INL/CON-24-81581-Revision-0

Advanced Manufacturing for Harsh Environments at Idaho National Laboratory

October 2024

Changing the World's Energy Future

Adrian R Wagner, Jorgen Fredrick Rufner, Andrea M Jokisaari, Michael D McMurtrey

Idaho National Laboratory

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Advanced Manufacturing for Harsh Environments at Idaho National Laboratory

(ICAM2024)

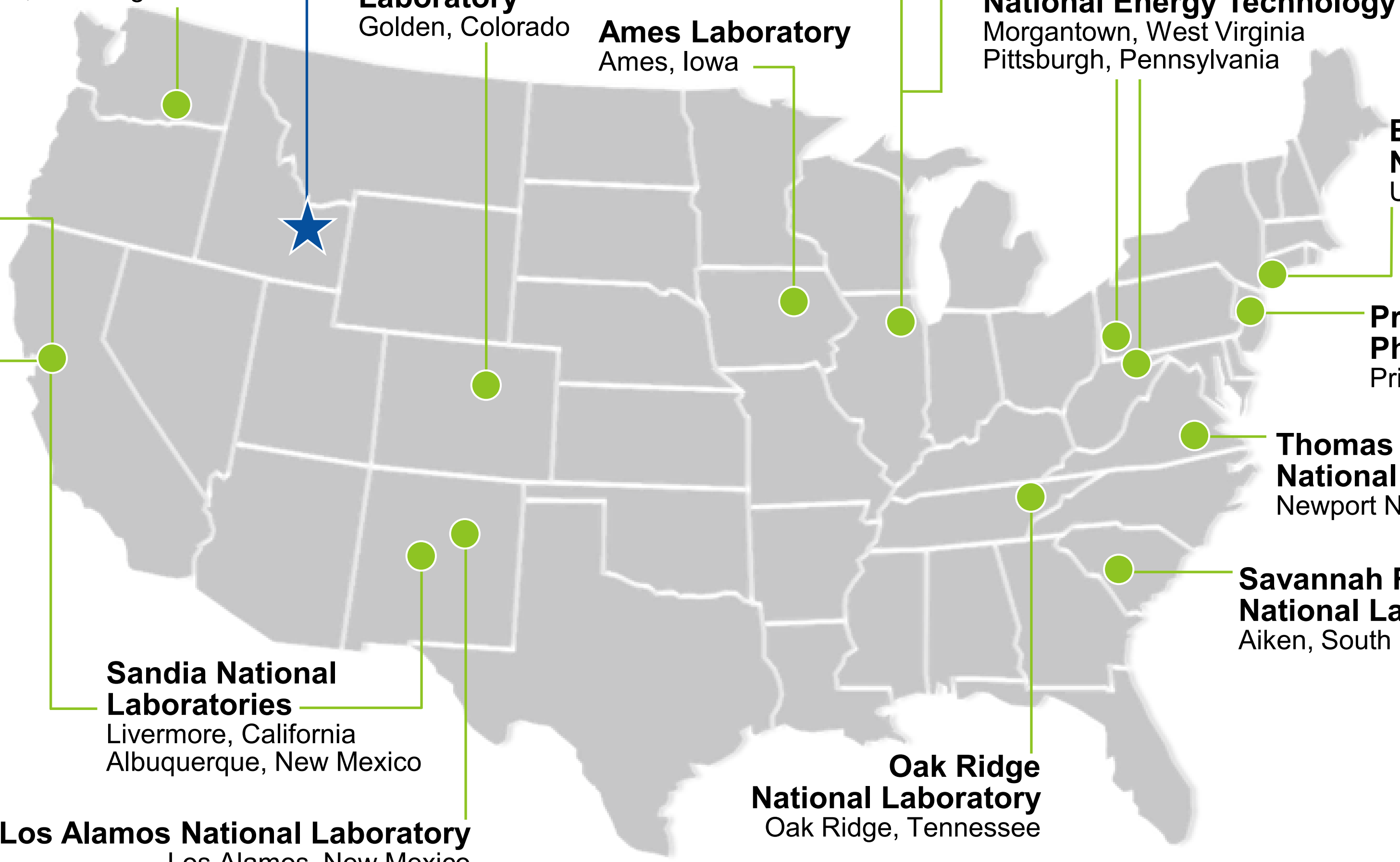
INL/CON-24-81581

Idaho National Laboratory

Agenda

- INL Overview
- Additive Manufacturing
  - Structural materials
  - Uranium bearing
- Electric Field Assisted Sintering
  - Non-nuclear materials
  - Uranium bearing

INL's Position Nationally



Unique INL site, infrastructure, and facilities enable energy and security RD&D at scale

6,000+ Employees
569,178 Acres
890 Square Miles

4 Operating reactors

12 Hazard Category II & III non-reactor facilities/ activities

50 Radiological facilities/activities

17.5 Miles railroad for shipping nuclear fuel

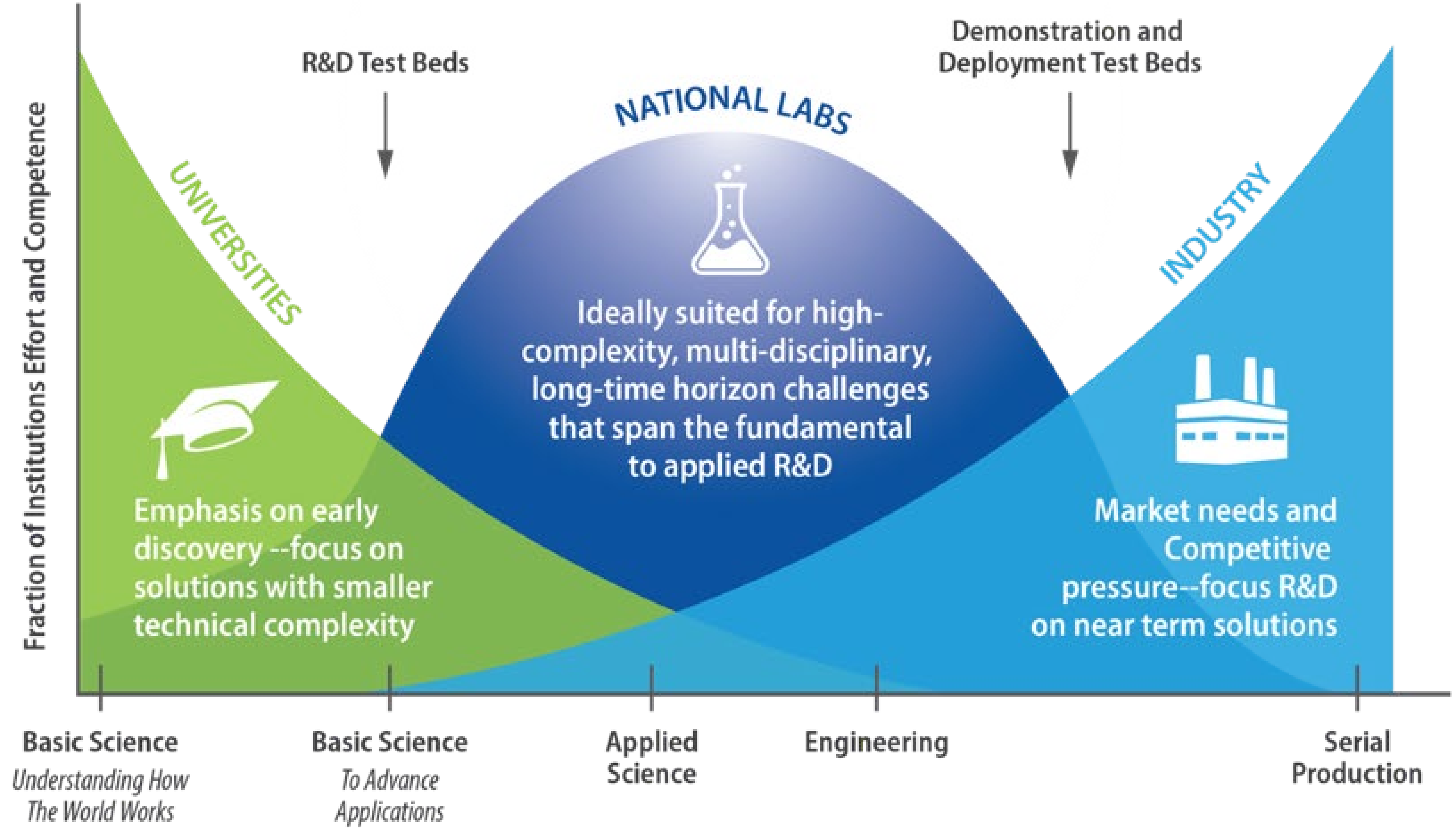
44 Miles primary roads (125 miles total)

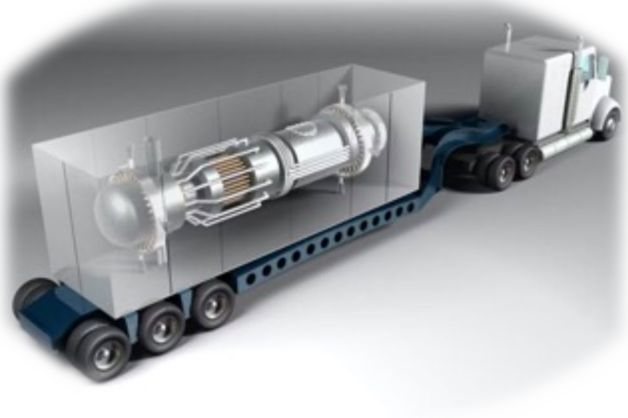
9 Substations with interfaces to two power providers

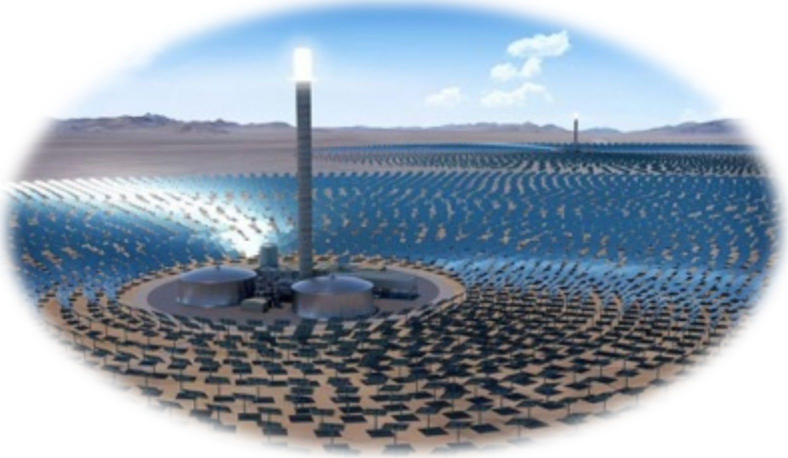
126 Miles high-voltage transmission lines

3 Fire Stations

DOE labs support the entire technology lifecycle and bridge academia and industry







High Performance Materials for Harsh Conditions

High strain rates

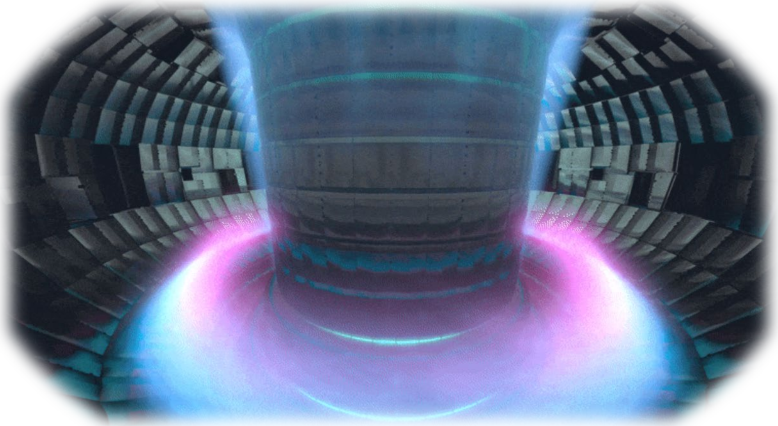
Corrosion / Ablation

Radiation

Pressure

Temperature / Enthalpy

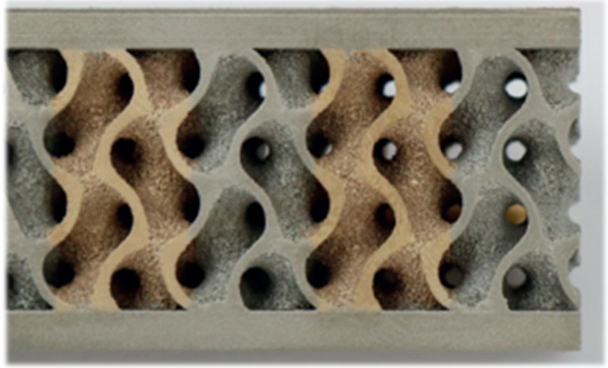
Reactions / Energetics



Additive Manufacturing overview

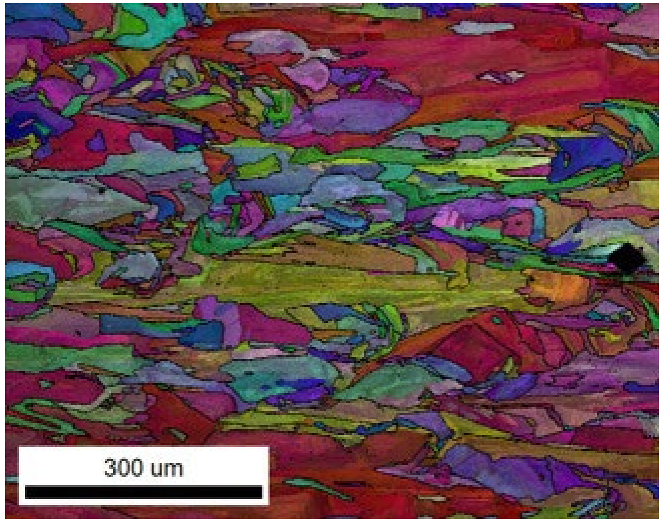
- Current focus on development of process capabilities and material optimization
- Environmental testing
  - Corrosion behavior
    - Molten salts: fluorides and chlorides
  - Mechanical testing
- Novel fuel forms
  - Uranium bearing concepts
  - Unique fuel and cladding geometries

