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Accelerated Qualification of Nuclear Structural Materials

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THE ENTIRE AMMT TEAM

AMMT Vision, Mission, and Goals

Vision

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

Mission

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

Goals

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

This talk focuses on the *accelerated qualification* aspect of the program.

Introduction

- “**Qualification**” refers to the process of systematically testing and evaluating materials to ensure their performance reliability under reactor operating conditions, such as high temperatures, radiation exposure, and corrosive environments.
- Qualifying an alloy for its use in nuclear applications involves complying with Codes, standards and regulations due to the critical safety requirements of nuclear components.

ASME Codes and NRC Regulations

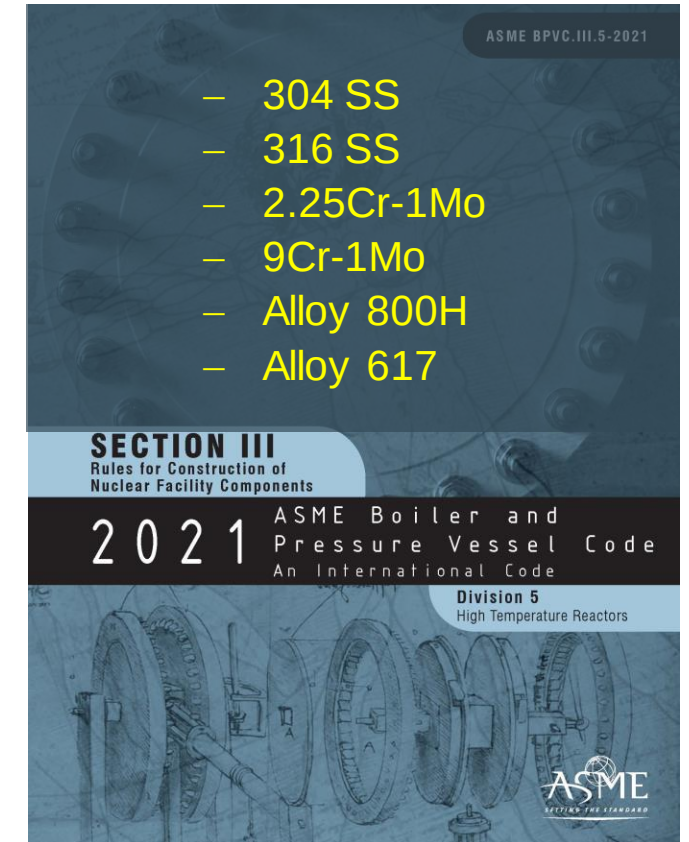
- ASME Codes
 - ASME Boiler and Pressure Vessel Code (BPVC) provides rules used for the design, fabrication, and inspection of boilers and pressure vessels, including structural components used in nuclear reactors.
 - BPVC, Section III provides rules for constructions of nuclear facility components.
- NRC Regulations
 - NRC performs regulatory reviews and approvals of new designs for nuclear power plants.
 - NRC's regulations for nuclear power plants are codified in Title 10 of the Code of Federal Regulations (10 CFR). Regulations in 10 CFR Part 50 and 10 CFR Part 52 include requirements for material qualification and performance monitoring.

Accelerating the material qualification process of new materials for nuclear use is crucial for timely innovation in the nuclear industry.

Our Challenge: Qualification of High Temperature Materials

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.
- NRC endorses the use of ASME Section III, Division 5.
- 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



Our Challenge: Qualification for Environmental Effects

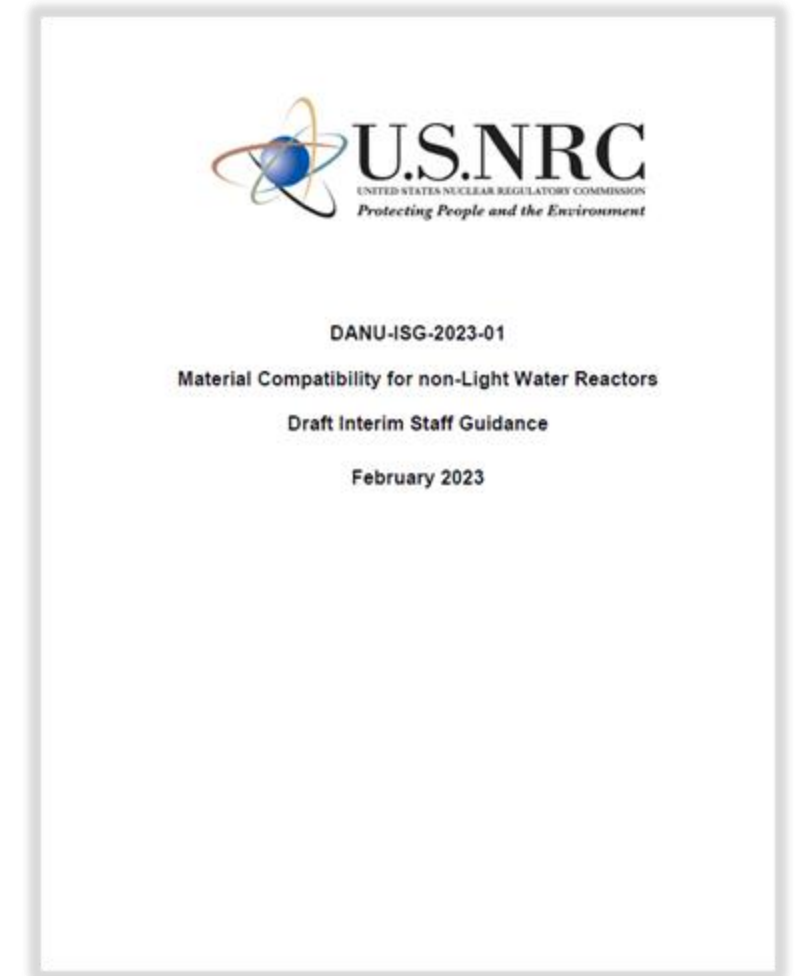
- ASME Code rules do not provide methods to evaluate deterioration from corrosion, mass transfer phenomena, radiation effects.
- NRC has issued “Material Compatibility for non-Light Water Reactor Draft Interim Staff Guidance” as part of its evaluation of a non-LWR application to review applicable design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

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

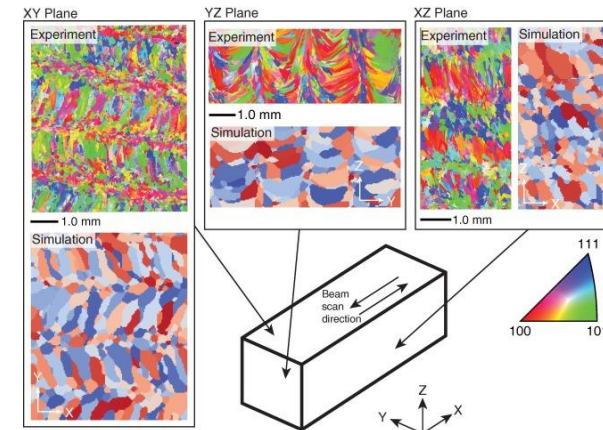
- Comprehensively evaluate the corrosion performance of materials in various reactor coolant environments (e.g. molten salt, sodium, lead, helium) is a challenge.



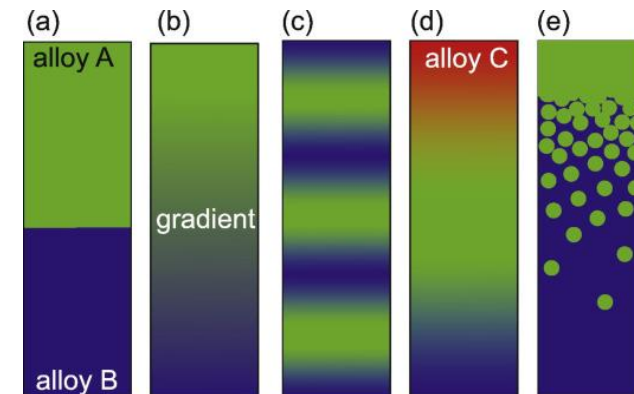
Our Challenge: Qualification of AM Materials

Need a paradigm shift to qualify materials and components made by advanced manufacturing technologies for use in nuclear systems.

