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Corrosion testing needs and considerations for additively manufactured materials in nuclear reactors

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Changing the World's Energy Future

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Corrosion testing needs and considerations for additively manufactured materials in nuclear reactors

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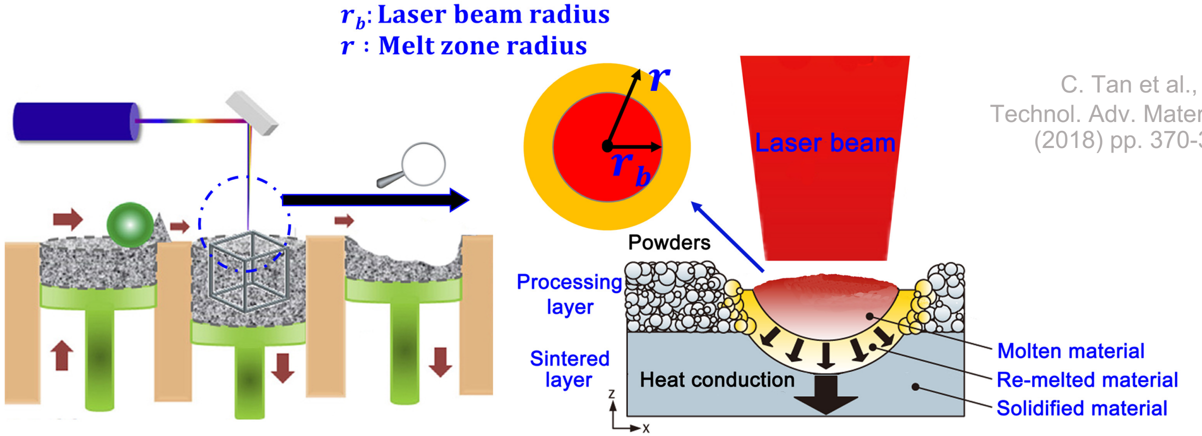
Advanced Materials and Manufacturing Technologies

IAEA Technical Meeting on Compatibility Between Coolants and Materials for Fusion Facilities and Advanced Fission Reactors, 30 October – 3 November 2023, Vienna, Austria

What is metal additive manufacturing?

- **Metal additive manufacturing is a means of building a component by adding material using energy input**
  - Layer-by-layer fabrication process
  - Net-shape or near net-shape
- **Common additive manufacturing techniques relevant for nuclear energy include:**
  - Laser powder bed fusion (LPBF)
  - Laser directed energy deposition (L-DED)
  - Electron beam (EB) welding
  - Powder metallurgy – hot isostatic pressing (PM-HIP)
  - Cold spray
- **Many processing parameters exist, including laser power, scan speed, material feed rate, hatch spacing…**

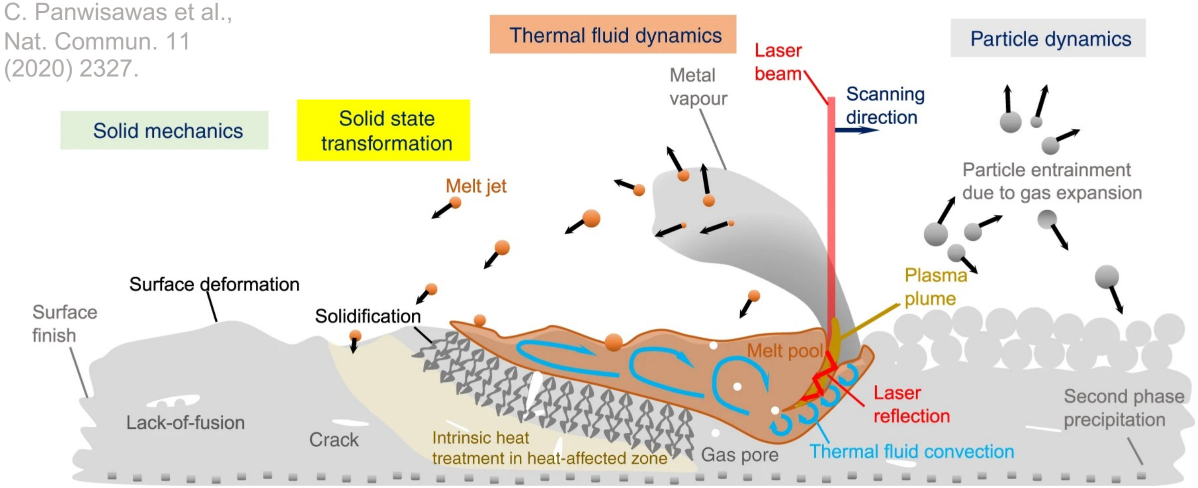
Laser powder bed fusion schematic



C. Tan et al., Sci. Technol. Adv. Mater. 19 (2018) pp. 370-380.

Phenomena and flaws in powder AM

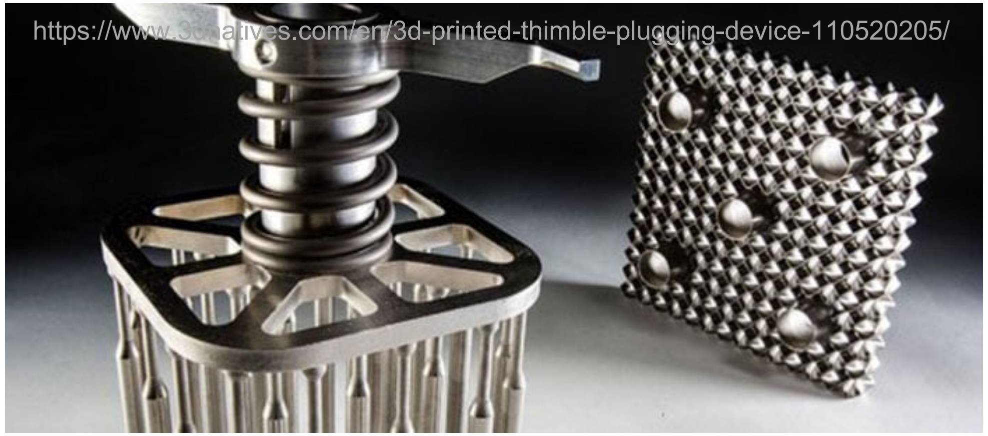
C. Panwisawas et al., Nat. Commun. 11 (2020) 2327.