- Materials
  - Haynes 282
  - 316H
  - Alloy 625
  - Zircaloy
  - Uranium alloys



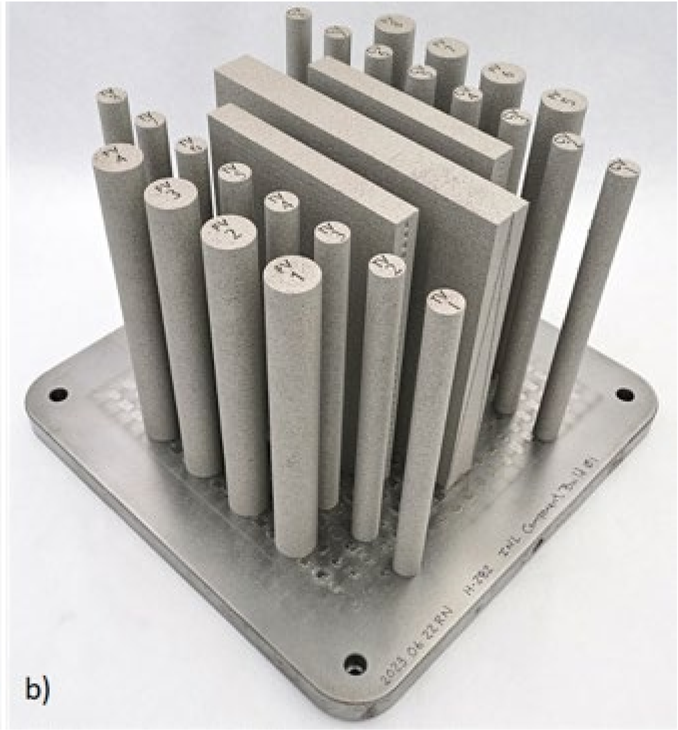
Structural materials testing

- Material testing is underway on various alloys
  - Samples fabricated by various organizations
- 316H made by LPBF, to qualify the material for high temperature nuclear
- Haynes 282 samples have been made by DED and LPBF
- Analyze and compare techniques
  - DED samples made by INL
  - LPBF samples made ORNL and EOS



Haynes 282 - LPBF

Idaho National Laboratory
laser-based DED

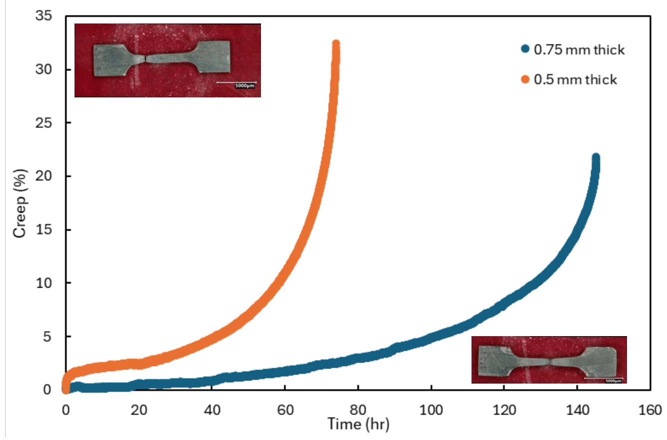


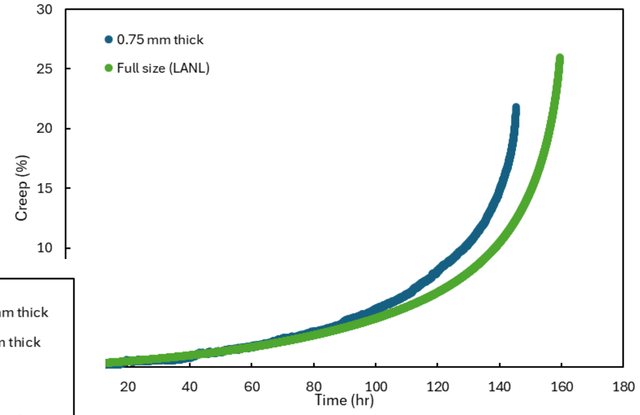
Oak Ridge National
Laboratory - LPBF

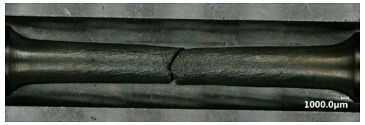
LPBF 316H Code Case Testing

- Nuclear structural materials often refer to the ASME Boiler and Pressure Vessel Code for material qualification
- INL, in partnership with ANL, ORNL, and LANL, are generating tensile, fatigue, creep, and creep-fatigue data to support qualification of the manufacturing process.
- Explored the use of sub-sized specimens for creep testing

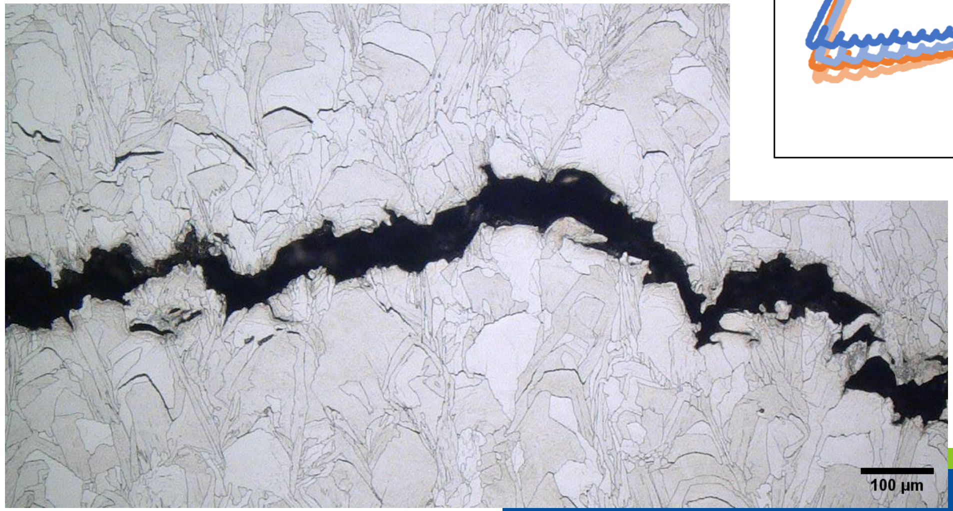
600 °C, 248 MPa
creep testing

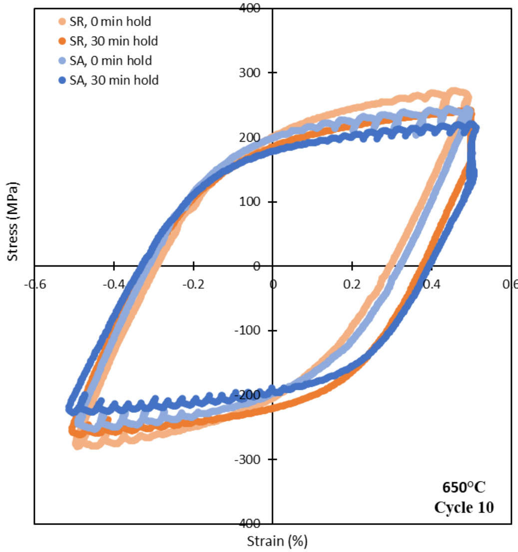






650 °C, 1% Δε
creep-fatigue testing



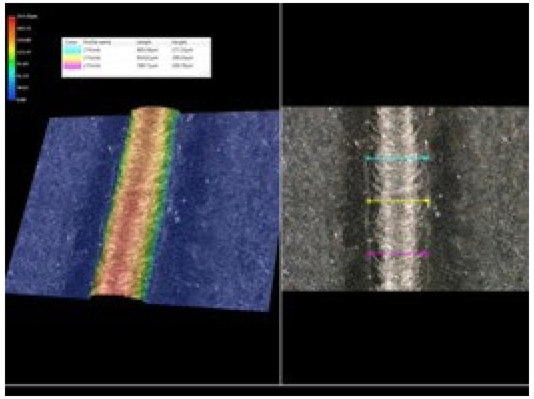


IDAHO NATIONAL LABORATORY

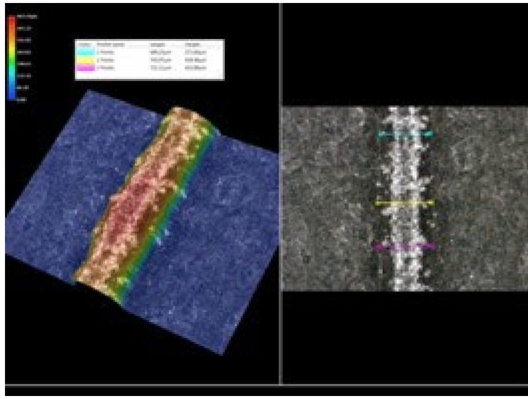
Haynes 282 - DED

- Single track depositions were made with various process parameters
- Defect density/porosity was analyzed on bulk specimens
- Mechanical testing samples were made from right cylinders

Ideal parameter sets that show uniform width and height.



Poor parameter sets have non-uniform height with partially melted powder stuck to the outer surface.



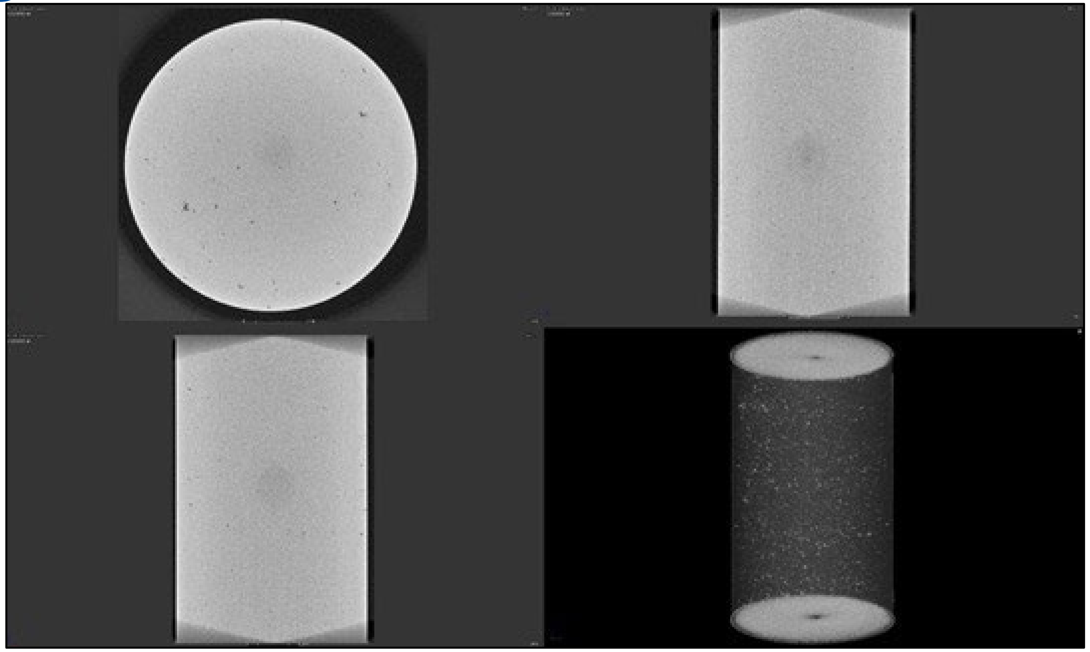
Total porosity: 0.03%
Average size: 3.22E-4 mm

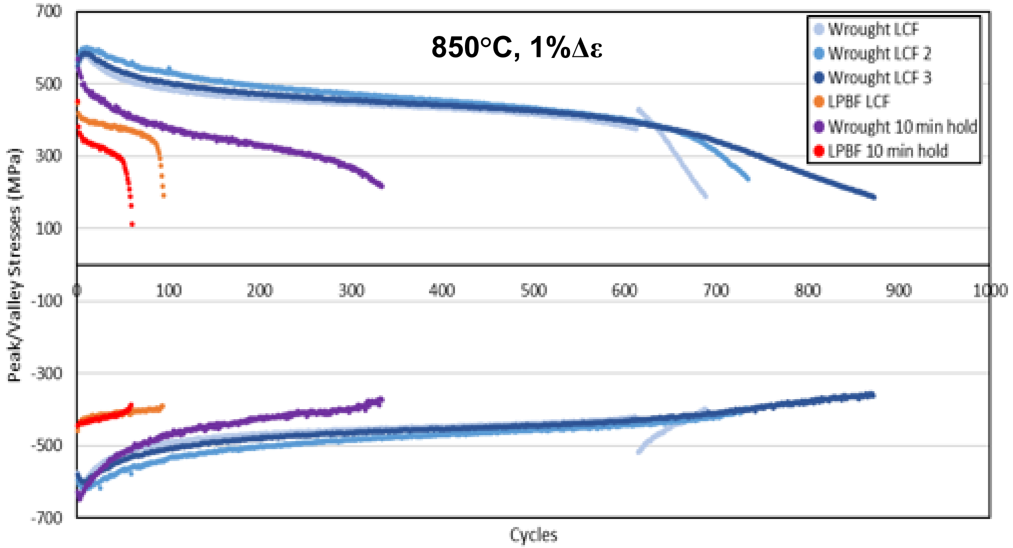
Laser Power: 300 W
Deposition rate: 9.3 mm/sec
Powder flow rate: 9.5 g/min
Hatch distance: 380 um
Layer Height: 127 um
Hatch angle: 67°

As deposited HY282 creep specimens.

