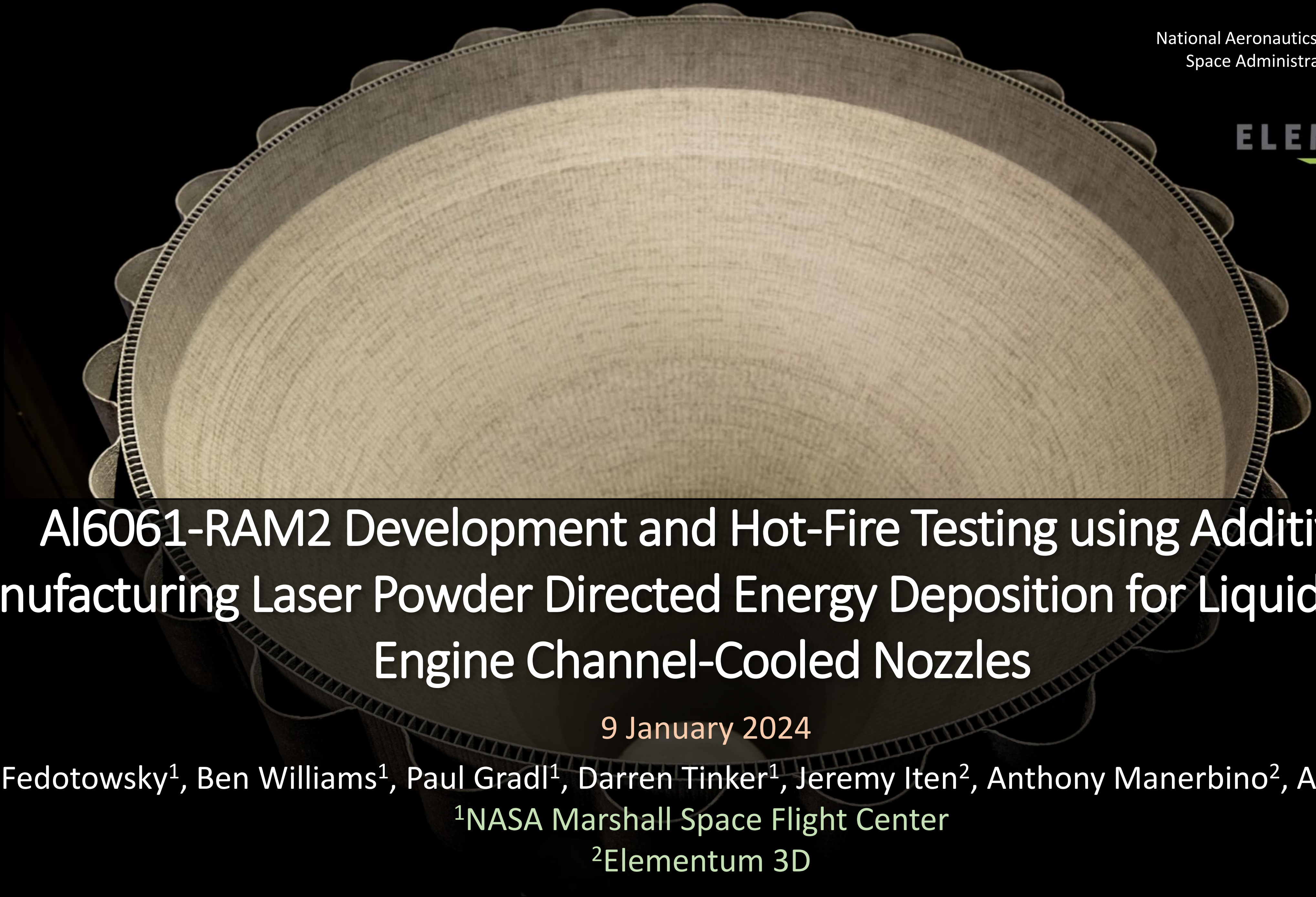
National Aeronautics and
Space Administration



Al6061-RAM2 Development and Hot-Fire Testing using Additive Manufacturing Laser Powder Directed Energy Deposition for Liquid Rocket Engine Channel-Cooled Nozzles

9 January 2024

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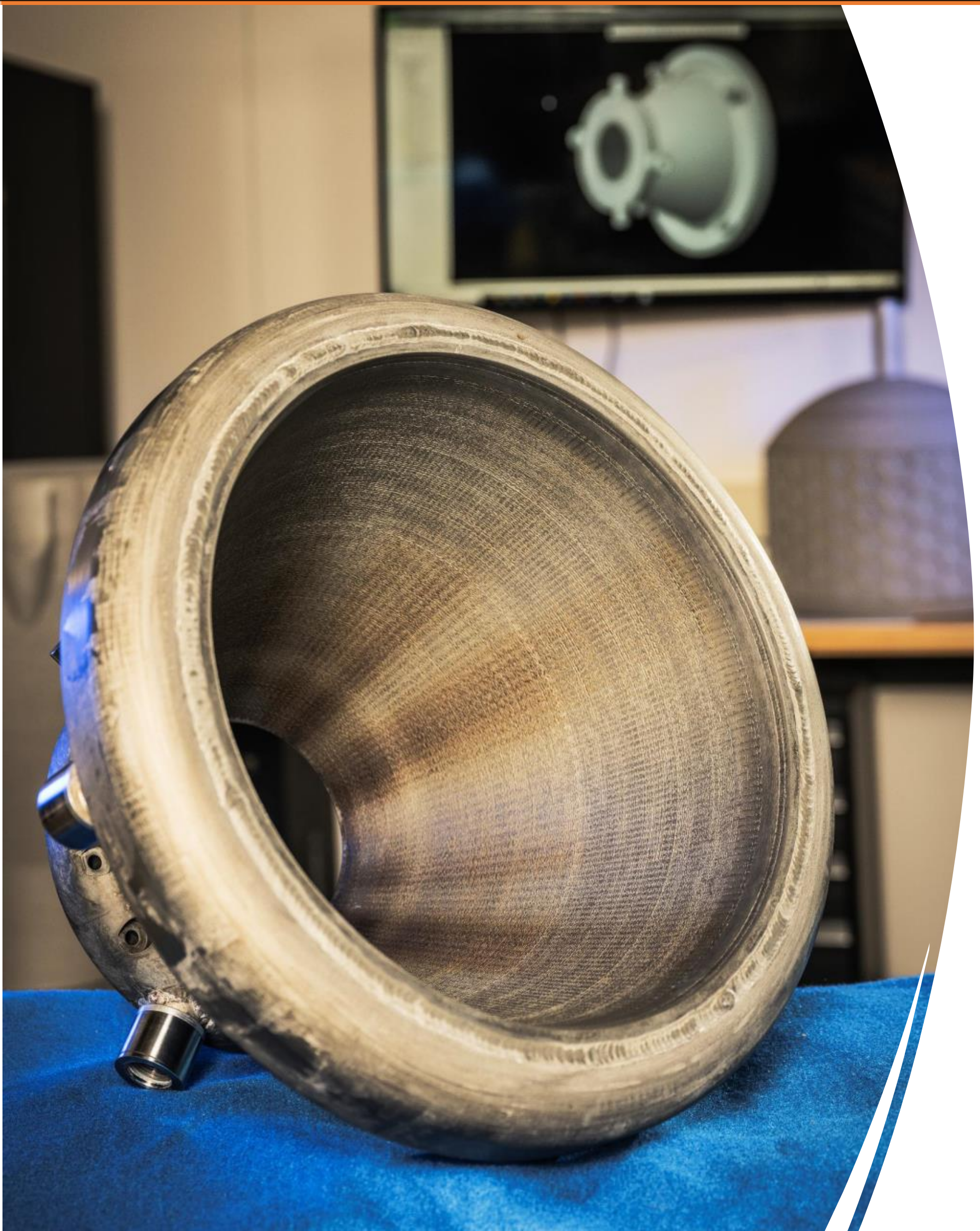
Introduction

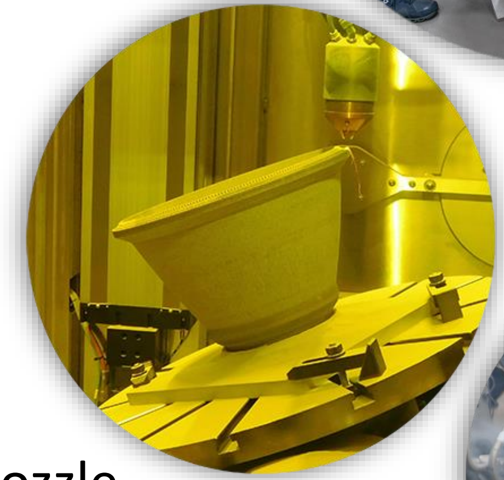


- Objectives
- LP-DED Development of Al6061-RAM2
- Microstructure and Material Properties
- Nozzle Manufacturing
- Supplemental Development
- Large-scale Demonstration Hardware
- Hot-Fire Testing
- Summary

Project Objectives

- NASA partnership called **Reactive Additive Manufacturing for Fourth Industrial Revolution Exploration systems (RAMFIRE)** matured large-scale lightweight additively manufactured (AM) aluminum alloys.
  - Specifically, Laser Powder Directed Energy Deposition (LP-DED) Al6061-RAM2
  - Partnered with Elementum 3D under NASA STMD Announcement for Collaborative Opportunities (ACO)

- **Project Objectives:**
  - LP-DED Al6061-RAM2 feedstock specification and validation
  - LP-DED process development and validation
  - Microstructural and mechanical property characterization
  - Hot-fire testing of a 7k-lbf thrust class regeneratively cooled nozzle
  - Printing of a large-scale regeneratively cooled demonstrator nozzle



