

Embedded Sensors for Advanced Reactor Systems Workshop, August 27- 28, 2024

# Advanced Materials and Manufacturing Technologies (AMMT) Program

**Meimei Li**, National Technical Director, Argonne National Laboratory

**Isabella van Rooyen**, Technical Area Lead for Advanced Materials & Manufacturing, Pacific Northwest National Laboratory

**Ryan Dehoff**, Technical Area Lead for Rapid Qualification, Oak Ridge National Laboratory

**Andrea Jokisaari**, Technical Area Lead for Environmental Effects, Idaho National Laboratory

**Dirk Cairns-Gallimore**, Federal Program Manager, Department of Energy, Office of Nuclear Energy

# Mission, Vision and Goals

## Mission

Accelerate the development, qualification, demonstration, and deployment of advanced materials and manufacturing in support of U.S. leadership in a broad range of nuclear energy applications.

## Vision

Expansion of reliable and economical nuclear energy enabled by advanced materials and manufacturing technologies.

## Goals

- Develop advanced materials & manufacturing technologies.
- Establish and demonstrate a rapid qualification framework.
- Evaluate materials performance in nuclear environments.
- Accelerate commercialization through technology demonstration.

# Technical Areas

## Advanced Materials & Manufacturing

- Advanced Materials Development
- Advanced Manufacturing Technologies
- Traditional Manufacturing Methods

## Rapid Qualification

- Processing-Structure-Property-Performance Qualification Framework
- High-temperature Materials Qualification
- Advanced Manufacturing Qualification

## Environmental Effects

- Radiation Effects
- Corrosion Effects in Nuclear Environments
- Materials Surveillance Technologies

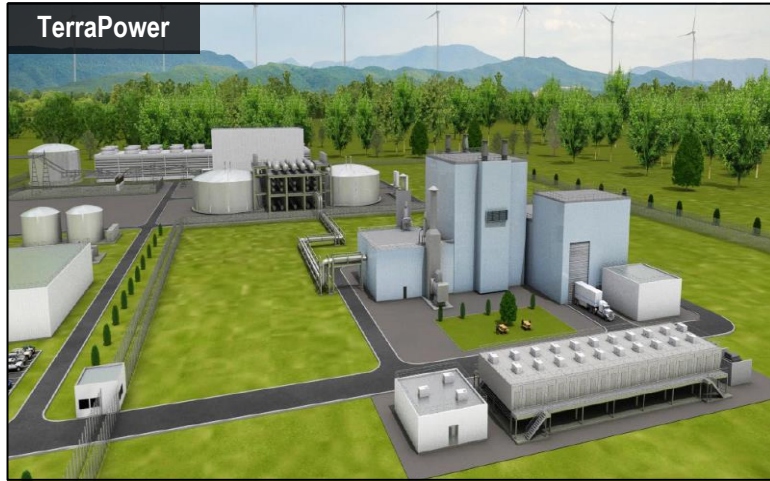
## Technology Demonstration

- Component Fabrication & Evaluation
- Large-scale Manufacturing & Supply Chain
- Codes, Standards & Regulatory Acceptance

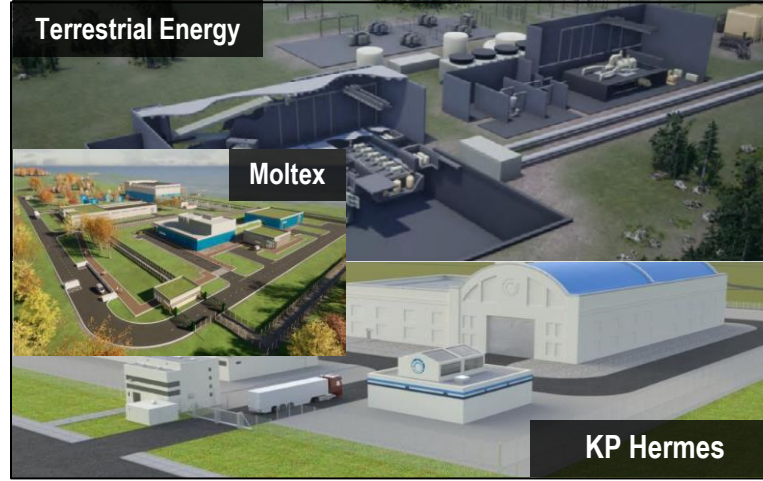
**Impact:** The AMMT program will provide the nuclear industry with next-generation high-performance materials and novel fabrication methods for expanded supply chains and demonstrate new technologies within the next decade.



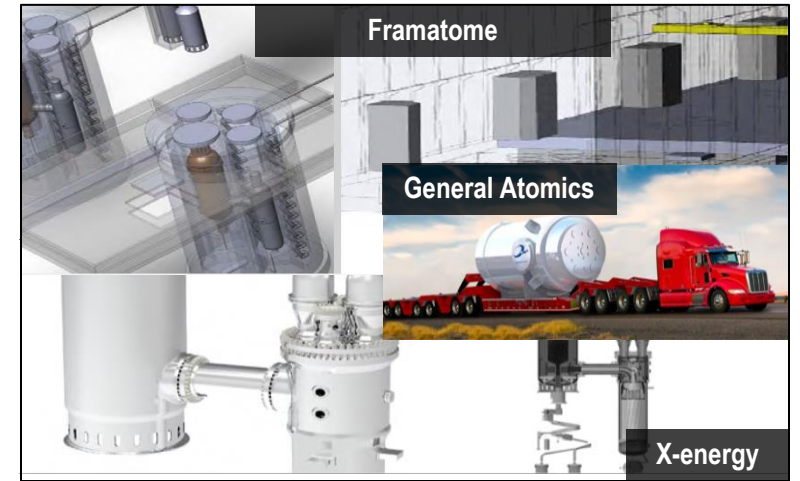
# AMMT Supports a Broad Range of Reactor Technologies



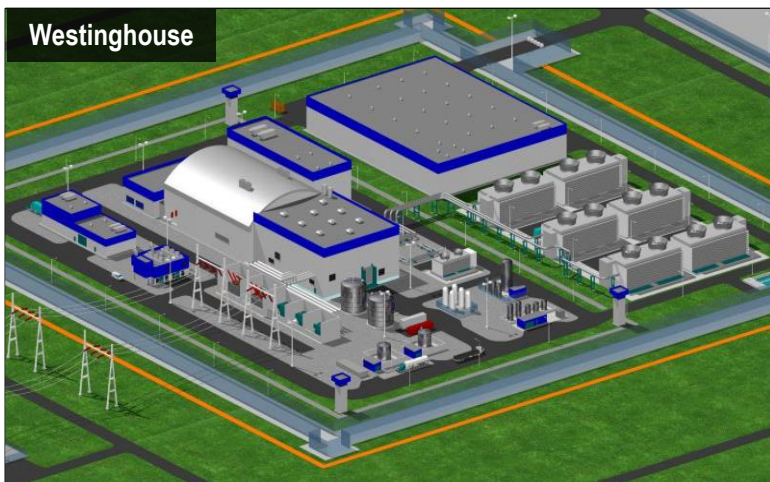
Sodium-cooled Fast Reactor (SFR)



Molten Salt Reactor (MSR)



Gas-cooled Reactor (GCR)



Lead-cooled Fast Reactor (LFR)



Advanced Light Water Reactor (ALWR)



Microreactor

# Advanced Materials & Manufacturing

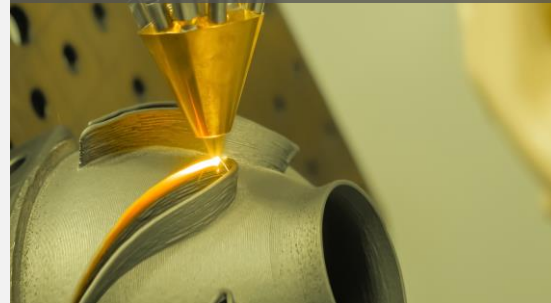


# Materials Processing for Nuclear Components

## Laser Powder Bed Fusion



## Direct Energy Deposition



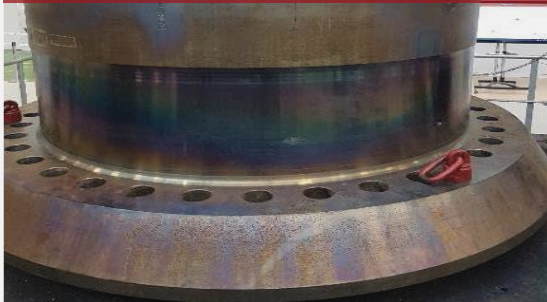
## PM-HIP



## Coating Technologies



## Advanced Materials Forming



## Advanced Materials Joining/Welding



## Hybrid Manufacturing



## Integrated Manufacturing Development

- Functional requirements, design and manufacturing integration
- Process integration & optimization
- Data integration (in-process monitoring & AI)

# Integrated Materials & Manufacturing Development

Develop and optimize materials through composition and processing to achieve high performance.

- **Expand applications of nuclear reactor materials**
  - Optimize advanced manufacturing processes to expand the applications of current reactor materials.
- **Optimize non-nuclear commercial materials for nuclear applications**
  - Optimize materials for enhanced resistance to nuclear environments to enable their nuclear applications.
- **Develop innovative new materials**
  - Explore new material design concepts.
  - Explore multi-material, multi-functional designs and fabrication enabled by advanced manufacturing.