- 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.
- Materials made by conventional manufacturing (e.g., casting, forging, rolling, etc.) rely on a well-defined thermal and mechanical processing condition that defines the material properties.
- Materials and components are formed simultaneously vs. sequentially in conventional manufacturing; AM qualification involves parts, materials, equipment, operators, processes.
- Additive manufacturing is a highly-localized process, creating spatial variations in response to differences in geometry and processing conditions. A large test matrix is required to determine the bounds of mechanical properties.
 - Defect variations
 - Microstructure and property variations
 - Compositional variations (e.g. functionally graded materials)



Spatially heterogeneous microstructures of an additively manufactured alloy revealed by experiments and modeling (T. M. Rodgers, et al, Comput. Mater. Sci. 135 (2017) 78).



Composition of different alloys to form functionally graded parts (N. G. March, et al, Add. Manu. 64 (2023) 103415).

Rapid Qualification Framework

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



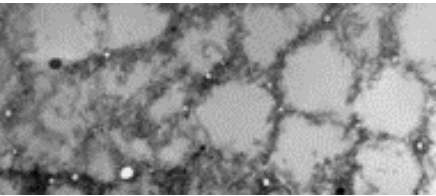
Develop **Processing-Structure-Property-Performance** based qualification framework

A P-S-P-P based qualification framework involves tailoring processing methods to achieve desired structural characteristics to meet the property and performance requirements for specific applications.



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.

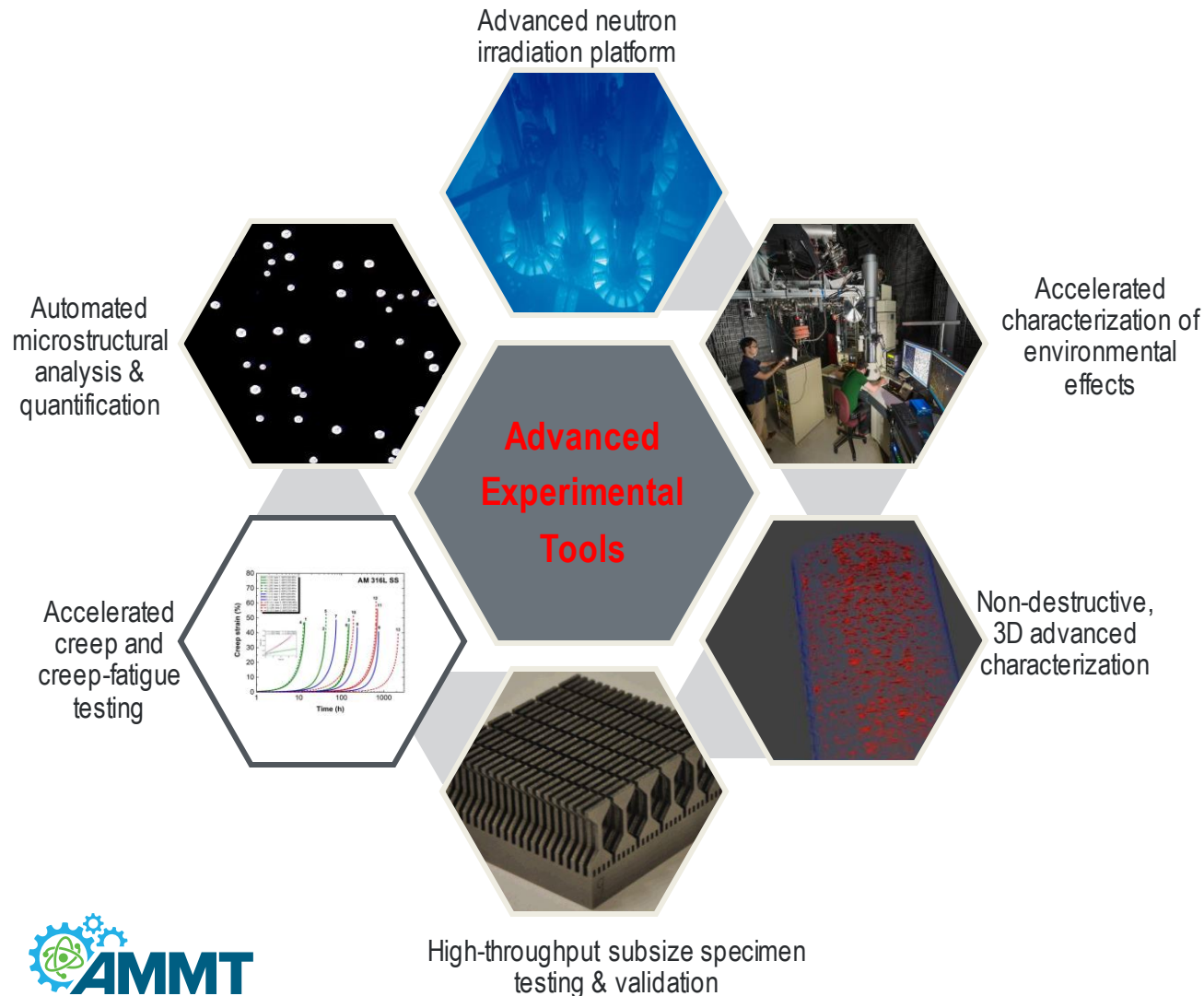
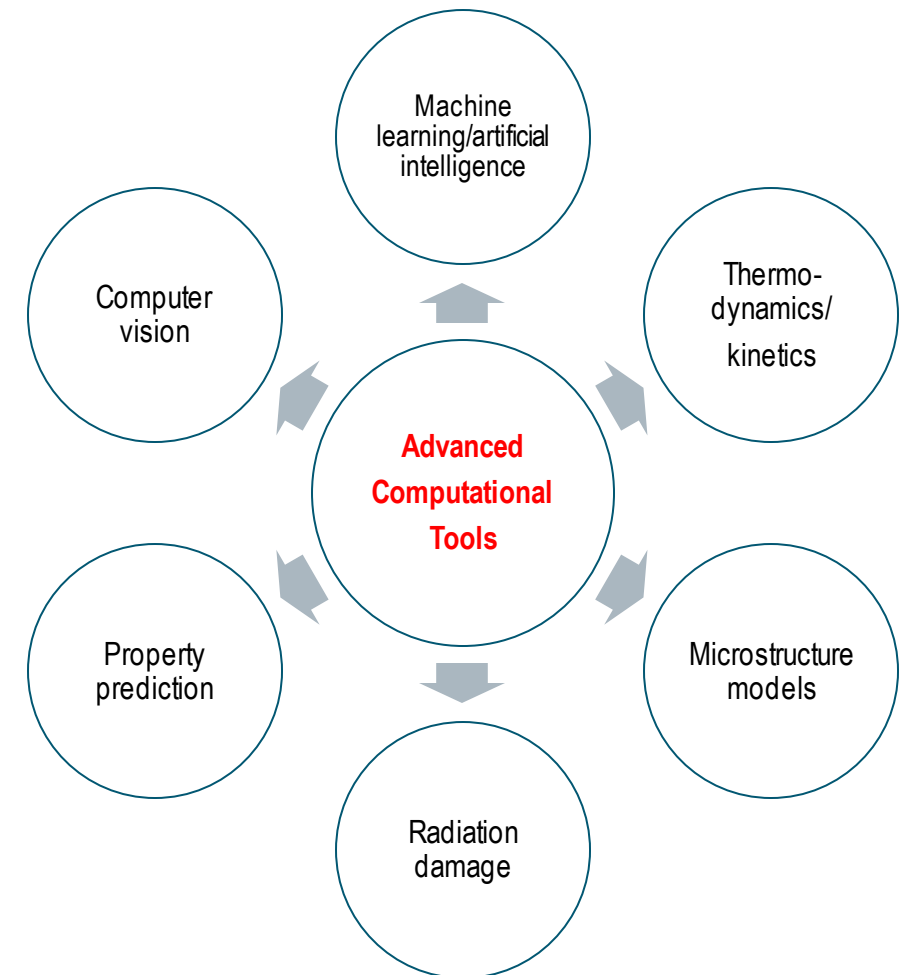


Demonstrate new qualification framework through qualifying LPBF 316H SS

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

Advanced Experimental & Computational Tools

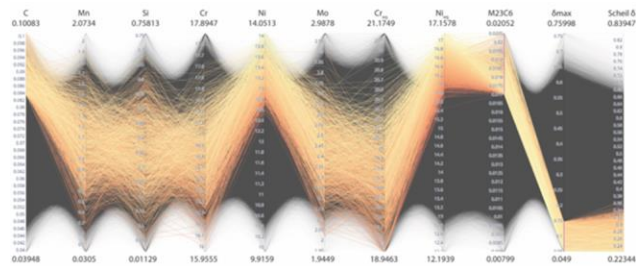
Accelerating the qualification of new alloys requires an integrated approach combining advanced experimental and computational methods.