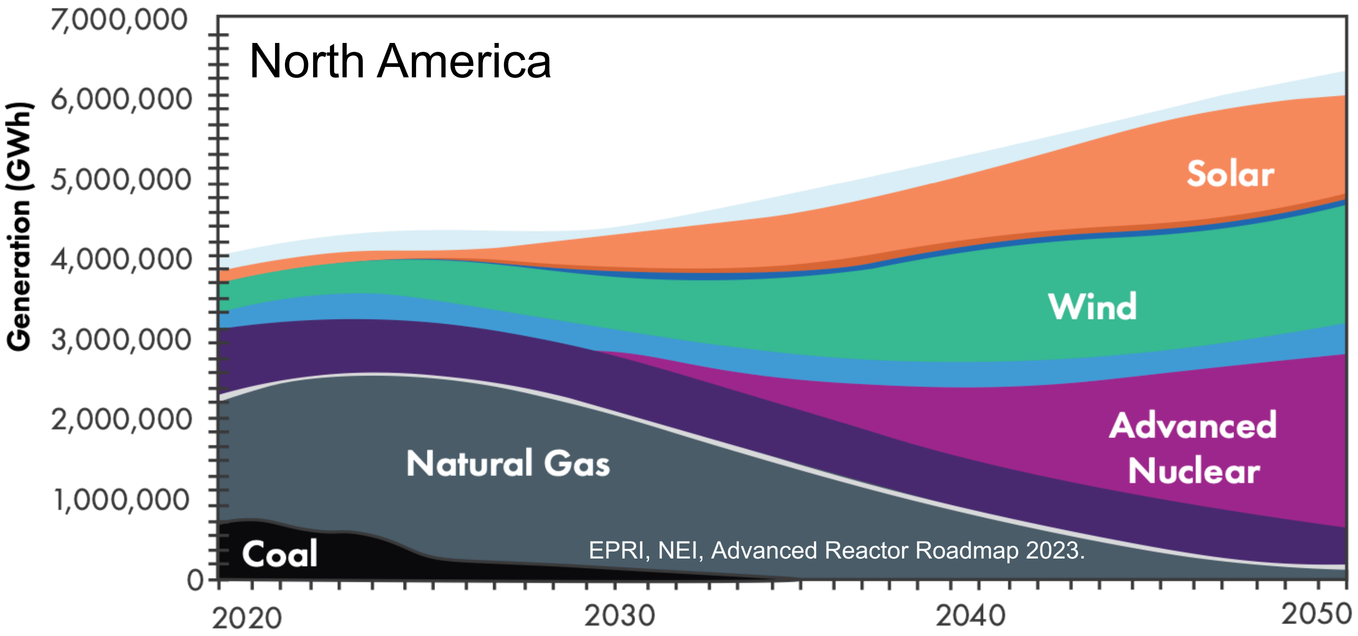


Additive manufacturing is already employed in the nuclear industry

- **Additive manufacturing has already been deployed in non-safety-critical nuclear energy applications**
  - Naval reactor door hinges
  - Fuel debris filters
  - Thimble plugging devices…
- **Nuclear energy deployment is projected to grow – how will all that capacity be built?**

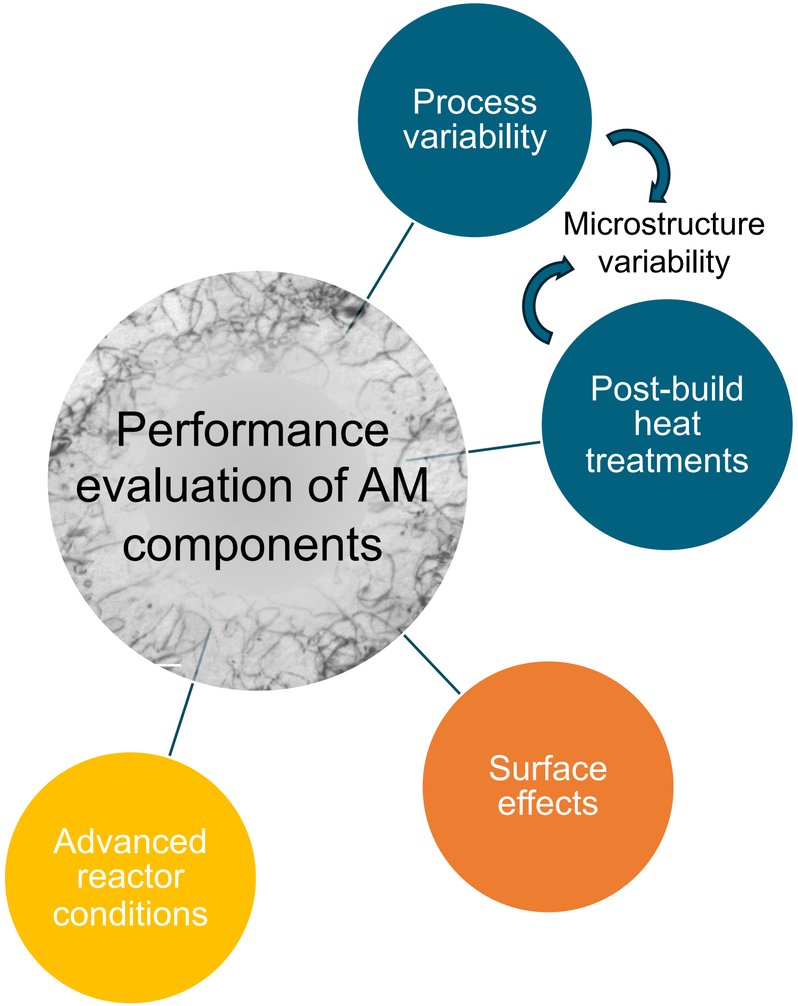
Additive manufacturing may be a critical technology for deployment of nuclear energy through 2050





What’s the big deal for environmental effects testing of additively manufactured materials?

- **Corrosion costs the US LWR fleet approximately $4B/year**
- **Material lifetime in harsh advanced reactor environments must be part of a material development and qualification program**
  - Irradiation, corrosion, and high-temperature loading conditions
  - Complex damage processes that are often coupled phenomena
  - Experiments can be time-consuming and costly
- **Additive manufacturing results in different microstructures and properties versus a wrought material of the same composition**
- **Evaluating corrosion performance of new materials is one of the most critical technical hurdles for their rapid adoption in nuclear energy systems**



Goal: Rapid and effective qualification of the effect of process variability on performance and degradation of AM materials

