U.S. DEPARTMENT OF ENERGY | Office of NUCLEAR ENERGY

# Materials Testing, Characterization, and NDE

Michael McMurtrey, INL

AMMT Industry Workshop

May 23rd, 2023

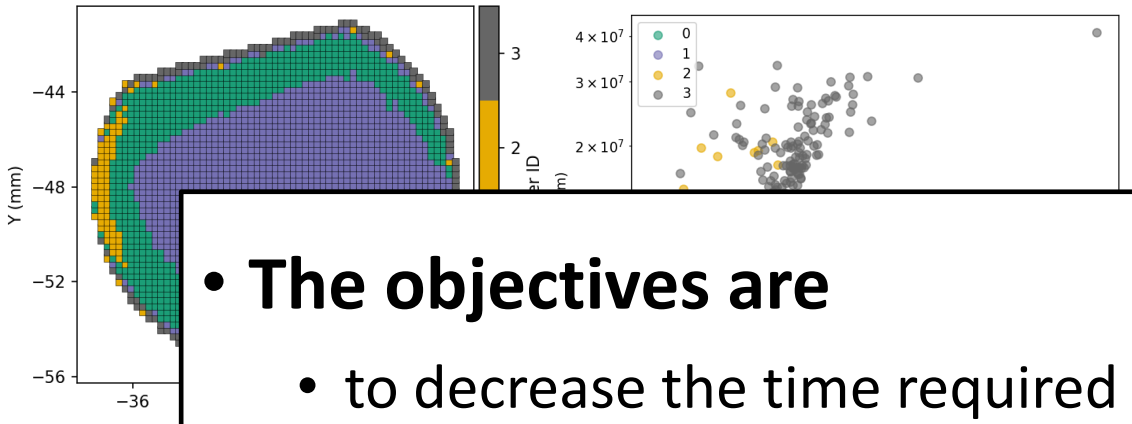
# Multi-lab team

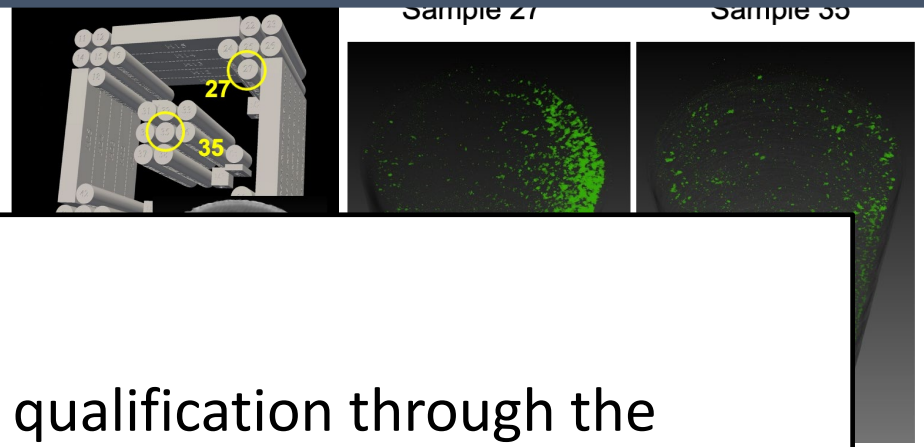
- Teams from Argonne, Idaho, Oakridge, and Pacific Northwest National Laboratories collaborating in this area.
- Acknowledge input from Yiren Chen (ANL), Xuan Zhang (ANL), Bill Chuirazzi (INL), Amir Ziabari (ORNL), Matthew Olszta (PNNL), Robert Montgomery (PNNL) and other teams supporting this work at each laboratory

# Validated Informatics for multi-scale characterization

Complex thermal signatures cause variations in properties

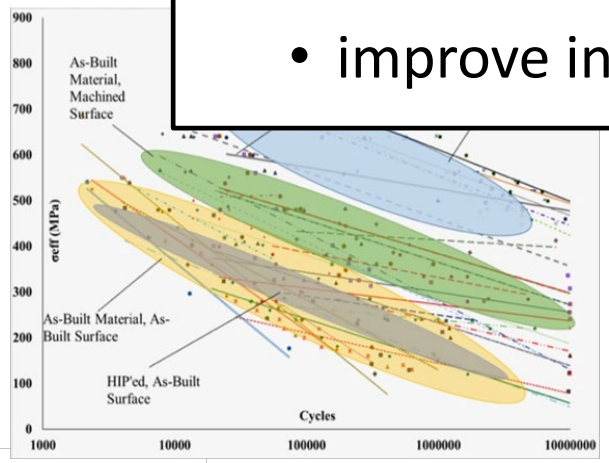
Defect size, density & distribution vary for uniform process conditions



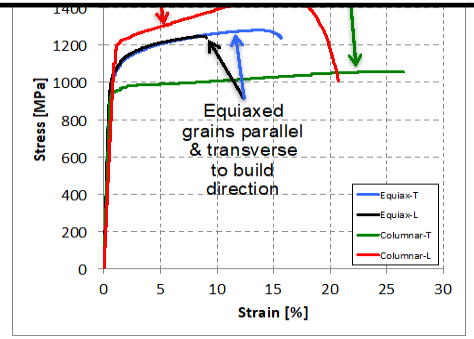




- **The objectives are**
  - to decrease the time required for code qualification through the development of accelerated testing and characterization techniques
  - improve inspectability through nondestructive evaluation

etc.)

