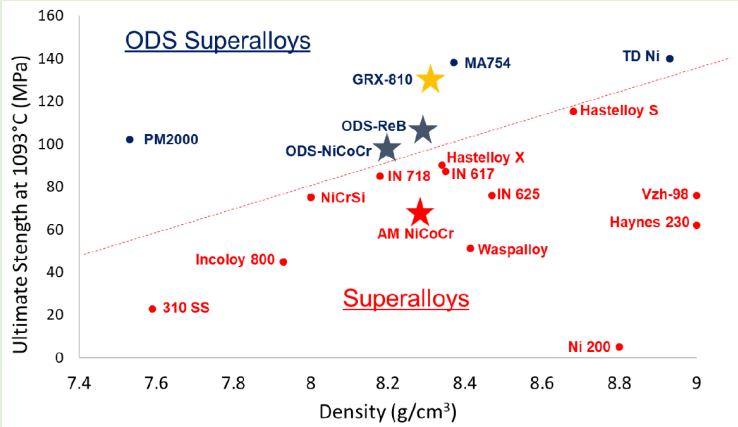
DIRECTED ENERGY DEPOSITION



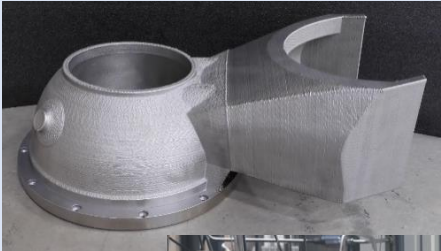
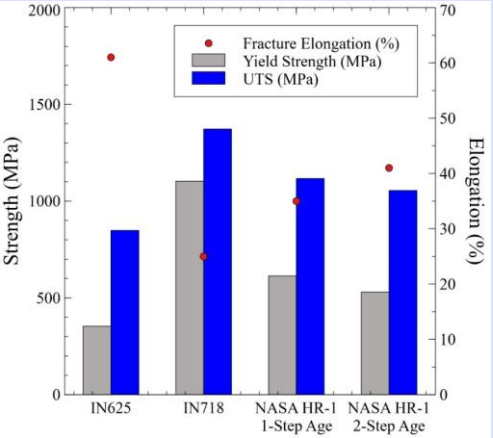
GRCo-42, High conductivity and strength for high heat flux applications



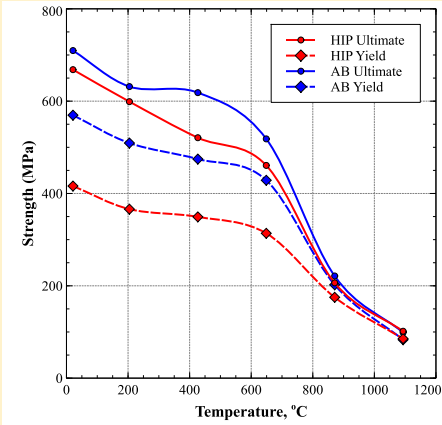
GRX-810, high strength, low creep rupture and oxidation at extreme temperatures



NASA HR-1, high strength superalloy for hydrogen environments



ORCAIloy-21, high strength burn-resistant alloy for oxygen-rich staged combustion





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 50,000 seconds of hot-fire time and 2,000 starts on >40 chambers

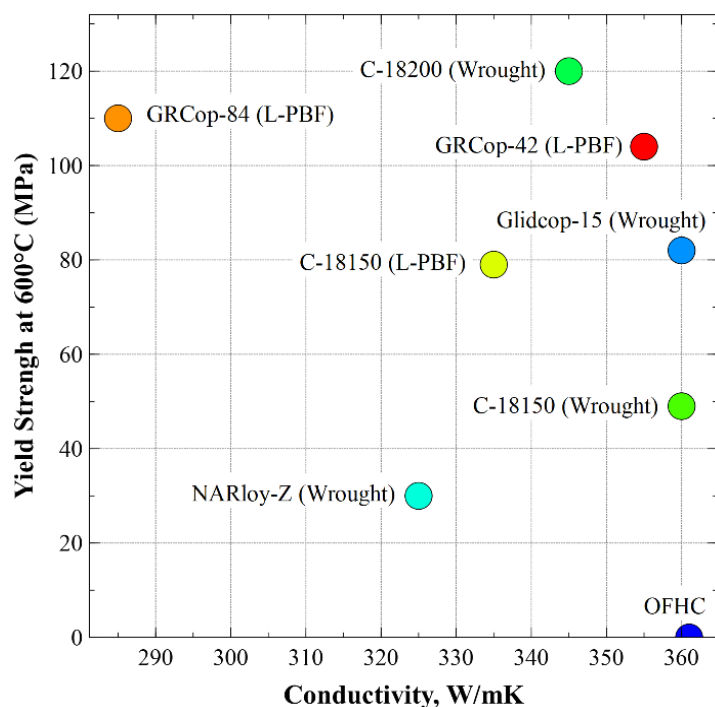
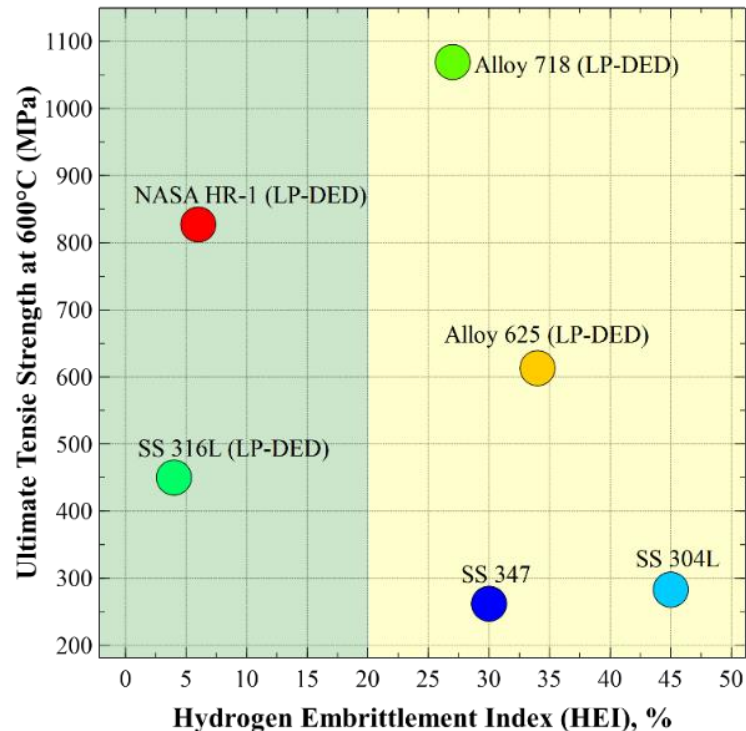


Image courtesy: Relativity Space



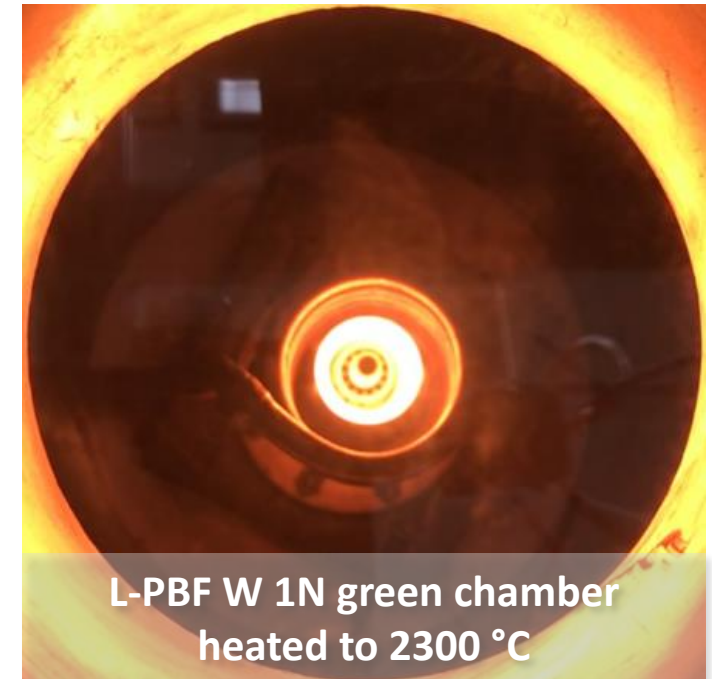
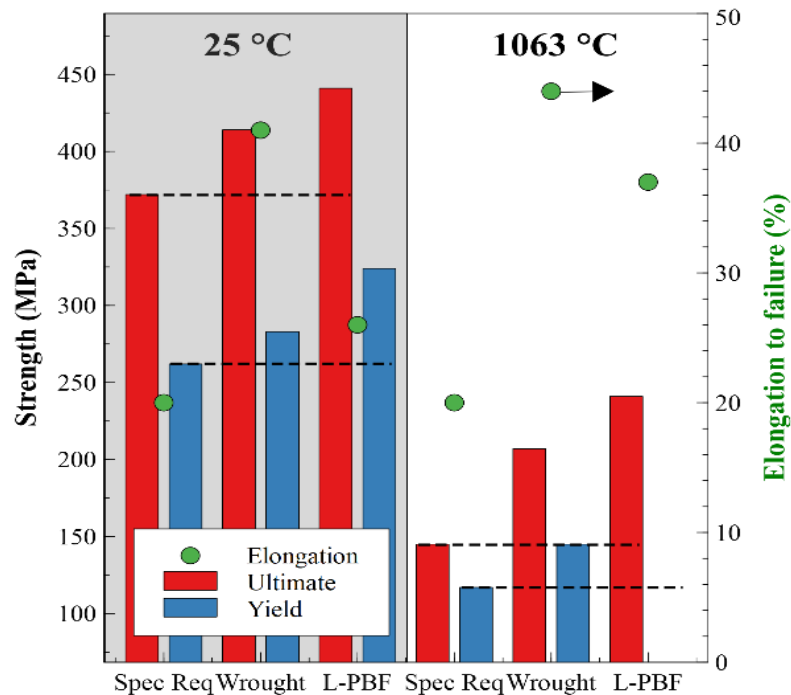
NASA HR-1 Hydrogen Resistant Alloy

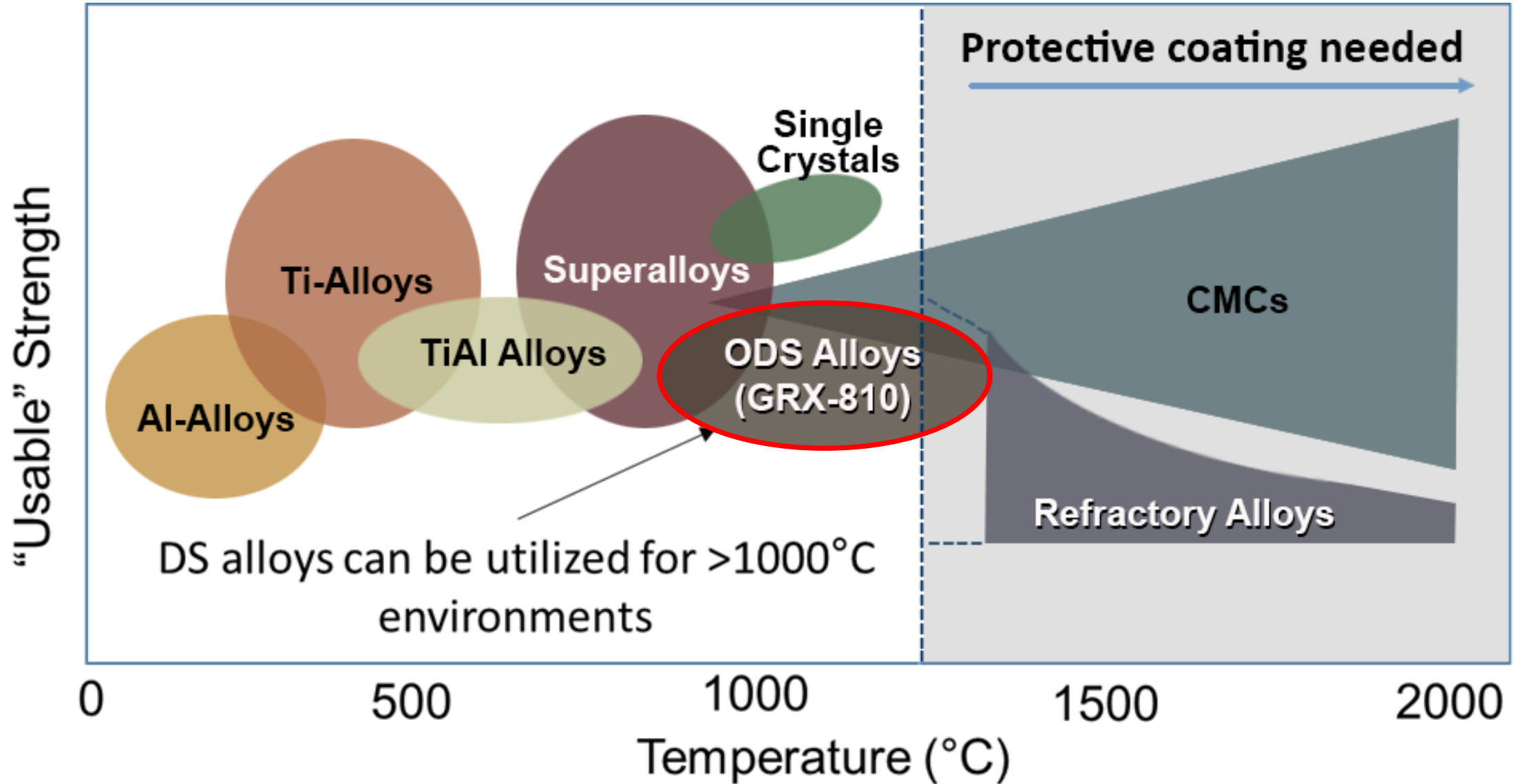
- NASA HR-1 (Fe-Ni-Cr) is a hydrogen resistant high strength superalloy.
- Formulated for AM processes for low cycle fatigue, ductility, and H₂ resistance properties.
- Targeted use is Laser Powder Directed Energy Deposition (LP-DED) for large scale nozzles.
- Supply chain maturity for powder feedstock, build parameters, and demonstrator builds.
- Single NASA HR-1 LP-DED nozzle accumulated 207 starts and >6,800 secs.
- NASA HR-2 being developed for improved strength in L-PBF applications.