Understanding Processing-Structure-Property Relationship

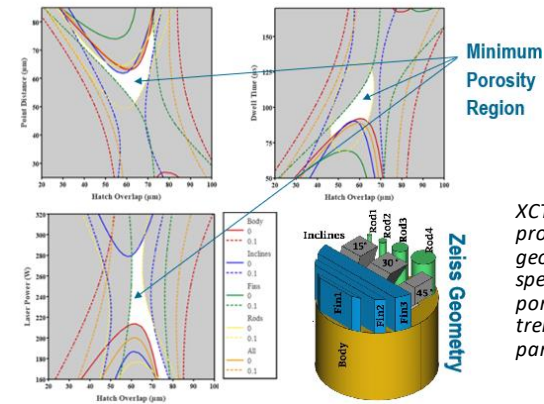
Process Understanding and Optimization of AM 316H SS

Feedstock Chemistry



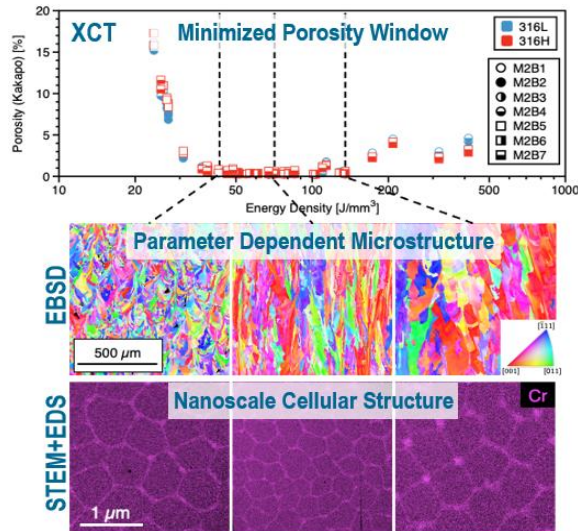
Chemistry on sensitivity to energy density, and data mining highlighting 316H chemistries to maximize carbides and minimizing δ -ferrite.

Geometry Effects



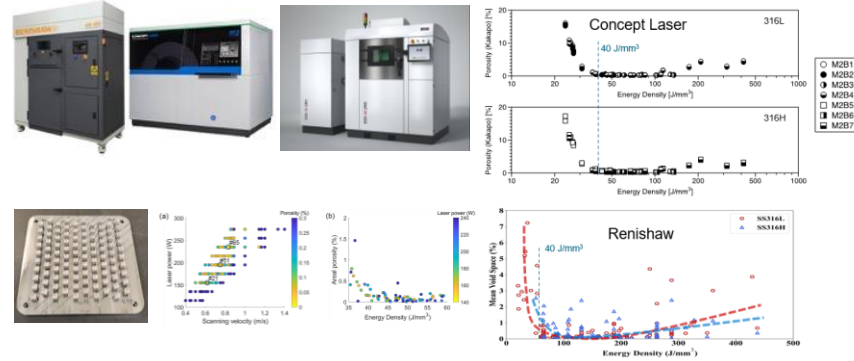
XCT used to probe geometry-specific porosity trends in AM parts.

Process Variables



Multiscale characterization using XCT, EBSD and STEM-EDS reveals variations in porosity, grain structure, & nanoscale segregation as a function of varying processing parameters

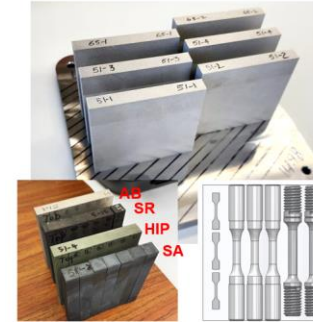
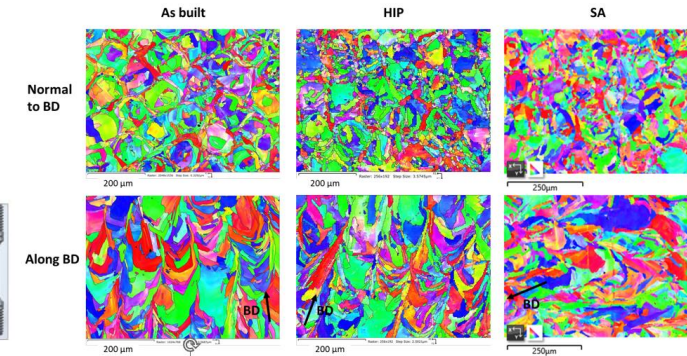
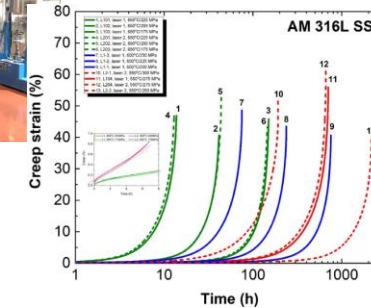
Machine Variability



- Volumetric energy density provides a reasonable first-pass optimization parameter for porosity minimization.
- Investigate the "windows" of optimal parameters between machines.

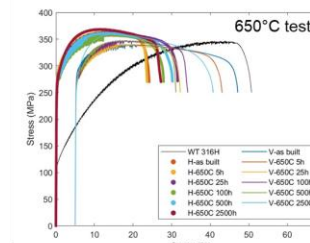
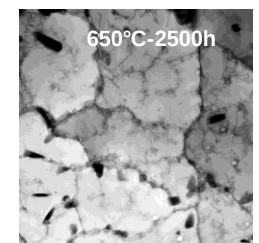
P. Nandwana, et al., ORNL/TM-2023/3031 (2023); C. Massey, et al. ORNL/TM-2023/3115 (2023); X. Zhang, et al. ANL-AMMT-004 (2023);

Post-build Treatment and Property Testing



Evaluate high-temperature fatigue, creep, and creep-fatigue tests of LPBF 316H SS.

Conduct thermal aging study of LPBF 316H SS to understand material stability.



High-throughput Automated X-ray CT NDE

Rapid printing parameter selection and optimization

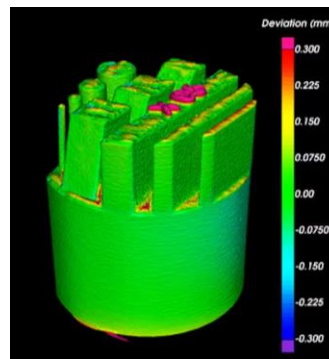
- Design of Experiment (DoE)
- Print the build plate (~100 parts per plate)
- X-ray CT at fraction of optimal scan time
- Fast, consistent and accurate AI-based XCT reconstruction
- Analysis (segmentation, flaw detection, porosity, morphology, metrology)



