

# **NIST Additive Manufacturing Fatigue and Fracture Project: Research Highlights**

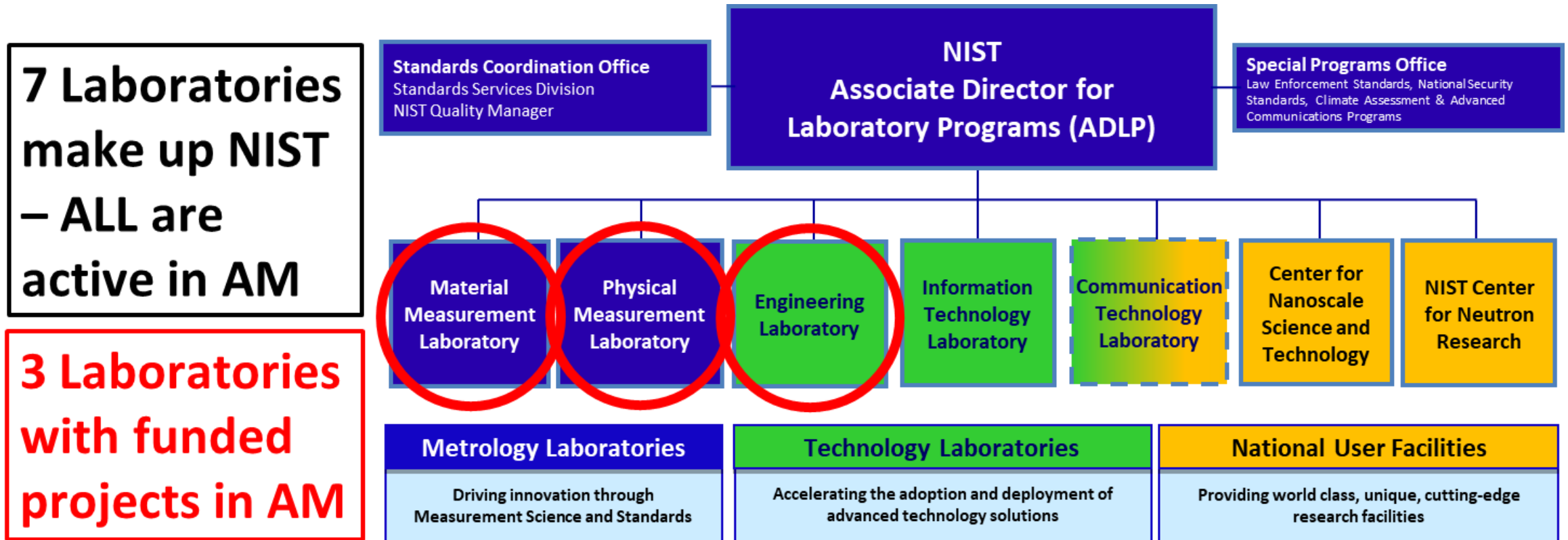
Nik Hrabe, Ward Johnson, Jake Benzing, Nicholas Derimow, Newell Moser,  
Orion Kafka, Alec Saville

National Institute of Standards and Technology (NIST), Boulder, CO, USA



# Introduction

- NIST is a national laboratory within the US Department of Commerce
- **MISSION:** To promote U.S. innovation and industrial competitiveness by advancing measurement science, standards and technology in ways that enhance economic security and improve our quality of life.



# Introduction

**MML**

**Lyle Levine – Metal Program Lead**  
**Igor Levin – Ceramics Program Lead**  
**Kalman Migler – Polymer Program Lead**

**Phase Evolution and Corrosion**

Mark Stoudt

IN625 delta phase

**Fatigue and Fracture**

Nik Hrabe

Small-scale mech. testing

**Alloy Development**

Carelyn Campbell

Co-based AM alloys

**X-ray CT**

Ed Garboczi

Powder/Pore shape

**In Situ Nanomechanics**

Jason Killgore

Atomic Force Microscopy

**Process Modeling**

Jon Guyer

CHiMaD

Phase Field/FEM

**Acoustic NDE**

Ward Johnson

**Synchrotron X-ray**

Fan Zhang

Full stress tensors

**Extrusion Process**

Russell Maier

**Cold Sintering Process**

Andrew Allen

**Process Monitoring & Control – Polymer**

Jonathan Seppala

**EL**

**Shawn Moylan – Measurement Science for AM Program Lead (Metals)**

**Feedstock and Machine Qualification**

Shawn Moylan

**In Situ Monitoring**

Brandon Lane

**Data for Model Validation**

Thien Phan

**Data Management**

Yan Lu

AM Materials Database

**Part Qualification**

Jason Fox

Surface roughness, XCT, NDE, material properties

**AM Control**

Ho Yeung

AM Metrology Testbed (AMMT)

**Data-Driven Decision Making for AM**

Paul Witherell

**3D Printed Concrete**

Scott Jones

**PML**

**Thermal Emittance of Melts and Powders**

Sergey Mekhontsev

AMMT - Emissivity

**In Situ Metrology**

Brian Simonds

Energy Coupling and Spectroscopy

# Additive Manufacturing Fatigue and Fracture Project Summary

## Problem Statement

- Metal additive manufacturing (AM) is not used in fatigue and fracture critical applications despite industrial need

## Goal

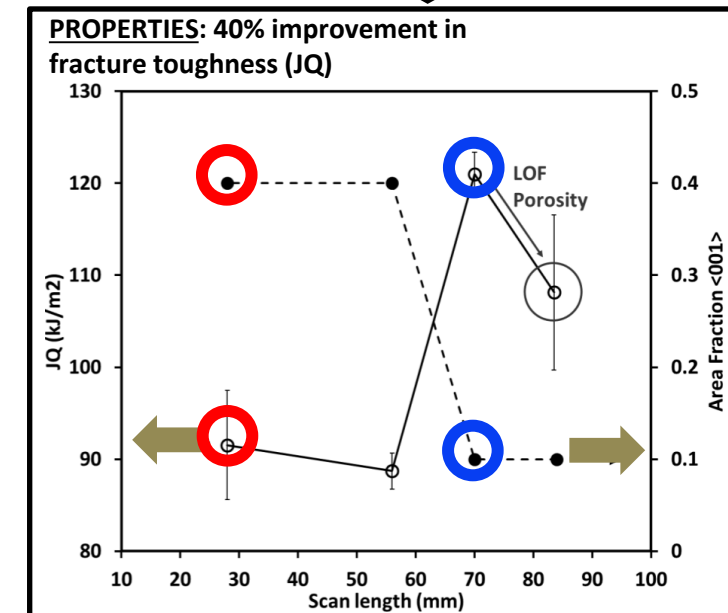
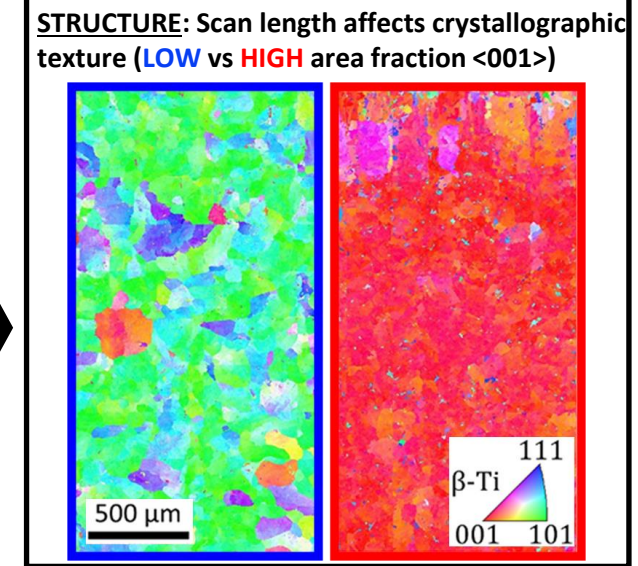
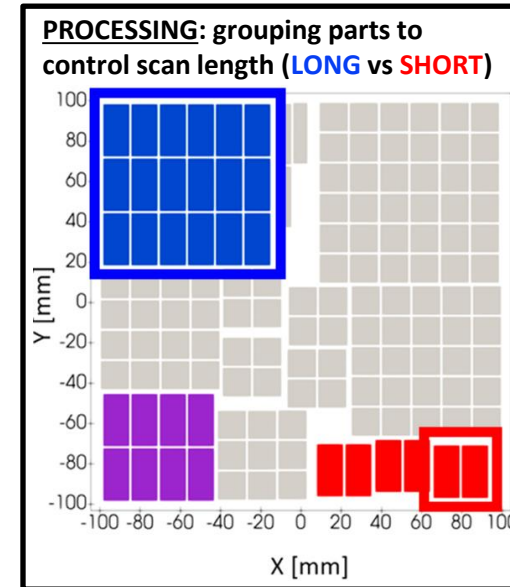
- Enable confident use of metal AM in critical applications through reducing material variability, improving performance, reducing qualification cost, developing appropriate part inspection techniques, and developing necessary standards

## Methodology

- Develop/advance metrological practice for AM-specific performance metrics encompassing the full processing-structure-properties-performance spectrum and the full AM lifecycle
- Develop process and post-process control methods to reduce material variability and improve performance
- Develop new non-destructive evaluation (NDE) techniques that are fast, inexpensive, precise, and capable of inspecting larger parts with complex geometries common in AM
- Develop a rapid qualification framework based on digital twin to reduce cost and time to production
- Develop consensus AM standards with key stakeholders and various standards development organizations