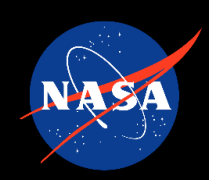


Refractory Alloys for Ultra High Temperature

- 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%.
- New refractory alloys being explored and developed under Refractory Alloy Additive Manufacturing Build Optimization (RAAMBO) project focused on Nb, Mo, W.



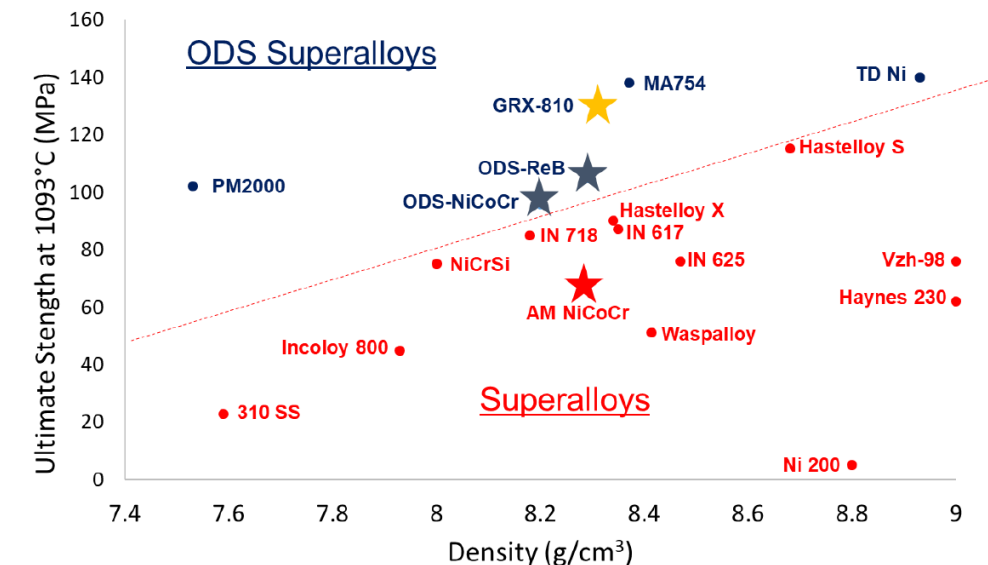
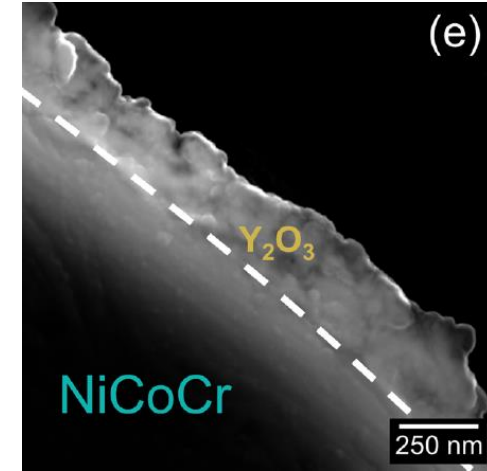
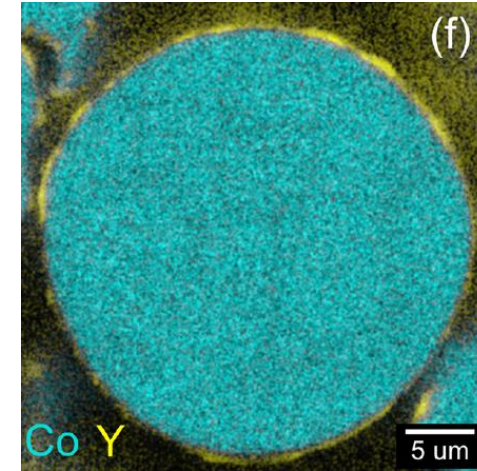


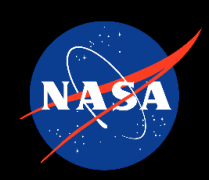


GRX-810 Overview

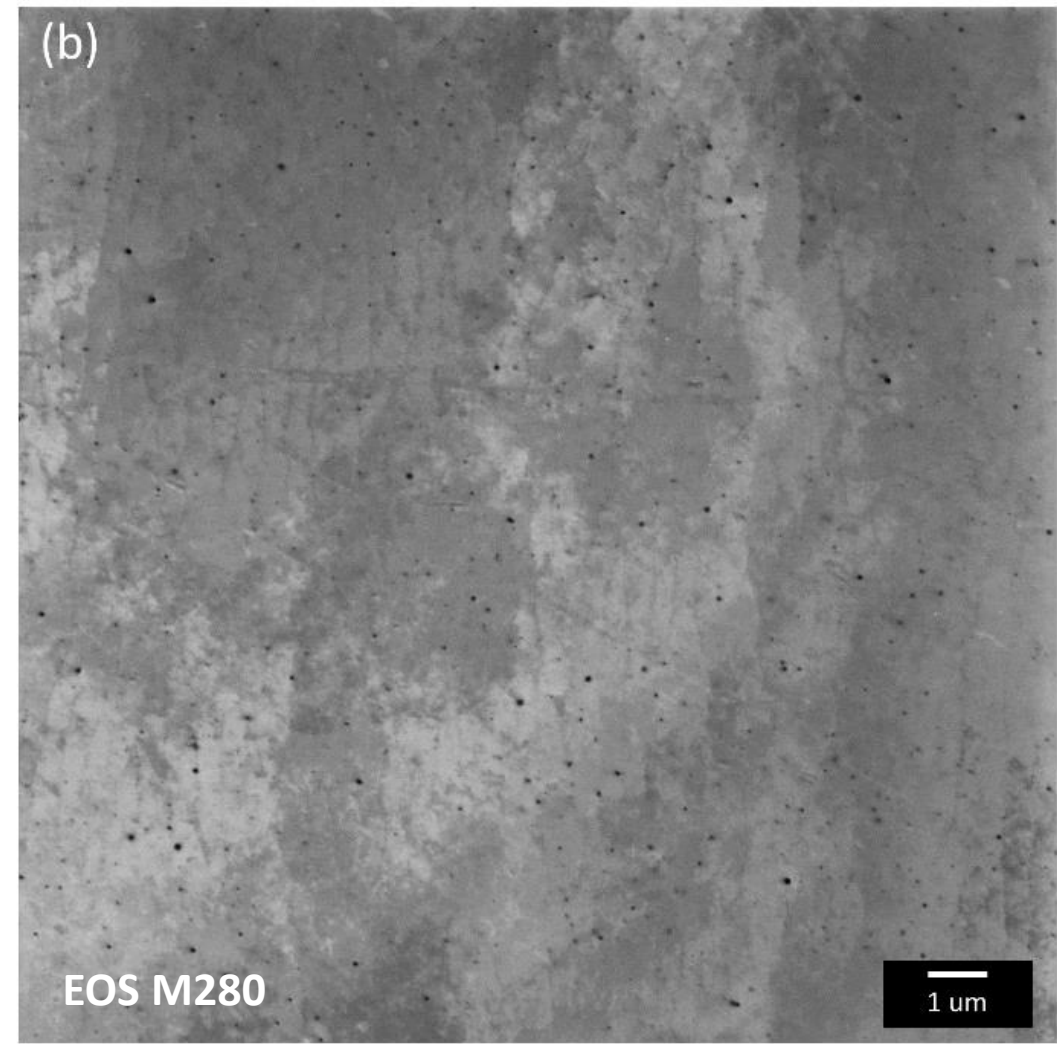
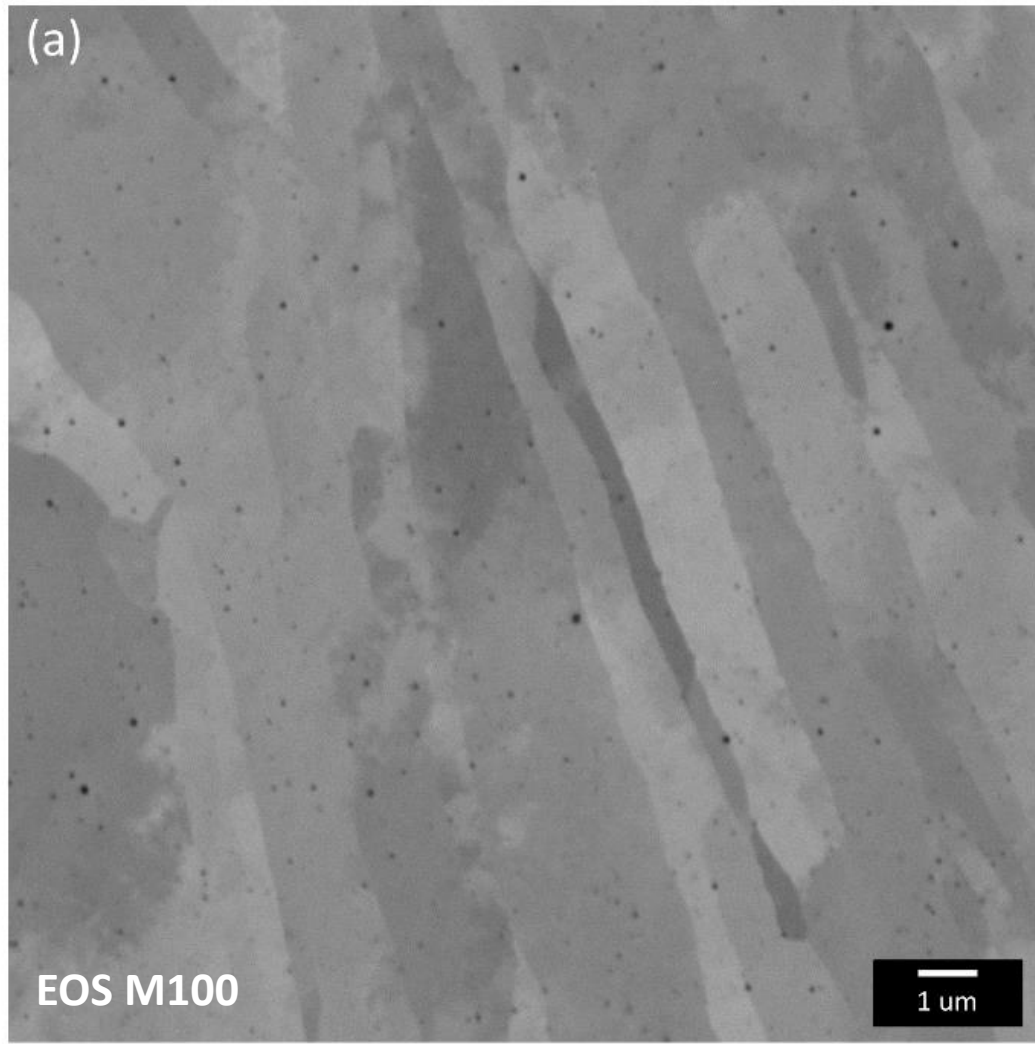


- **Glenn Research Center eXtreme** temperature alloy (GRX-810) was developed using Integrated Computational Materials Engineering (ICME) – significantly reducing development time.
- Ni-Co-Cr medium entropy alloy using Laser Powder Bed Fusion (L-PBF).
- Uses unique resonant mixing ODS powder coating process with nano-scale Y_2O_3 .
- Significantly improved properties:
 - 2x strength at elevated temperatures approaching $1300^{\circ}C$
 - 1,000x better creep rupture
 - 2x better resistance to oxidation
- Designed for simplified heat treatment – as-built or Hot Isostatic Pressing (HIP).