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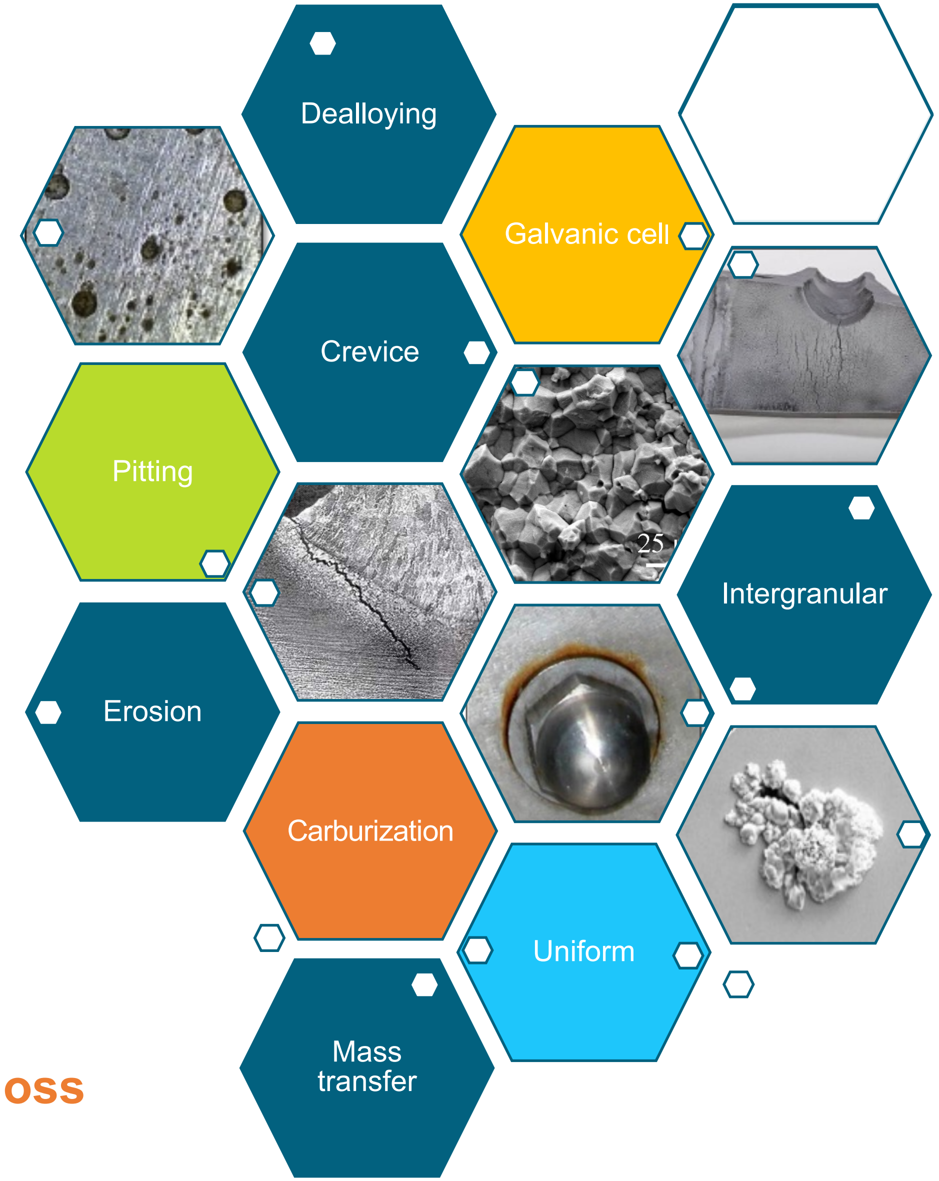
Corrosion presents multiple engineering concerns and comes in many forms

- **Corrosion can occur practically anywhere in the reactor**
  - Any type of containment vessel (RPV, molten salt fuel pins…)
  - Coolant system components
- **Corrosion can compromise structural integrity of components, limiting lifetimes**
  - Loss of load-bearing capacity
  - Embrittlement
  - Leakage
- **Corrosion can release corrosion products that deposit in other parts of the plant, resulting in loss of functionality**
- **Materials must be selected for their service properties AND their compatibility with environment and each other**

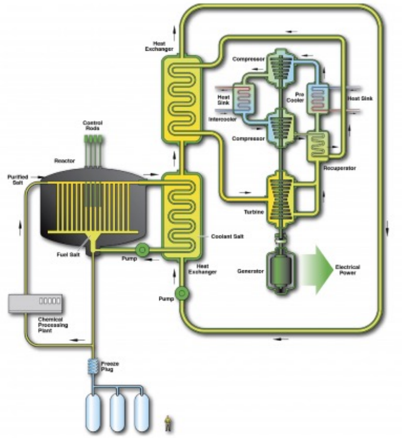


Radiation affects corrosion by changing the material microstructure, defect density, and environment chemistry

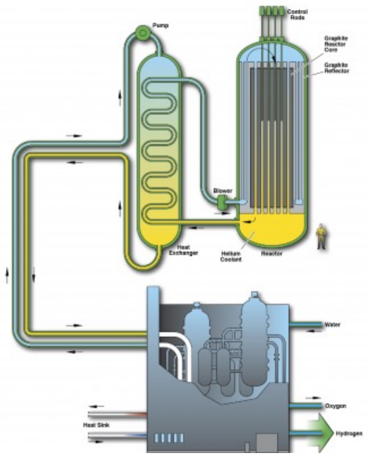
Corrosion of austenitic stainless steel is largely driven by the loss or damage of protective oxide

Reactor environments are varied, and corrosion issues are specific to reactor type

- **Molten salt reactors**
  - Highly corrosive fluoride or chloride salts, possibly mixed with actinides and other fission products
- **Liquid metal fast reactors**
  - Sodium fast reactors: dissolution and mass transfer, carburization/decarburization, Na impurities (C, O, N…)
  - Lead and lead-bismuth: corrosive coolant, liquid metal embrittlement
- **High temperature gas-cooled reactors**
  - Coolant gas impurities: H_2, CH_4, N_2, O_2, H_2O, CO_2/CO…
- **Water-based advanced reactors**
  - Water cooled reactors: aqueous corrosion, galvanic corrosion, intergranular corrosion, SCC
  - Supercritical water reactors: oxidation and dissolution from hydrothermal corrosion, SCC …



