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Environmental Degradation in Advanced Reactor Environments

July 2024

Changing the World's Energy Future

Andrea M Jokisaari

Idaho National Laboratory

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

AMMT ADVANCED MATERIALS AND MANUFACTURING TECHNOLOGIES

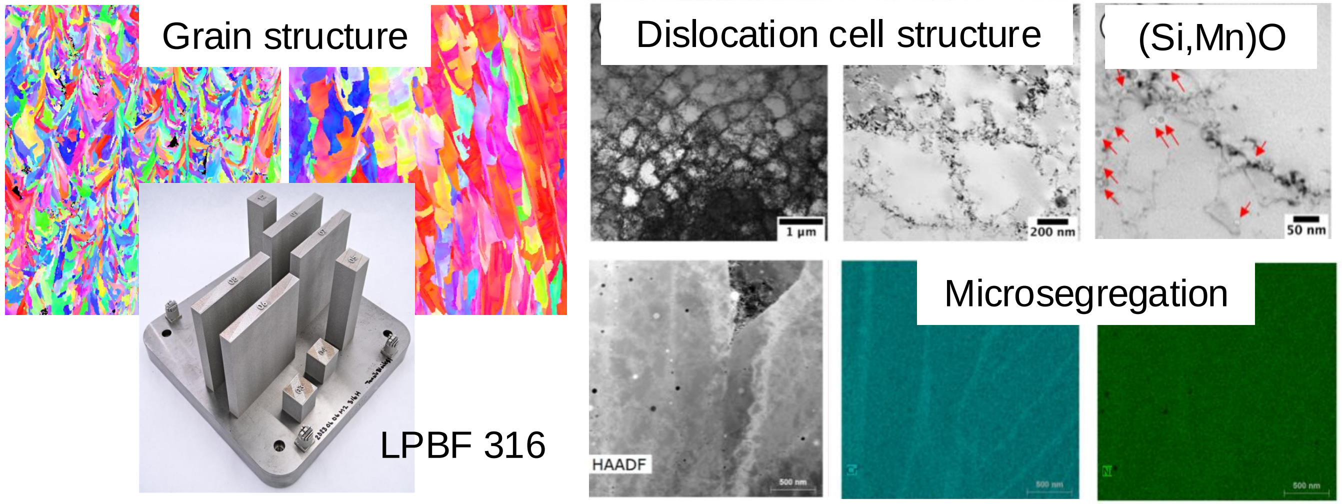
Environmental Degradation in Advanced Reactor Environments

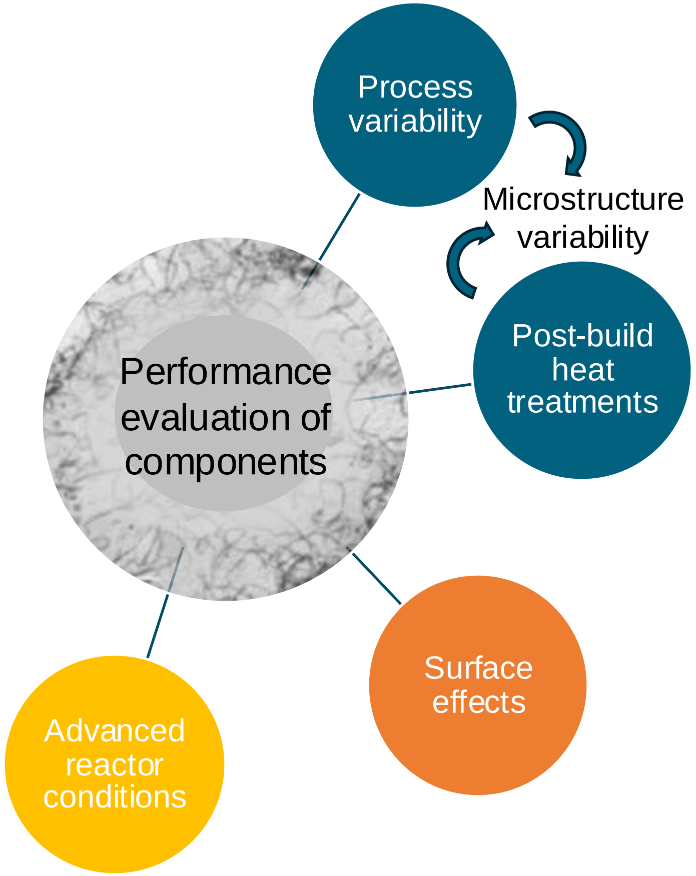
Andrea Jokisaari

With contributions from: TS Byun, Yiren Chen, Trishelle Copeland-Johnson, Michael Woods, Grace Burke, Annabelle Le Coq, Kory Linton, Stephen Taller, Caleb Massey, Xuan Zhang, Weiying Chen, Tim Lach, Drew Johnson

2024 AMMT Industry Workshop, Idaho Falls, Idaho, July 10-11, 2024

AM-specific microstructures can affect material behavior in-reactor





Evaluating environmental performance of new materials is one of the most critical technical hurdles for their rapid adoption in nuclear energy systems outside of ASME code qualification

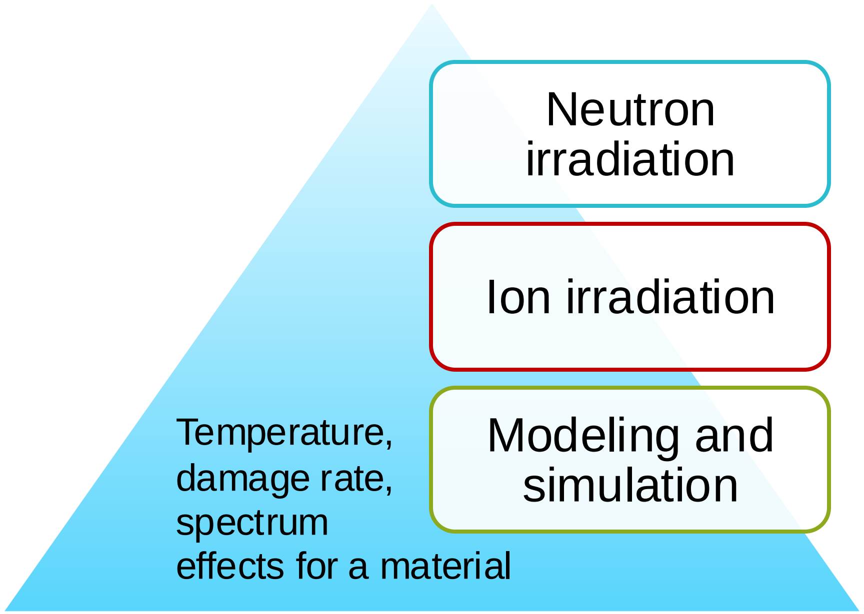
The Environmental Effects technical area has four broad goals:

- First-of-a-kind degradation data on new materials and components
- Effect of microstructure variability on degradation
- Getting to the answer faster (faster tests, less tests…) for rapid and effective qualification on materials performance and degradation in reactor environments
- Establishing a technical basis for regulatory acceptance by providing needed data and models to support reactor design and operation

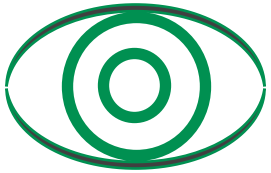
Ex: Corrosion rate **4.6 ± 0.7** µm/year

Environmental effects are reactor-specific…

…But there are ways to be cross-cutting



Computer vision

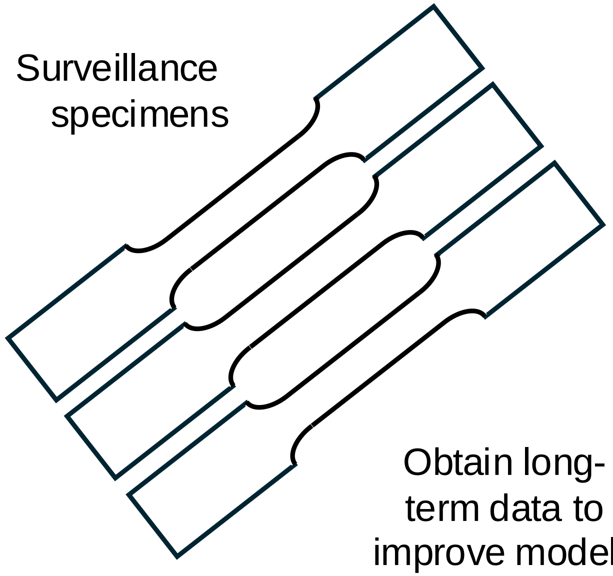


Rapidly gather quantitative data sets

Corrosion testing

Consider the same material in different environments

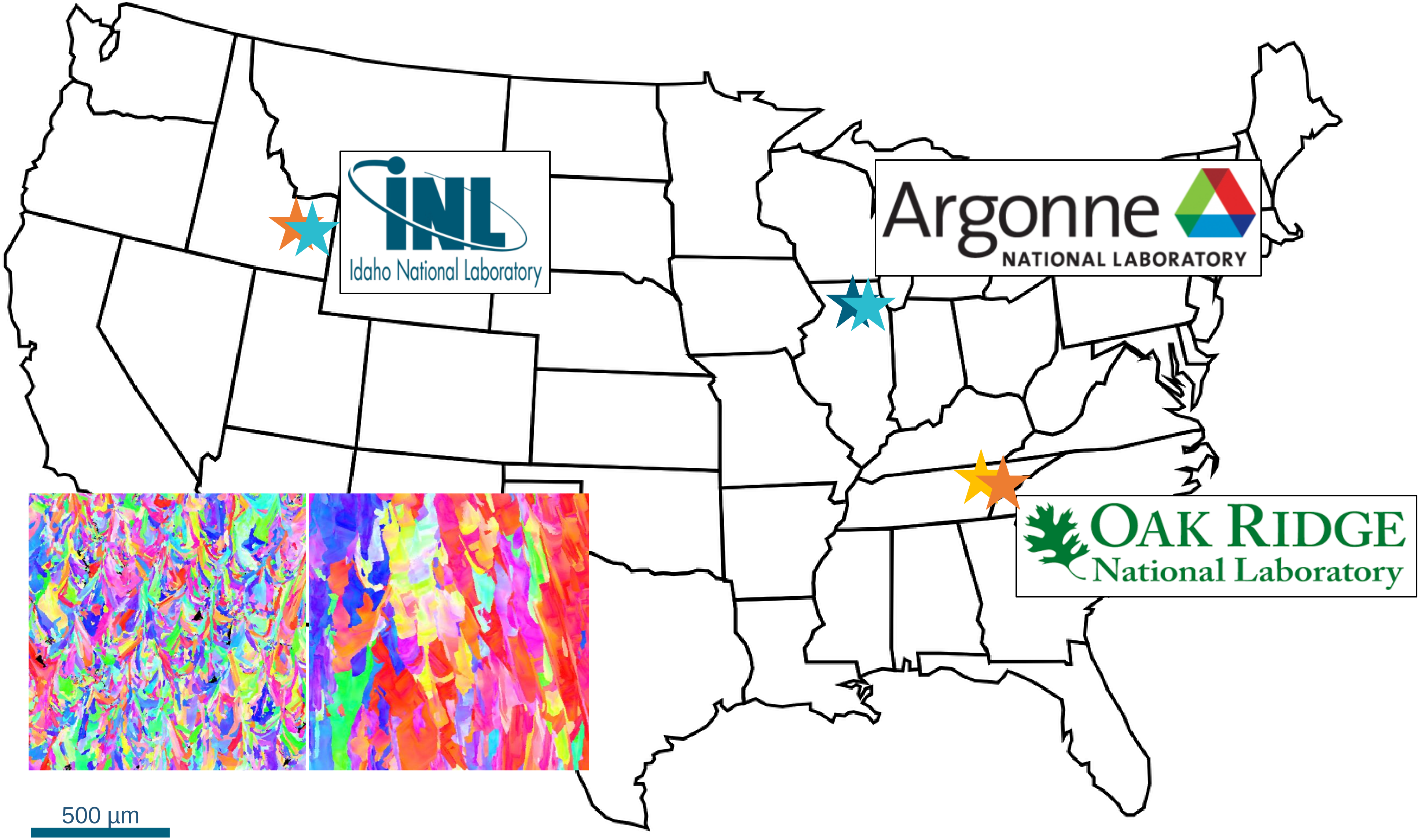
Surveillance specimens

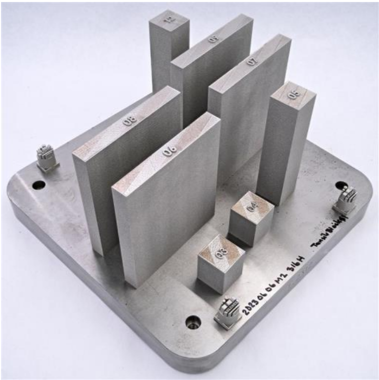


Obtain long-term data to improve models

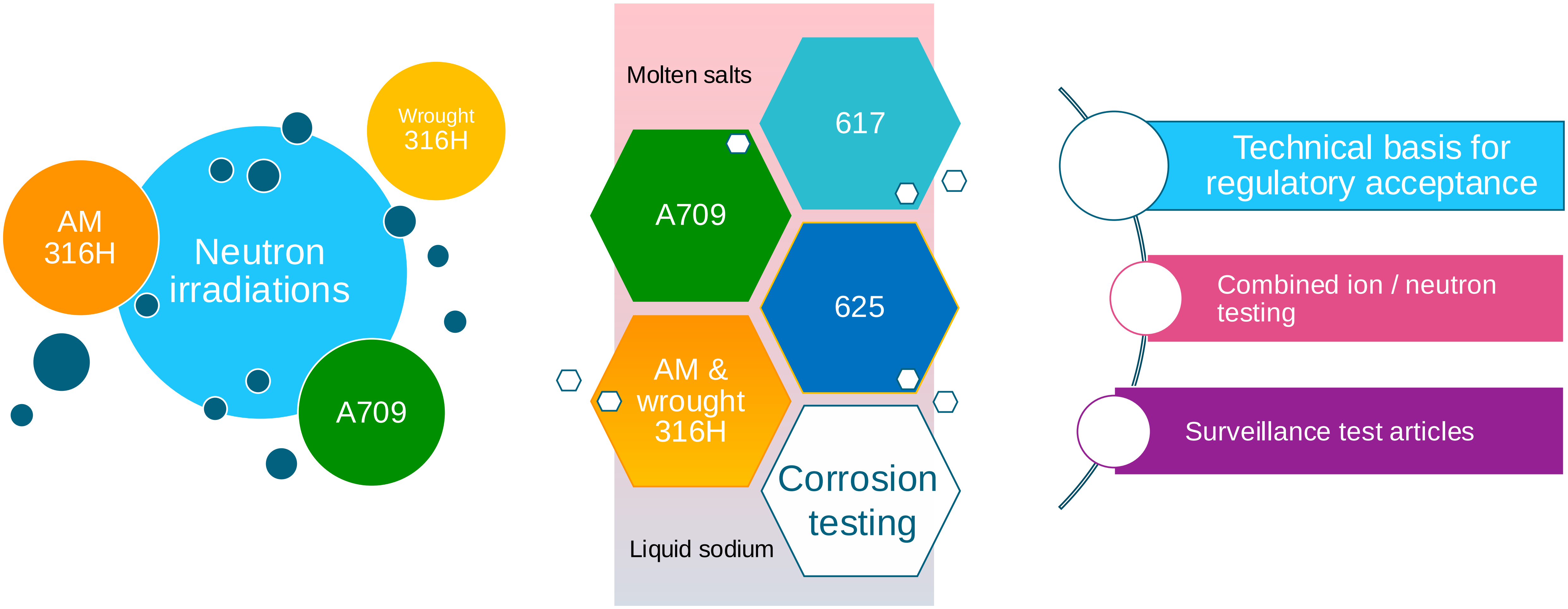
An integrated environmental effects testing strategy for AM 316H and other materials