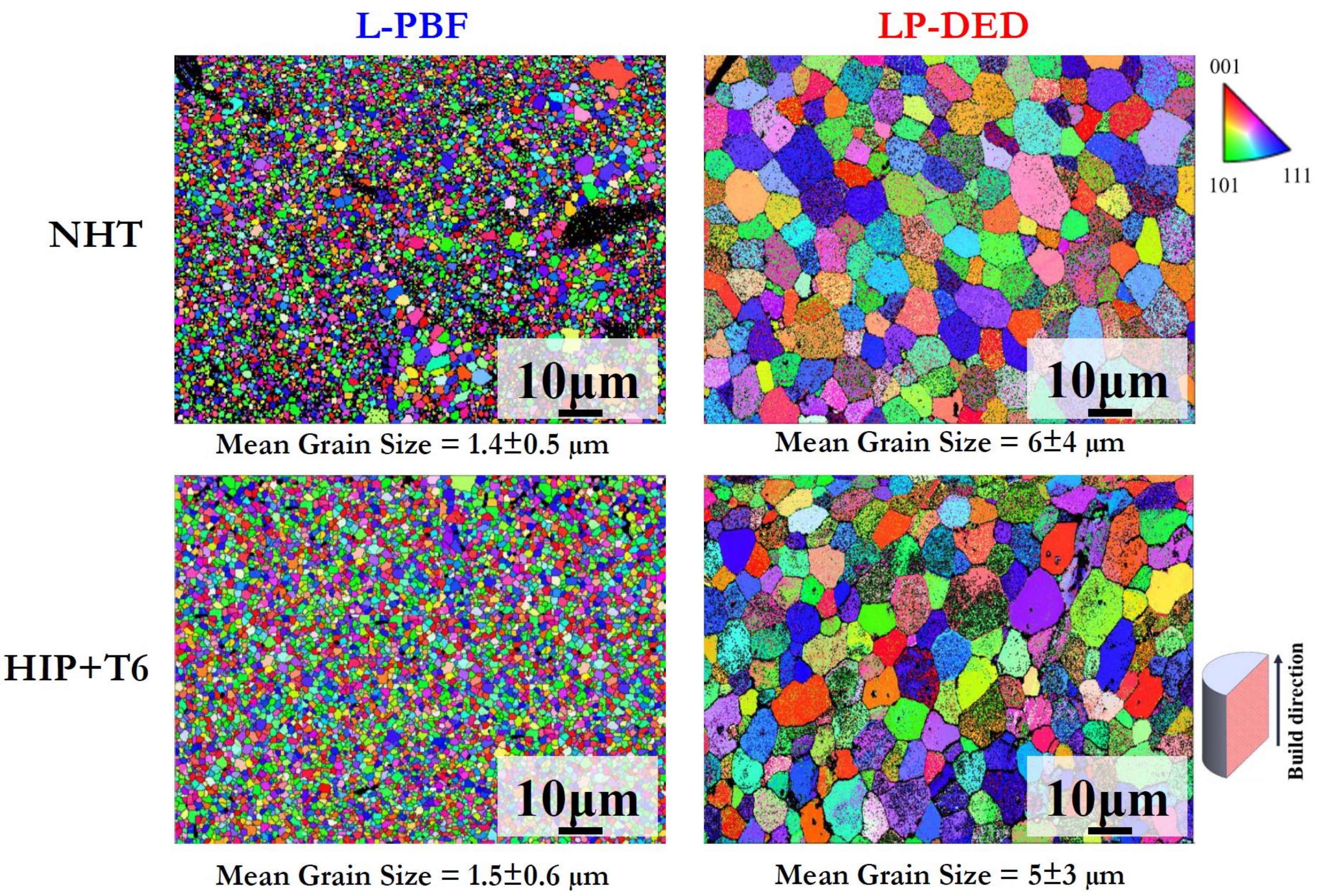
Microstructural Characterization

- The addition of the Boron Carbide (B4C) and Titanium (Ti) inoculant particles provides heterogeneous nucleation sites for solidification

- Equiaxed, fine grains were observed in all heat-treated conditions

- Mean grain sizes were relatively larger for LP-DED compared to L-PBF

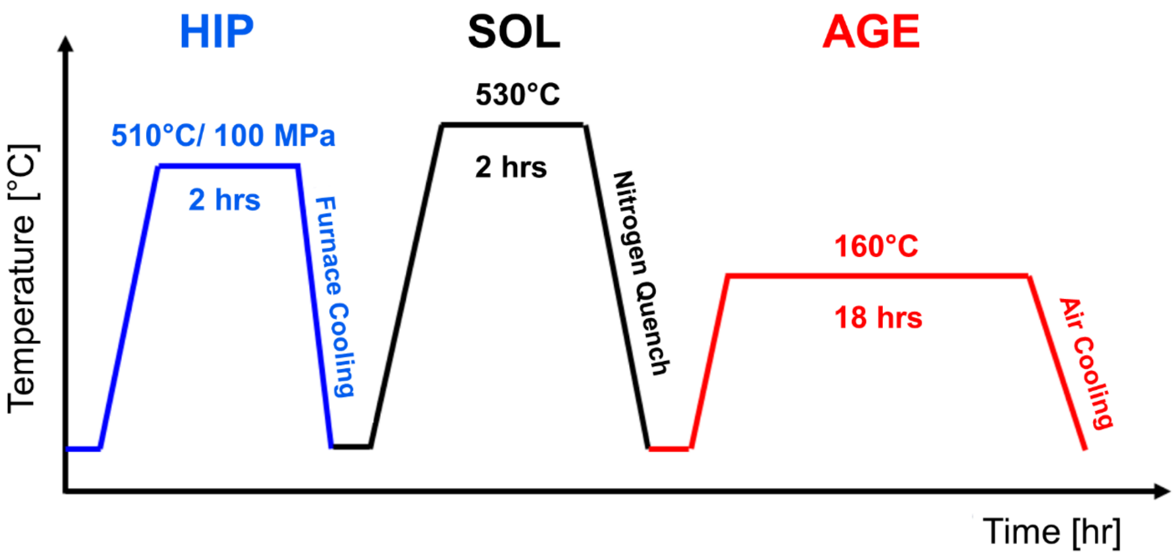
- Grains were stable after heat treatment because the inoculant particles acted as grain boundary pinners

