Evaluation of Additively Manufactured Candidate Alloys for Hydrogen Turbine Fuel Injectors

NATIONAL ENERGY TECHNOLOGY LABORATORY

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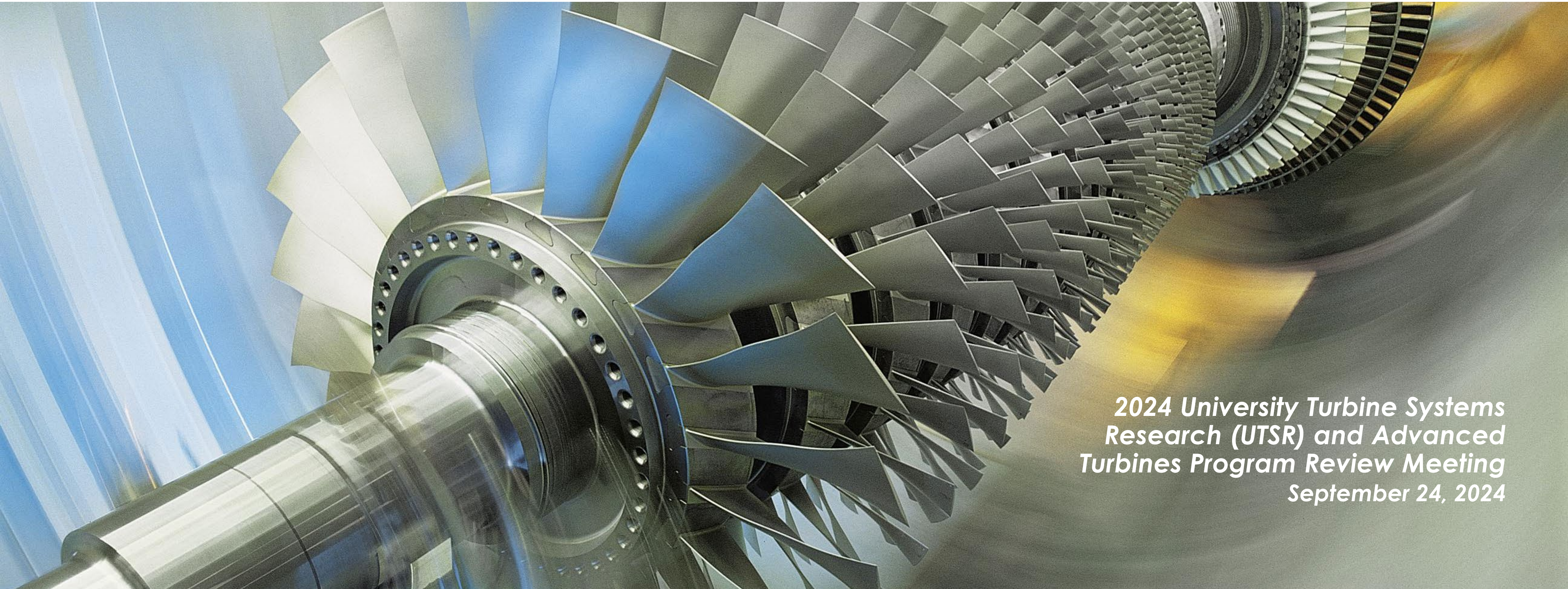

2024 University Turbine Systems Research (UTSR) and Advanced Turbines Program Review Meeting
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Acknowledgements

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Motivation & Approach

<u>Hydrogen turbine technology</u>: Hydrogen and hydrogen/natural gas blends are being pursued to reduce emissions and improve engine operating efficiency

<u>Laser powder bed fusion (L-PBF) fabrication</u>: Design of fuel injectors with cooling passages & fuel channels for both premixed and non-premixed gas turbine combustion systems

Goal: Document AM candidates in operation ranges that are undocumented and relevant to hydrogen service

Approach: Property-Microstructure Evaluation L-PBF Ni-Superalloy for Industrial Gas Turbine Fuel Injectors

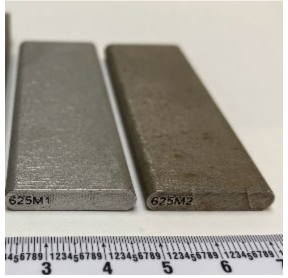
- Alloys**:** Solid Solution (IN625), γ' Precipitate (Haynes 230), γ'/γ''/δ Precipitate (IN 718) strengthen
- Compare L-PBFproperties to wrought properties
- Screen the **<u>tensile, creep, and fatigue</u>** properties up to 815 °C in air.
  - Porosity / defects - Location specific microstructure – Impact of minor phases
  - Failure mechanisms with fractography, cross-section analysis, and TEM
- **<u>Assess hydrogen embrittlement (HE) susceptibility</u>** using slow strain-rate tensile testing
  - Ex-situ electrochemically charged and then test to failure
  - Extend to in-situ testing, examine elevate temperature hydrogen attack and damage
- Screen materials behavior under conditions that mimic service
  - Coupon exposure various fuel environments at elevated temperature, pressure, and H_2O vapor
  - Capture prior thermal history& assess impact on select properties

On-going & future work

Laser Powder Bed Fusion (L-PBF) Processing

- IN625, IN718, Haynes 282
- **L-PBF printing with Ar gas-atomized powders**
  - Siemens
  - **EOS M290 machine**
  - Optimized parameters consistent with EOS specifications,
  - Bidirectional scan strategy
  - 40 μm layer thickness
  - Vertical Z-direction test bars and blanks in the build direction
  - Stress Relief on the build plate
  - Heat treated to industrial practice.

