National Aeronautics and Space Administration

Heat Treatment Optimization of Laser Powder Bed Fusion Additive Manufacture C103

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Presentation Outline

- Motivation for Work
- Background on Refractory Metals (C103), Heat Treatments, HIP-Q
- Initial Heat Treatment Schedule
- Metallographic Results and Comparison
- Mechanical Property Results and Comparison
- Summary and On-Going Work
- Acknowledgements

AM is an appealing manufacturing technique for refractory metals

Traditional manufacturing of refractories remains difficult due to demanding fabrication processes and costs

- **Additive manufacturing (AM) allows for near net shape fabrication, therefore reduces the need for machining and post-processing**

Refractory alloys are known for high melting temperature, high corrosion resistance, and elevated mechanical property performance up to 1400-1800 °C

C103 (Nb-10Hf-1Ti wt%) is a refractory metal with BCC crystal structure, but remains ductile at room temperature

C103 Applications:

- Green Propulsion Thrusters
- Thrust augmenter flaps in jet engines
- Rocket nozzles

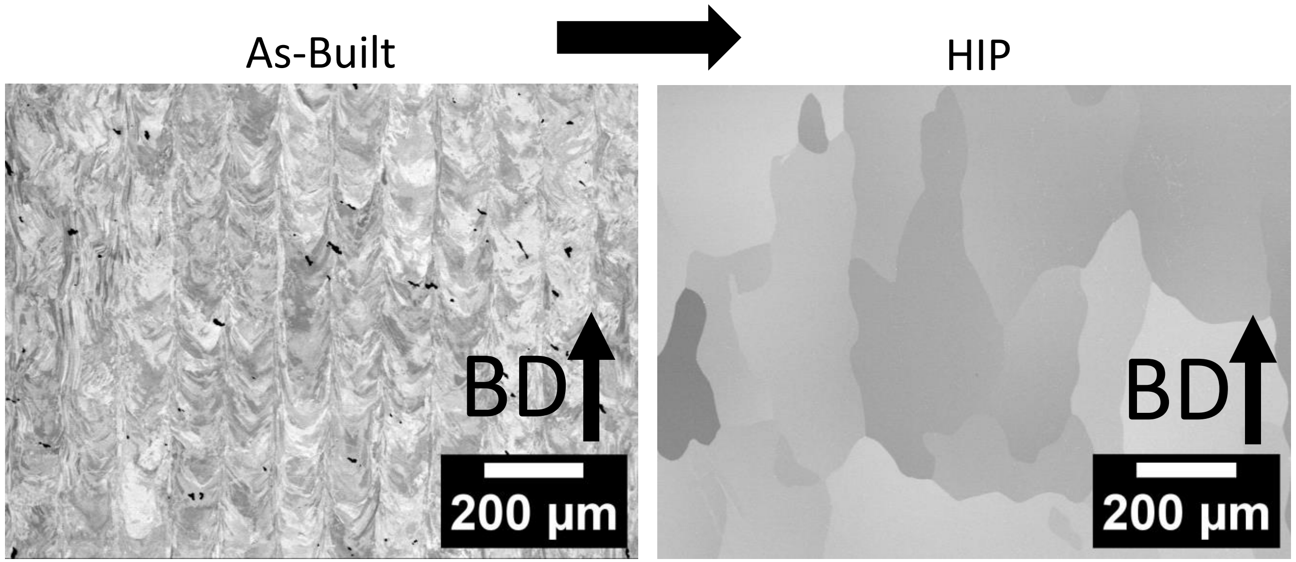
Examples of L-PBF and EBAM C103 components.

AM C103 Hot Fire Test

AM C103 MSFC Green Propulsion Thruster and Stand-off.

Photos: Additive Manufacture C103 for In-Space Propulsion - NASA Technical Reports Server (NTRS)

Heat treatment affects material properties



L-PBF AM C103 100X

Stress Relief *Reduce or mitigate residual stresses*

Hot Isostatic Pressing (HIP) *Reduce porosity to increase density and fatigue life*

Solution or Anneal *Alloy Specific*

Aging *Alloy Specific*

HIP greatly improves density, but in the case of C103, fatigue life is decreased after HIP

- **The associated grain growth results in a reduction in mechanical properties**
- **HIP is required to meet NASA Standard 6030 Section 5.4.3.3**

HIP-Q Heat Treatment

Uniform Rapid Cooling, URC®, is a Quintus invention that enables high speed, uniform cooling of the payload

- Cold gas in the HIP is exchanged with hot gas in the furnace at a high rate, effectively quenching the payload in a uniform way with minimal induced stress
- Cooling rate depends on the furnace
  - Max cooling rate for a Mo URC is ~200 °C/min

Photo Courtesy of Quintus Technologies: Quintus HIP Machine
[High Pressure Technology | Quintus Technologies]

HIP-Quench (HIP-Q) may address the problem of grain growth. It introduces a “quench” to limit grain size enlargement during post-processing

Heat Treatment Schedule

All specimens are sonicated in IPA, compressed air dried, and wrapped in Ta foil (twice) before heat treatment

Heat Treatment	Temperature (°C)	Hold Time (hour)	Pressure (MPa)	Cooling Rate (°C/min)
SR [1]	1100	1	Vacuum	-
HIP [2]	1600	3	104	-
HIP-Q	1400-1500	3	150-200	200

*HIP required to meet NASA Standard 6030 Section 5.4.3.3.

C103 Specimens & Ta wrap (pre-SR) [1]

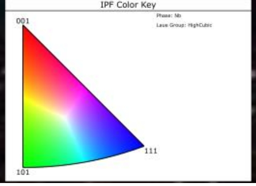
Post-HIP Ta wrap brittle from C &O [1]

[1] Mireles, Omar, et al. "Additive manufacture of refractory alloy C103 for propulsion applications." AIAA Propulsion and Energy 2020 Forum, 17 Aug. 2020, https://doi.org/10.2514/6.2020-3500.

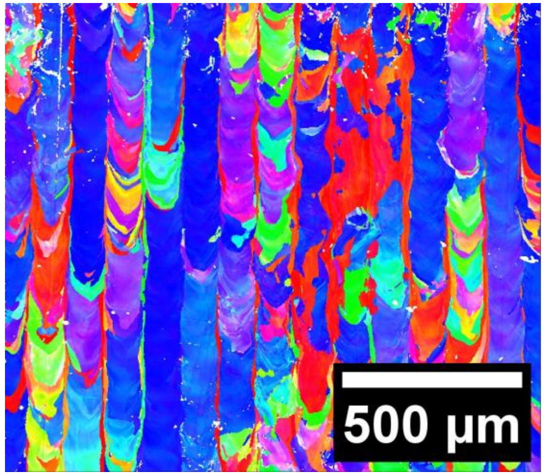
[2] Wadsworth, J., et al. "Creep behaviour of hot isostatically pressed niobium alloy powder compacts." Journal of Materials Science, vol. 17, no. 9, Sept. 1982, pp. 2539–2546, https://doi.org/10.1007/bf00543885.

Grain size changes after HIP

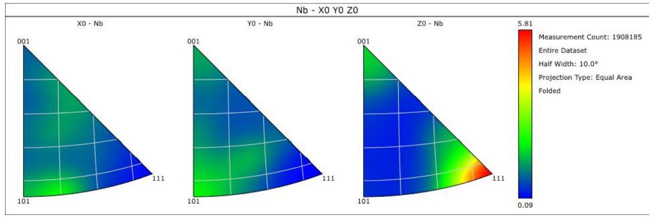
BD



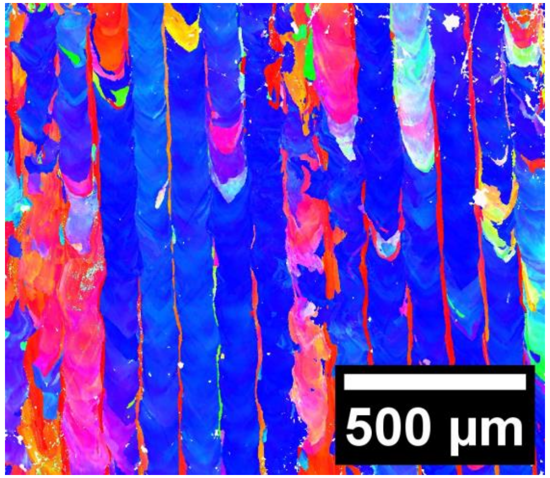
As-Built Condition



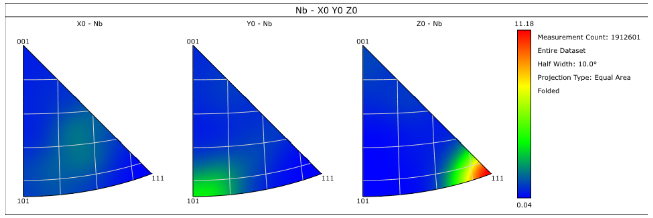
Average Grain Size: 1803.82 μm^2
Standard Deviation: 7255.57