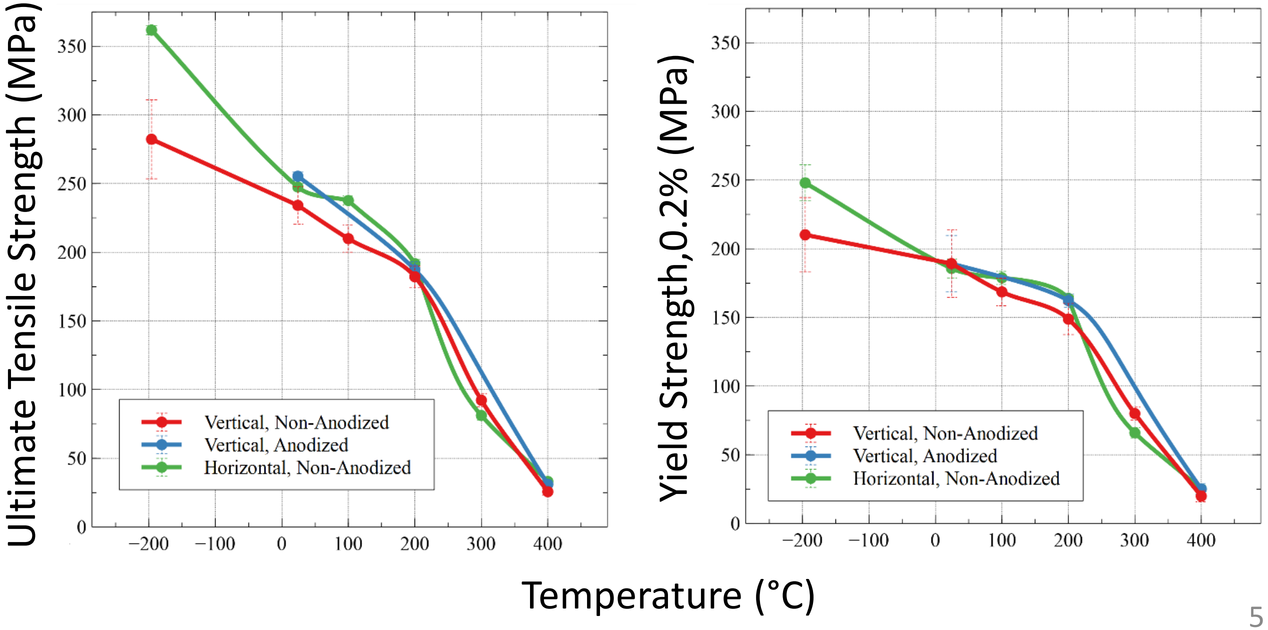


Heat Treatment and Mechanical Properties

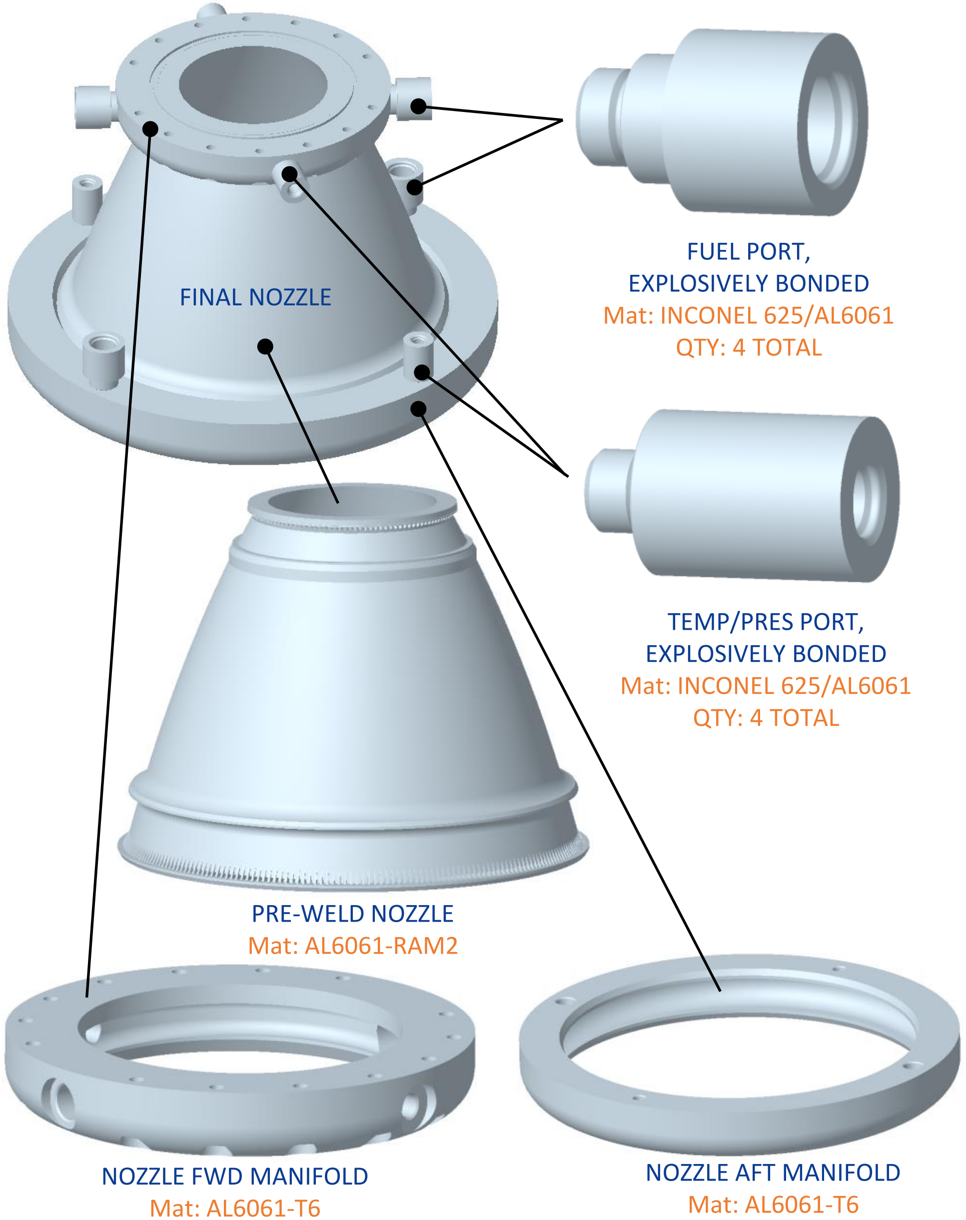
- Heat Treatment is critical to obtain desired material properties
- Hot isostatic pressing (HIP) is a requirement of NASA-STD-6030 to obtain full density and desired fatigue properties
- Solution and aging processes per industry standard T6 cycle

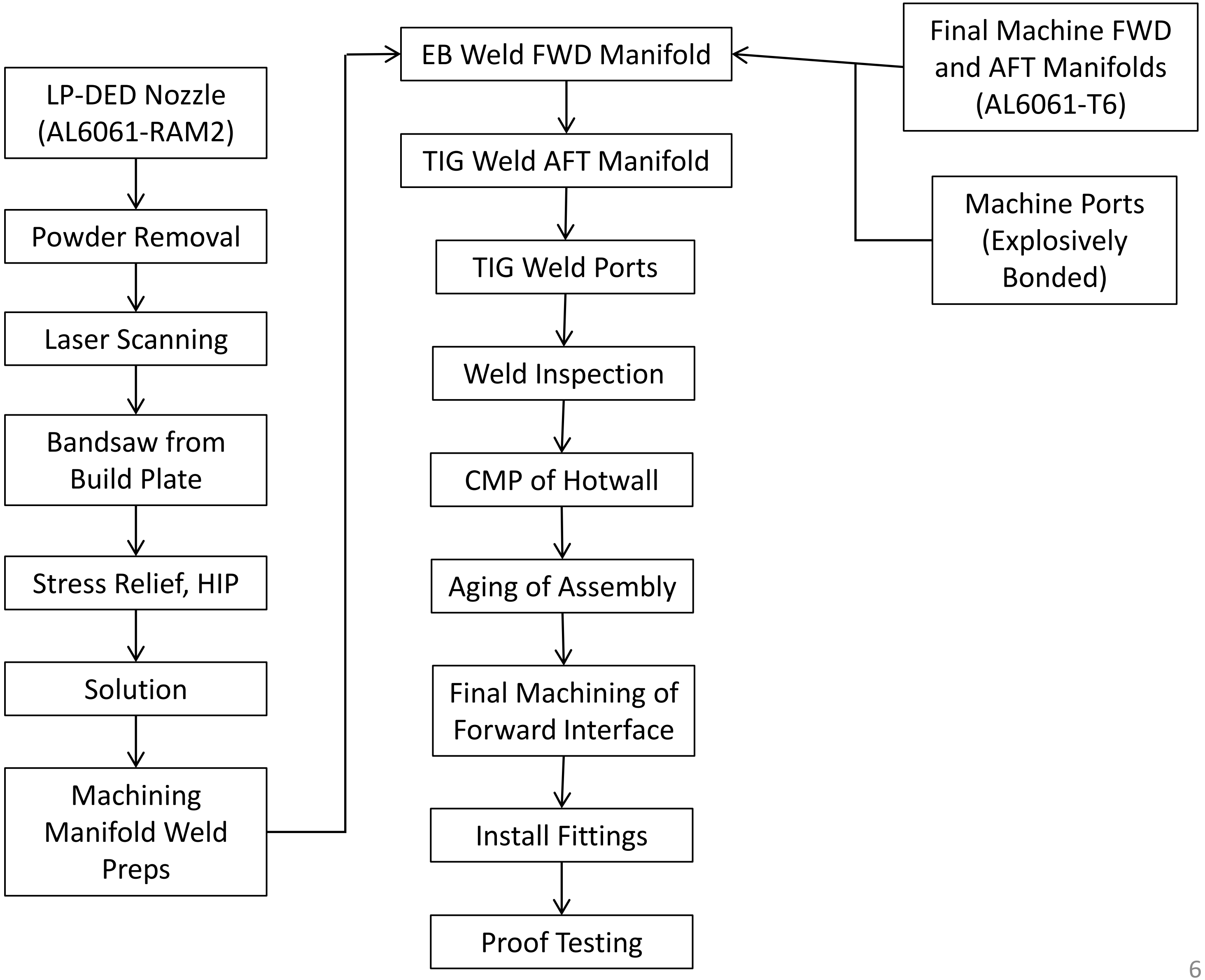
- Anodization condition has little effect on mechanical properties
- The vertical and horizontal (in relation to the build direction) conditions showed minor differences on mechanical properties





Nozzle Manufacturing





LP-DED Nozzle (AL6061-RAM2)

Powder Removal

Laser Scanning

Bandsaw from Build Plate

Stress Relief, HIP

Solution

Machining Manifold Weld Preps

EB Weld FWD Manifold

TIG Weld AFT Manifold

TIG Weld Ports

Weld Inspection

CMP of Hotwall

Aging of Assembly

Final Machining of Forward Interface

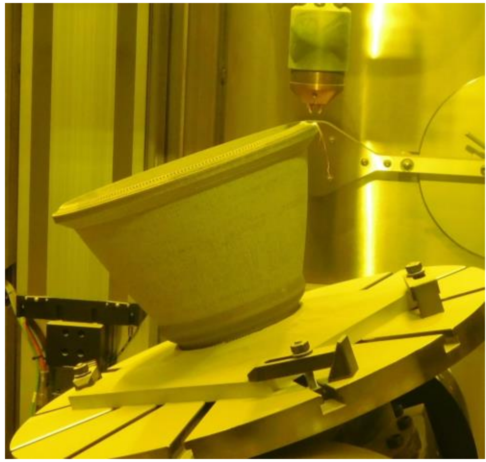
Install Fittings

Proof Testing

Final Machine FWD and AFT Manifolds (AL6061-T6)

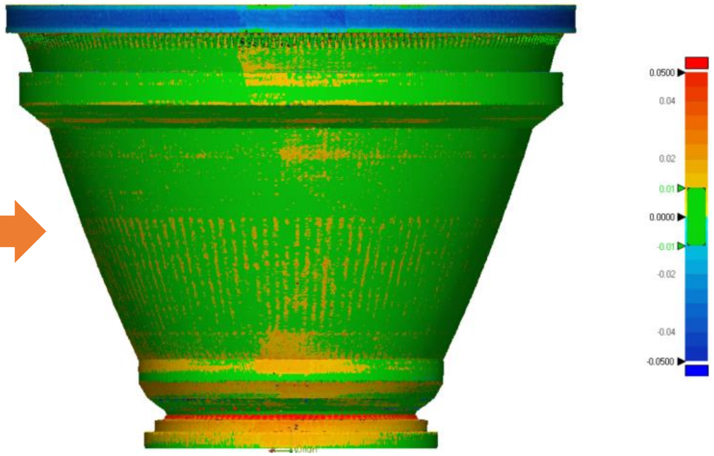
Machine Ports (Explosively Bonded)

Nozzle Manufacturing



LP-DED Nozzle
(AL6061-RAM2)