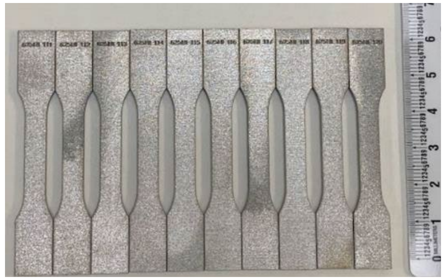
(1) Met. as-printed
(1) Met. solutioned



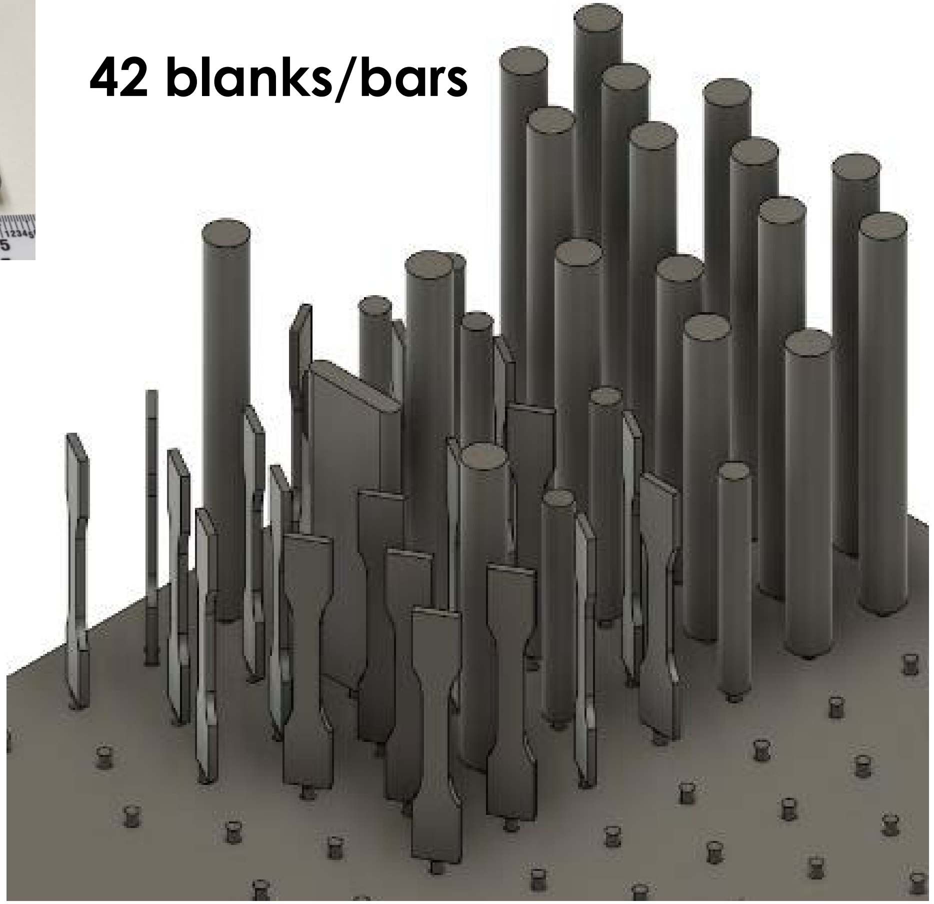
(5) Tensile / Creep Z-blanks

(13) Oversized Z-blanks

(20) Z-dog bones HE Tests



42 blanks/bars



Materials & Microstructures

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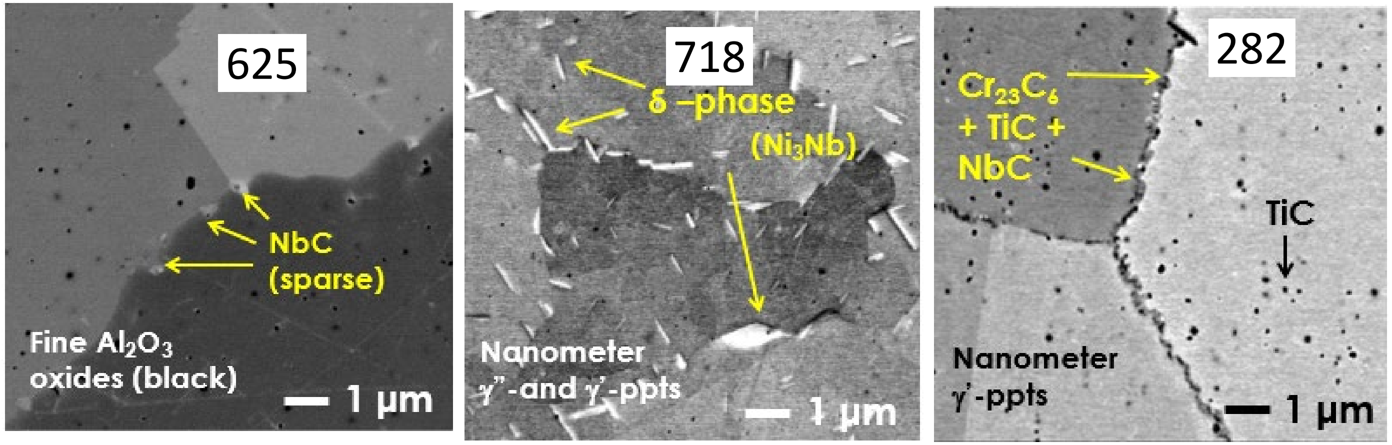
Solid Solution vs. γ'/γ''/δ Precipitate vs. γ' Precipitate Strengthen



WDXRF/LECO Measured Compositions on L-PBF Material

Wt.%	Ni	Cr	Mo	Nb	Fe	Ti	Al	Co	C
L-PBF 625	61.5	21.3	9.1	3.7	4.0	0.08	0.06	0.08	0.0124
L-PBF 718	52.9	18.7	3.1	5.16	18.3	0.96	0.48	0.21	0.0525
L-PBF H282	58.1	19.2	8.8	0.05	0.11	2.12	1.22	10.3	0.0459

Composition Compared to Specifications

Carbon specification

Low C content L-PBF 625

Wt.%	C
625	**0.10 max**
718	**0.08 max**
H282	**0.06**

Heat Treatment:

- Stress-relieved on the build plate
- **L-PBF 625 (no HIP)**: Solution heat treatment at 1175 °C for 1 hour.
  As-printed porosity measured to be 0.04 + 0.02 % → **HIP does not appear necessary**
- **L-PBF 718 (w/HIP)**: Modified AMS 5663 specification schedule
  Solutioning at ~980 °C for 1 hour (**below δ-solvus**) + similar holds/temps for 2-step aging
- **L-PBF 282 (w/HIP)**:
  Two-step solutioning above 1150 °C with 1 hour holds each
  Standard single step aging 788 °C for 8 hours

Alloy specifications
https://www.specialmetals.com/documents/technical-bulletins/inconel/inconel-alloy-718.pdf
https://www.specialmetals.com/documents/technical-bulletins/inconel/inconel-alloy-625.pd
https://haynesintl.com/en/datasheet/haynes-282-alloy/#alloy-brochuref

Grain structure after heat treatment

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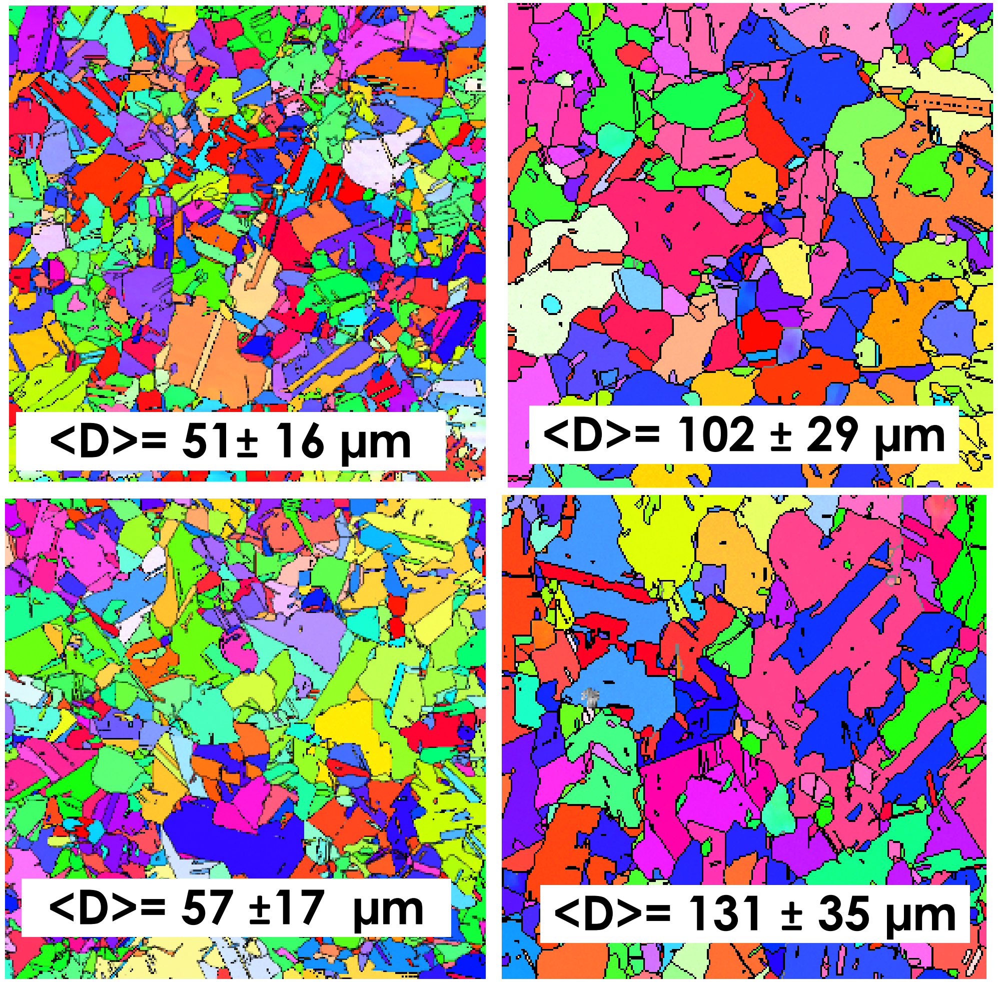
Near equiaxed: ***Grains recrystallize***