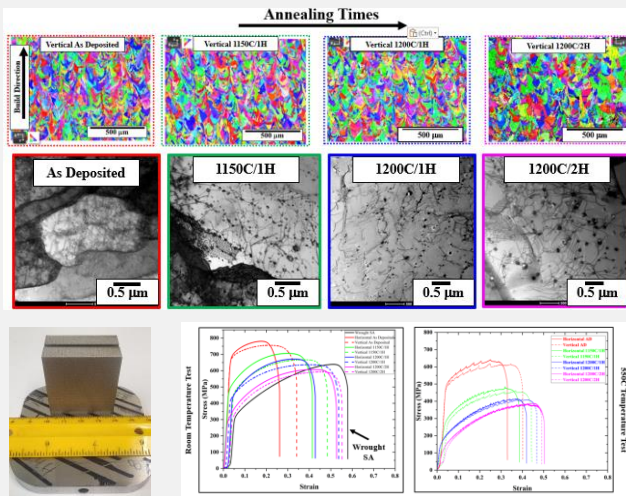
## Materials of interest

- Fe-based alloys
- Ni-based alloys
- Refractory alloys
  
- ODS alloys
- High entropy alloys
  
- Functionally graded materials
- Cladding/coating materials

# Development of Fe-based Alloys

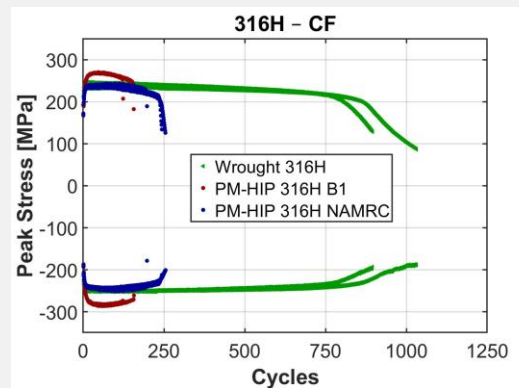
## AM A709, G91/G92

- Enable additive manufacturing of Code-qualified materials.
- Optimize LPBF and DED process and post-process treatment to achieve better high temperature properties than wrought alloys.



## PM-HIP 316H

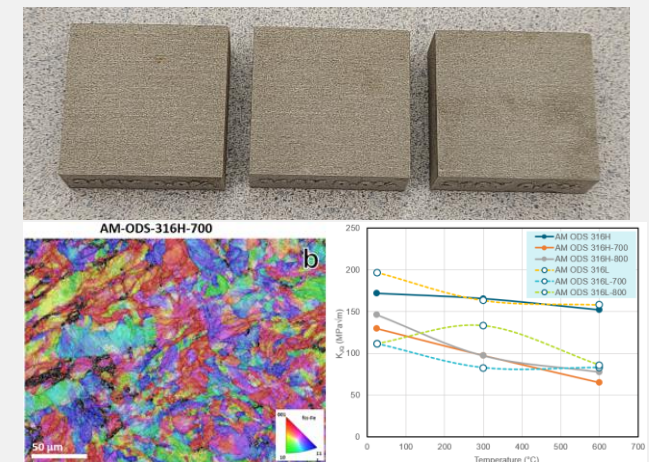
- Support microreactor high-temperature structural component fabrication.
- Achieve high temperature mechanical properties of PM-HIP comparable to or better than wrought materials.



PM-HIP 316 showed drastically reduced creep-fatigue lives compared to wrought 316.

## LPBF ODS 316

- Demonstrate high-quality ODS alloys can be manufactured using alternative processing routes without the need for mechanical milling.
- Successfully demonstrated the LPBF processing of 316Y + oxide mixture.





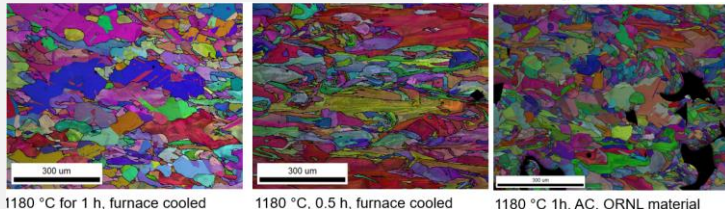
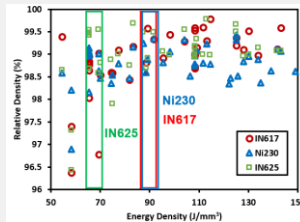
# Development of Ni-based Alloys

- Currently considered three Ni-based alloy categories based on potential applications.

Low-Cr molten salt compatible	Low-Cr for reactor internals	High-temperature strength
Hastelloy N	Alloy 718	Alloy 617
Alloy 244	Alloy 625	Alloy 282

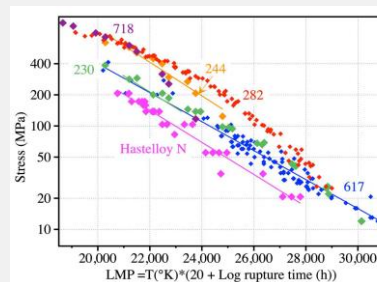
## Process Optimization

- Optimize LPBF processing parameters & post-process treatment.



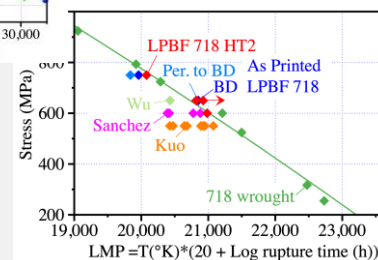
LPBF 282 post-build heat treatment optimization.

## Creep Property Evaluation



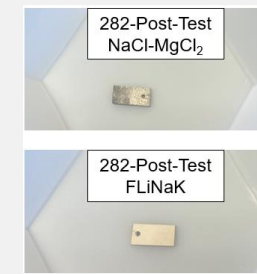
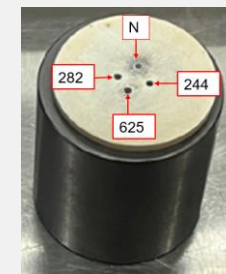
- AM and wrought alloys show similar creep strength.

- AM alloys have low creep ductility.



## Molten Salt Corrosion Tests

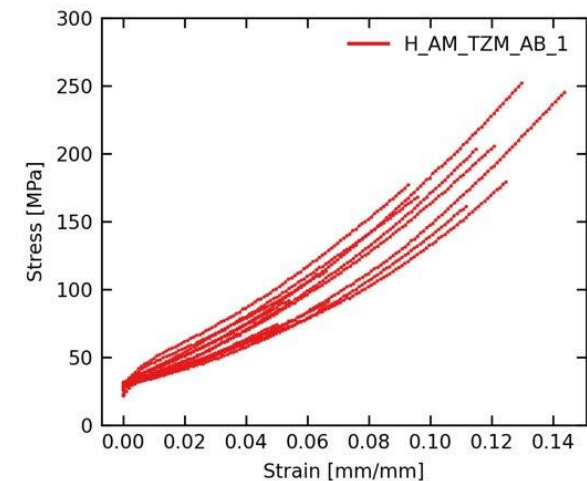
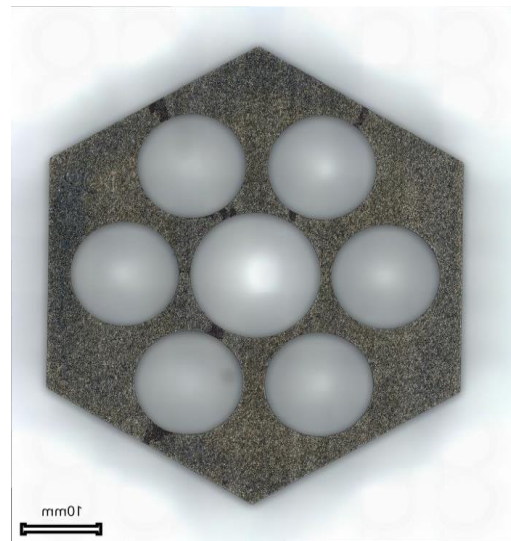
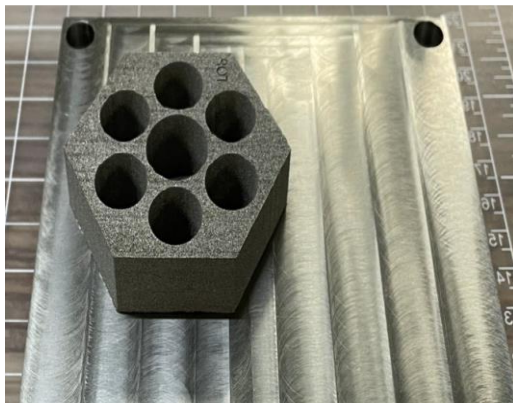
- Corrosion at 750°C for 1,000 h in NaCl-MgCl<sub>2</sub> and FLiNaK.
- Weight loss: 625 > 282 > 244 ≈ N