Post-DED



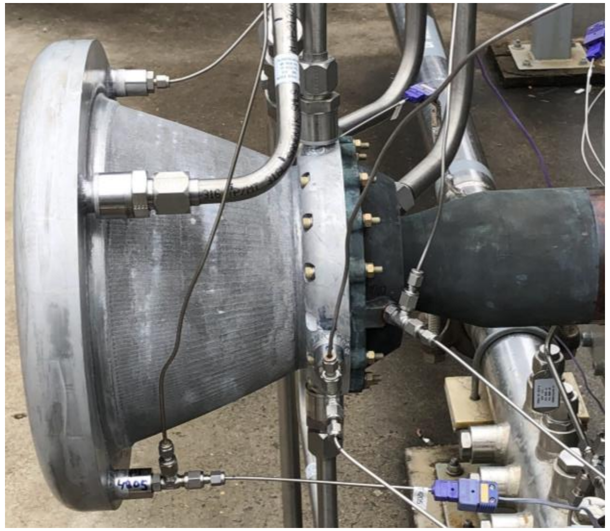
Laser Scanning

TIG Welding

Post-Aging

Final Machining of Forward Interface

Flow/Proof Testing



Hardware Installation

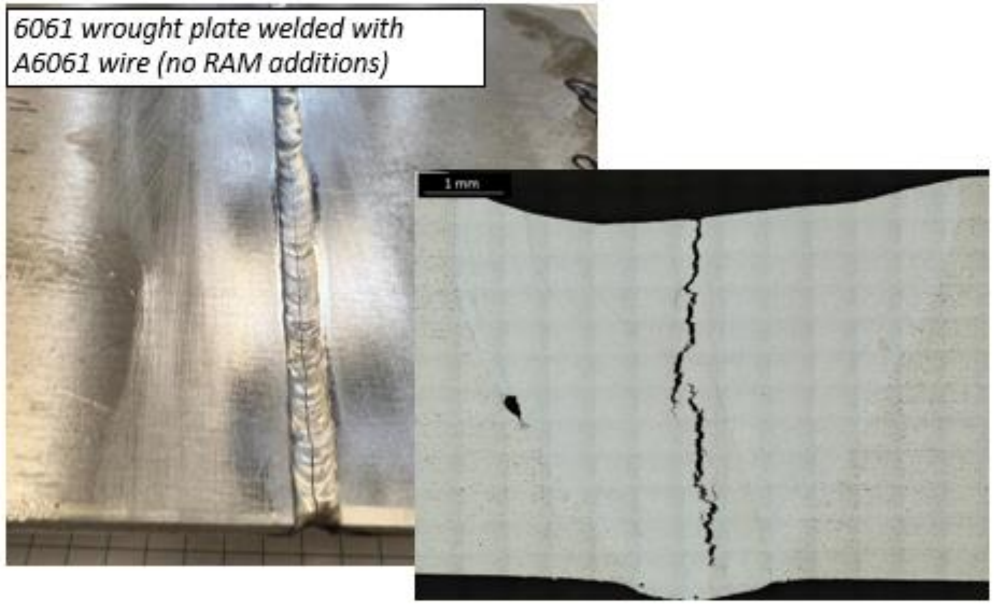
Supplemental Development - Welding

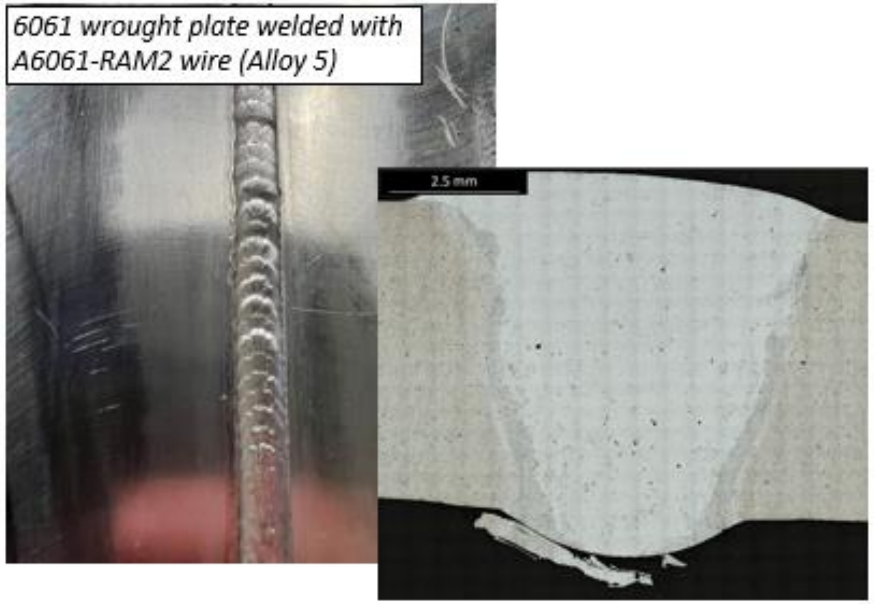
EB Welding

- 6000 series aluminum alloys typically requires Al4043 filler material to avoid cracks
- Development determined that filler material was no longer required due to the RAM nano-dispersoids
- The RAM particles in the nozzle were mixed into the fusion zone of the EB weld

TIG Welding

- The RAM technology was leveraged to make filler wire with Fortius Metals
- Al6061-RAM2 wire showed drastic improvement in weldability; noticeable grain refinement, no cracking, and higher mechanical properties compared to traditional 4043 filler metal

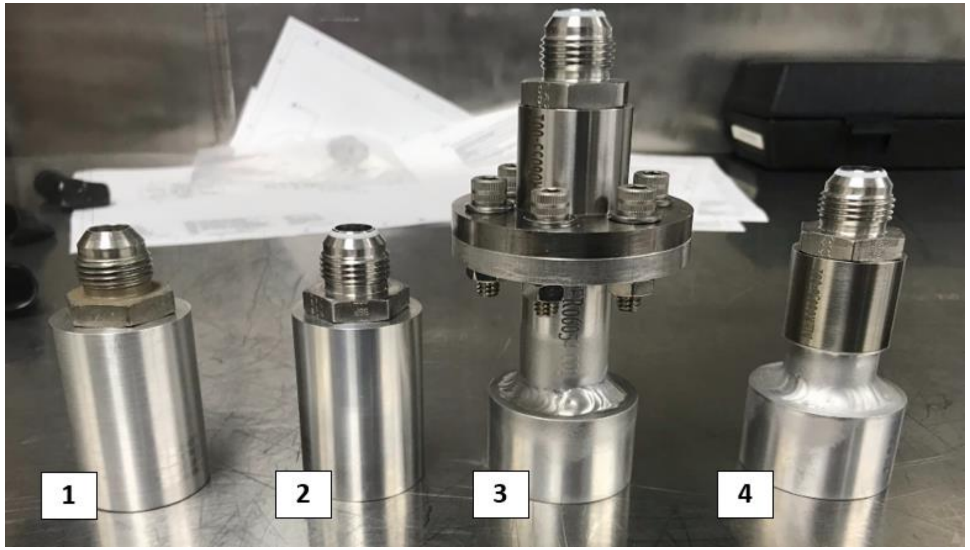

6061 wrought plate welded with A6061 wire (no RAM additions)


6061 wrought plate welded with A6061-RAM2 wire (Alloy 5)

Both nozzle's welds were nominal with no gross defects and had no issues throughout hot-fire testing

Supplemental Development - Ports

- A risk was identified for installing SST fluid fittings to the aluminum manifolds due to galling and continued cycling.
- 4 fixtures were submerged and pressure cycled with LN2, and burst tested with ambient water.
- The first two fixtures burst at the interface; the threads of the port galled, and the sealing O-Ring deformed to release the pressure.
- The last two fixtures burst at the aluminum welds.
- Explosively bonded ports were selected for the nozzle.



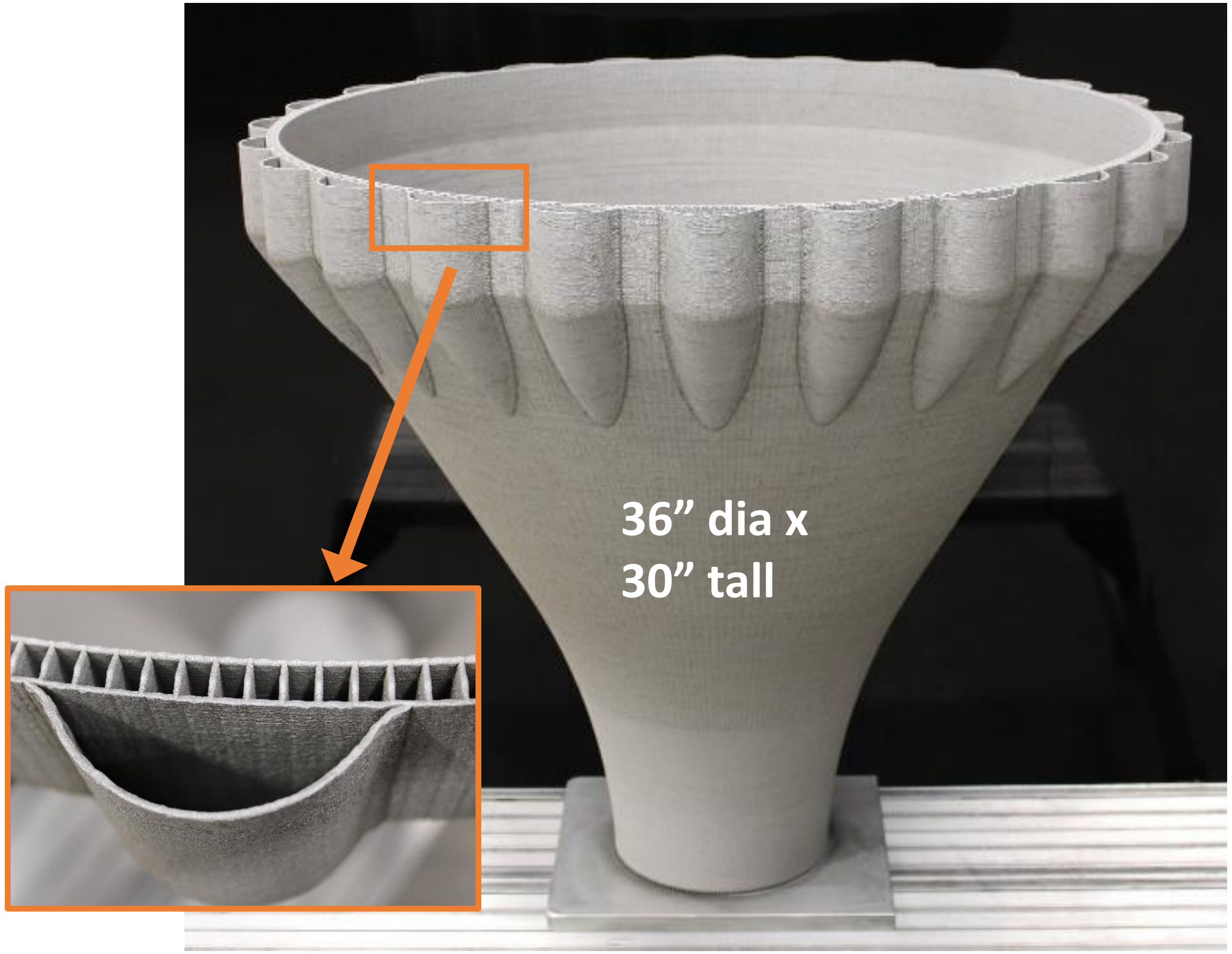

O-Ring Split


Threads rolled over each other – allowing pressure to reach O-ring and burst

The explosively bonded ports remained intact with no leaks or weld cracks throughout hot-fire testing