## Key Findings and Significance

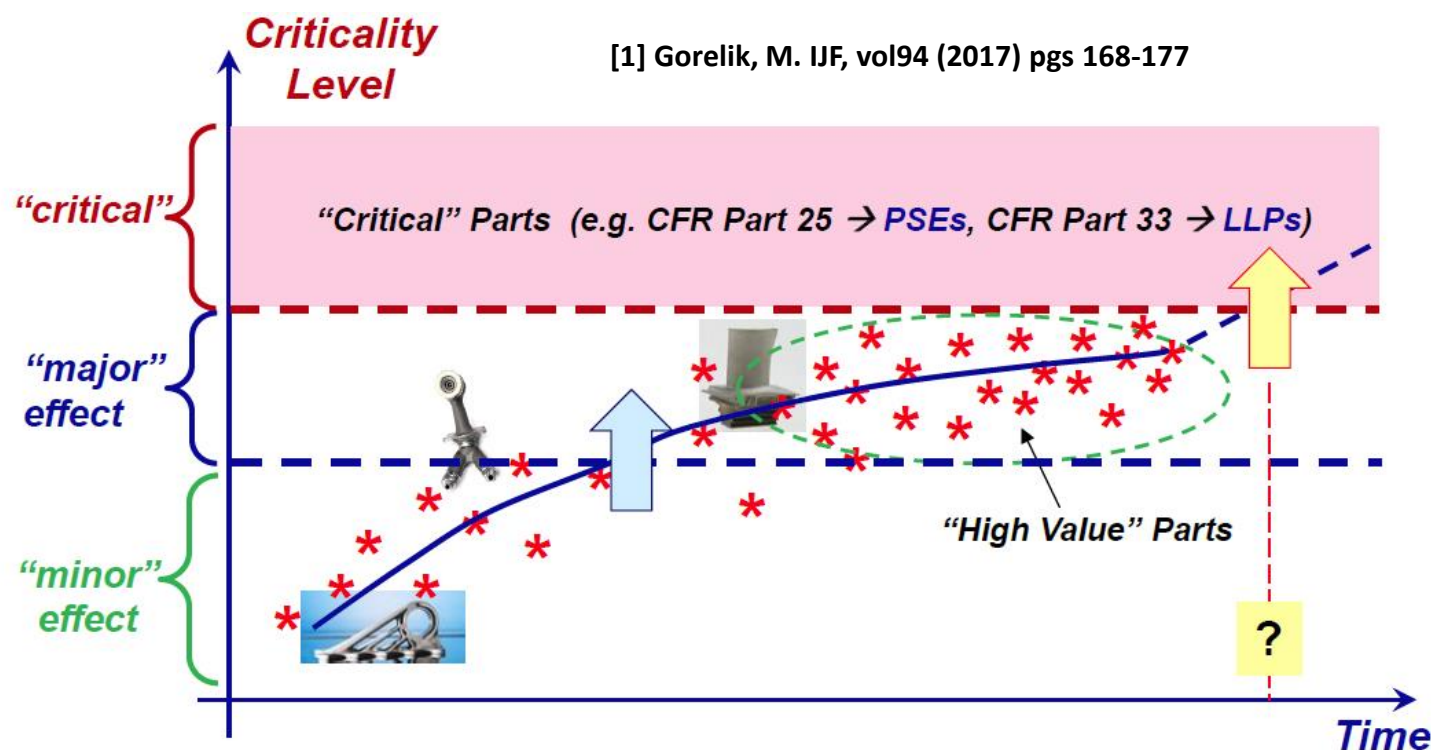
- Organized workshop that has grown into ASTM International Conference on Advanced Manufacturing, with more than 1,000 annual attendees
- Created NIST Metal AM Powder Consortium, with 11 members as of 2024
- Developed new post-process powder recovery blasting control technique for AM titanium that led to 30x improvement in fatigue lifetime and first-time use in the most critical medical device application (Collaborator: Stryker)
- Developed new process powder reuse quarantining method for AM titanium that led to 15% lower chemistry heterogeneity and part-to-part strength variability and standard ASTM F3592 (Collaborators: UTEP, Sandia)
- **Developed new process-based technique to control crystallographic texture for AM titanium, leading to 40% improvement in fracture toughness and standard ISO/ASTM 52911-3 (Collaborator: AFRL)**
- Developed new post-process heat treatment for AM titanium leading to 100x improvement in fatigue lifetime and revision of standard AMS 7028 (Collaborators: Lynntech, Quintus, 3D Systems)
- Developed new post-process heat treatment for AM Inconel 718 leading to 65% reduction in heat treatment time and 10x improvement in fatigue lifetime (Collaborators: SLM Solutions, Quintus, Sandia)
- Developed a resonant acoustic technique for rapid, inexpensive NDE of AM aluminum, stainless steel, and cobalt-chrome-molybdenum parts (Collaborators: Elementum 3D, Colorado State)



project website with  
FREE publications

# Motivation

- Confident use of metal additive manufacturing (AM) in critical applications is still lacking [1]
- NIST/ASTM workshop [2] identified one of the greatest needs
  - Deeper understanding of processing-structure-properties relationships focusing on fatigue and fracture behavior



[2] Hrabec, N. et al. NIST Advanced Manufacturing Series 100-4. (2016)

NIST Advanced Manufacturing Series 100-4

## Findings from the NIST/ASTM Workshop on Mechanical Behavior of Additive Manufacturing Components

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Material Measurement Laboratory  
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Auburn, AL

This publication is available free of charge from:  
<https://doi.org/10.6028/NIST.AMS.100-4>

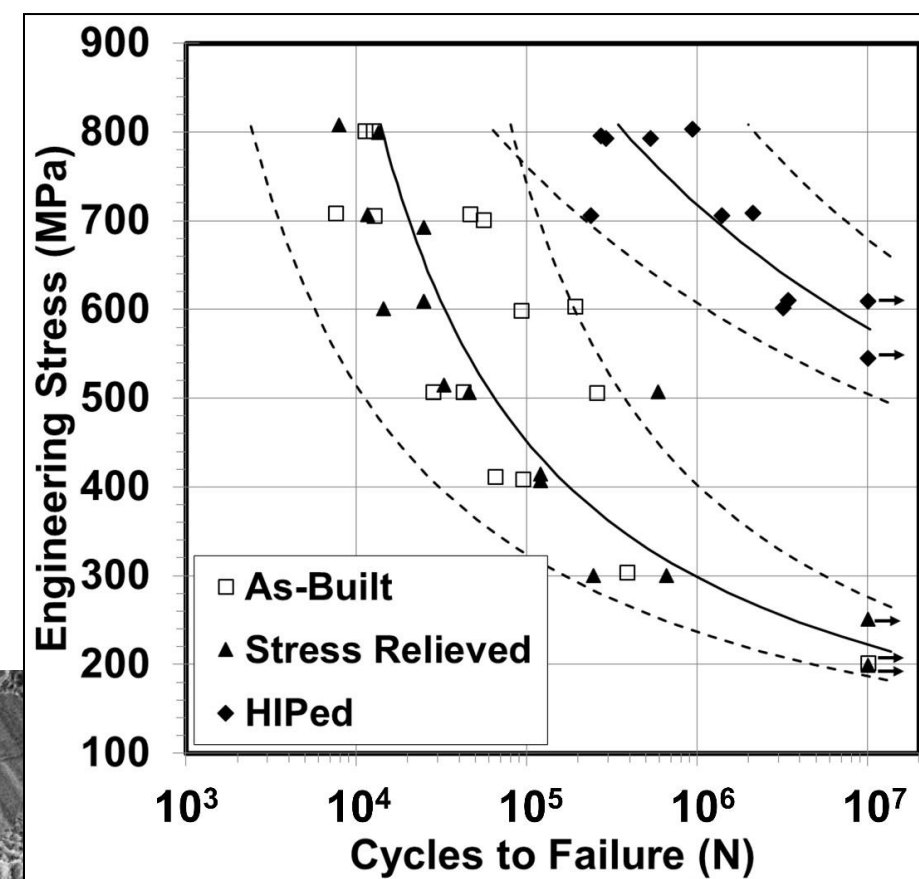
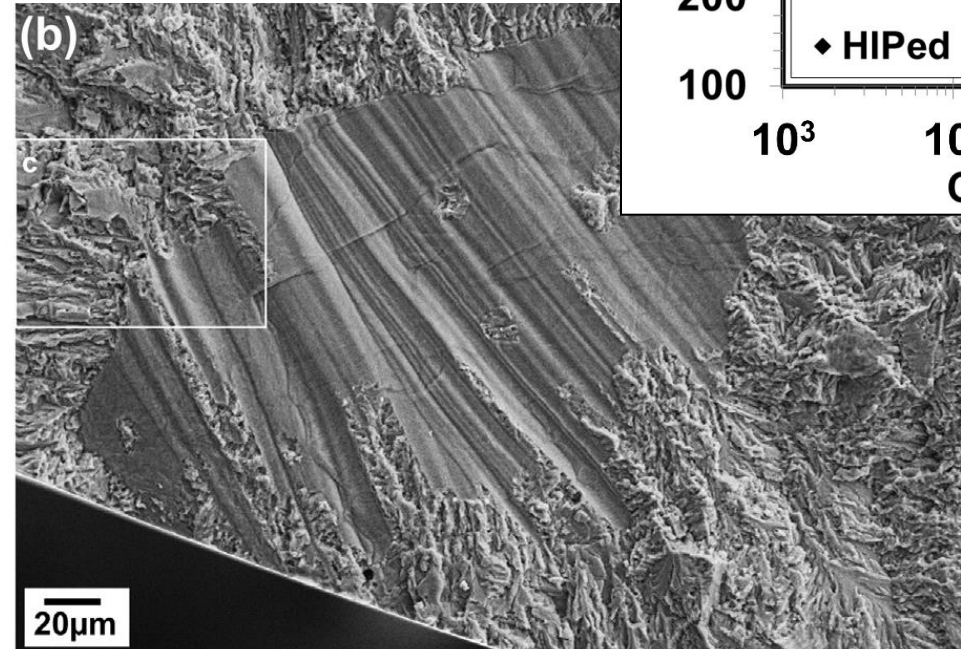
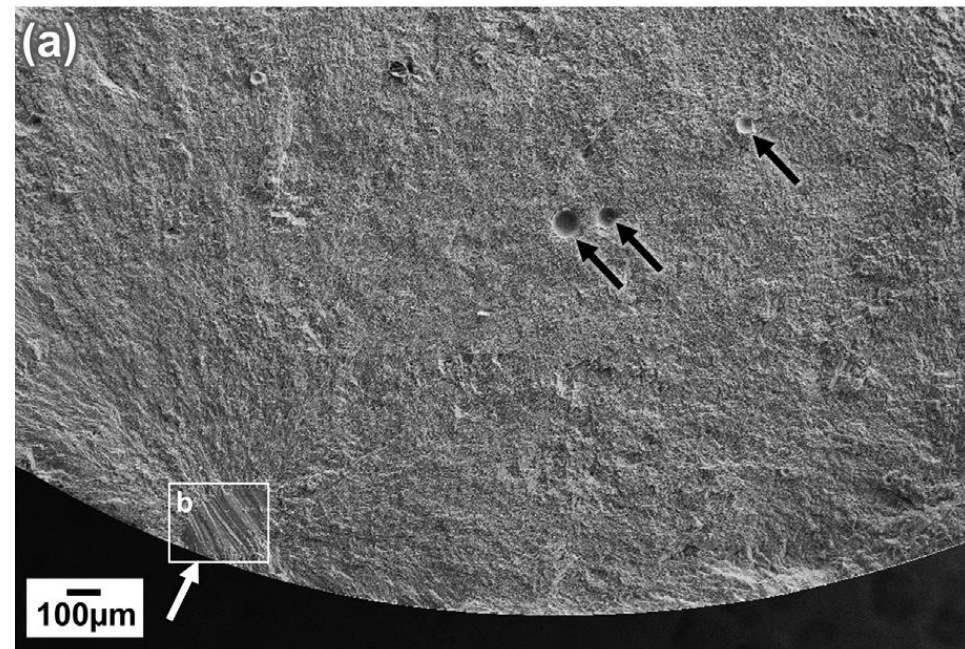


# Outline

- Effect of internal defects (porosity)
- Effect of surface defects (surface roughness)
- Effect of microstructure
- Effect of chemistry
- Role of non-destructive evaluation (NDE)
- Role of qualification
- Role of standardization

# Effect of internal defects (porosity)

- PBF-EB Ti-6Al-4V, axial HCF,  $R = 0.1$
- Pores cause fatigue crack initiation and lower fatigue lifetime
- Hot isostatic pressing (HIP) closes internal pores and improves fatigue lifetime



# Effect of internal defects (porosity)

- **NIST-BAM Workshop** to discuss suitability of probabilistic damage tolerance techniques (e.g. Kitagawa-Takahashi) to predict fatigue performance
- Review paper resultant from workshop

Progress in Materials Science 121 (2021) 100786

Contents lists available at ScienceDirect

Progress in Materials Science

journal homepage: [www.elsevier.com/locate/pmatsci](http://www.elsevier.com/locate/pmatsci)



## Damage tolerant design of additively manufactured metallic components subjected to cyclic loading: State of the art and challenges