Crucible test

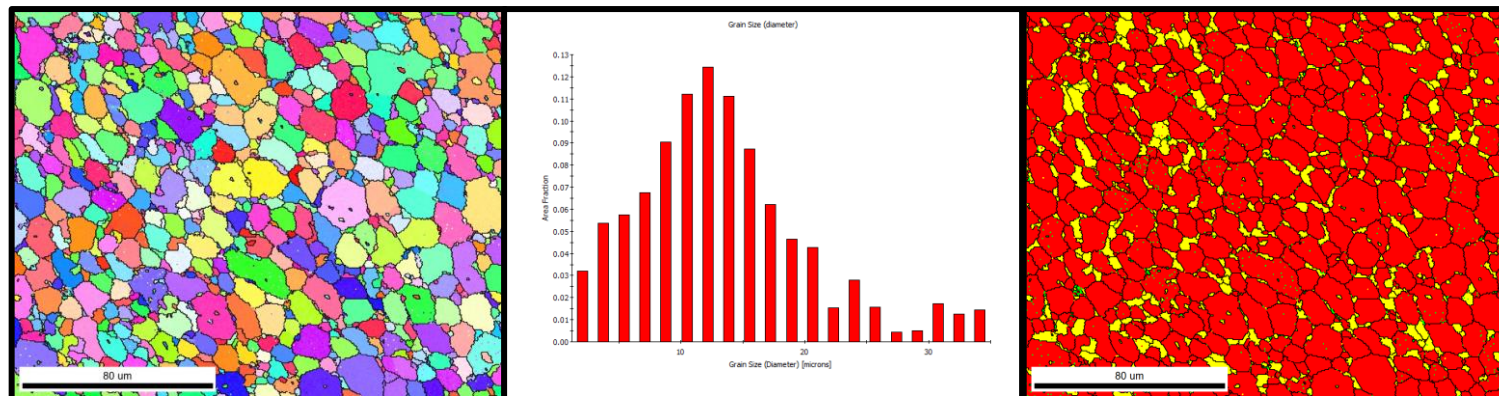
# Development of Refractory Alloy – AM TZM

- Refractory alloys are considered to provide material option with higher operating temperatures.
- Molybdenum alloys
  - Offer high corrosion resistance and higher thermal conductivity. Reduce thermal constraint of fuel elements
  - Drawback is more expensive and difficult to fabricate.
- Explore additive manufacturing of refractory alloys to overcome difficulty in conventional fabrication.
- Demonstrate capability of additive manufacturing of TZM in support of microreactors.



# Development of High Entropy Alloys (HEAs)

- **US-Czech Bilateral Project: Additive Manufacturing of High Entropy Alloys.**
  - Project focuses on additive manufacturing of HEAs for high-temperature nuclear applications, in particular on manufacturing of near net-shape products. Tasks include:
    - HEA design - AlMoNbTiZr with Cr, Fe, W additions.
    - AM manufacturing of HEAs and process optimization.
    - Microstructure and mechanical property testing to select the most promising alloys and processing conditions.
    - Corrosion studies.
    - Neutron and ion irradiation studies.
    - Near-net shape AM of components.



Phase	Total Fraction	Partition Fraction
Body Centered Cubic	0.857	0.857
Titanium (Alpha)	0.010	0.010
Titanium Carbide	0.133	0.133

- Matrix phase is BCC.
- Particles are indexed as TiC but needs validation.

Characterization of as sintered Al<sub>15</sub>Mo<sub>30</sub>Nb<sub>30</sub>Ti<sub>25</sub>



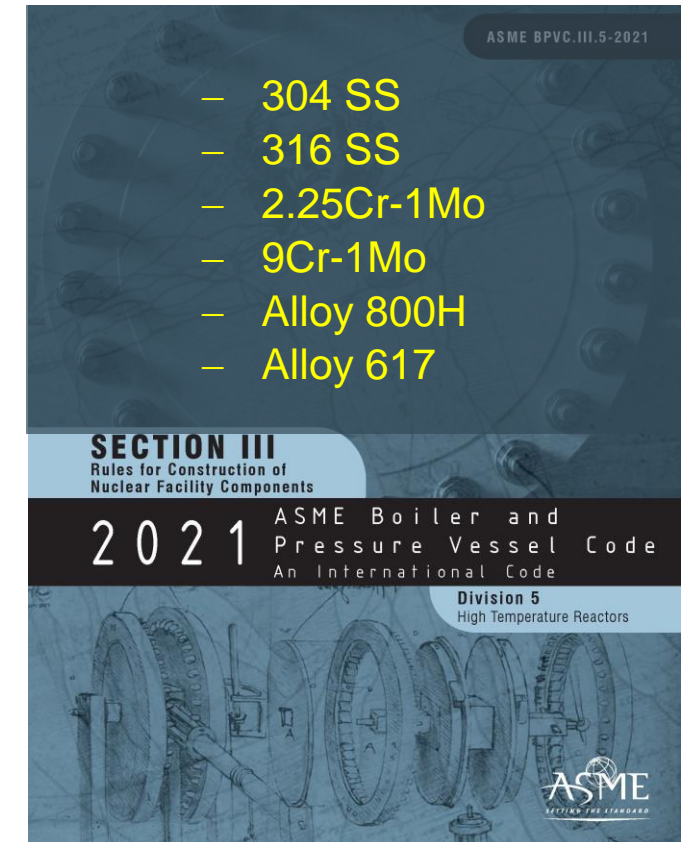
# Rapid Qualification

# Focus on Qualification

## Qualification of High Temperature Materials for Nuclear Construction

Code qualifying a new material for high temperature nuclear structural design is a lengthy process.

- ASME Section III, Rules for Construction of Nuclear Facility Components - Division 5, High Temperature Reactors.
  - Governs the construction of structural components for use in high temperature reactors including GCR, SFR, LFR, MSR.
  - Specifies the mechanical properties and allowable stresses to be used for design of components in high temperature reactors.
- Only **six materials** have been approved for elevated-temperature nuclear construction in Section III Division 5.
- Approval of a new material under the Code involves rigorous testing, documentation, and approval processes.
  - Specification, product form, size/thickness, heat treatment, metallurgical structure.
  - Data are generated from at least 3 different “heats” of the material.
  - Exhaustive mechanical property tests (tension, creep, fatigue, creep-fatigue, etc.).
  - For time-dependent properties (e.g. creep), allowed time extrapolation factor, 3-5.
  - Consider long-term properties stability, e.g. structural stability due to thermal aging.



# Focus on Qualification

## Qualification for Nuclear Environmental Effects

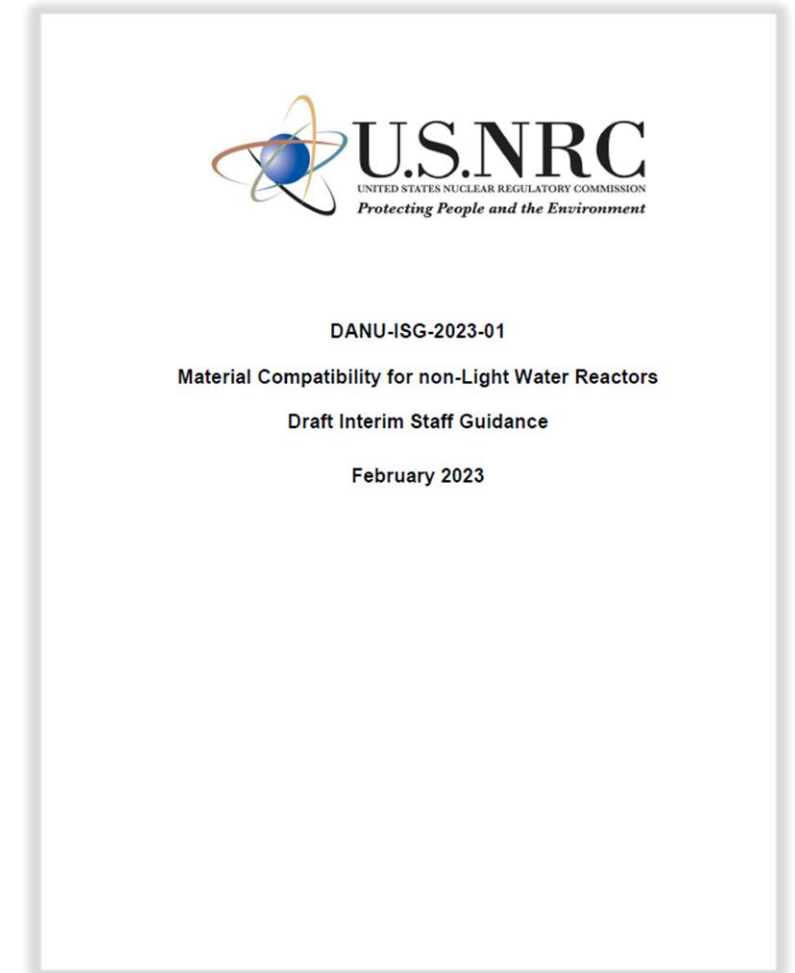
- ASME Code rules do not provide methods to evaluate deterioration from corrosion, mass transfer phenomena, or radiation effects.
- NRC has issued “**Material Compatibility for non-Light Water Reactor Draft Interim Staff Guidance**” as part of its evaluation of a non-LWR application to review applicable design requirements including environmental compatibility, qualification and monitoring programs for safety-significant structures, systems, and components (SSCs).
- Demonstrates material performance to satisfy regulatory requirements.

### Irradiation Effects

- The cost and time required for neutron irradiation and post-irradiation examinations only allow for exploration of a limited set of metallurgical and irradiation conditions. Extrapolation of data beyond testing conditions and prediction of long-term performance is a challenge.

### Corrosion Effects

- Comprehensively evaluate the corrosion performance of materials in various reactor coolant environments (e.g. molten salt, sodium, lead, helium) is a challenge.





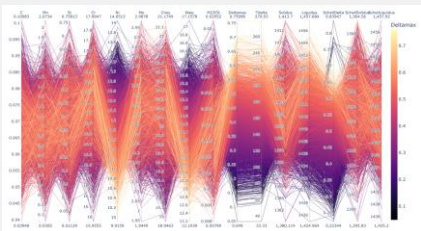
# Focus on Qualification

## Qualification of AM Materials for Nuclear Applications

### Qualification of AM materials/components for nuclear applications needs a paradigm shift.

- Adding AM materials into ASME Sec III Div 5 is critical to the wide adoption of AM technologies in advanced reactors.
- Qualification of materials made by advanced manufacturing (AM) processes (e.g. laser powder bed fusion, directed energy deposition) for nuclear applications presents a new challenge.
- Unlike traditional manufacturing, additive manufacturing creates geometric forms simultaneously with the material.
- Additive manufacturing is a highly-localized process, creating spatial variations in response to differences in geometry and processing conditions.
- Materials properties are related to the component geometries and may vary throughout the component.
- AM plays a crucial role in accelerating qualification by introducing new challenges requiring innovative approaches.