- ★ Build
- ★ Ions
- ★ Neutrons
- ★ Corrosion





Activities in the Environmental Effects area



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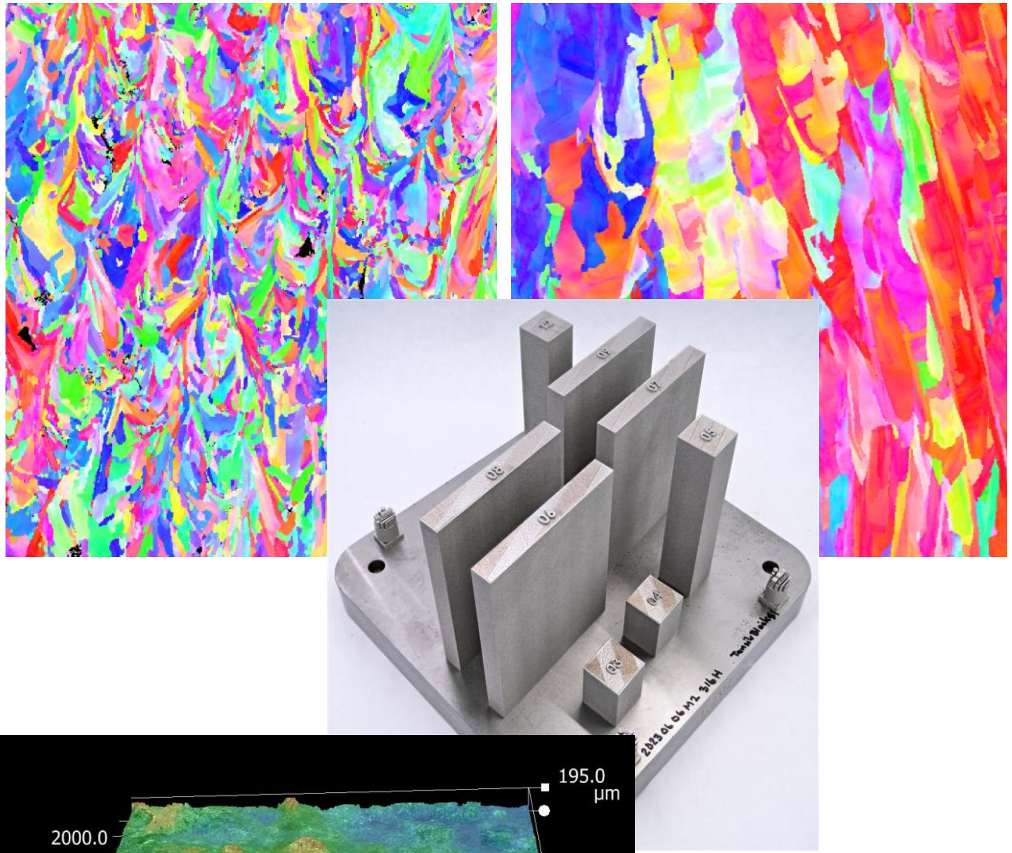
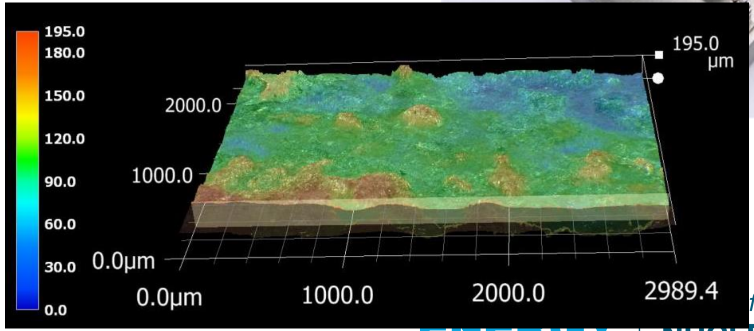
Corrosion testing

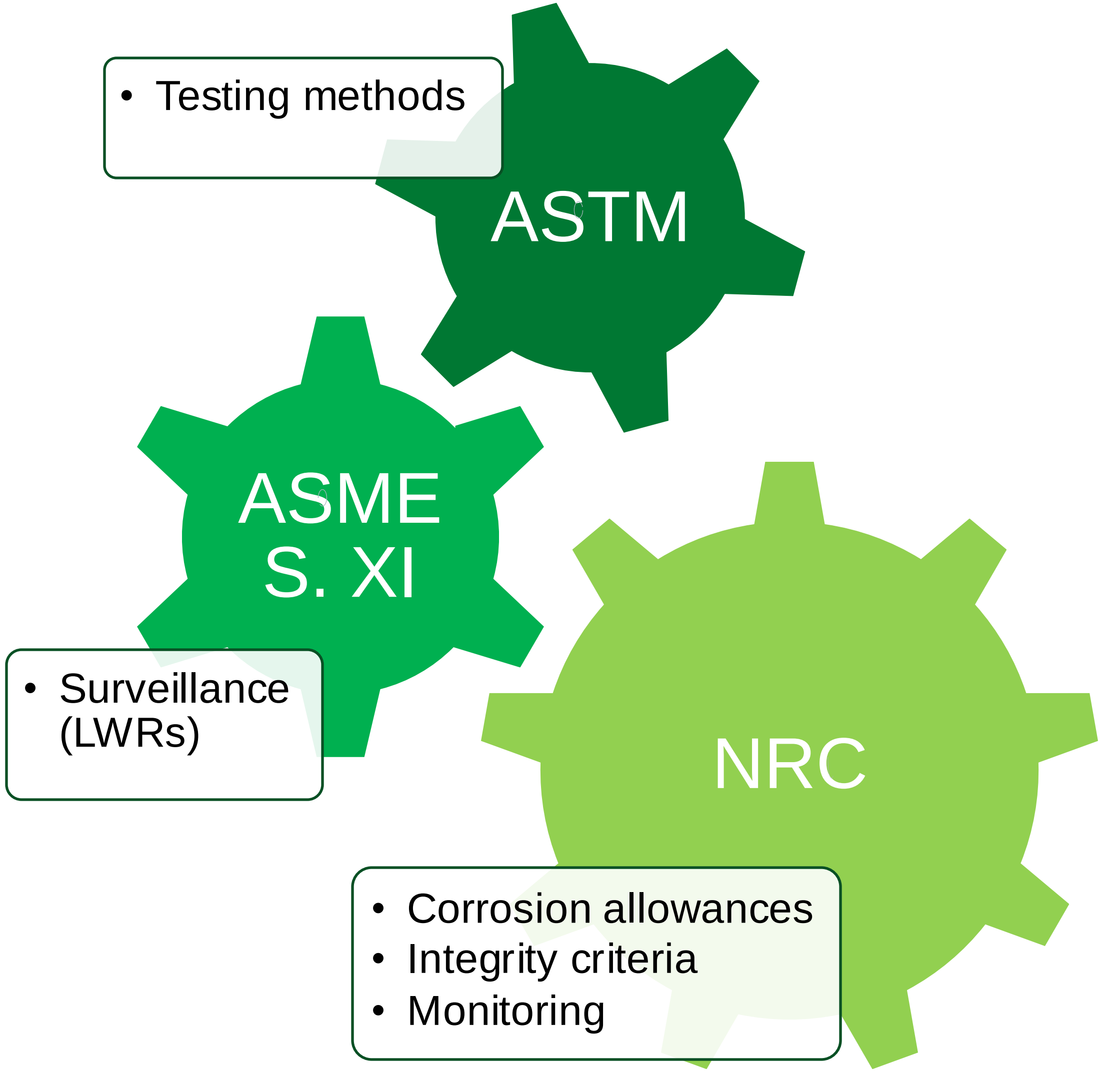
Unique aspects of AM components in nuclear reactor applications must be considered for corrosion

- **Components may be deployed without additional surface finishing**
  - As-built surface may improve or degrade corrosion properties
- **AM-specific features can intersect surface and alter corrosion vs wrought counterparts**
  - Build porosity, oxides, atypical inclusions, residual stresses, dislocation cells with chemical segregation
  - Melt pool boundaries, anisotropic grain structure
  - Uniform, pitting/crevice, electrochemical, corrosion fatigue corrosion may all be affected
- **Build process variability is inherent to AM materials**
  - Variations in as-built microstructure due to component geometry and build parameters
  - Feedstock lots and storage/handling





Corrosion is considered in a regulatory perspective

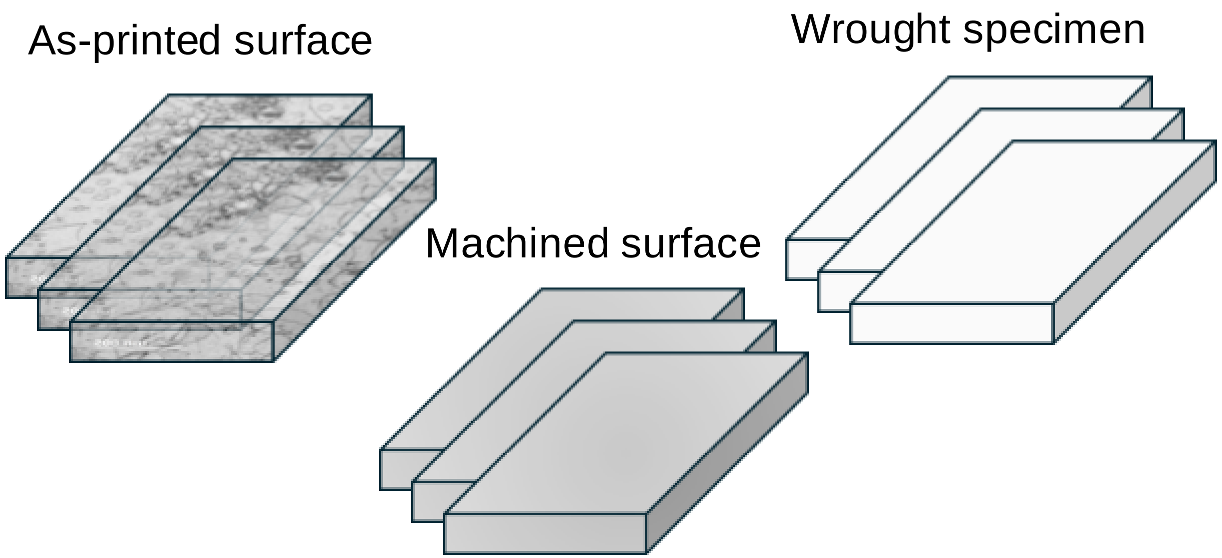


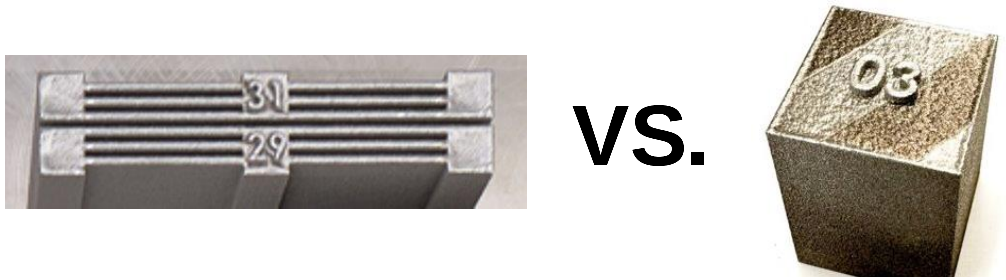
- **ASTM G1** – "Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens"
- **ASTM G31 G31/A** – "Standard Guide for Laboratory Immersion Corrosion Testing of Metals" including static and dynamic testing
- **ASTM A282** – "Standard Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels"
- **ASTM A763** – "Standard Practices for Detecting Susceptibility to Intergranular Attack in Ferritic Stainless Steels"
- **ASTM G48** – "Standard Test Methods for Pitting and Crevice Corrosion Resistance of Stainless Steels and Related Alloys by Use of Ferric Chloride Solution"
- **ASTM G129** – "Standard Practice for Slow Strain Rate Testing to Evaluate the Susceptibility of Metallic Materials to Environmentally Assisted Cracking"

Integrated workflow for molten chloride salt and liquid sodium testing

- **Develop machining and sample preparation workflow strategies**
  - Sample handling
  - Specimen machining and material tracking
  - Cleaning and descaling
- **Characterization of AM SS316H: prioritized processing conditions**
  - Heat treatment (as-printed vs. SA, etc.)
  - Surface condition
  - Orientation: build direction
  - Compare to wrought