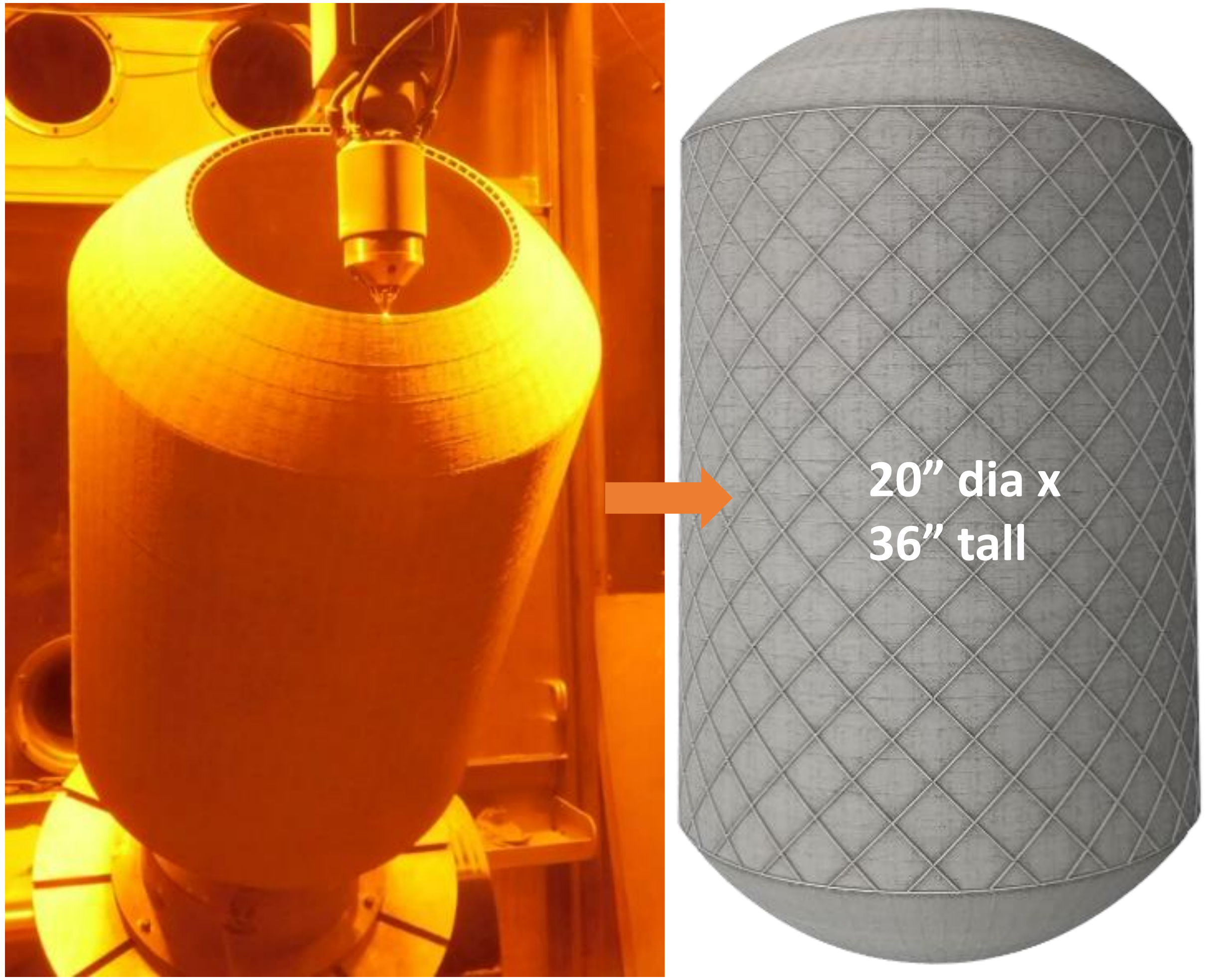
Large Scale Demonstration Hardware

LP-DED printed Al6061-RAM2 with 1.5 mm single-bead wall thickness



36” dia x
30” tall

18 day build time



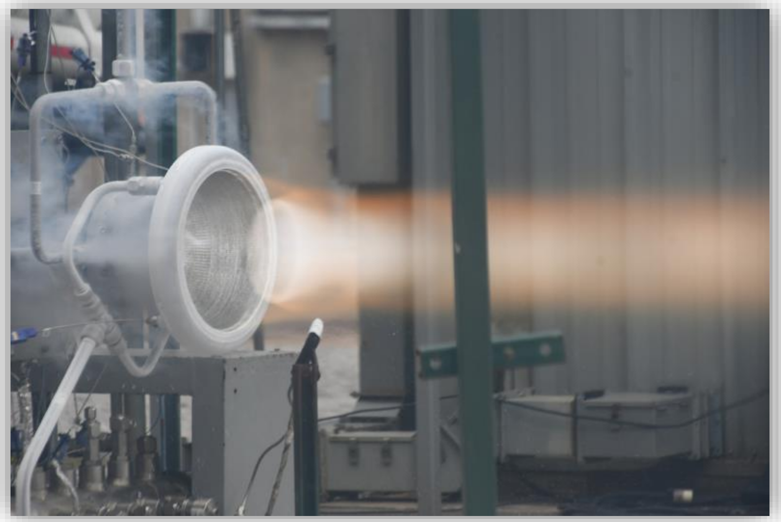
20” dia x
36” tall

15 day build time

Hot-Fire Testing

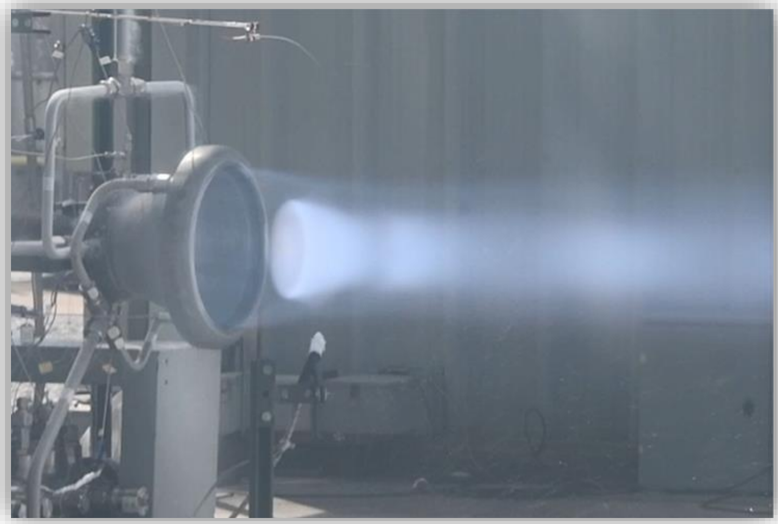
Hydrogen Testing

- 9 hot-fire tests, 302.8 seconds total mainstage
- ~5.7 MPa max Pc, ~7.02 max MR



Methane Testing

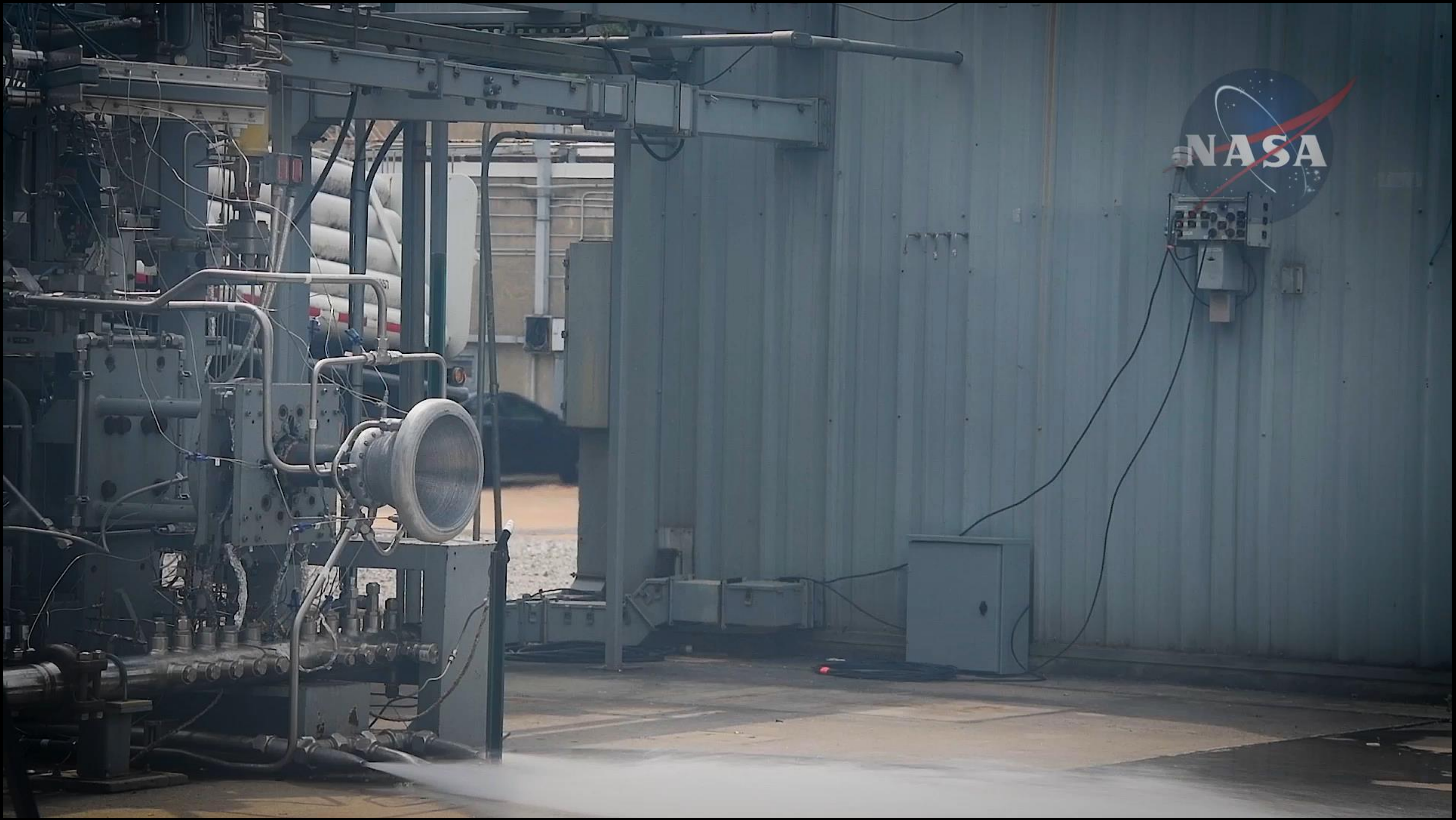
- 11 hot-fire tests, 254.2 seconds total mainstage
- ~5.2 MPa max Pc, ~3.65 max MR