Build plate
each coupon a
process parameter

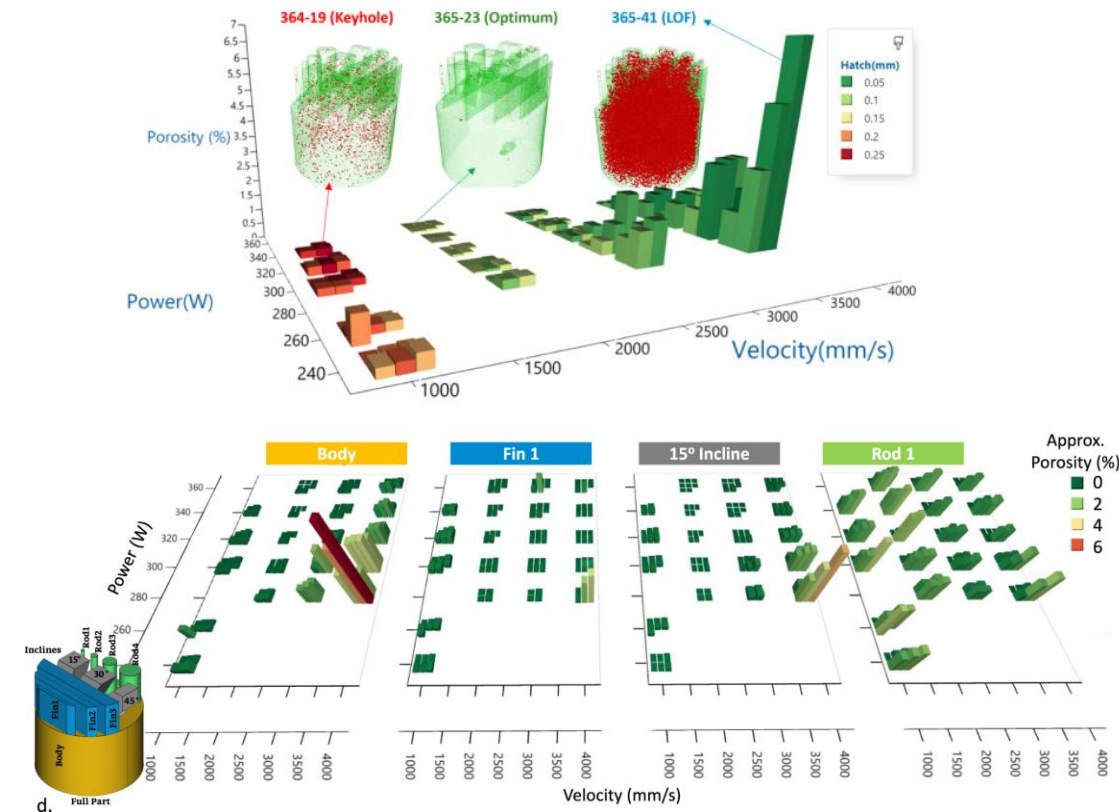


Segmented flaws



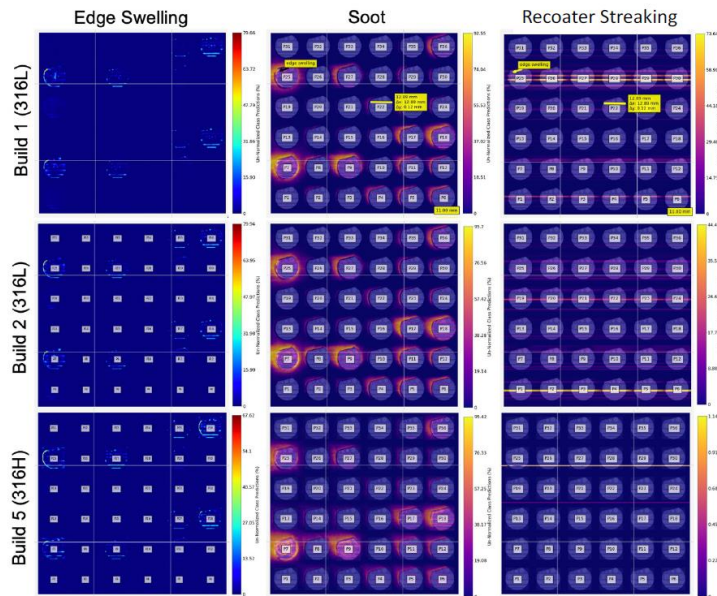
Deviation Map

Window of desired process parameters

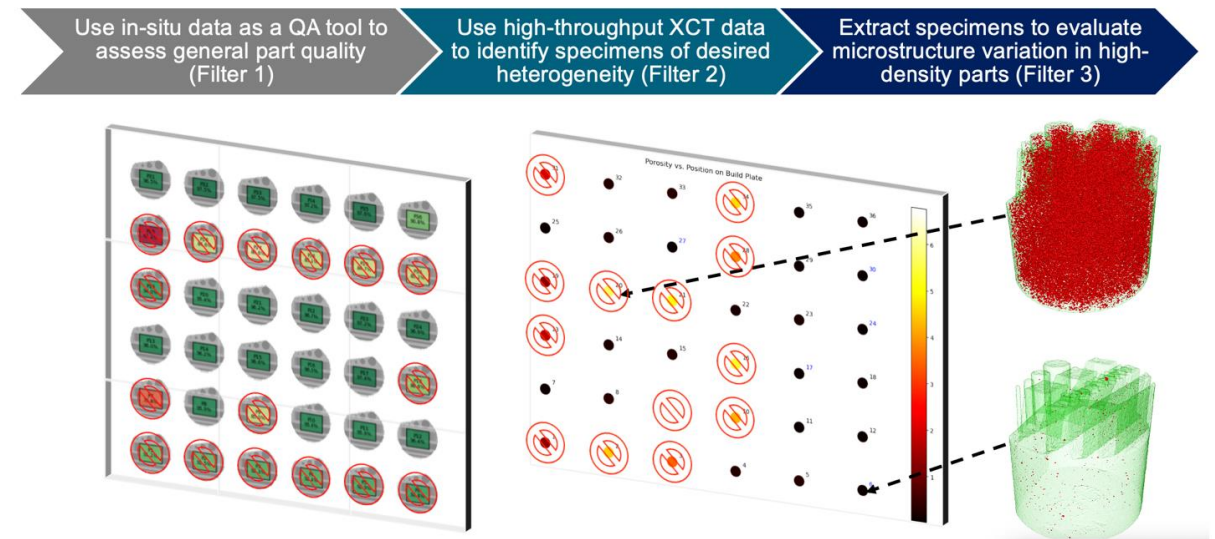


In situ Process Monitoring for AM Qualification

- Use *in situ* process monitoring data to detect defects and as a QA tool to assess part quality.



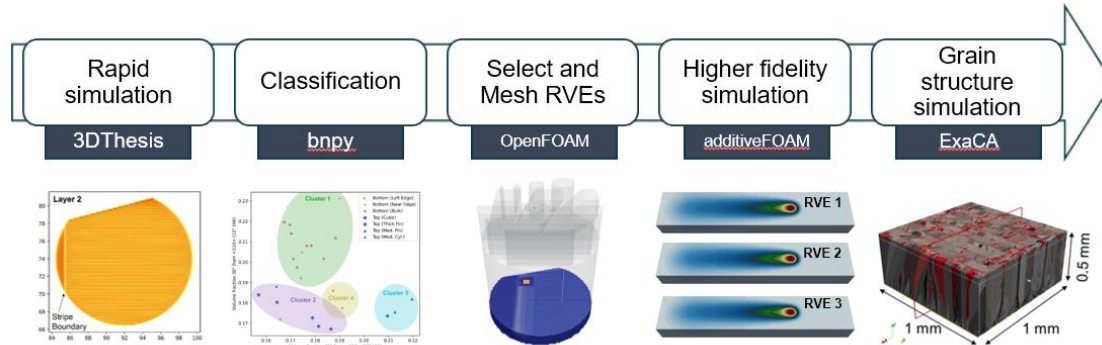
Apply *in situ* sensors and ML algorithms to determine the quality of a build. Identify three classes of characteristic anomalies through *in situ* data collection.



- Integrate *in situ* process monitoring data into the qualification process and work with ASME to identify a qualification pathway.

Processing-Structure-Property Modeling

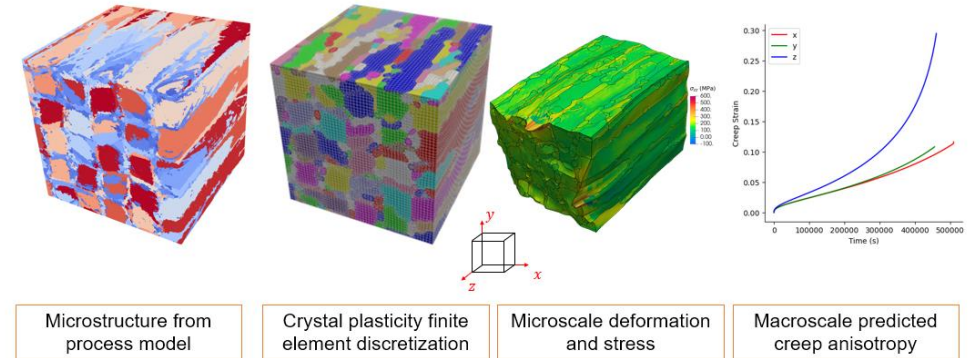
Process modeling to predict AM microstructure