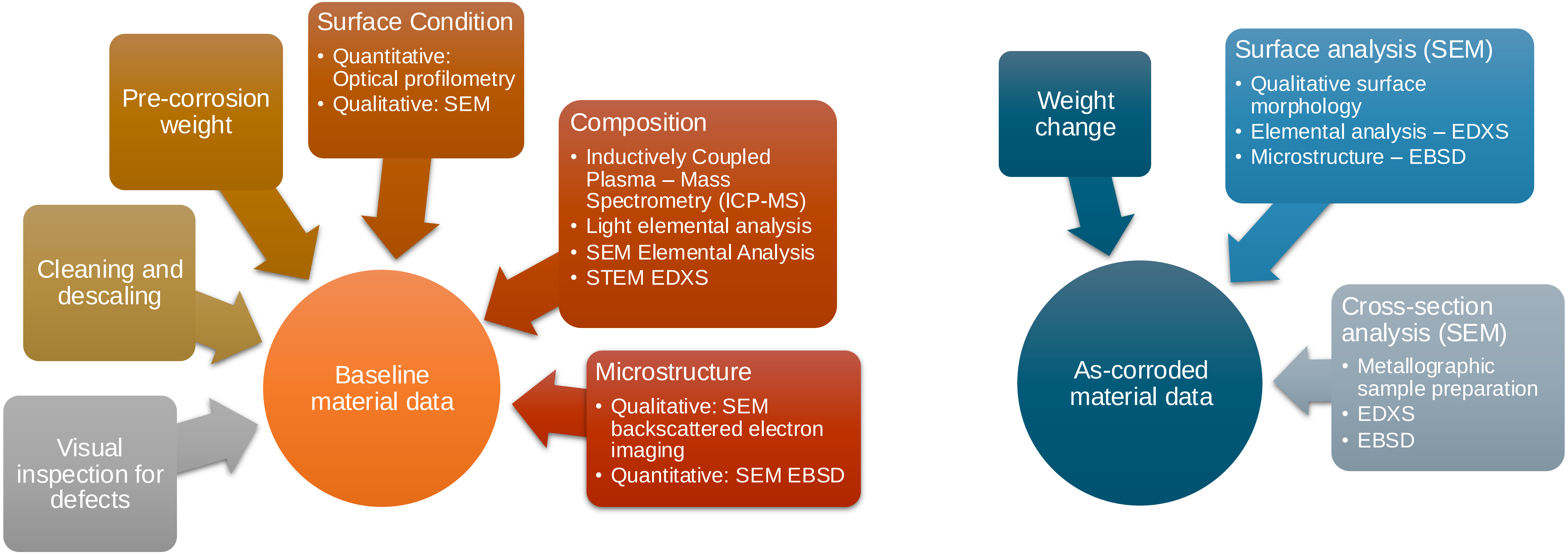
Goal: Determine the unique impact of AM processing conditions on the corrosion performance of AM 316H SS components





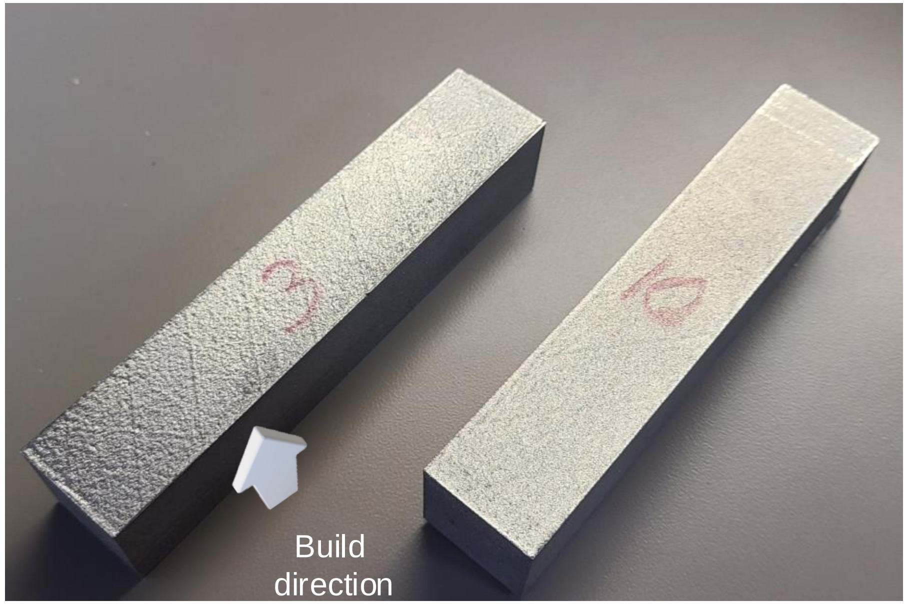
Thin-walled versus thick geometries may result in different microstructures, impacting corrosion performance despite being built using the same powder and processing conditions.

Pre- and post-test material evaluation

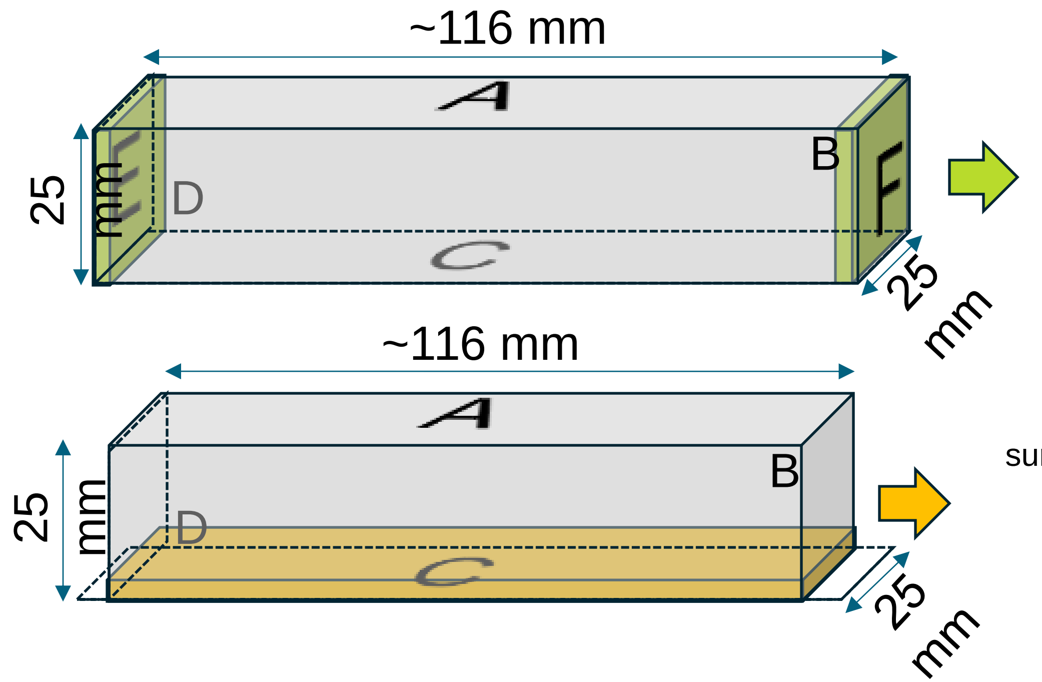


Molten chloride testing

- **Experimental design: static corrosion tests**
  - NaCl-$MgCl_2$ eutectic salt
  - Initial test period: 500 h at 550 C and 650 C
  - Include samples to assess thermal aging effects
  - Include wrought specimens for comparison

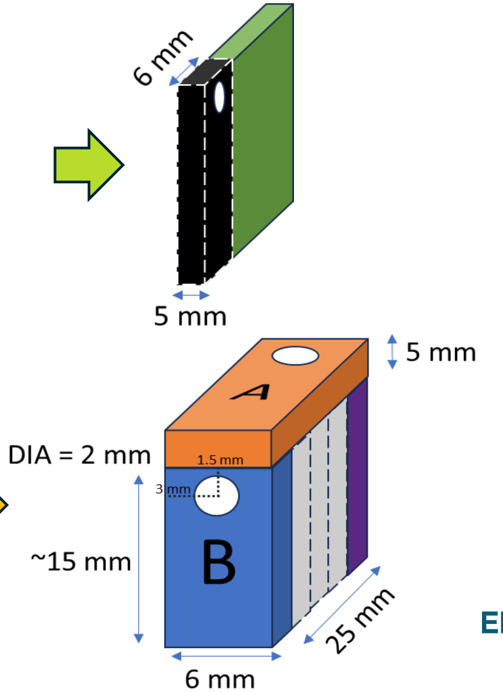


- **25 x 25 x 126 mm (1 x 1 x 6") LPBF SS316H samples**



Remove 5 mm from surfaces E and F

Remove 5 mm from surface C, then section the remainder as shown:



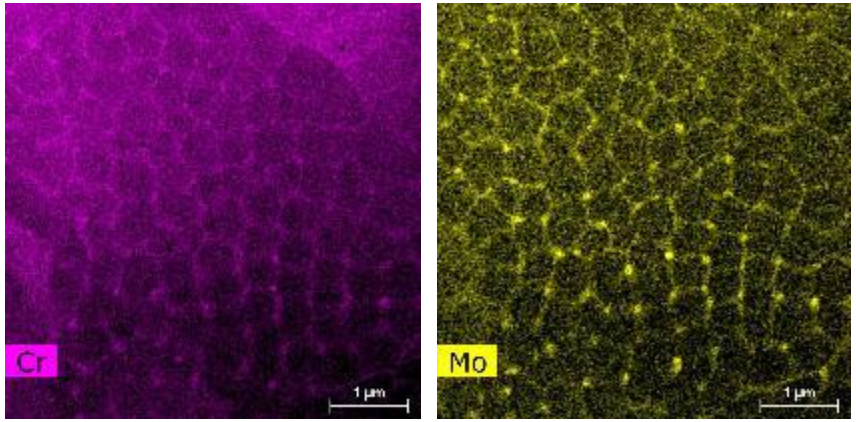
ERGY

Sodium exposure for AM 316H SS

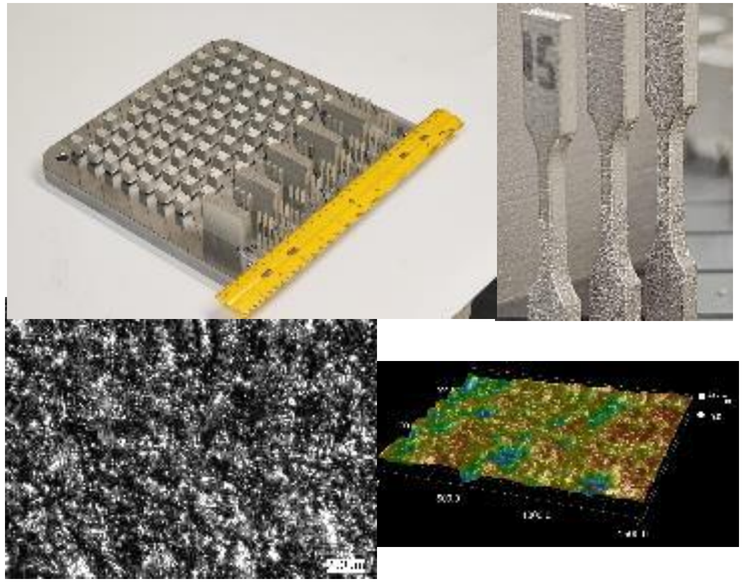
Objective – to assess the impact of AM characteristics on performance in flowing sodium

- Rough surface finish
- Porosity, anisotropic microstructure, cellular microstructure with segregation, post-build treatments
- Performed thermodynamic analysis and determined AM 316H will carburize in typical SFR environment

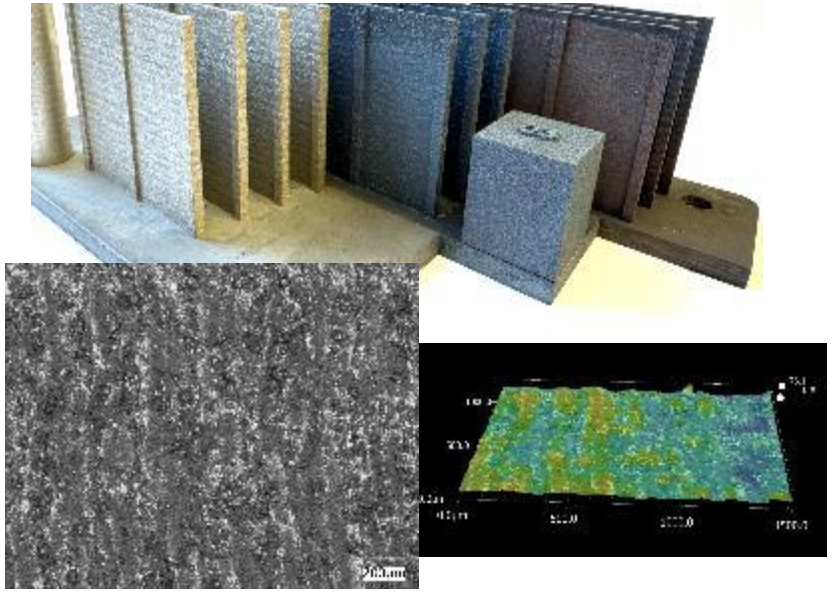
Cellular microstructure with segregation



As-printed tensile samples, provided by Zhang & Mantri (ANL)

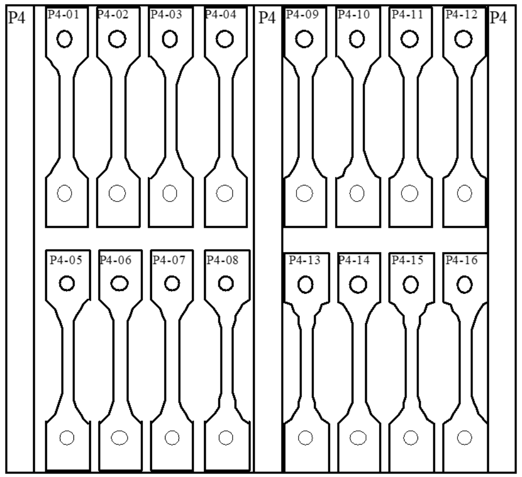


Plates and cube samples with as-printed surface finish, provided by Massey (ORNL)



Flat tensile samples are being machined to prepare for sodium exposure tests.