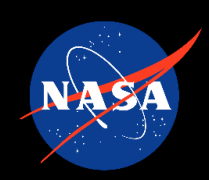




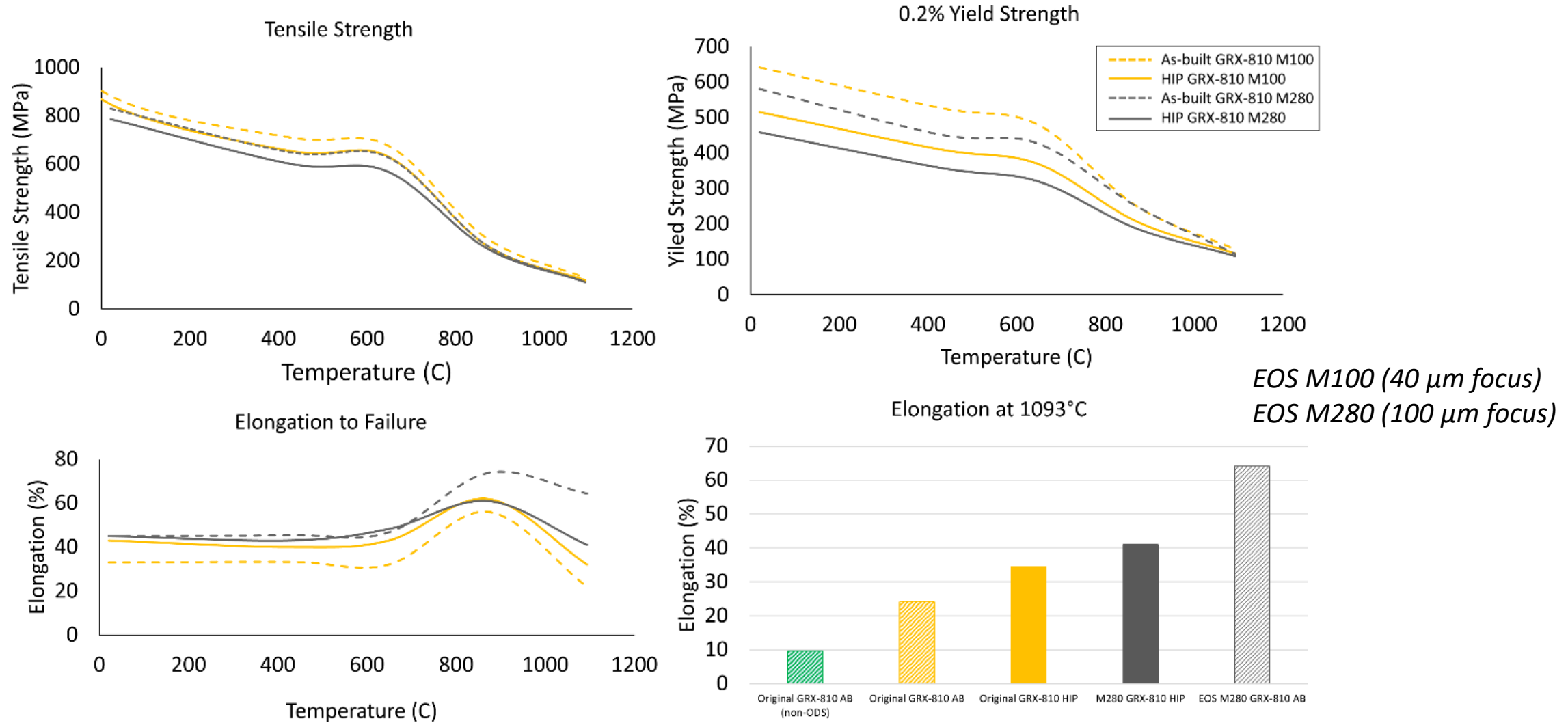
SEM Images of GRX-810 using L-PBF



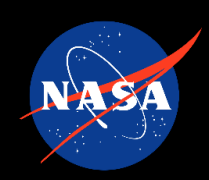
Secondary electron SEM micrographs of (a) Lot 1 GRX-810 produced using an EOS M100 and (b) Lot 2 GRX-810 produced using an EOS M280. The fine circular dark features are nano-scale Y₂O₃ particles.



GRX-810 Mechanical Properties



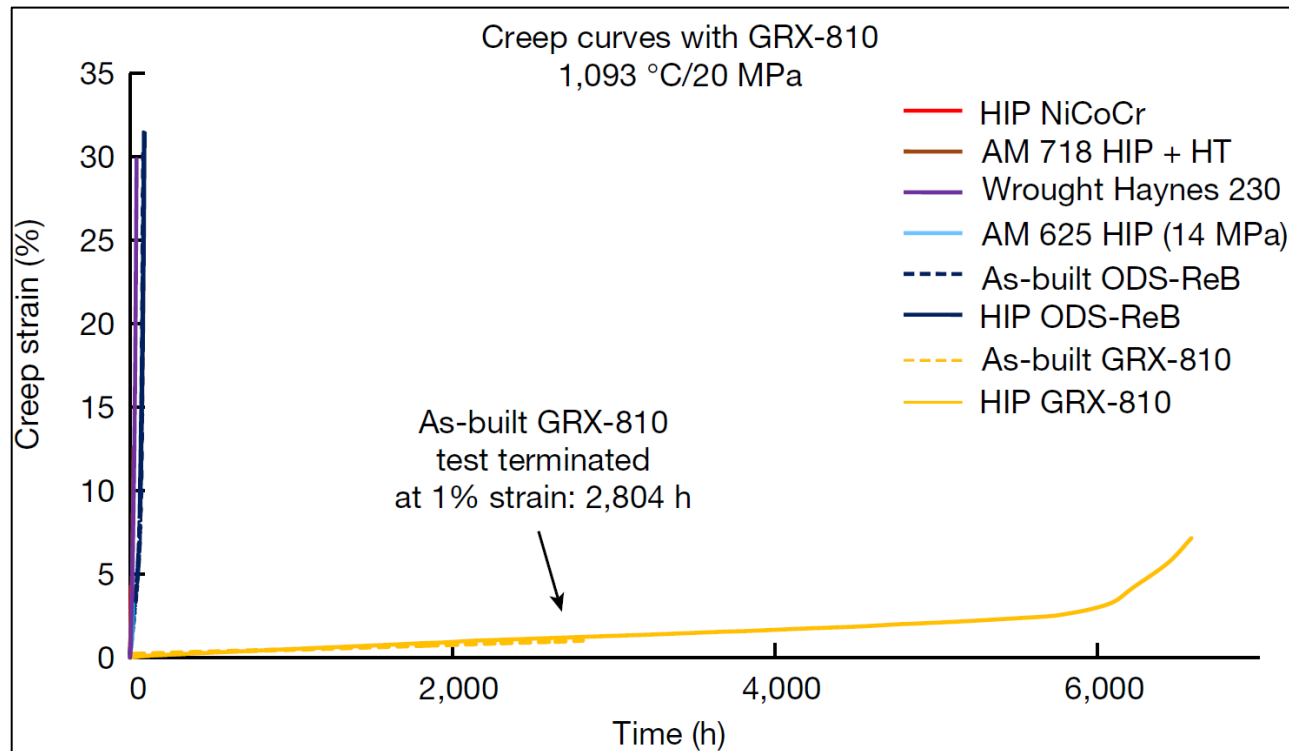
Tensile properties of GRX-810 produced using an EOS M100 and using an EOS M280. Notable differences in room temperature strength and high temperature elongation were observed between the different lots.



GRX-810 Stress Rupture and Oxidation

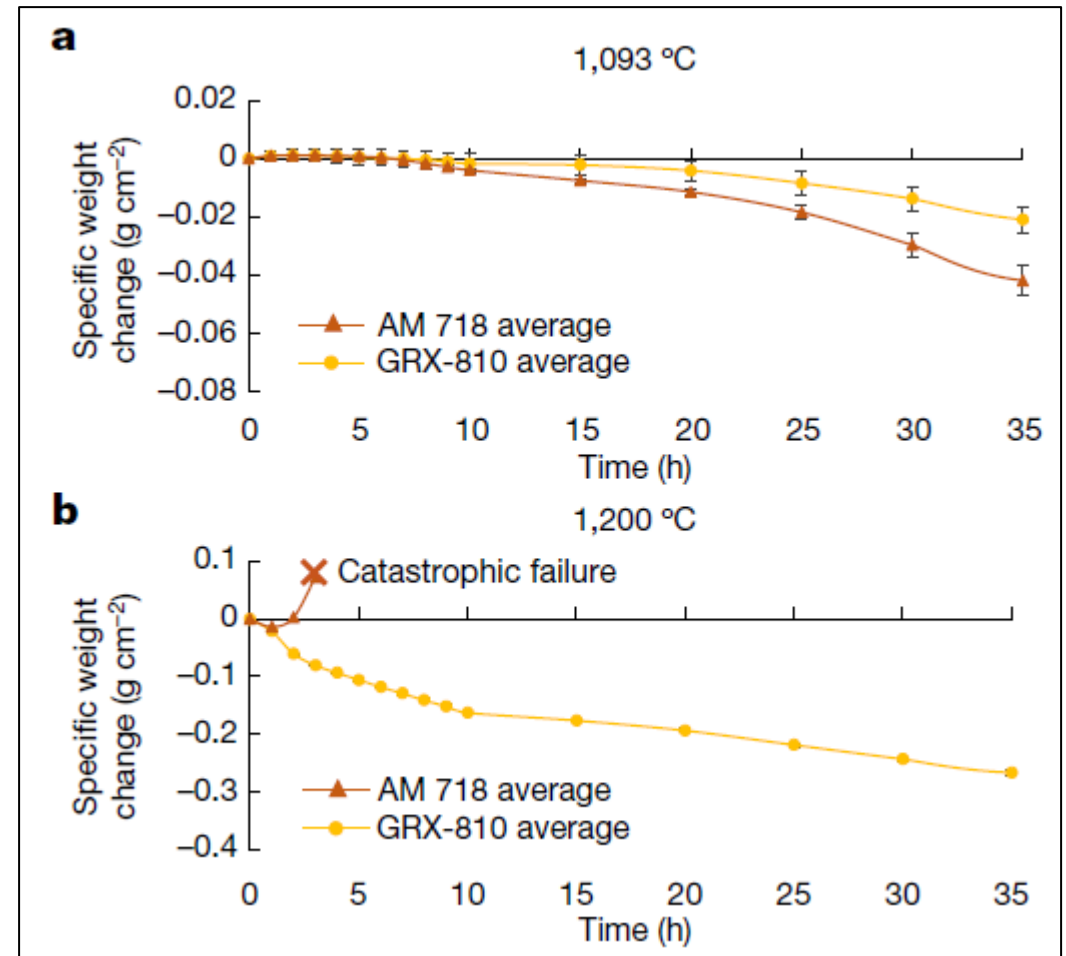


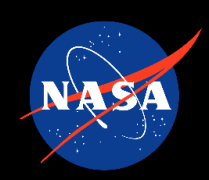
Stress rupture shows 1,000x improvement over typical Ni-based superalloys



Creep results from M100 and M280 showing similar trends, although M280 performing better