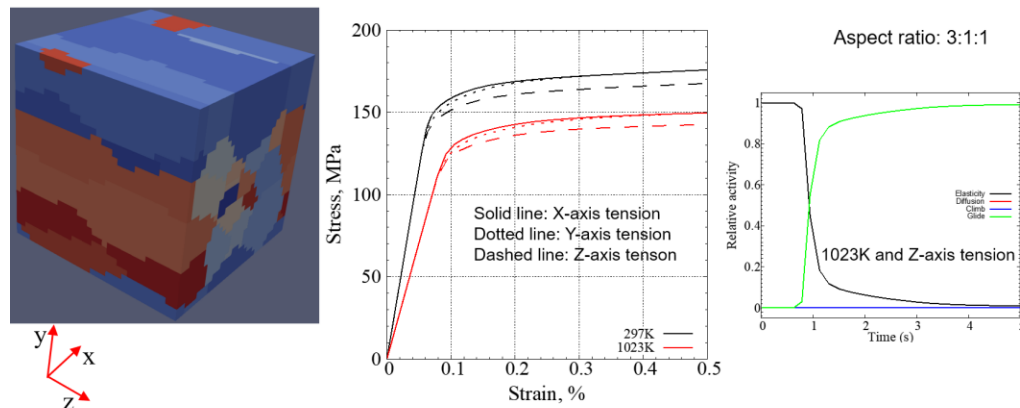
Output: Generate representative grain structure statistics for variation found in real parts

Microstructure-Property Modeling

- Process model may be used to generate synthetic microstructures

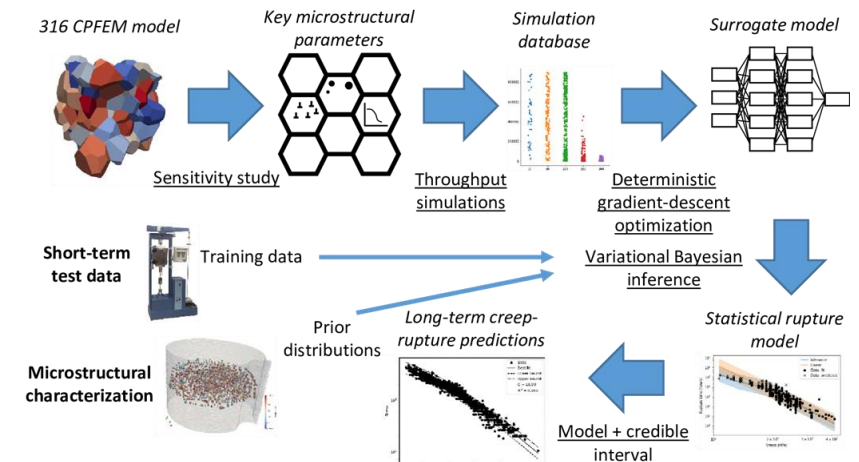


Model tensile behavior of AM with columnar grains



Columnar grain structure develops significant anisotropy in the mechanical responses.

Predict long-term creep strength



Thermal Creep Challenge Problem

Problem Definition

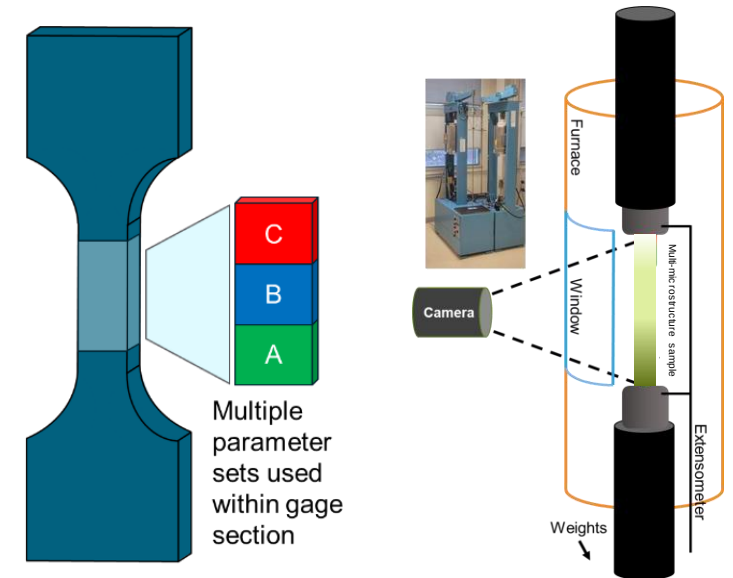
- Predict the creep behavior of an additively-manufactured 316H specimen that has a range of microstructure within the specimen gauge resulted from different processing conditions.

Goals

- Actively integrate multiple facets of the program such as materials development, advanced manufacturing, characterization and testing, computer modeling, and digital data to accelerate the qualification process.
- Provide a testable example to evaluate key technical challenges, assess the state-of-the art, and identify critical gaps that must be addressed to meet the program goals.

Potential Impact

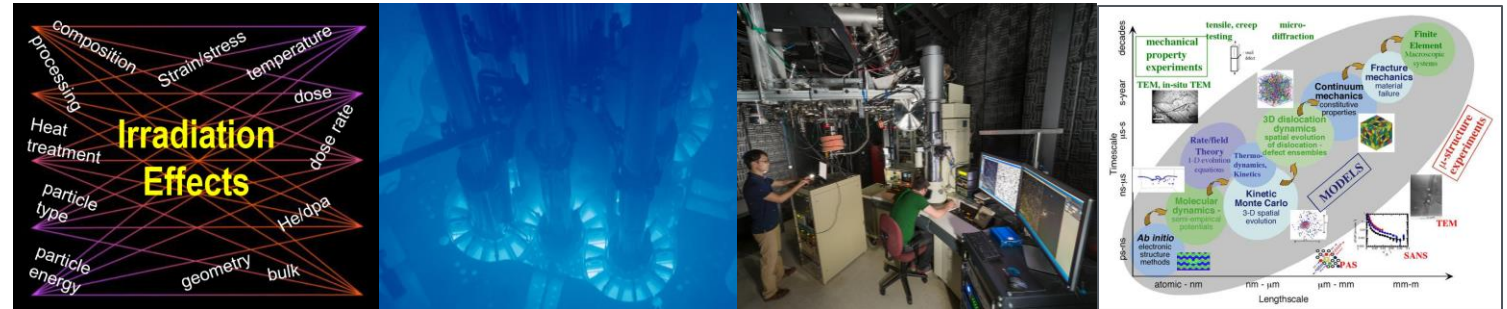
- Successful completion of the challenge problem will yield a critical benchmark to gauge the ability of model predictions. The developed methodology could be applied to various materials, mechanical property tests, and environmental conditions.



Accelerated Qualification for Environmental Effects

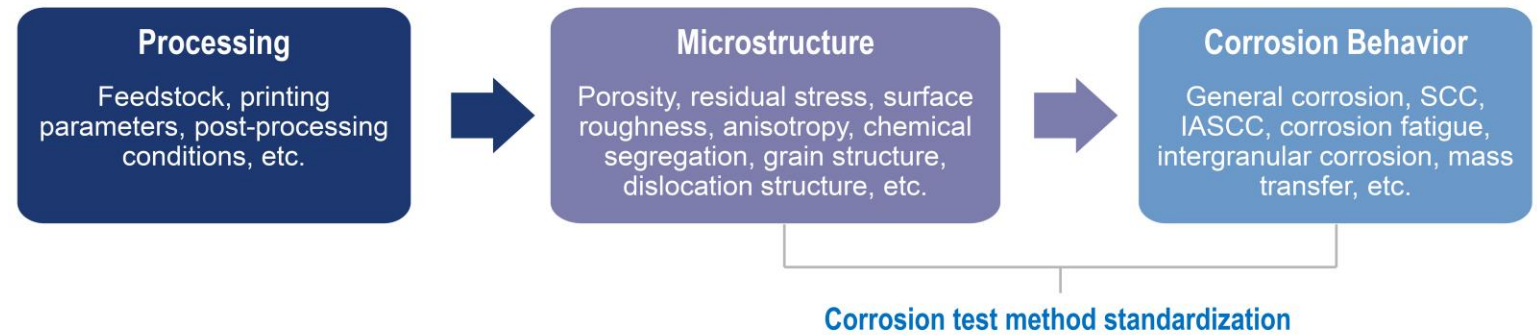
Irradiation Effects

- Conduct neutron irradiation and PIE to evaluate the materials performance.
- Use combined ion & neutron irradiation and modeling data to cover a wide parameter space to develop a comprehensive understanding 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.