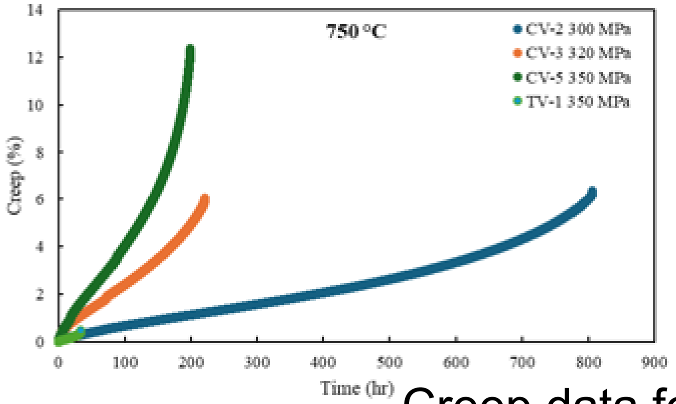
LPBF H282 mechanical testing

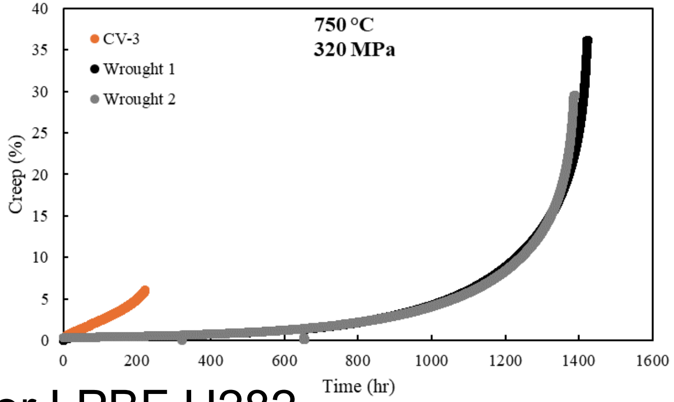
- Testing high porosity LPBF H282
  - Creep, and cyclic testing
  - Decrease in mechanical properties





Cyclic data for LPBF H282

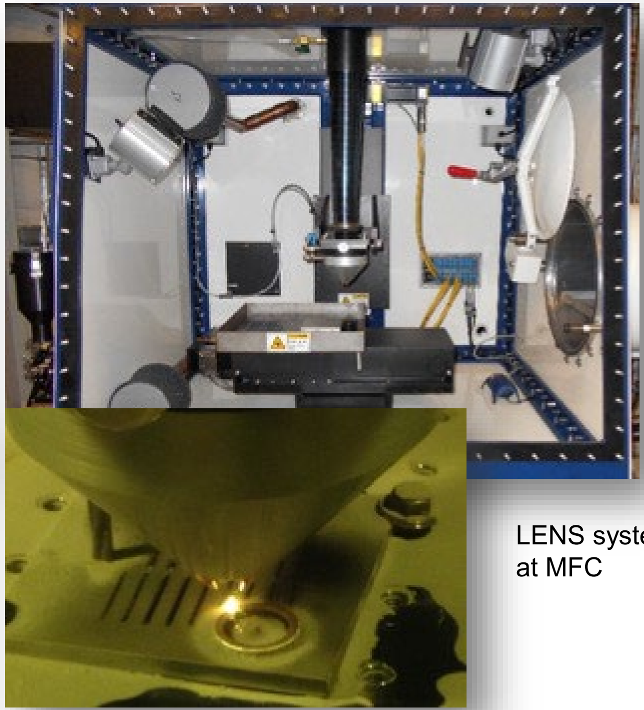




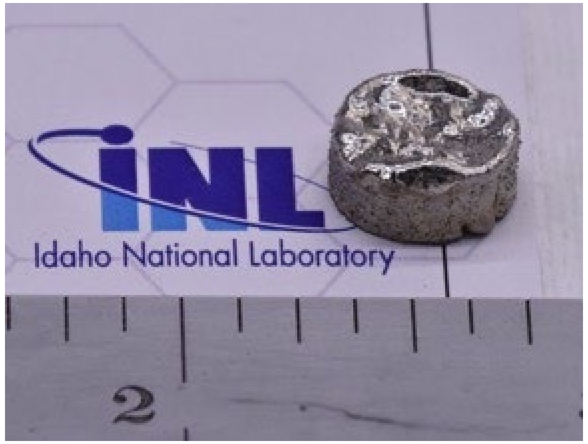
Creep data for LPBF H282

Additive Manufacturing: Uranium bearing

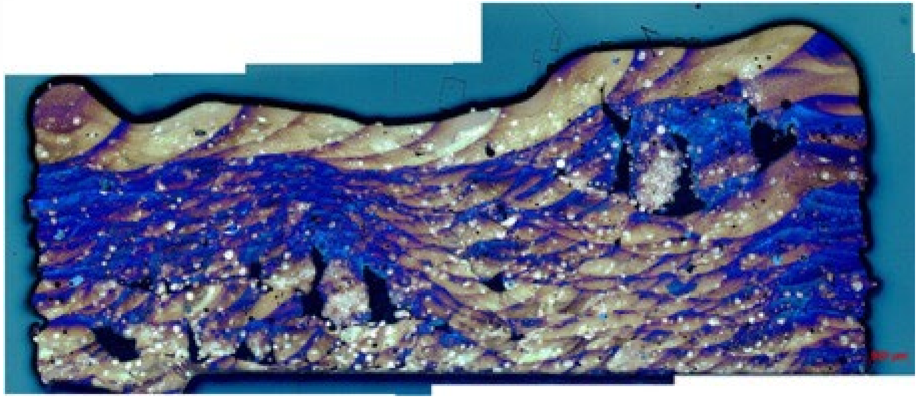
- Additive manufacturing techniques being pursued at MFC
  - Directed Energy Deposition—DED technique
  - Digital Light Processing—DLP

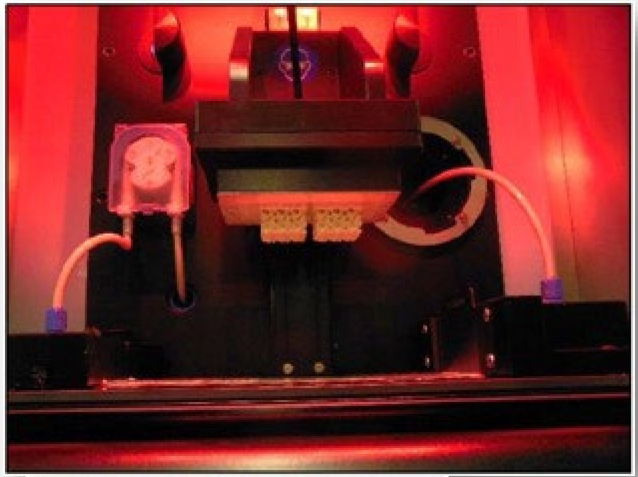


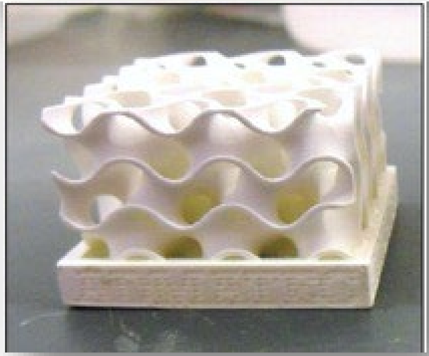
LENS system
at MFC



U-10Zr pellet fabricated
by LENS technique



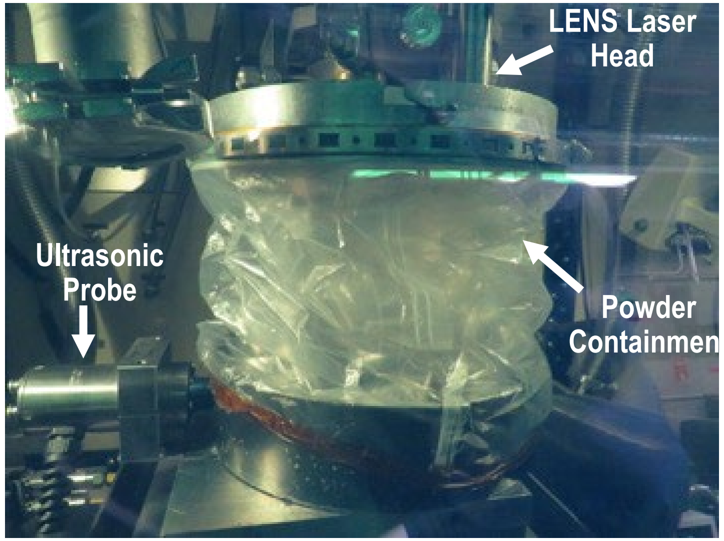


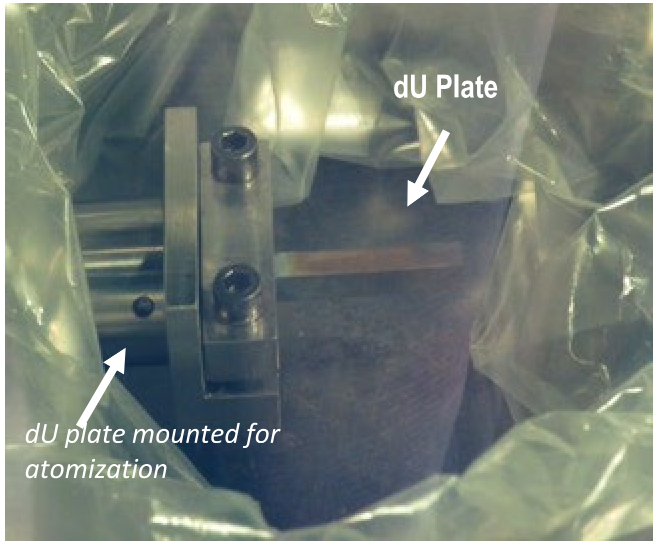


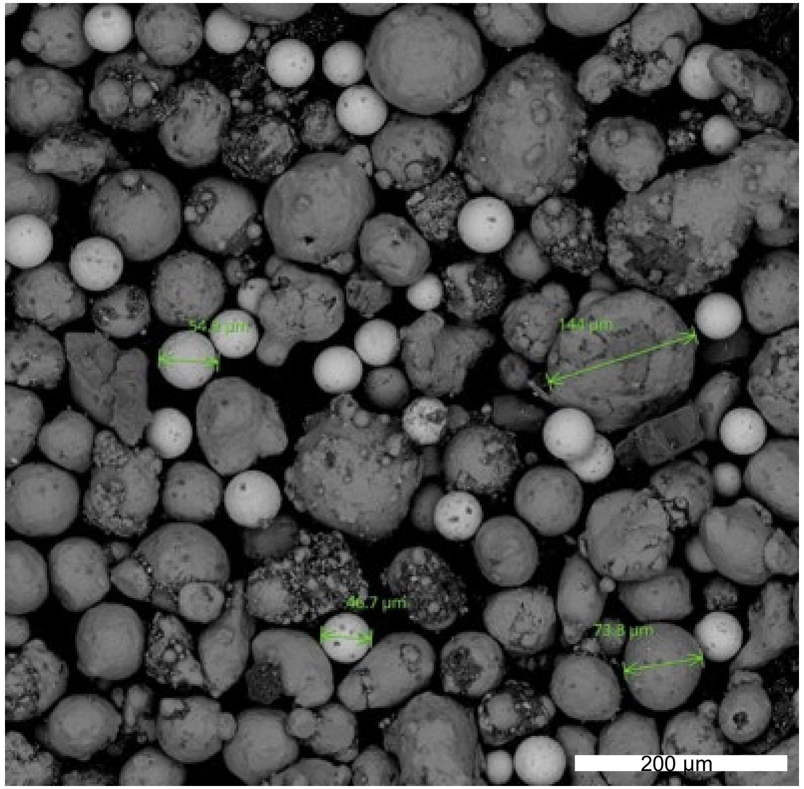
Admatech 130
alumina lattice

Atomization Capabilities: Ultrasonic Atomizer

- Atomized uranium feedstock development
  - Cannot procure spherical U powder
  - Use existing DED system to fabricate U powder
  - Further development is needed, but initial results are promising