Plate-4, Solution-annealed



Neutron & ion irradiation

AMMT neutron irradiation goals and strategy

Support industry deployment of new AM / conventional materials

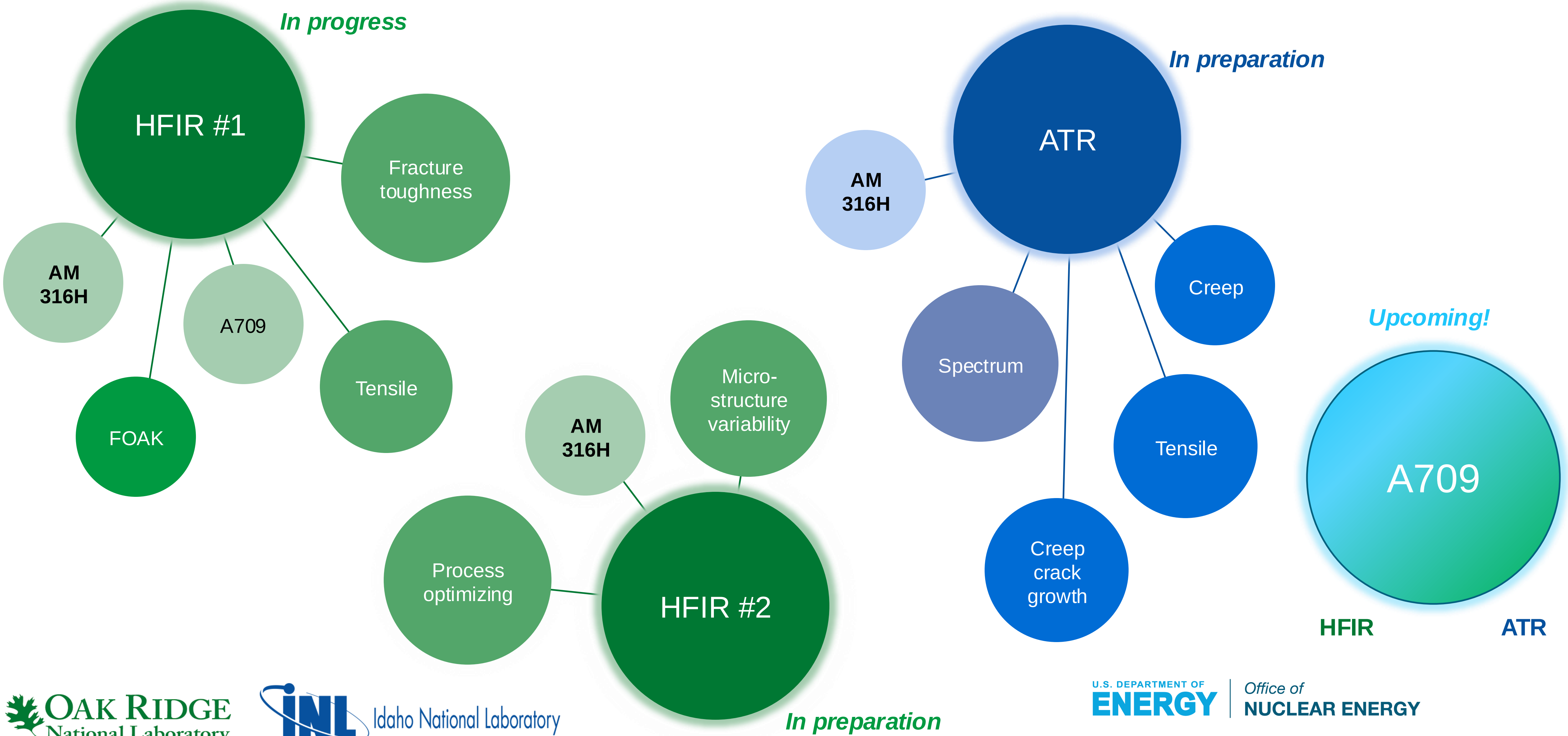
- Provide first-of-a-kind neutron irradiation data of AM 316H in the range of 400 °C – 600 °C up to 10 dpa
- Support the deployment of A709 in advanced reactors
- Provide information about process/microstructure variability on neutron irradiation behavior
- Support development of accelerated qualification processes using combined neutron and ion irradiation data set methodology

Data of engineering importance supported by scientific understanding

Combined campaign leveraging both INL and ORNL capabilities and expertise to maximize the information gained for materials within the program

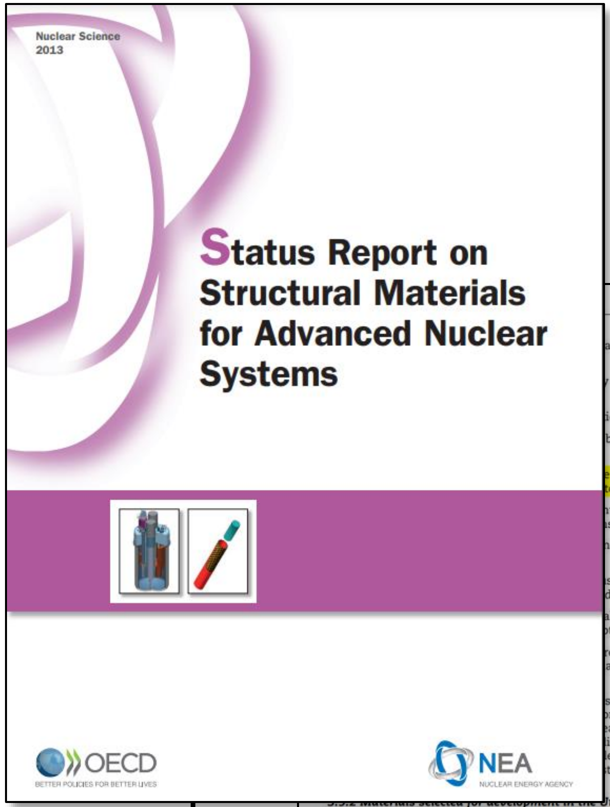
AMMT | INL Idaho National Laboratory | Oak Ridge National Laboratory | U.S. Department of Energy, Office of Nuclear Energy

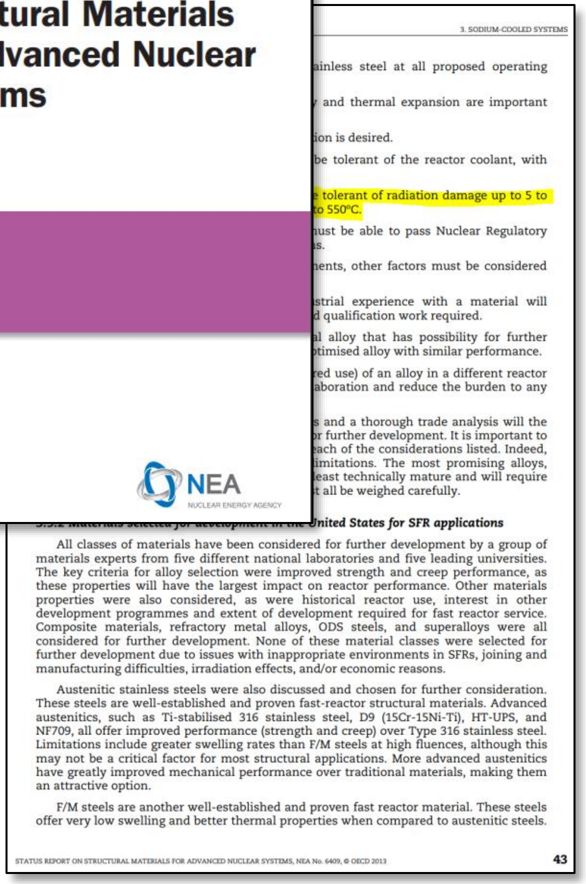
Neutron irradiation campaign overview



HFIR #1 irradiation test matrix

- **2 levels of irradiation damage: <u>2 and 10 dpa</u>**
  - HFIR irradiation < 1 year
  - In line with the NEA Status report on structural materials for Advanced Nuclear Systems
- **2 irradiation temperatures: <u>400°C and 600°C</u>**
  - Targeting the expected temperature range to deploy 316H (versus 316L)





3. SODIUM-COOLED SYSTEMS

...United States for SFR applications

All classes of materials have been considered for further development by a group of materials experts from five different national laboratories and five leading universities. The key criteria for alloy selection were improved strength and creep performance, as these properties will have the largest impact on reactor performance. Other materials properties were also considered, as were historical reactor use, interest in other development programmes and extent of development required for fast reactor service. Composite materials, refractory metal alloys, ODS steels, and superalloys were all considered for further development. None of these material classes were selected for further development due to issues with inappropriate environments in SFRs, joining and manufacturing difficulties, irradiation effects, and/or economic reasons.

Austenitic stainless steels were also discussed and chosen for further consideration. These steels are well-established and proven fast-reactor structural materials. Advanced austenitics, such as Ti-stabilised 316 stainless steel, D9 (15Cr-15Ni-Ti), HT-UPS, and NF709, all offer improved performance (strength and creep) over Type 316 stainless steel. Limitations include greater swelling rates than F/M steels at high fluences, although this may not be a critical factor for most structural applications. More advanced austenitics have greatly improved mechanical performance over traditional materials, making them an attractive option.

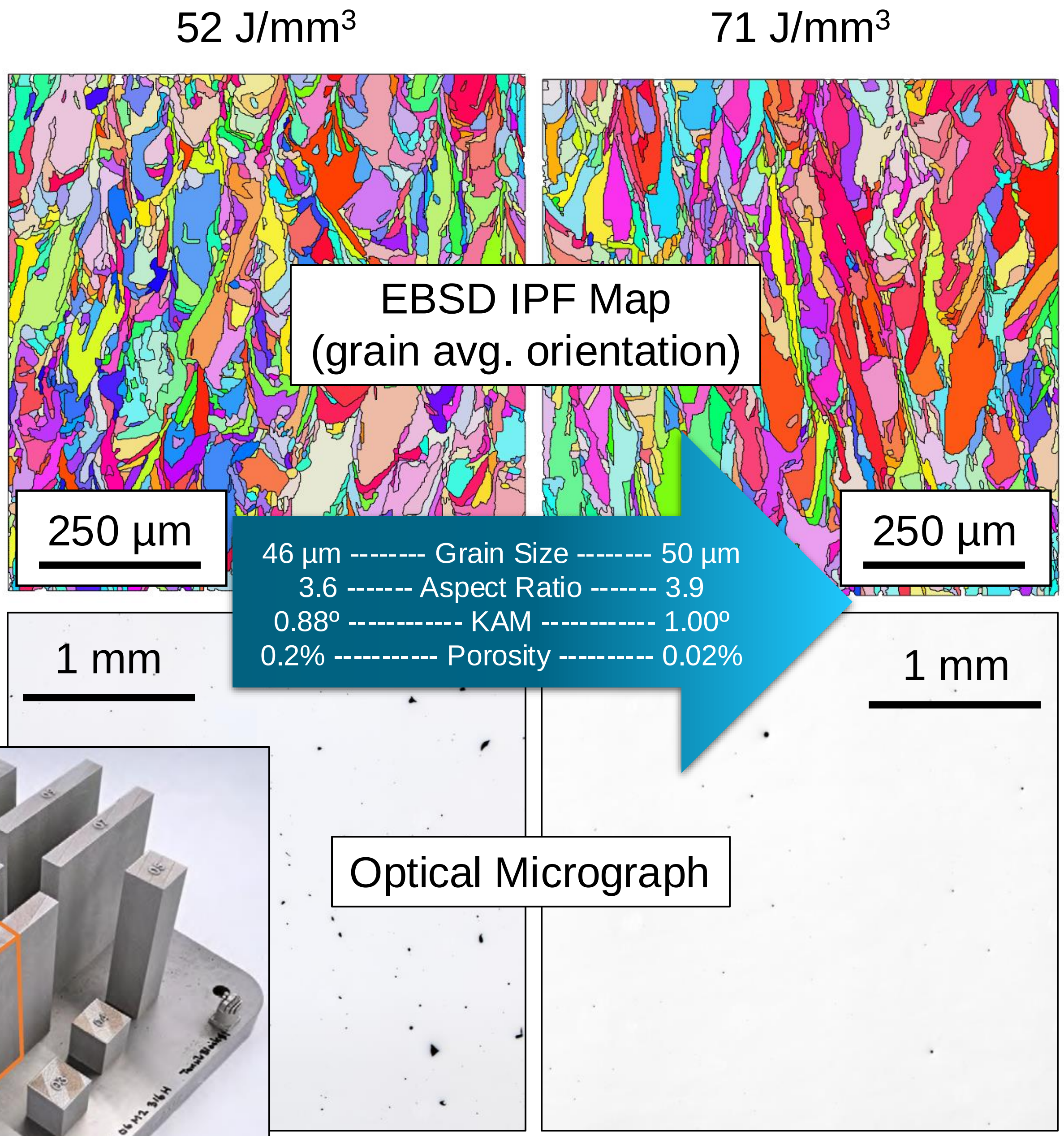
F/M steels are another well-established and proven fast reactor material. These steels offer very low swelling and better thermal properties when compared to austenitic steels.