### Sources of Variability

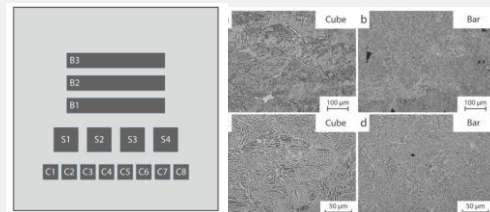


Kannan et al. IMMI, 2022 (SS316L)

Feedstock Chemistry

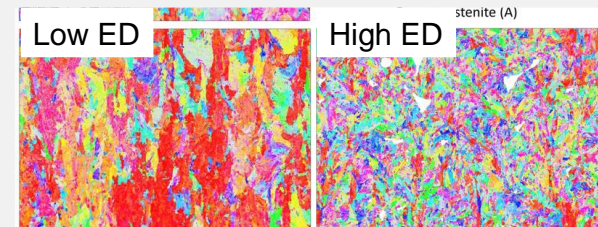


Machine Variability



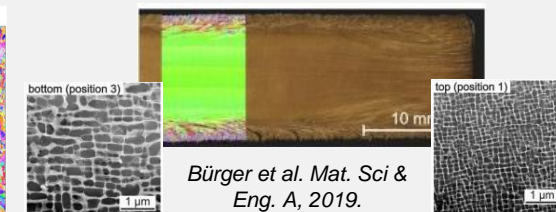
Nandwana et al. Mat. Today Comm., 2020 (Ti64)

Geometry Effects



Kannan et al. Met Trans A, 2023 (M300 steel)

Process Variables



Bürger et al. Mat. Sci & Eng. A, 2019.

Spatial Variation Within Build

# Rapid Qualification Framework

A science-based engineering approach: combining scientific understanding with fundamental database development.



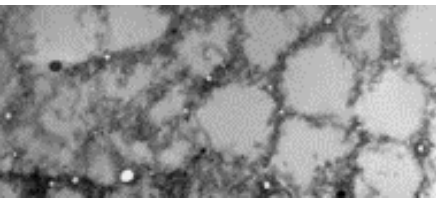
## Develop **Processing-Structure-Property-Performance** based qualification framework

A P-S-P-P based qualification framework requires a fundamental understanding of processing-structure-property-performance relationship through experimental and computational approaches.



## Use integrated experimental, modeling and data-driven tools

Capitalize on the wealth of digital manufacturing data, integrated computational materials engineering (ICME) and machine learning/artificial intelligence (ML/AI) tools, and accelerated, high-throughput testing and characterization techniques.



## Integrate *in situ* process monitoring data into the qualification process

Monitor and analyze the printing process in real-time to ensure the quality and integrity of the fabricated part. Use *in situ* process monitoring data to detect defects and as a QA tool to assess part quality.



## Demonstrate accelerated qualification methods through qualifying LPBF 316H SS

Laser powder bed fusion 316H stainless steel (LPBF 316H SS) serves as a test case for demonstration of a new qualification framework.

# Technical Basis For Qualification of AM 316H SS

## Process Understanding

- Understand the effects of variations in feedstock materials.
- Understand the effects of processing and post-processing conditions on residual stress, defects, and microstructure.
- Understand the location- and orientation-dependence of microstructure and properties.
- Understand the effects of component geometry on component quality and consistency.
- Understand machine-to-machine variability and repeatability.

## In-process & Post-process NDE

- Develop integrated in-process sensing, monitoring, and control technologies and demonstrate in situ detection of processing anomalies (dimension, surface finish, density, hot spots, defects).
- Develop advanced reliable and high-resolution NDE techniques for post-build inspection of parts with complex geometry.
- Use in situ processing data to complement/guide post-process NDE.
- Develop a pathway that will allow use of in-situ processing data records for nuclear component qualification and certification.

## Process-Structure-Property Correlation

- Generate microstructure and baseline mechanical property data of as-built and post-treated materials for model validation & verification and determine performance limits to support the ASME Code Case.
- Develop the correlation of in-situ process data with microstructure and mechanical properties.
- Establish defect acceptance criteria of fabricated components to meet performance requirements.
- Establish the correlation of mechanical properties with microstructural and material processing parameters with uncertainty quantification.

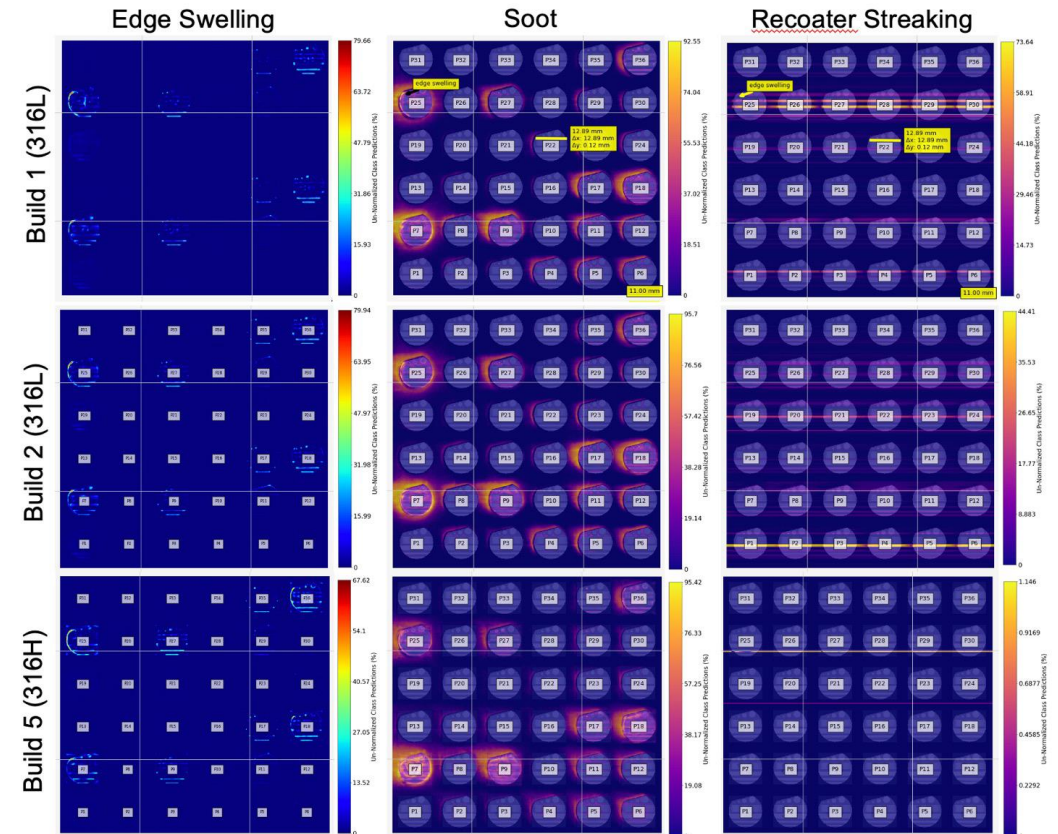
## Environmental Effects

- Understand the effects of composition and microstructure on irradiation creep, swelling, high-temperature He embrittlement and microstructural stability.
- Understand corrosion mechanisms in reactor environments.
- Understand the effects of microstructural variations resulting from different processing and post-processing conditions.
- Establish performance limits in nuclear environments using experimental data and modeling results.



# In-Process Monitoring

- Use *in situ* process monitoring data to detect defects and as a QA tool to assess part quality.
- Integrate *in situ* process monitoring data into the qualification process for AM nuclear applications.
- Work with ASME to understand the pathway for utilizing *in situ* process monitoring data for AM qualification.



Three classes of characteristic anomalies identified through *in situ* data collection.