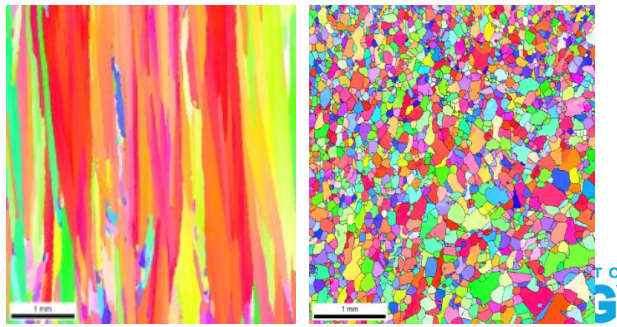



**Fatigue Behavior**

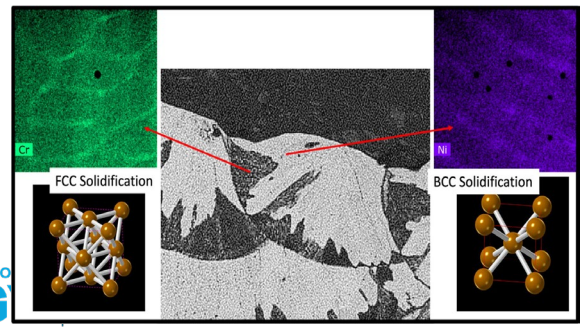



**Tensile Properties**

**Texture Variation**



**Solute Variation**

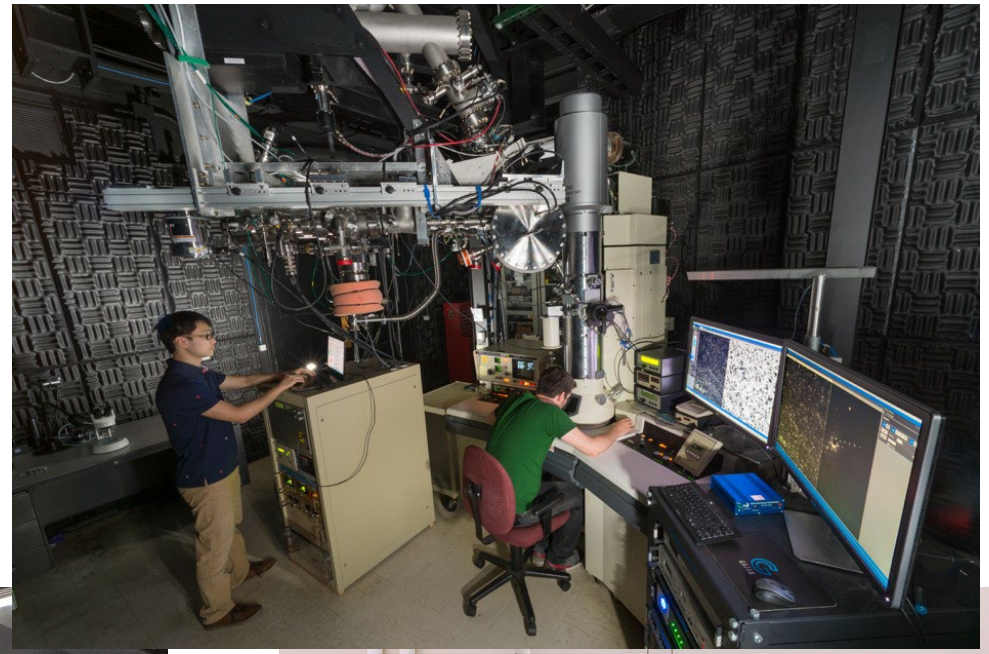


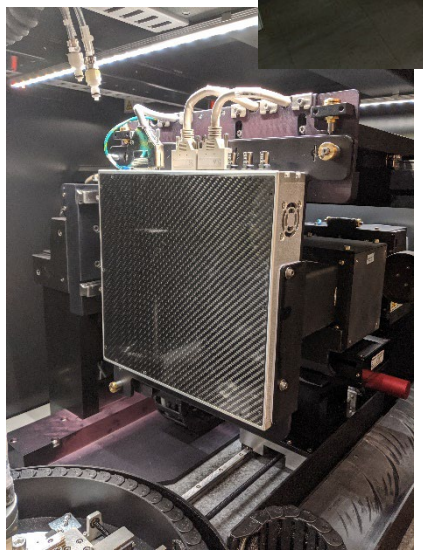
# Decrease time requirements for qualification

- Accelerated testing
- Process monitoring/control as part of the qualification
- Accelerated characterization
  - Part of the qualification process
  - Provide data for modeling/simulation

# Capabilities at National Laboratories

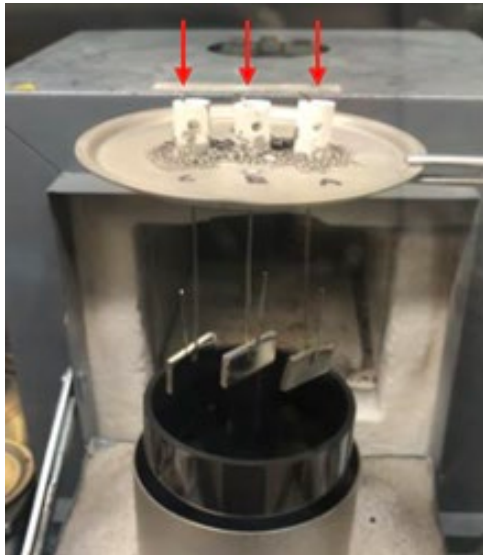
- Microscopy
  - Transmission Electron Microscopy
  - Scanning Electron Microscopy
  - Atom Probe
  - Focused ion beam
  - Optical/Laser
- NDE
  - X-ray/neutron Computed Tomography
  - X-ray/Neutron diffraction
  - Resonant ultrasound spectroscopy