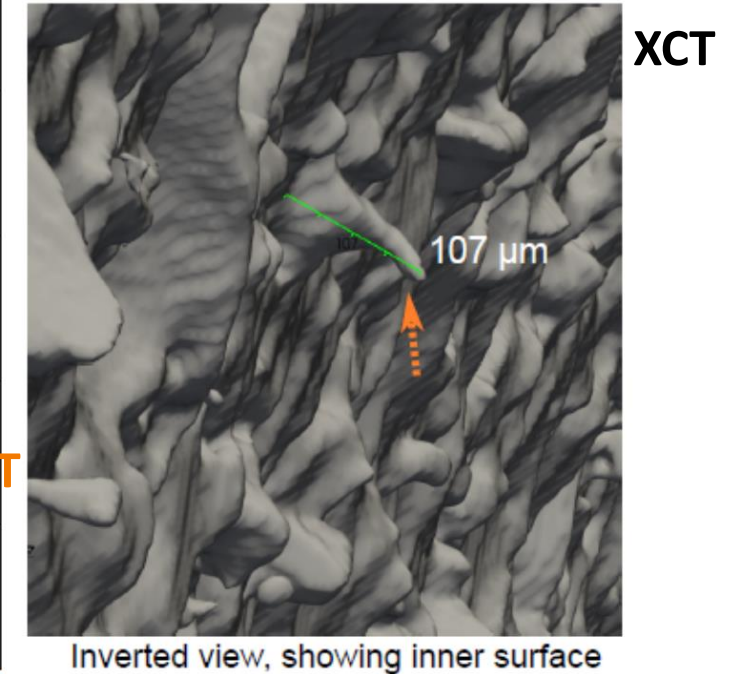
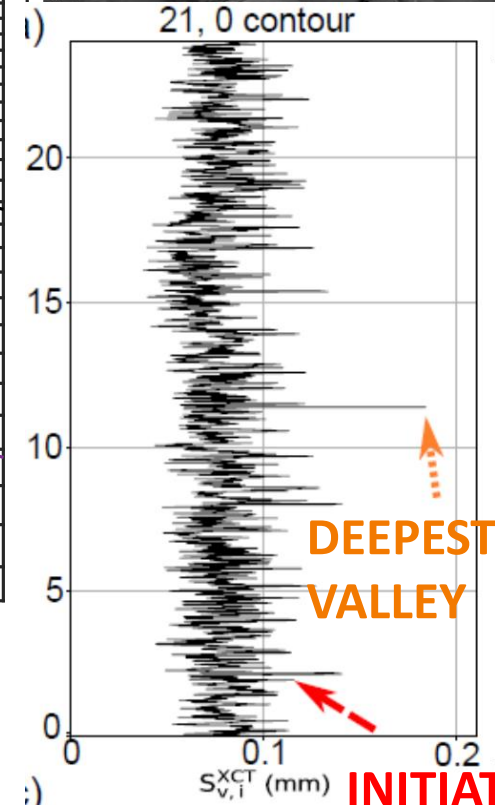
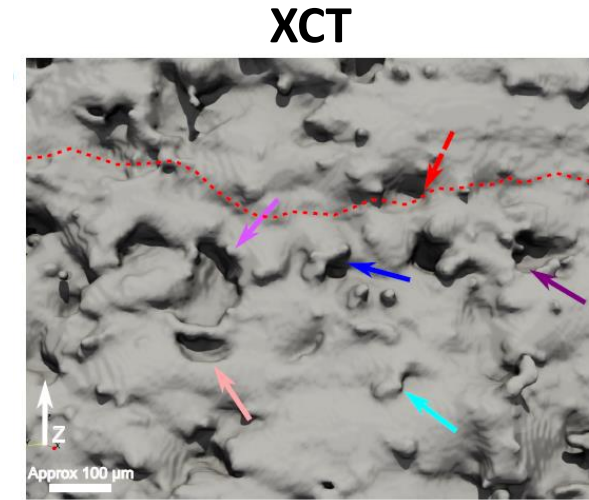
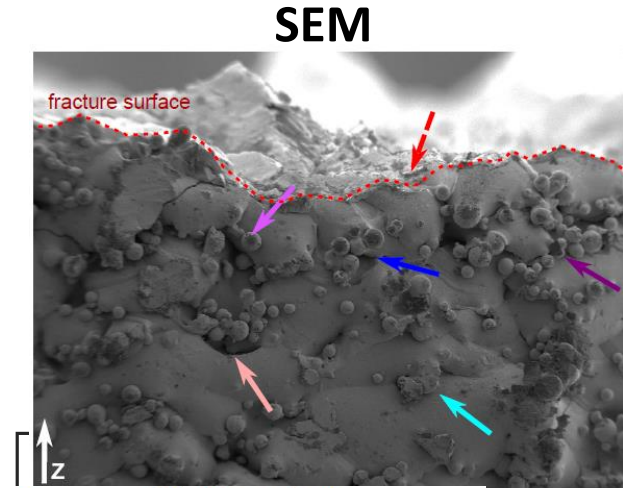
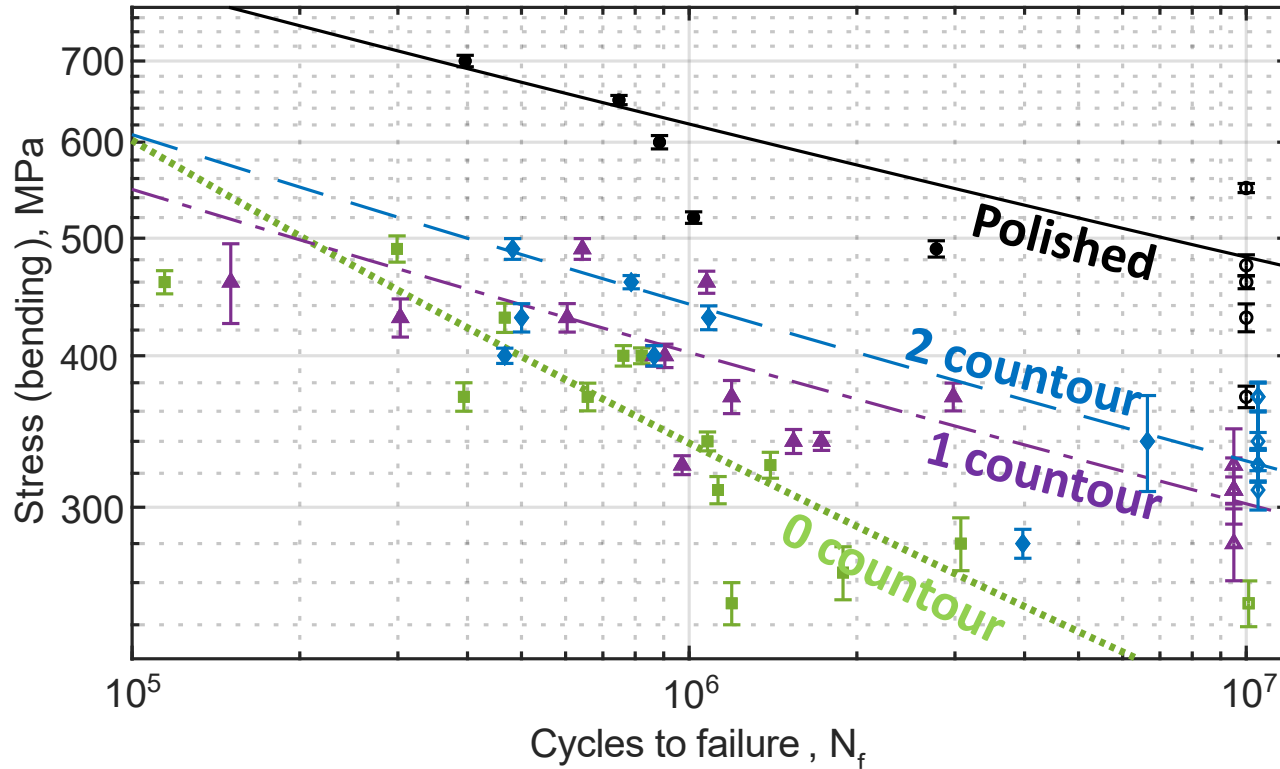
Uwe Zerbst<sup>a,\*</sup>, Giovanni Bruno<sup>a,\*</sup>, Jean-Yves Buffière<sup>b</sup>, Thomas Wegener<sup>c</sup>, Thomas Niendorf<sup>c</sup>, Tao Wu<sup>c</sup>, Xiang Zhang<sup>d</sup>, Nikolai Kashaev<sup>e</sup>, Giovanni Meneghetti<sup>f</sup>, Nik Hrabe<sup>g</sup>, Mauro Madia<sup>a</sup>, Tiago Werner<sup>a</sup>, Kai Hilgenberg<sup>a</sup>, Martina Koukolíková<sup>h</sup>, Radek Procházka<sup>h</sup>, Jan Džugan<sup>h</sup>, Benjamin Möller<sup>i</sup>, Stefano Beretta<sup>j</sup>, Alexander Evans<sup>a</sup>, Rainer Wagener<sup>i</sup>, Kai Schnabel<sup>i</sup>

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Zerbst (2021) Prog. Mat. Sci., 121, 100786