# High-throughput NDE Characterization

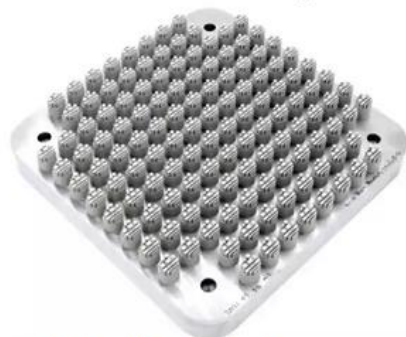
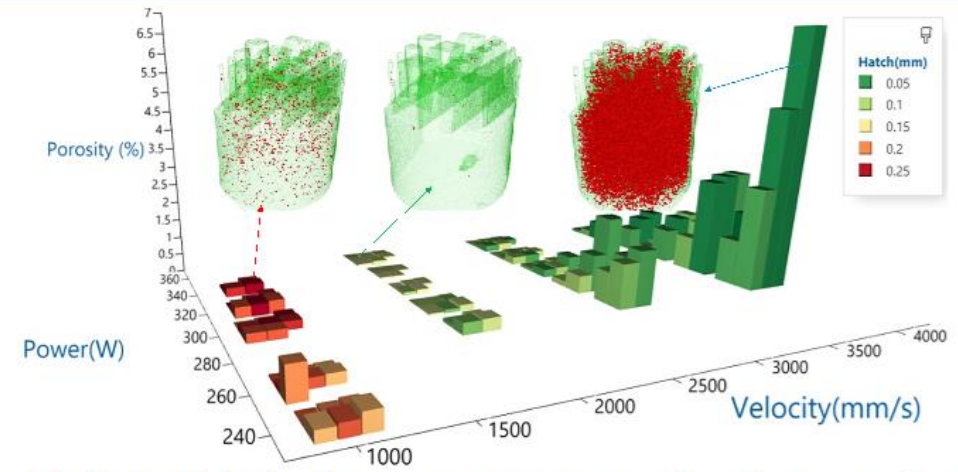
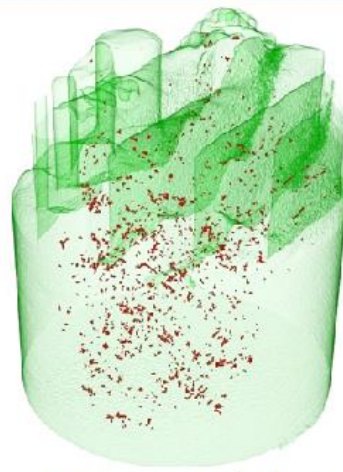
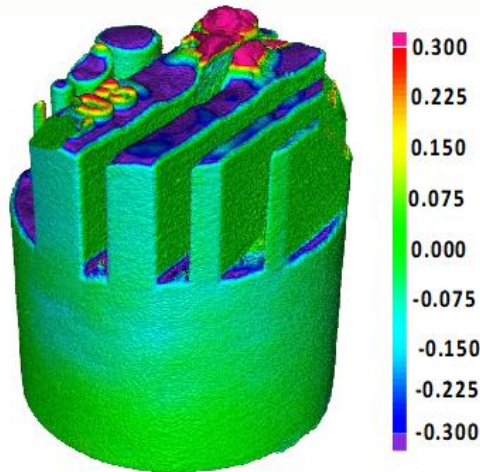
1. Design of Experiment (DoE)  
Parameters (**A Novel Material**)

2. Print the build plate. Remove parts from plate: (~100 parts per plate)

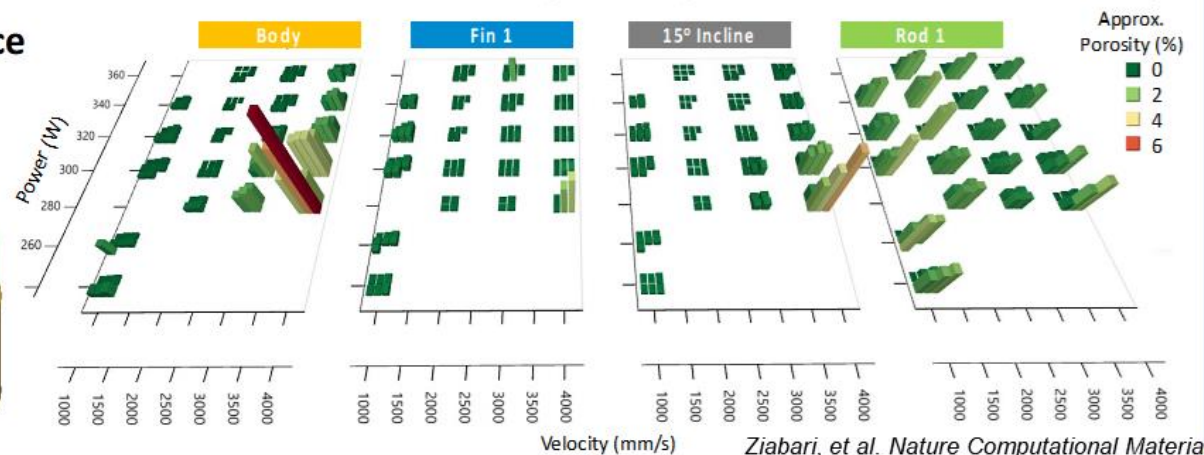
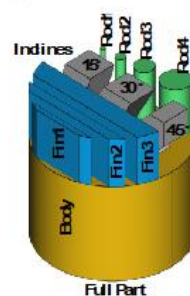
3. X-ray CT **at fraction** of optimal scan time based on material

4. **Fast, consistent and accurate AI-Based X-ray CT Reconstruction**

5. Analysis (segmentation, flaw detection, porosity & morphology, Metrology)



Geometry dependence of optimum process parameters

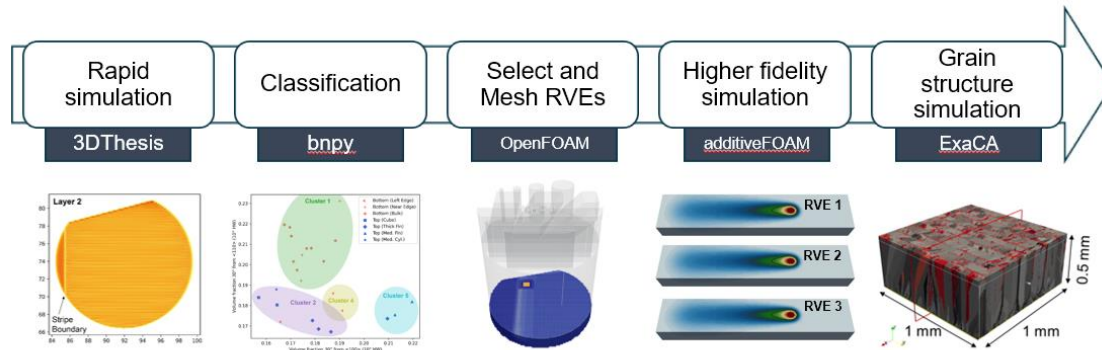


- Rapid automated characterization for process parameter selection
- Integrated with in-situ monitoring process



# Processing-Structure-Property Correlation

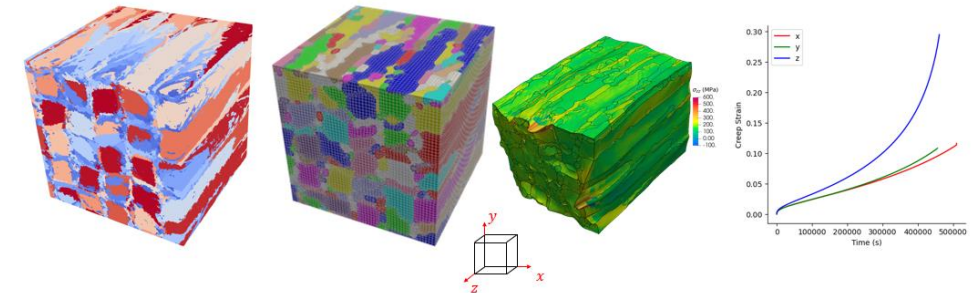
## Process modeling to predict AM microstructure



**Output:** Generate representative grain structure statistics for variation found in real parts

## Connecting process modeling to property predictions

- Process model may be used to generate synthetic microstructures



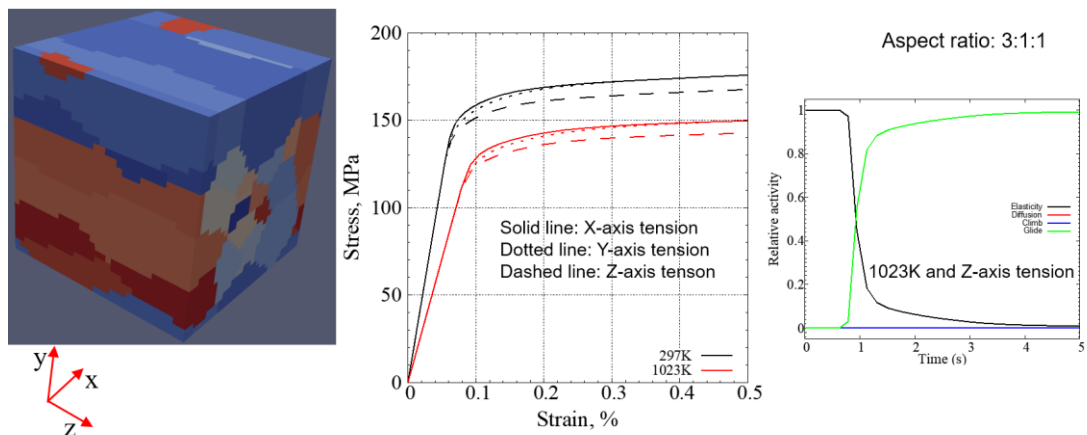
Microstructure from process model

Crystal plasticity finite element discretization

Microscale deformation and stress

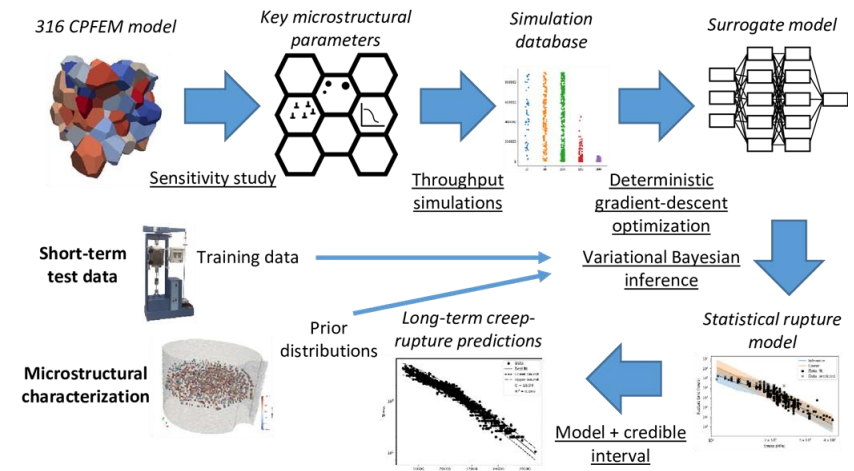
Macroscale predicted creep anisotropy

## Simulate tensile behavior of AM with columnar grains



Columnar grain structure develops significant anisotropy in the mechanical responses.