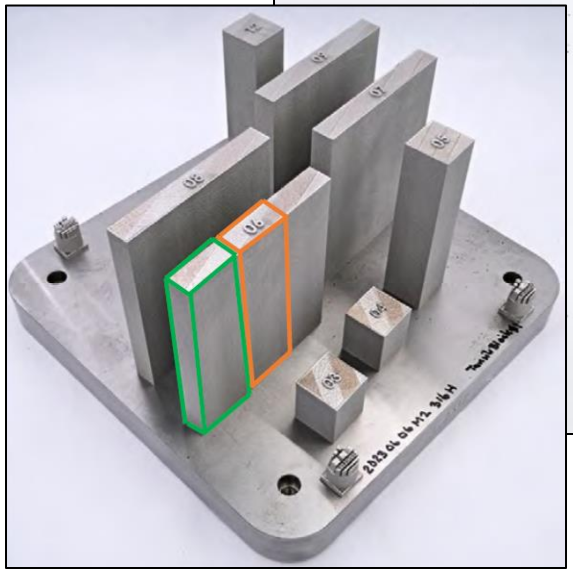
STATUS REPORT ON STRUCTURAL MATERIALS FOR ADVANCED NUCLEAR SYSTEMS, NEA No. 6409, © OECD 2013 43

Radiation resistance: the material must be tolerant of radiation damage up to 5 to 10 dpa, at temperatures ranging from 400 to 500°C

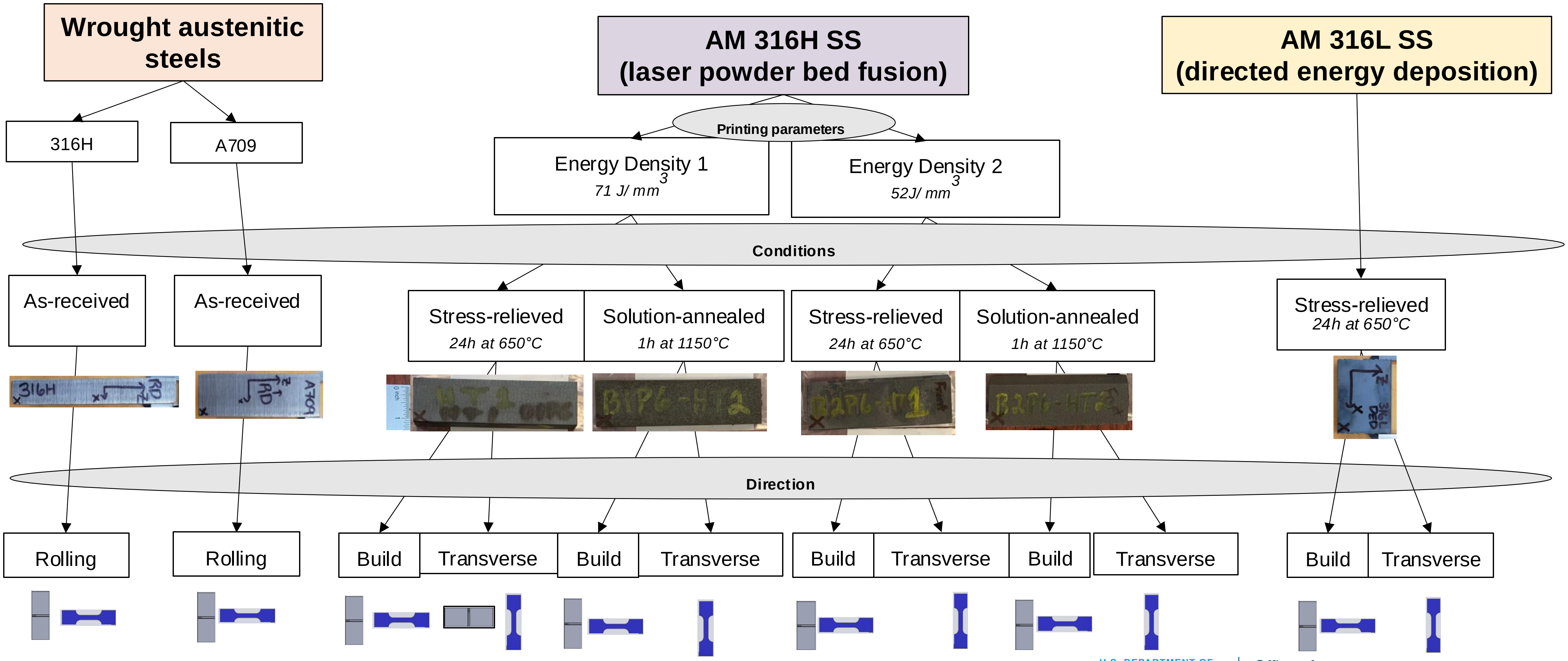
Variants of LPBF 316H Evaluated in HFIR #1

- Prior FY processing parameter optimization study produced **fully dense** material (0.2% and .02% porosity) at **intermediate energy density values** (52 J/mm^3 and 71 J/mm^3)
- However, this porosity minimization is accompanied by a transition from fine weld pool microstructures (chevron/globular) to more elongated columnar grains due to epitaxial growth at high energy densities.
- Consequently, two sets of processing parameters with **minimized grain growth, and low porosity**, respectively, are scoped in the current irradiation campaign.



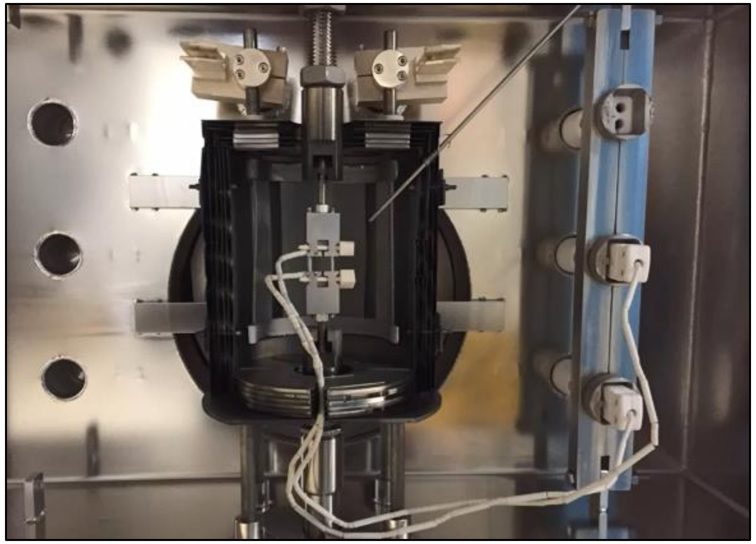


HFIR irradiation – materials of interest



HFIR irradiation testing status

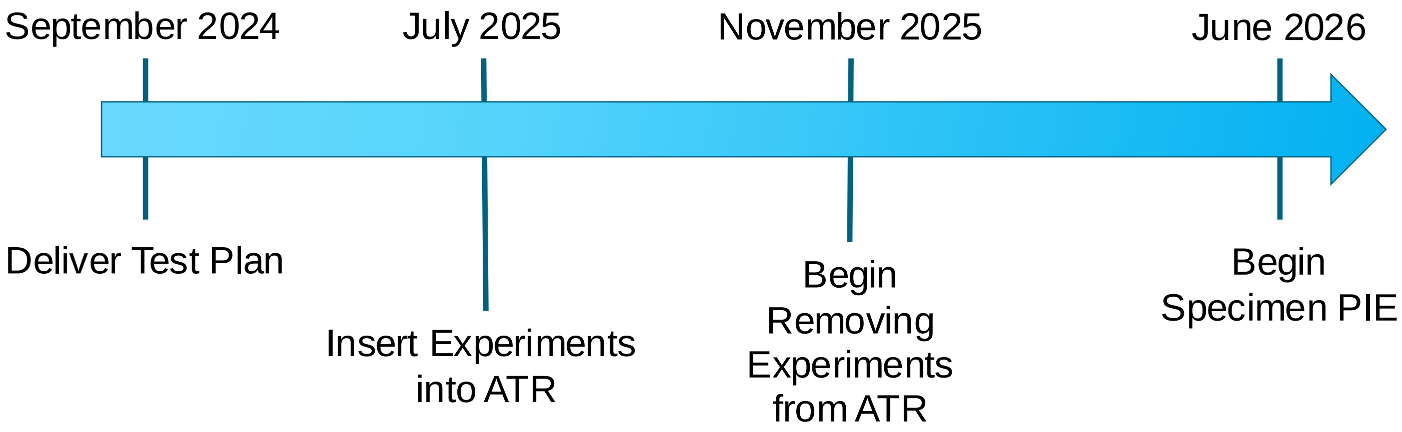
- **2 dpa capsules**
  - Irradiation complete for all tensile capsules and 2 of 12 bend bar
  - All other bend bar capsules to complete irradiation in July
  - PIE to start this summer and expected to be completed by the spring 2025
- **10 dpa capsules**
  - Irradiation will be completed for all capsules by May 2025
  - PIE expected to be completed by the spring 2026
- **PIE plan**
  - Tensile testing (2 specimens per condition) at room temperature and at irradiation temperature
  - Fracture toughness testing (1 specimen per condition) at room temperature and irradiation temperature
  - Thermal aging study at 600°C for comparison with 2 dpa neutron-irradiated data

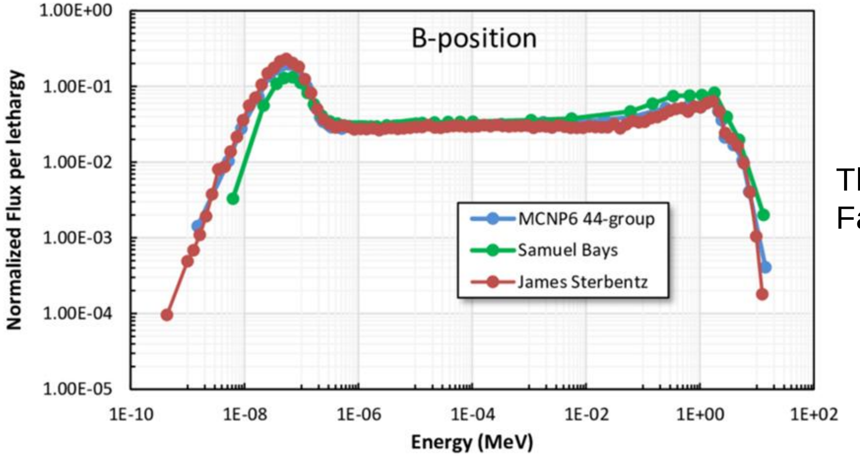


Irradiation in preparation: ATR

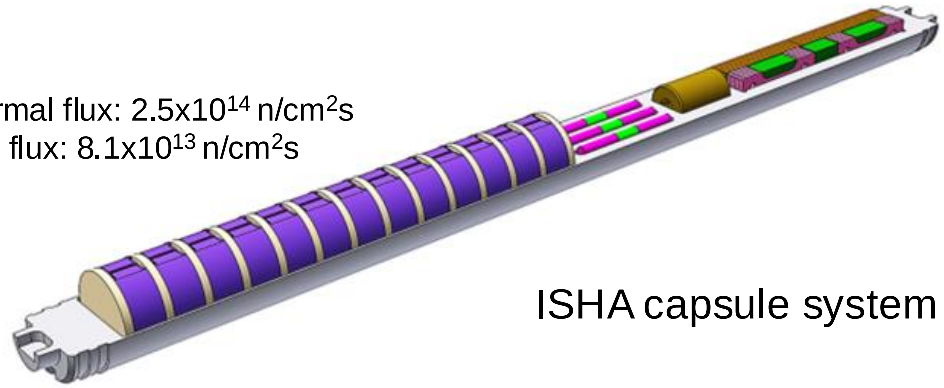
- **Purpose: Overlap AM 316H irradiation with HFIR irradiations up to 2 dpa and provide additional industry-relevant mechanical properties**
- **Lower dpa targets for ATR**
  - 60-day cycle, 0.5-1dpa/cycle
  - 400 °C and 600 °C
  - 1 and 2 dpa, 3 cycles total
- **AM 316H**
- **SSJ3 specimens**
  - Tensile tests
  - Creep tests
- **CT specimens**
  - Fracture toughness
  - Creep crack growth

Cycle 175-B





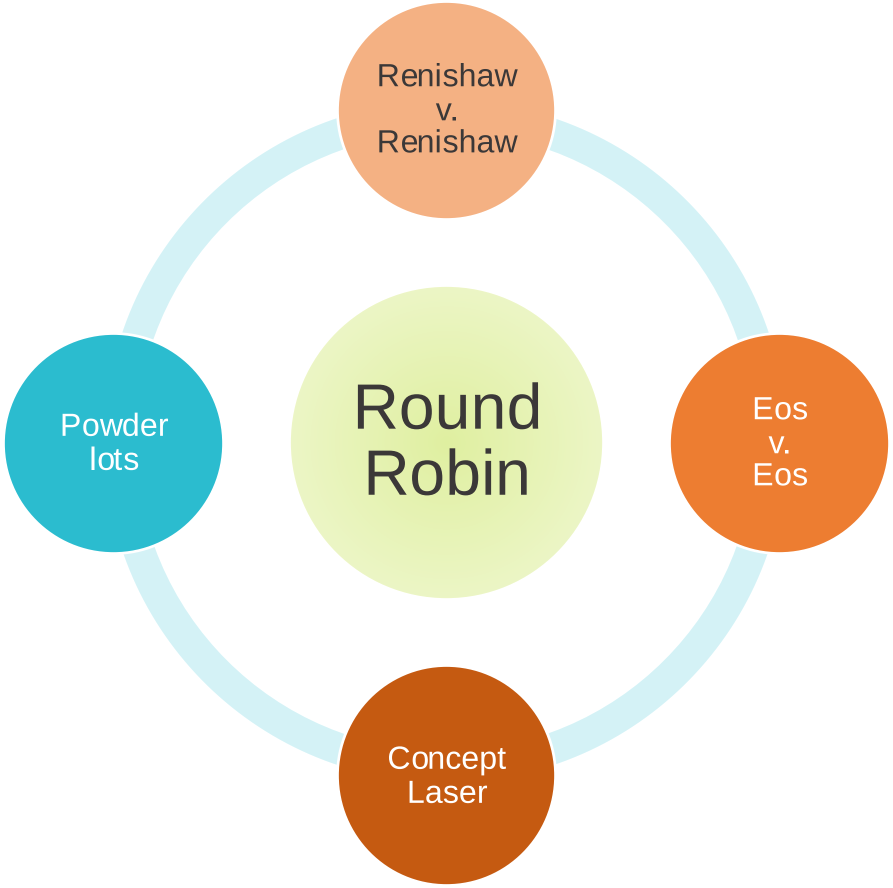
Thermal flux: 2.5×10^{14} n/cm^{2}s
Fast flux: 8.1×10^{13} n/cm^{2}s