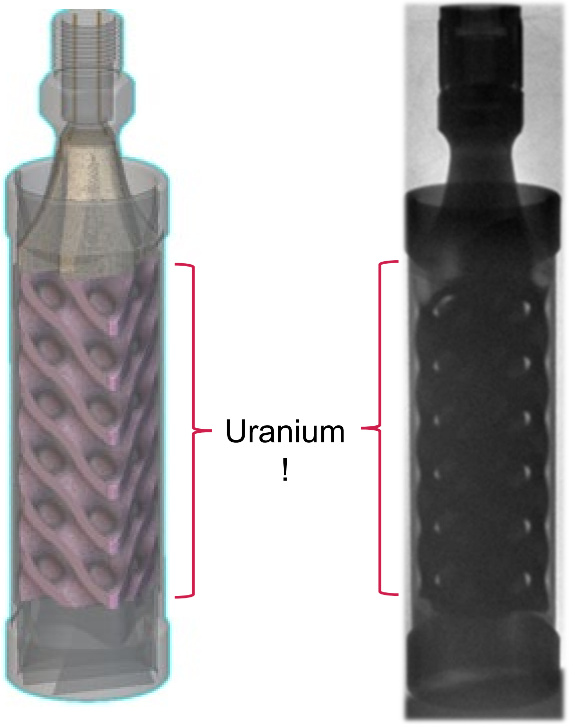




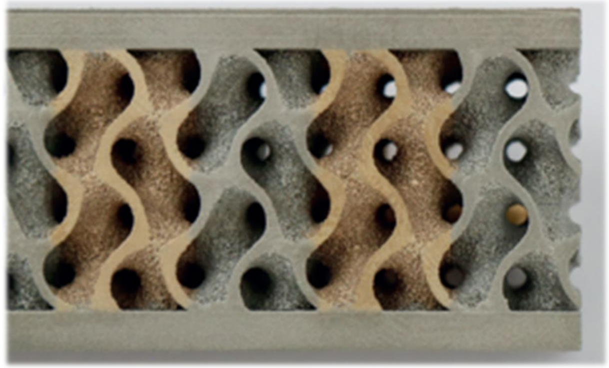
Additively Manufactured Fuel

- Greatest challenge is developing commercially-viable manufacturing technique
- Nuclear fuel typically multi-material systems, three fabrication categories:
  1. AM the fuel, then apply the cladding (candidate processes such as chemical vapor deposition are slow ways to apply a thin layer)
  2. AM the cladding, then add the fuel (limited to particles, liquids, flowable precursors), perhaps followed by consolidation processes
  3. Multi-material AM processes (specialized machines, probably with metal-metal systems)
- Will take a program with multi-year multi-million-dollar staying power to develop and qualify AM technologies

Sample Cladding Lattices Produced by Specialized L-PBF Process (StarHagen company)



Rendering and X-Radiograph of UO_2 Vibropac in Cladding Lattice, TREAT Test Specimen (Josh Zelina – MFC)



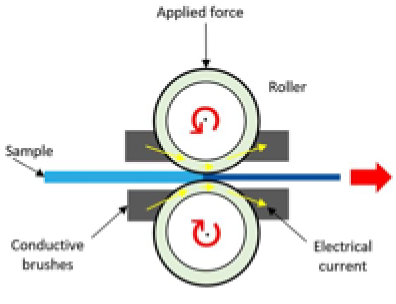
Example multi-material L-PBF lattice (Aerosint company)

Electric Field Assisted Sintering Technologies

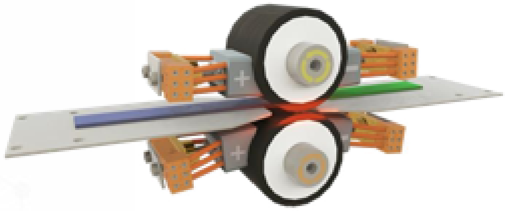
- Current focus on development of process capabilities and material optimization
- World's largest EFAS machine (DCS 800) and capabilities
- New capability - Continuous EFAS

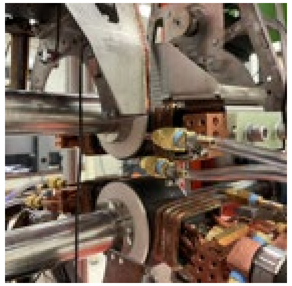
Center of Excellence for Electric Field Sintering Sciences

Continuous EFAS



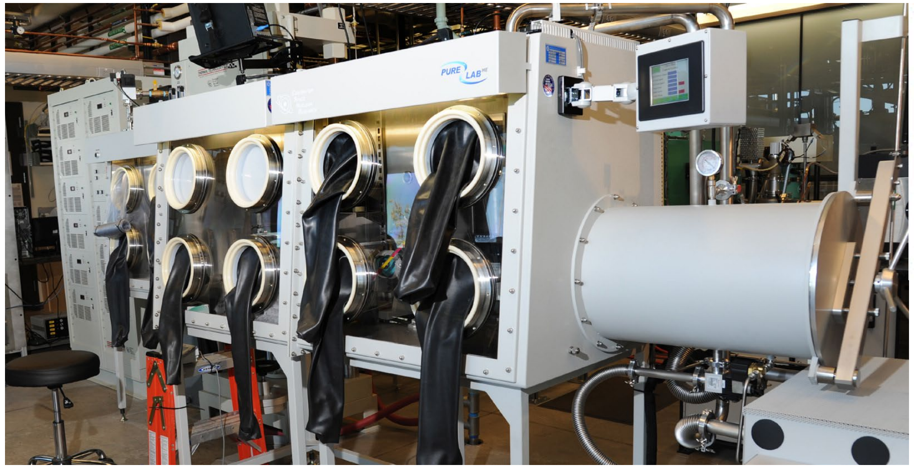
CEFAS: sample passes between two heated rollers.

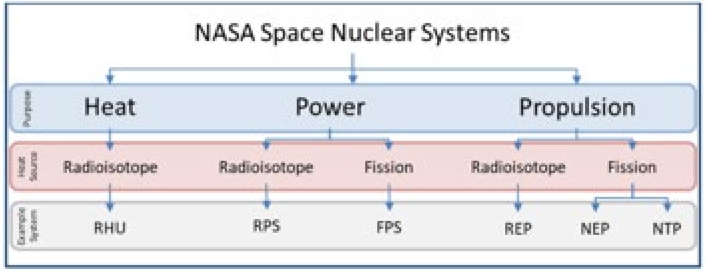


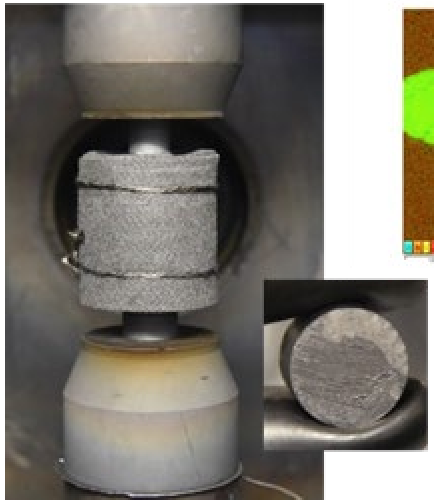


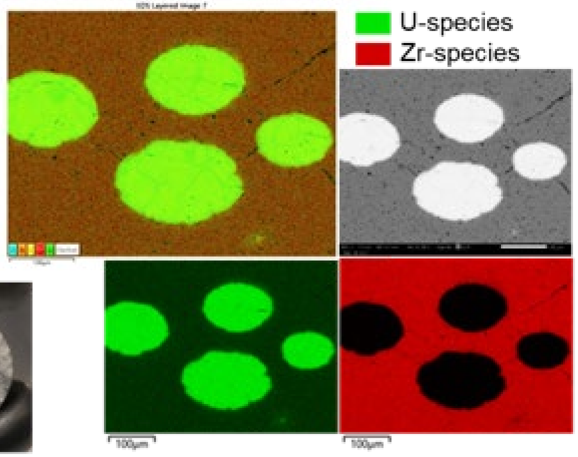
INL EFAS Capabilities
Radiological "SPS" (DCS 25-10)

- 25 ton / 10,000 Amp
- 20mm - 75mm sample size
- 2300°C Cont. / 2500°C Peak
- Advanced nuclear fuels fabrication
- Integrated within radiological work glovebox
- NTP and handling of special powders requiring controlled environment

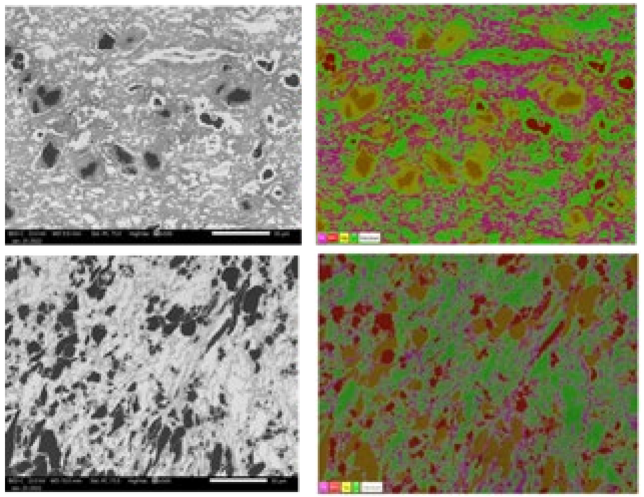








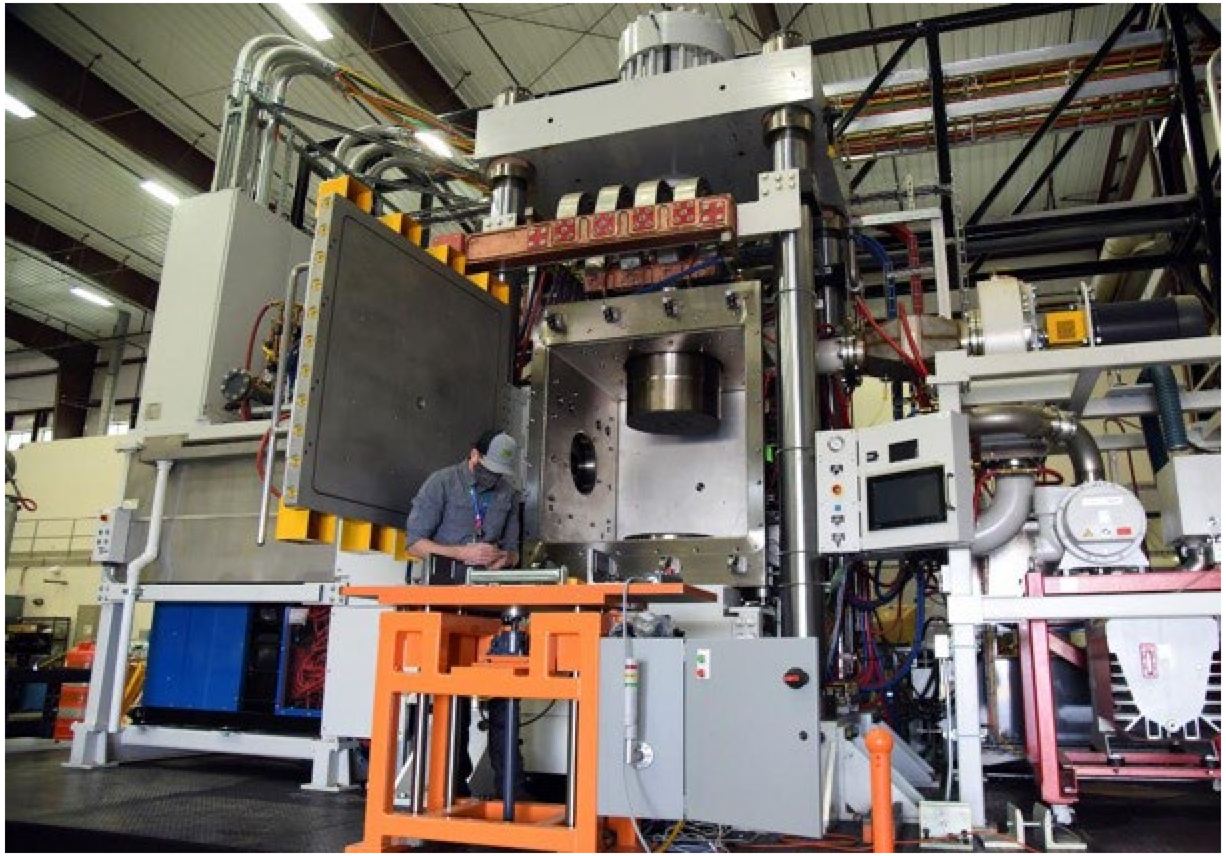
- UN/ZrC, UN/W-Re
- Samples tested in TREAT to 2500^{o}C



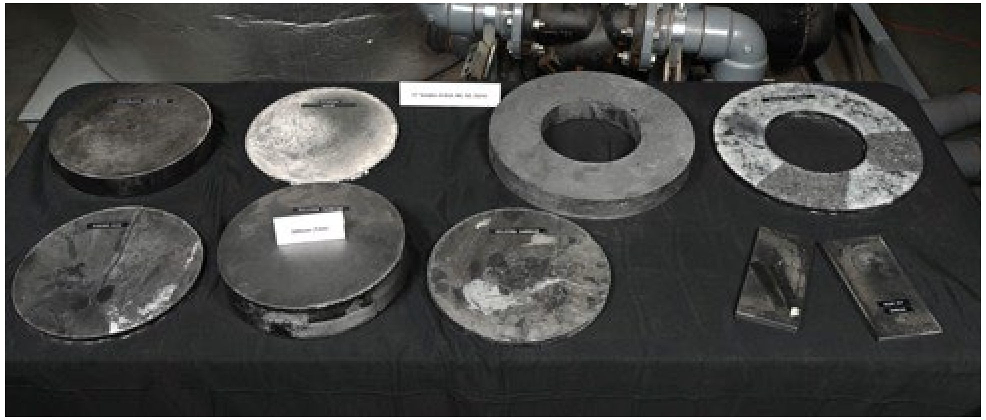
BSE SEM micrographs and EDS maps

RD&D of Industrial Scale EFAS Science

World's largest format, experimentally available, EFAS System.