MSR

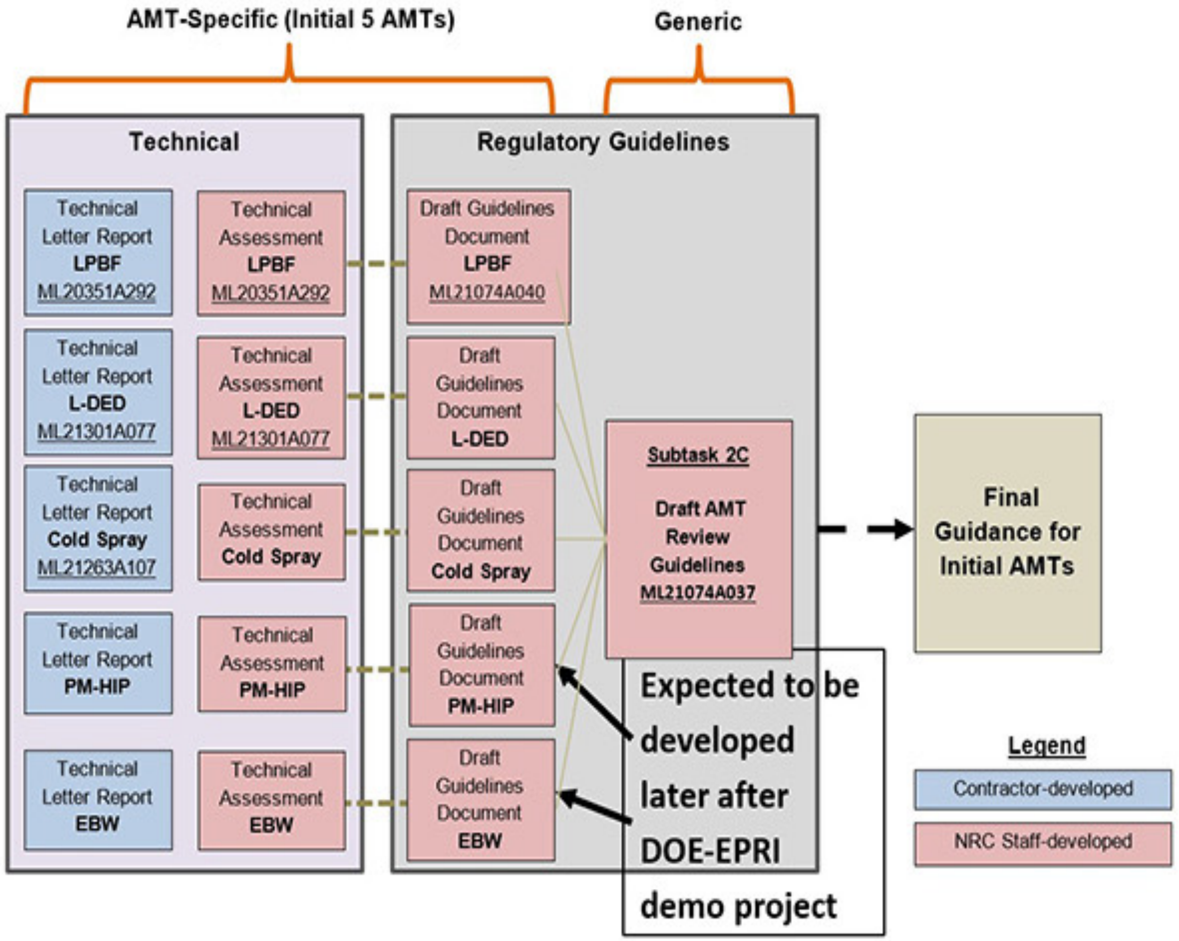


SFR

Reactor Type	Environment			Materials
	Coolant	Moderator	Temperature	
LWR	Water	Water	290 - 320 °C	SS, LAS, Ni alloys
MSR	Fluoride or chloride salt	Graphite, Zirconium hydride	600 - 750 °C	Ni alloys, SS
SFR	Sodium	-	500 - 550 °C	SS, F/M, Ni alloys
LFR	Lead, lead-bismuth	-	480 - 650 °C	F/M, ODS, SS, Ni alloys
HTGR	Helium	Graphite	750 - 950 °C	Graphite, SiC, Ni alloys, refractory metals

Corrosion is considered in a regulatory perspective

- **The U.S. Nuclear Regulatory Commission considers corrosion as a significant factor in ensuring the safe and reliable operation of nuclear power plants**
  - NRC's regulatory framework includes requirements to manage corrosion issues in reactors
  - Applicant must demonstrate that a material and environment will meet integrity criteria
  - May set corrosion allowances in terms of absolute loss of thickness and acceptable corrosion rates
  - Monitoring also required in accordance with ASME Section XI and other applicable corrosion standards
- **NRC also has an Advanced Manufacturing Technologies Action Plan**
- **ASME Section XI focuses on surveillance of corrosion in a nuclear plant**
  - Largely focused on light water reactors
  - The applicability of Section XI standards for advanced reactors must be assessed

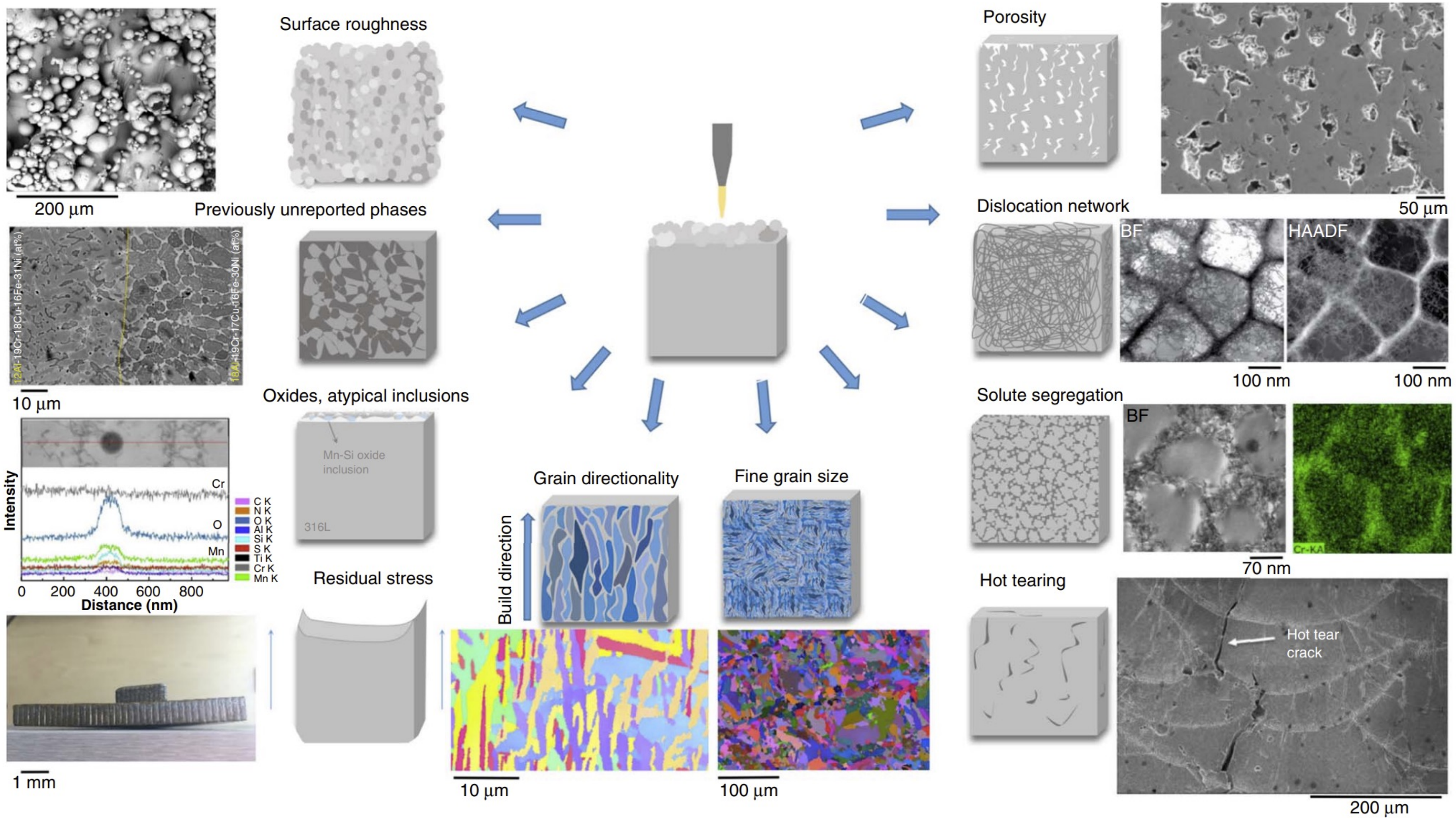


Corrosion testing should be considered in the context of codes and standards

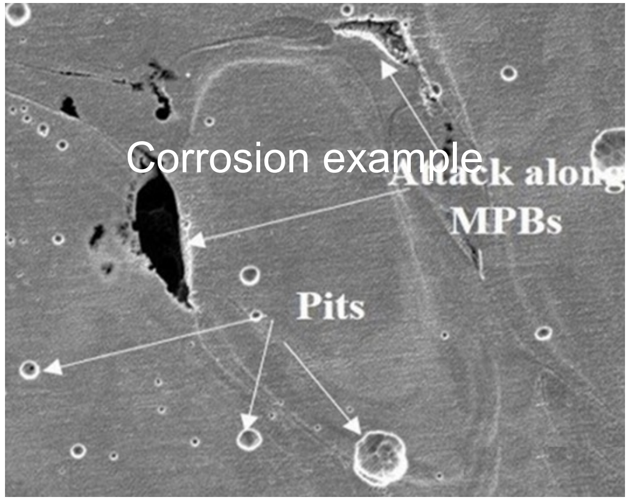
- **Laboratory testing of corrosion behaviors should be informed by regulatory considerations and applicable testing standards**
- **ASTM has extensive standards for corrosion testing**
  - Search for applicable ASTM standards before beginning corrosion testing
  - Not all corrosion tests that could be envisioned for advanced reactor material-environment systems have ASTM standards
  - No ASTM standard exists that is specific for testing additively manufactured materials
  - If a relevant standard does not exist, the program should develop an internal standardized methodology for testing and consider promoting a new ASTM standard