ISHA capsule system

Irradiation in preparation: HFIR #2

- **Target Insertion Q3 or Q4 FY25**
- **Include:**
  - Optimized builds in round robin prints
  - Build and transverse orientation tensile data
    - Build direction provides lowest strength, highest ductility
    - Transverse direction provides highest strength, lowest ductility
  - Fracture toughness in most conservative orientation
- **Overlap with some irradiation conditions of HFIR irradiation #1 to enable use of that data for batch-to-batch compositional variation**



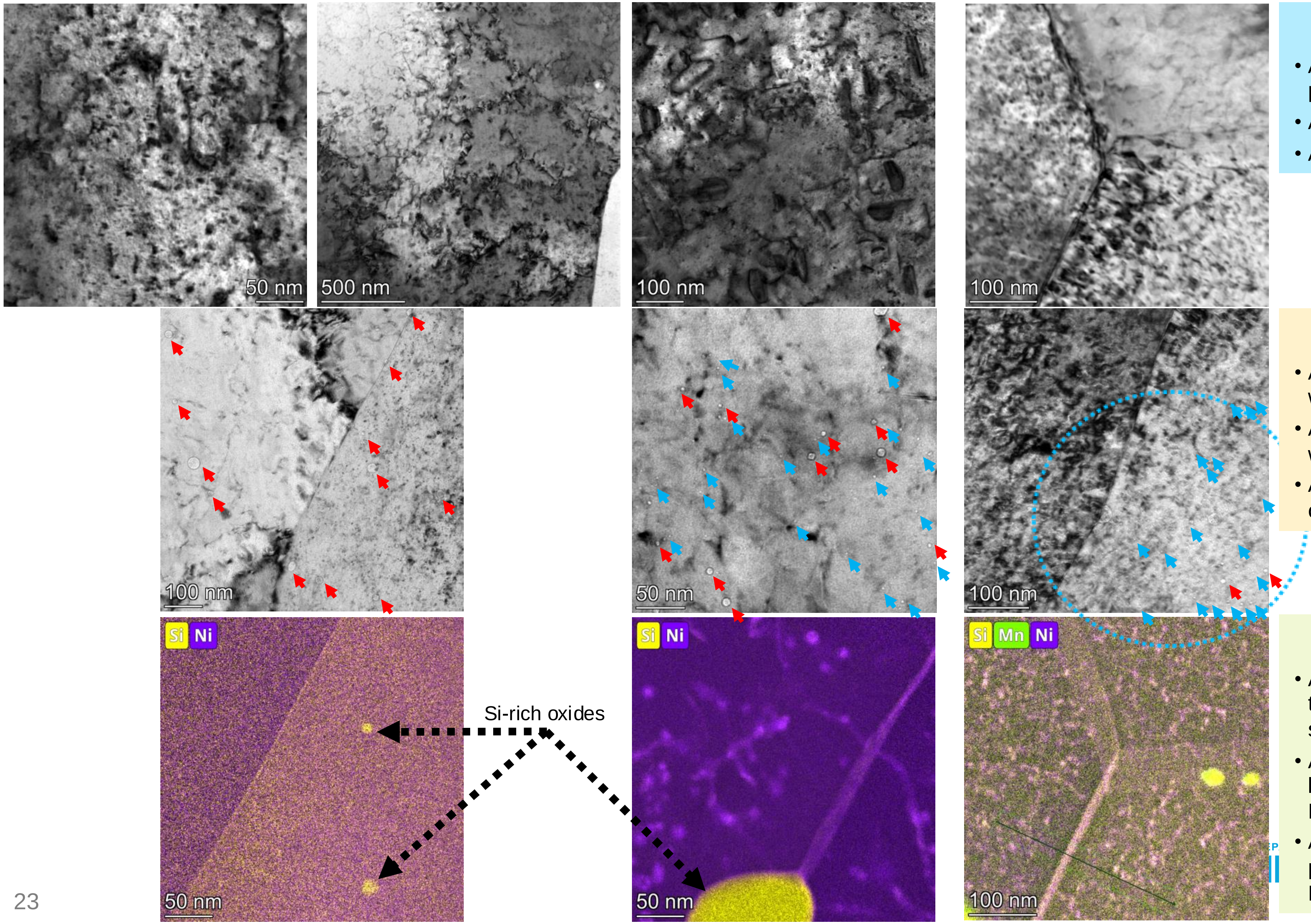
Results from HFIR irradiation of AM 316L SS

- **Radiation Effect and Microstructure:**
  - The fine grain, high dislocation density microstructure of AM 316L resulted in higher initial strength, lower ductility, and lower creep life when compared with the reference 316L.
  - In the AM 316L SS (in particular, stress-relieved condition), **high strength and ductility are retained at least up to 10 dpa at 600°C.**
  - **Loss of ductility was observed after irradiation at 300°C.**
  - Complete embrittlement (zero TE) might not occur in AM stainless steels until the dose reaches a few dozen dpa.
  - Ductilization was observed in 600°C irradiation, but at the lower doses (0.2 and 2 dpa) only.
- **Radiation Effect and Sampling Location:**
  - Irradiation increased the variation of tensile property data, particularly after 600°C irradiation.
  - **No clear dependence on build thickness or sampling location was observed.**
  - High temperature irradiation is believed to magnify the effect of initial variation in AM microstructure.

1. Byun, T. S., et al. "Mechanical behavior of additively manufactured and wrought 316L stainless steels before and after neutron irradiation." ***Journal of Nuclear Materials* 548 (2021)**: 152849.
2. Byun, T.S., et al. "Mechanical properties of additively manufactured 316L stainless steel before and after neutron irradiation – FY23". **ORNL/TM-2023/2919 (2023), OSTI**: 1974316.

As-printed LPBF 316L HFIR neutron irradiated at 300 °C target temperature

0.2 dpa, 250 °C | 2 dpa, 376 °C | 10 dpa, 277 °C



Dislocations

- At 0.2 dpa/ 250 °C , dislocation cell structure still partially present
- At 2 dpa/ 376 °C, larger dislocation loops
- At 10 dpa/277 °C, smaller dislocation loops

Cavities

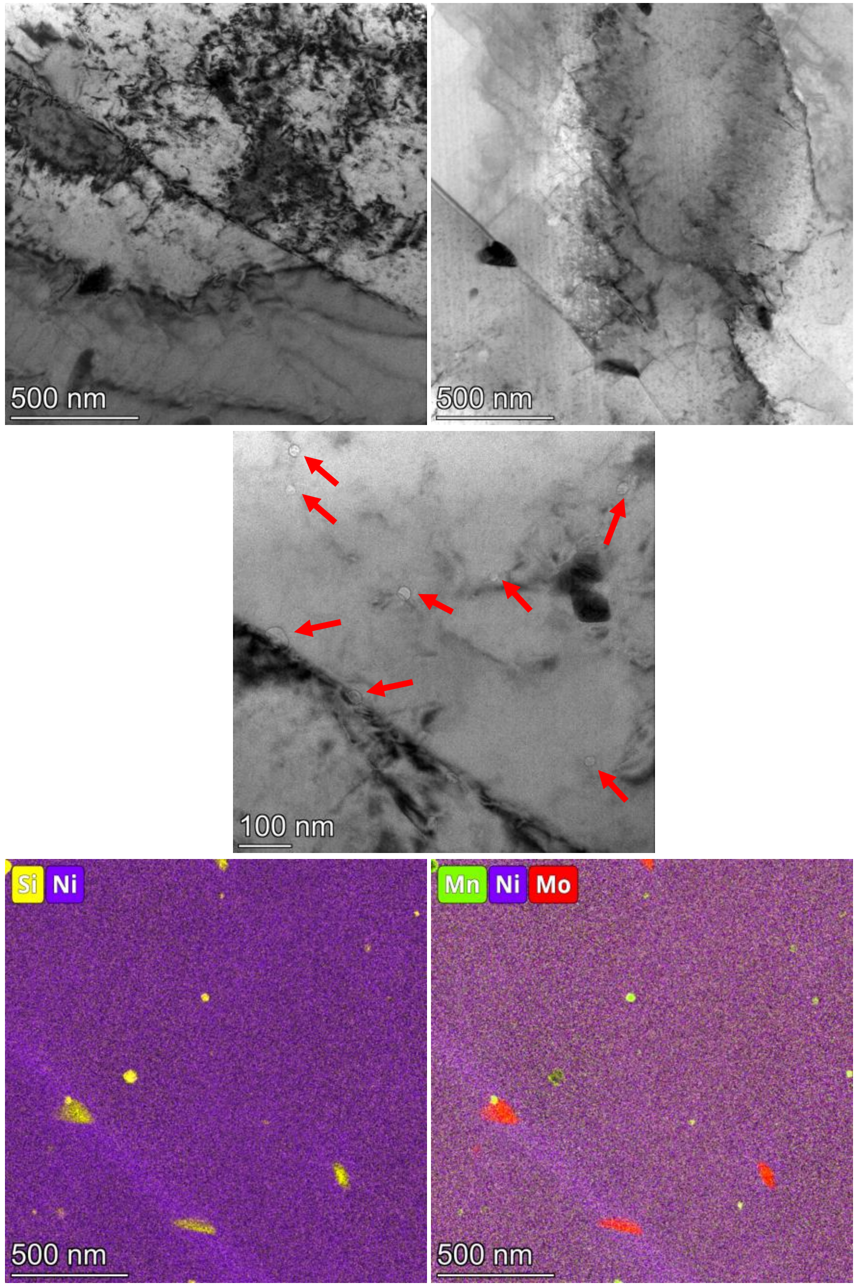
- At 0.2 dpa/ 250 °C, wide variation in cavity sizes with some at GB; not high density
- At 2 dpa/ 376 °C, wide variation in cavity sizes, with more visible on small end
- At 10 dpa/ 277 °C, high density of small < 3 nm cavities with some 5-10 nm cavities

Precipitation/ Segregation

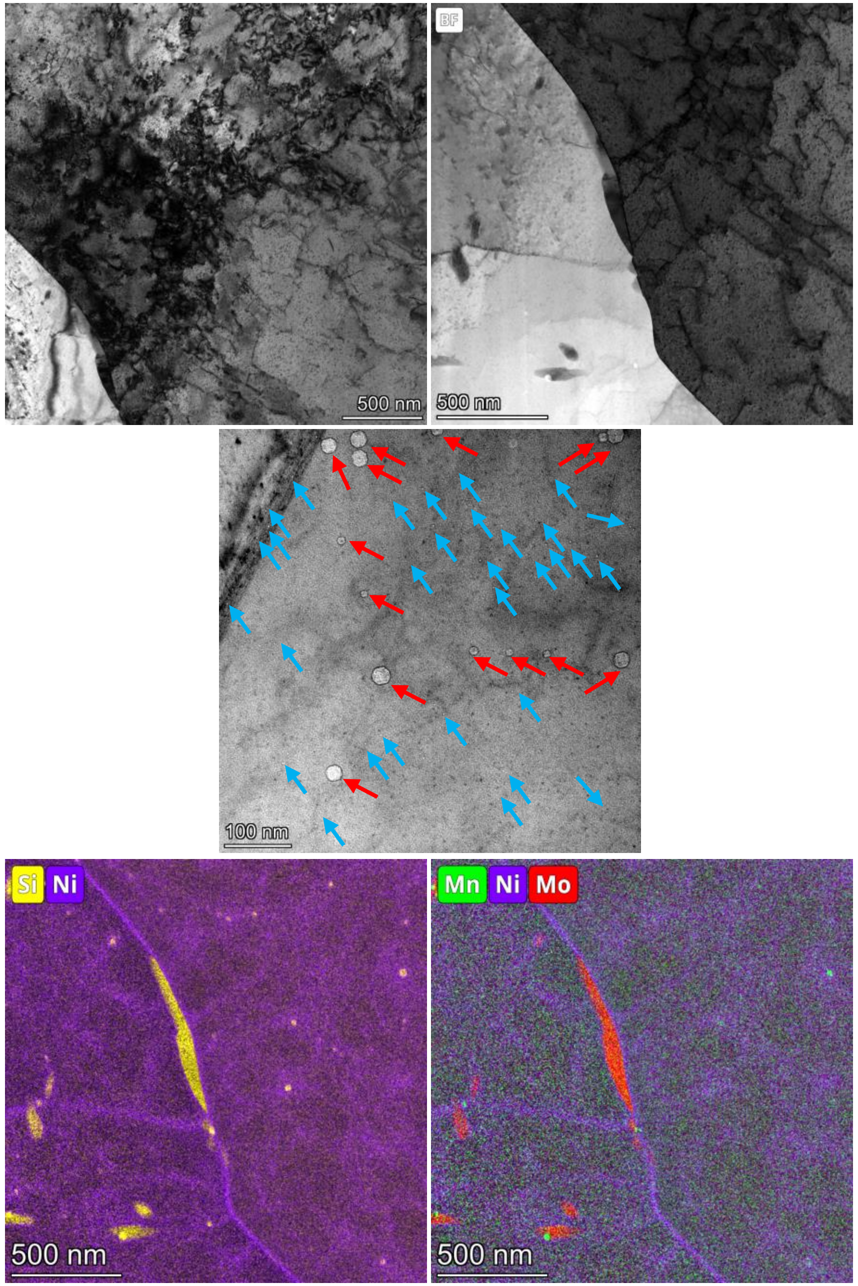
- At 0.2 dpa/ 250 °C, no precipitation, slight Si RIS to GB, as-printed cell walls still present but no segregation; Si-rich oxide particles
- At 2 dpa/ 376 °C, Ni/Si segregation to dislocation loops with precipitates on loops; and Ni/Si RIS to HAGBs; Si-rich oxide particles
- At 10 dpa/ 277 °C, high density of Ni/Si precipitation vs. segregation to dislocations; Ni/Si RIS to HAGBs; Si-rich oxide particles

As-printed LPBF 316L HFIR neutron irradiated at 600 °C target temperature

0.2 dpa, 673 °C



2 dpa, 600 °C



Dislocations

- At 0.2 dpa/ 673 °C, dislocation cell structure still partially present
- At 2 dpa/ 600 °C, network dislocations, no/limited cell structure

Cavities

- At 0.2 dpa/ 673 °C, only larger 10-20 nm cavities
- At 2 dpa/ 600 °C, bimodal distribution in cavity sizes, with high density of small < 3 nm cavities