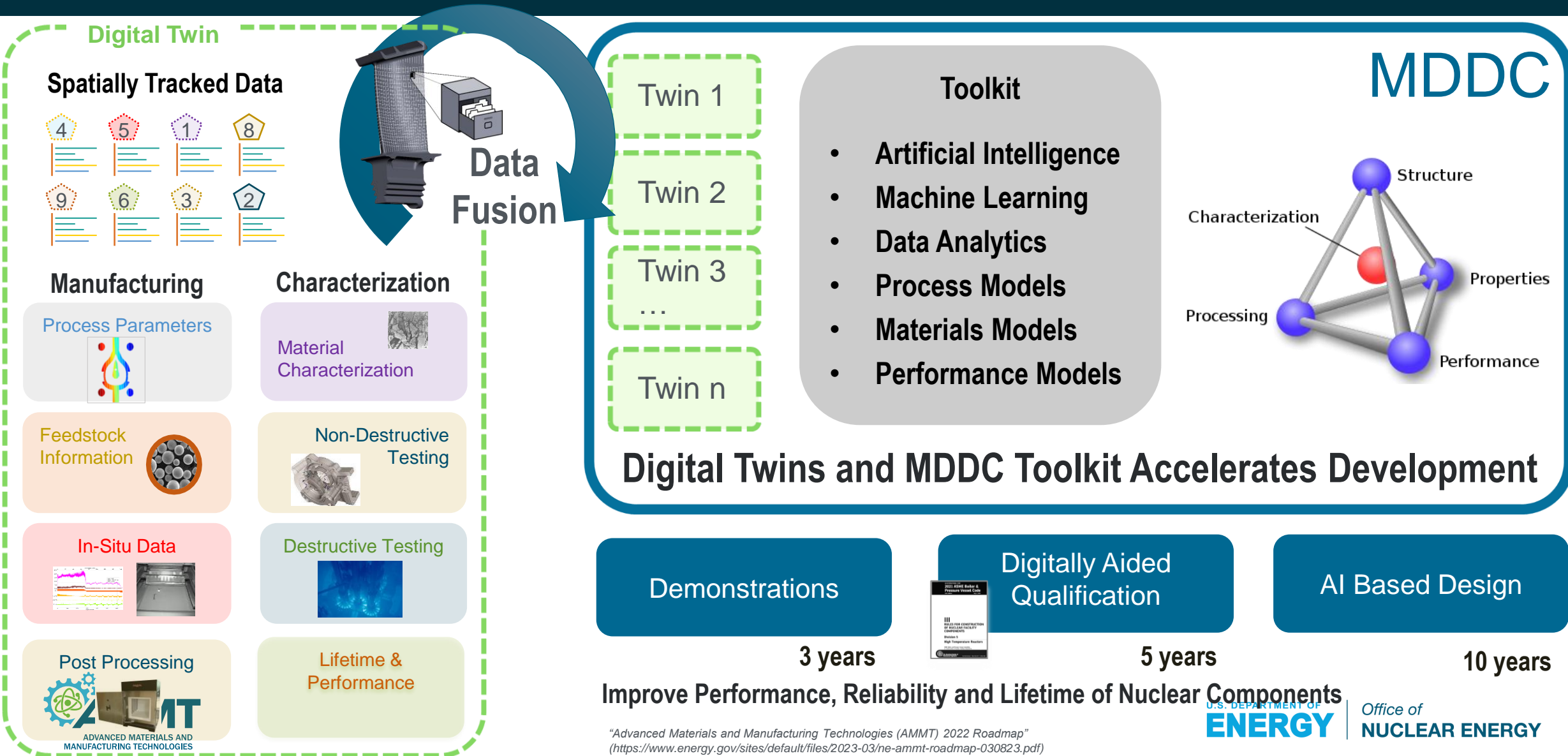
## Predict long-term creep rupture strength





# Data-Driven Qualification

Multi-Dimensional Data Correlation (MDDC)

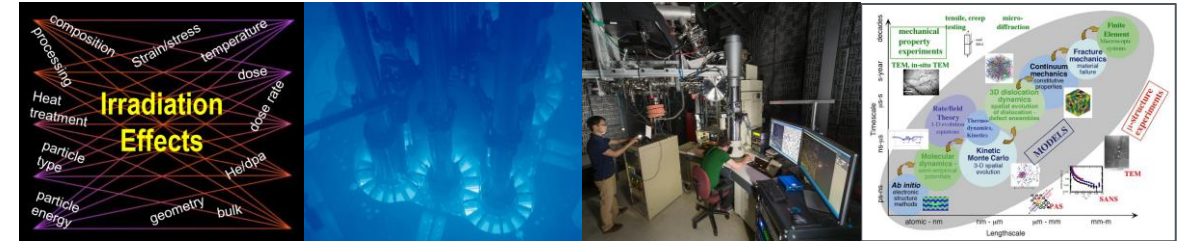


# Environmental Effects

# Environmental Effects

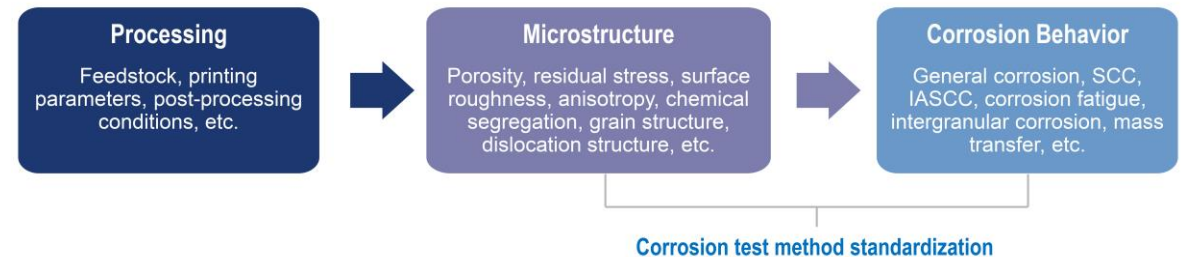
## Irradiation Effects

- Conduct neutron irradiation and PIE to evaluate the materials performance.
- Accelerate qualification for irradiation effects with the combination of neutron irradiation, ion irradiation and modeling data to cover a wide parameter space and understand and predict the material behavior beyond testing conditions.



## Corrosion Effects

- Evaluate corrosion performance in reactor coolant environments.
- Understand and predict the effects of defects and microstructural heterogeneities on corrosion behavior and determine the performance bounds.



## Materials Surveillance Testing

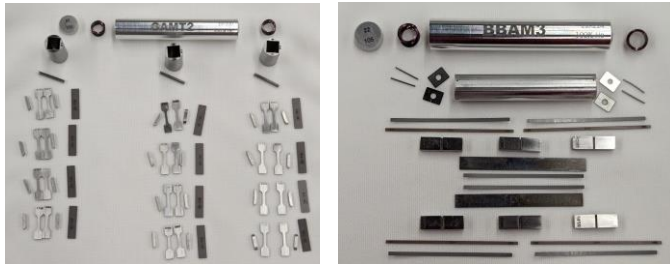
- Develop the technology necessary for implementing a surveillance program that monitors material degradation in service.



# Performance Evaluation under Neutron Irradiation

## AM 316H HIFR Irradiation

- 8 capsules were inserted in Cycle 506 (April 2024)
  - Irr. temp.: 400, 600°C
  - Doses: 2, 10 dpa
  - Materials: AM 316H, wrought 316H, wrought 709, AM 316L
  - Specimens: tensile, bend bars
- 12 capsules to be inserted in cycle 507 (June 2024)
- PIE of the 2 dpa capsules will start in July 2024.



## AM 316H ATR Irradiation

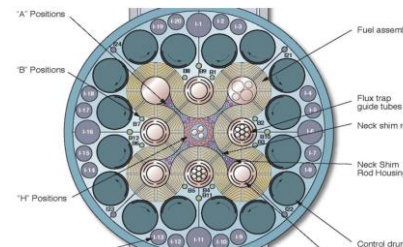
- Neutron irradiation experiments at the ATR are being planned (targeting Cycle 175-C, 06/2025).