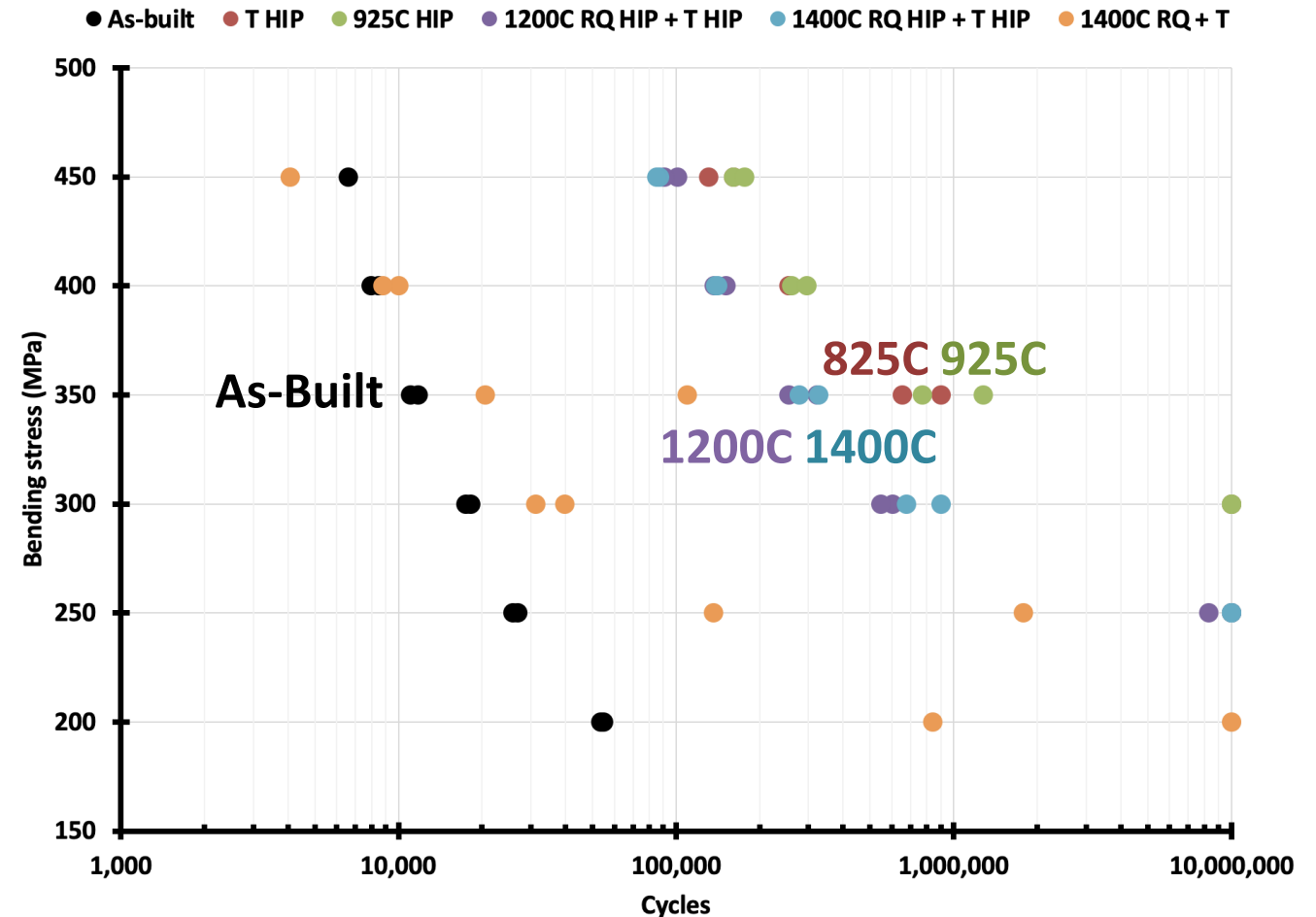
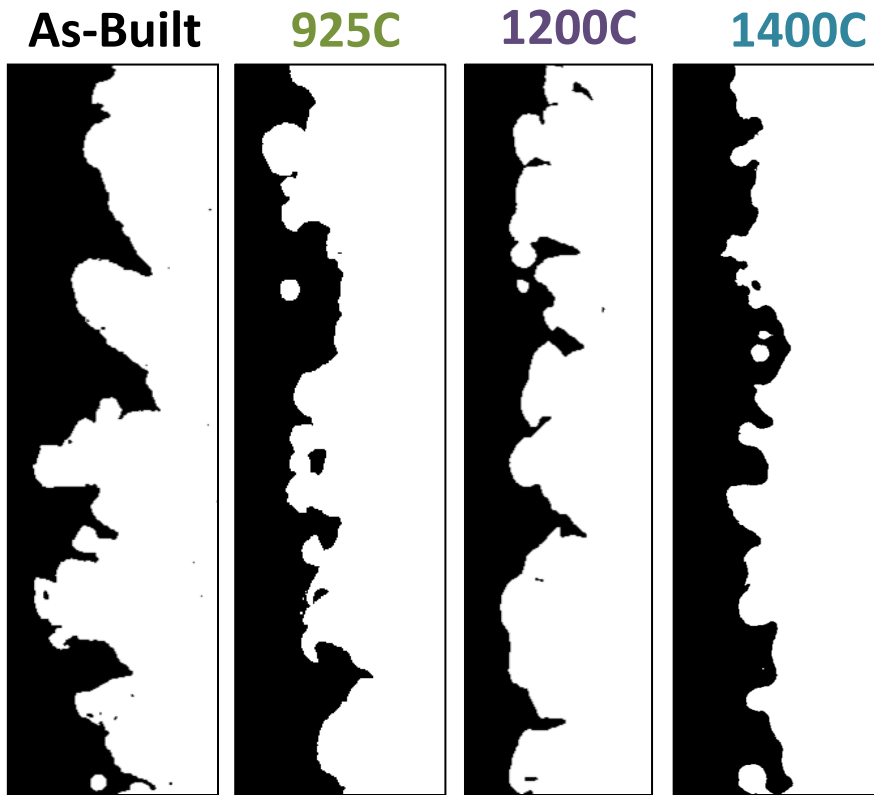
# Effect of surface defects (surface roughness)

- PBF-L Inconel 718, rotating bending fatigue (RBF),  $R = -1$
- In general, fatigue lifetime improves with reduced surface roughness
- Deepest valley does not always cause crack initiation (microstructure influence?)



# Effect of surface defects (surface roughness)

- PBF-L Ti-6Al-4V, RBF, R = -1
- Higher temperature HIP to reduce surface roughness and improve fatigue lifetime

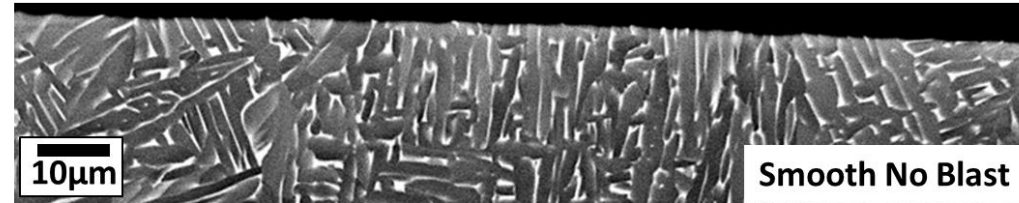


# Effect of surface defects (surface roughness)

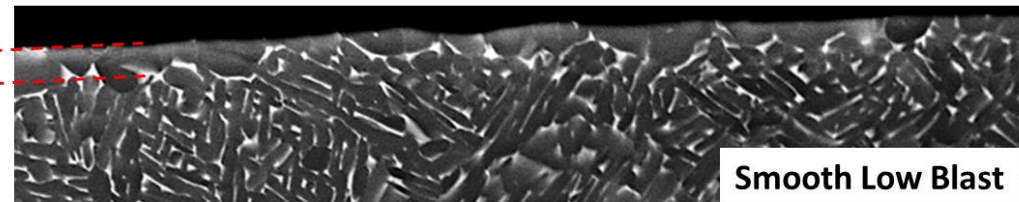
- PBF-EB Ti-6Al-4V, RBF, R = -1
- Controlled powder removal blasting intensity (working distance) to control depth of surface microstructure change (globular, equiaxed) and surface roughness, resulting in significant fatigue strength improvement (**10%**)



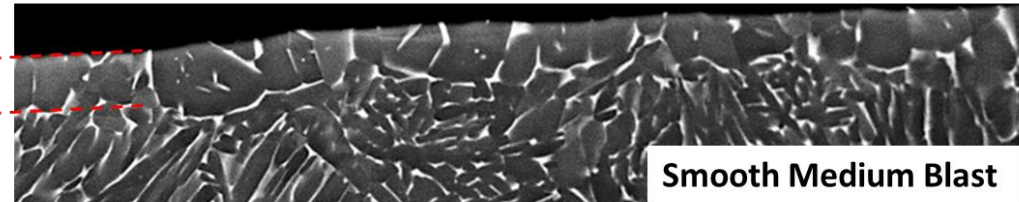
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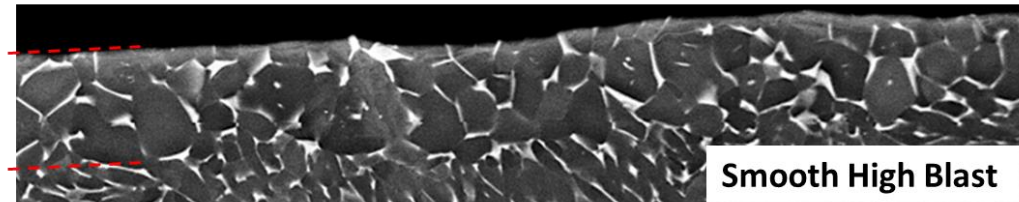
5 μm



8 μm

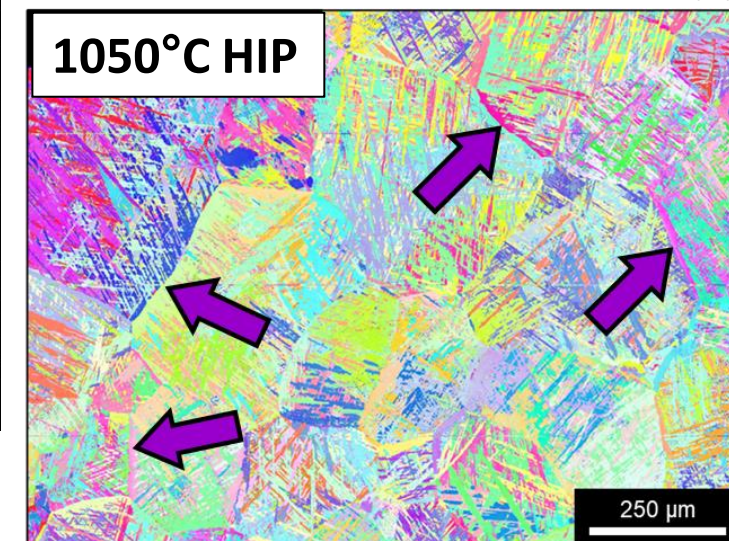
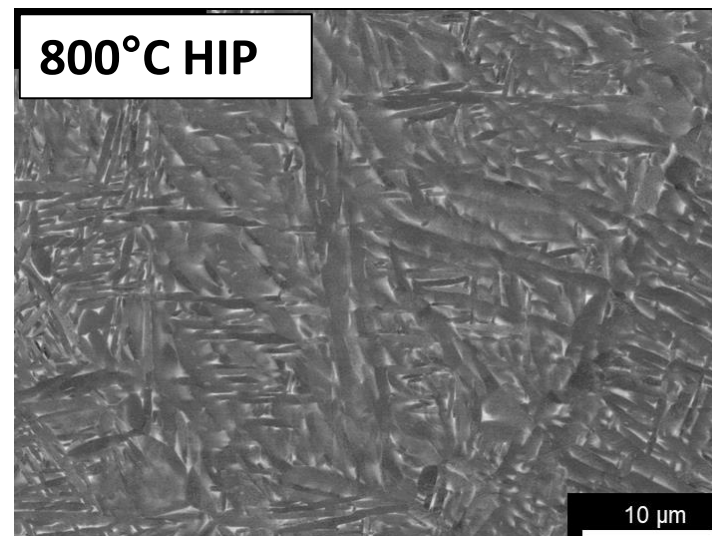
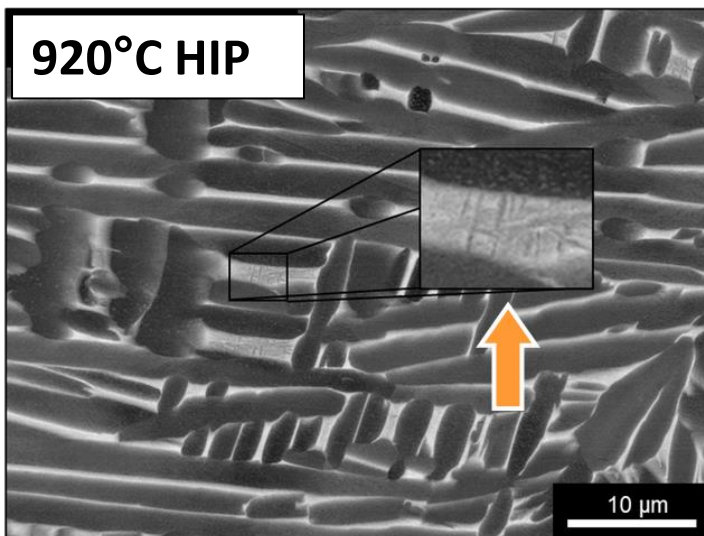
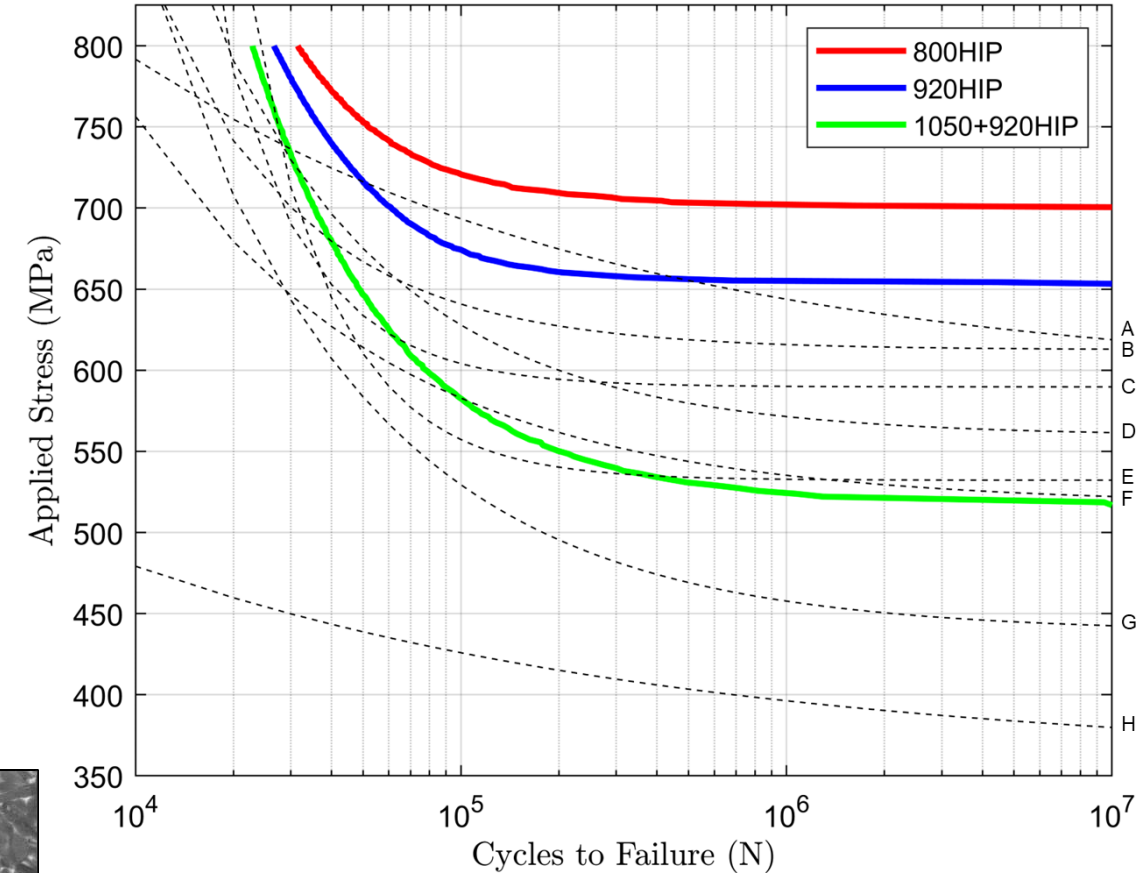