Performance Evaluation under Neutron Irradiation

Neutron Irradiation and Post Irradiation Examination (PIE) of Laser Powder Bed Fusion 316 SS

HIFR Irradiation

Flux Trap “Rabbits” Experiments

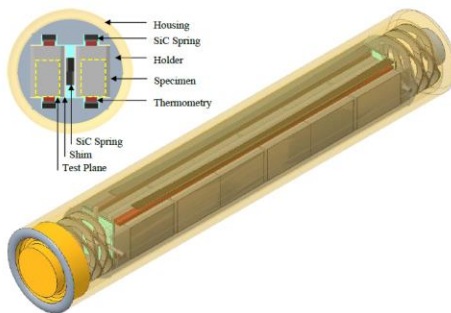
- Fast flux: 1.1×10^{15} n/cm²/s
- Thermal flux: 2.0×10^{15} n/cm²/s
- Small diameter: $\phi 9.1$ -11.3 mm
- 12.6 dpa/year (steels)
- Temperature: 60-1200°C
- Hydraulic tube for irradiation

Tensile specimens

- SS-J2 (16 x 4 x 0.5 mm)
- SS-J3 (16 x 4 x 0.75mm)

Bend bar specimens

- Dimensions: 14.8 x 3 x 4.5 mm



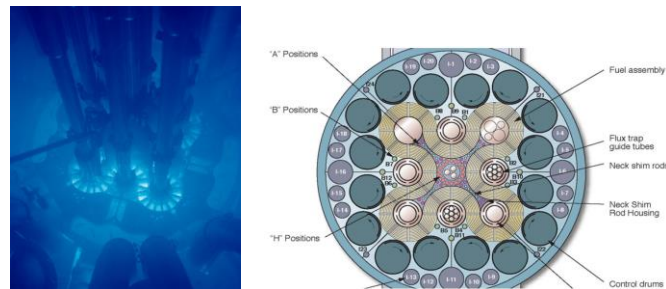
ATR Irradiation

Irradiation Experiments

- Primarily thermal spectrum
- Pressurized water at ~50°C
- Experiment positions vary between 5/8" to 3" diameter
- Fast flux: 8.1×10^{13} n/cm²/s
- Thermal flux: 2.5×10^{14} n/cm²/s

Specimens

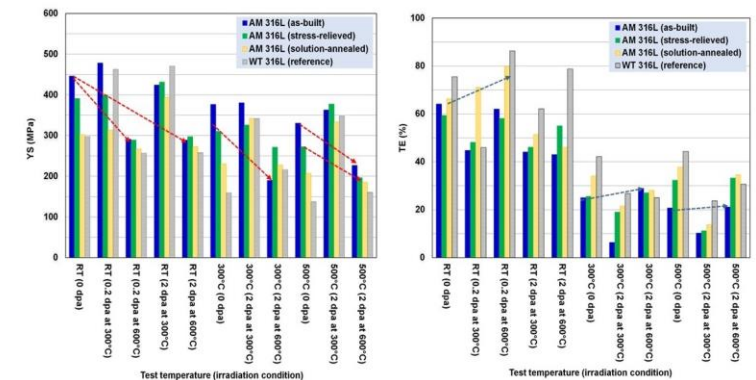
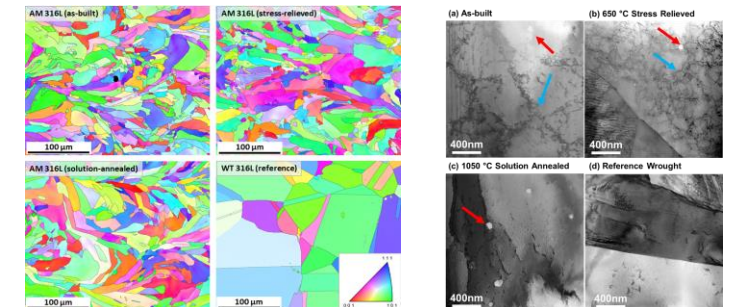
- SS-J tensiles
- Bend bars
- Round compact tension



P. A. Chanplin, et al., ORNL/TM-2023/3037 (2023).
G. J. Youinou, INL/EXT-17-40962 (2017).
T. S. Byun, et al., J. Nucl. Mater. 548 (2021) 152849.

Post Irradiation Examination

Comparison of tensile strength and ductility data for LPBF and wrought 316L in various test and irradiation conditions.

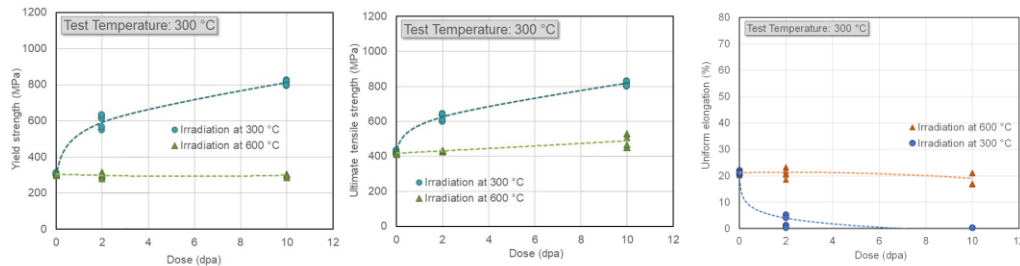


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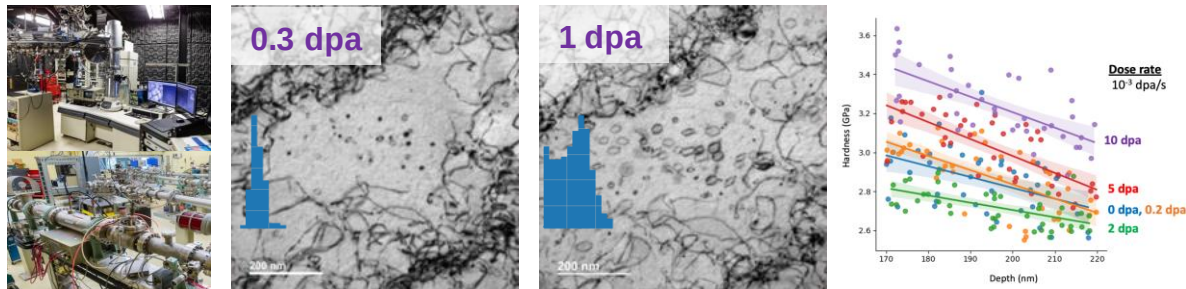
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Accelerate Qualification with Combined Ion & Neutron Irradiations and Modeling

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.

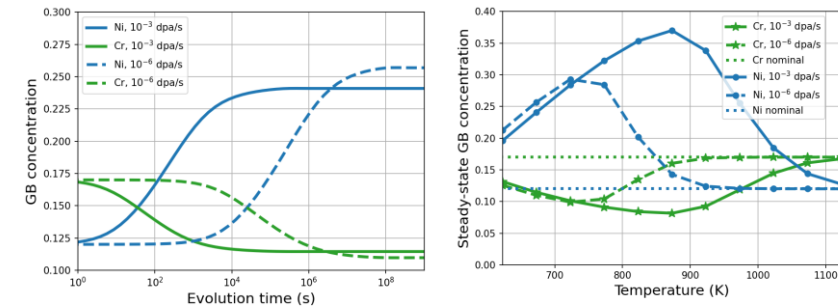


Ex situ/in situ Ion Irradiation: *Ex situ* ion irradiation creates a sample library of AM 316 SS to understand the effects of a large number of metallurgical and irradiation parameters; *in situ* ion irradiation with TEM provides high-fidelity data for modeling of irradiation-induced defect evolution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

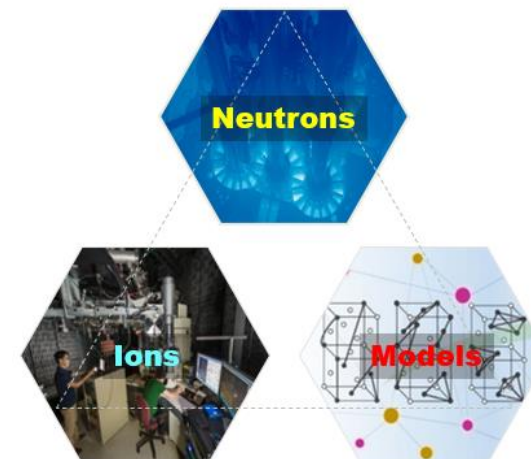
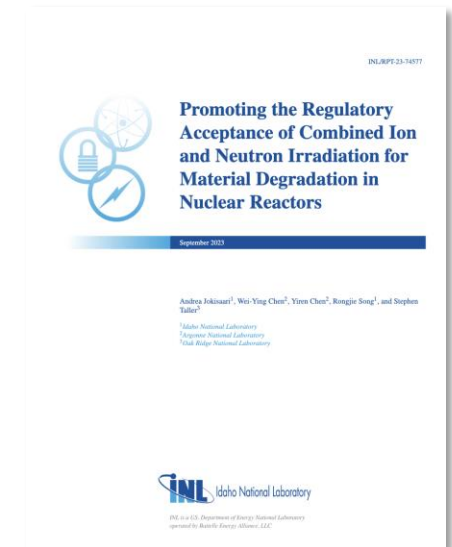