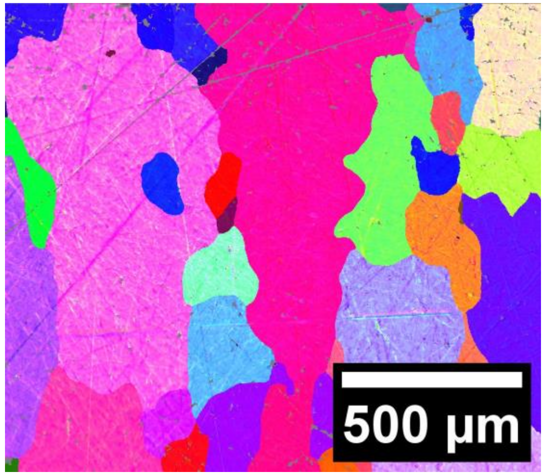
Stress Relieved Condition



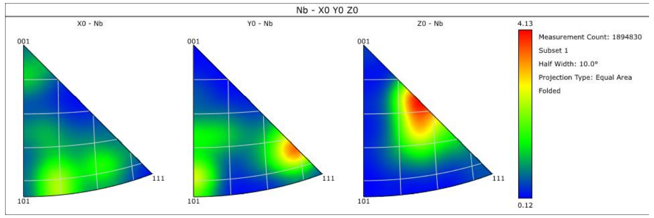
Average Grain Size: 2029.17 μm^2
Standard Deviation: 15144.10



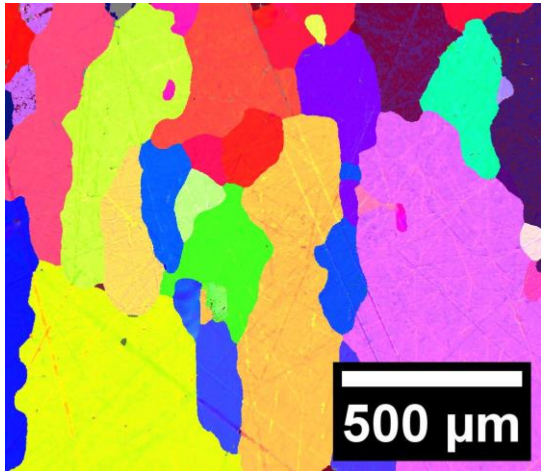
HIP Condition



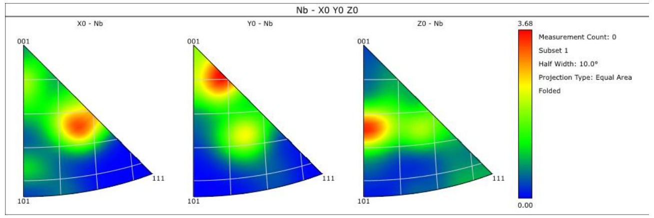
Average Grain Size: 55730.29 μm^2
Standard Deviation: 102168.11