15 μm



# Effect of microstructure

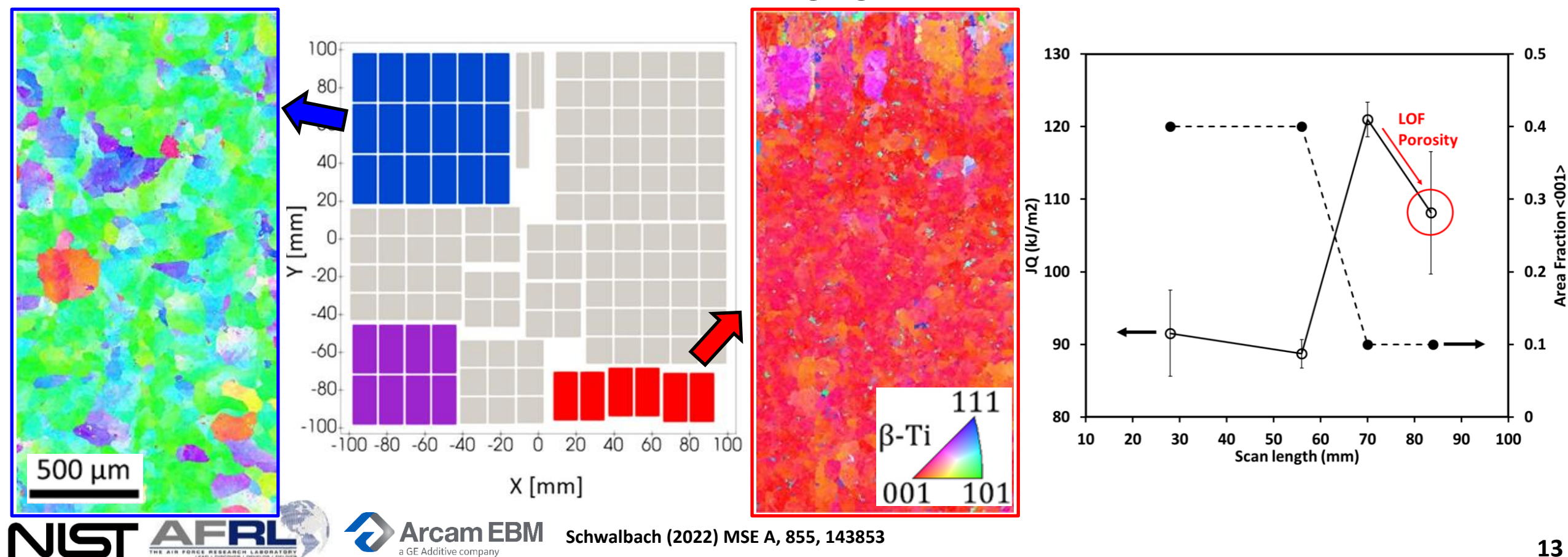
- PBF-L Ti-6Al-4V, RBF, R = -1
- Fatigue behavior of novel HIP treatments
  - 800°C > 920°C due to finer  $\alpha$  lath thickness
  - 920°C > literature due to **tertiary  $\alpha$**  (100°C/min cooling rate)
  - 1050°C < 920°C due to **grain boundary  $\alpha$**  (2000°C/min cooling rate)
- Contributed to AMS 7028



Derimow (2024) *IJF*,  
vol 185, 108362

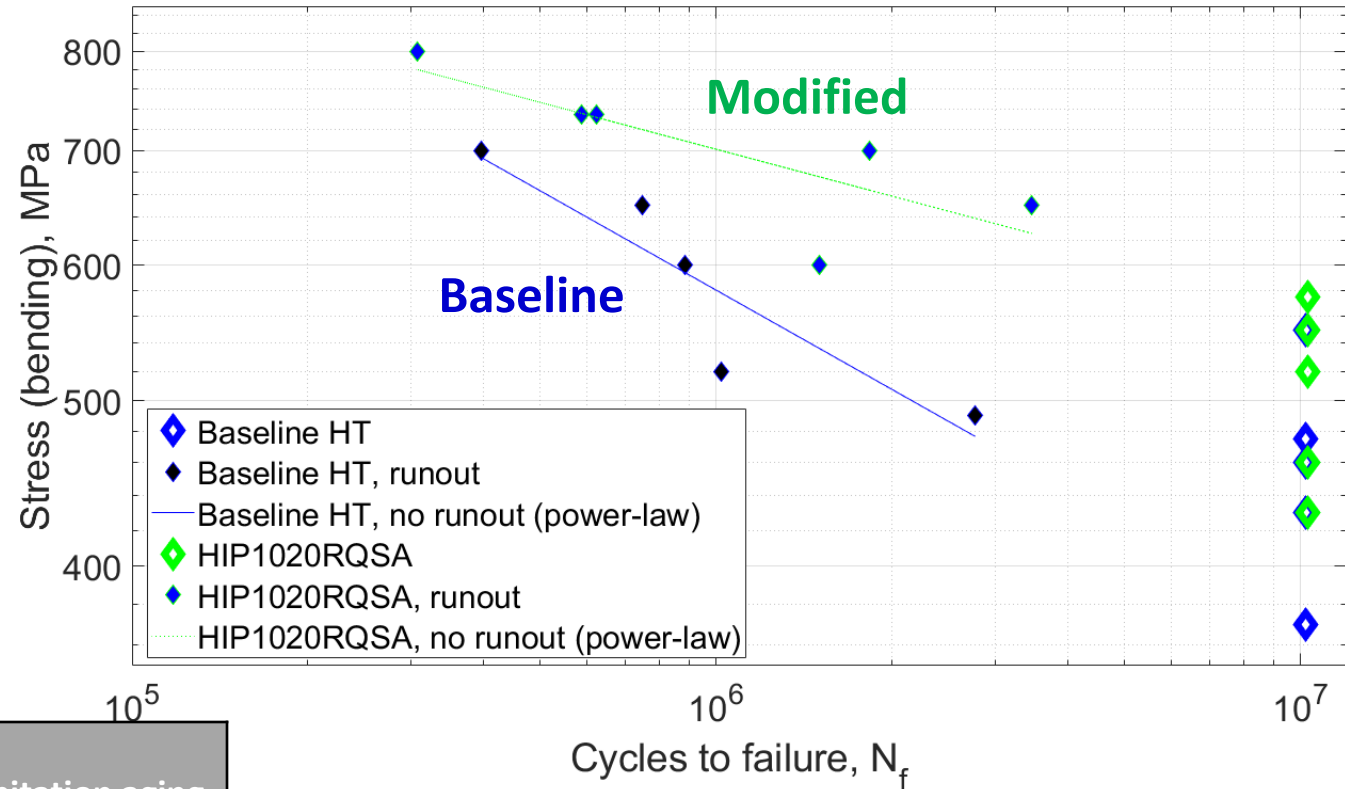
# Effect of microstructure

- Discovered texture variation in PBF-EB Ti-6Al-4V due to small changes in build layout (scan length) leading to 10% change in YS and **40% change in fracture toughness**
- Developed processing (scan length control) and post-processing (super- $\beta$  HIP to reset texture) mitigation strategies
- Contributed to ISO/ASTM 52911-3 PBF-EB design guide

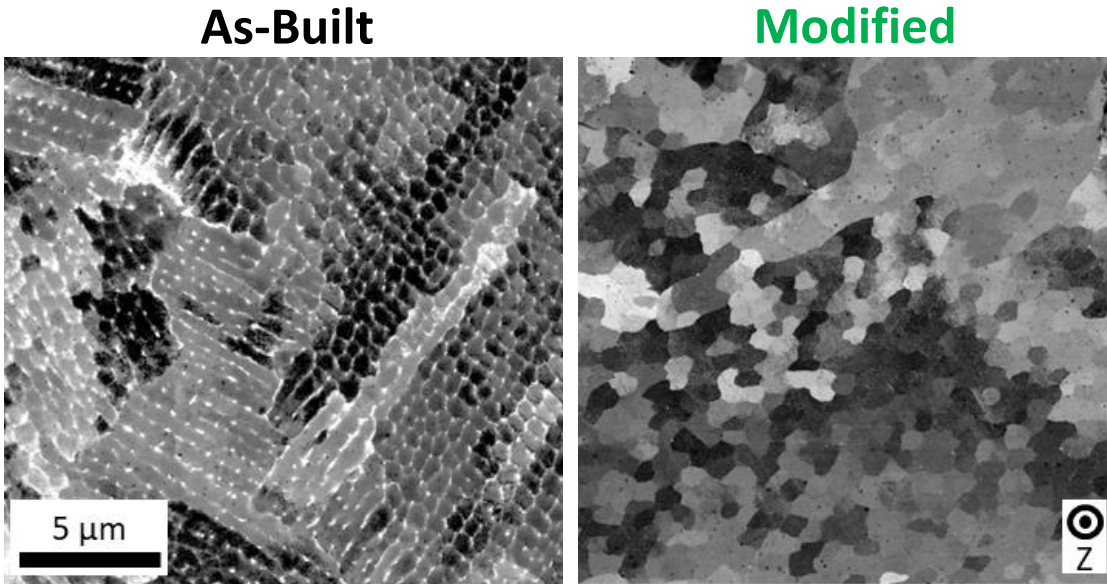


# Effect of microstructure

- Modified HIP for PBF-L Inconel 718
- Reduced heat treatment time (42h→15h) and steps (5→2)
- Lower temperature HIP (1020°C) and combining 3 Baseline steps: stress relief, HIP, and solutionizing
  - Retain as-built sub-grain structure (no/partial recrystallization)
  - No  $\delta$ , reduced Laves phases
  - Successful pore closure
- Single step aging still achieves desired  $\gamma'$  and  $\gamma''$  precipitates