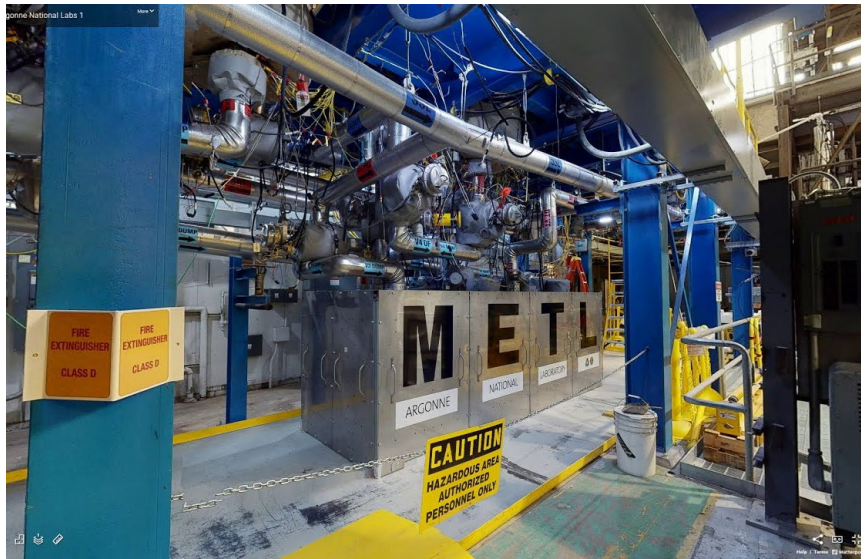


# Testing

- Mechanical testing
  - Electrical/hydraulic test frames
    - Tensile
    - Cyclic
    - Crack growth rate
  - Creep
    - Stress relaxation
    - Rupture
  - Charpy impact
  - Hardness

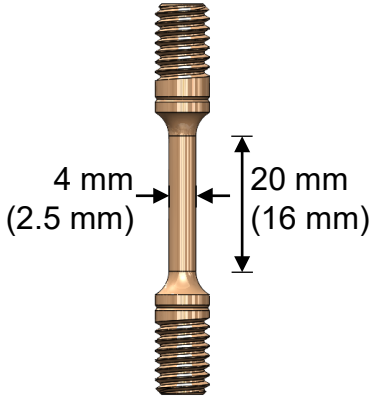


| Materials | Environments | Mechanical load | | | in-situ irradiation | |
|---|---|---|---|---|---|---|
| | | no load | static load | dynamic load | ions | gamma / neutrons |
| Unirradiated materials | Water | X | X | X | X | |
| | Sodium | X | | | | |
| | Helium | X | X | X | | |
| | Molten salt | X | | | | X |
| Irradiated materials (radioactive) | Water | X | X | X | X | |
| | Sodium | | | | | |
| | Helium | | X | | | |
| | Molten salt | | | | | |

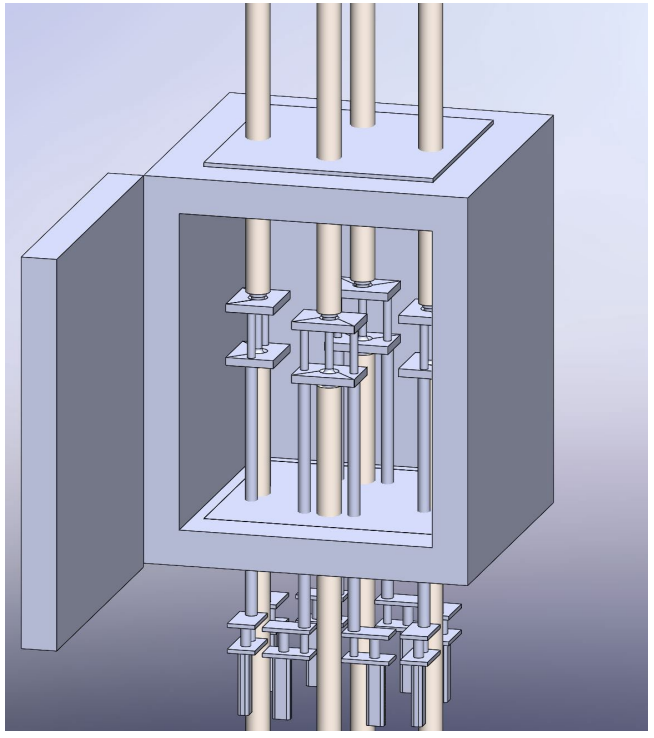


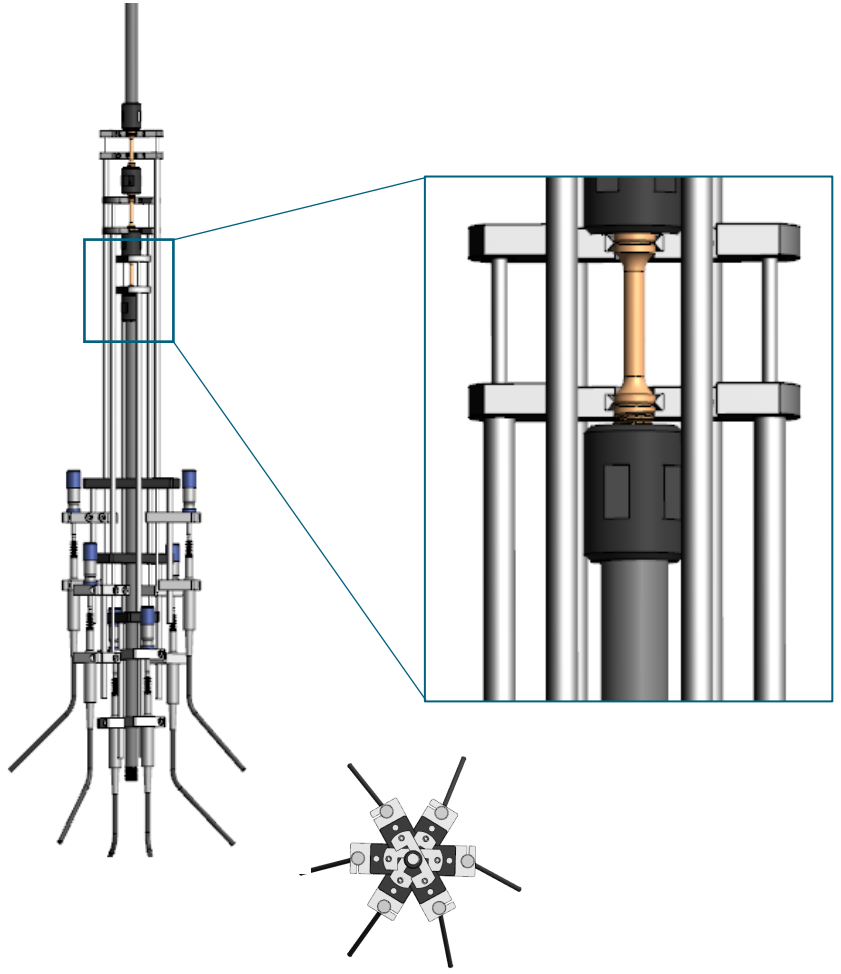
# Accelerating creep testing

- Challenges: Can't just use higher temperatures or higher stresses to accelerate testing
  - Potential for significantly different mechanisms at lower stresses/temperatures
  - ASME allows extrapolating rupture times out to a factor of 3-5
- Design concept
  - Parallel loading
  - Series loading