Overall

- The project completed 22 cycles at ~557 seconds of cumulative duration
- Progressively harsher heat fluxes were imparted on the nozzles during testing
- The Al6061-RAM2 nozzles performed well and survived numerous cycles of relevant heat flux environments

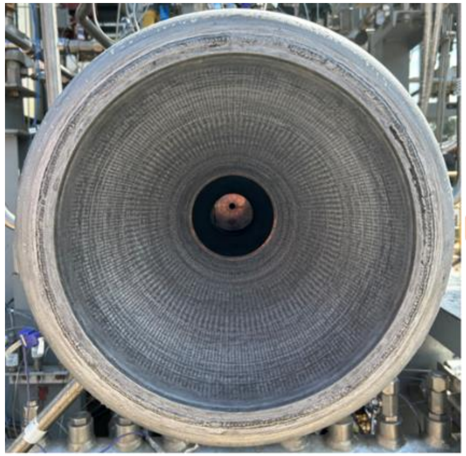
Hot-Fire Testing



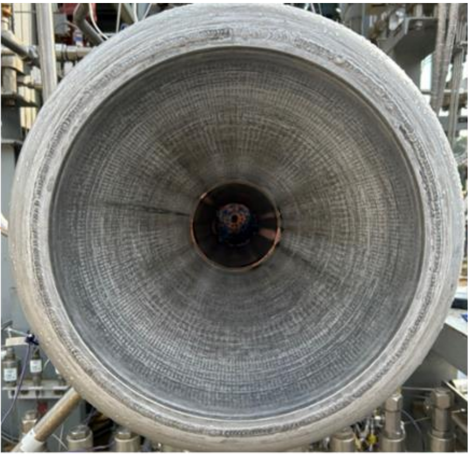
Hot-Fire Testing

Non-Anodized Nozzle

- 16 starts, 457 seconds mainstage
- Discoloration identified along a single coolant channel during hydrogen
- CT scan verified trapped powder
- Cooling was adequate and no erosion was observed during methane testing
- ~10x increased thermal conductivity (vs. superalloys), allowed adequate cooling through adjacent ribs



Pre-Test



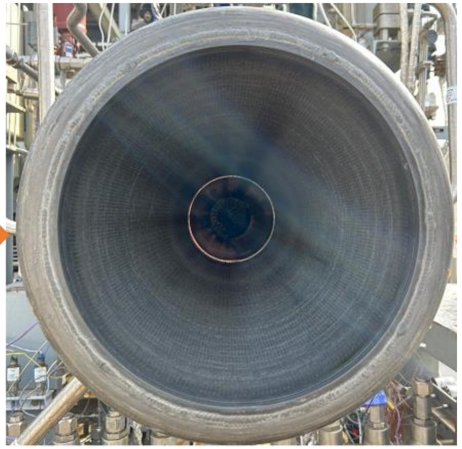
Post-Hydrogen

Post-Methane

Anodized Nozzle

- 6 starts, 120 seconds mainstage
- Discoloration was observed on the hot-wall which propagated from the injector to the chamber and nozzle
- No roughening or erosion of the hot-wall identified

Pre-Test



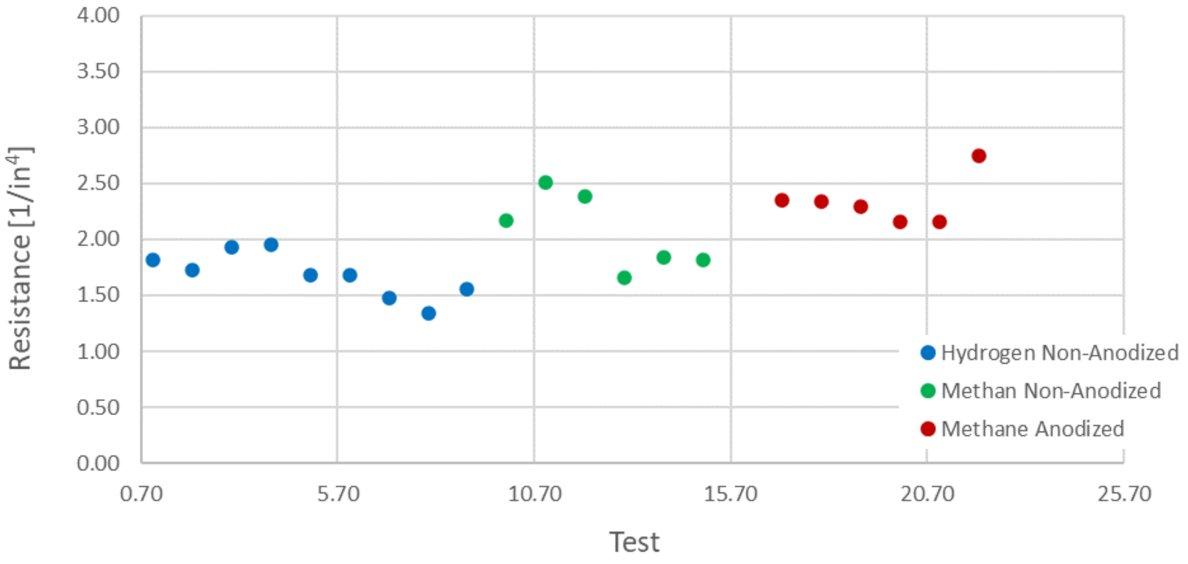
Post-Methane

Similar hot-wall discoloration was observed on the anodized & non-anodized nozzle with no significant differences

Hot-Fire Testing

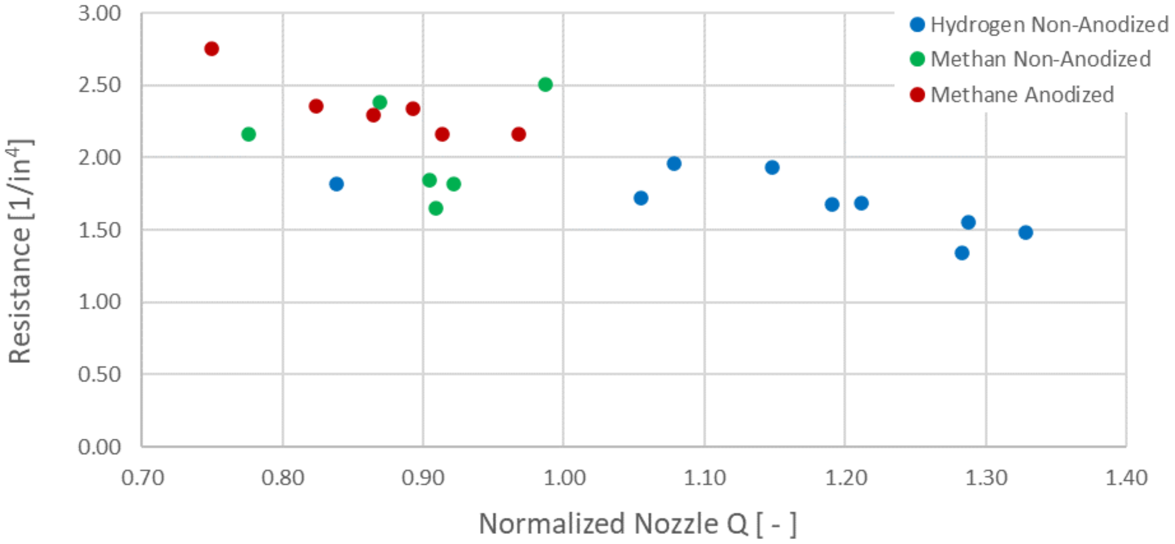
Nozzle Channel Resistance Per Test

- Regardless of propellant or nozzle, the resistance through the channels remained relatively constant throughout the test program



Nozzle Channel Resistance vs. Normalized Heat Load

- Channel resistance remains relatively unchanged as heat load increases. As operating conditions changed, the nozzle performance for channel resistance was highly repeatable.



Proven repeatability between tests and reproducibility between nozzles.

Summary

- Developed LP-DED Al6061-RAM2 powder feedstock and process to build thin-wall components with integral channels.
- Advanced supplemental manufacturing technology including use of the RAM alloy in TIG and EB welding and bimetallic explosively bonded ports.
- Achieved 22 total starts and 579 seconds accumulated time between LOX/LH2 & LOX/LCH4.
- Weight reduction of 23% compared to superalloy regen nozzle.
- Completed scale-up of various components including rocket nozzles and cryogenic tanks.
- Data is available to industry partners