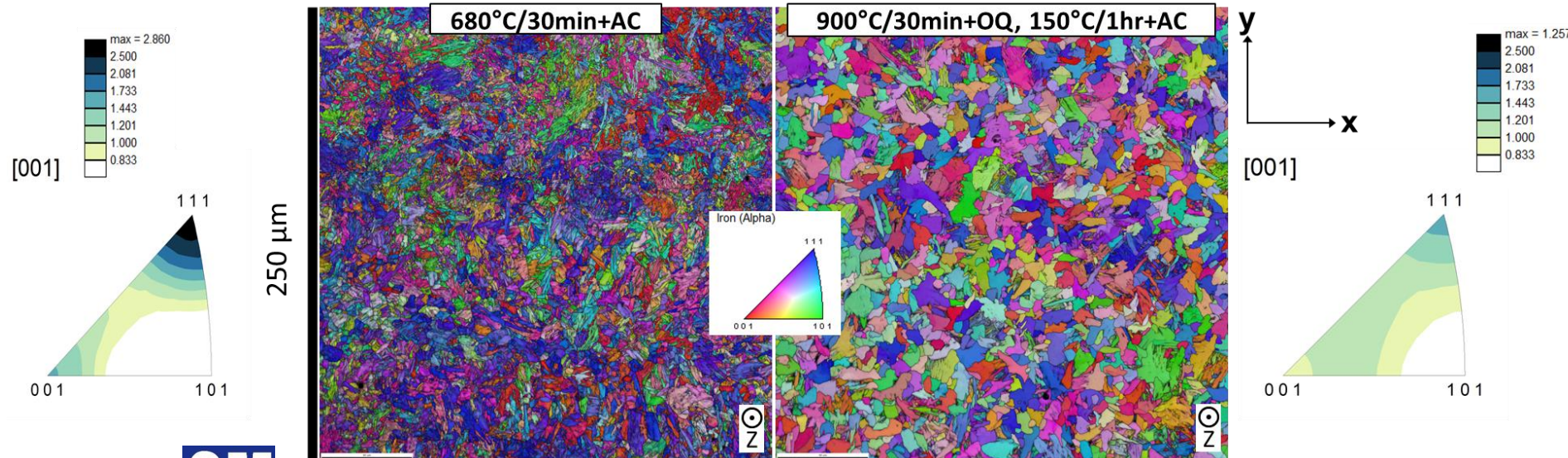
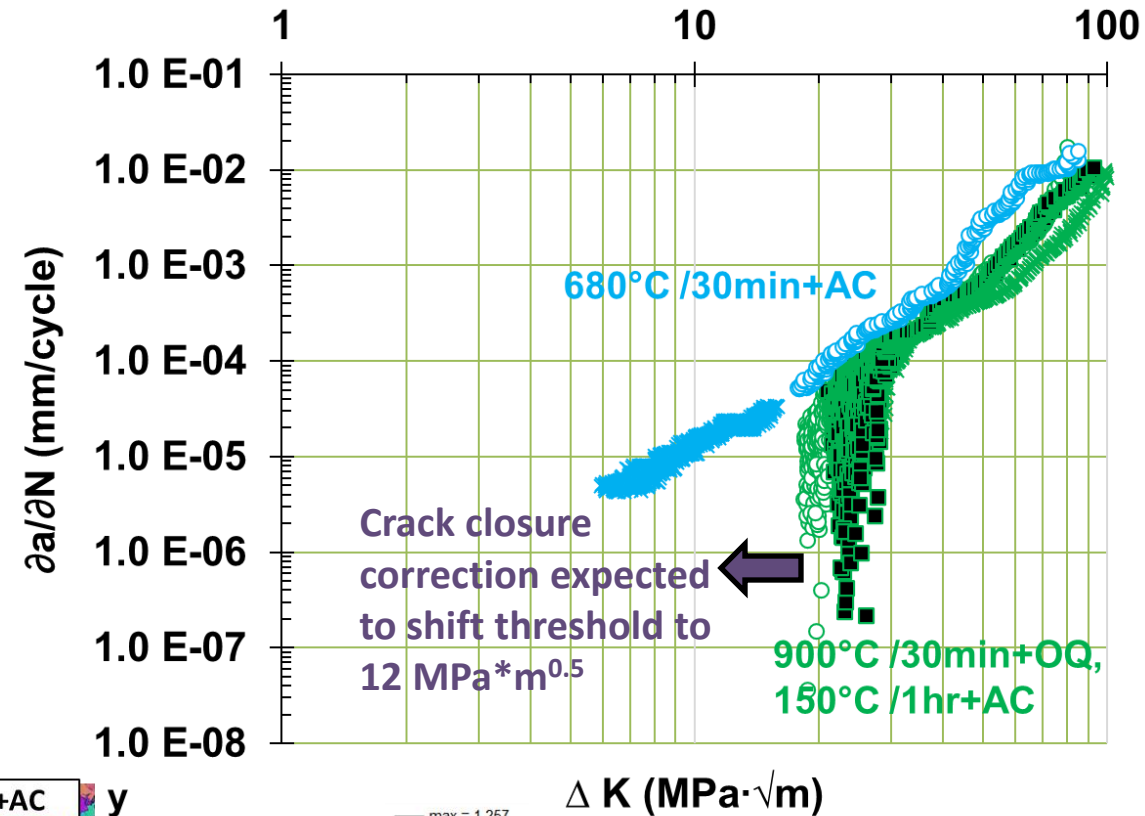


| HIP-HT variant      | Stress relief (furnace) | Seal porosity (HIP)                                  | Solution (HIP or furnace)                               | Precipitation aging (furnace)                                    |
|---------------------|-------------------------|------------------------------------------------------|---------------------------------------------------------|------------------------------------------------------------------|
| Baseline (AMS 2774) | 1065 °C, 1.5 h, AC      | 1150 °C, 4 h, 100 MPa, FC                            | Vacuum furnace: 1066 °C, 1 h, quench to 95 °C, AC to RT | 2-STEP AGING 718 °C, 8 h, FC to 621 °C, hold for additional 10 h |
| Modified (V4)       | -                       | 1020 °C, 0.5 h, 200 MPa, cool to RT at 2,000 °C /min | -                                                       | 1-STEP AGING 700 °C, 12 h, WQ                                    |



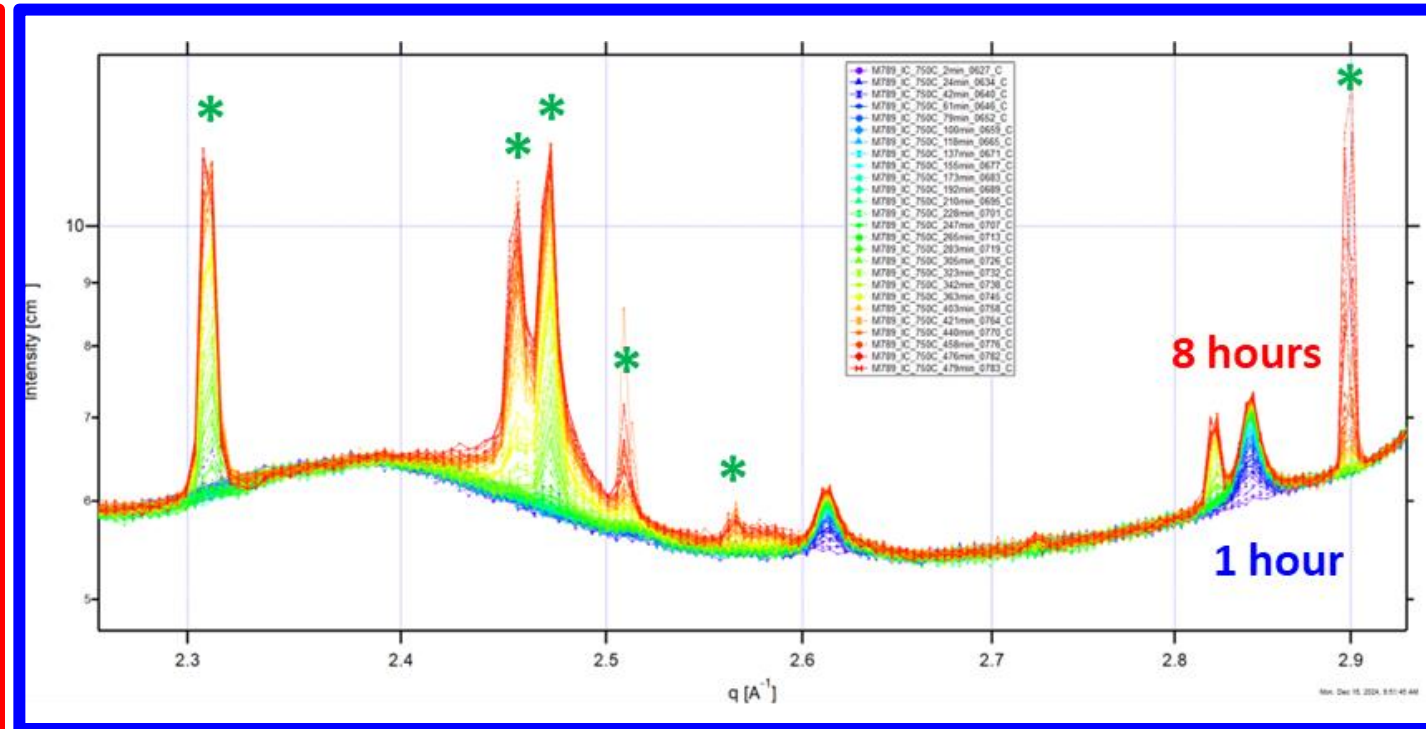
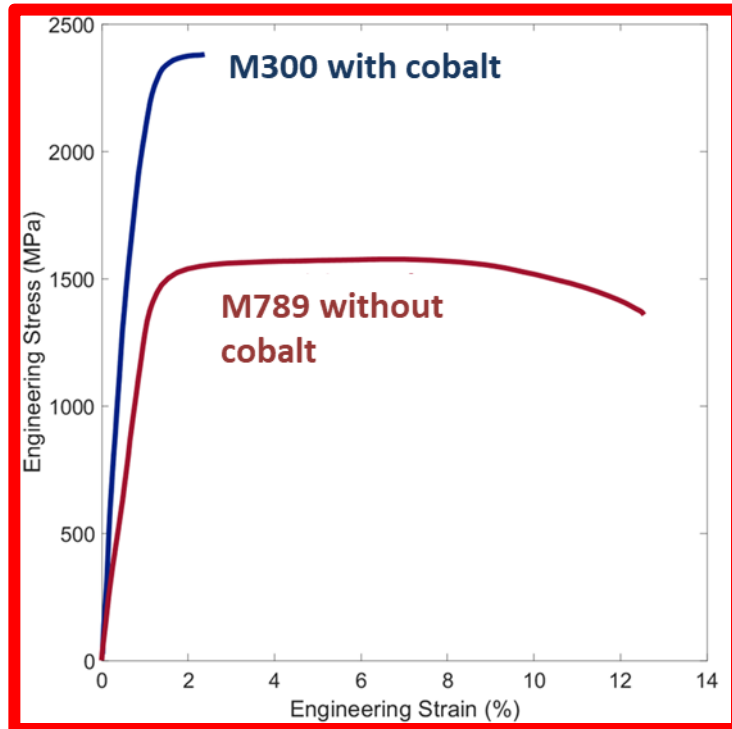
# Effect of microstructure