### Irradiation Experiments

- Primarily thermal spectrum
- Pressurized water at ~50°C
- Experiment positions vary between 5/8" to 3" diameter
- Fast flux:  $8.1 \times 10^{13}$  n/cm<sup>2</sup>/s
- Thermal flux:  $2.5 \times 10^{14}$  n/cm<sup>2</sup>/s

### Specimens

- SS-J tensiles, bend bars, round compact tension

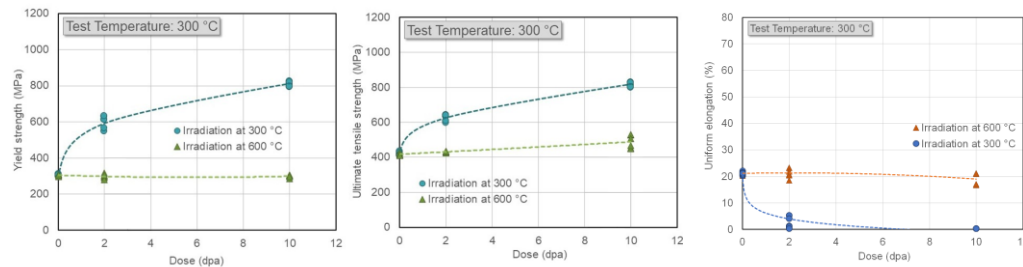


## A709 Irradiation Planning

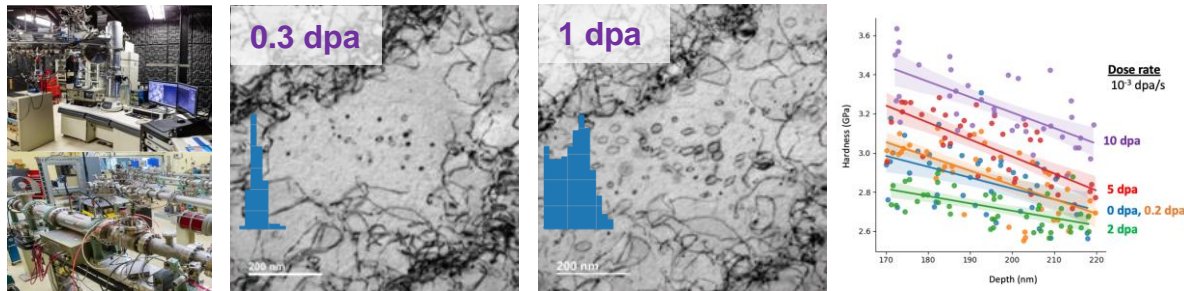
- Two work packages have been created to develop a neutron irradiation and PIE plan for A709.
- Funding was provided by the ART Fast Reactor Program.
- INL and ORNL will jointly develop a detailed plan for generating A709 irradiation data in support of advanced reactor license application and its integration with the AMMT current neutron irradiation campaign plan.

# Accelerate Qualification with Combined Ion & Neutron Irradiations and Modeling

**Neutron Irradiation:** Conducted neutron irradiation at 300 and 600°C at 0.2, 2 and 10 dpa. Included as-printed, stress-relieved, solution annealed, and wrought samples. Developed a preliminary understanding of microstructural evolution and irradiation effects on tensile properties of AM 316L SS.

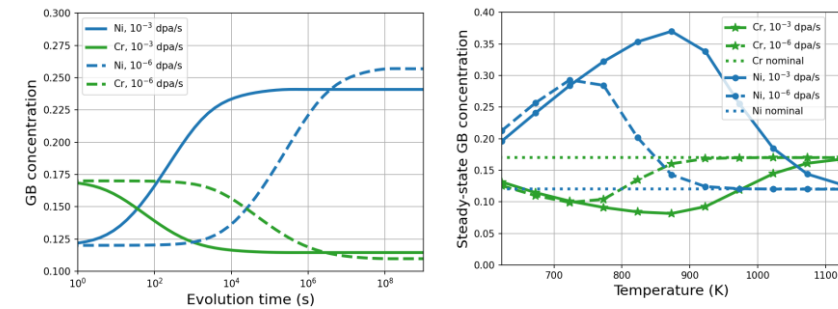


**Ex situ/in situ Ion Irradiation:** *Ex situ* ion irradiation creates a sample library of AM 316 SS to understand the effects of a large number of metallurgical and irradiation parameters; *in situ* ion irradiation with TEM provides high-fidelity data for modeling of irradiation-induced defect evolution in AM 316 SS.

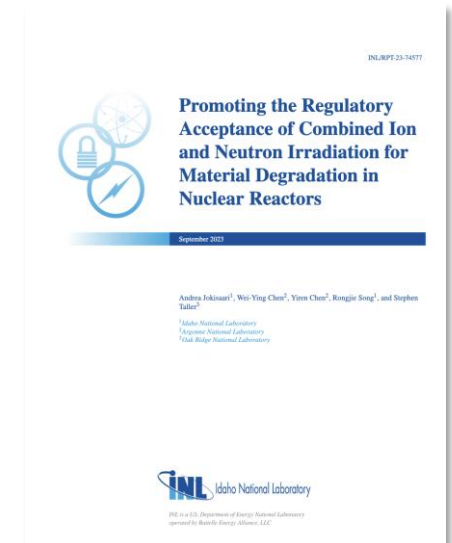
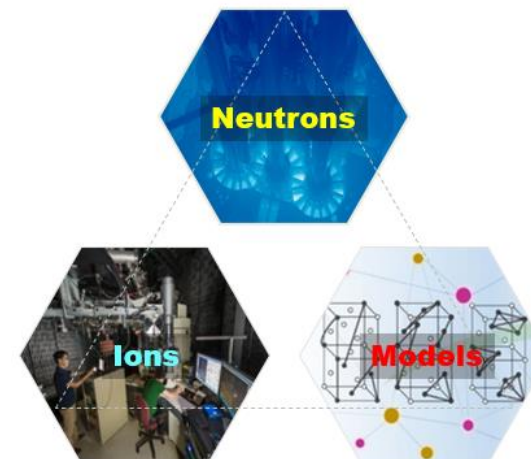


## Computer Modeling

Modeling of radiation-induced segregation (RIS) in AM 316 SS.



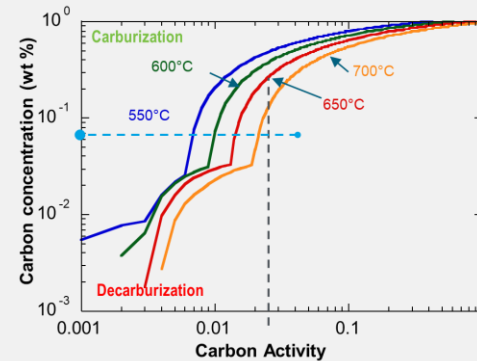
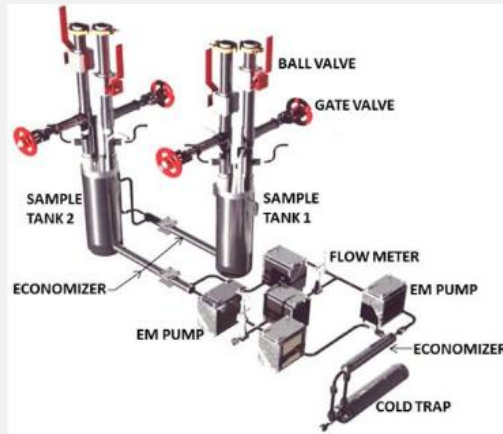
Effect of dose rate or particle type on RIS. a) Time to reach steady state concentration at 673 K. b) Combined effect of temperature and dose rate on the steady-state GB concentration.



# Corrosion Effects in Nuclear Environments

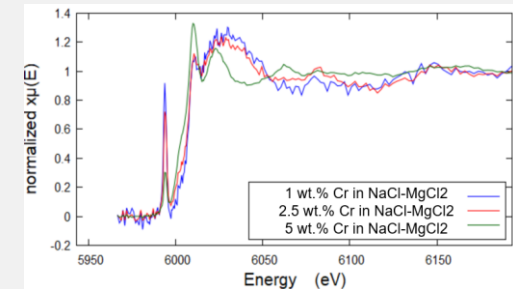
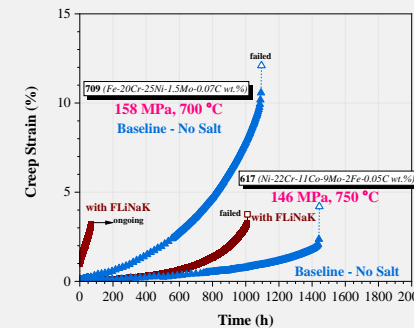
## Sodium Corrosion

Conduct A709 and AM 316H sodium exposure tests, corrosion measurements, microstructural characterization, high-temperature tensile tests, thermodynamic and kinetic calculations of carbon transfer behavior.



## Molten Salt Corrosion

- Evaluate impact of molten salts on creep rupture lifetime of 316H, A709, A617.
- X-ray absorption spectroscopy (XAS) of chemical speciation of Cr, Fe, Ni to understand corrosion kinetics of 316H and A617 in chloride salt.
- Evaluating corrosion performance in chloride molten salts for AM 316H processing parameters.

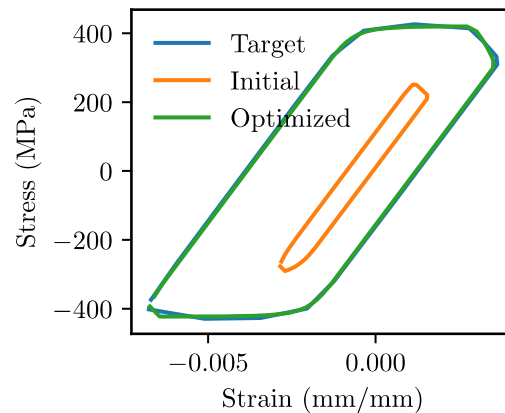


# Material Surveillance Development

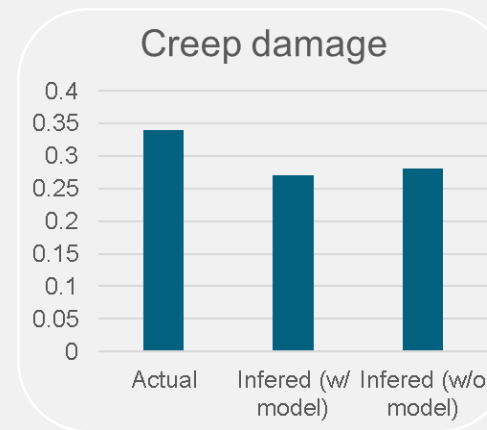
- Develop surveillance test articles and generate thermal cycling data for establishing acceptance criteria of MSR materials surveillance program in support of NRC staff guidance on materials compatibility.
- A material surveillance program would monitor material degradation in service to mitigate the risk posed by the limited up-front test data.
- It provide reactor vendors and regulators a means to ensure the long-term reliability of MSR materials in the operating environment.