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"

AM-fabricated material has features that can affect corrosion versus wrought material



Potential impact of AM processing (without post treatment) on microstructure and corrosion behavior



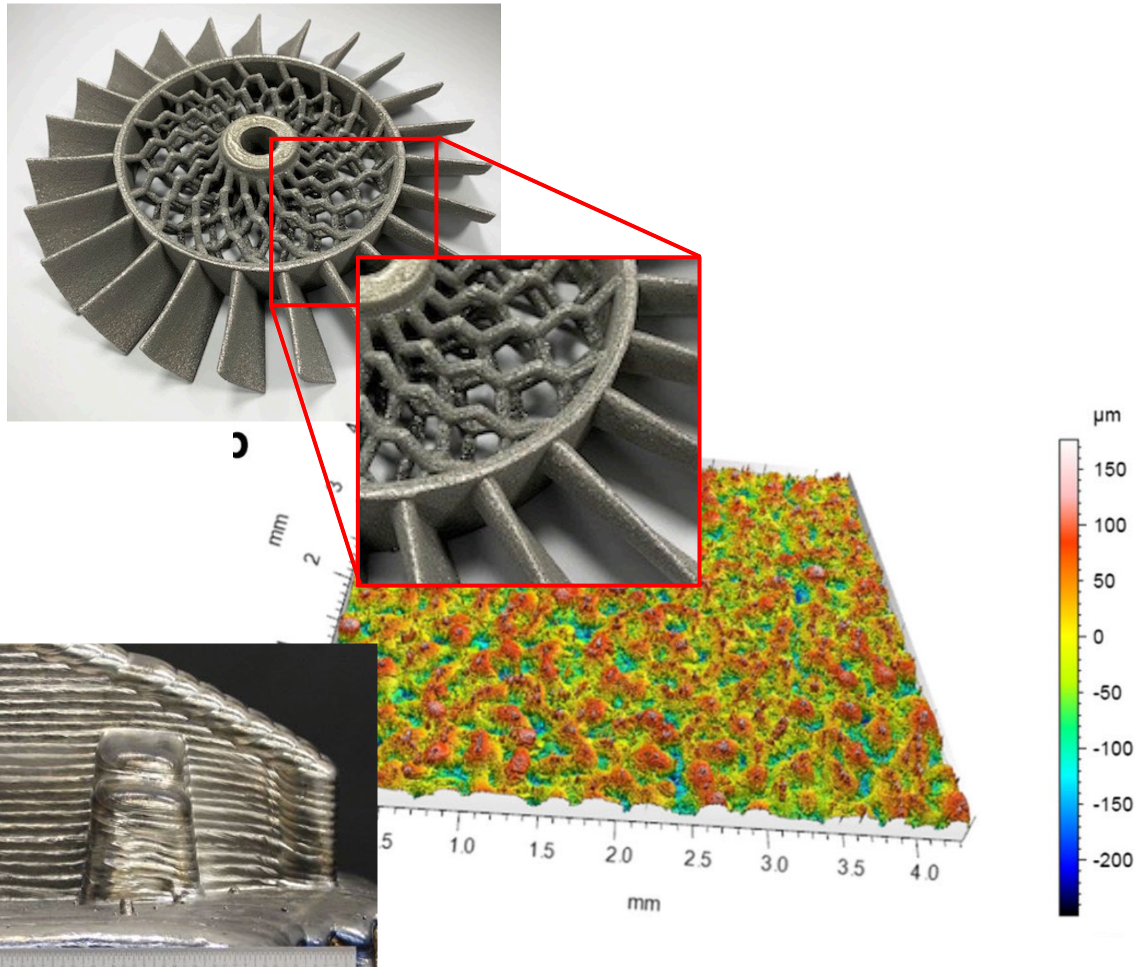
MPB: molten pool boundaries

- **Can affect:**
  - Uniform corrosion
  - Pitting
  - Crevice
  - Electrochemical properties
  - Corrosion fatigue

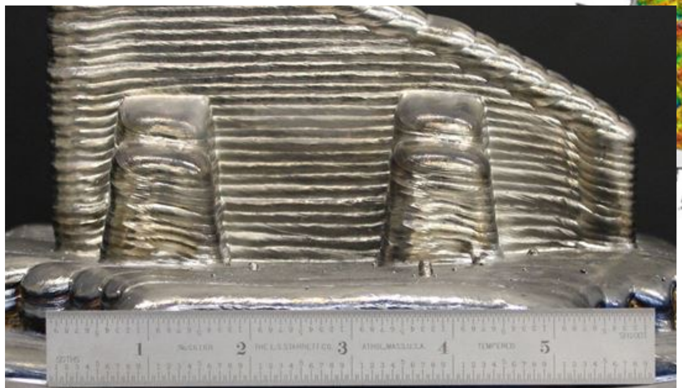
G. Sander, et al. Corrosion 74(2018)1318. // D. Kong, et al. npj Materials Degradation 24(2019)1.

Surface properties must be considered for AM components in nuclear reactor applications

- **Components may be deployed without additional surface finishing**
  - Surface roughness may improve corrosion behavior ***or*** degrade crevice corrosion properties
- **Build porosity, oxides, atypical inclusions can also intersect surface and affect corrosion**
- **Residual stresses can directly affect electrochemical potential**
- **Testing may include:**
  - Macroscale weight change measurements
  - Microscale chemical evolution



K. Jahns, et al., Inter. J. Adv. Manuf. Tech. (2020) 107:2151–2161



https://www.additivemanufacturing.media/articles/additive-manufacturing-with-wire%282%29

Many more corrosion studies of AM materials in reactor coolants are needed

- **Corrosion studies to date focused on non-nuclear water (saltwater) and LWR environments**
- **Most corrosion studies of AM materials focus on the effect of microstructure on milled and polished coupons**
  - Very little work on the effect of surface roughness or build orientation on corrosion behavior
- **Inconclusive results regarding corrosion resistance of AM stainless steels versus wrought material**
  - Some studies show improved behavior for AM material, hypothesized to result from improved chromium oxide film thickness arising from the high density of cellular dislocation walls with chemical segregation
  - This mechanism would be subject to process parameters that impact dislocation cell wall formation and chemical segregation
- **Increasing porosity and pore size may:**
  - Increase stress corrosion cracking growth rate
  - Decrease the breakdown voltage of stainless steels
- **The presence of other phases may alter the corrosion of AM 316, mechanisms still need to be determined**
  - δ-ferrite/γ-austenite phase boundaries affects IASCC, including crack propagation along phase boundaries
  - Pitting and uniform corrosion rates influenced by anisotropy of δ-ferrite distribution and size of δ-ferrite grains, presence of σ phase
  - Heat treatment generally improves corrosion behavior, likely due to the dissolution of deleterious phases