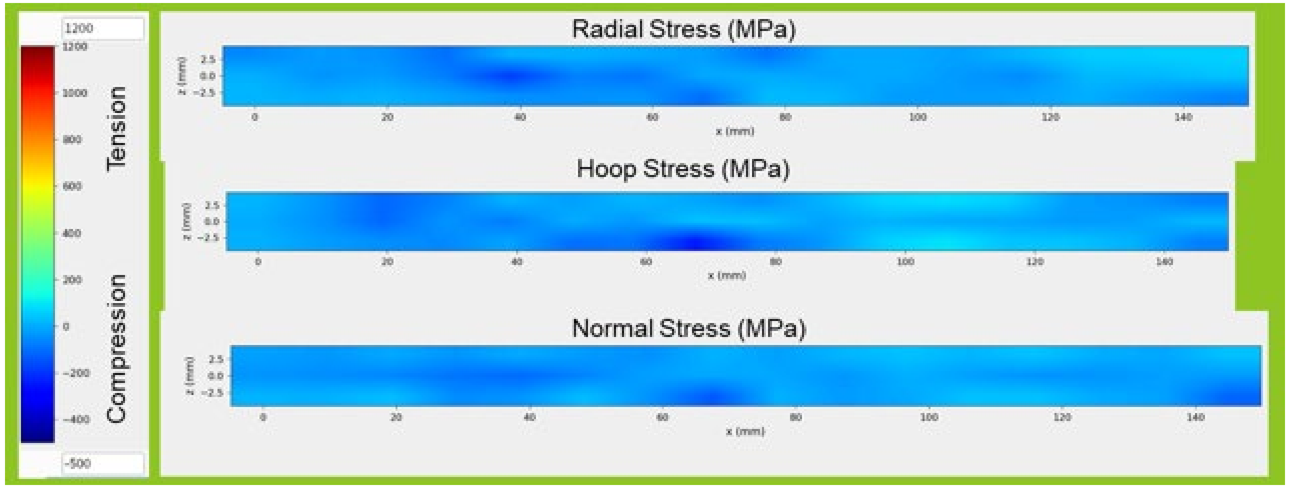


- Operational
- Densifies hard to sinter materials quickly (70-90% faster)
- Customers
  - Industry
  - DOE/NNSA
  - DoD



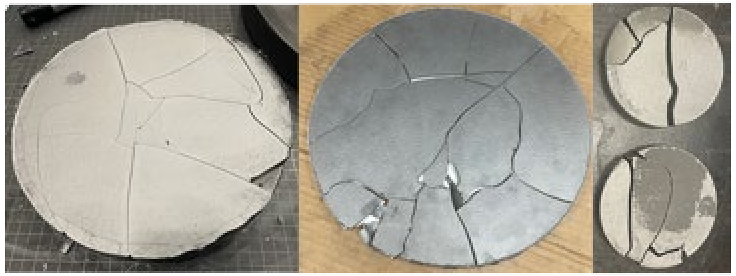
Must manage thermal gradients ←→ internal stress

Jeff Bunn @ ORNL HIFR Neutron Beamline HB-2B | HIDRA (High Intensity Diffractometer for Residual Stress Analysis)

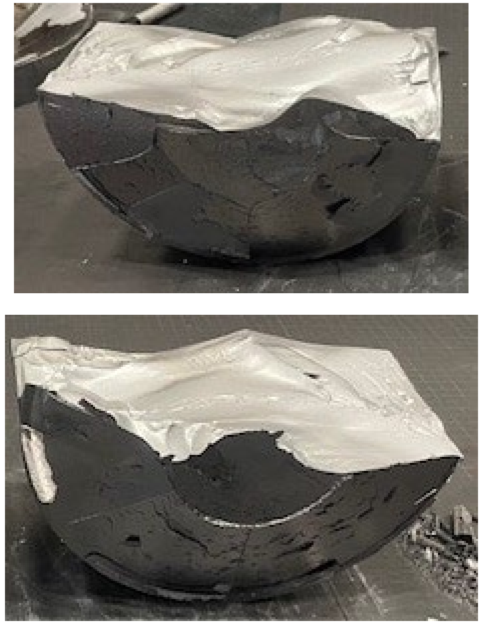


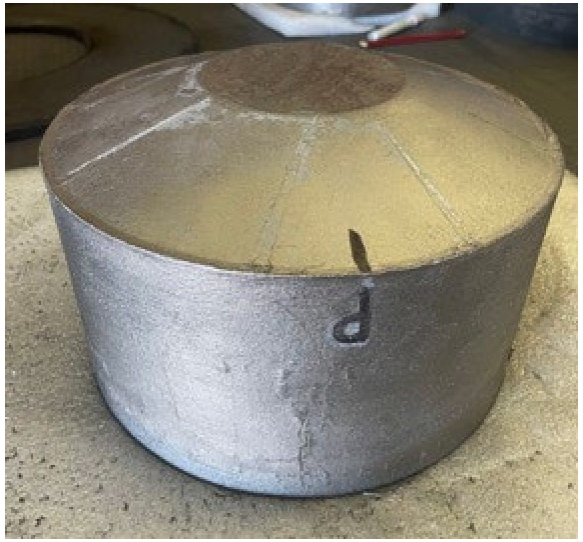
Processing parameters and careful setup reduce internal residual stresses and temperature gradients.

12" 12" 3"



Intact 12" diameter ceramics



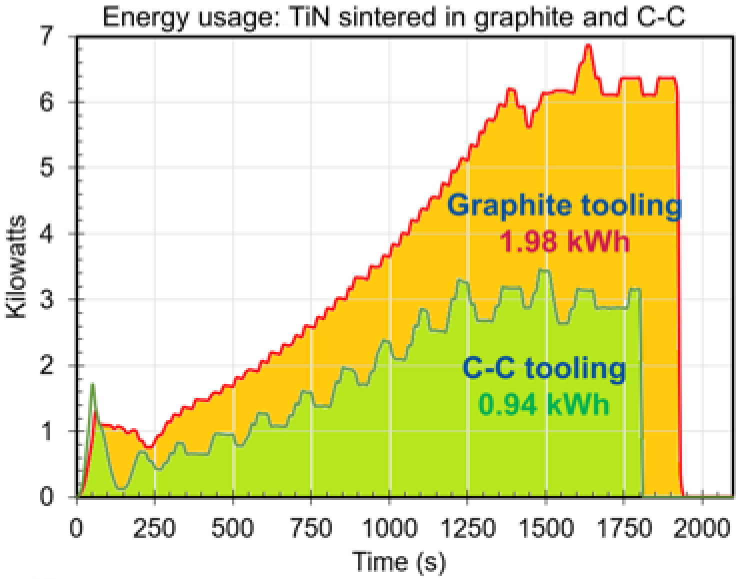


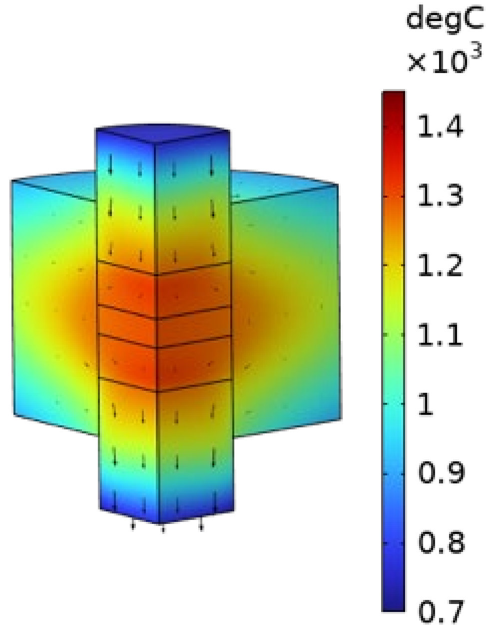
3D Printed CMC's for Advanced Molds, Reactors, and Aerospace

- Controllable anisotropic properties.
- Ultra high temperatures

	INL Carbon-Carbon	Commercial Graphite
	Electrical Resistivity (μΩ*m)	
X/Y Plane	16.5	17
Z-Direction	121.2	13
	Thermal Diffusivity (25 °C)	
X/Y Plane	75	63
Z-Direction	5	82

- Enhanced thermal transport in tailored directions.
- Thermal shock resistant.
- Put the hot zone where you want it
- 50% energy savings by leveraging anisotropic properties of C-C as tooling.





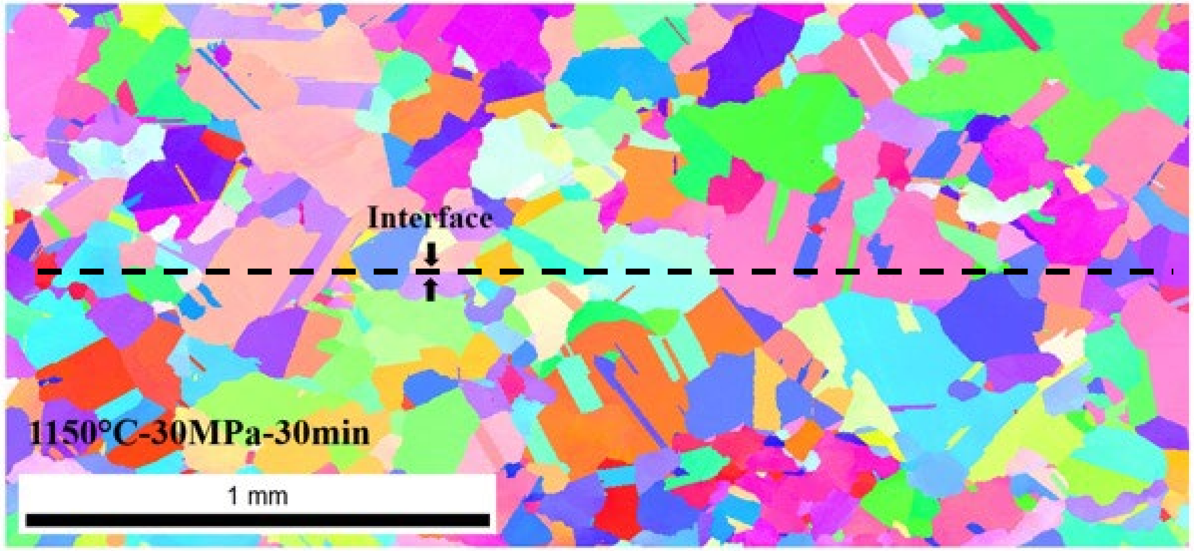
Composites Part B: Engineering Volume 283, 15 August 2024, 111679

Joining of metals with poor weldability and ceramics

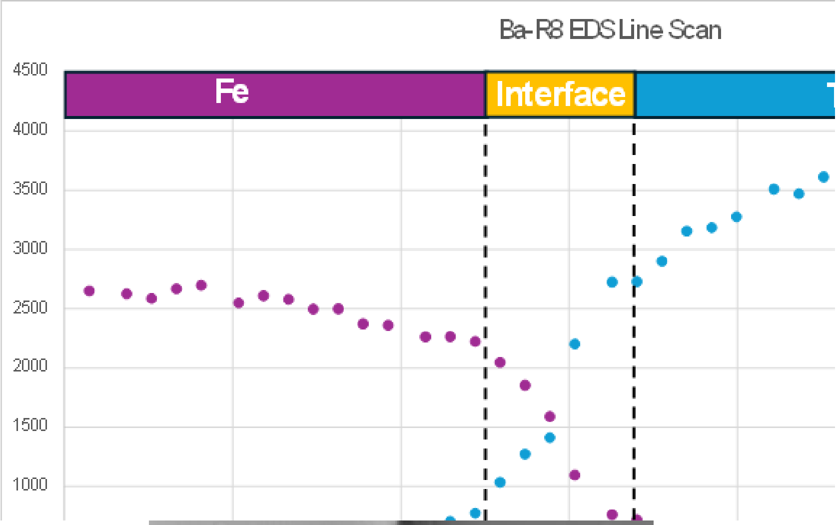
Similar Materials (Inconel 617)

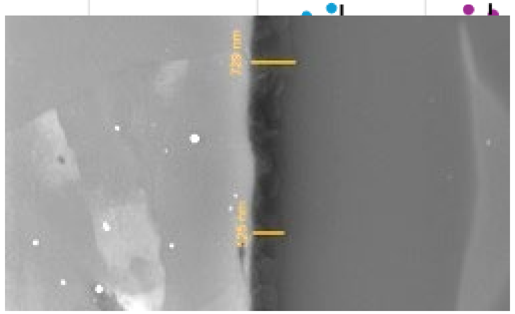
Part 1

Part 2



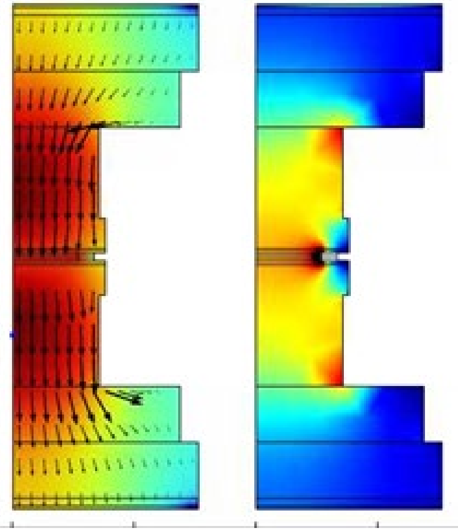
Dissimilar Materials



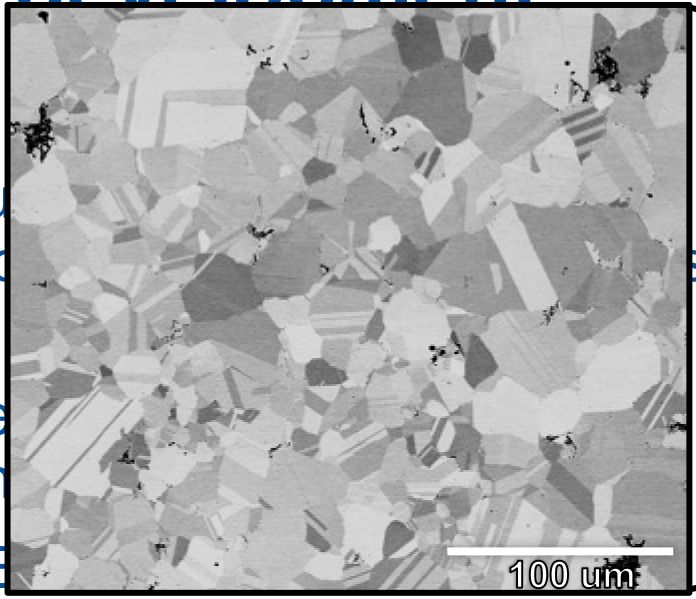


- The intense current during EFAS can have a significant influence on precipitate formation and grain boundary migration in metallic joining.