Precipitation/ Segregation

- At 0.2 dpa/ 673 °C, no precipitation, slight Ni RIS to GB, as-printed cell walls still present but no segregation; Si-rich oxide particles and Cr/Si/Mo/P inclusions
- At 2 dpa/ 600 °C, Ni segregation to dislocation lines and grain boundaries (very faint Si and P to grain boundaries); Si-rich oxide particles and Cr/Si/Mo/P inclusions

Damage rate effect

- <u>LPBF316L-AP</u>
- 10^{-3}, 10^{-4}, 10^{-5} dpa/s
- 0.2, 2 dpa
- 300°C, 600°C

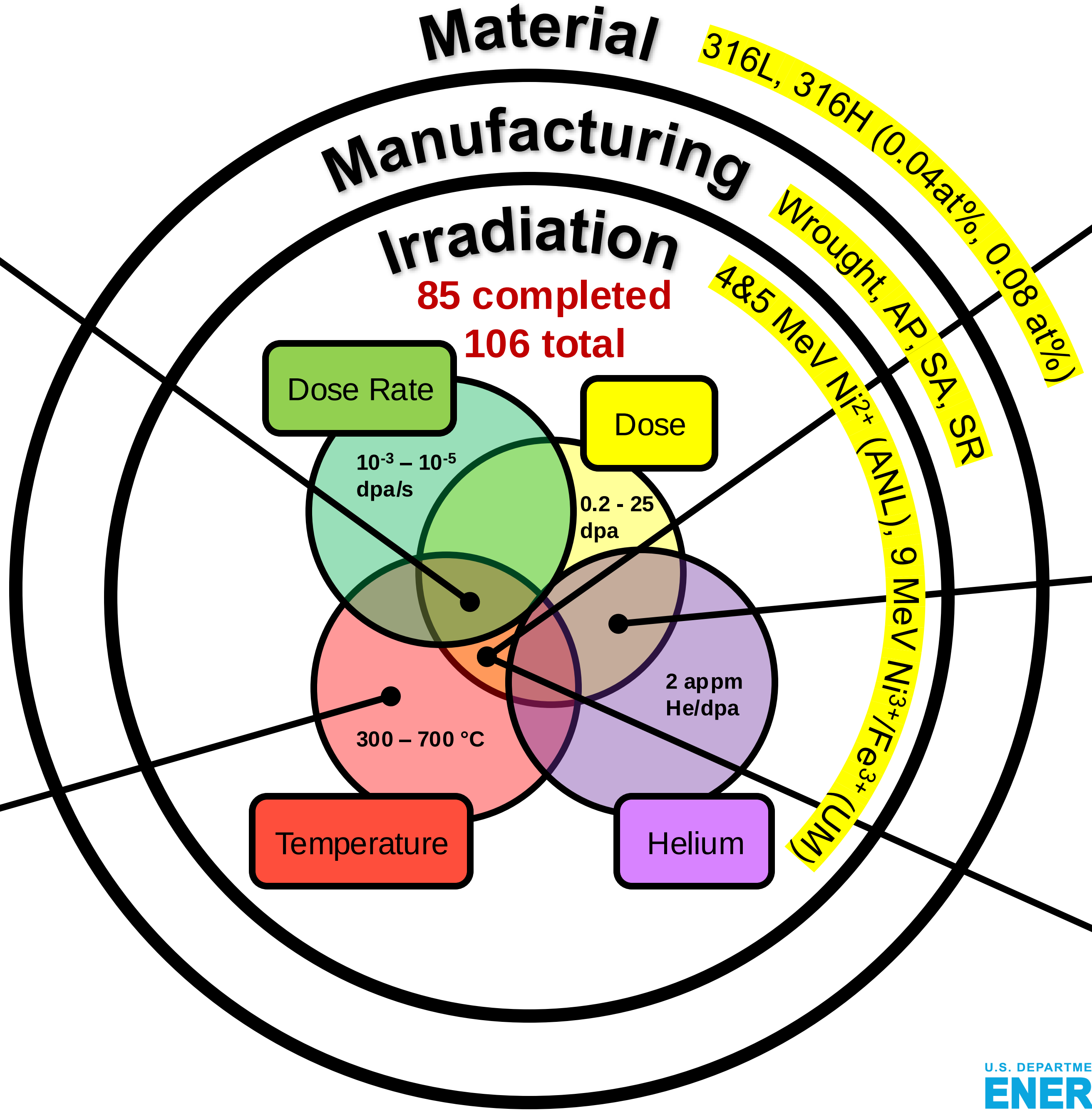
- <u>LPBF316H-SA/SR</u>
- 10^{-3}, 10^{-4} dpa/s
- 2 dpa
- 400°C, 600°C

Temperature Dependence

- LPBF316H-SA/SR
- 10^{-3} dpa/s
- 10 dpa
- 300, 400, 500, 600, 700 °C

- <u>LPBF316H-AP</u>
- 10^{-3} dpa/s
- 5 dpa
- 300, 400, 500, 600 °C

- <u>W316L</u>
- 10^{-3} dpa/s
- 10 dpa
- 300, 400, 500, 600 °C



Carbon effect

- <u>LPBF316L-AP</u>, <u>LPBF316H-AP</u>
- 10^{-3} dpa/s
- 300, 600 °C
- 0.2, 2, 5, 10, 25 dpa

- <u>W316L</u>, <u>W316H</u>
- 10^{-3} dpa/s
- 600 °C
- 2, 10

Helium effect

- LPBF316H-AP/SR/SA, W316H
- 7×10^{-4}+2 appm He/dpa
- 575 °C
- 2, 10, 25 dpa

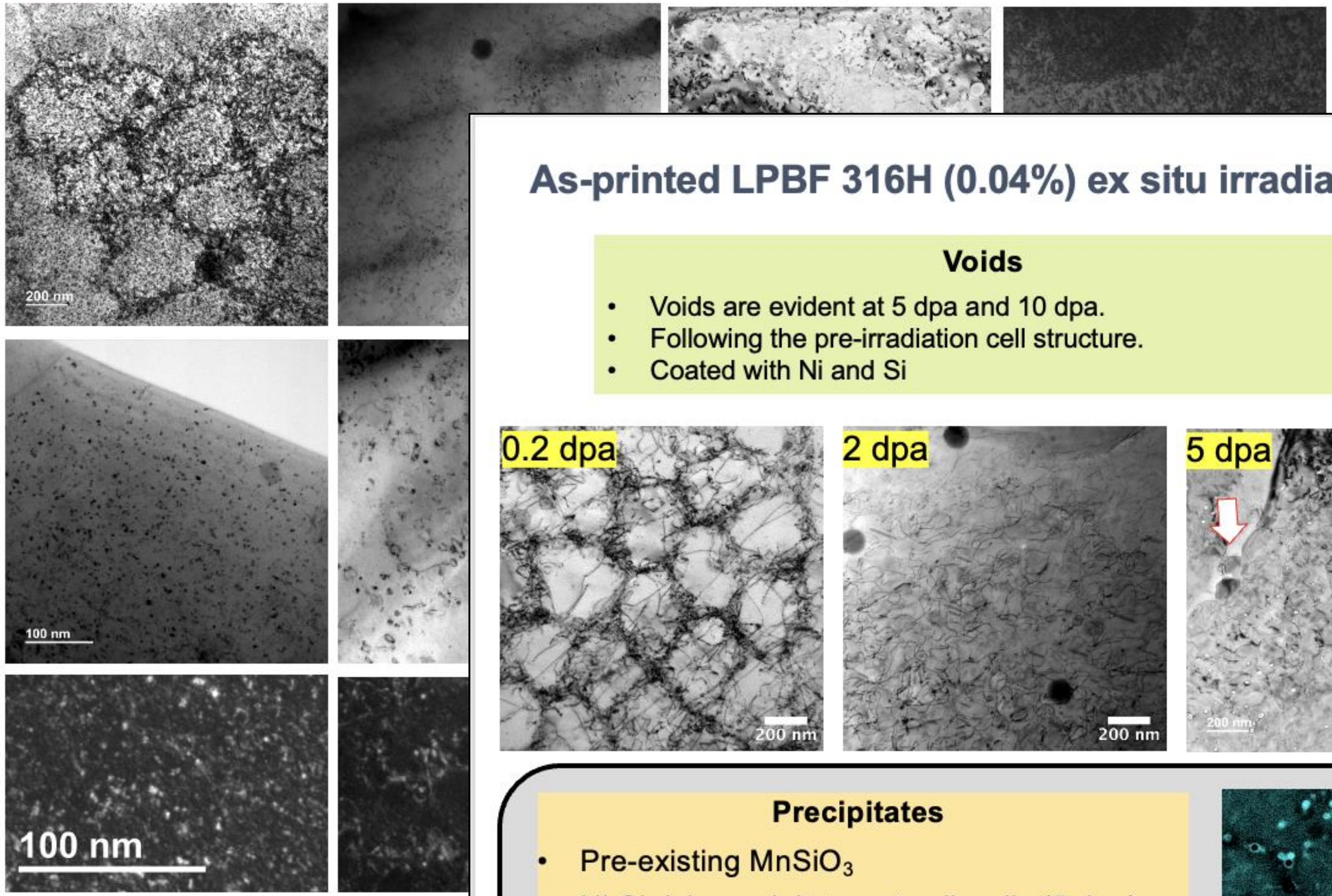
Heat Treatment Effect

- <u>LPBF316L-AP/SA</u>
- 10^{-3} dpa/s
- 300, 600°C
- 0.2, 2, 5, 10

- LPBF316H-SR/SA
- 10^{-3} dpa/s
- 400, 600°C
- 0.5, 2, 10 dpa

<u>**Underlined**</u>: irradiation complete

As-printed LPBF 316H *ex situ* irradiated with 4 MeV Ni ions at 300°C (10^{-3} dpa/s)



Dislocations

- Dislocation cell structure

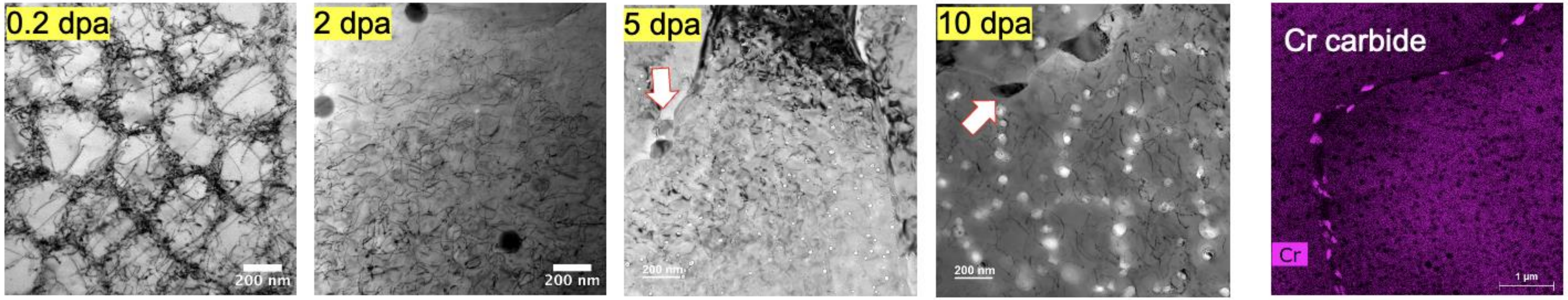
As-printed LPBF 316H (0.04%) ex situ irradiated with 4 MeV Ni ions at 600°C (10^{-3} dpa/s)

Voids

- Voids are evident at 5 dpa and 10 dpa.
- Following the pre-irradiation cell structure.
- Coated with Ni and Si

Dislocations

- Dislocation cell structure still observed at 0.2 dpa.
- Uniform dislocation network well developed at 2 dpa
- Low density of dislocation loops

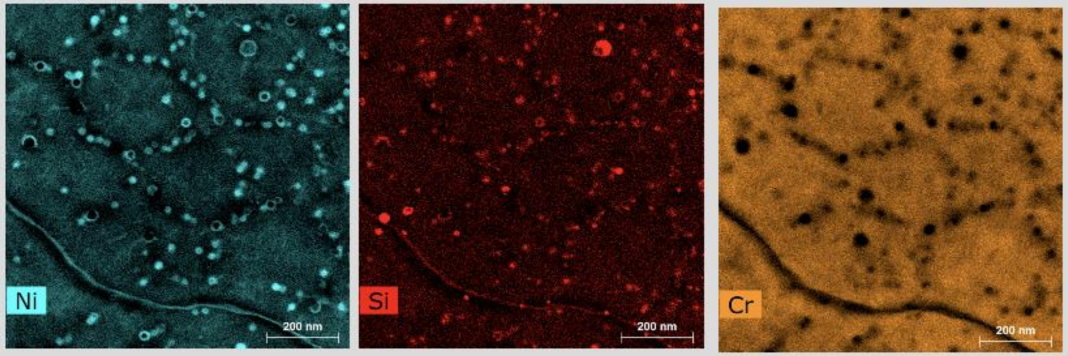


Precipitates

- Pre-existing $MnSiO_3$
- Ni-Si rich precipitates at cell walls (5 dpa)
- Cr carbide at GB (starting at 2 dpa)

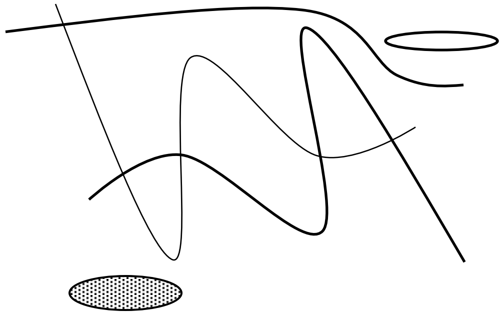
RIS

Ni, Si Enriched, Cr depleted



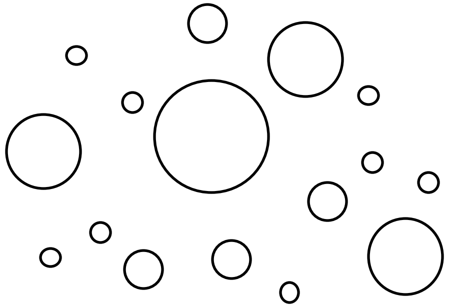
Ion vs neutron irradiation – summary of results