ASTM E8M small-size specimen #4 (#5)

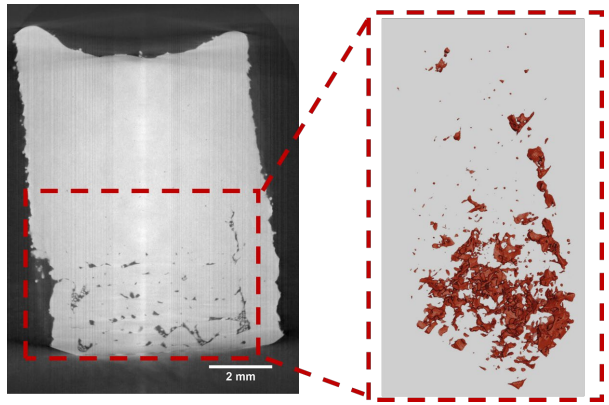




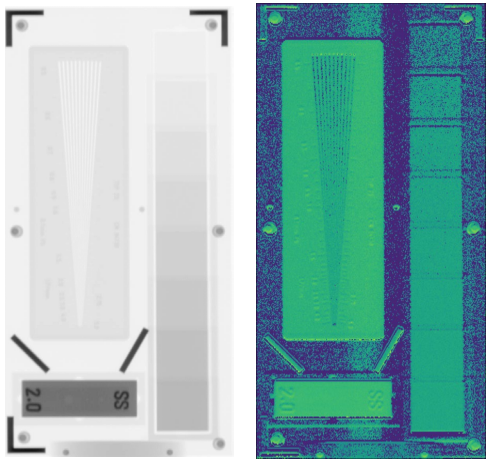
U.S. DEPARTMENT OF ENERGY | Office of NUCLEAR ENERGY

# NDE Techniques and Importance

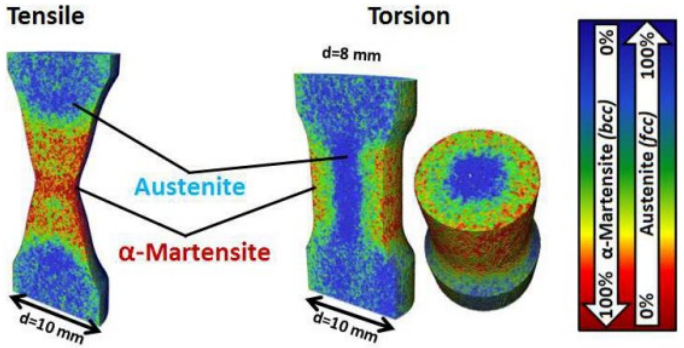
- Neutron/X-ray Radiography (2D) and Computed Tomography (3D)
  - Volumetric Imaging
- Dual-Energy X-ray Tomography
  - Material Identification
- Neutron Scattering
  - Microstructure Information
  - Phase Identification
- Photo-Thermal Radiography
  - Thermal Properties Measurements
  - Porosity, Grain Size, Grain Boundary Density




**(Right)** A reconstructed slice of a DED SS 316 part with intentional defects. **(Left)** Segmented porosity can be quantitatively analyzed.

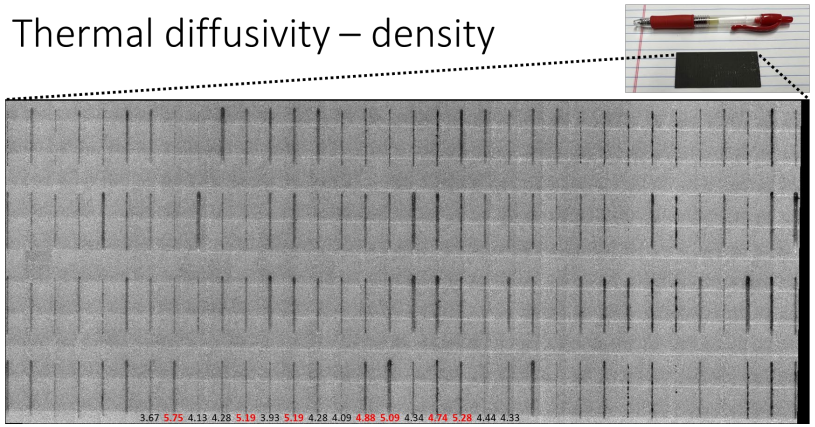



X-Ray image of the Image Quality Indicator (IQI) Phantom **(left)** and associated material identification results **(right)** displaying a map of the effective atomic number (Zeff) at each point in the region of interest.



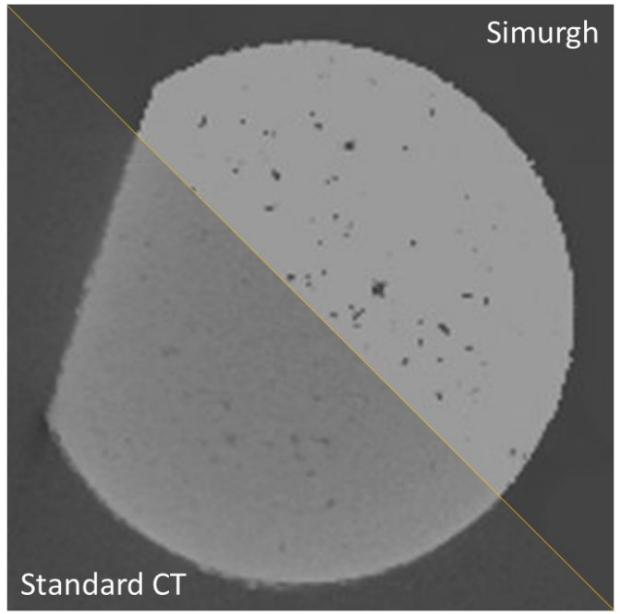

Example of Bragg Edge imaging.