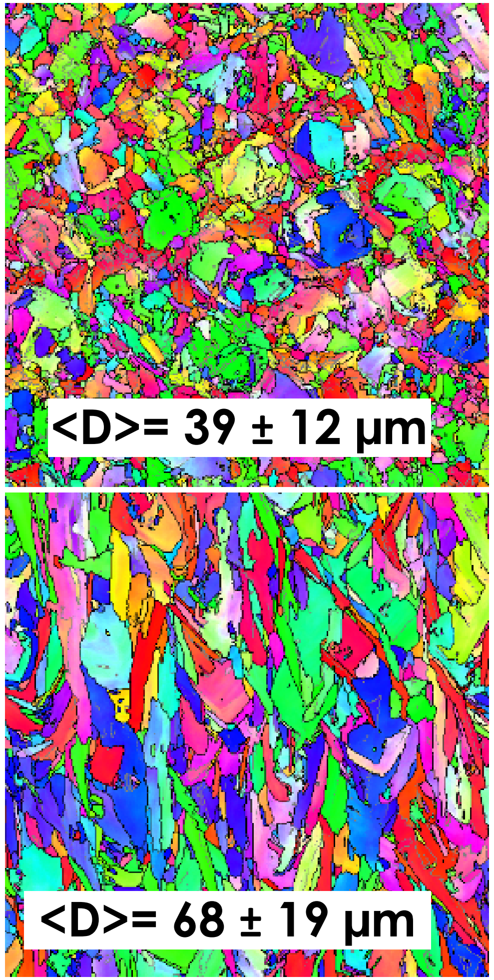
625 **282**

In-Plane (XY)

Along Build Direction (XZ)

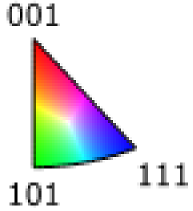


718



Elongated like As-printed:

Grains restrained by δ-ppts at GBs



200 µm

Mechanical Tests

L-PBF 625, 718, 282 property tested in fully heat-treated condition

Three tensile tests (E8/E21)

- **Room Temp, 650°C, 750°C**
- 1 test each at 2.17x 10^{-3} mm/s to1.2%, then 2.17 x 10^{-2} mm/s thereafter

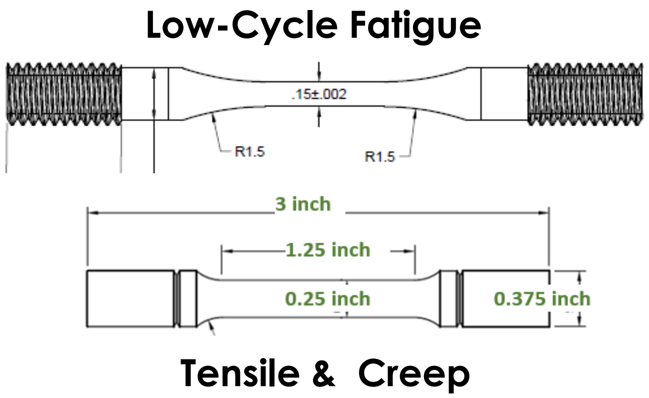
Four creep rupture tests (E139)

- **650 – 815 °C / 100 – 600 MPa**

Eleven strain-controlled low cycle-fatigue tests

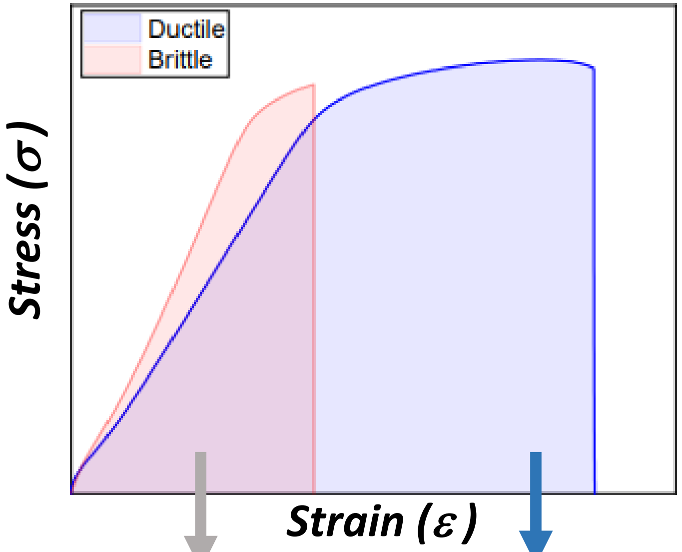
- **650 °C**
- Strain range vs. Fatigue Life (N_f) curve (S-N curve)
- R= 0.05, f= 0.2 Hz for strain range up to 1.5%

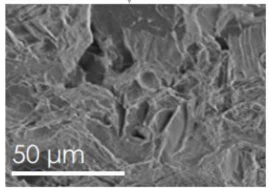
Machined from Z-Blanks



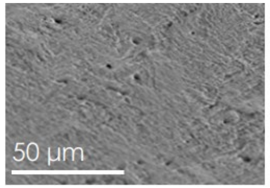
Hydrogen embrittlement – 5 slow strain tensile tests at RT

- Surfaces milled to 600 grit finish - good electrical contact for charging.
- **Ex-situ H_2 charging** at 1mA/cm² in 0.1 M H_2SO_4 with +1 g/L CH_4N_2S for 72 h
- Test to failure using **6.3×10^{-6} s^{-1}** strain rate