- Develop models for large plates & stacks.



High throughput tooling strategies for rapid material development



- Cu
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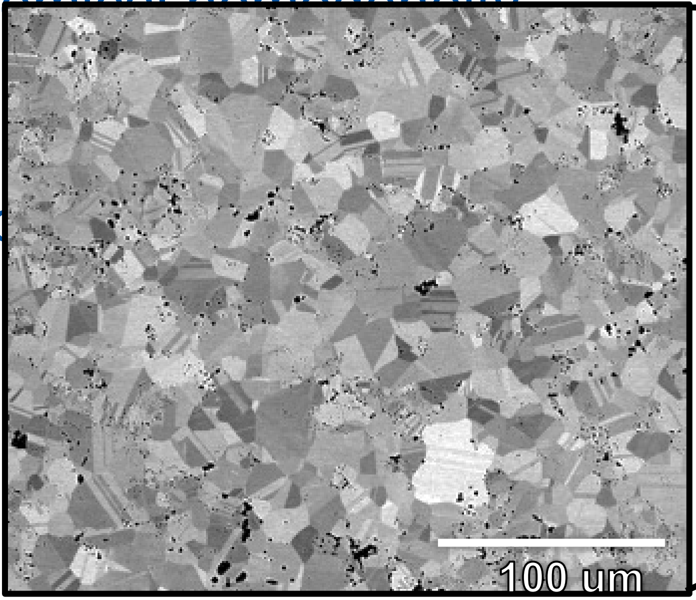
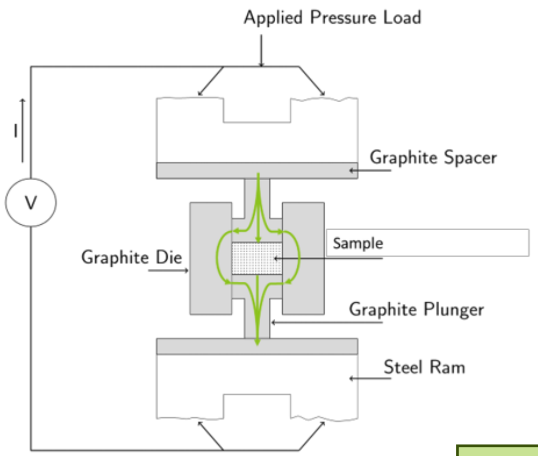
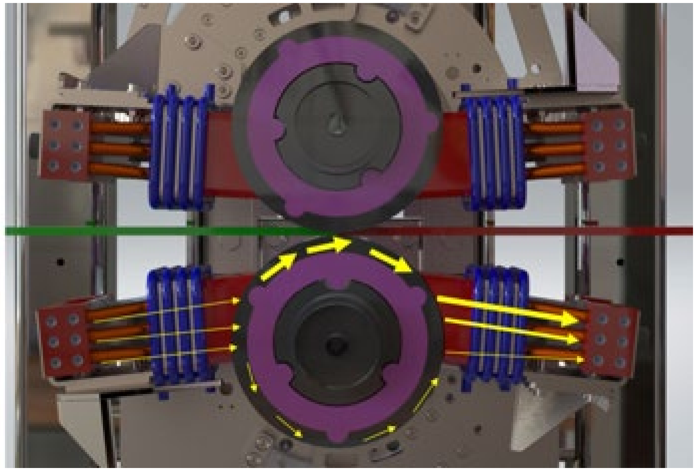
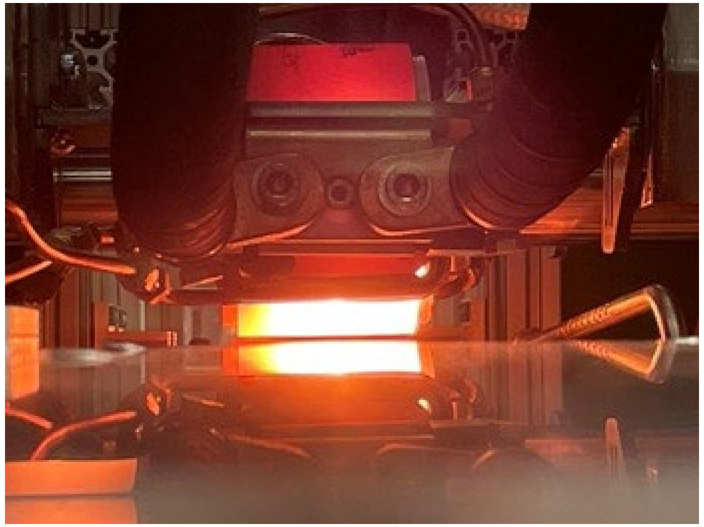


Continuous Electric-Field Assisted Sintering (CEFAS)





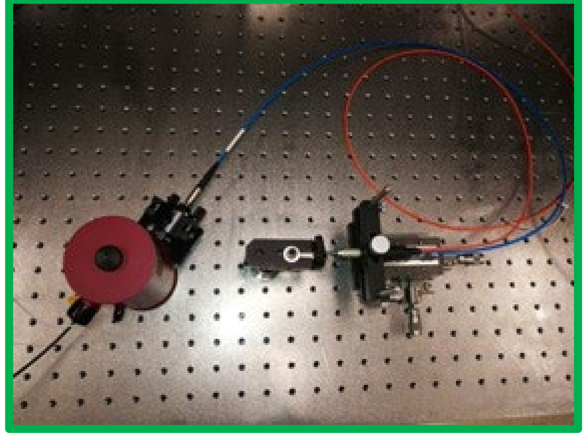


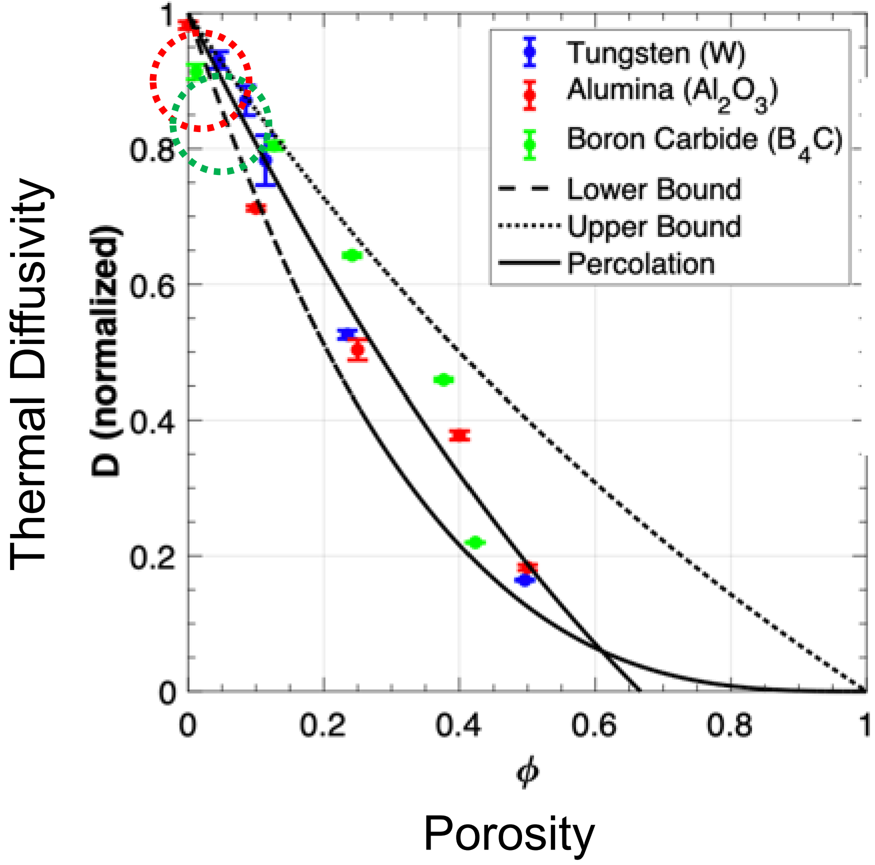
GOAL	*Develop a continuous sintering process that retains the beneficial aspects of traditional SPS/EFAS for processing unlimited sample area.*
Operating Parameters	T_{max} = 1300 °C+ *dia_{roller} = 5”* *V ≤ 10*

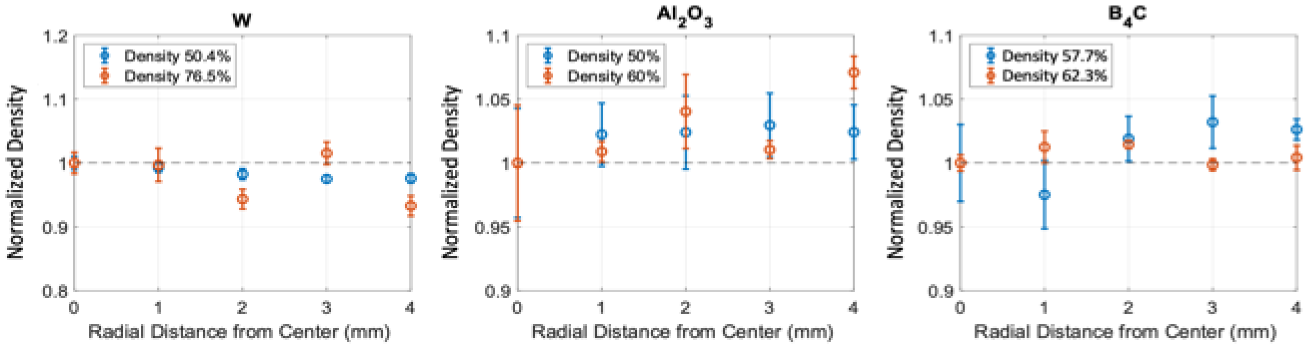
Local Porosity of SPS Samples

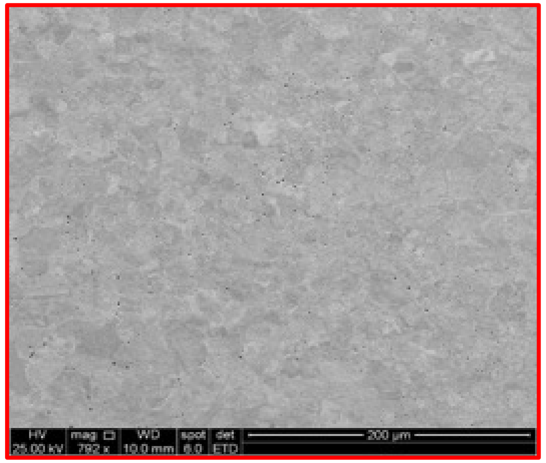
Photothermal Radiometry (PTR)