Corrosion Science 128 (2017) 140–153

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journal homepage: www.elsevier.com/locate/corsci

On the stress corrosion crack growth behaviour in high temperature water of 316L stainless steel made by laser powder bed fusion additive manufacturing

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ENVIRONMENTA

Pitting Corrosion in 316L Stainless Steel Fabricated by Laser Powder Bed Fusion Additive Manufacturing: A Review and Perspective

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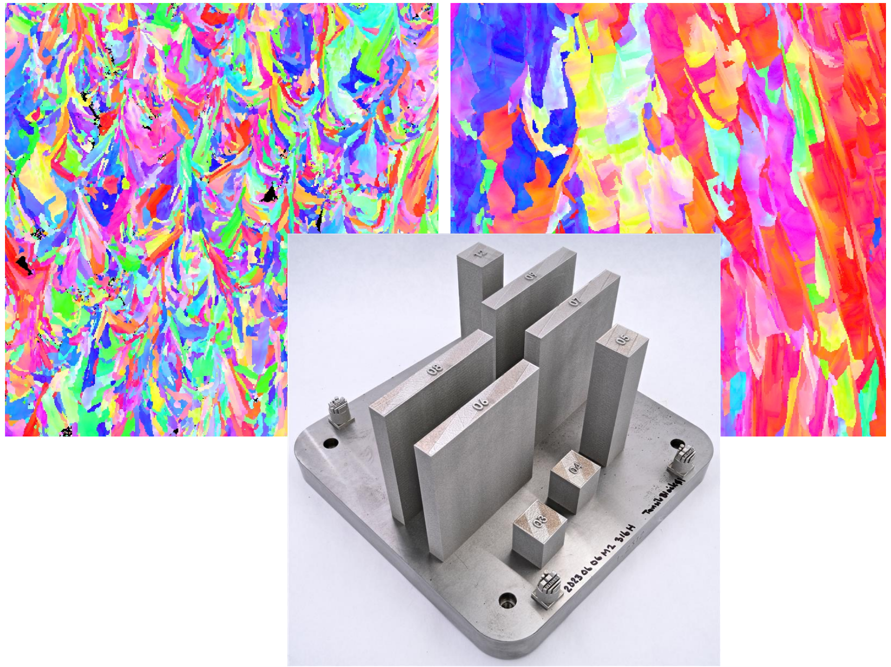
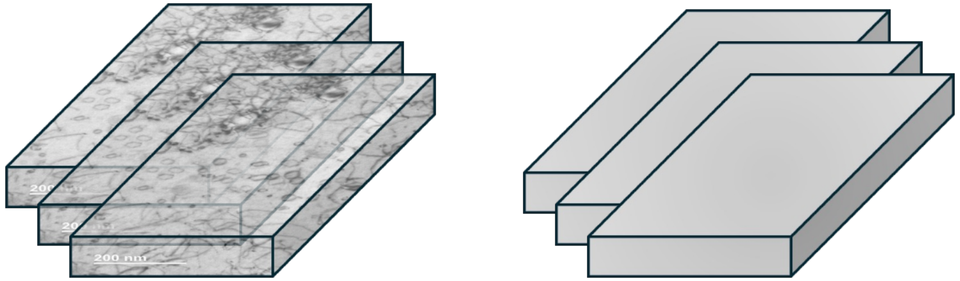
Build process variability and specimen geometry must be considered

- **Build process variability is inherent to AM materials**
  - Variations in as-built microstructure from a single specific machine due to geometry, built plate heating, atmosphere and humidity, and machine-to-machine variability
  - Process variables such laser power, scan pattern, scan speed, post-build treatment
  - Feedstock lots and feedstock storage/handling
- **Address process variability with experiments and data analytics: link process variability sources with microstructure statistics and corrosion test results**
- **Microscale: understand statistical distribution of a corrosion behavior with respect to key microstructural features**
- **Macroscale: take samples from different locations within a component**
- **The geometry of the specimen from which corrosion samples are removed should be considered as part of process variability**
  - Small specimens with limited build volume vs large components with different thermal history
  - May need corrosion specimens sectioned from specimens of representative geometries and volumes of the actual component

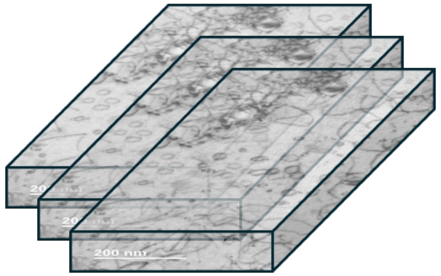




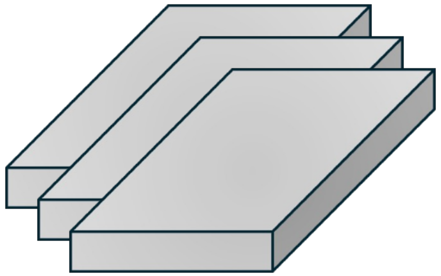
A "two-surface" test can provide a more complete understanding

- **A "two-surface" test with as-printed and machined coupons can help determine macroscopic and microscopic behaviors and determine mechanisms**
  - Smooth surface specimens can assess underlying corrosion mechanisms and how corrosion is localized to or affected by the rough surface, while as-printed surfaces provide the engineering baseline
  - Wrought specimens can be added to further determine effect of AM build process and microstructure (especially if aiming to replace conventional material)
- **The two-surface test strategy is applicable to a variety of tests (static corrosion, flow, stress corrosion cracking…)**
- **3D non-destructive examination before and after corrosion testing can evaluate AM build porosity impact**
  - X-ray computed tomography
  - Comparative characterization of pre- and post-corrosion porosity at the surface and within the same sample
  - Section specimens to study microstructure and local composition variation with SEM, TEM...