Woracek, Robin, Dayakar Penumadu, Nikolay Kardjilov, Andre Hilger, Mirko Boin, John Banhart, and Ingo Manke. Physics Procedia 69 (2015): 227-236.



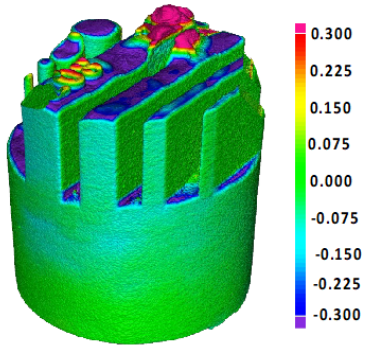

**(Right)** Measured thermal diffusivity [$mm^2/s$] at several tracks. The values larger than 4.6 $mm^2/s$ (average thermal diffusivity of the measured tracks) are highlighted in red. Denser tracks generally have higher thermal diffusivity.

# Fast Automated Characterization

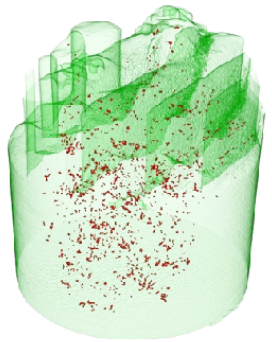



- Faster Scan
- Lower Cost and Labor
- Higher Quality at no computational cost
- GAN-based synthetic data, no data curation
- High-throughput Non-destructive Characterization
- Enhanced Detection and reduced analysis complexity

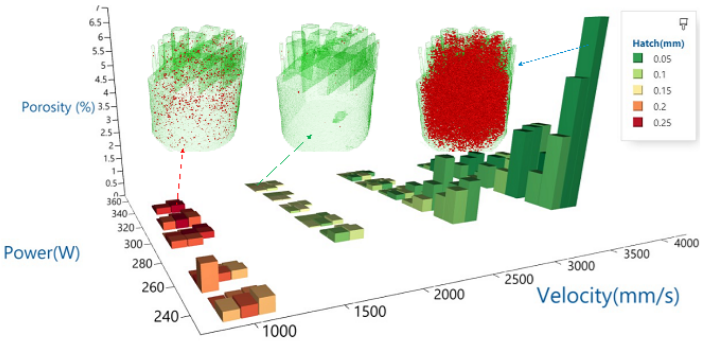
1. Design of Experiment (DoE) Parameters (**A Novel Material**)
2. Print the build plate. Remove parts from plate: (~100 parts per plate)
3. X-ray CT **at fraction** of optimal scan time based on material
4. **Fast, consistent and accurate AI-Based X-ray CT Reconstruction**
5. Analysis (segmentation, flaw detection, porosity & morphology,Metrology)



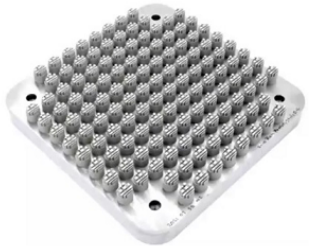

Deviation Map



Segmented Flaws

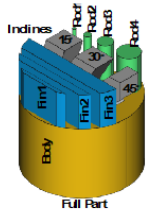


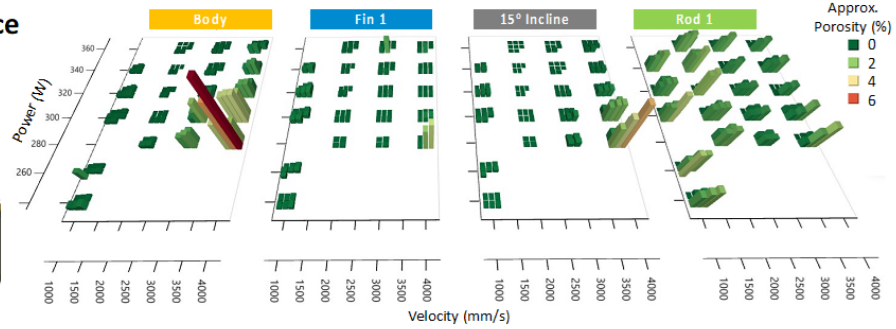

Window of desired process parameters for the new alloy



Build Plate: Each coupon a Process Parameter

Geometry dependence of optimum process parameters



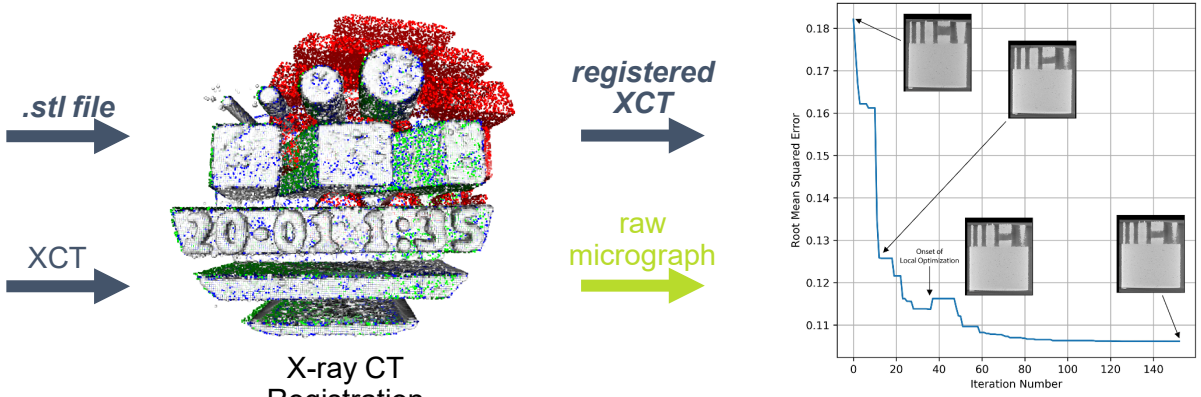

- Rapid Automated Characterization for Process Parameter Selection
- Integrated with in-situ monitoring process