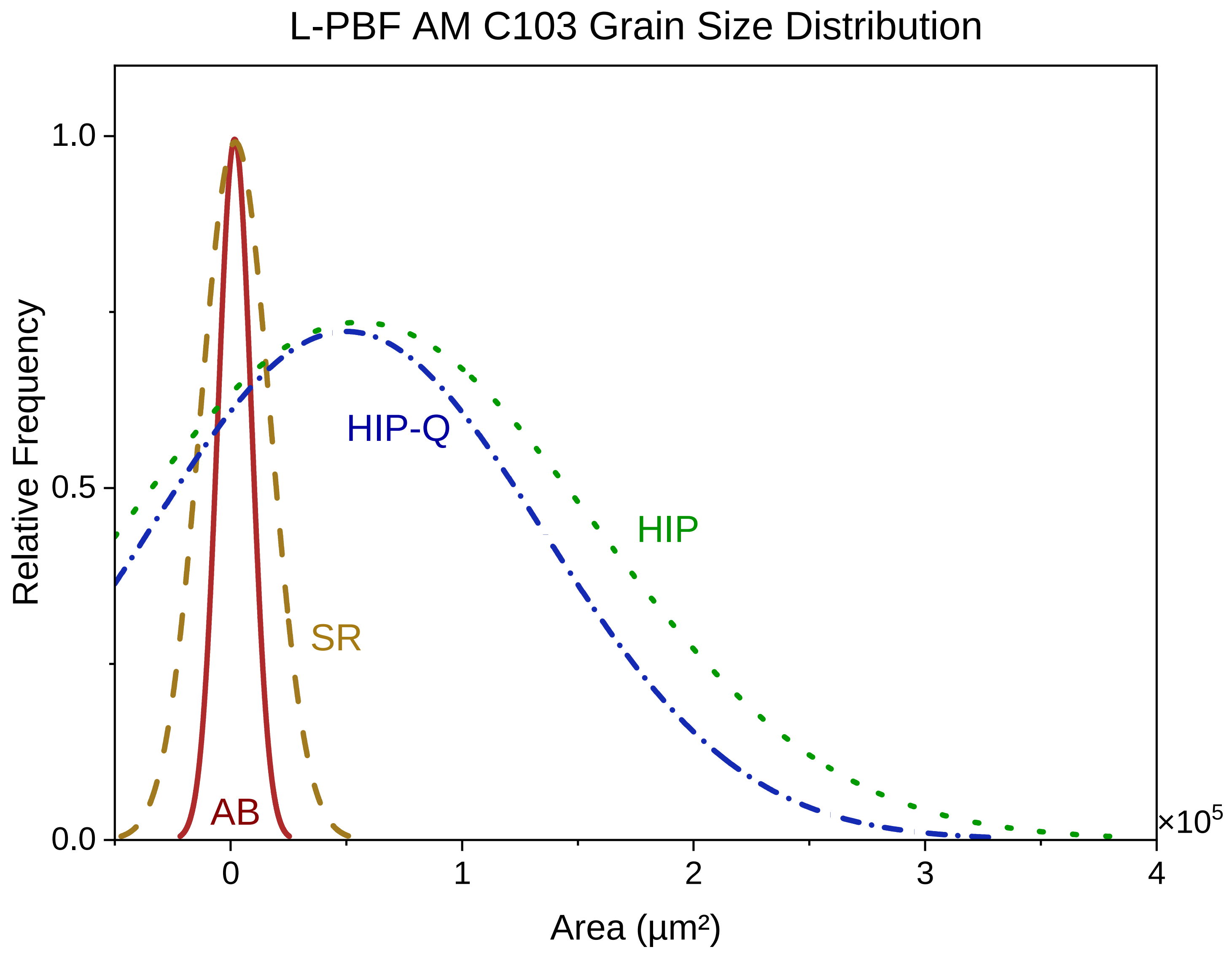
HIP-Q Condition



Average Grain Size: 49928.08 μm^2
Standard Deviation: 85288.67



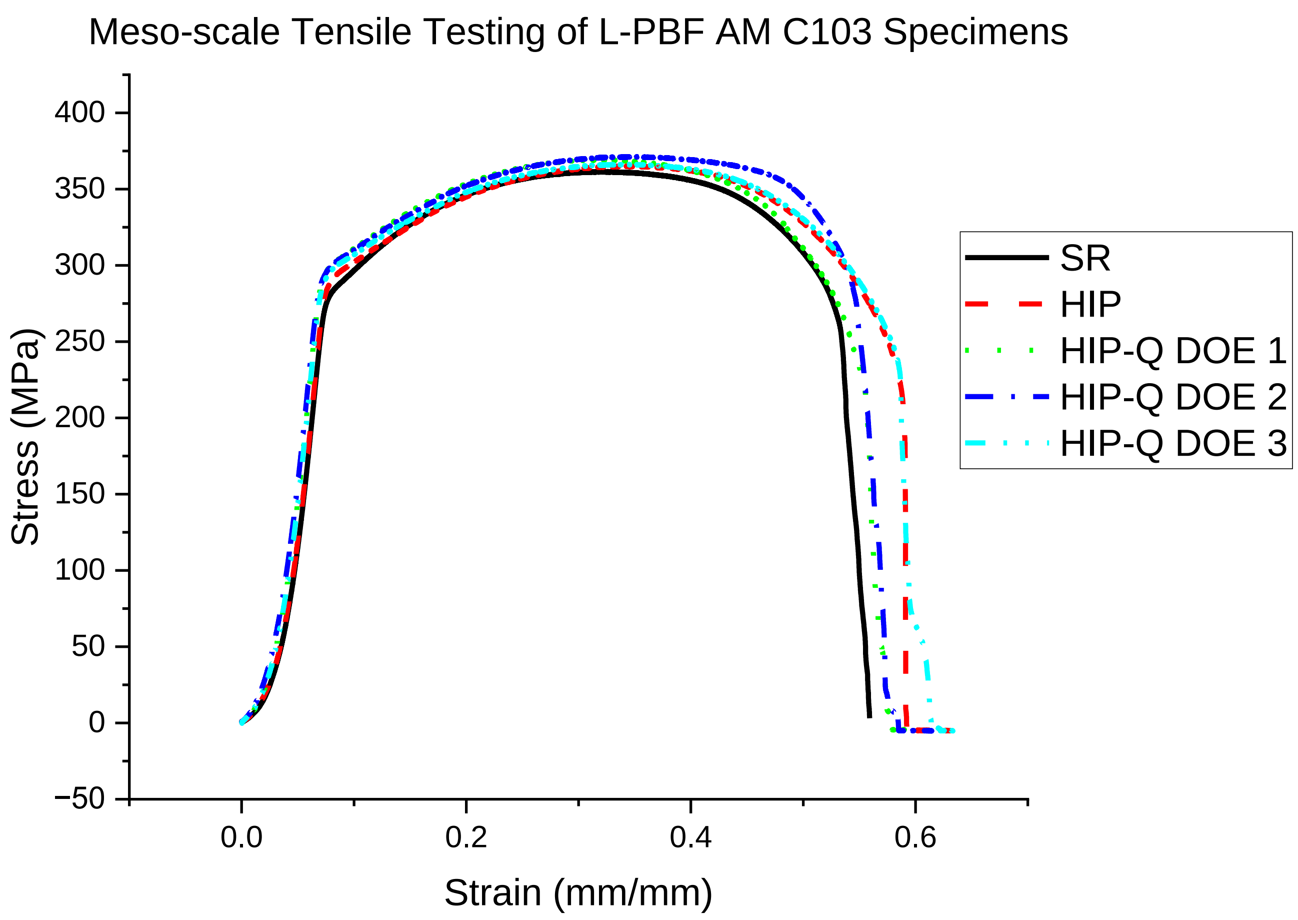
Current HIP-Q Schedule Ineffective

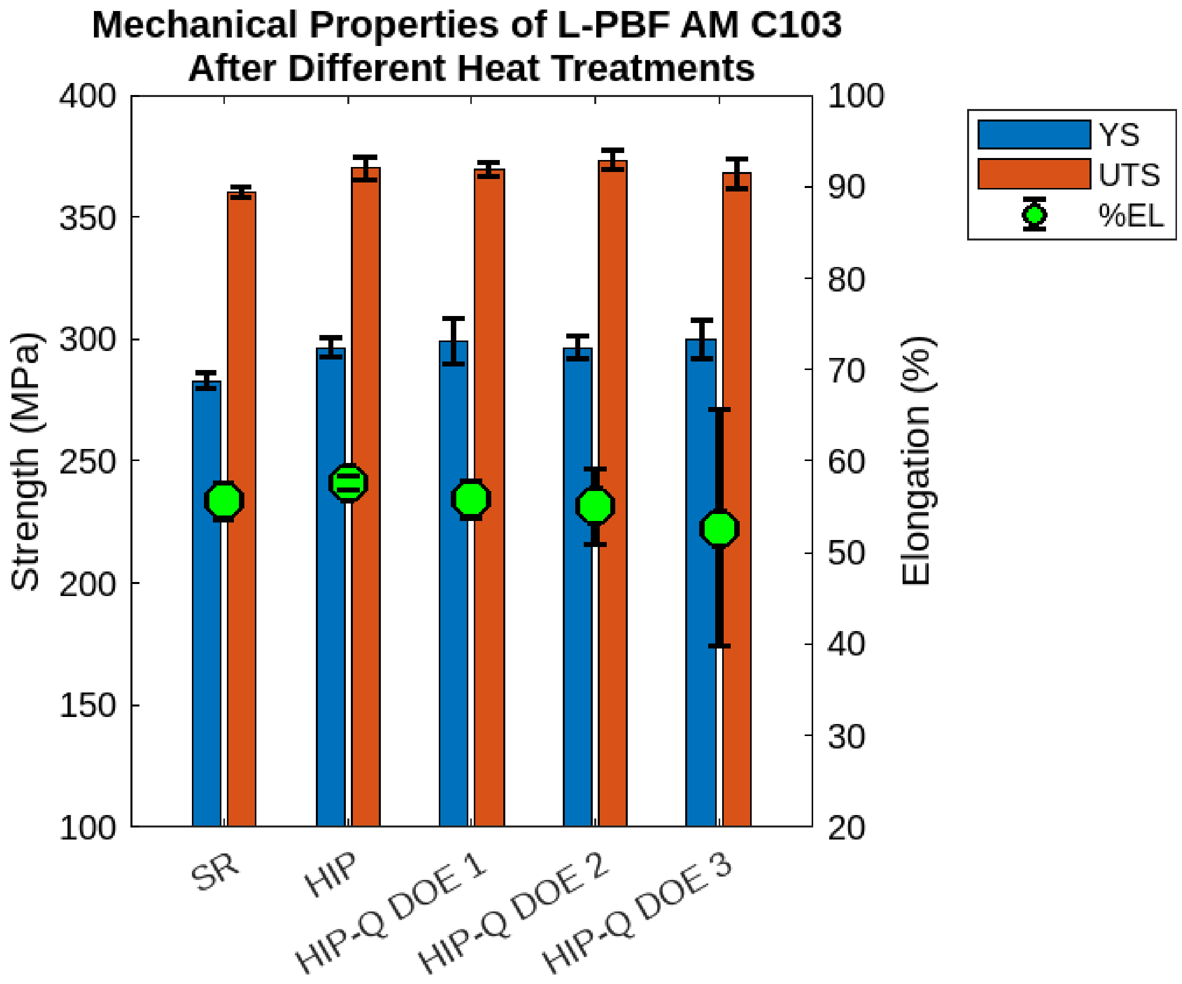


Heat Treatment	Average Grain Size - µm² ($\mu \pm \sigma$)
AB	1803.2 ± 7255.57
SR	2029.17 ± 15144.10
HIP	55730.29 ± 102168.11
HIP-Q	49928.08 ± 85288.67

No significant grain size reduction observed when comparing HIP and HIP-Q microstructures