Brittle fracture

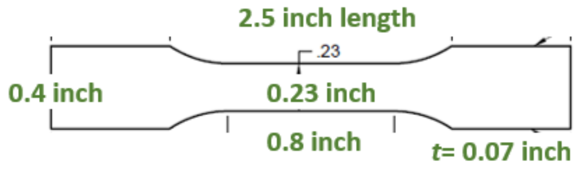


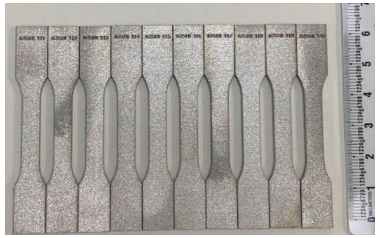
Ductile fracture

$$HE_{index} = \frac{\varepsilon_{AR} - \varepsilon_{HE}}{\varepsilon_{AR}}$$

AR = As-received condition (no charging)
HE= Charged condition

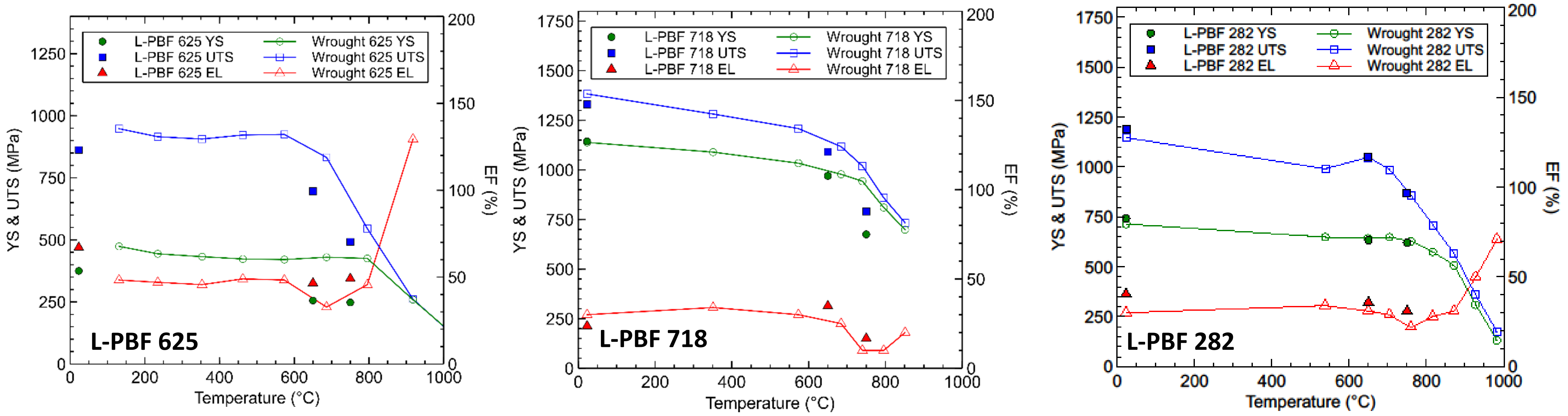
Near-net shape Z- dog bones





Tensile Properties

Room temperature, 650 °C, and 750 °C



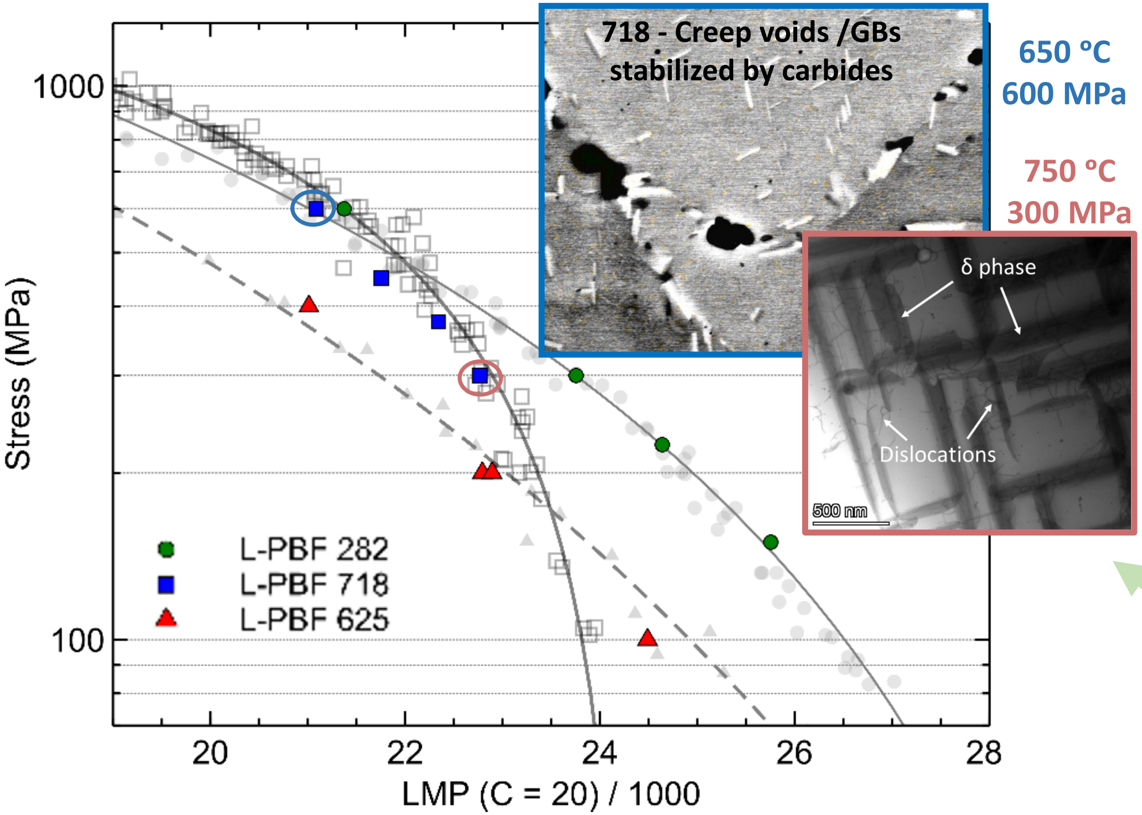
Compared to wrought product:

- **All L-PBF alloys** shows modestly **higher ductilities** over the temperature range
- **L-PBF 625/718** Drop-off in **UTS** shifted to lower temperatures by ~50 °C ← ***refined grain structures***
- **L-PBF 625** **yield strength** significantly lower, possibly due to124 ppm C content → **fewer MC carbides**
- **L-PBF 282** has very comparable **UTS** and **yield-strength** ← ***similar grain structure to wrought***

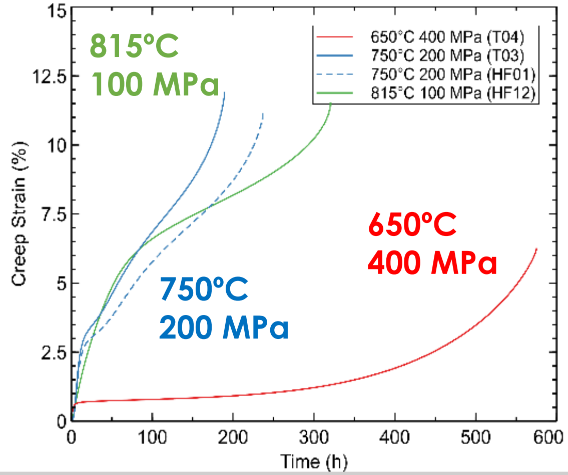
Creep Behavior

Investigation of deformation behavior and failure modes is underway

Larson-Miller Plot: All Alloys Compared

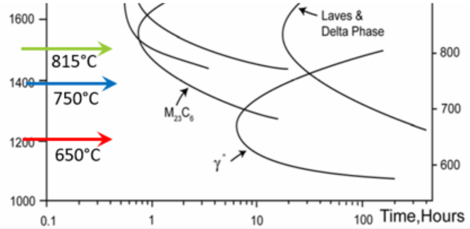


- **All three alloys perform like wrought product**
- **L-PBF 718** is within statistical deviation; however, on the lower side → likely a result of fine, restrained grains



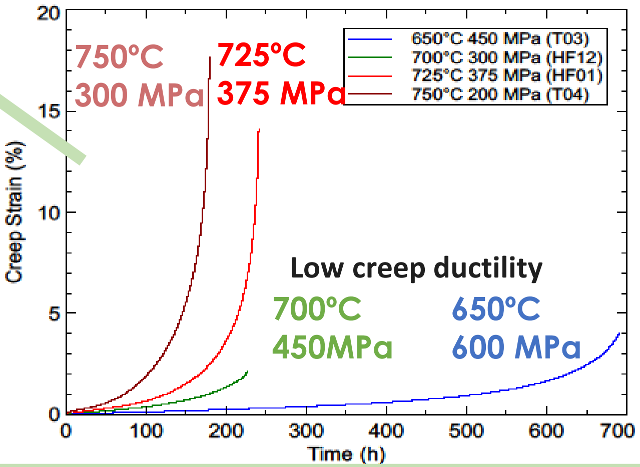
- Double minima for 750°C and 815°C creep curves
  - 1st minimum γ” emerges
  - 2nd minimum δ emerges

L-PBF 625

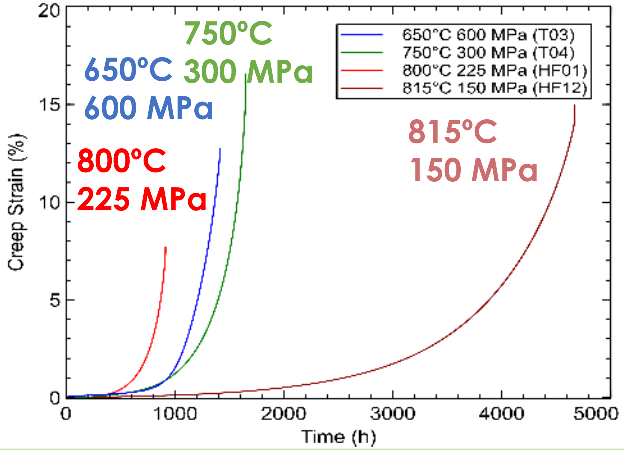


Well-defined primary, secondary and tertiary stages

L-PBF 718



L-PBF 282



Strain-Controlled Low Cycle-Fatigue

Detail examination of fracture surfaces complete; microstructural linkage and evaluation of hysteresis planned