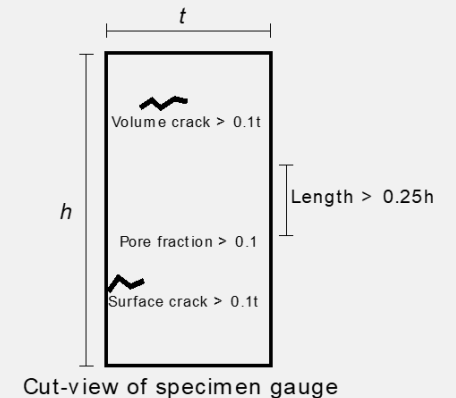
Test article development



Specimen sizing to match component loading



Damage inference



Acceptance criteria:  
component remaining life



# Technology Demonstration

# Technology Demonstration & Deployment

## Component Fabrication and Evaluation

- Identify reactor components that could take advantage of new materials or AM technologies for demonstration.
- Evaluate component performance in relevant reactor environments.
- Demonstrate rapid qualification framework and methods.

## Large-Scale Manufacturing & Supply Chain

- Address technical challenges in large-scale manufacturing.
- Identify large-scale component manufacturing capabilities, and potential supplies and manufacturers.
- Consider vendor qualification for nuclear components and NQA-1 or equivalency certification requirements.

## Codes, Standards, and Regulatory Acceptance

- Include new materials and manufacturing methods into ASME Codes and other Standards.
- Develop technical bases for gaining regulatory acceptance.
- Engage with stakeholders and gather feedback.

# IHX Liner Assembly for MARVEL

Goal was to fabricate full-scale IHX liner assembly that meets the requirements of the design using AM capabilities available within AMMT & industry partnerships.

## MARVEL



- Demonstrate proof of concept
- Evaluate materials and techniques



GE Concept M2 LPBF  
Liner: 316L SS  
Size: 250 x 250 x 350 mm<sup>†</sup>



EOS 290M LPBF  
Downcomer: In 625  
Size: 250 x 250 x 325 mm<sup>†</sup>



Liner/Downcomer Assembly



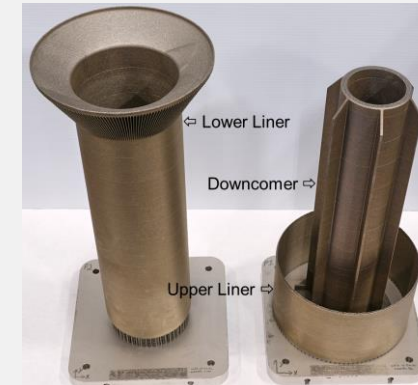
AddUp BeAM Modulo 400 DED  
Liner: 316L SS  
Size: 400 x 400 x 650 mm<sup>†</sup>

## Full-scale Liner Assembly



GE Concept Xline 2000R LPBF  
Liner & downcomer: In 718  
Size: 800 x 400 x 500 mm<sup>†</sup>

<sup>†</sup> - Denotes maximum theoretical build height; the actual build height achievable may be less.

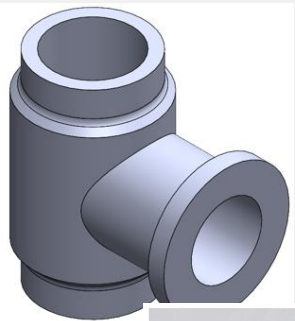


# Large-scale Additive Manufacturing

- Feasibility demonstration of large-scale additive manufacturing of nuclear components (e.g. pressure vessel, valves).
- Smart Manufacturing: incorporate *in-situ* monitoring data into component qualification and certification.

## T-valve (50% scale)

- Hybrid AM
- Material: 316L SS
- Weight: 25lbs
- Volume: 53 in<sup>3</sup>



CAD file  
(CAD design provided by EPRI.)

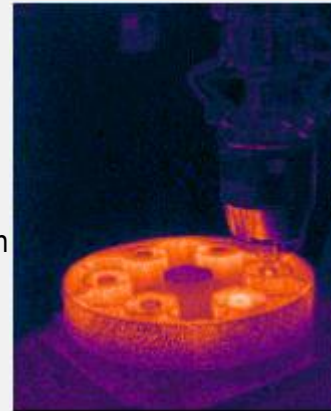


Actual Build

## Process Monitoring



Digital Image Correlation (DIC)



IR camera

### Monitor and control stress evolution via in-situ DIC+IR

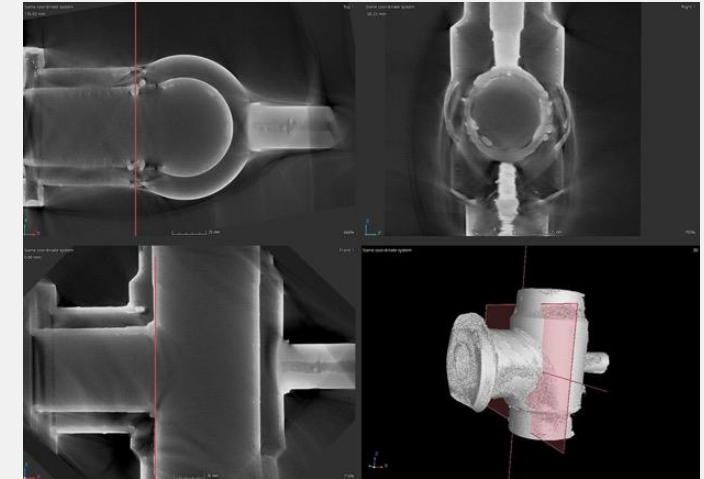
- DIC tracks surface features in 3D over time.
- Multispectral IR measures temperature and emissivity.

### Target detectable information

- Cracking and delamination, volumetric phase changes.
- Thermophysical properties, CTE, thermal conductivity.

## Post-Process NDE

### CT Scan (Volumax)



Overall Resolution ~88  $\mu$ m

- 48hr scanning yielded much good contrast across and between walls.
- Certain regions show build discrepancies along weld beads – none seems critical.

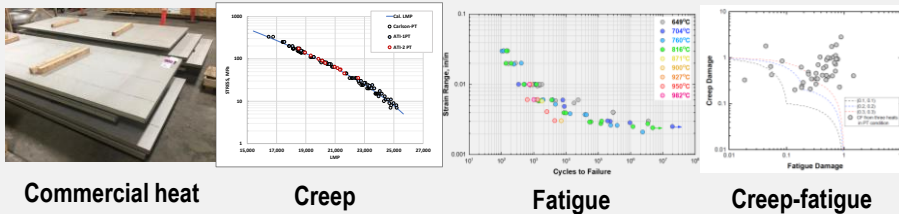


# Codes and Standards

- ASME Section III Division 5, High Temperature Reactors Code Case development
  - 100,000 h code case of new materials (A709, LPBF 316H)
  - 500,000 h code cases of Code-qualified materials
- Incorporate accelerated qualification methods into the current Code Case development.

## 100,000 h Code Case for A709

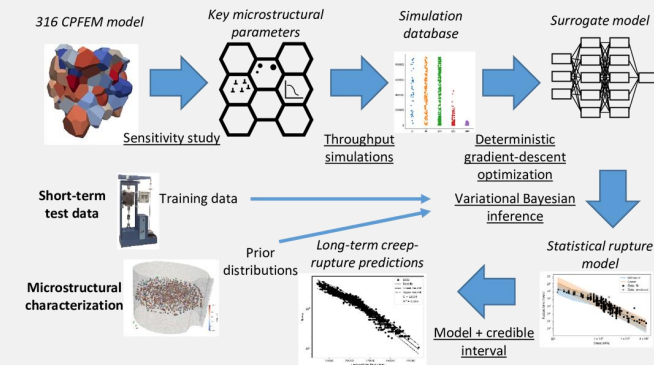
- A709 ASME Code qualification requires property testing for **three commercial-scale heats**
  - 1<sup>st</sup> Commercial Heat Plate by G. O. Carlson, 40,000 lbs.
  - 2<sup>nd</sup> & 3<sup>rd</sup> heats from ATI Flat Rolled Products, each 45,000 lbs.
- Recommended heat treatment
  - 1150°C/ SA & 775°C/10 hr.
- Code case testing
  - Tensile, creep, fatigue, creep-fatigue.
  - Base metal and weldment.



## 500,000 h Code Cases for Code Materials

- Extension of allowable stresses to longer lifetimes for existing Section III Division 5 Class A materials (e.g. 304H/316H to 500,000 hours, 617 to 150,000 hours)
- A709 Code Case > 100,000 hours.

Physics-based/Active Learning for Creep Data Extrapolation for Allowable Stress Development



# Summary

- The overarching vision of the AMMT program is to accelerate the development, qualification, demonstration, and deployment of advanced materials and manufacturing technologies to enable reliable and economical nuclear energy.
- Integrated materials and manufacturing development strategy
  - Expand the use of existing reactor materials through advanced manufacturing methods
  - Enable the use of non-nuclear commercial materials in nuclear environments
  - Develop innovative, high-performance materials enabled by advanced manufacturing
- Accelerated qualification is the program focus
  - Qualification of high temperature materials
  - Qualification of materials for environmental effects (irradiation and corrosion)
  - Qualification of materials made by advanced manufacturing
- Technology demonstration is crucial for accelerating the deployment of advanced materials and manufacturing technologies.
- We tackle these complex materials challenges to enable reactor vendors to adopt and utilize these advanced materials and fabrication methods into their reactor designs.

**AMMT website**  
**<https://ammt.anl.gov>**