Current HIP-Q Schedule Ineffective

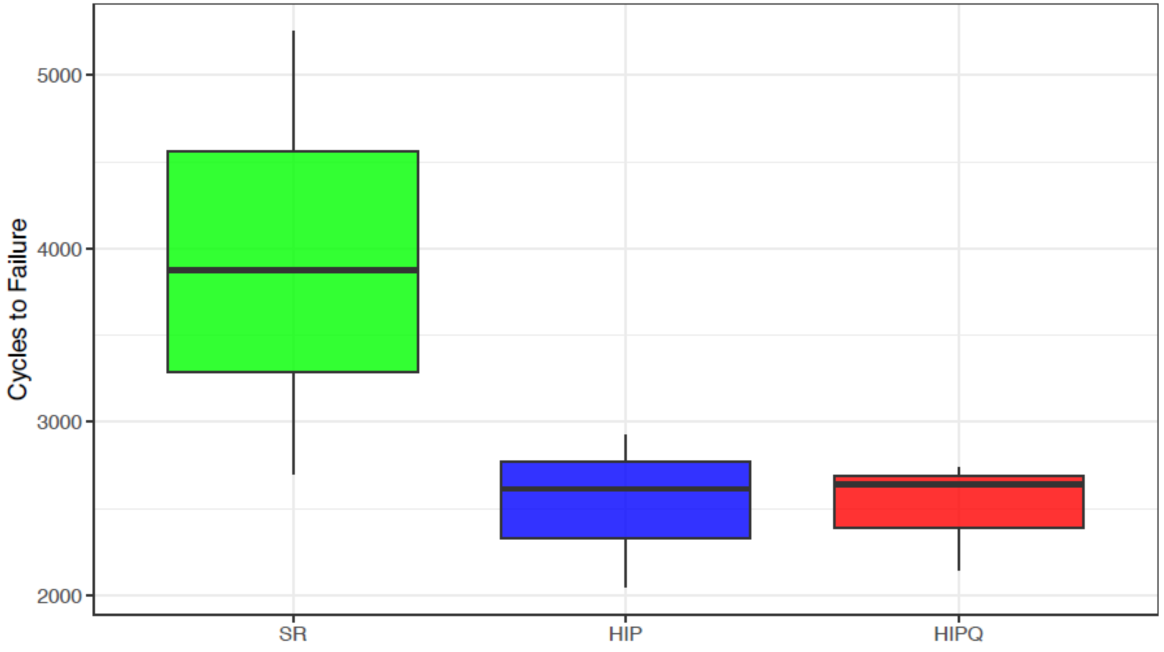




No statistically different UTS, YS, or %E between HIP and HIP-Q specimens

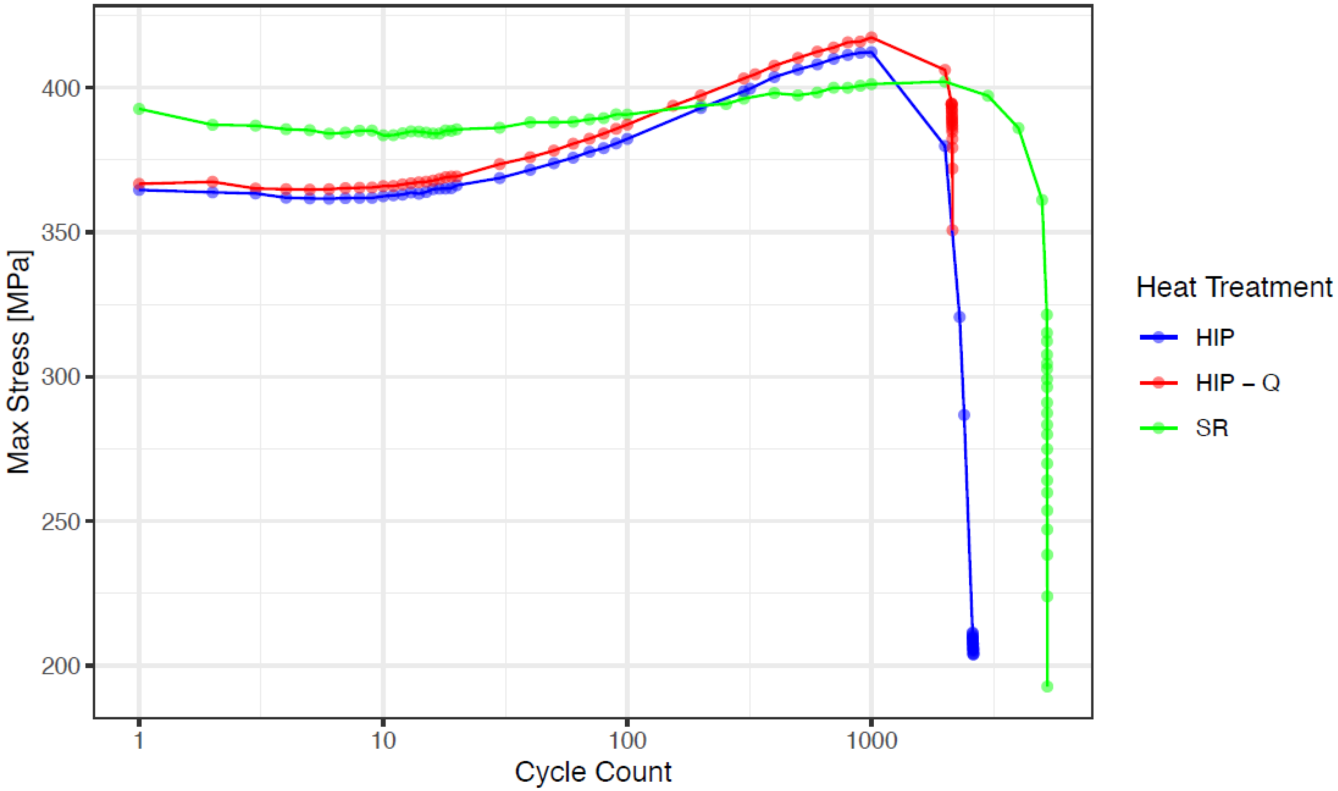
Current HIP-Q Schedule Ineffective

Low Cycle Fatigue (LCF) Testing of L-PBF C103 – Cycles to Failure



- SR condition exhibits higher cycles to failure and the largest variation amongst heat treatments

Maximum Stress observed under LCF Testing of L-PBF C103



- SR condition remains relatively constant until end of life
- HIP & HIP-Q exhibit hardening behavior until end of life

Lower LCF life in HIP and HIP-Q conditions compared to SR condition

Summary and On-Going Work

Goal of this work is to look at the potential benefit of leveraging HIP followed by rapid cooling to minimize grain growth

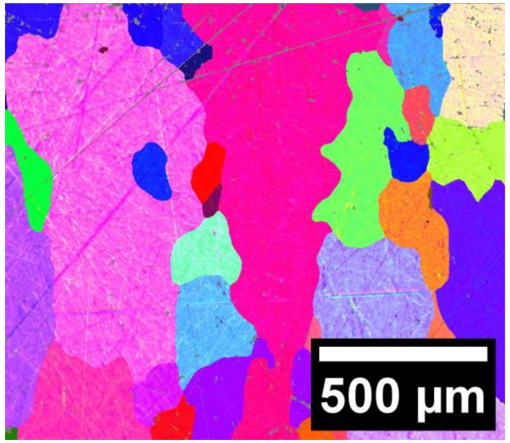
Current HIP-Q schedule (varying temperature) is ineffective

- No significant grain size reduction
- Mesoscale tensile tests reveal no statistically different UTS, YS, or %E between HIP and HIP-Q specimens
- LCF life reduced following HIP and HIP-Q heat treatments

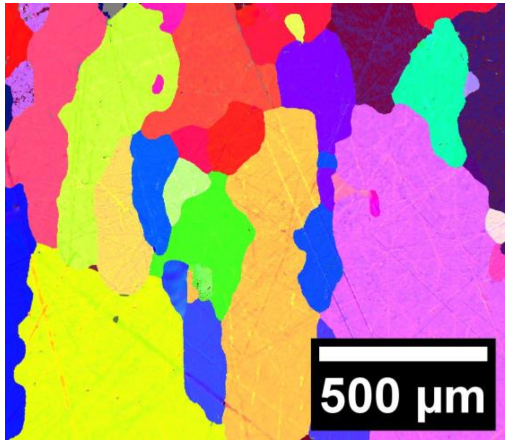
Updated HIP-Q schedule to reduce hold-time

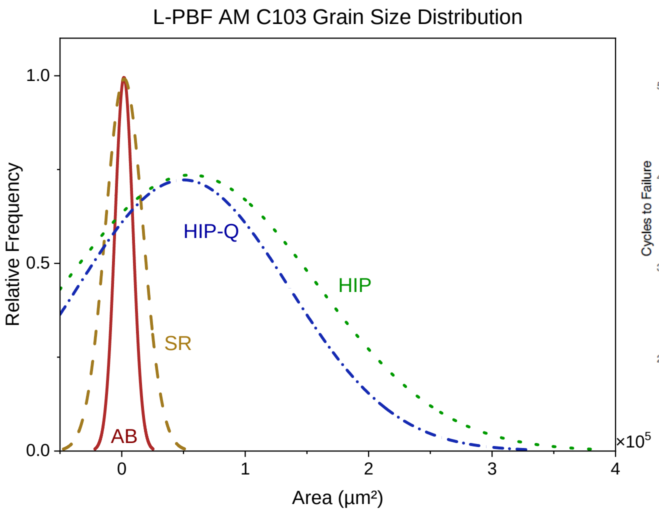
- Expect grain size difference after shorter hold

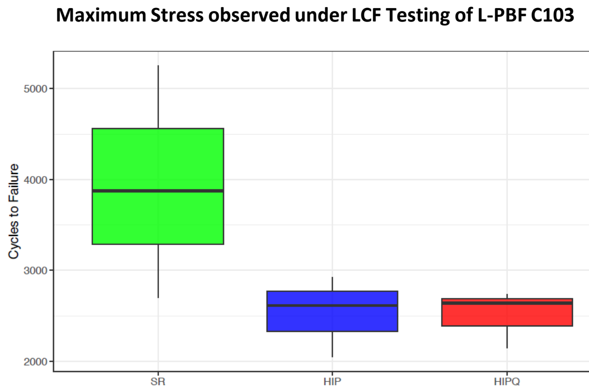
HIP



HIP-Q







Acknowledgements

Special Thanks to:

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Brandon Colon – University of Texas at El Paso

Omar Mireles - Los Alamos National Laboratory

Thank you for your attention!

Feel free to contact me with additional questions:

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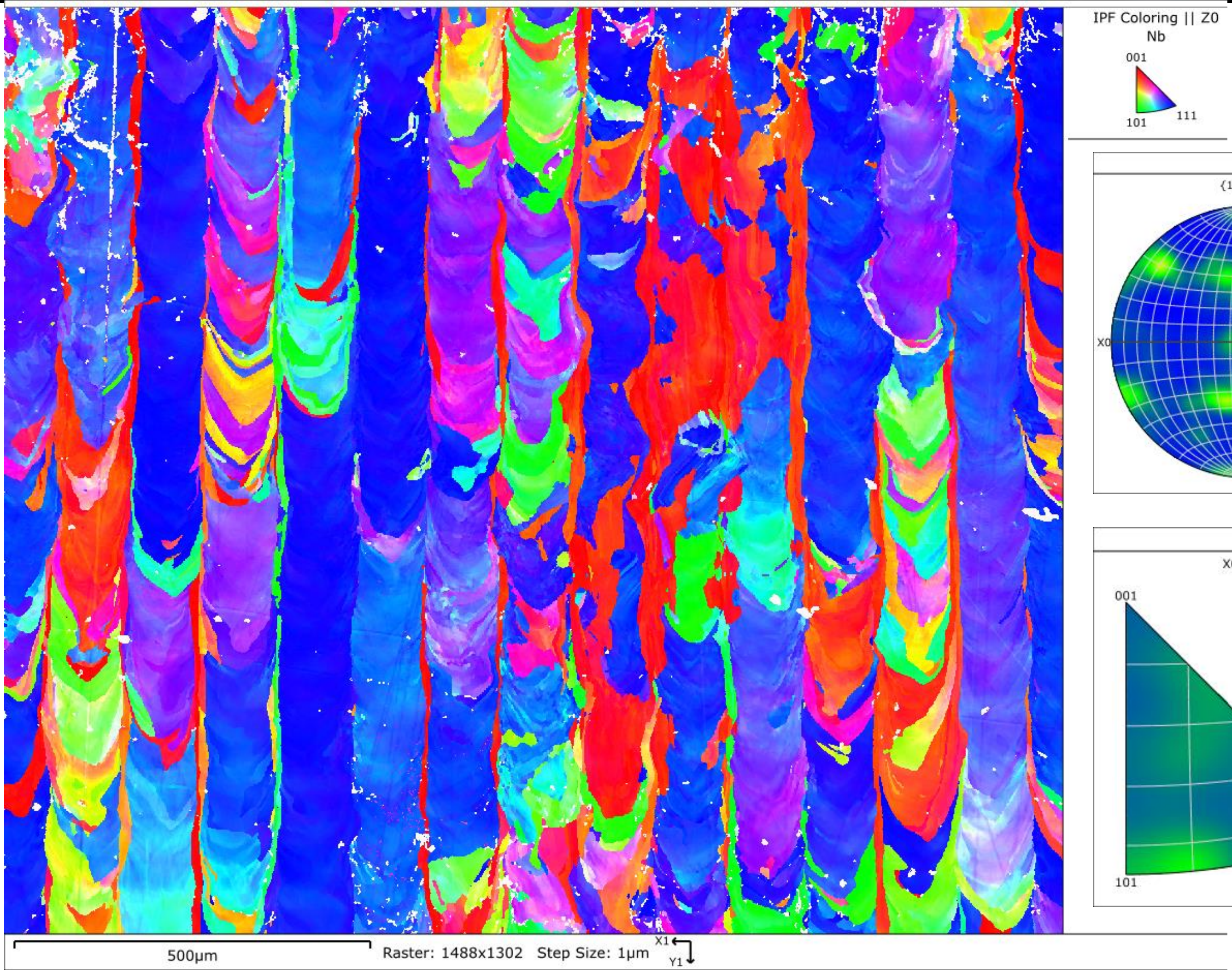
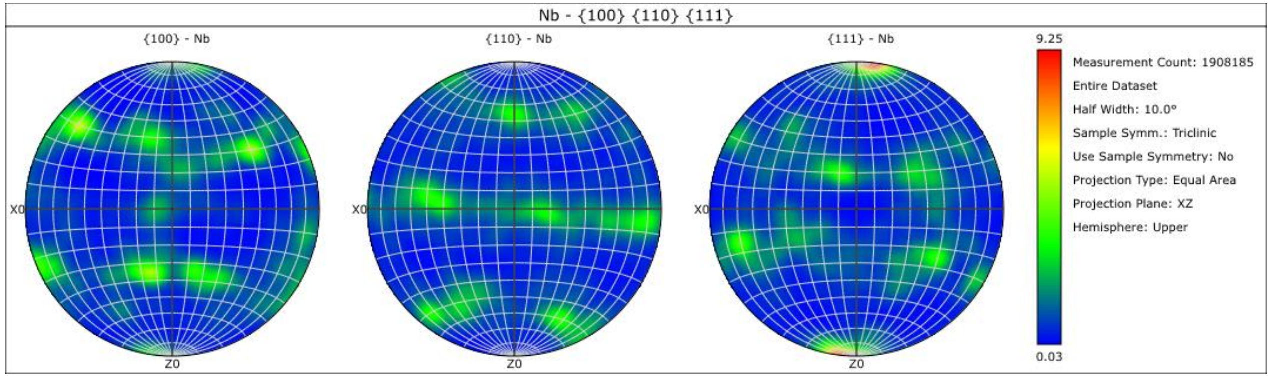
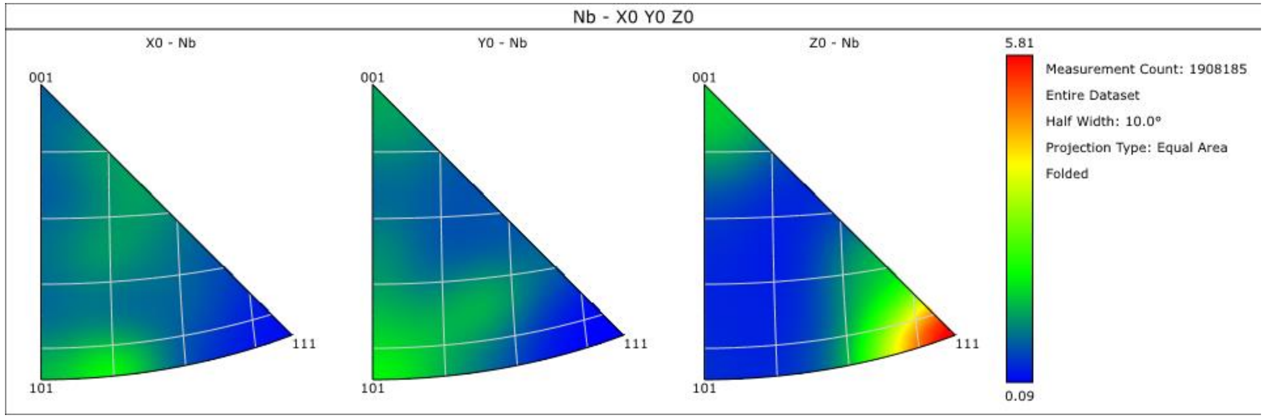
Backup Slides

As- Built Condition



Preferential texture parallel to <111> in the XZ plane (parallel to build direction)

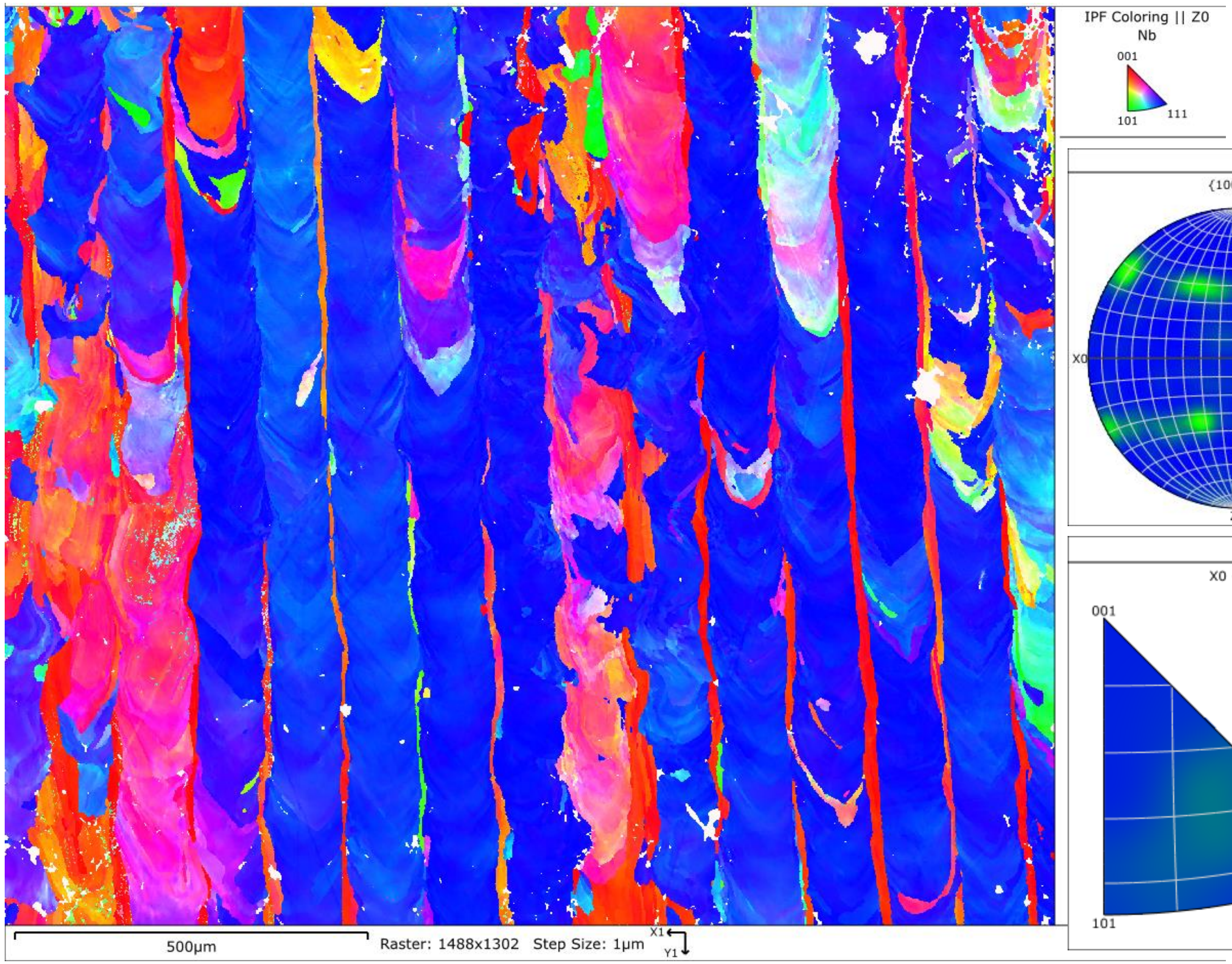




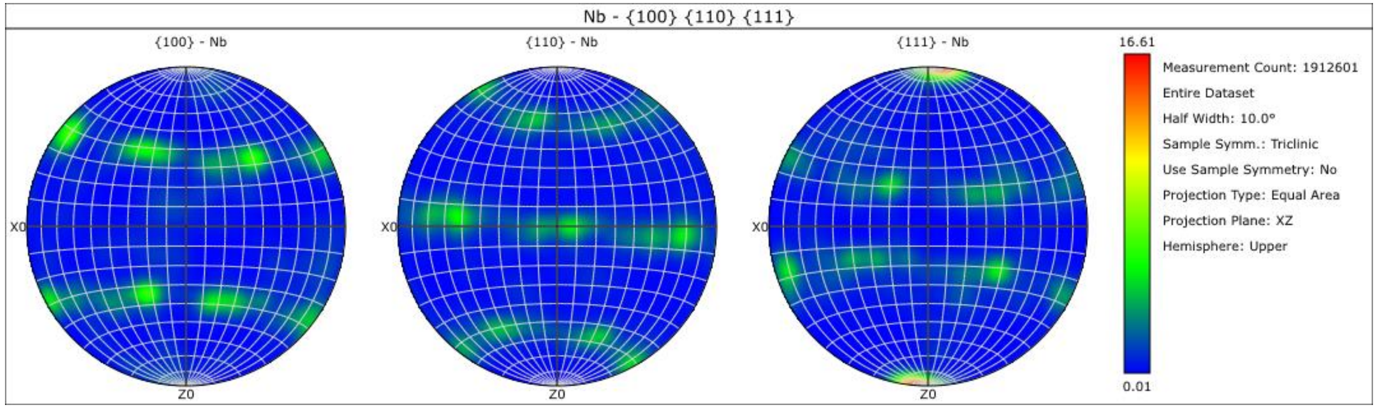
Average Grain Size: 1803.82 μm²
Standard Deviation: 7255.57