- **Custom PBF-L automotive steel alloy**
  - Minimize alloy content (0.2C-0.8Mn-1Si wt%)
  - Quench and temper (Q&T) condition [900°C/30min+OQ, 150°C/1hr+AC]
  - Stress Relieved (SR) condition [680°C/30min+AC]
  - No HIP
  - Avoid  $\delta$ -ferrite phase transformation
- **Q&T condition shows weaker texture and improved fatigue crack growth rate threshold behavior compared to SR condition**



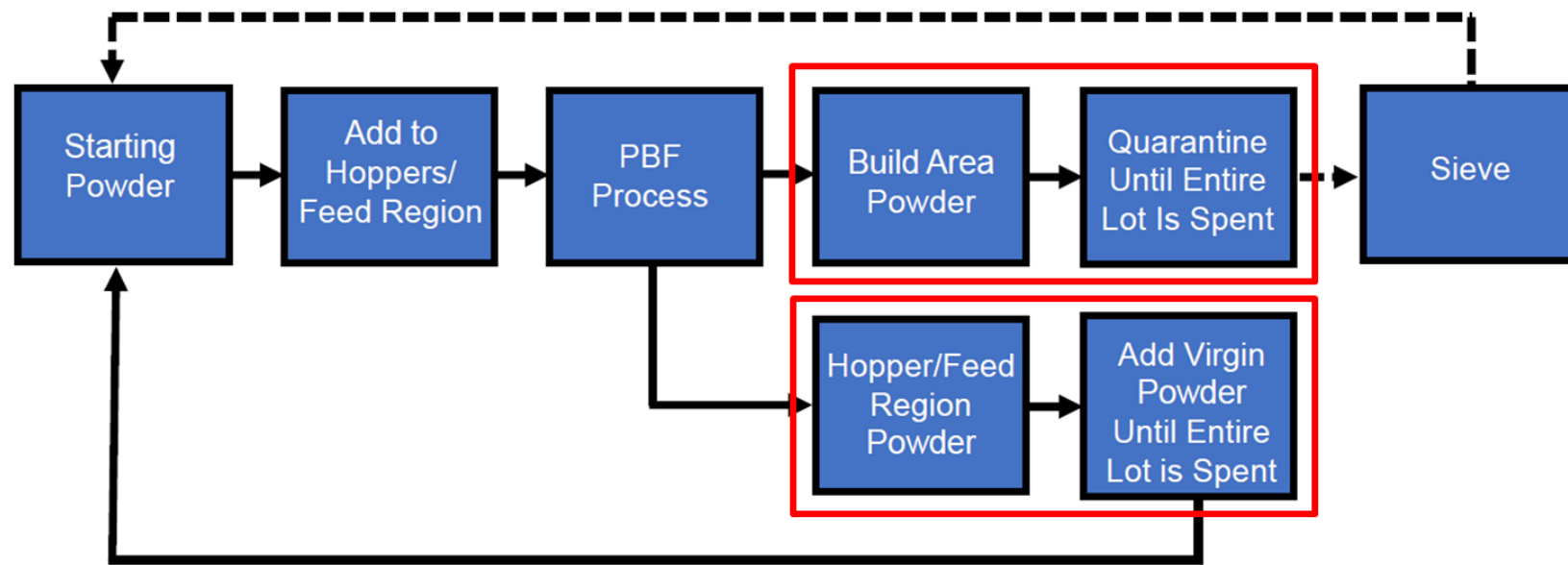
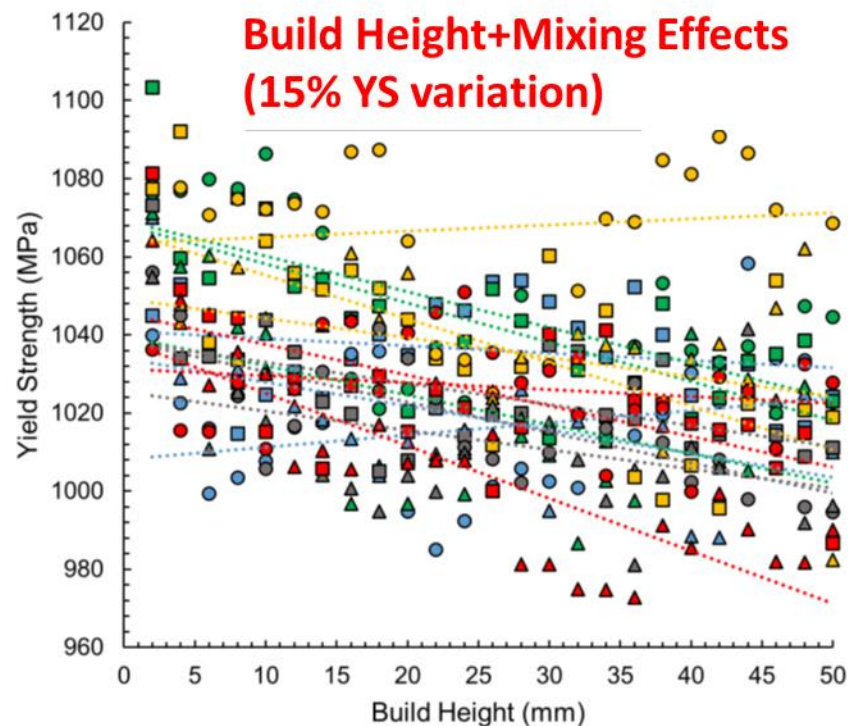
# Effect of microstructure

- M789 cobalt-free maraging steel
- Removal of critical mineral leads to **significant strength reduction**
- AM rapid heating/cooling promotes nickel partitioning to form metastable austenite ( $\gamma$ ) with potential for transformation induced plasticity (TRIP) and improved strength
- Optimize processing and **post-process heat treatment** to maximize  $\gamma$
- Mechanical testing at cryogenic (4°K liquid helium) temperatures, and in pressurized hydrogen



# Effect of chemistry

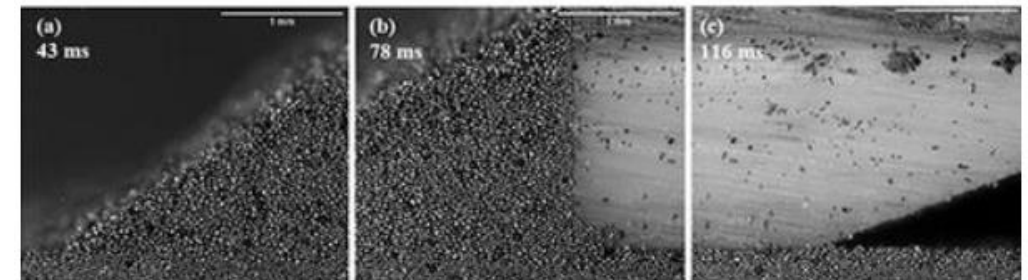
- Discovered significant chemistry and intra-build strength variation (**15% YS variation**) for PBF-EB Ti-6Al-4V due to current industrial powder reuse (i.e. mixing of powder with different oxygen content)
- Developed mitigation strategy (i.e. **powder quarantining**)
- Contributed to ASTM F3592 guide for PBF powder reuse



# NIST Metal AM Powder Consortium (MAMP)

- MAMP brings together stakeholders to develop deeper understanding of AM metal powder characteristics, metrics, and measurement techniques, and how they affect various AM processes and resultant materials and components
- Current Challenges
  - Incomplete understanding of powder characteristics, metrics, measurement techniques, and their effect on process, material, and component performance
  - Lack of **prescriptive** standards for effective powder use, re-use, and measurement techniques
  - Inconsistent or undefined repeatability or reproducibility of various powder metrics
  - Lack of common protocols for powder measurement techniques across different equipment manufacturers

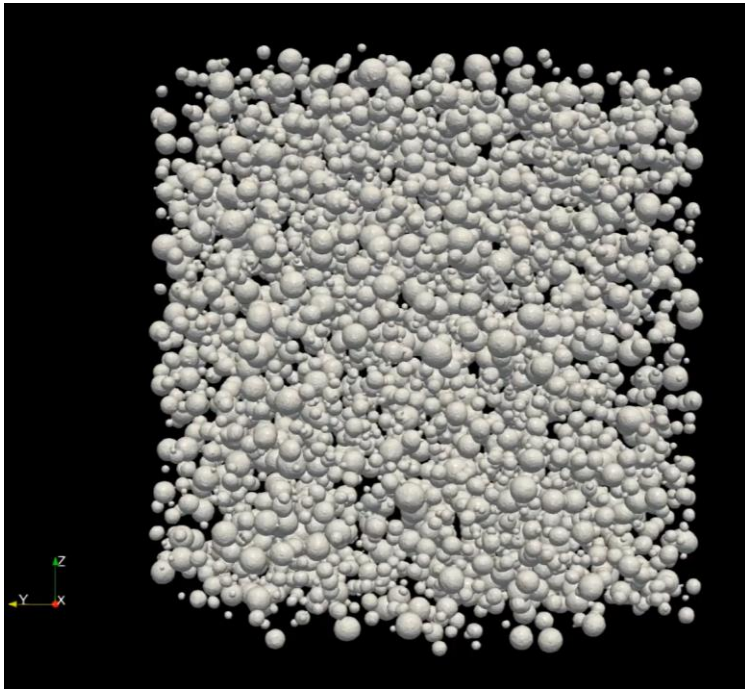
NIST Powder Spreading Testbed (PST)



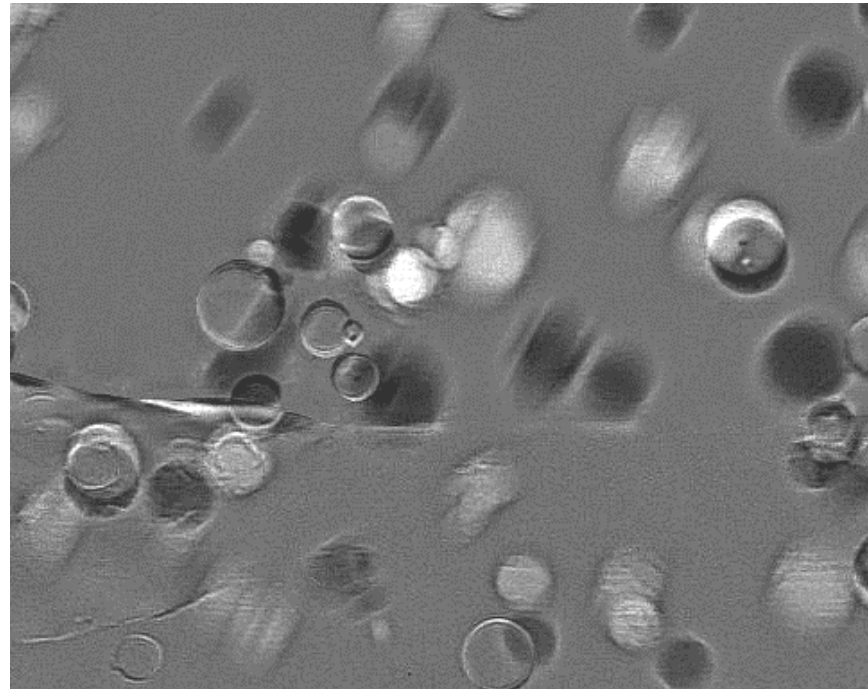
Timelapse Results from PST

# Role of non-destructive evaluation (NDE)

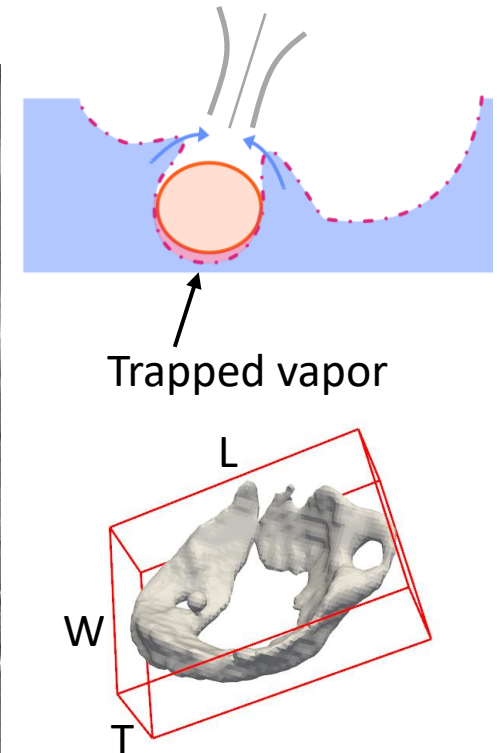
- Developed an (open source) Python library for X-ray CT data processing
  - IMPPY3D
- Enables:
  - Dimensional & shape measurements of internal & external defects & features
  - 3D rendering visualization