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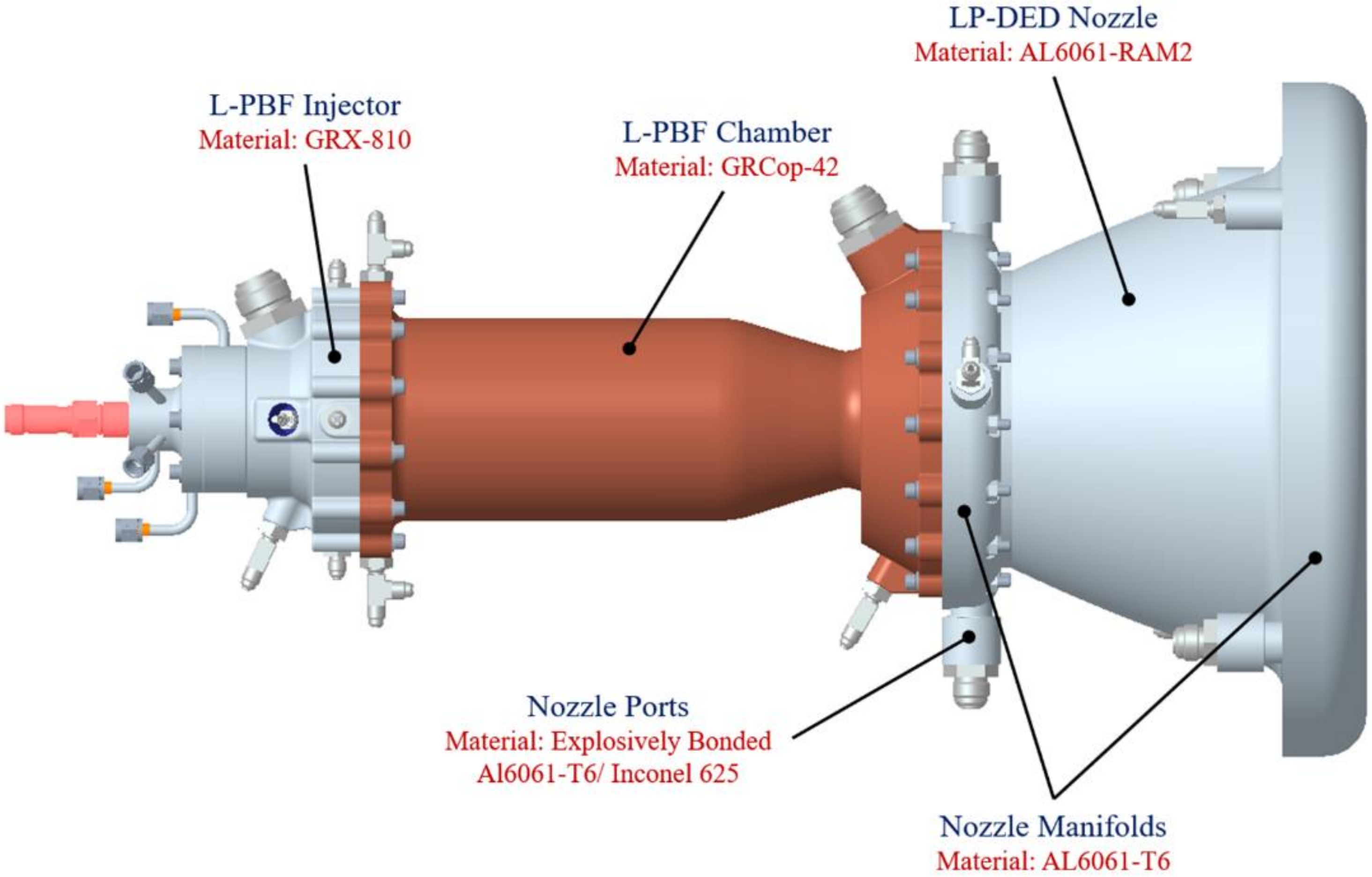
Backup

Thrust Chamber Assembly



Powder Feedstock

Chemical composition of Al6061-RAM2 (LP-DED) vs. (L-PBF).
***From a one powder lot; not specification values**

Element	Al6061-RAM2 (LP-DED)*	Al6061-RAM2 (L-PBF)*
	Wt. %	
Al	94.61	94.67
B	0.74	0.74
C	0.18	0.18
Cr	0.07	0.10
Cu	0.26	0.28
Fe	0.17	0.09
Mg	0.83	0.84
Mn	0.01	0.00
Si	0.56	0.54
Ti	2.45	2.44
Zn	0.00	0.01
Others, Each	< 0.05	< 0.05
Others, Total	< 0.16	< 0.16

Powder feedstocks for Al6061 and Al6061-RAM2 (LP-DED).

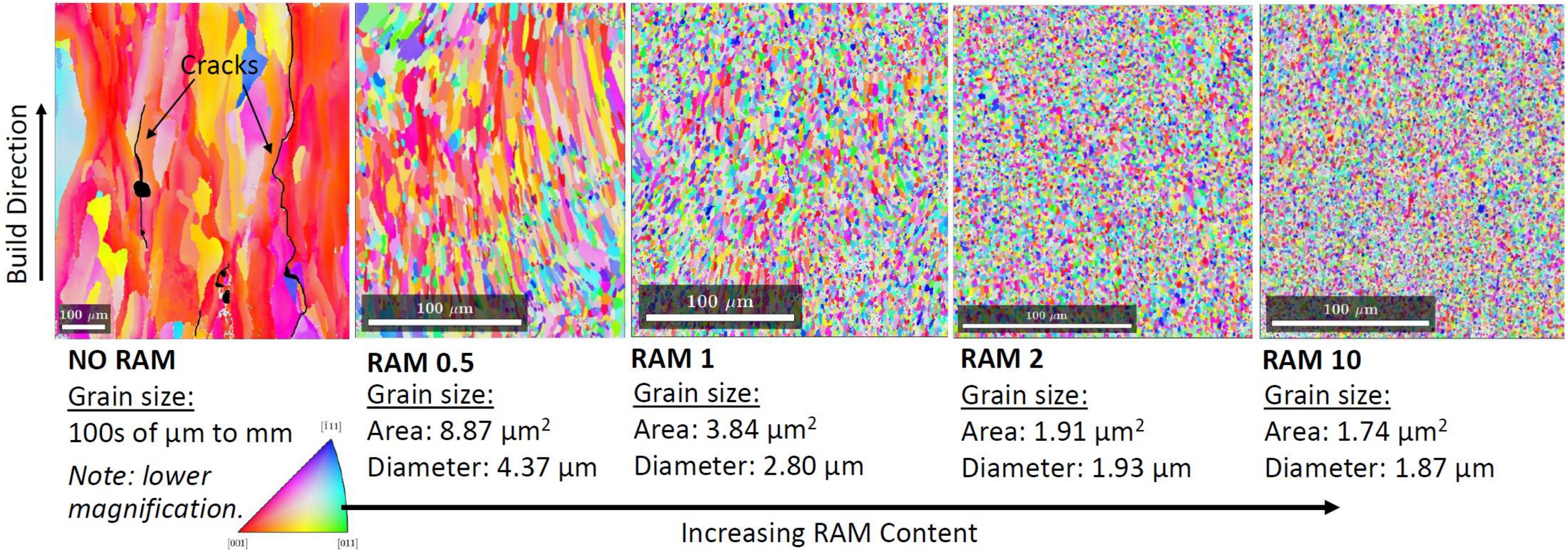
Material	Powder Size Distribution			Average Hall Flow Time [s]
	D10 [μm]	D50 [μm]	D90 [μm]	
Base 6061 (LP-DED)	46.4	67.6	96.0	59.6
Al6061-RAM2 (LP-DED)	44.3	67.3	96.3	59.7

Coefficient of Thermal Expansion properties.

Material	CTE [μm/m/°C]	
	50°C	100°C
Al6061-RAM2 (LP-DED)	21.8	22.2
Al6061-RAM2 (L-PBF)	22.4	-
Al6061-T6	22.6	23.0

LP-DED Development of Al6061-RAM2

- **Reactive Additive Manufacturing (RAM) process:**
  - Developed by Elementum 3D; initially developed for Laser Powder Bed Fusion (L-PBF)
  - Inoculates alloys against hot tearing (solidification cracking) due to nano-dispersoids that creates nucleation sites
  - Promotes weldability and maintains strength
  - For Al6061-RAM2, gas-atomized 6061 base powder is blended with ceramic particulates



NO RAM
Grain size:
100s of μm to mm
Note: lower magnification.

RAM 0.5
Grain size:
Area: 8.87 μm^2
Diameter: 4.37 μm

RAM 1
Grain size:
Area: 3.84 μm^2
Diameter: 2.80 μm

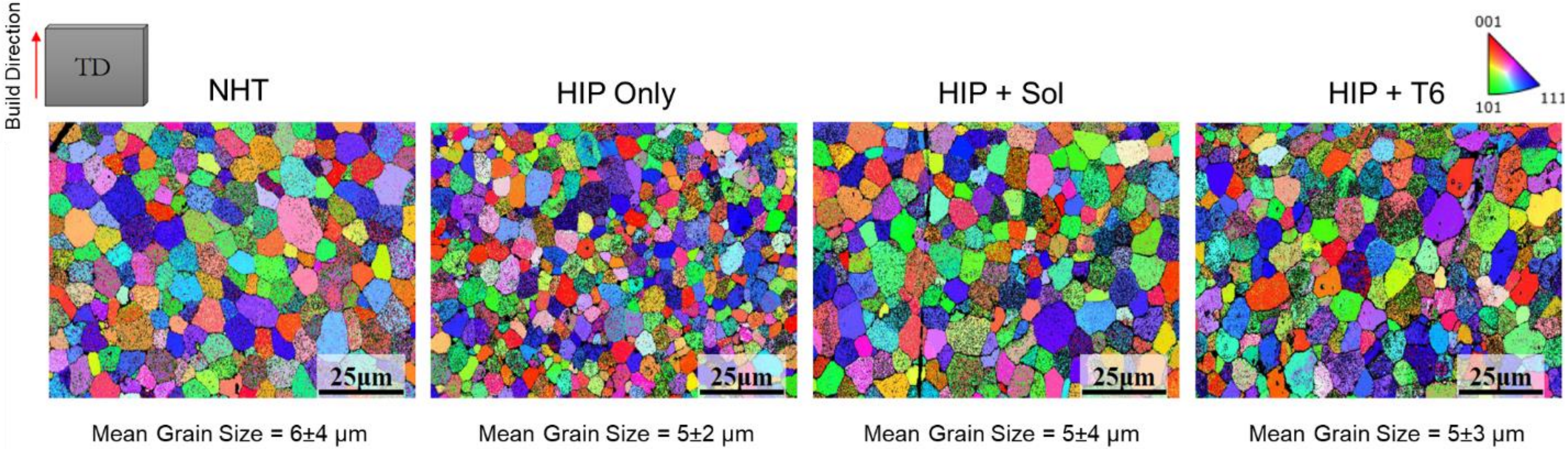
RAM 2
Grain size:
Area: 1.91 μm^2
Diameter: 1.93 μm

RAM 10
Grain size:
Area: 1.74 μm^2
Diameter: 1.87 μm

Increasing RAM Content

Microstructure

Material characterization was completed including microstructure, evaluation of heat treatment steps, and mechanical testing. Microstructure and mechanical properties were obtained from thin-wall (~1.5 mm / 0.060”), single bead build boxes. Microstructure samples were obtained after each heat treatment step to characterize the as-built, post-hot isostatic pressing (HIP), post-Solution, and fully heat-treated (age hardened) condition. Equiaxed, fine grains were observed in all the conditions and the mean grain size for LP-DED Al6061-RAM2 is ~5 µm. The addition of B4C and Ti inoculant particles provides heterogenous nucleation sites for solidification, which causes refinement of the grain structure and formation of equiaxed grains. Mean grain sizes were larger for LP-DED samples, 5 µm, compared to L-PBF samples, 1.5 µm. The grain size from both printing processes did not significantly change after heat treatment because the inoculant particles acted as grain boundary pinners.