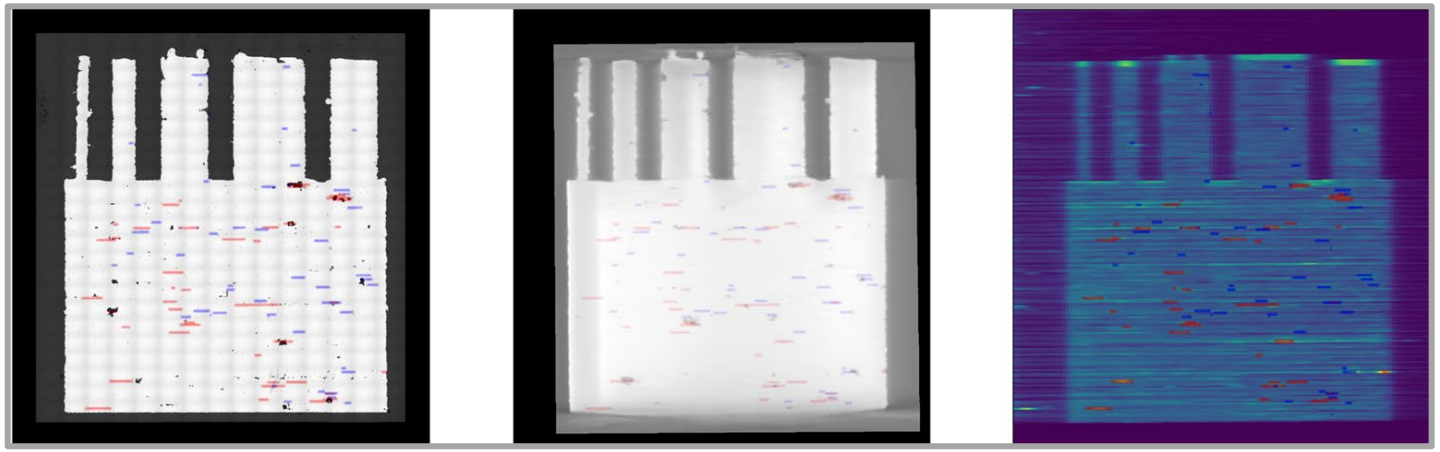
U.S. DEPARTMENT OF ENERGY | Office of NUCLEAR ENERGY

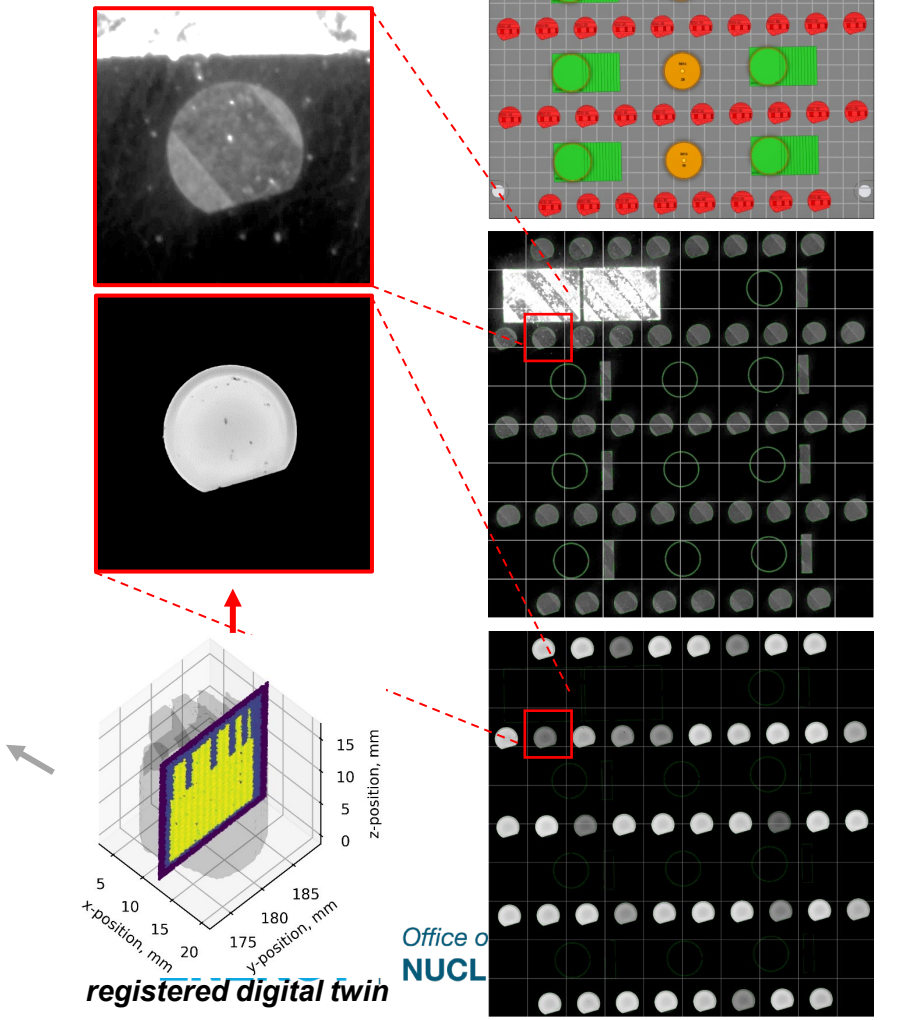
# Registration of In Situ Sensing and Characterization Data