Similar performance to wrought alloys

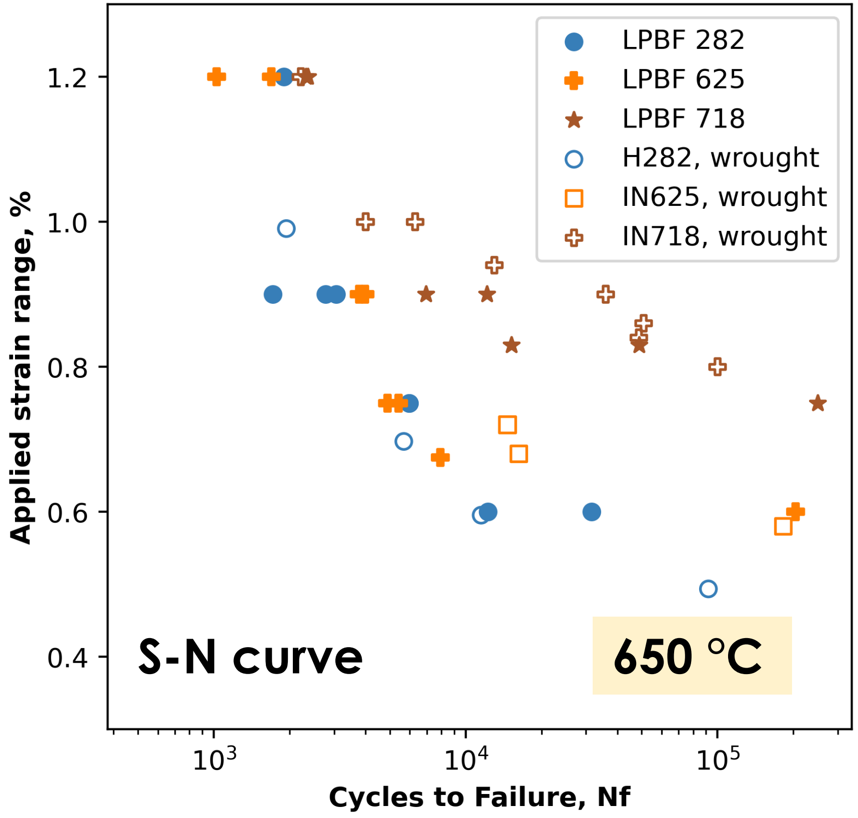
- L-PBF 718 somewhat lower

For L-PBF 625

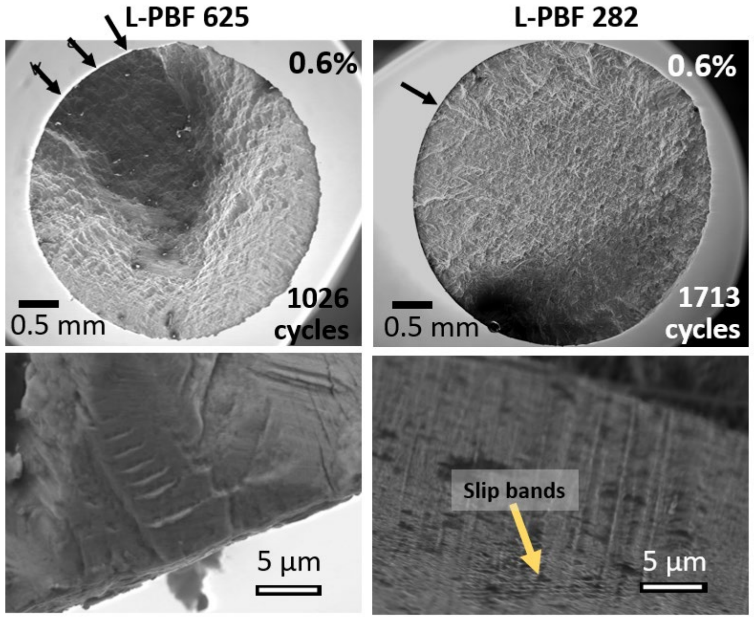
- Low applied strain ranges show stage one crystallographic initiation
- High applied strain ranges show initiations at dislocation egress from the surface

Further work on other alloys and hysteresis underway

L-PBF (R= 0.05) vs Wrought (R =-1)



Crack initiation and propagation

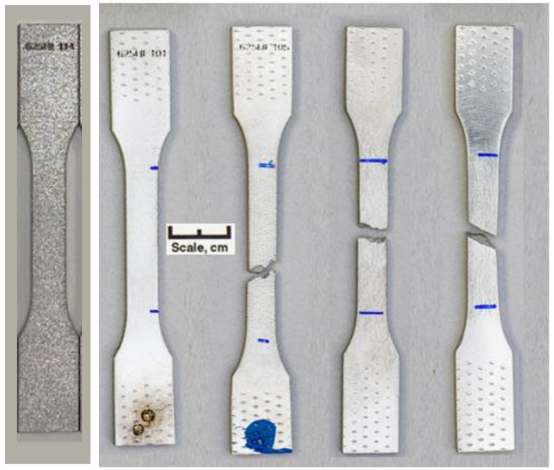


Slow strain-rate testing to screen H_2-embrittlement (HE)

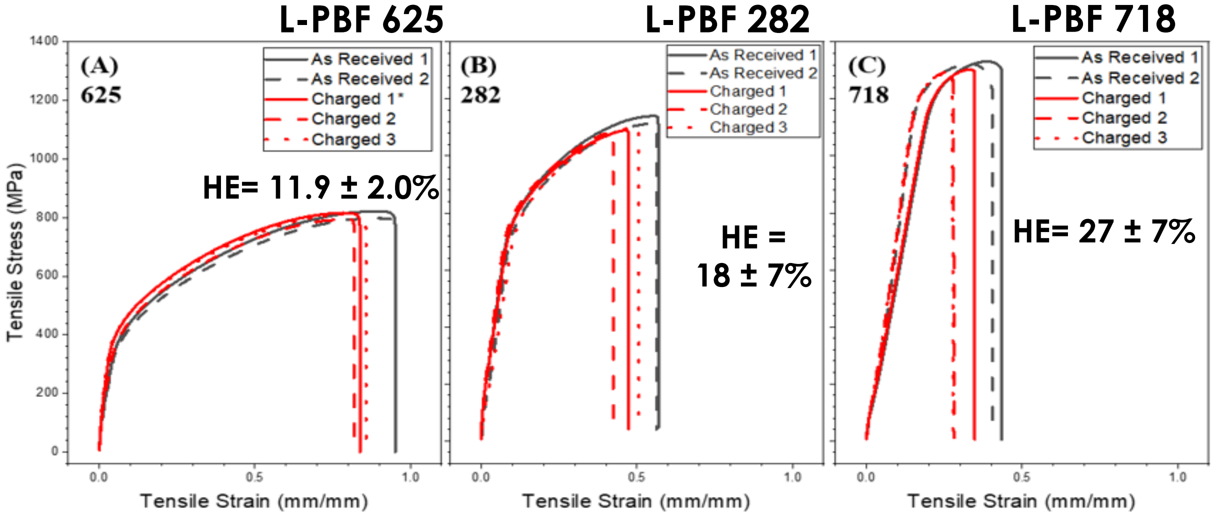
2 Tests on Uncharged / 3 Tests on Charged Test Bars

Ex-situ H_2 charging

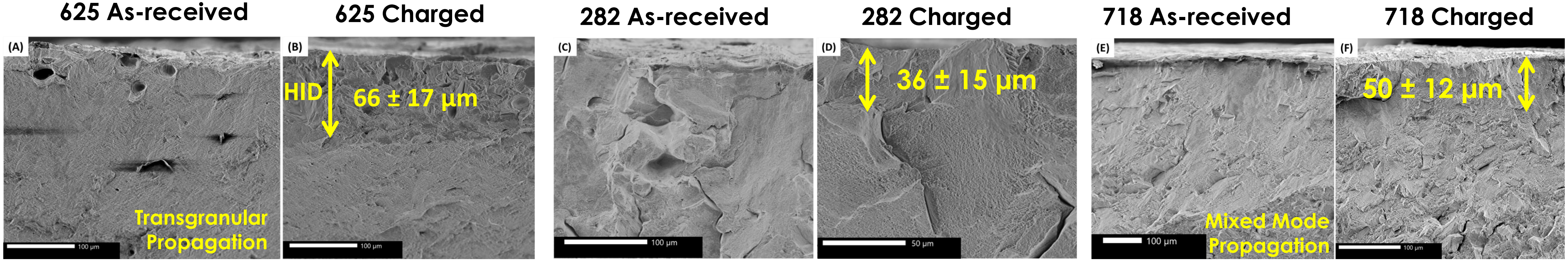
- Largest hydrogen ingress depth (HID) for L-PBF 625
- Brittle features in HID for all three alloys
- Brittle cleavage extends past HID in L-PBF 718



HE Index
L-PBF 718 (most HE) > 282 > 625 (least HE)



Edge of fracture surfaces:



Recrystallized grains after FHT — Restrained grains

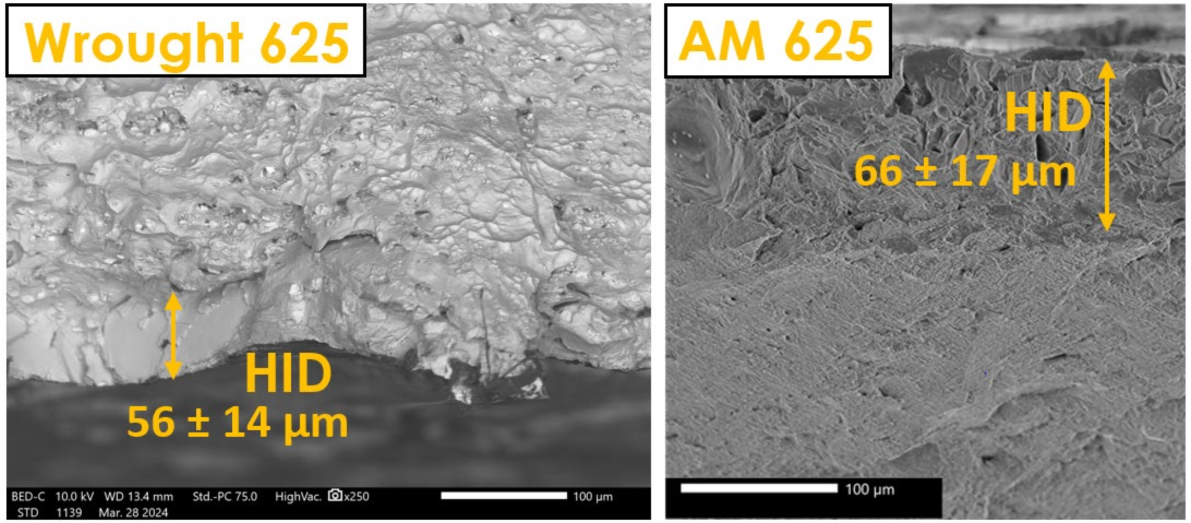
L. Teeter, M. Detrois, K. Rozman, and C.K. Sudbrack, *Oxidation and Hydrogen Embrittlement Behavior of Several Additively Manufactured Ni-Based Superalloys*. AMPP 2024-21117, New Orleans, LA, (2024)

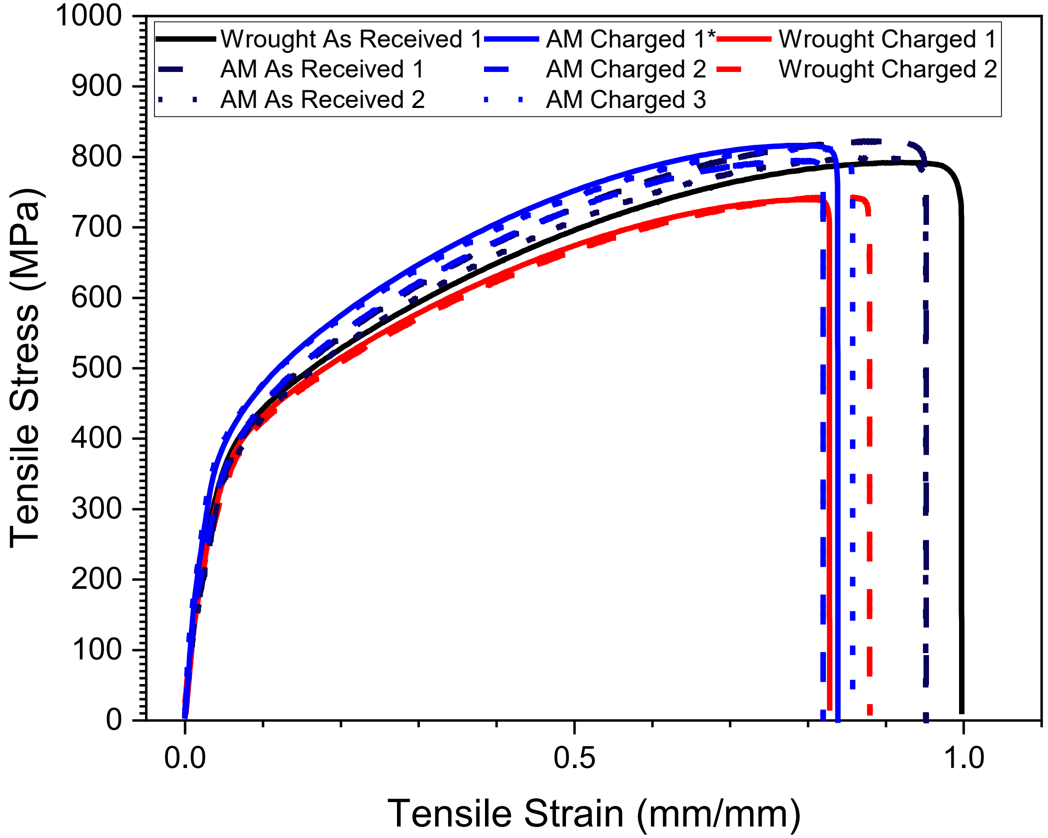
Wrought Alloy Comparison

Hydrogen Embrittlement and Fractography

- AM and wrought behavior is similar; however, AM 625 does seem to experience slightly reduced mechanical degradation in comparison to the wrought 625.
- Brittle features on both samples inside HID. HID slightly larger for AM 625

Material	HE Index
Wrought IN625	14.4% ± 3.7%
AM IN625	11.9% ± 2.0%

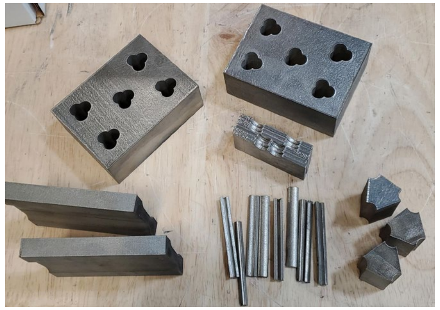




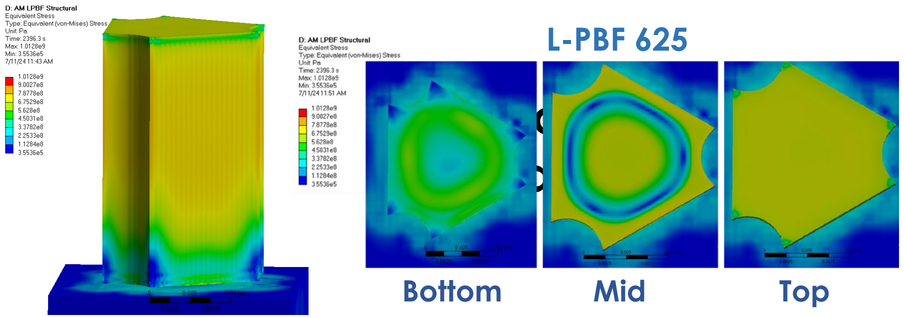
Geometric Stress Concentration Effect on AM Parts Performance

Objective: Understand the effect of geometric residual stress on the materials-hydrogen interactions and performance of fuel injector candidates

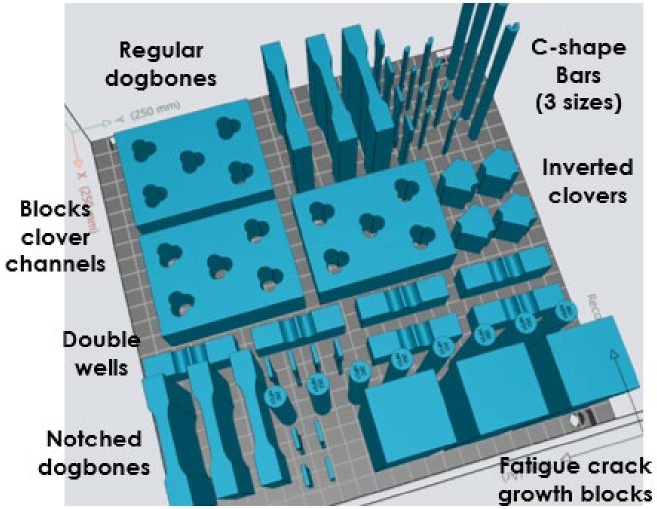
First Build Set Delivered



Initial predictions of residual stress distribution



*Lab-scale articles that capture realistic injector features: **54 pieces per alloy***



Highlights:

- ✓ **Continued printing partnership** with Siemens Energy
- ✓ **Designed lab-scale articles** with realistic injector geometric features **and completed printing of first alloy**, L-PBF 625
- ✓ **Completed initial residual stress simulations with ANSYS** of select lab-scale articles
- ✓ **Successfully awarded beam time** under am ORNL Neutron Sciences General User Proposal to measure residual stress using Spallation Neutron Scattering (SNS)

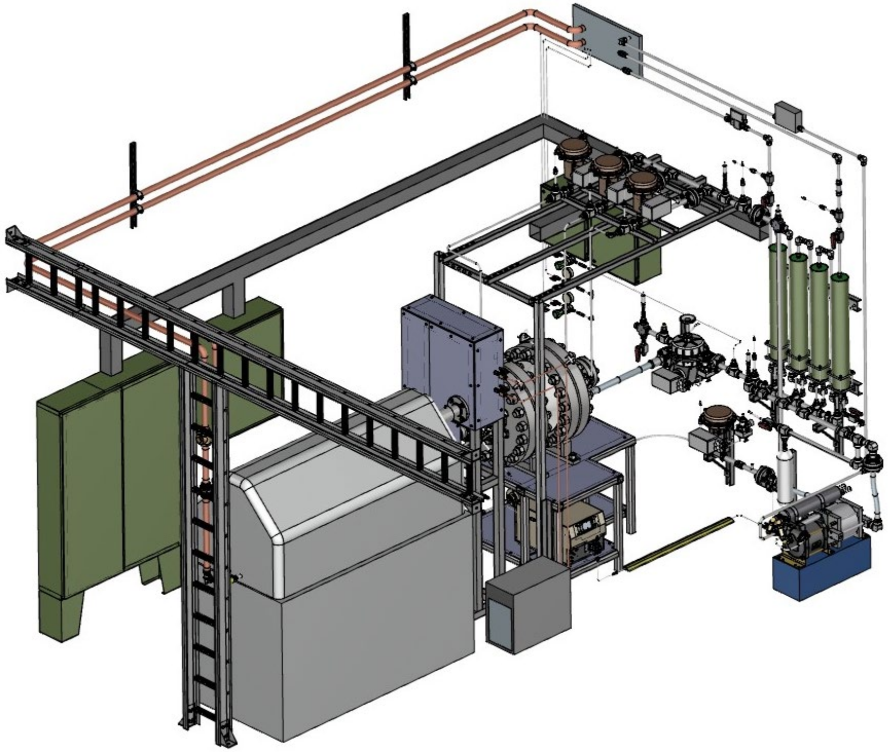
Next steps:

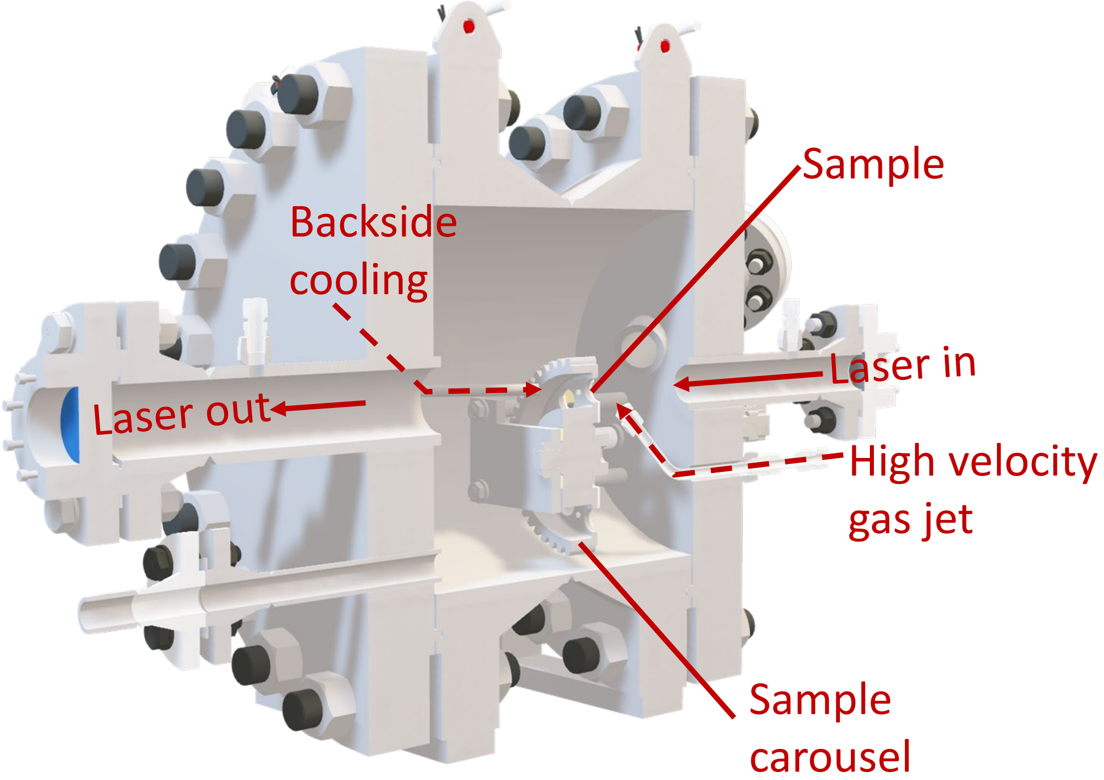
- **Measure location-specific residual stress of selected geometries using neutron scattering**
- **Assess the impact of stress concentration on H_2-damage and performance debits**

Gas Turbine Combustion Simulation Rig

NETL-Research and Innovation Capabilities

Environmental performance testing of T/EBCs, CMCs, and other high temperature materials





- ✓ Ultrahigh surface temperatures
- ✓ High gas velocity
- ✓ High pressure
- ✓ Complex gas mixtures
- ✓ Backside cooling (thermal gradient)
- ✓ Long exposure times (unattended operations)

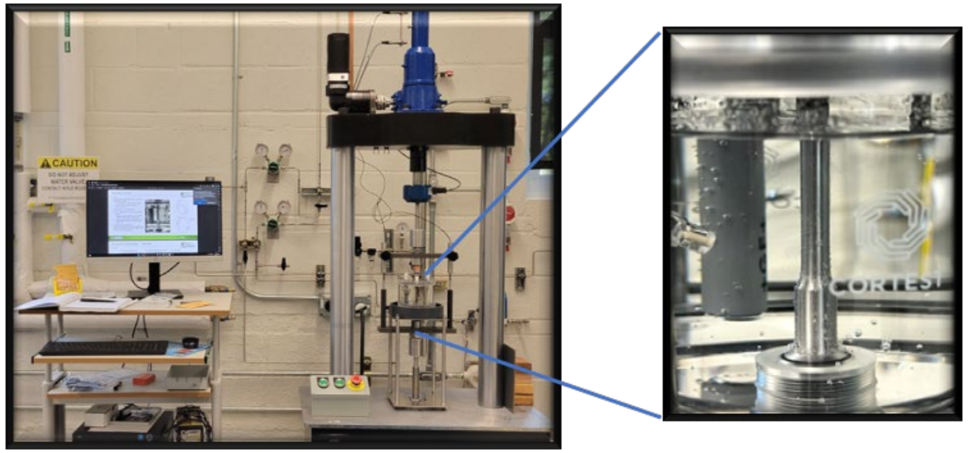
→ **Realistic gas turbine environments**

Materials Performance in H_2

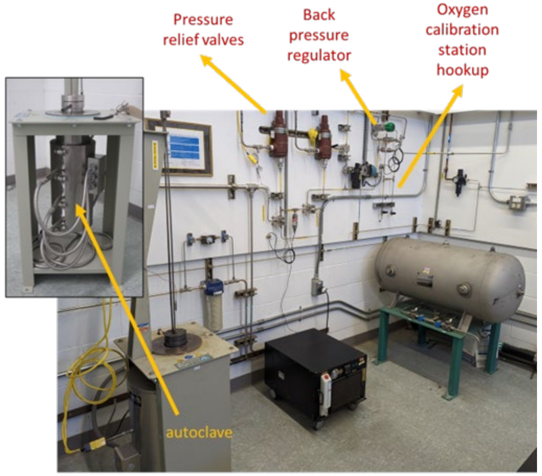
NETL-Research and Innovation Capabilities