Computer Modeling

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



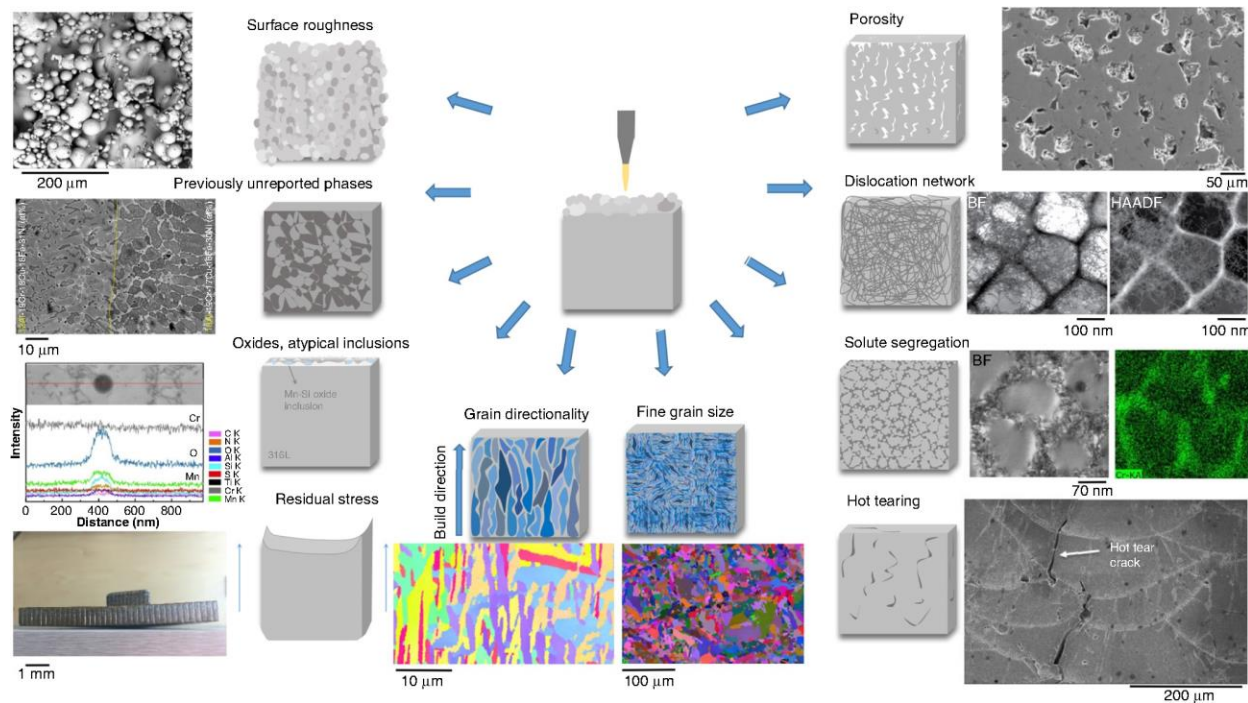
Effect of dose rate or particle type on RIS. a) Time to reach steady state concentration at 673 K. b) Combined effect of temperature and dose rate on the steady-state GB concentration.



Corrosion Performance Evaluation of AM Materials

- Identified the research needs and strategies to characterize the corrosion behavior of AM materials in nuclear environments.
- Initiated corrosion studies of AM materials in molten salt and sodium environments.

Potential impact of AM processing on microstructure and corrosion behavior



CORROSION. 2018;74(12):1318-1350. doi:10.5006/2926



High-throughput Automated Microstructure Quantification

Goal

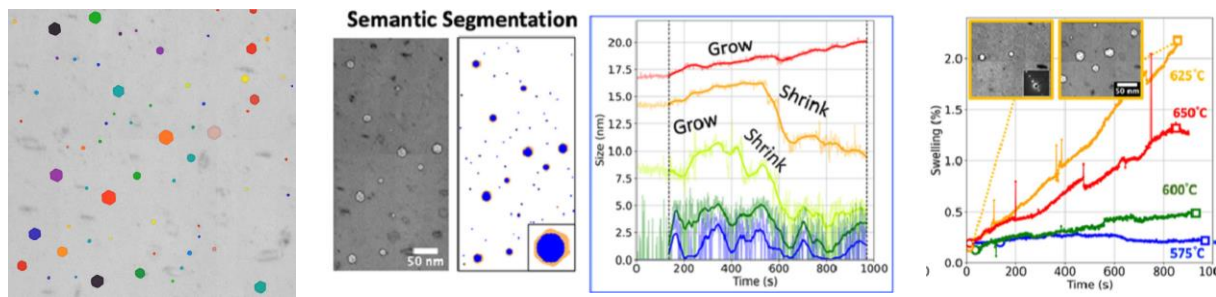
- To provide quantitative microstructural data to establish the processing-structure-property-performance relationships to accelerate material development and qualification for nuclear applications.

Application of AI/ML

- AI models identify and analyze microstructural features in microscopy images, enhancing data accuracy and efficiency.
- These models, trained on expert annotations, segment images to generate quantitative microstructural data such as size and density.

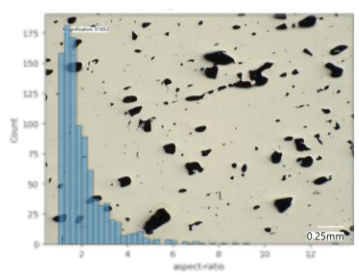
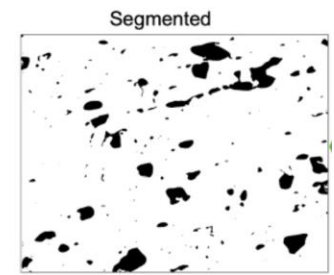
Irradiation Defect Dynamics: CV enables real-time analysis of nanometer-sized defects generated during *in situ* ion irradiation with TEM.

Track individual voids and monitor void swelling during irradiation.



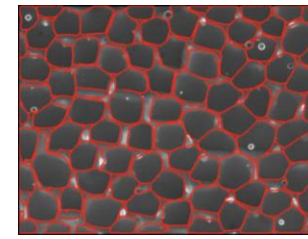
Multi-scale Microstructure Quantification: CV enables rapid, consistent microscopic analysis and provides quantitative microstructural data at various length scales.

Optical Microscopy

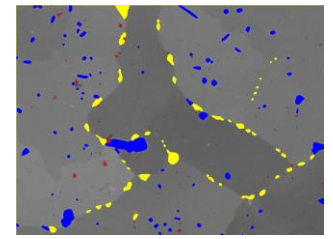


Python-based computer vision tools to analyze porosity size, density and shape in LPBF 316 SS.

Scanning Electron Microscopy (SEM)

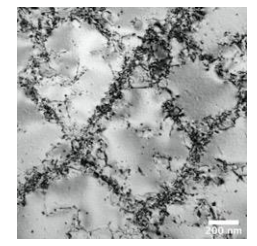
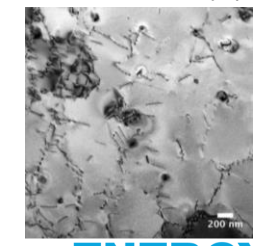


Segmentation of cell structure in LPBF 316 SS.



Annotation overlay of CrCO-rich phase, MnSiO₃, and MoSi rich precipitates highlighted in yellow, red and blue, respectively.