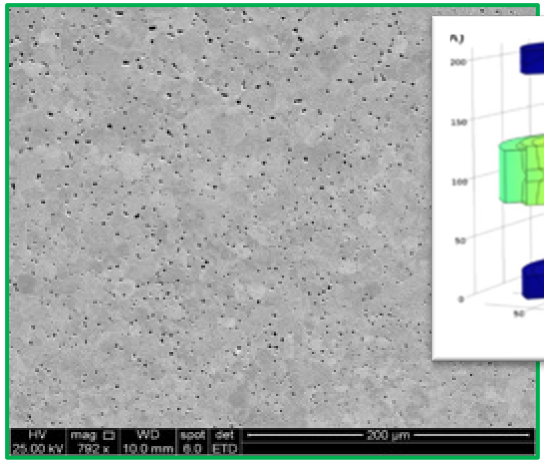
Blackbody radiation

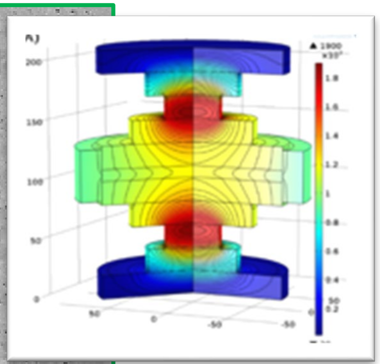








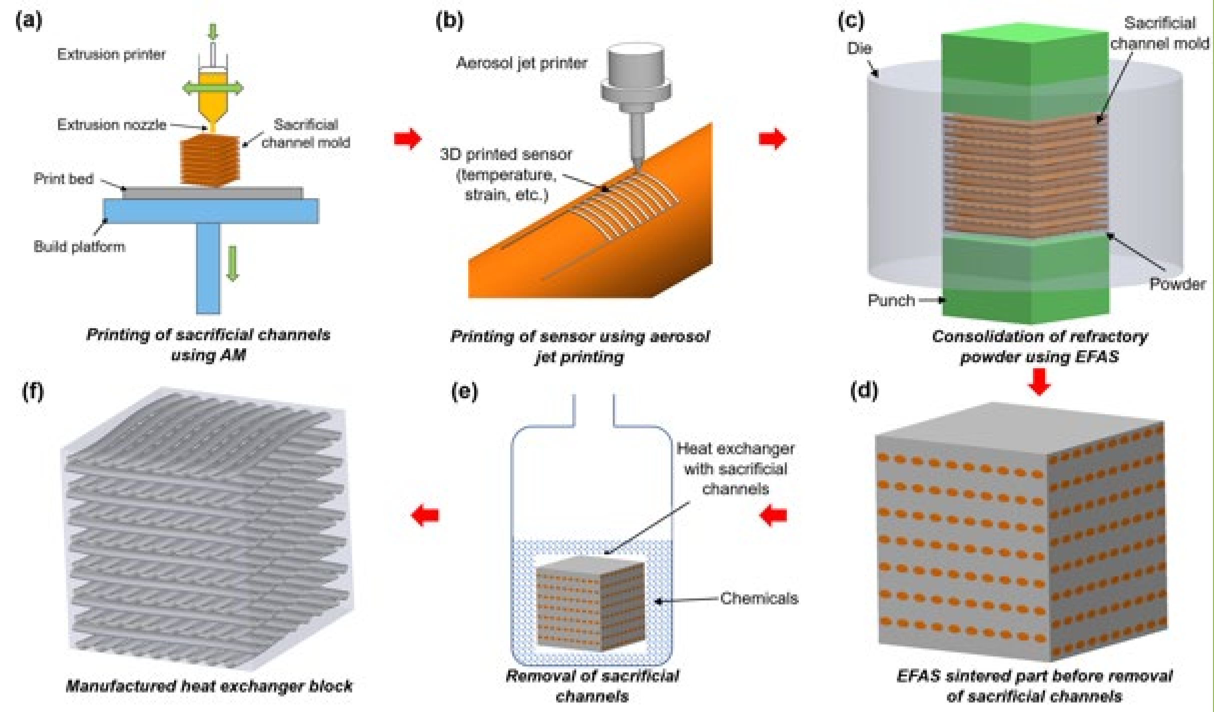




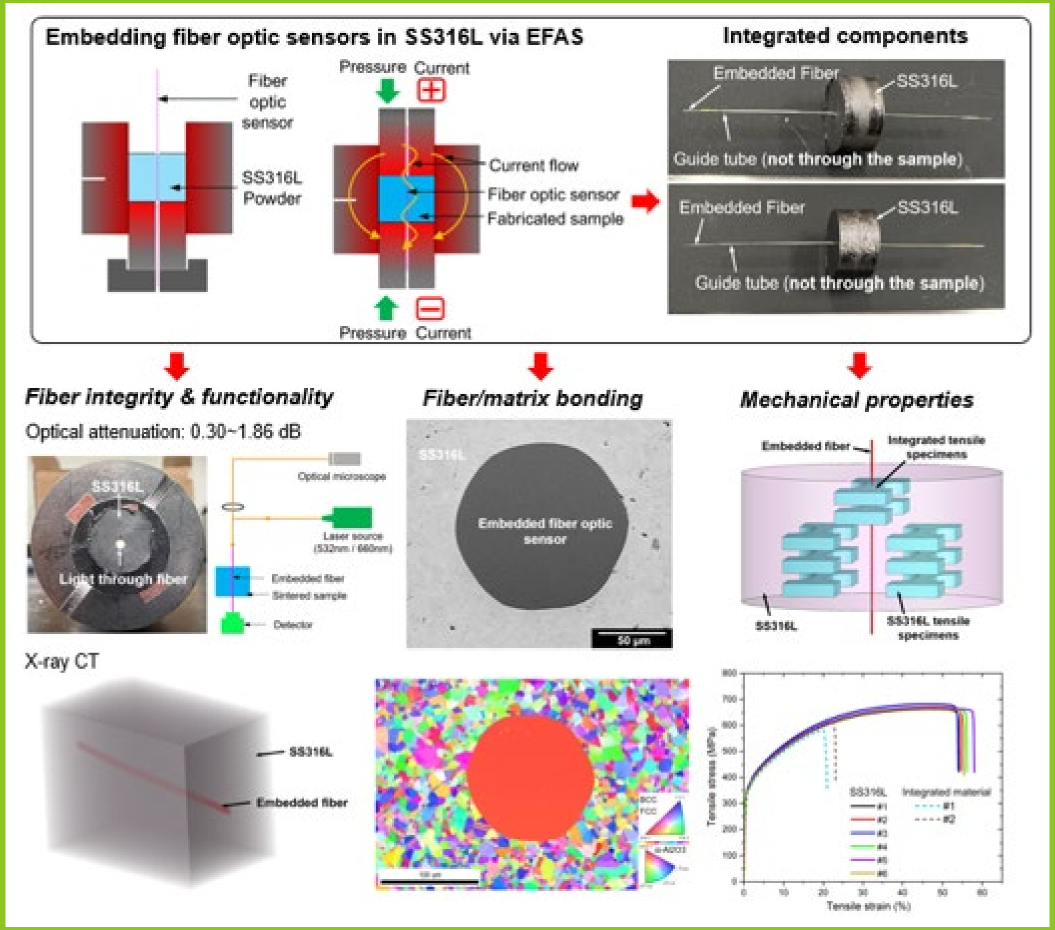
Z. Hua, et al., "Microstructure characterization of electric field assisted sintering (EFAS) sintered metallic and ceramic materials using local thermal diffusivity measurement", AIP Advances, 13, pp095220 (2023)

Embedded Sensors for In-Situ Environmental and Material Monitoring

Refractory metal materials with 3D printed, removable shape holders and embedded sensors for material and system monitoring. Molten salt applications.

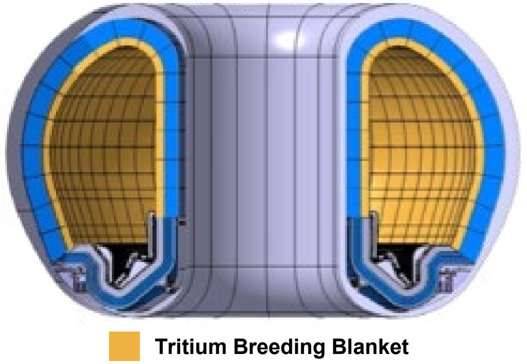


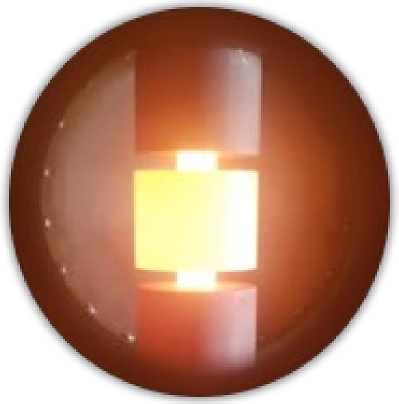
Fiber optic Bragg grating sensors for nuclear applications embedded into metal matrices.



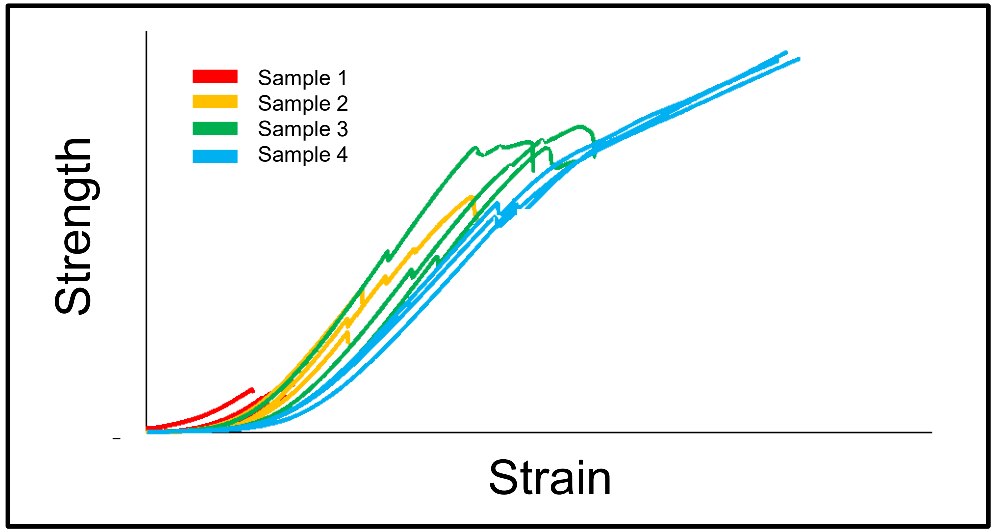
Solid Tritium-Breeder for Fusion Reactors

Fusion reactor cross section





Tritium breeder material during processing

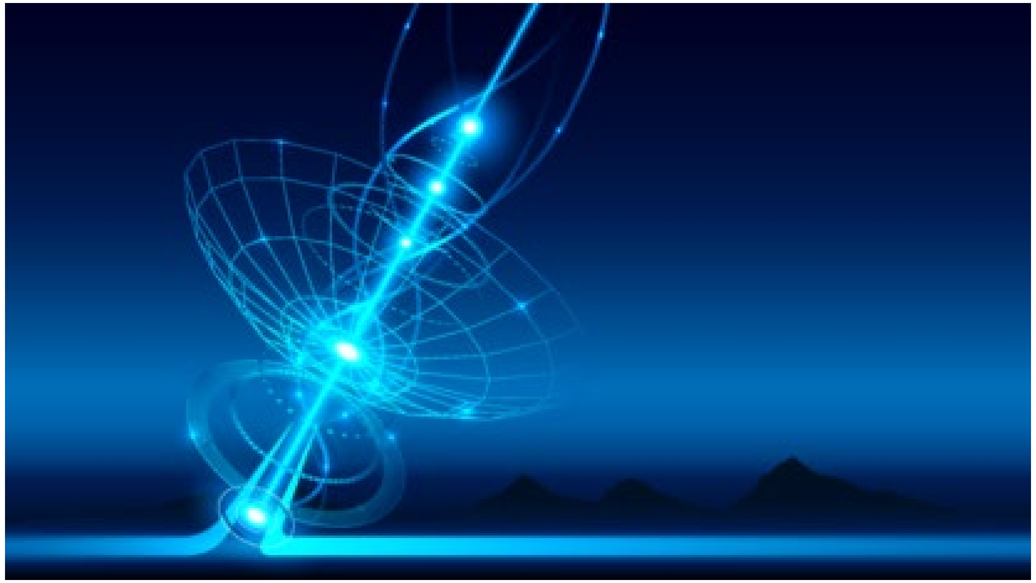


Destructive mechanical testing of new breeder pellet

- Fusion reactors “breed” tritium on-site for sustainable self-fueling.
- Lithium produces tritium when irradiated with neutrons.
- Goal to increase lithium density and mechanical integrity.