- **Fatigue Crack Growth**
  - Tests can be performed in aqueous electrolyte or in pure hydrogen gas
  - Autoclave pressures up to 1500 psi
  - Autoclave temperatures up to 288°C
- **Slow strain rate testing**
  - In-situ (loading while charging)
- **Creep Testing**
  - Ar-2.8% H_2 Up to 1200°C
  - 100% H_2 future
- **Hydrogen autoclave for gaseous pre-charging**
  - H_2 (pure) (600 °C / 1600 psi)
- **Hydrogen Permeation**
  - Devanathan – Stachurski cell
  - Hydrogen gas permeation
- **Hydrogen absorption / desorption**
  - Scanning Electrochemical Microscopy
- **Analytical Capabilities**
  - Thermal Desorption Mass Spectrometry
  - Hydrogen microprinting

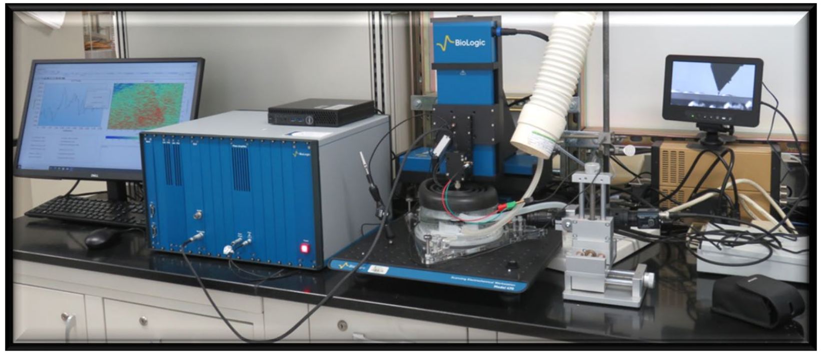
Slow Strain Rate Testing



Fatigue Crack Growth



Hydrogen autoclave for pre-charging



Scanning Electrochemical Microscopy

Alloy Fabrication Current Capabilities

AASC Advanced Alloy Signature Center

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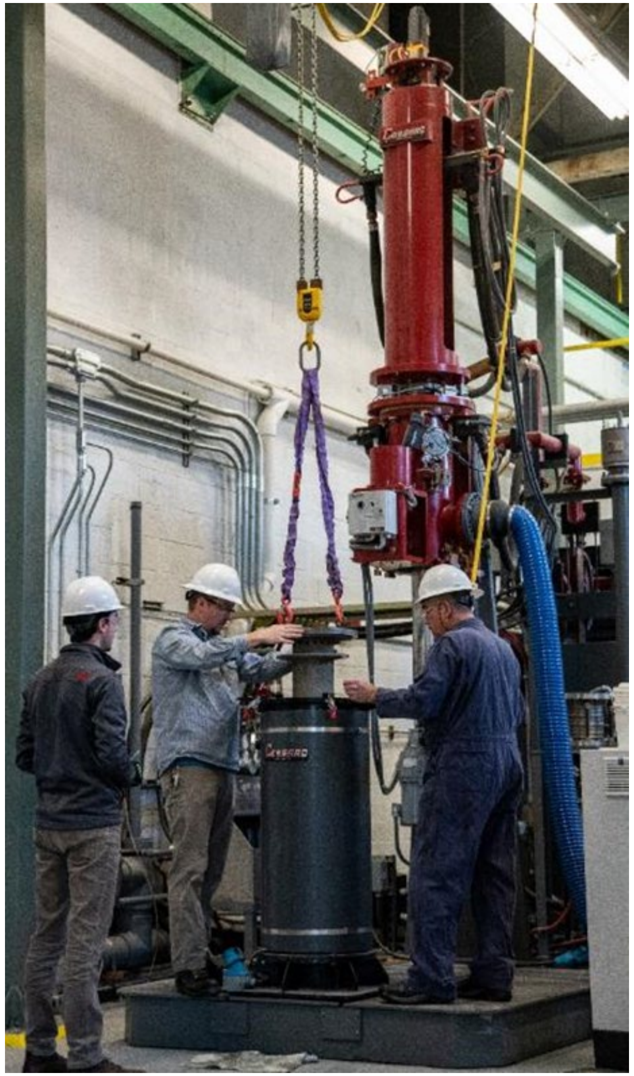
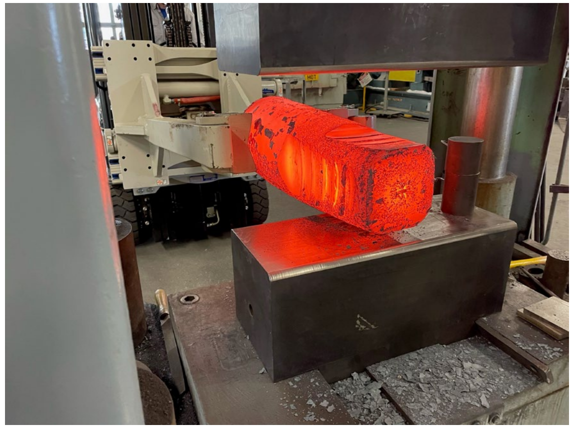
NETL-Research and Innovation Capabilities



Melt Processing Capabilities
- Air Induction Melting: up to 300 lbs
- VIM: 15, 50 and 500 lbs
- Vacuum Arc Remelt/Electro-Slag Remelt 3-to-8-inch diameter ingots

Thermo-Mechanical Processing Capabilities
- Heat-treatment furnaces:1650ºC, inert atmospheres and controlled cooling.
- Press Forge: 500 Ton
- Roll mills: 2 and 4 high configurations.



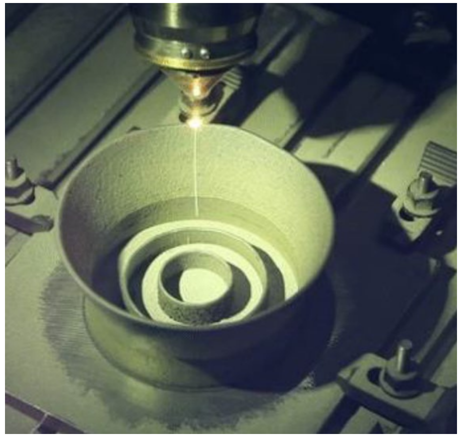
Alloy Fabrication Future Capabilities

NETL-Research and Innovation Capabilities

Melt Processing Capabilities
- Alloy Development Research Building, completion in 2026.
- Enhanced melt processing capabilities for high temperature and ultra-high temperature alloys.
- Operational Fall 2026.

Thermo-Mechanical Processing Capabilities
- Operational in 2025/2026.
- 1500 Ton Press Forge
- 800 Ton Extrusion Press
- Wire Drawing Equipment
  - ★ Experimental wire-based/solid feedstocks for additive manufacturing

Additive Manufacturing Capabilities
- Operational in 2025
- Lased Direct Energy Deposition Dual Wire/Powder Feed Tool



Summary & Concluding Remarks

Good progress towards screening the tensile, creep, and fatigue properties up to 815 °C in air and the native hydrogen embrittlement susceptibility at room temperature.

Examination of L-PBF superalloy candidates revealed:

- **Microstructure**: Precipitate & carbide phases observed are consistent with conventional alloys after selected heat treatments. L-PBF 625 and 282 show near equiaxed grain structure
  (1) Fine Al_2O_3 oxide inclusions in L-PBF 625, which are highly stable with no noticeable impact on properties
  (2) Grain boundary stabilization with densely distributed δ-precipitates along grain boundaries in L-PBF 718
- **Tensile behavior** is consistent with wrought, particularly L-PBF 282 behavior, while L-PBF 625 & 718 showed modest differences, indicating good potential to apply within IGT.
- **Creep behavior studied (650 – 815 °C / 100 – 600 MPa)**: L-PBF alloys performed consistently with reports for wrought counterparts, with L-PBF 718 on lower end statistically, likely due to refined grain structure.
- **LCF behavior at 650 °C**: S-N curve comparison with literature data looks promising.
- **Hydrogen embrittlement**: As-received L-PBF 625 is the least prone to hydrogen embrittlement, while L-PBF 718 is the most prone

Thank you for your attention! Follow up questions: chantal.sudbrack@netl.doe.gov