MDDC

Post-build characterization data (e.g., rapid X-ray CT, microscopy) can be registered to the build plate coordinate system to link to sensing data and bolster understanding of **process-structure-property-performance** relationships



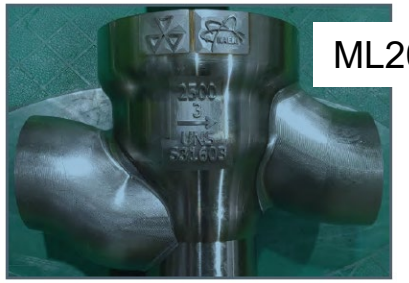



# Adapting to Post-Process NDE Industry Applications

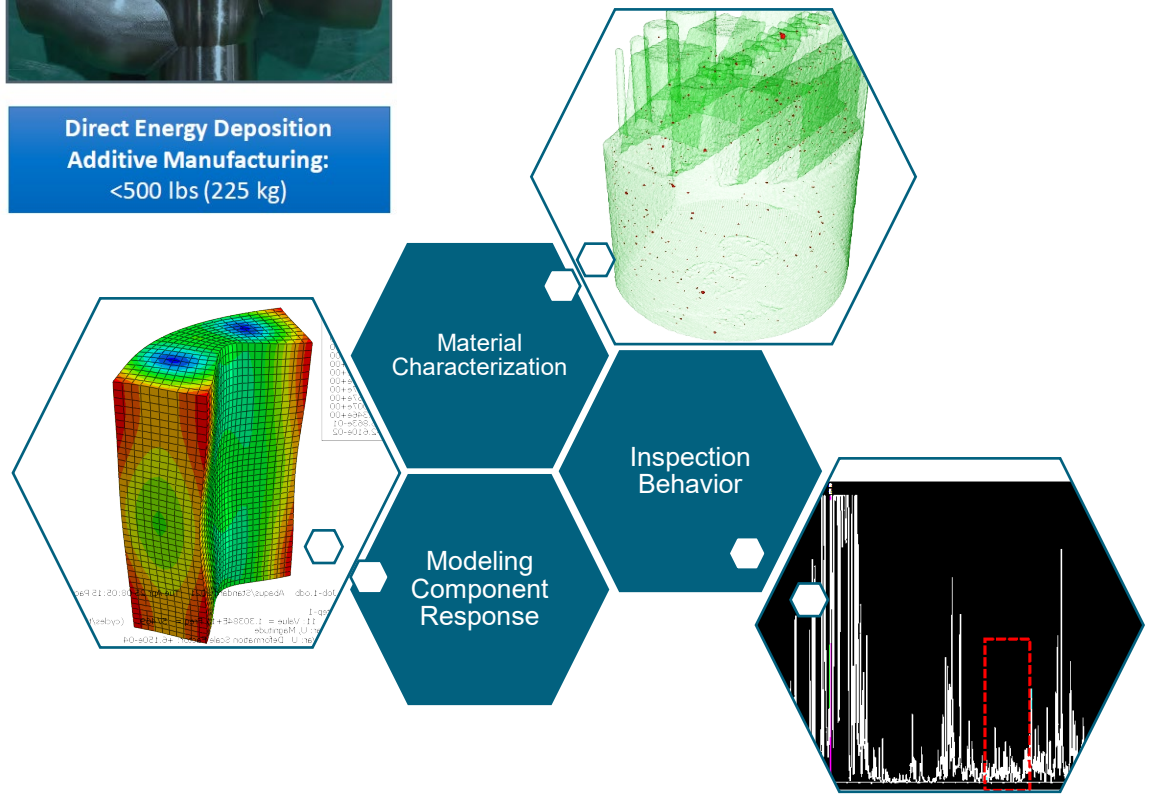
- Address the challenges of scalability
  - Component size and complexity
  - In-service conditions, access, etc.
  - Changes/features of interest

- Multi-scale/multi-discipline approach is needed
  - Use micro X-CT/n-CT to characterize microstructural features of AM materials
  - Computational modeling to understand effects on material/component performance
  - Use resonance ultrasound approaches to assess impacts of microstructural variations or defects

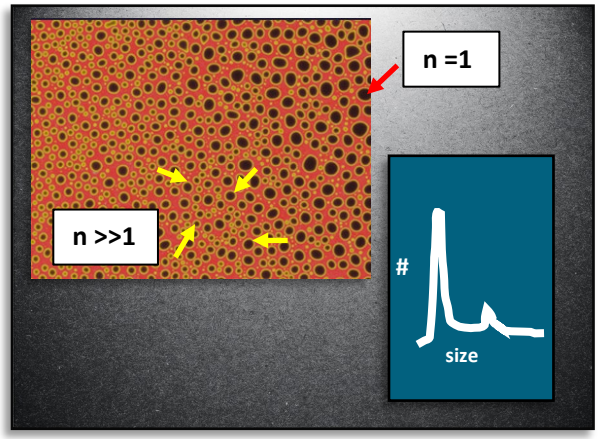


ML20342A035

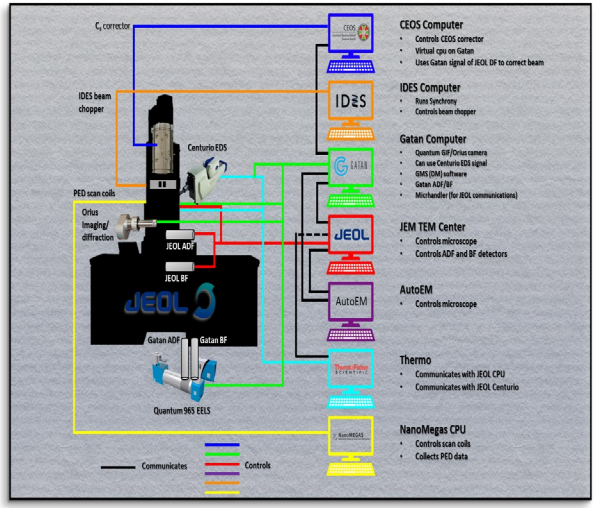
**Direct Energy Deposition Additive Manufacturing:**
<500 lbs (225 kg)