Transmission Electron Microscopy (TEM)

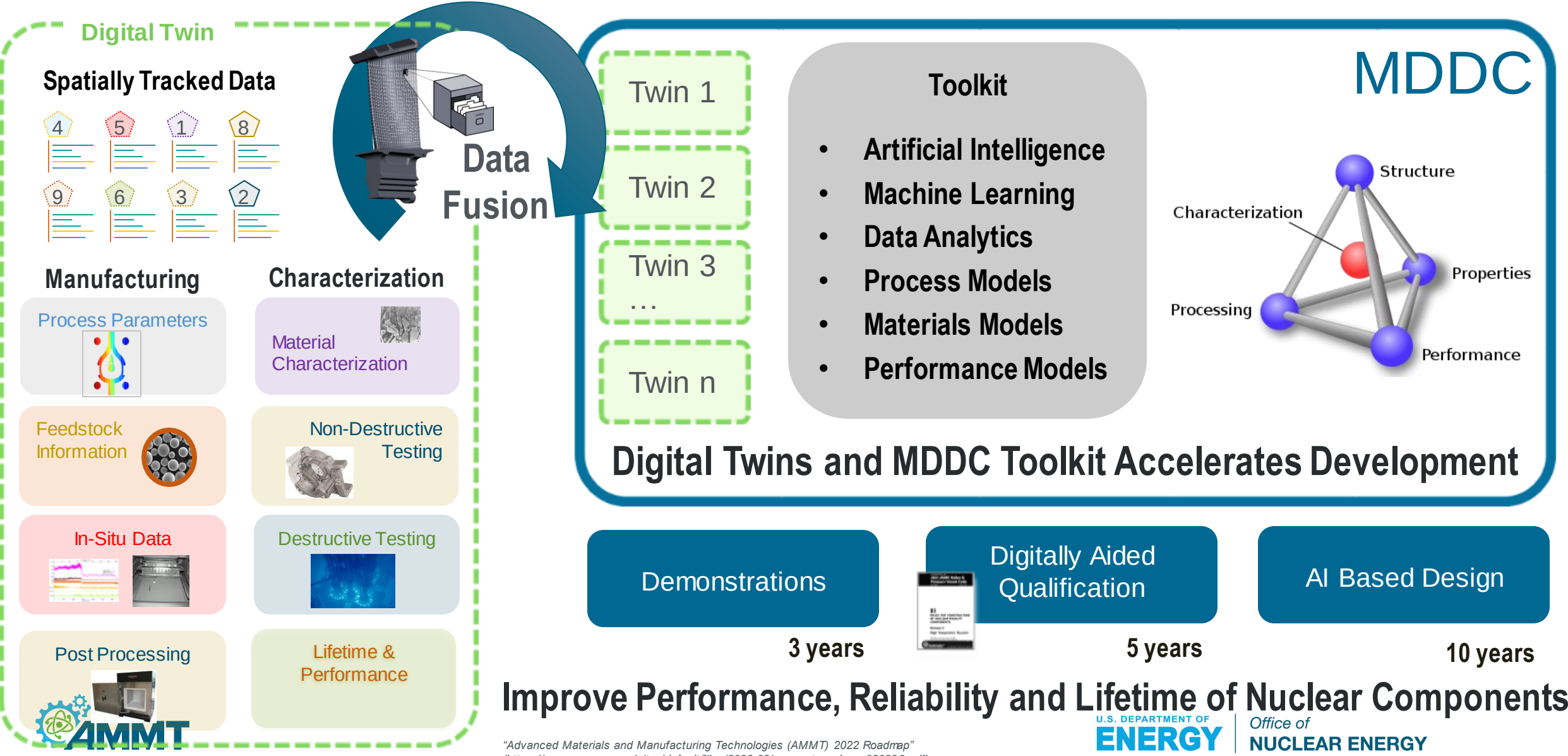


CV models to analyze TEM images to measure dislocation cells, dislocation lines and dislocation loops.



W. Chen, et al., Acta Mater. 254 (2023) 119013.
W. Yiren, et al., ANL-AMMT-10 (2023).
M. Li, et al., J. Nucl. Mater. 559 (2022) 153469.

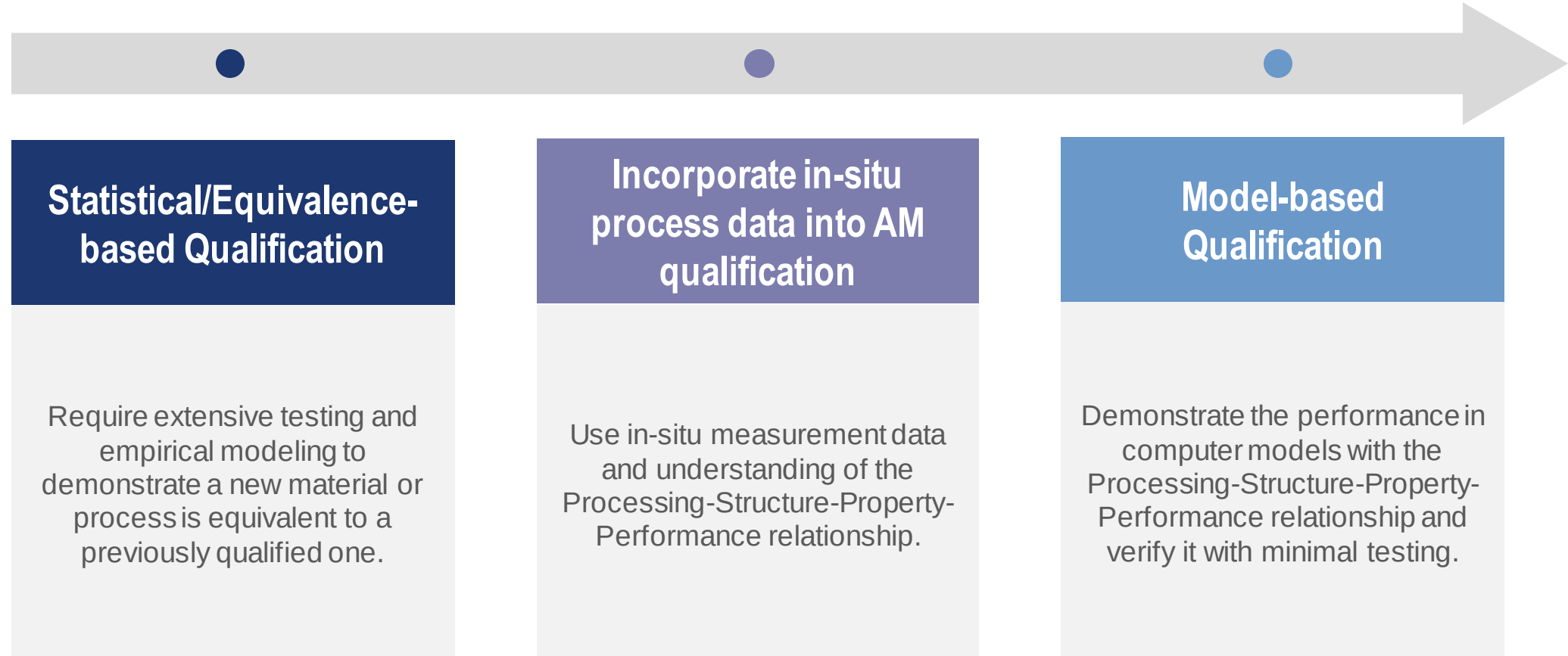
Multi-Dimensional Data Correlation (MDDC) Platform



"Advanced Materials and Manufacturing Technologies (AMMT) 2022 Roadmap"
(<https://www.energy.gov/sites/default/files/2023-03/ne-ammt-roadmap-030823.pdf>)

Accelerated Qualification: Staged Approach

Consider multiple qualification pathways and take a staged approach.



Summary

- Major challenges in the qualification of nuclear structural materials:
 - Qualification of high temperature materials
 - Qualification of materials for environmental effects (irradiation and corrosion)
 - Qualification of AM materials
- Introduce a framework to enable accelerated qualification of nuclear structural materials, focusing on understanding the Processing-Structure-Property-Performance relationships.
- Integrate novel research methodologies, e.g. advanced manufacturing, advanced characterization, high-throughput testing, computational modeling, and ML/AI, to expedite the qualification process.
- Leverage the extensive dataset and knowledge to establish design allowables and performance boundaries for materials subjected to irradiation, corrosion and high-temperature stresses.
- Establish and demonstrate the new qualification framework through qualifying LPBF 316H SS for applications in high temperature reactors.
- Actively engage with ASME and NRC early to understand their requirements and expectations. Regularly update them on progress and interim results to build trust and facilitate approval processes.

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Questions