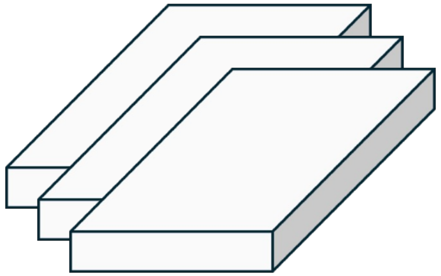
As-printed surface



Machined surface

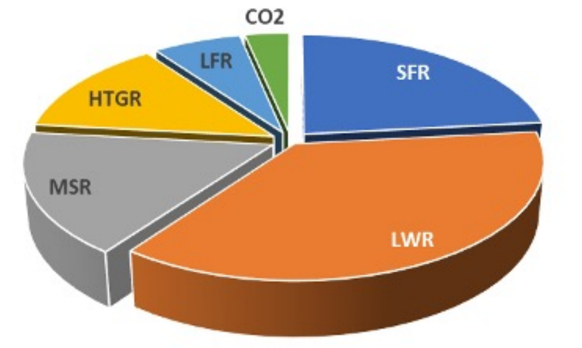


Wrought specimen



Feasible experimental campaigns must consider available facilities

- **Types of materials:**
  - Unirradiated material in laboratory environment
  - Irradiated material in PIE corrosion test (unirradiated environment) to determine effect of radiation-induced microstructure on corrosion
  - *In situ* or in-reactor irradiation testing to provide prototypical results and to probe synergistic effects between the radiation environment and the material
  - May test ion-irradiated material as a means of accelerated investigation on the impact of radiation-induced microstructure evolution on corrosion behavior, but ion irradiation results will necessarily be focused on understanding corrosion mechanisms due to the small sample volumes achievable and differences in ion and neutron irradiation on material degradation
- **Types of tests:**
  - Immersion tests (static capsule, flow loop)
  - Electrochemical tests
  - Stress-affected corrosion tests



Corrosion system		Testing capability				
		With mechanical stress			With *in-situ* irradiation	
Material	Reactor environment	No load	Static load	Dynamic load	Ions	Gamma/neutrons
Unirradiated materials	Water	++++	+++	+++	+	
	Molten salt	++				+
	Sodium	+++				
	Helium	+	+			
Irradiated materials (radioactive)	Water	+++	++	++	+	
	Molten salt	+				+
	Sodium					
	Helium		+			

An AM material corrosion test strategy for nuclear applications must be focused

Determine AM material system

- Does material have a conventional analogue?

Determine environmental system

- What coolant?
- What temperature?
- What other materials (in reactor, in test system) are involved?

Determine AM build process

- Does the material need post-build treatment?
- Is process optimization needed?

Determine goal of AM corrosion study

- Does scientific understanding need to be developed?
- Is an engineering or licensing data set being created?

Determine component surface

- Will the component retain as-built surface?
- Will the component be machined?

Determine component geometry and microstructure statistics

- Is the geometry simple or complex?
- What is the distribution of the microstructure statistics?

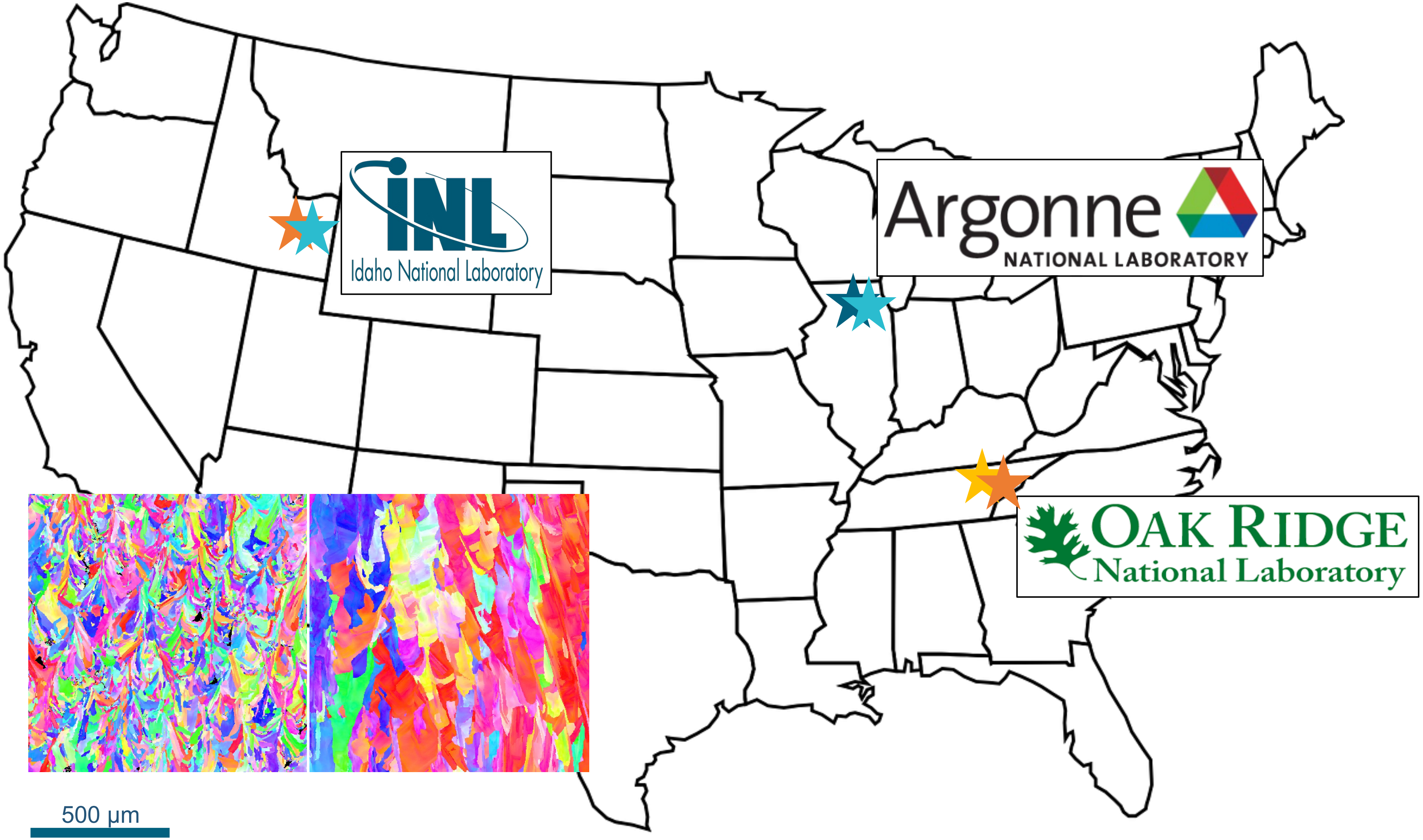
Determine applicable testing standards

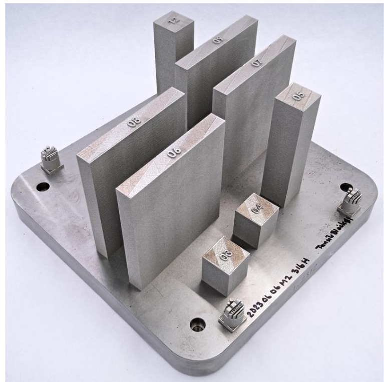
- Do existing standards apply?
- Does a standardized method need to be developed?