Dislocations



Dislocation cell structure disappears by 2 dpa in both ion and neutron irradiated AM 316 at both low (~300 °C) and high (~600 °C) temperatures

Cavities



Qualitative similarity, but cavities observed at lower temperature in neutron irradiation than in the ion irradiation, likely from helium generation

Segregation

Qualitative similarity for elemental segregation type (depletion / enrichment) for grain boundaries and line dislocations

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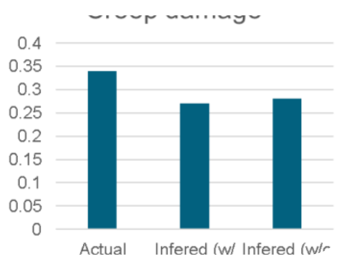
Materials surveillance test articles

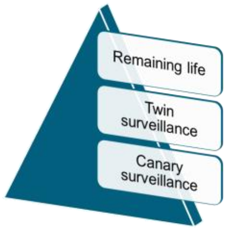
- **Structural materials experience both mechanical and environmental degradation**
- **A material surveillance program can monitor material degradation in service to mitigate the risk posed by the limited up-front test data**
- **This work seeks to develop the technology required to implement a surveillance program in an operating plant**

Robust test articles

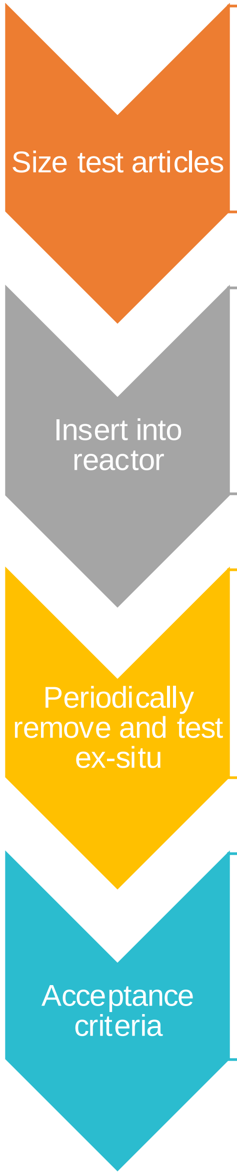

Sizing procedures



Damage inference



Acceptance procedures



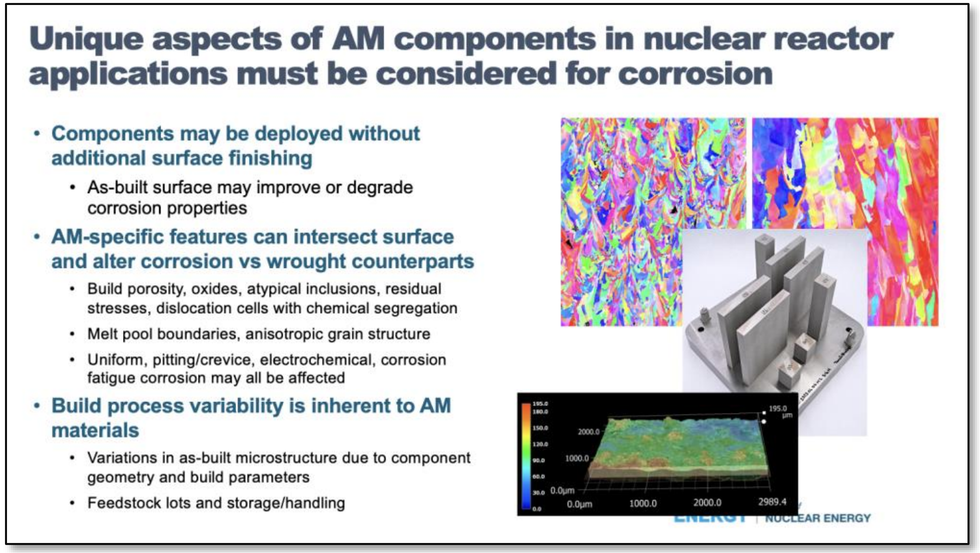
Size test articles
- Sizing procedures to mimic component loading
- Passively actuated test articles

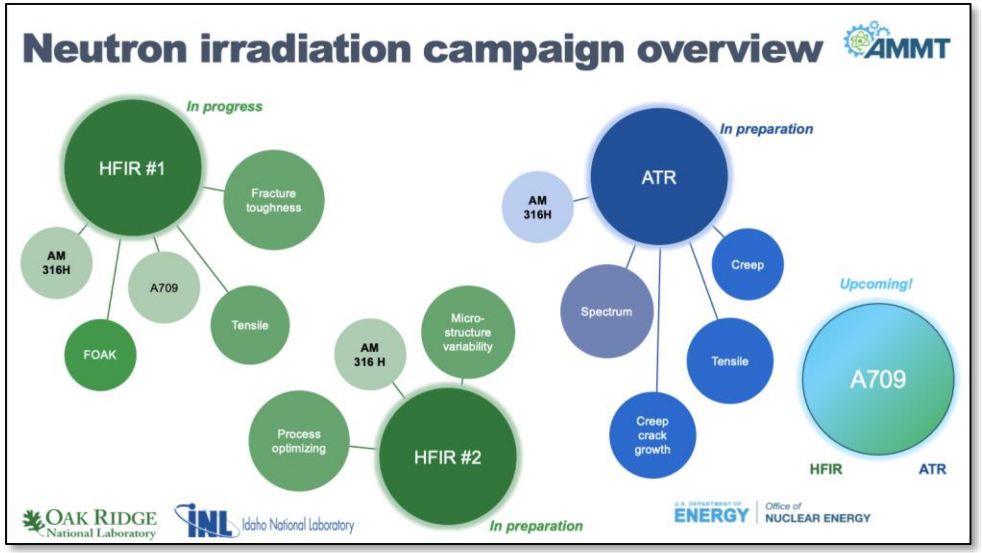
Insert into reactor
- Decide on locations/quantity
- Design test articles to minimize impact on component operation

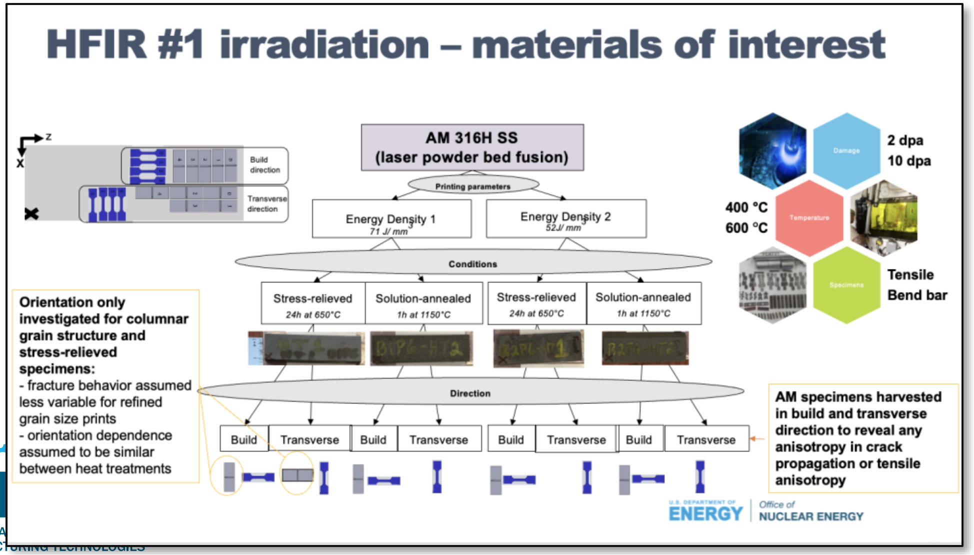
Periodically remove and test ex-situ
- How often to remove and how many?
- Robust article design
- Instrumented out of reactor testing – strain measurement

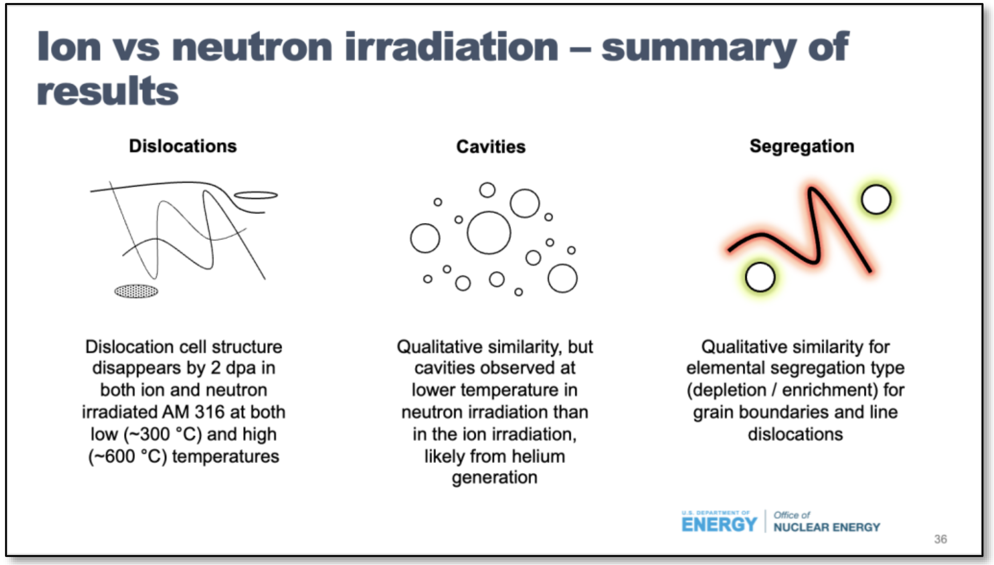
Acceptance criteria
- Damage inference based on out of reactor testing
- Acceptance criteria: what is the component remaining life?

Summary









ADVANCED MANUFACTURING TECHNOLOGIES

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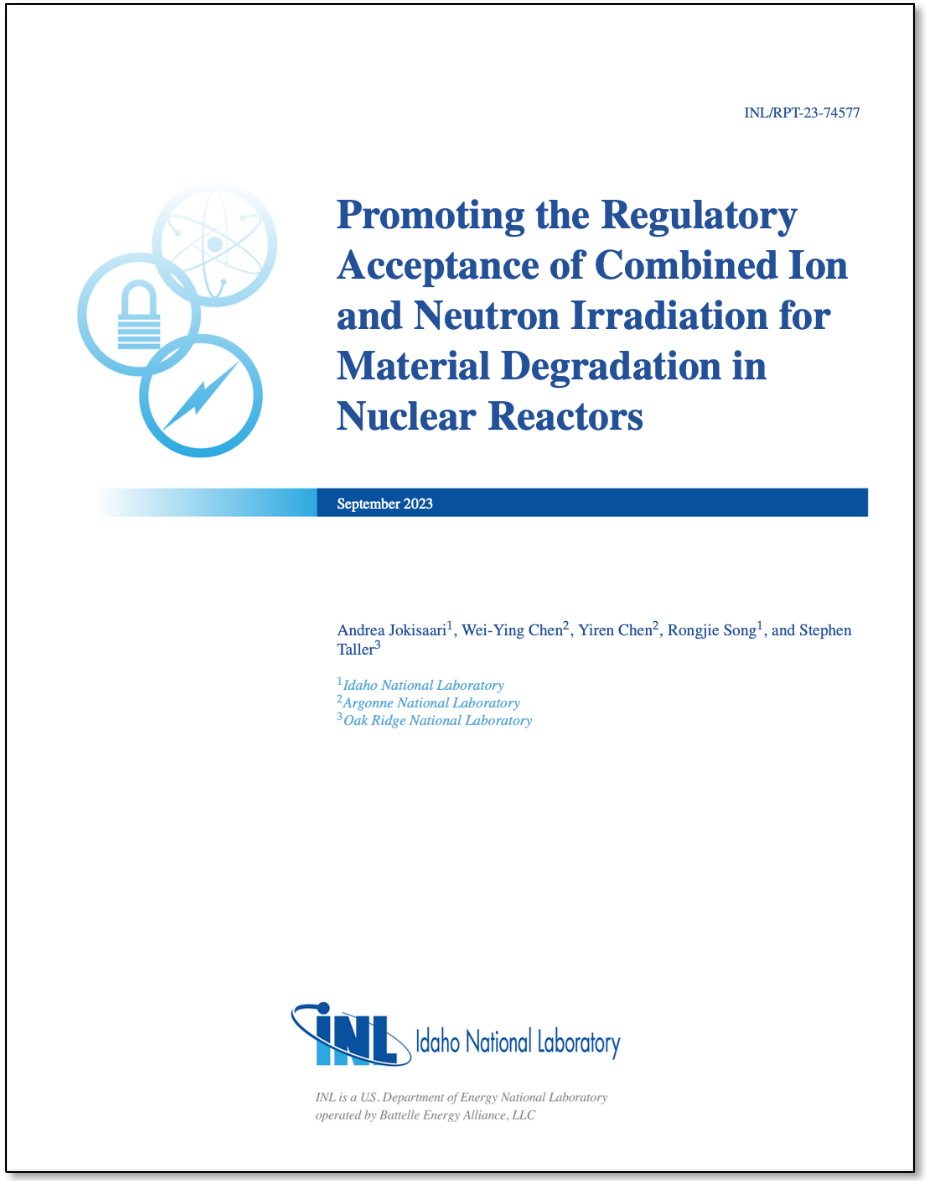
White paper for combined ion and neutron testing

- Provides background on the scientific basis for combined ion and neutron testing of structural materials
- Develops a recommendation for promoting the use of combined ion and neutron irradiation data for the accelerated qualification of nuclear reactor materials
- Describes a collaborative path forward for academia, industry, and national laboratories developed with input from the Office of Regulatory Research within the U.S. Nuclear Regulatory Commission
- Intended for a broad audience and to provide a technical, generalist-level overview on complex interplay of topics



INL/RPT-23-74577

Promoting the Regulatory Acceptance of Combined Ion and Neutron Irradiation for Material Degradation in Nuclear Reactors

September 2023

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