# High Res TEM Data: On a Budget

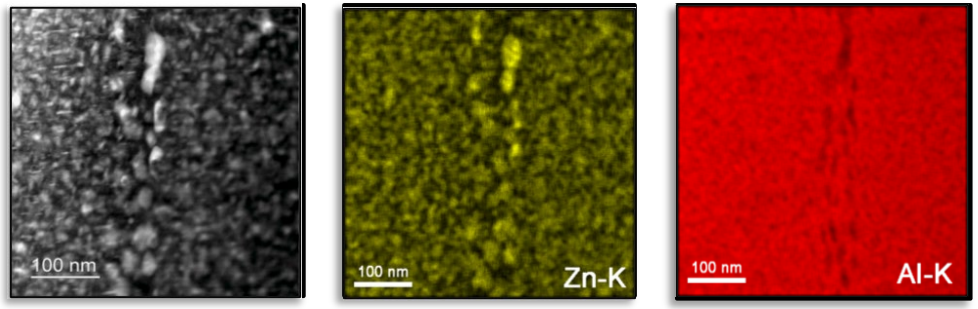



**The ability to collect large TEM data sets**

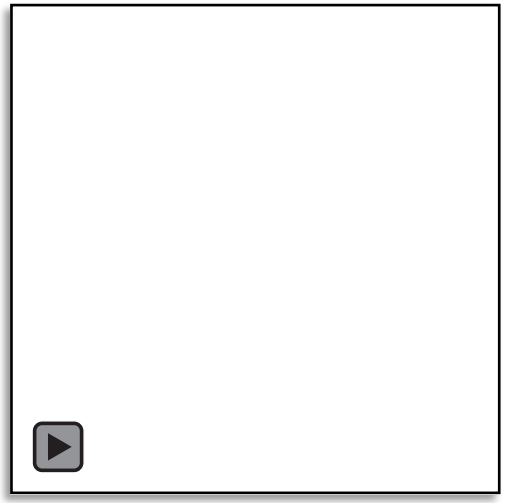


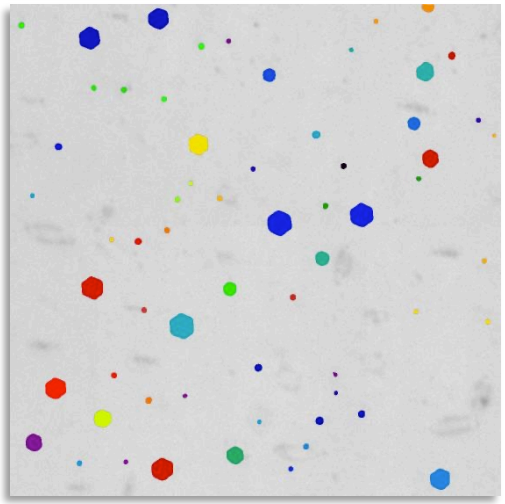

**Complex/Multimodal Control**

- An Automated Solution:
  - Overcome n = 1
  - Create an automated framework that provides customizable data analysis packages
  - ML/AI integration
- Providing necessary data for development of fundamental models
- Predicting Failure Before it Happens




**Nano-Chemistry in Motion**



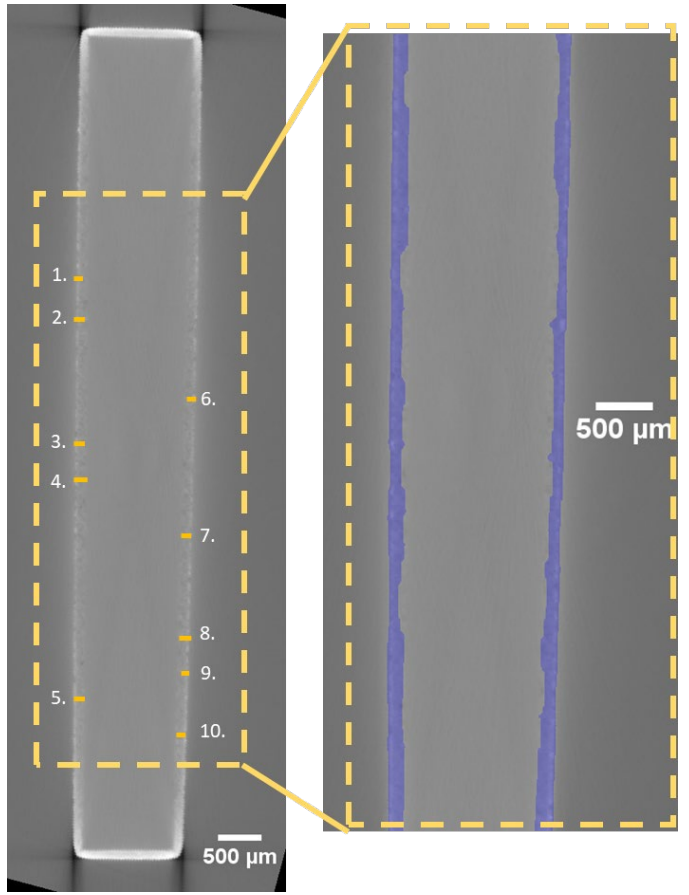


U.S. DEPARTMENT OF ENERGY

**Computer Vision: Auto Data Labeling**

# Future Work

- Continue developing accelerated testing and characterization techniques and work with Code Committees for methods for inclusion in qualification processes.
- Determine appropriate NDE methods for in-situ fabrication monitoring, QA/QC applications, and in-service inspections.
- Incorporating characterization and NDE data into long-term models to predict part lifetime, failures, etc.
- Correlate in-service NDE monitoring to initial destructive testing of AM parts.
- Link qualification pathways with accelerated testing and characterization methods




U.S. DEPARTMENT OF
ENERGY
Office of
NUCLEAR ENERGY