Cyclic oxidation at 1093°C and 1200°C

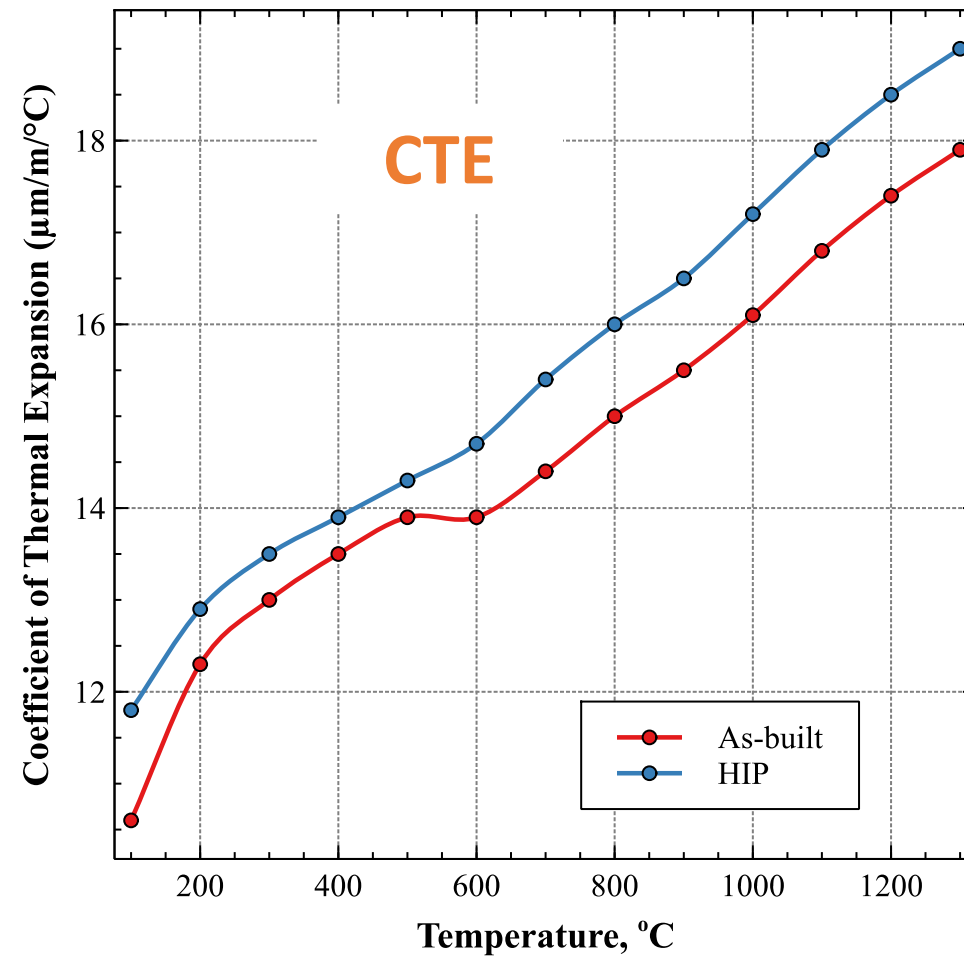
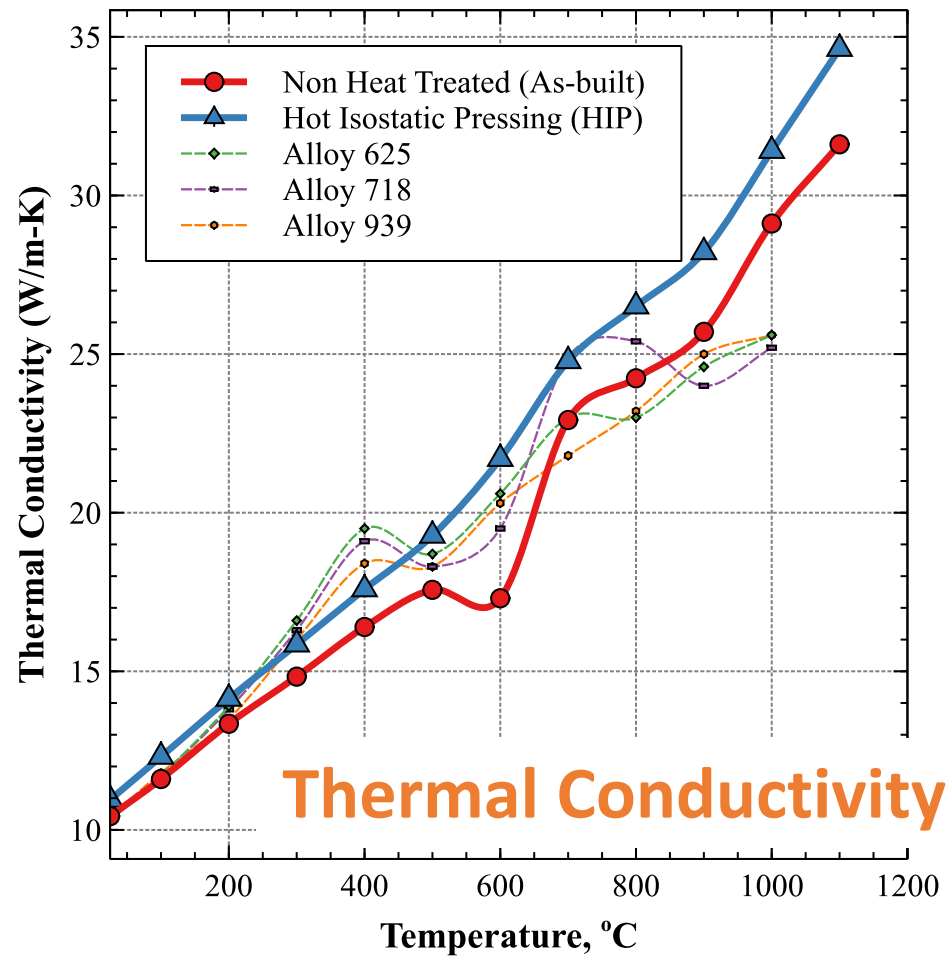




GRX-810 Thermophysical Properties

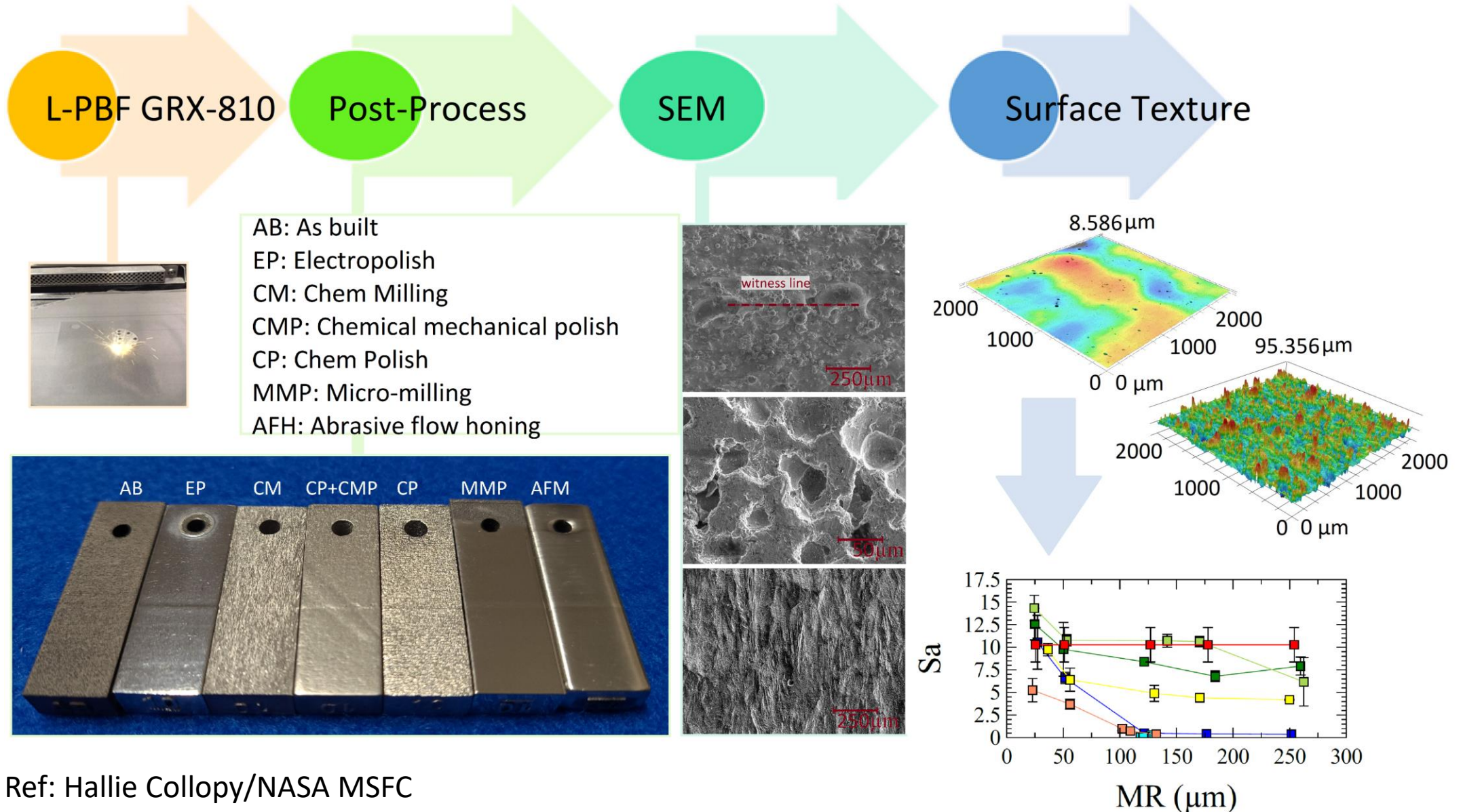


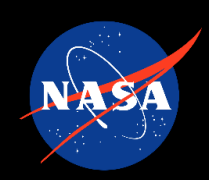
Thermal Conductivity and Coefficient Thermal Expansion (CTE) are similar to Ni-based superalloys