Garboczi (2020) Add. Manuf., vol 31, 100965

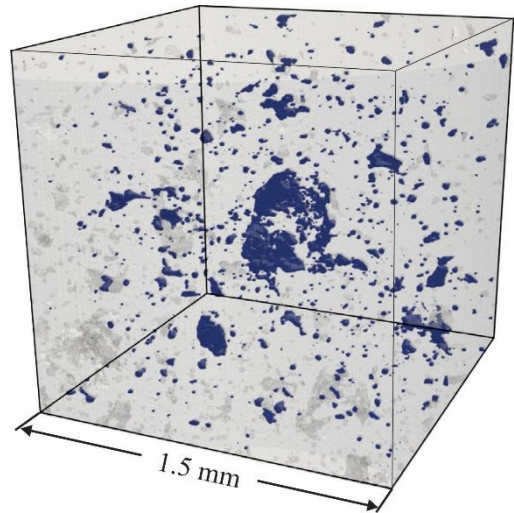


Webster (2023) PNAS Nexus, vol 2, issue 6

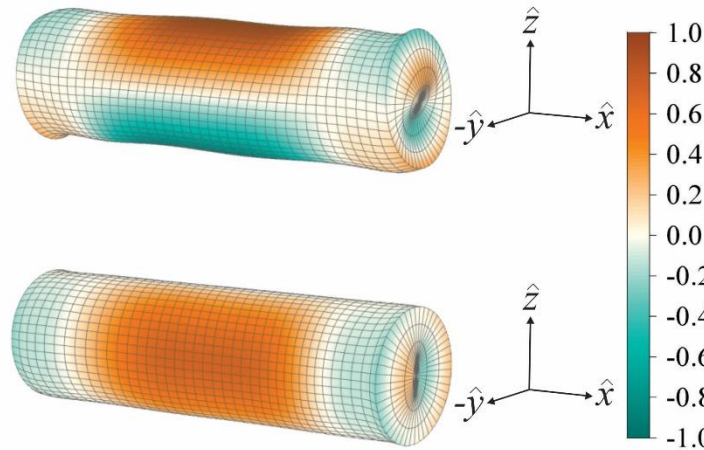


# Role of non-destructive evaluation (NDE)

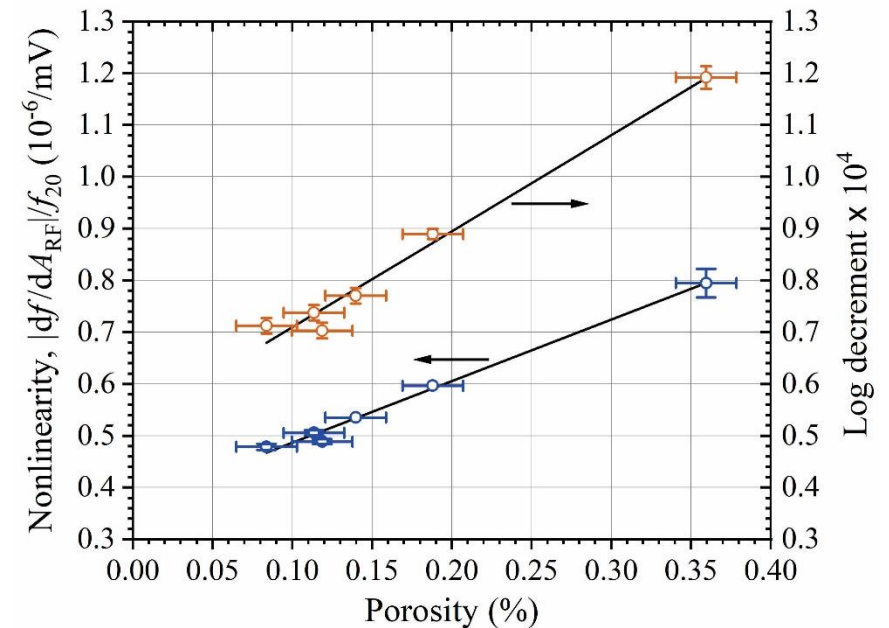
- Developed an innovative acoustic-resonance technique that is sensitive to variations in porosity in AM metals at industrially relevant levels.
- This technique offers a unique set of advantages for industrial post-process AM part inspection, including speed, high precision, low cost, and applicability to complex geometries.



Xray-CT image of pores in AM Al with non-optimal build parameters

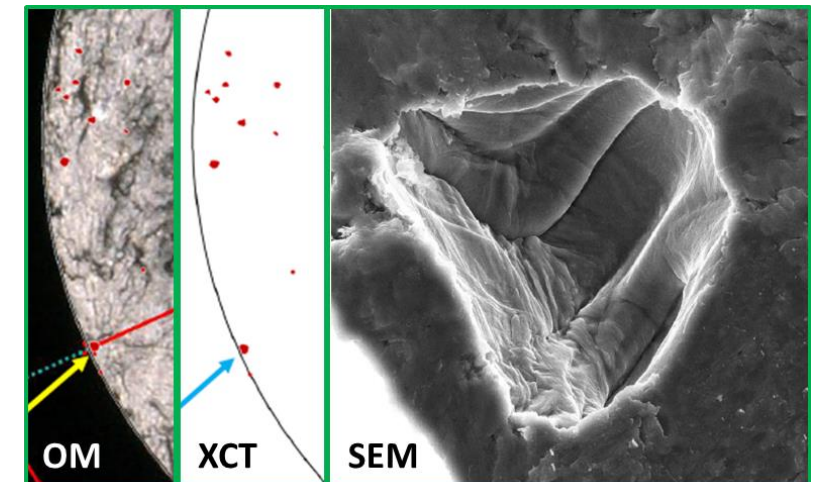
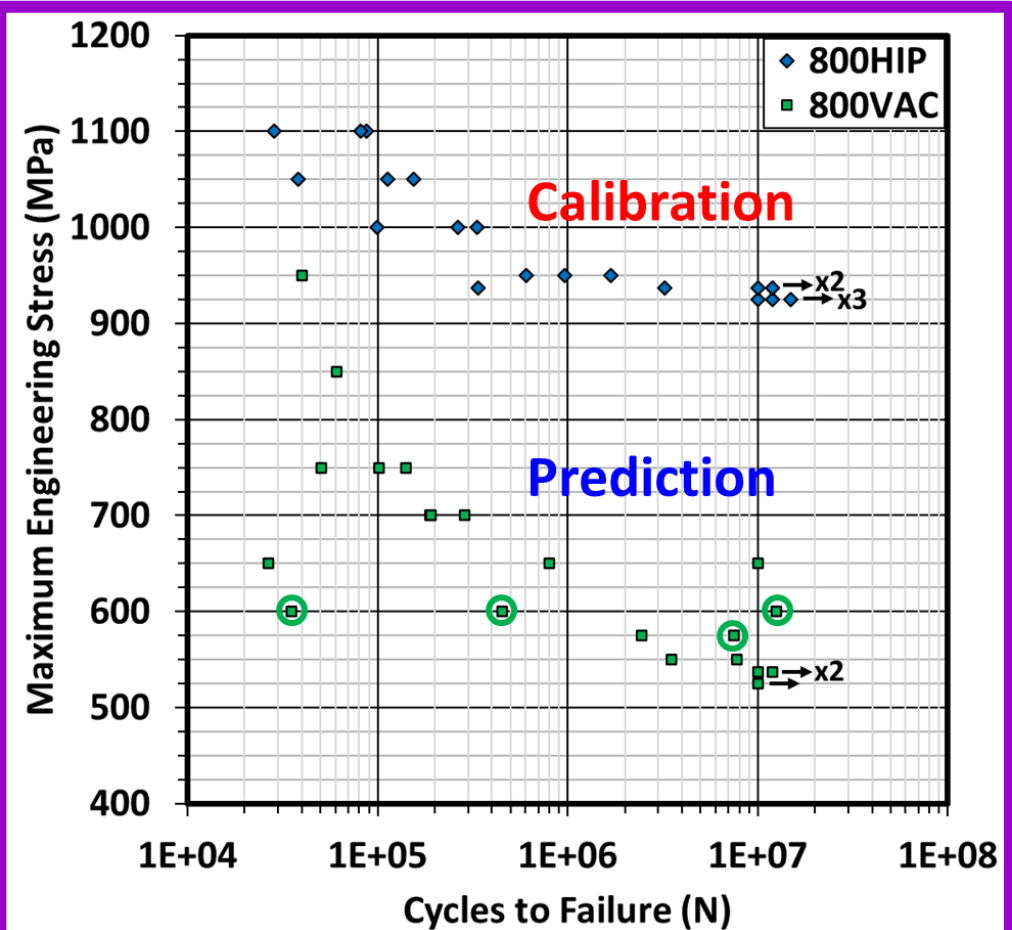


Vibrational displacement patterns of resonant modes near 670 kHz in AM Al cylinders.



# Role of qualification

- “Adoption is limited by qualification”
  - Quote from 2024 NASEM Workshop on Statistical and Data-Driven Methods for AM
- Can computational techniques enable rapid qualification?
  - NASA/FAA/NIST Computational Materials for Qualification and Certification (CM4QC)
  - **NIST AM Bench** - experimentally supports computational technique development for AM



# Role of standardization

- Documentary standards development requires volunteer experts
  - Many standards development organizations (SDOs) have AM committees/activities
  - Examples: ASTM+ISO, SAE, AWS
- America Makes and ANSI Additive Manufacturing Standardization Collaborative (AMSC)
  - Standardization Roadmap for AM



# Opportunities at NIST

- **Undergraduate Students**
  - NIST Summer Undergraduate Research Fellowship (SURF), <https://www.nist.gov/surf>
- **Graduate Students**
  - Email me to discuss collaboration ideas
  - Guest Researcher status at NIST is possible
- **Postdoctoral**
  - NRC Research Associateship Program (RAP), <https://sites.nationalacademies.org/PGA/RAP/index.htm>
  - 2yr postdoc with competitive pay (institutions other than NIST available)
  - 10pg proposal developed with potential advisor is main component of application
  - 2 application deadlines: Feb 1 (start date range July-December), Aug 1 (start date range January-June)

**Project Website,  
FREE Publications**



**QUESTIONS:  
[nik.hrabe@nist.gov](mailto:nik.hrabe@nist.gov)**