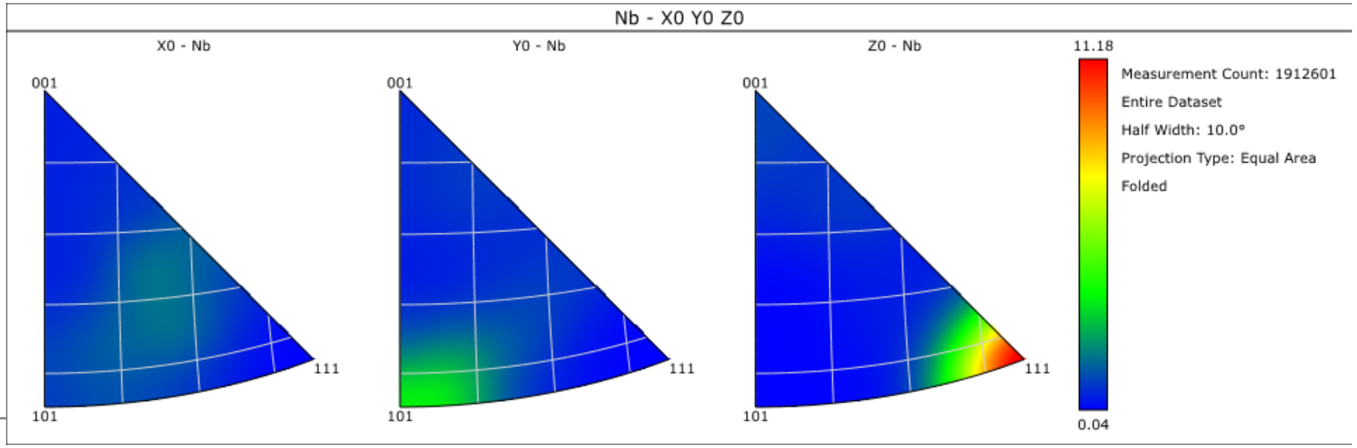
Grains are oriented on the <111> direction along the build direction. Material shows a highly <111> texture along the build direction

Stress Relieved Condition



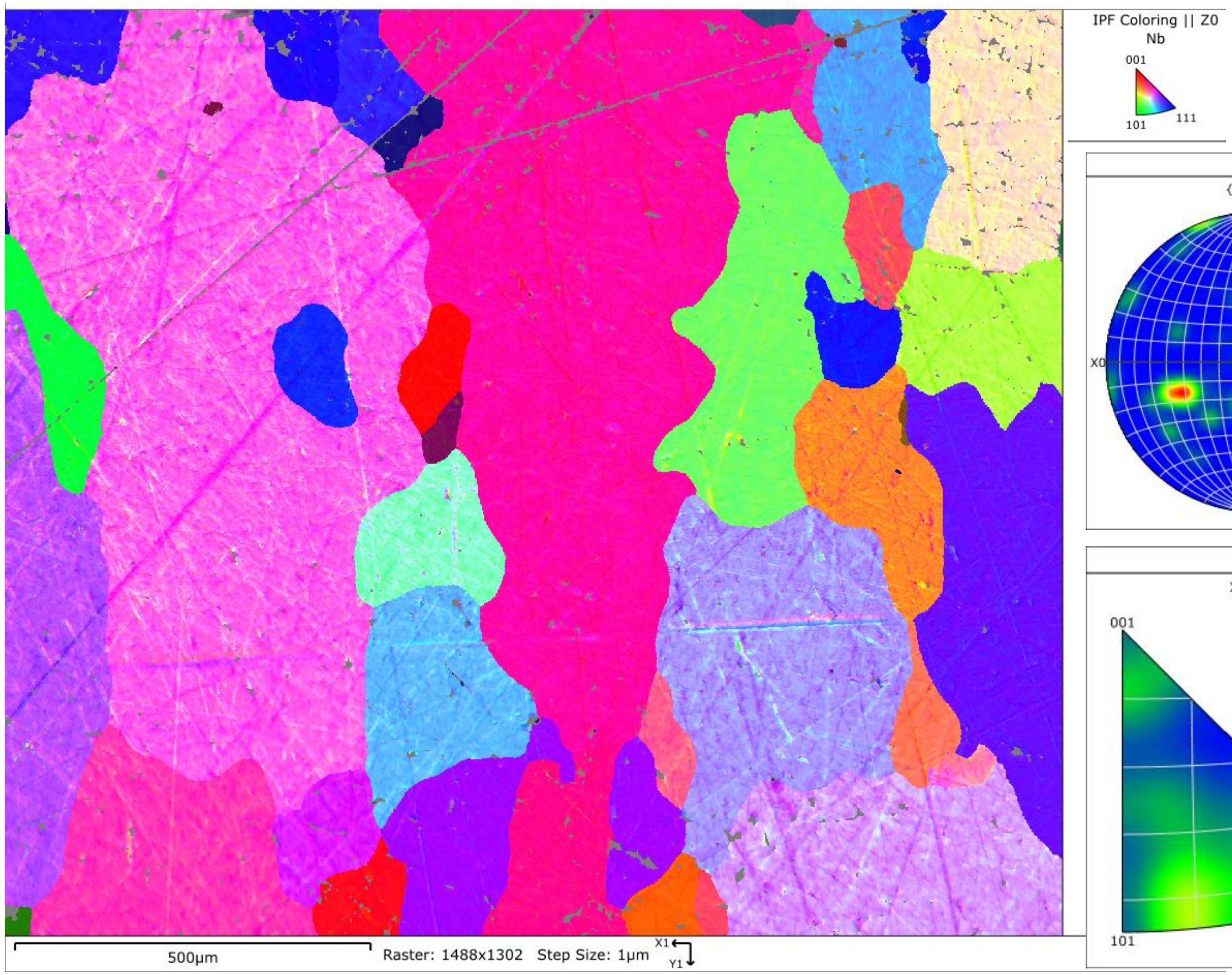
Preferential texture parallel to <111> in the XZ plane (parallel to build direction)-