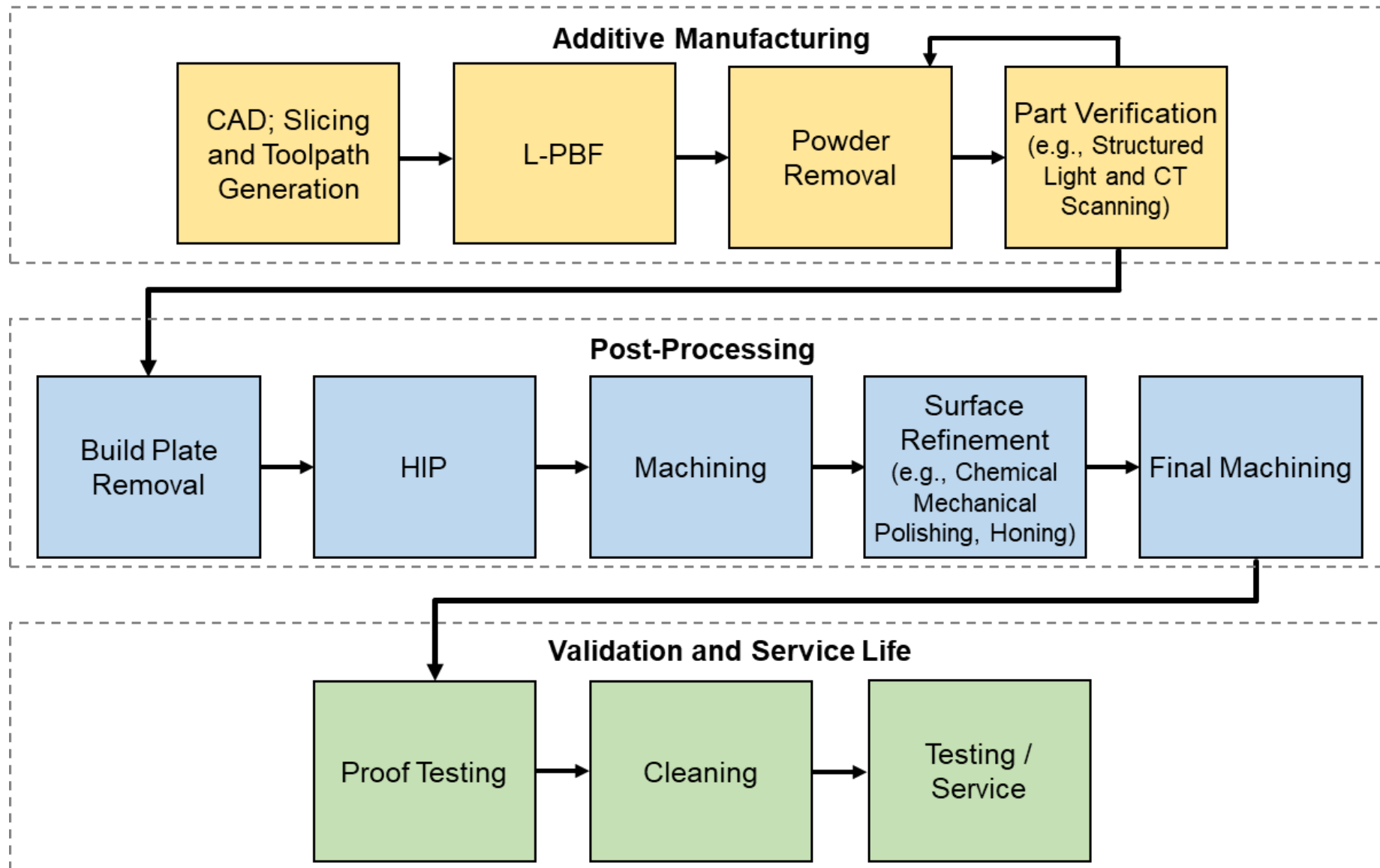


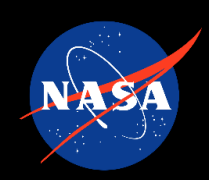
GRX-810 Post-Processing Development



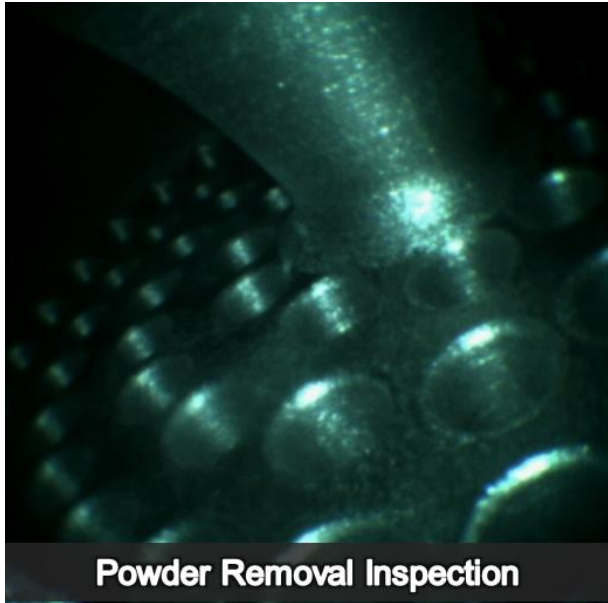


Typical Process Flow of GRX-810 Hardware

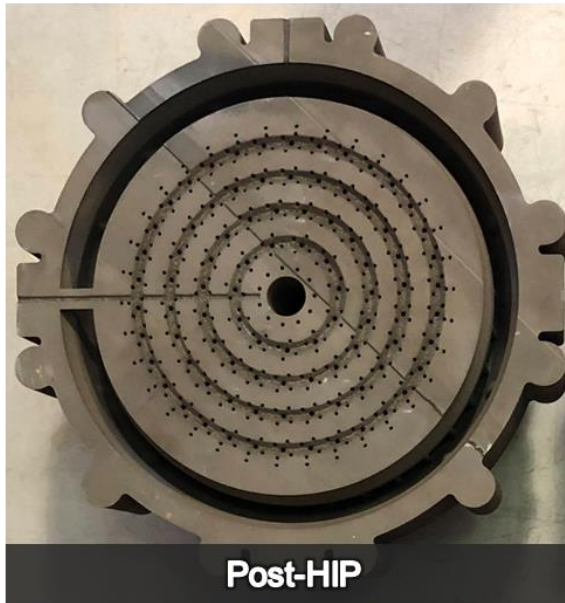




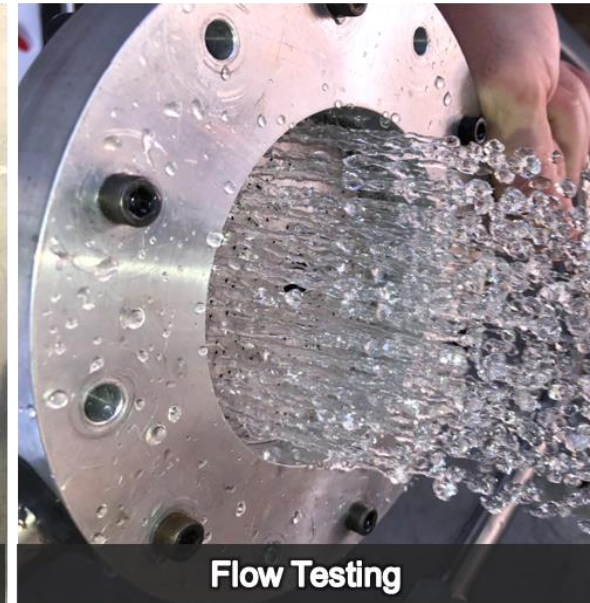
Typical Process Flow of GRX-810 Hardware



Powder Removal Inspection



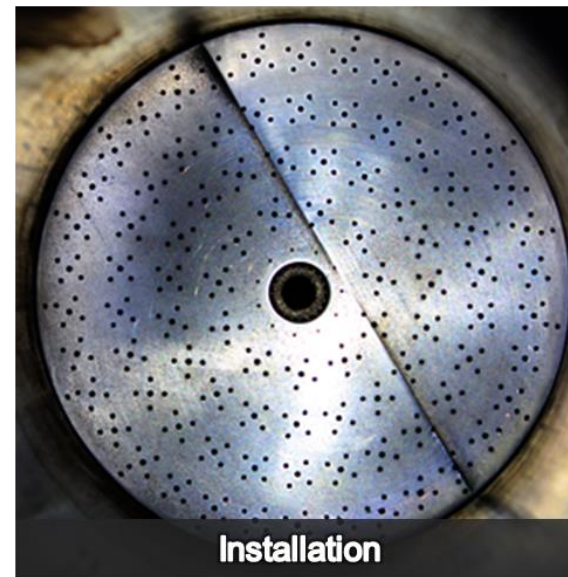
Post-HIP



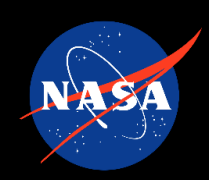
Flow Testing



Face Polish



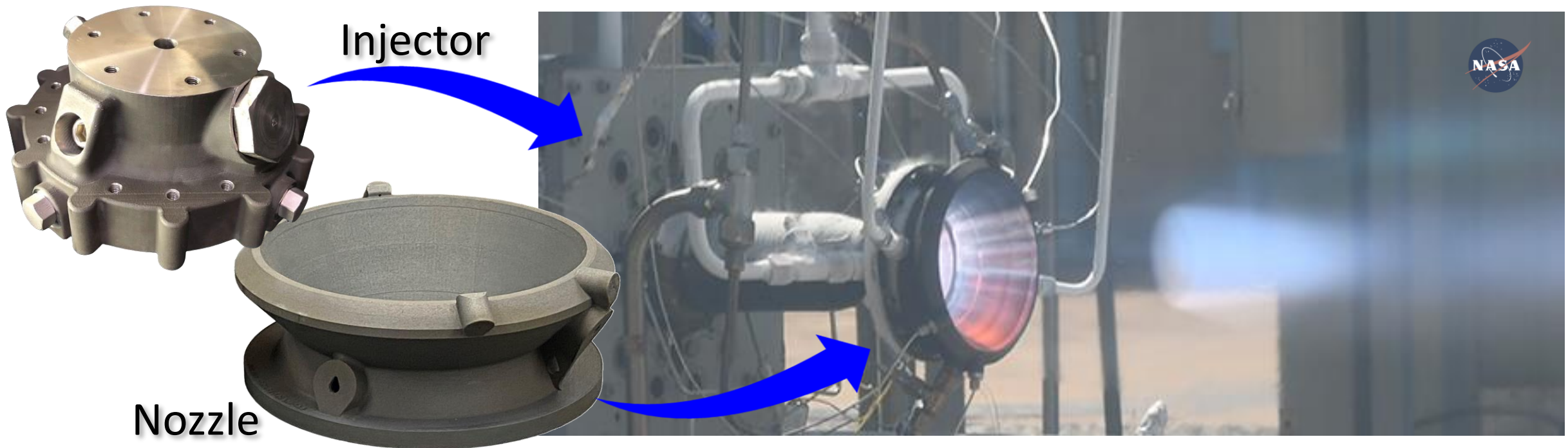
Installation



AM Maturation Through Rigorous Testing

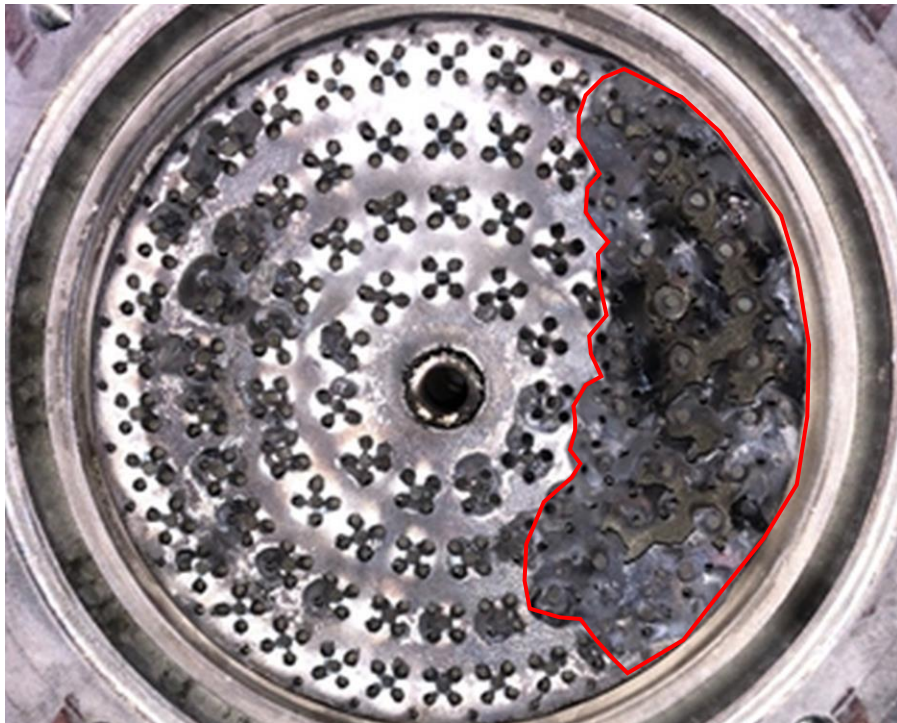


- Hot-fire testing of GRX-810 pentad injectors and channel-cooled nozzles using L-PBF GRX-810
- Increase TRL from 3 → 5
- **GRX-810** Injector achieved 84 starts and 2,228 sec (Liquid Oxygen/Methane)
- Similar injectors with Inconel 718/625 would see erosion after 8-10 starts
- **GRX-810** nozzle achieved 91 starts and 2,309 sec
- Average temperatures more than 820°C for sustained durations, local temps >1080°C

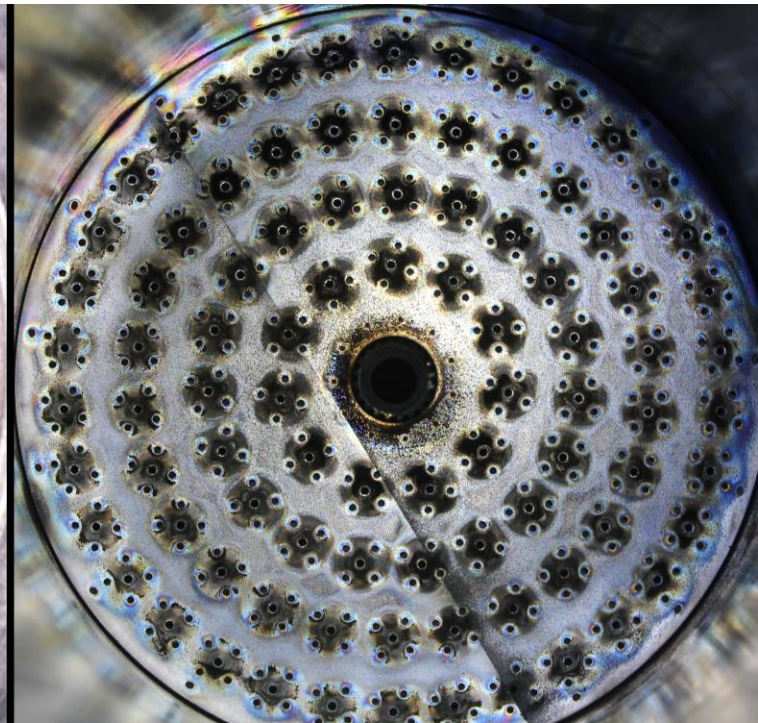


Injector Test Results

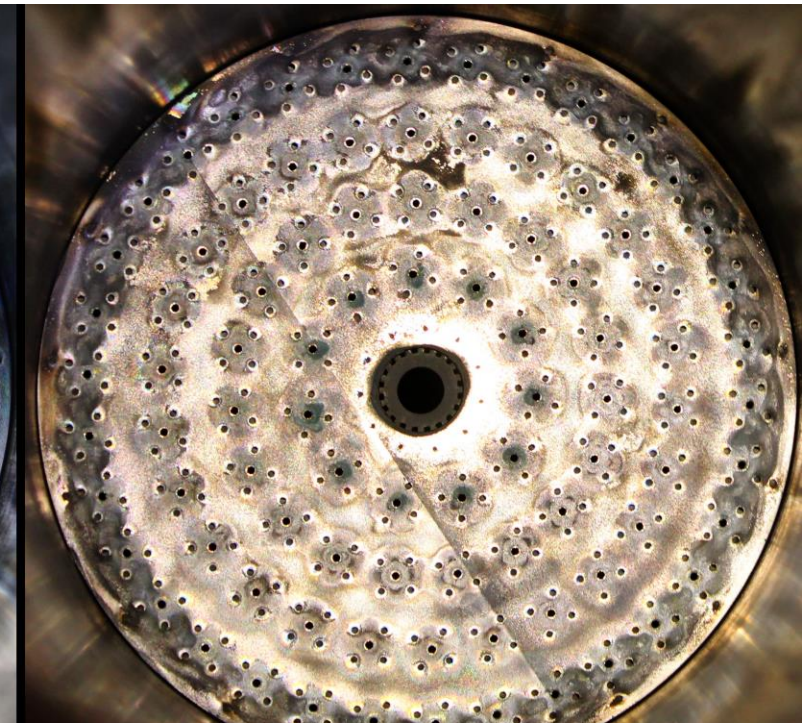
- GRX-810 Injector A achieved 84 starts and 2,228 sec (LOX/LCH4)
 - Demonstrated greater life than Inconel 625/718 equivalent
- GRX-810 Injector B achieved 30 starts and 591 sec (LOX/LCH4)
- GRX-810 Injector C achieved 9 starts and 303 sec (LOX/LH2)



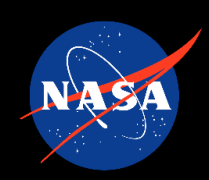
Inconel 625 Injector after 10 starts (Erosion)