This "decision-making process" must be centered about the characteristics of AM materials: unique surface finishing, compositional and crystallographic inhomogeneities, and residual stress

The AMMT solution: an integrated environmental effects testing strategy for AM 316H

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

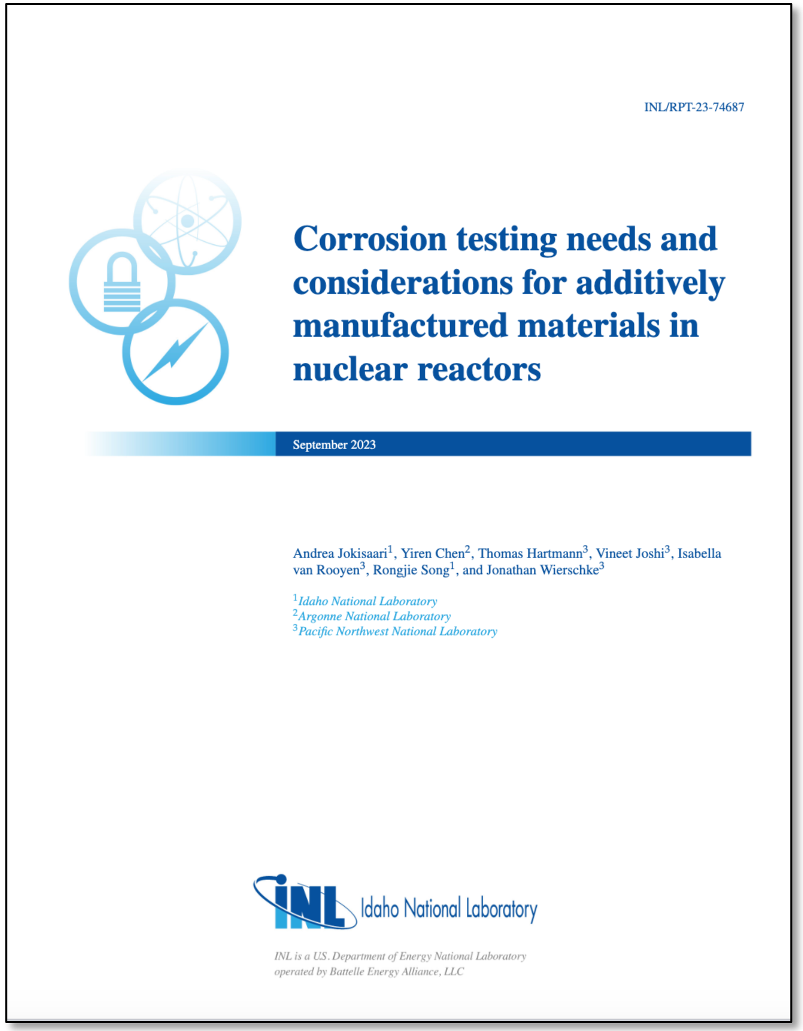




500 µm

Suggested path forward for corrosion testing of AM materials for advanced nuclear reactors

- **Clear identification of corrosion system (material-environment combination) and synergistic degradation mechanisms**
- **Start with an experimental program: modeling and simulation of corrosion is a particularly large challenge**
- **Staged approach:**
  - Unirradiated material in radiation-free environment
  - Irradiated material in unirradiated environment
  - Prototypical tests in-reactor
- **Post-build processing of AM material usually occurs, so behavior should be assessed for each instantiation of the AM-built and postprocessed material**
  - AM corrosion behavior should be assessed from a statistical standpoint to quantify build and process variability
- **Two-surface test can be used to assess the impact of surface roughness versus microstructure on corrosion**
- **Corrosion testing should assess phenomena of engineering interest and mesoscale mechanisms**
- **The specific corrosion testing strategy employed to assess an AM material for a reactor environment depends upon the amount of prior knowledge about the behavior of the material composition in the environment**
  - Targeted tests if mechanisms are generally known for conventional material of the same composition
  - Rapid assessment campaign of static coupon tests if a material-environment system are not well understood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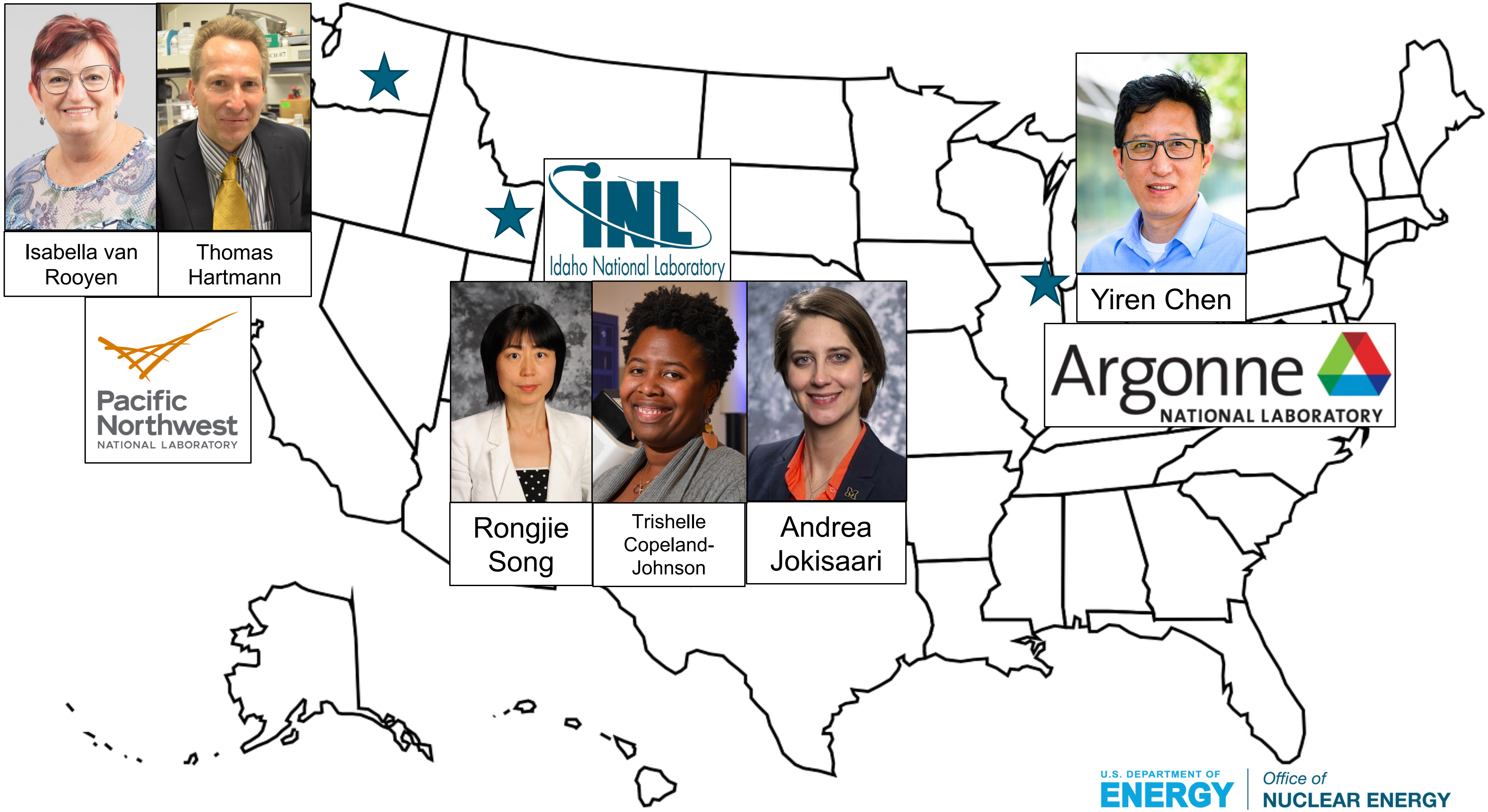

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