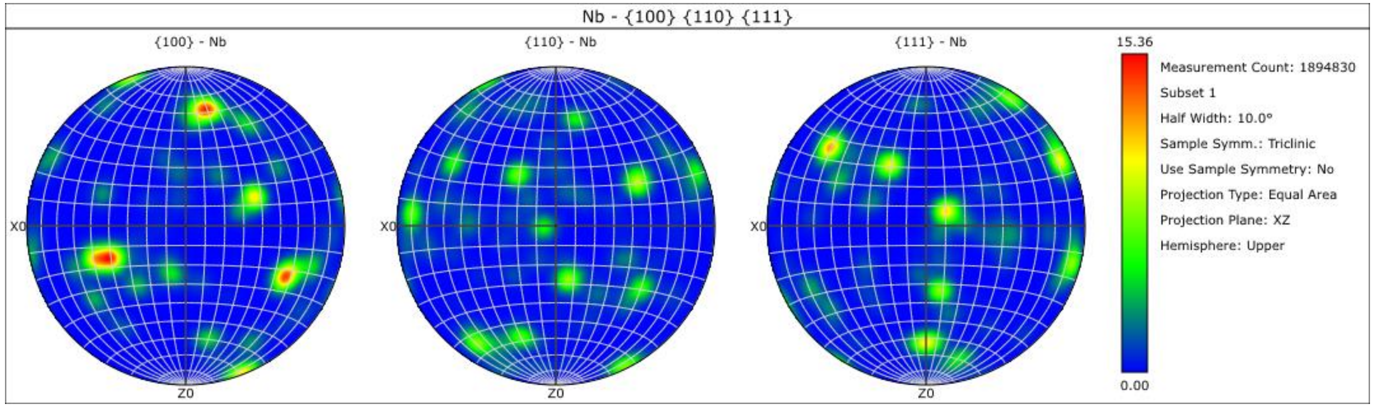


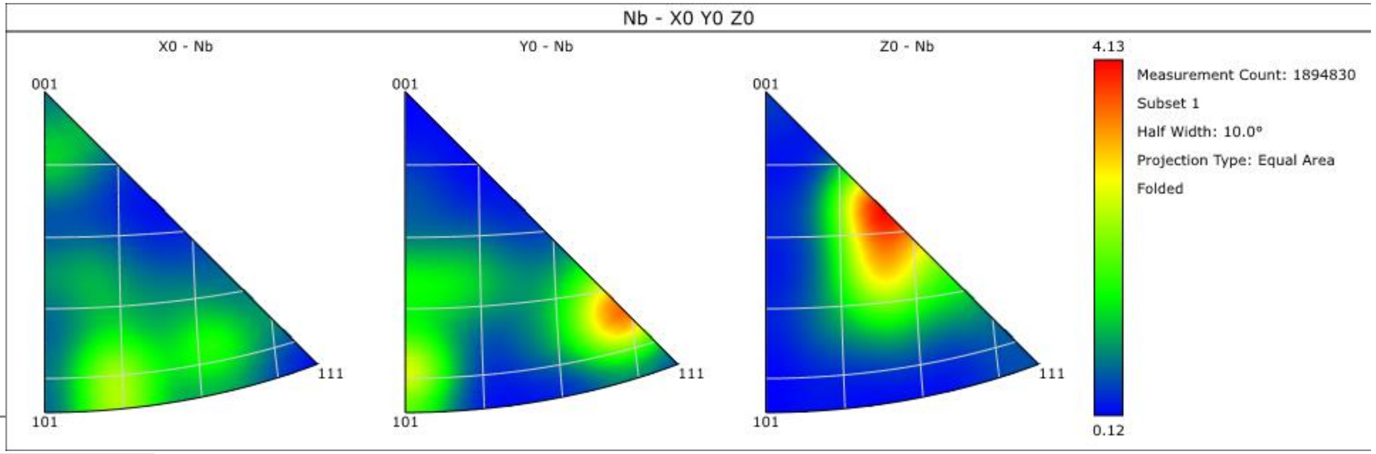
Average Grain Size: 2029.17 µm²

HIP Condition



No preferential texture, the sample has homogenized

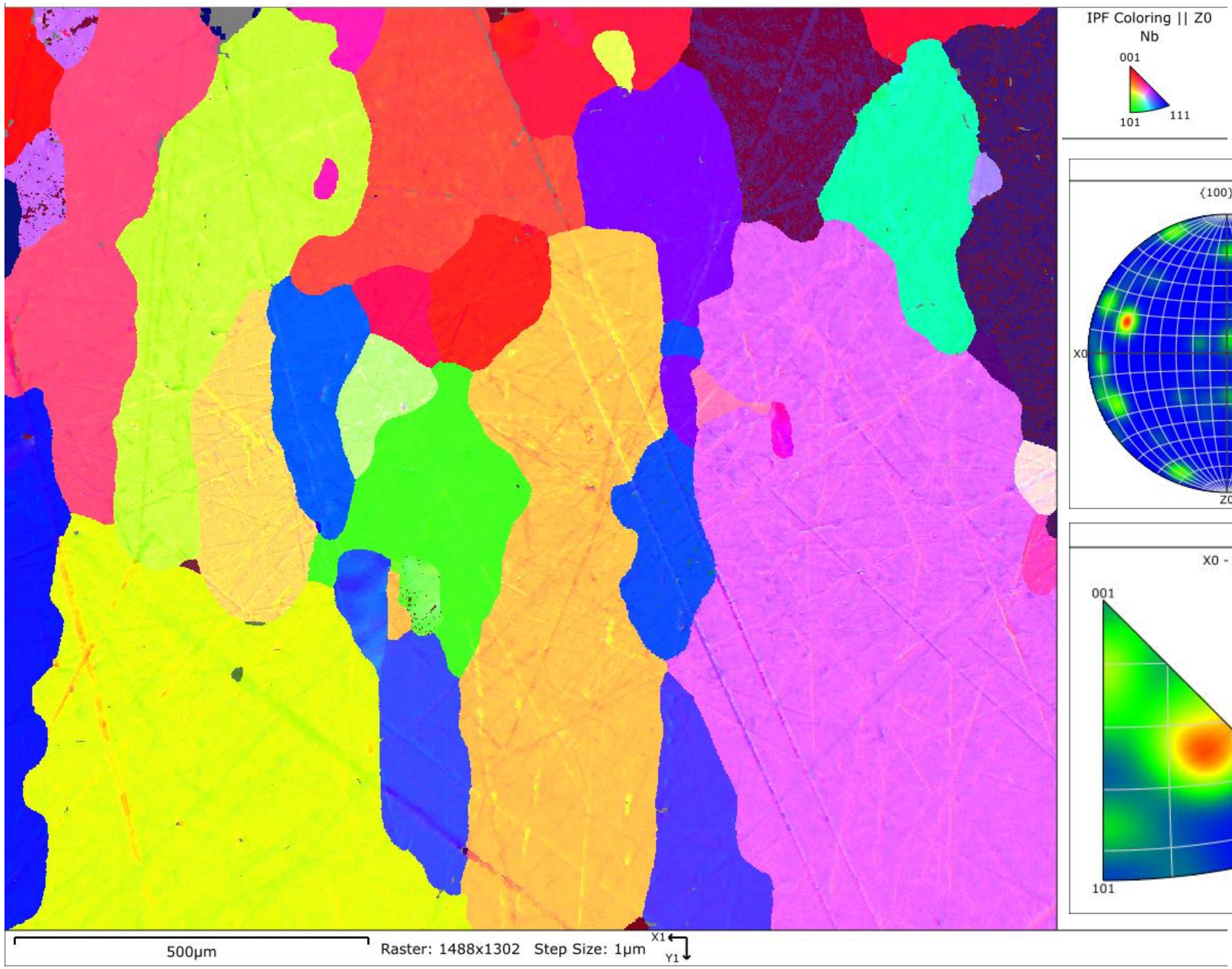


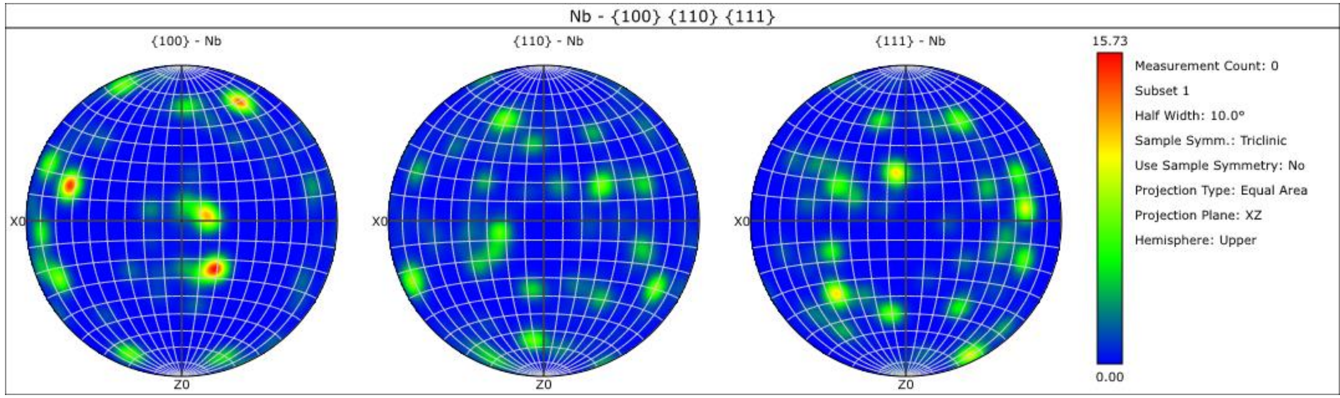


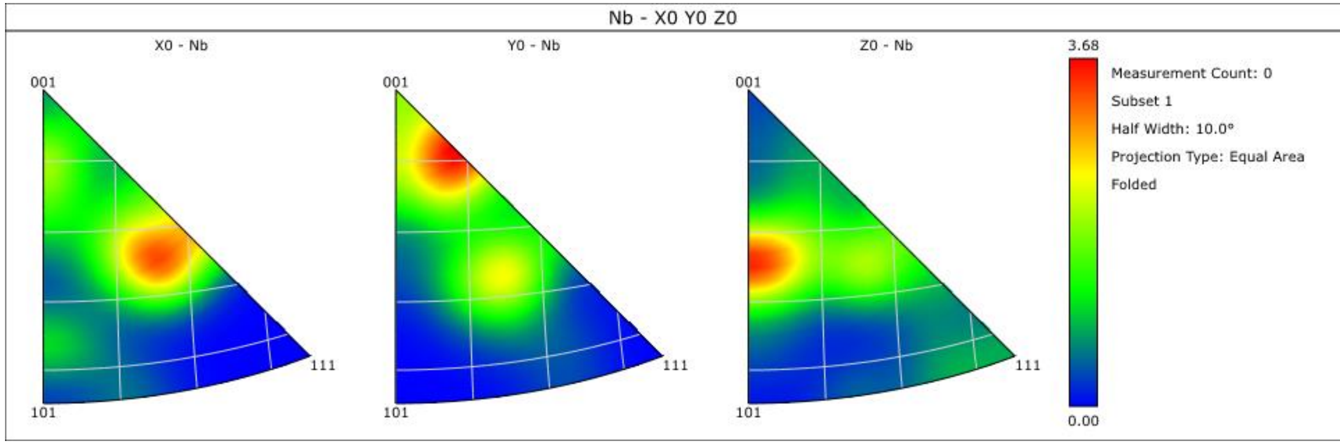
Average Grain Size: 55730.29 µm²

HIP-Q Condition

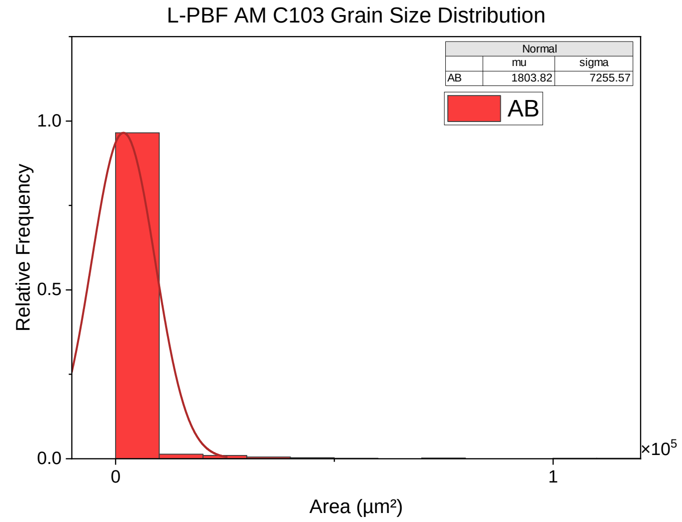
No preferential texture; the same has homogenized. Visually smaller grains than the HIP condition