GRX-810, LOX/LCH4 – 13 starts



GRX-810, LOX/LCH4 – 84 starts

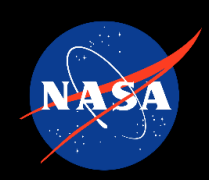


Hot-fire Testing



GRX-810 Nozzle and Injector Testing





L-PBF GRX-810 Component Examples



Turbine Blisk
Demo



Inducer with flow
passages



Pentad Injector



Shrouded
Blisk



Regen Nozzle



Turbine Blade with integral
instrumentation ports



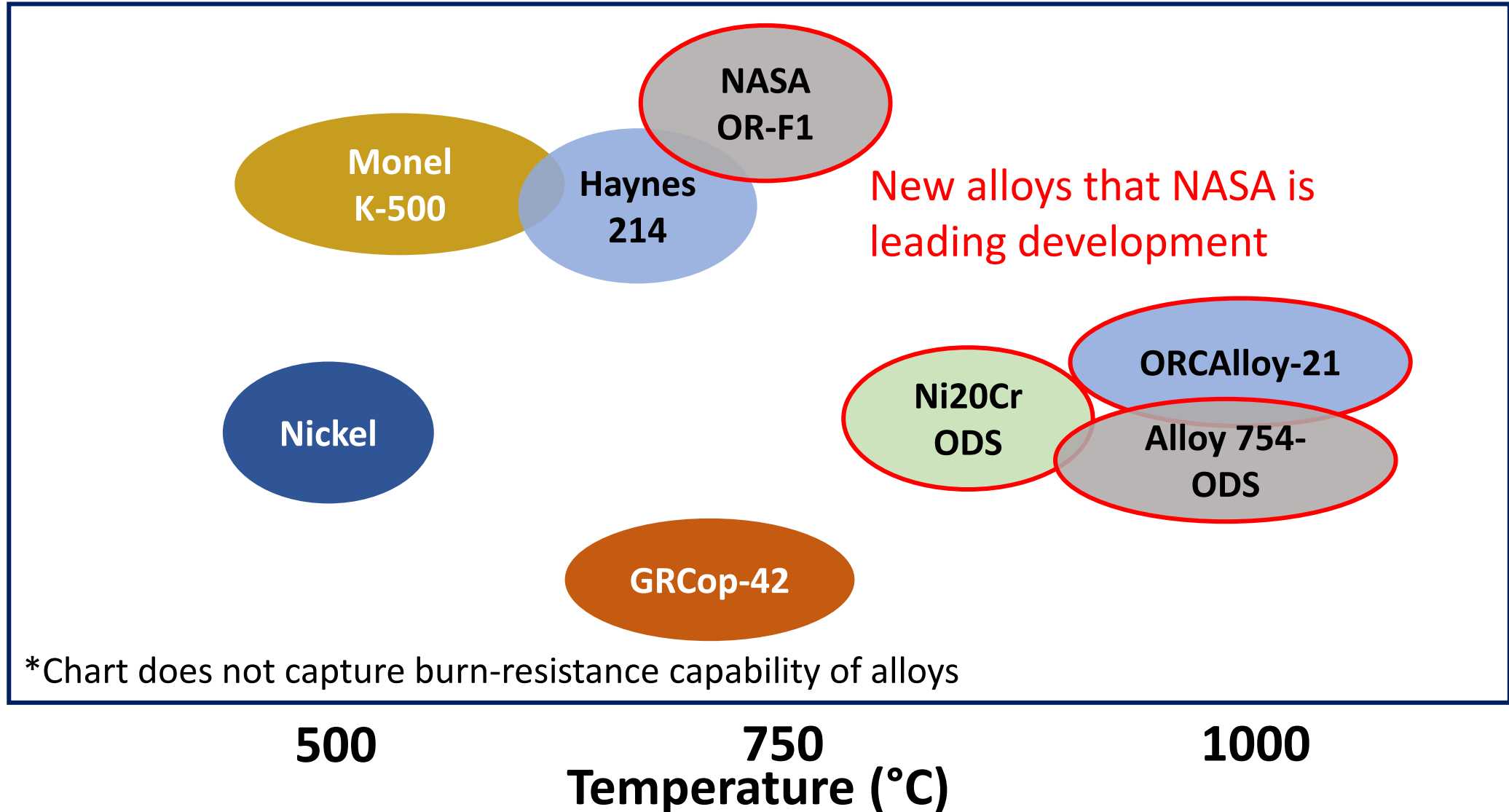
Toroidal
Inducer



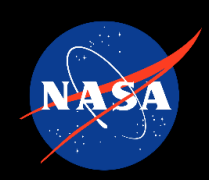
Landscape of Burn Resistant Alloys - Strength



“Usable” Strength



Proprietary Alloys Not Included



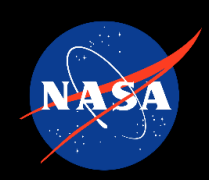
Alloy Development for High Pressure Oxygen Environments



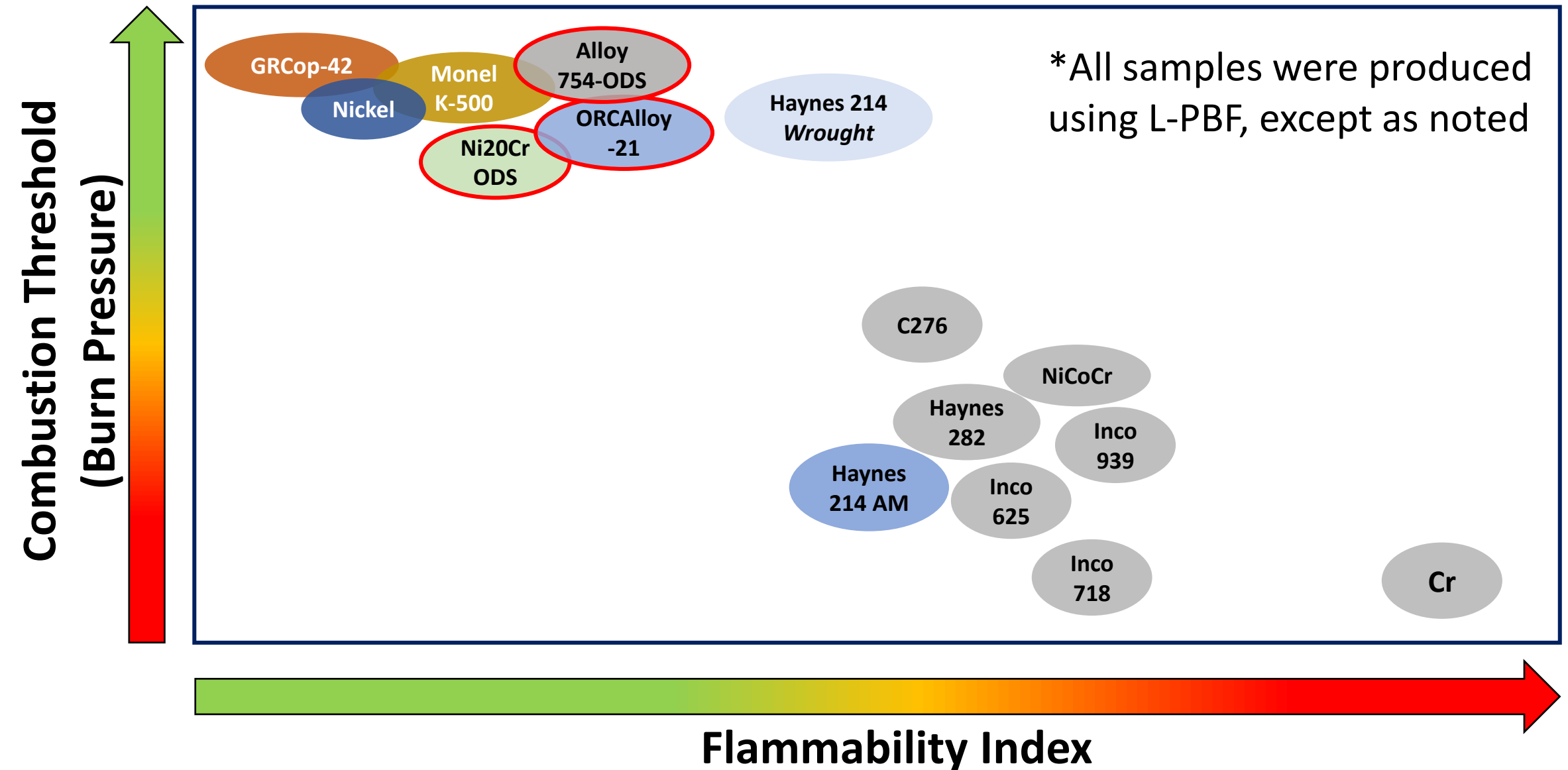
- Promoted combustion testing determines an alloys susceptibility to self-extinguish in pure O₂
- Alloys must be specifically designed to limit elements with high burn potential (e.g. Hf, Ti, Al, Nb), while maintaining other properties like strength, oxidation.



Example of an alloy that burns in pure O₂, White Sands Test Facility
(Courtesy: J. Tylka)



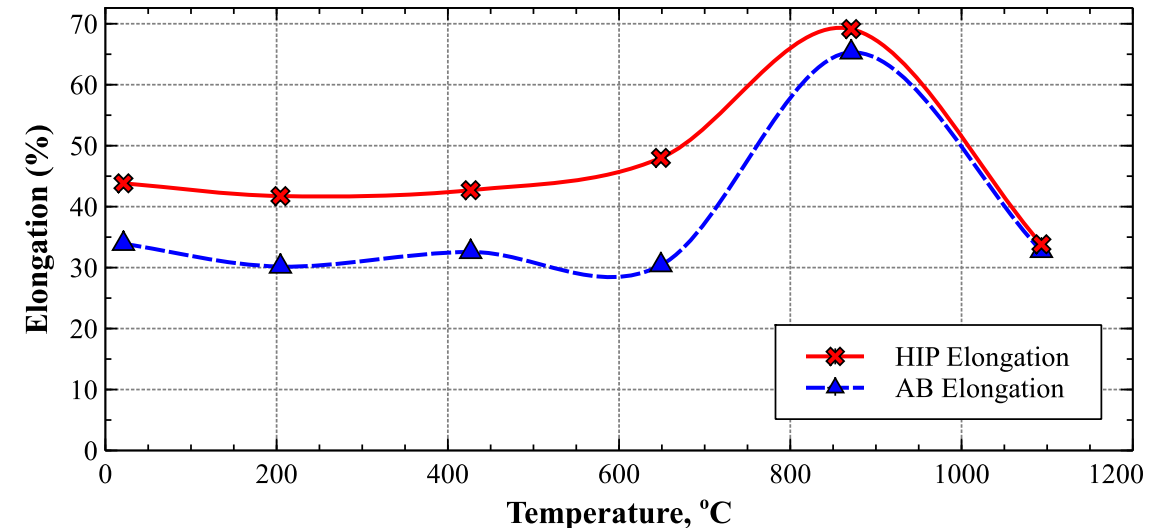
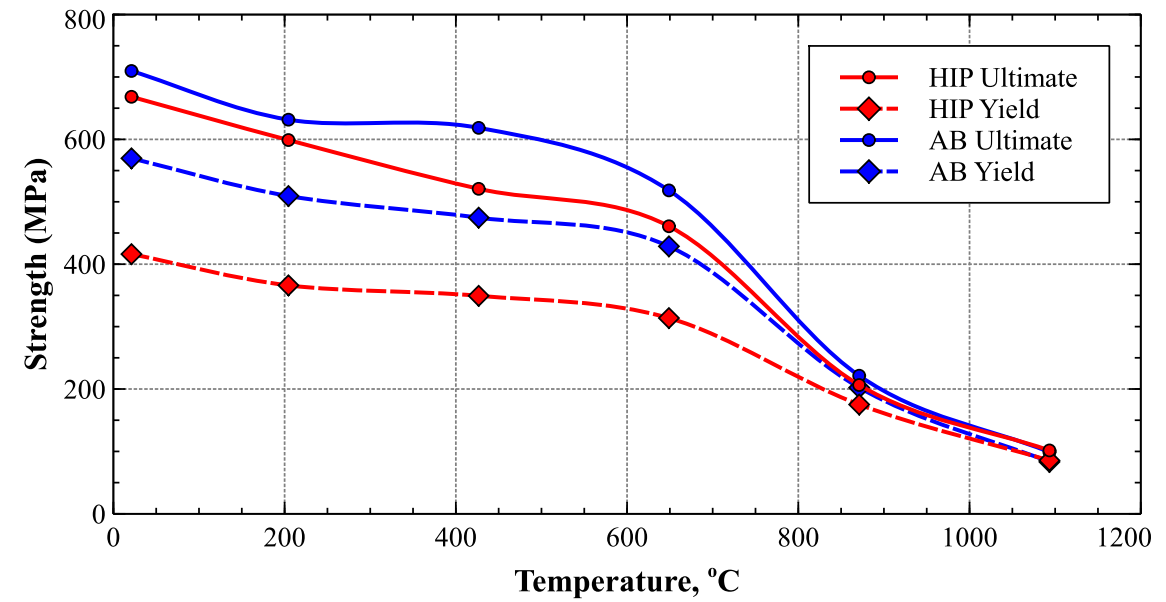
Combustion and Flammability Thresholds (Traditional and ODS)





ORCAIloy-21 Alloy Mechanical Testing Results

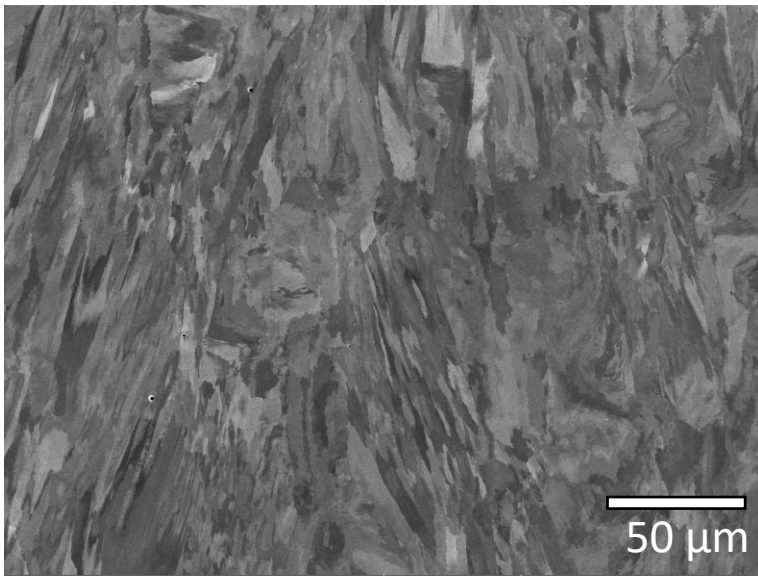
- **Oxygen Rich Compatible Alloy (ORCAIloy-21)** was developed using ICME and custom model validated with promoted combustion data.
- Ni-Co-Cr-Re medium entropy alloy with L-PBF and ODS with Y_2O_3 .
 - Similar composition as GRX-810, but not flammable.
- Designed for high strength at 2100°F (1149°C) operating temperature
- Designed for simplified heat treatment – as-built or Hot Isostatic Pressing (HIP).