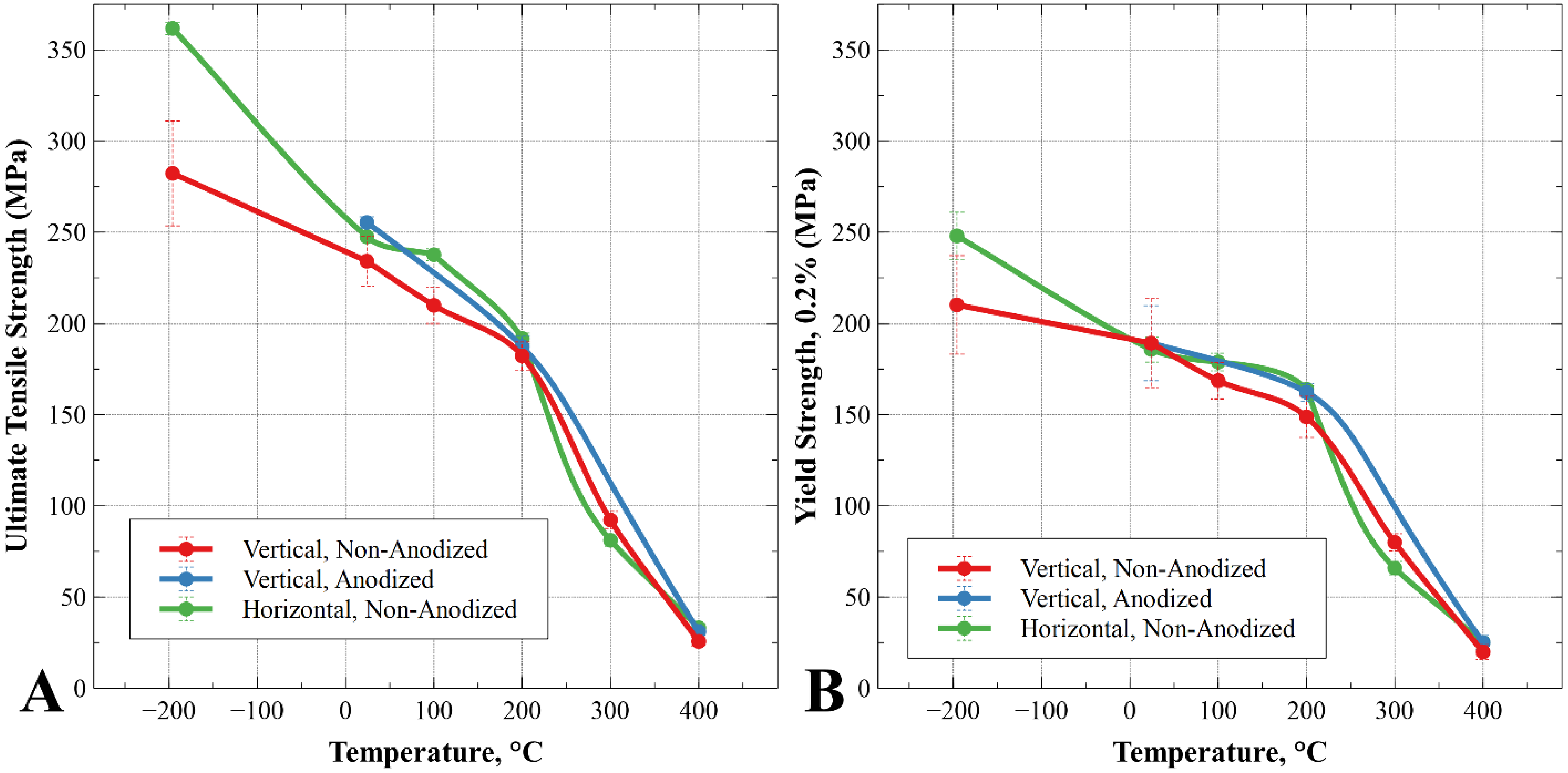
Mean Grain Size = 6±4 µm

Mean Grain Size = 5±2 µm

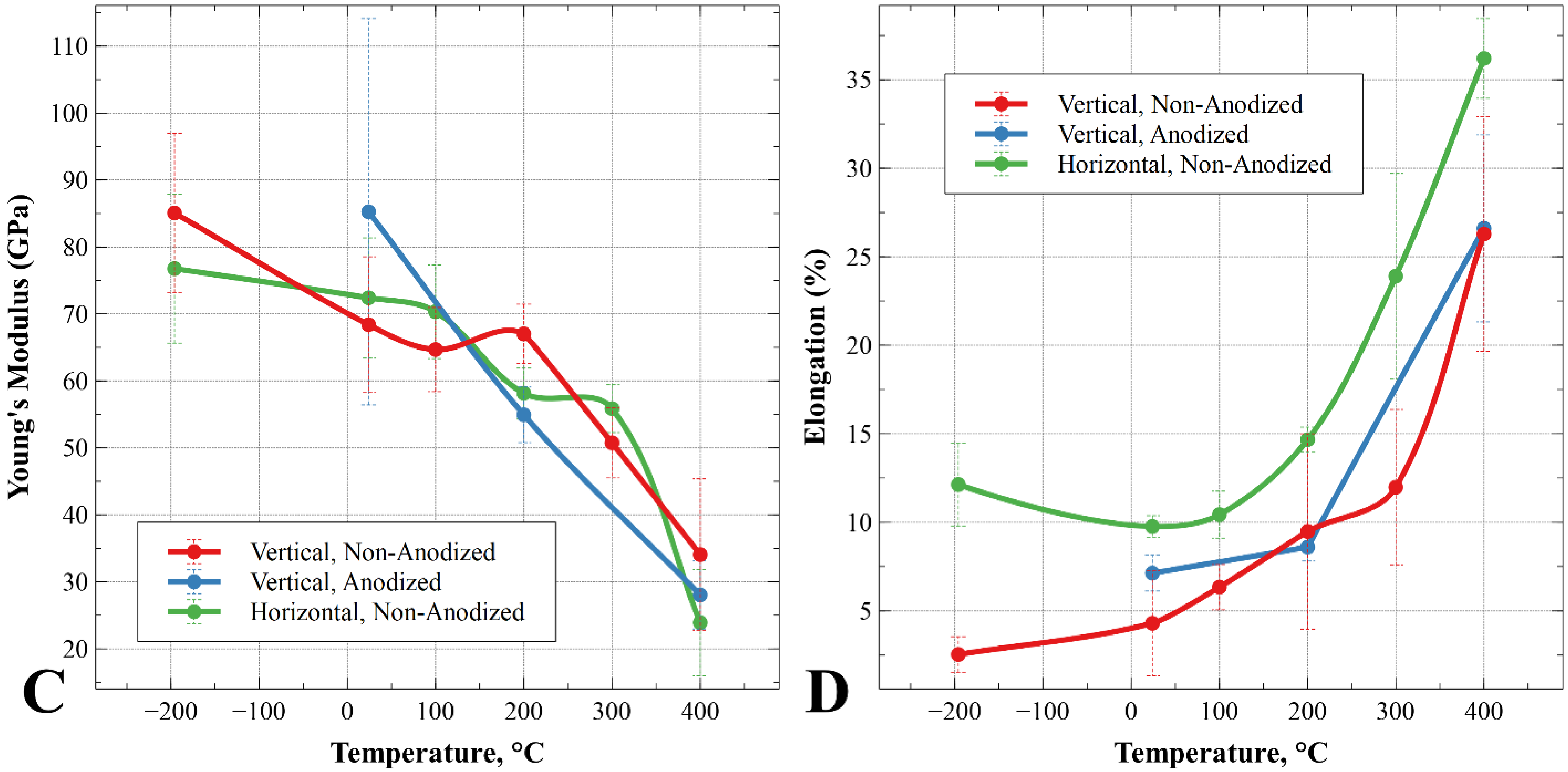
Mean Grain Size = 5±4 µm

Mean Grain Size = 5±3 µm

Material Properties



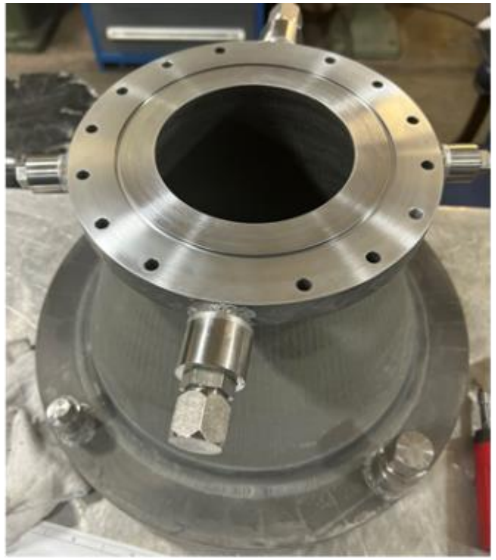
Material Properties



Anodization

Pre-Anodization Setup

Post-Anodization



Post-Final Machining Touch-up

Anodization is an electrochemical process that converts the surface of an aluminum material into a natural anodic oxide coating. This oxide coating provides a durable, corrosion-resistant surface and the idea was that this surface would potentially serve as a thermal barrier coating for additional insulation of the hot-wall. Aluminum is typically not used in combustion devices in the hot gas flow path, and there was limited experience with anodization for this environment. The nozzle was delivered to anodization with the explosively bonded Inconel 625 ports already welded onto the manifolds. Anodization is strictly for aluminum, and other materials such as Inconel 625, could be damaged if anodization is attempted. Other (non-aluminum) materials could also damage the anodization bath/mechanism. Therefore, masking was required on the Inconel 625 ports and the nozzle was successfully anodized per MIL-PRF-8625F Type III Class I anodization and a 0.076 mm (0.003 in.) layer thickness

Port Testing

Test Fixture #	Description	# of Cycles	Burst Pressure	
			[MPa]	[psig]
1	Aluminum Threads	30	132.3	19,193
2	Aluminum Threads	23	146.4	21,228
3	Bolted Flange	24	56.1	8,142
4	Explosively Bonded	22	46.5	6,745