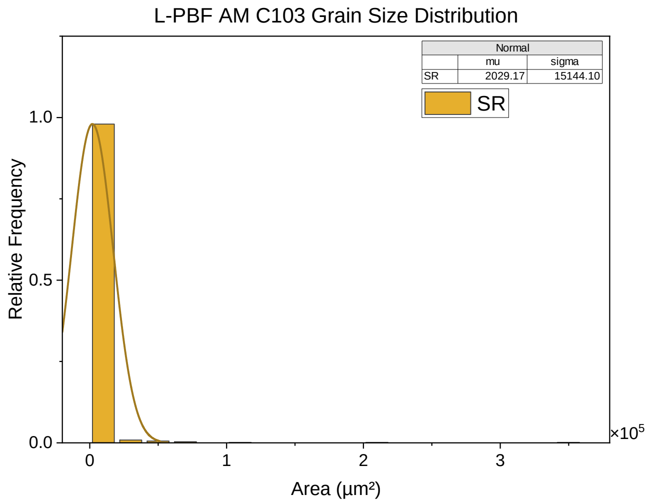


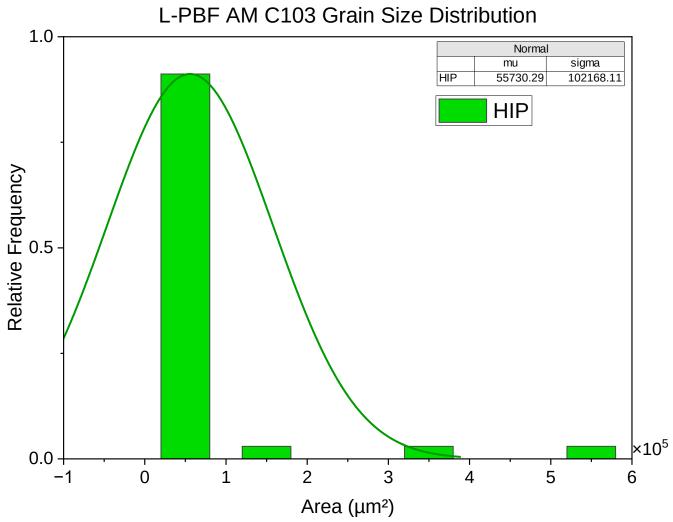


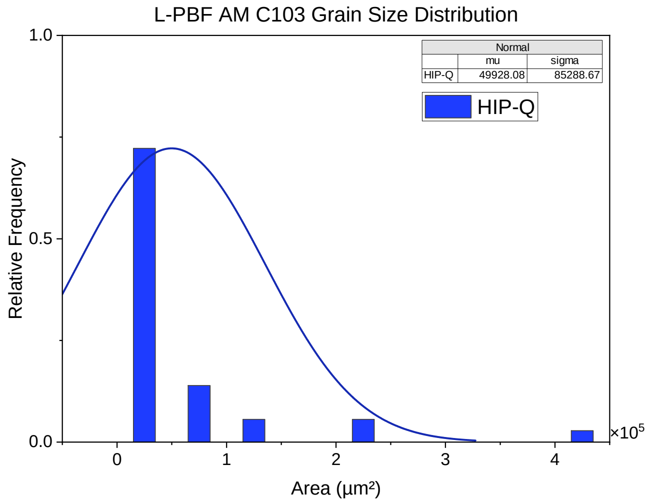


Average Grain Size: 49928.08 µm²

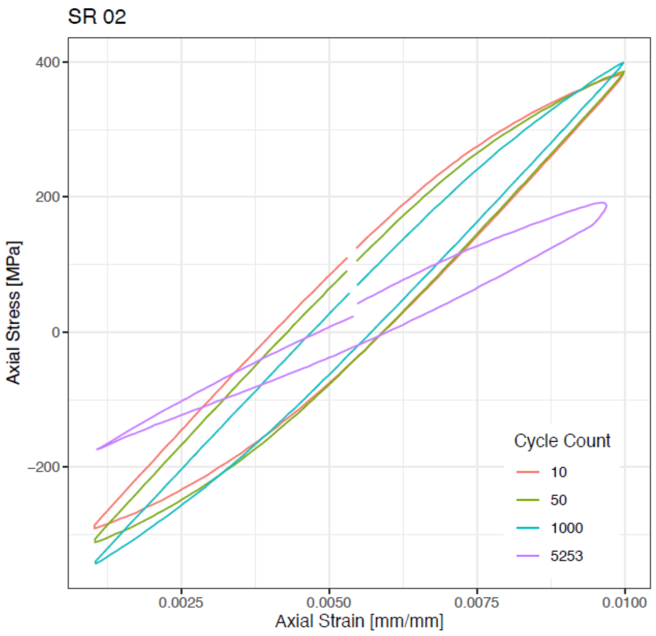

L-PBF AM C103 Grain Size Distribution
Normal
mu
sigma
AB 1803.82 7255.57
AB
Relative Frequency
Area (µm²)
×10^5
0.0
0.5
1.0
0
1

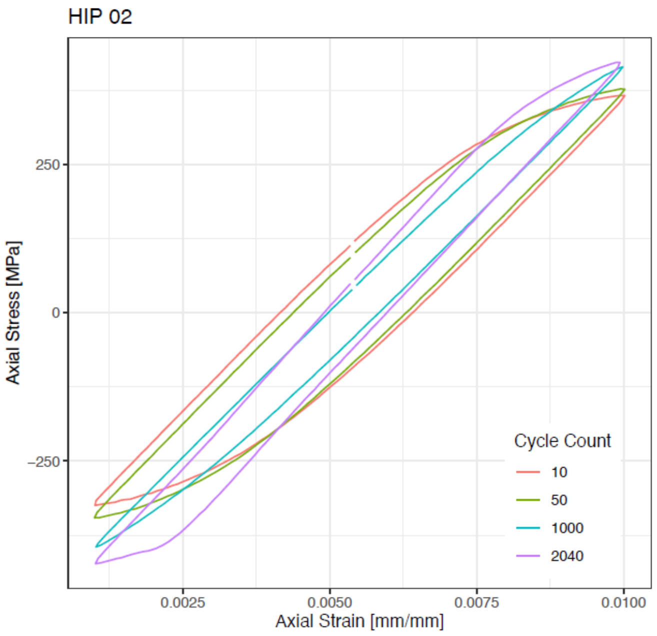

L-PBF AM C103 Grain Size Distribution
Normal
mu
sigma
SR 2029.17 15144.10
SR
Relative Frequency
Area (µm²)
×10^5
0.0
0.5
1.0
0
1
2
3

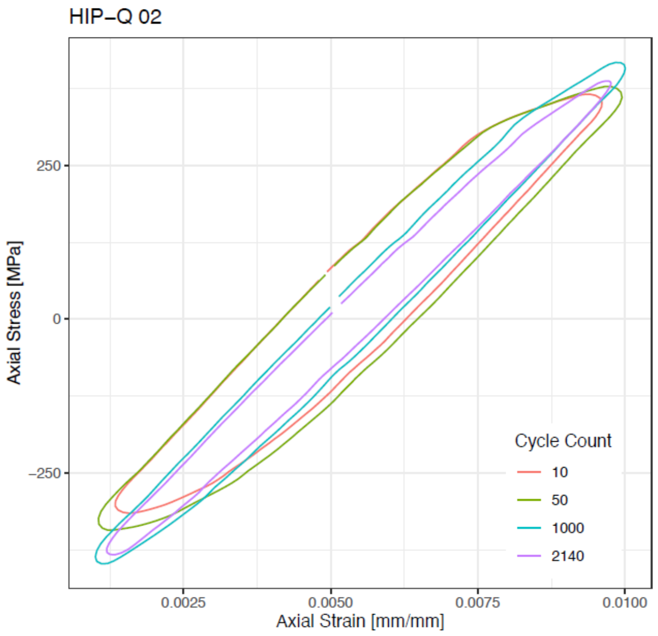

L-PBF AM C103 Grain Size Distribution
Normal
mu
sigma
HIP 55730.29 102168.11
HIP
Relative Frequency
Area (µm²)
×10^5
0.0
0.5
1.0
−1
0
1
2
3
4
5
6


L-PBF AM C103 Grain Size Distribution
Normal
mu
sigma
HIP-Q 49928.08 85288.67
HIP-Q
Relative Frequency
Area (µm²)
×10^5
0.0
0.5
1.0
0
1
2
3
4

Comparisons - LCF







HT	Max Stress [MPa]	First Stress [MPa]	HR
SR	404.791	391.606	0.034 ± 0.009
HIP	410.915	367.111	0.119 ± 0.040
HIP - Q	418.189	370.247	0.130± 0.008