ORCAIloy-21 Development and Results

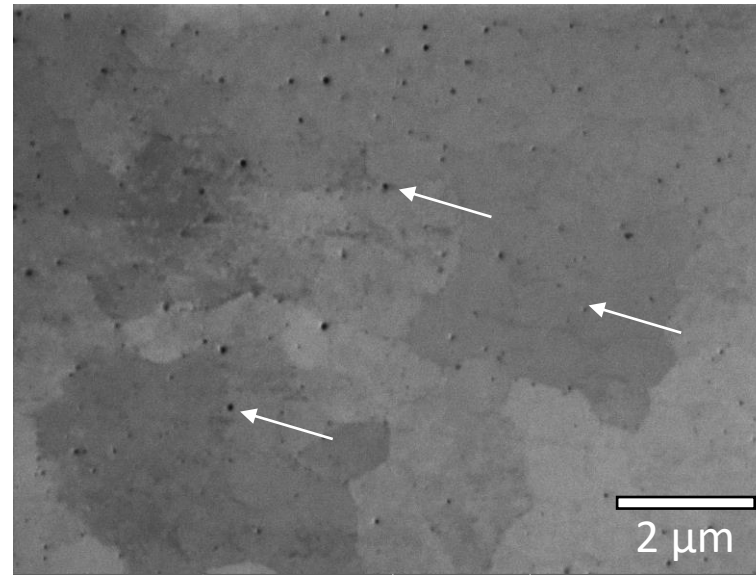


- Powder feedstock and coating process fully demonstrated and print parameters are mature.
- Microstructure is similar to GRX-810 with nano-scale Y_2O_3 dispersed.
- Components built successfully with parameters similar to Ni-based.



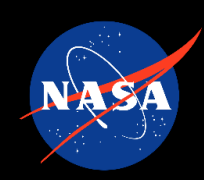
As-built microstructure

BD
↑



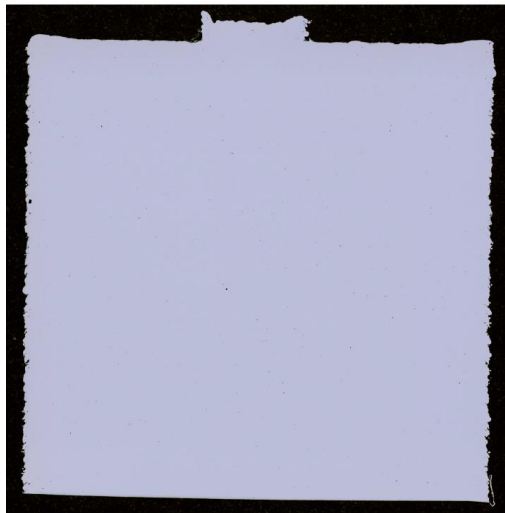
Nano-scale Y_2O_3



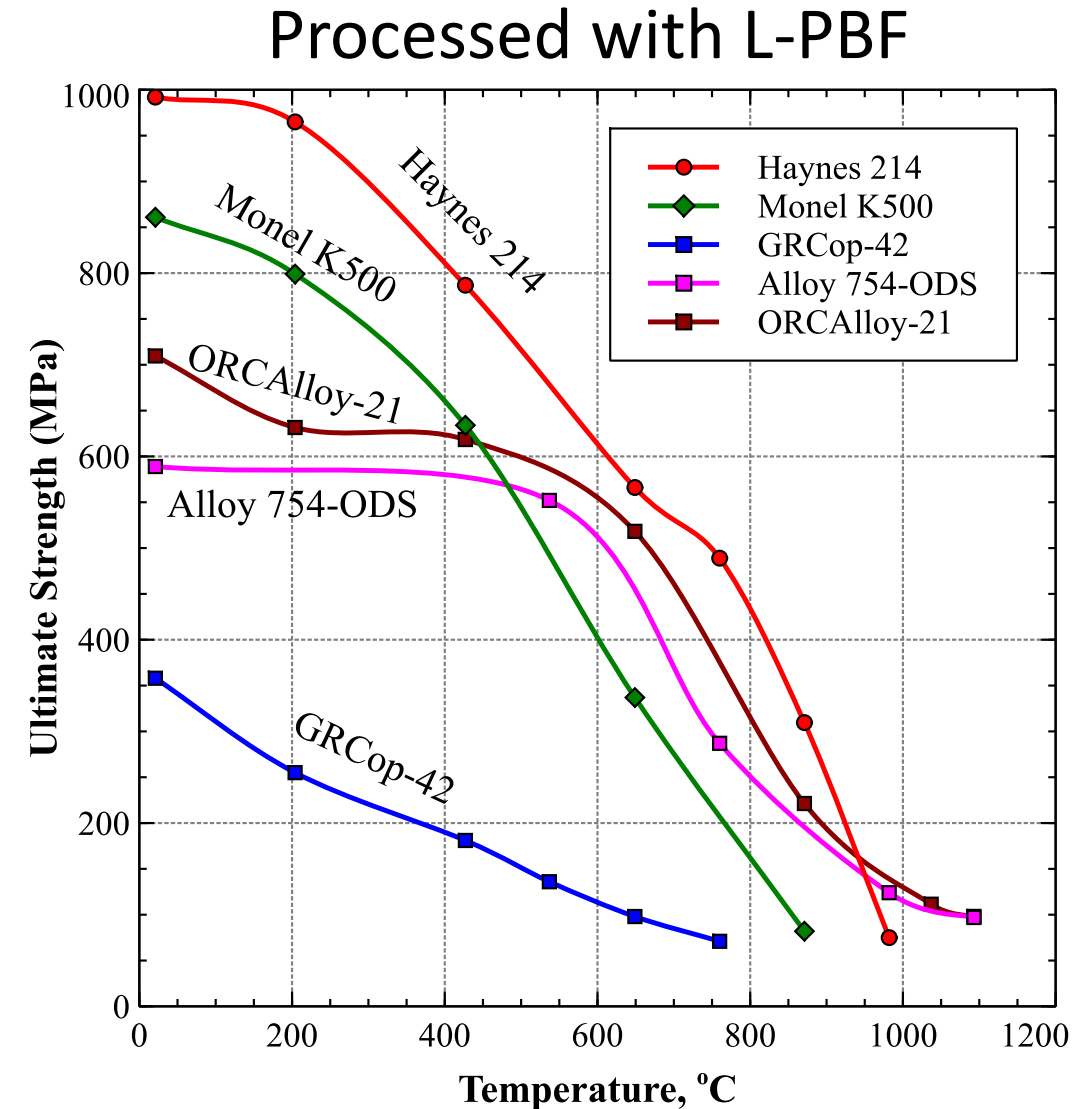
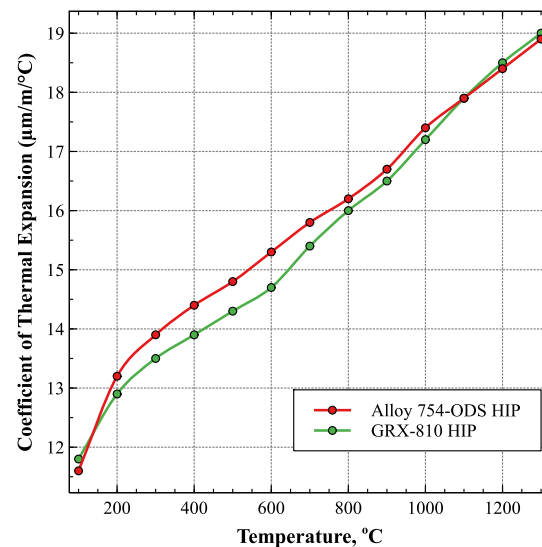


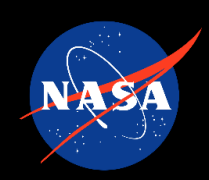
Alloy-754-ODS and Burn Resistant Alloy Comparison

- Alloy 754-ODS is Ni-Cr-Fe.
 - Based on mechanically alloyed MA-754.
- Acoustic resonant mixing Y_2O_3 ODS applied to gas-atomized powder
- Alloy comparison indicates strength retained at high temperature.



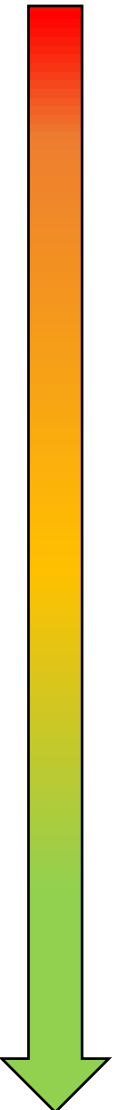
99.97% dense





What is required to properly mature aerospace alloys?



- 
- A vertical arrow on the left side of the list, pointing downwards. It has a color gradient from red at the top to green at the bottom, with yellow and orange in between.
1. Basic Concept of Material
 2. Chemical Composition Established through detailed Modeling
 3. Feedstock Produced and Characterized
 4. Parameters and sensitivities established with mature feedstock, geometric limitations
 5. Mechanical and thermophysical properties developed and characterized
 6. Parts demonstrators and supplemental processing
 7. Testing in a relevant environment with actual operational loads
 8. Production established and qualified metallurgy
 9. Flight production and maturity.



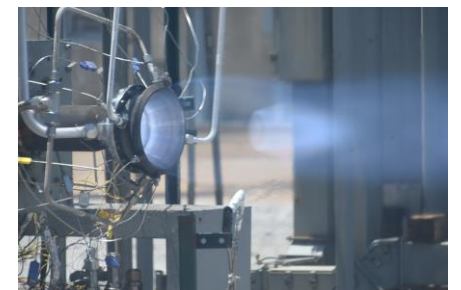
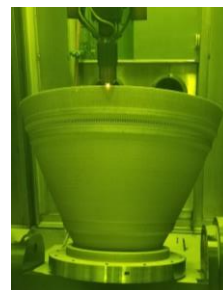
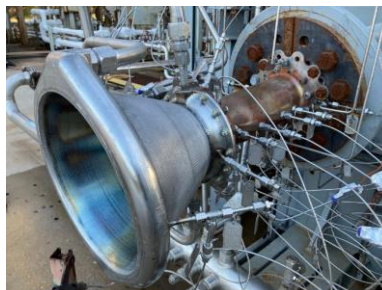
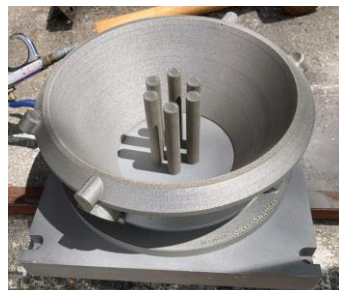
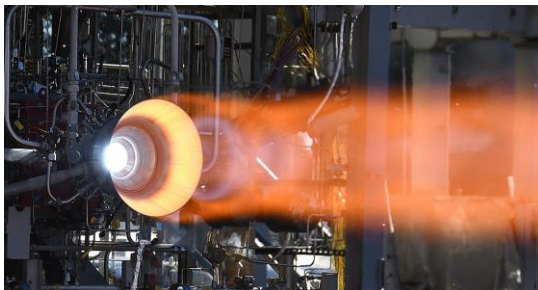
Summary and Future Work



- Several extreme environment NASA alloys have matured for aerospace component applications and actively being used.
 - GRCo-42, NASA HR-1, C-103, GRX-810, ORCAAlloy-21
- Demonstrated successful formulation, powder processing/coating, characterization, mechanical properties, component development and hot-fire testing of GRX-810 alloy.
- Successful scale up from M100 => M280/M290 => M400
- Used same ODS coating process and ICME to develop new high temperature burn resistant alloys including ORCAAlloy-21, Alloy 754-ODS, and OR-F1 (non-ODS)

Ongoing Development

- Openly available data and properties for commercial and government partners.
- Mature burn-resistant alloys through property databases and component testing.





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Appropriate Material Definition: Process → Performance

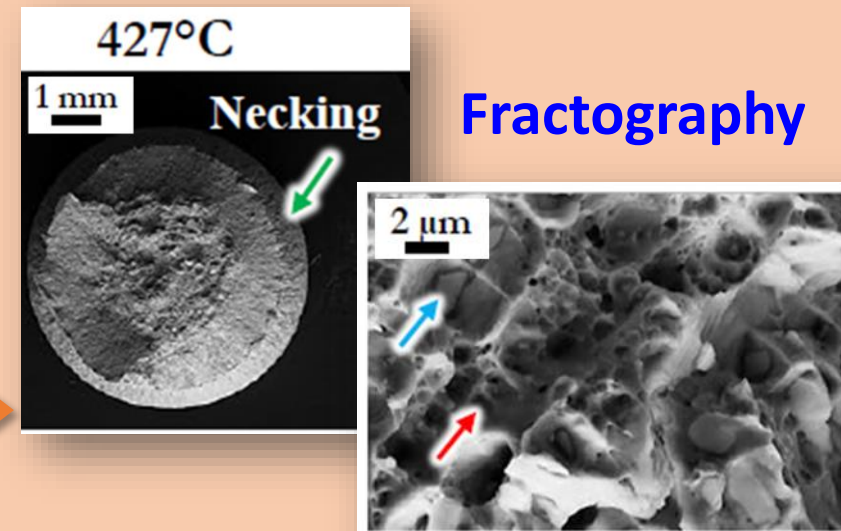
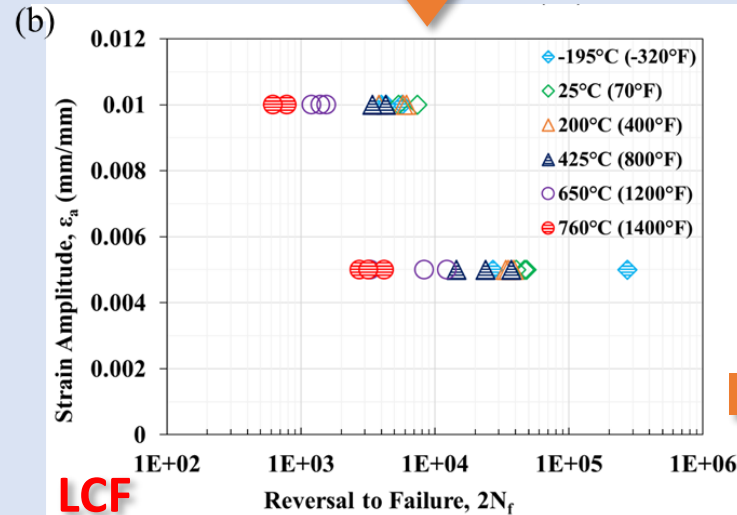
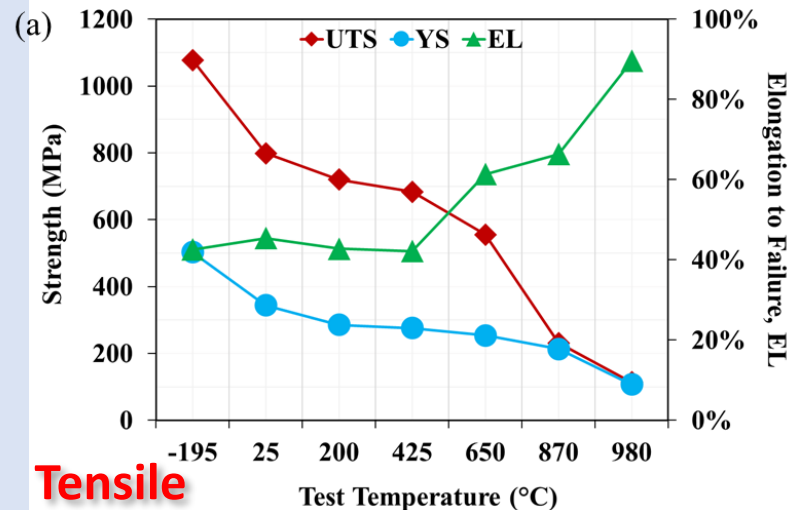
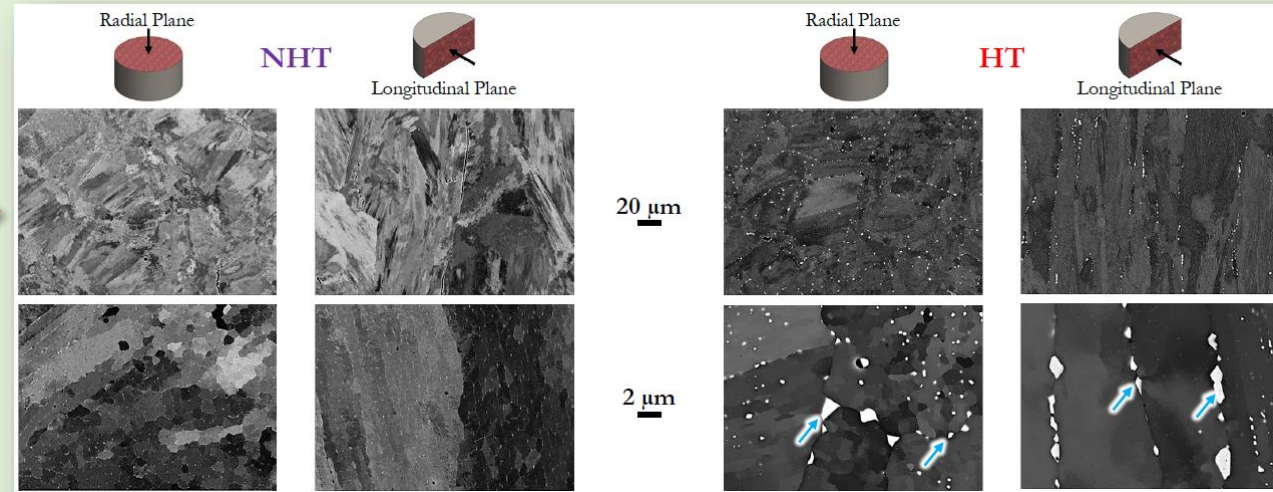
Data from Gradl, Mireles, Protz, Garcia. "Metal Additive Manufacturing for Propulsion Applications", AIAA Progress Series. (2022). Appendix A.

LP-DED Haynes 230

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

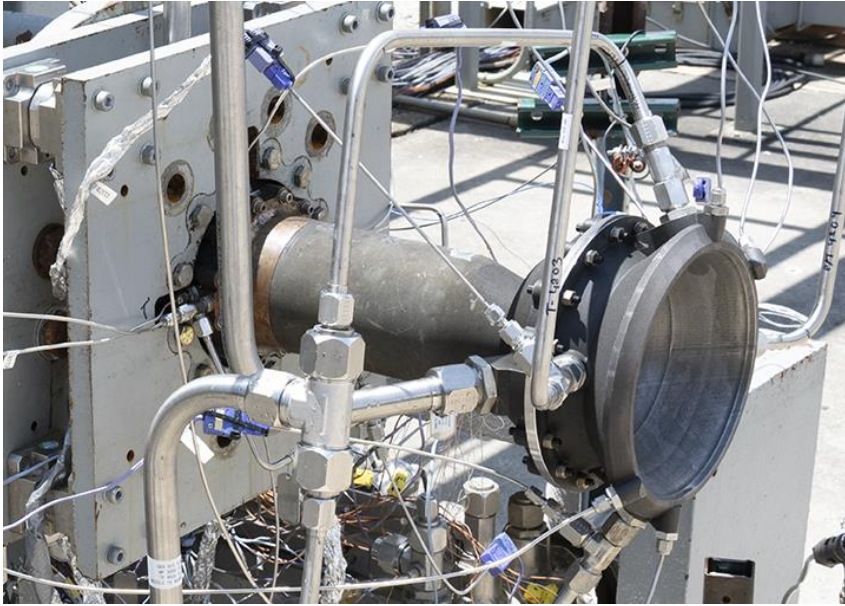
Procedure (Designation)	Temperature (°C)	Time (hrs)	Cooling
Stress Relief (SR)	1066	1.5	Furnace cool
HIP [2]	1163/103 MPa	3	Furnace cool
Solution Annealing (SOL)	1177	3	Argon quench

[2] HIP per ASTM F3301





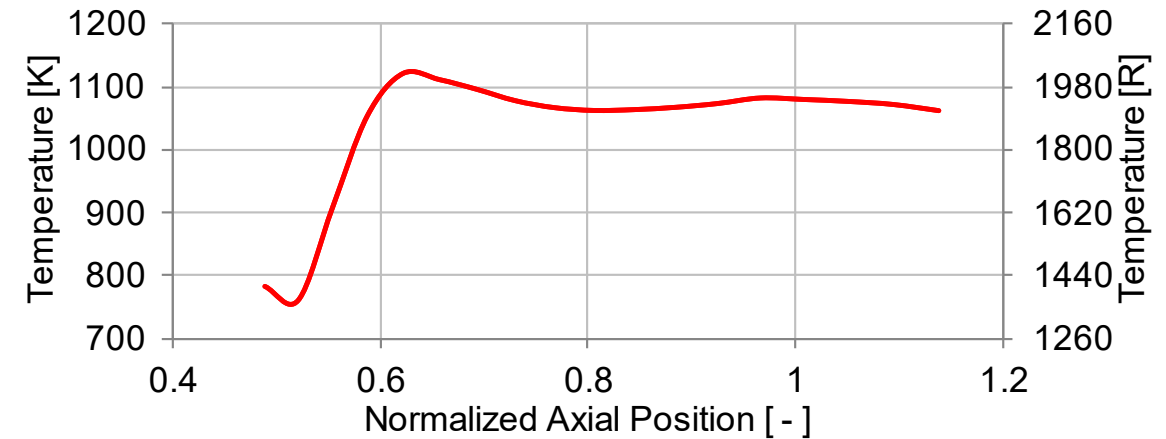
Hot-fire Testing of L-PBF GRX-810 Injector and Nozzle



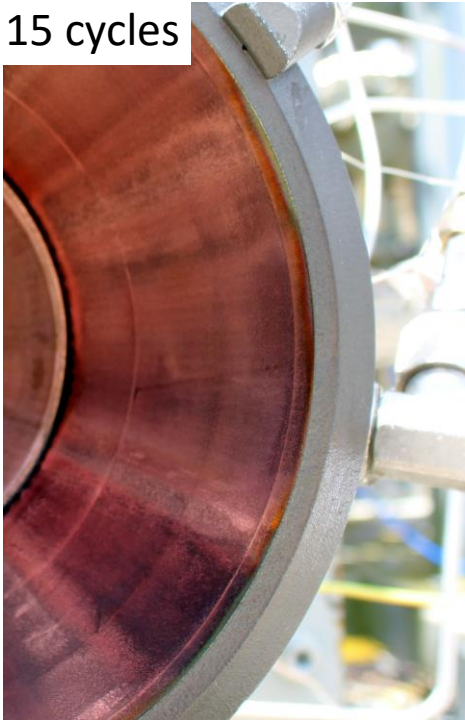
Component (-)		Starts (-)	Duration (s)	Pc		MR (-)
				(bar)	(psia)	
H2 Injector	SN01	9	302.8	49.5 - 57.2	718 - 829	5.33 - 7.02
CH4 Injector	SN02	29	586.5	37.4 - 52.4	542 - 760	3.03 - 3.65
CH4 Injector	SN03	84	2,227.9	43.2 - 52.1	626 - 756	2.68 - 3.19
Nozzle	SN04	91	2,309.4	37.4 - 52.1	542 - 756	2.68 - 3.11
Nozzle	SN05	8	149.1	49.0 - 50.5	711 - 732	3.00 - 3.19

GRX-810 Nozzle Test Results

- Regeneratively-cooled (LCH₄) nozzle accumulated 90 starts and 2,309 seconds.
- Local wall temperature $>1,000^{\circ}\text{C}$



15 cycles



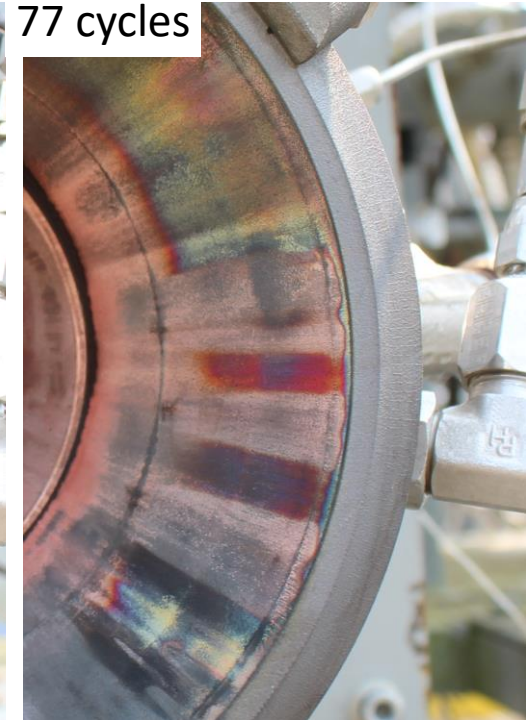
50 cycles



67 cycles



77 cycles





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