Advanced geometries

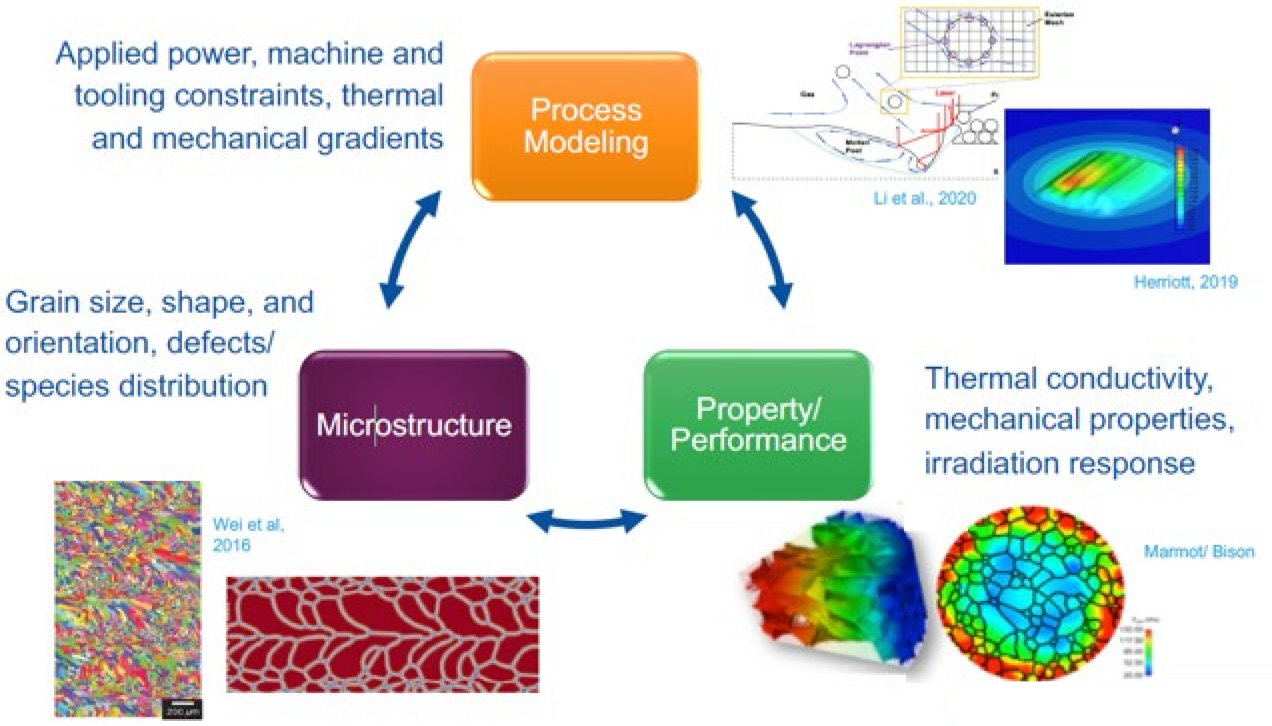
- Research material and pathways for making high temperature ceramics complex geometries.
- Creation of novel multi-component breakaway molds.
- Integration of 3D printed components and EFAS.



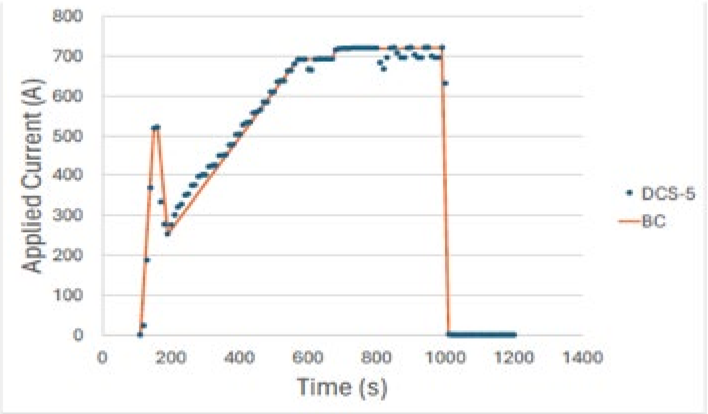
Example part shapes

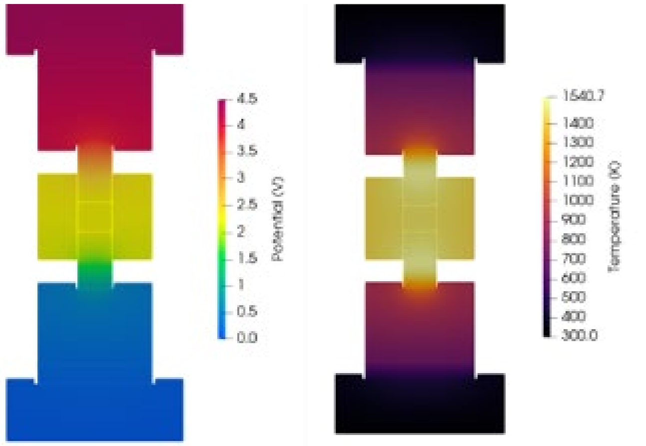
MALAMUTE
MOOSE Application Library for Advanced Manufacturing Utilities

- **Open-Source Advanced Manufacturing Modeling Software Application**
  - Focus on physics-based and extensible capabilities
  - Code development and validation efforts are on-going at INL
  - Builds on MOOSE multi-physics eco-system and multiscale coupling
- **Generalized approach** is adaptable to multiple advanced manufacturing techniques
- **Long-term objective is a validated, predictive modeling & simulation tool to shorten development time and cost to produce and qualify advanced materials**

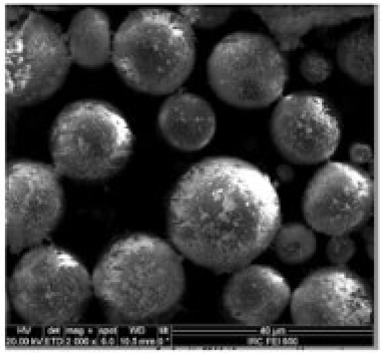


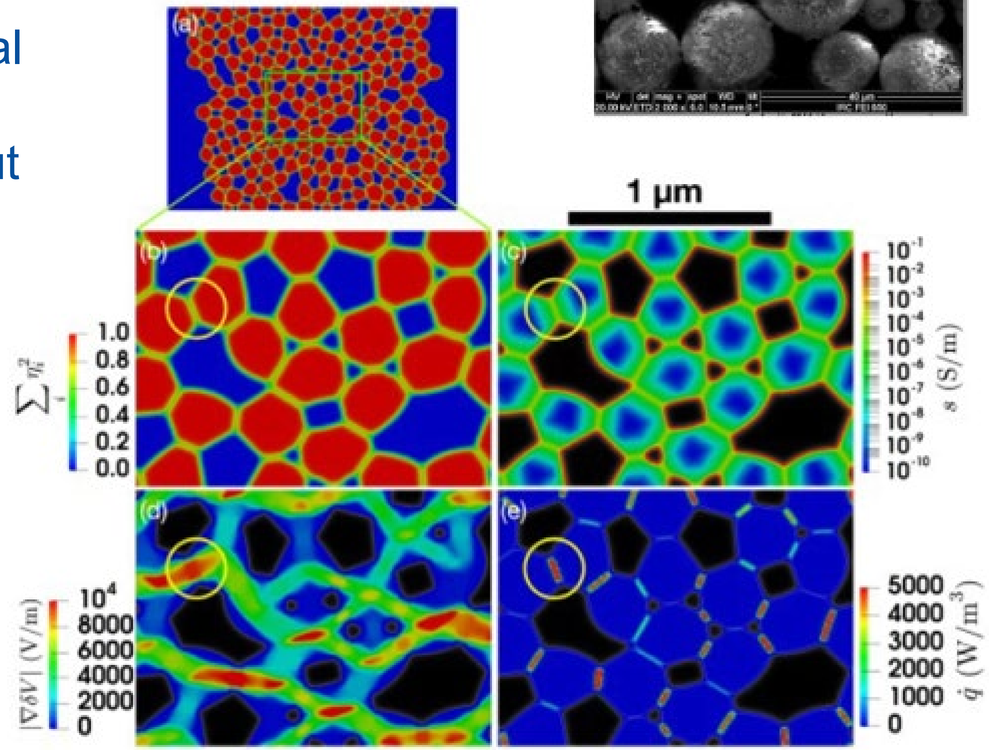
Engineering Scale and Microstructural Modeling





- Solve electrical, thermal, and mechanical PDE's of tooling and sample microstructure.
- Connect changing microstructural properties to distribution of field variables within molds throughout the sintering process.
- Phase field modeling to capture Joule heating along GB's
  - Species diffusion
  - Charged defects
  - Electric potential
  - Electric resistivity
  - Temperature
  - Thermal conductivity
  - Kapitza resistance





Aagesen et al. Acta Materialia 2024

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  - andrea.jokisaari@inl.gov

Question?

Mount Moran, GTNP

Crystal Park Montana

Mt. Biking, Pocatello, ID

Camping, City of Rocks, ID

City of Rocks, Idaho

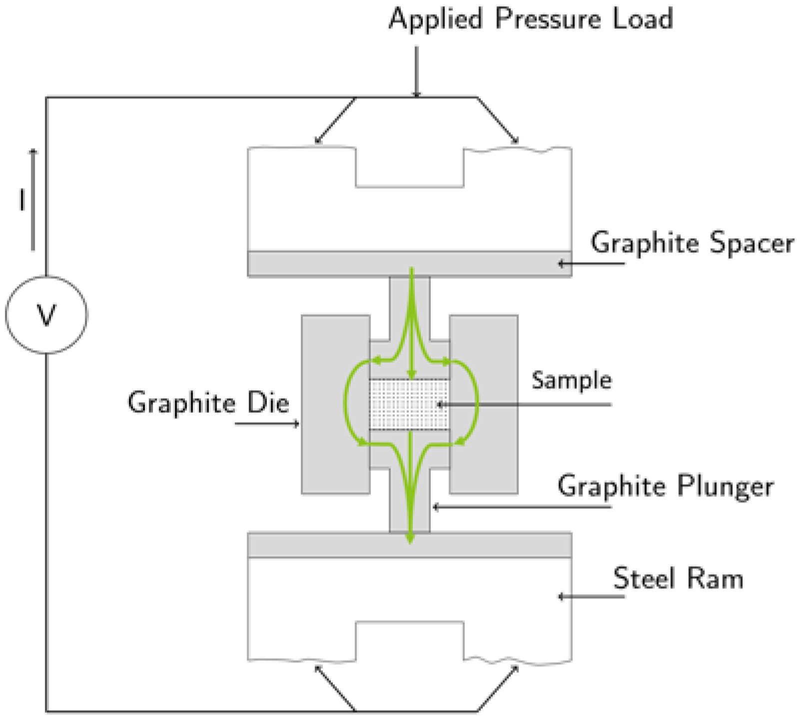
IDAHO NATIONAL LABORATORY 75TH ANNIVERSARY

Battelle Energy Alliance manages INL for the U.S. Department of Energy's Office of Nuclear Energy. INL is the nation's center for nuclear energy research and development, and also performs research in each of DOE's strategic goal areas: energy, national security, science and the environment.

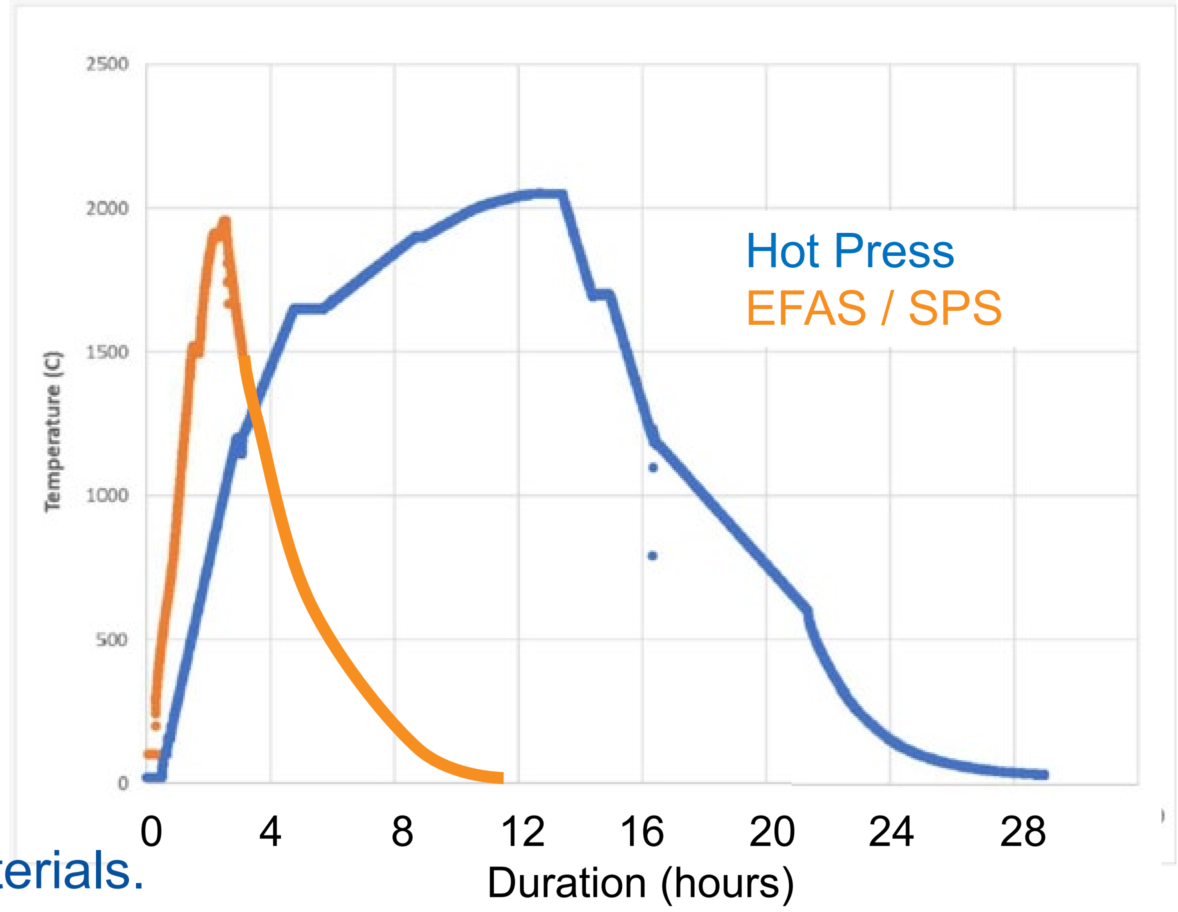
What is field assisted sintering and why do people care about it?

Joule Heating

Current Density



Schematic of EFAS technique rams, tooling, and powder[1]



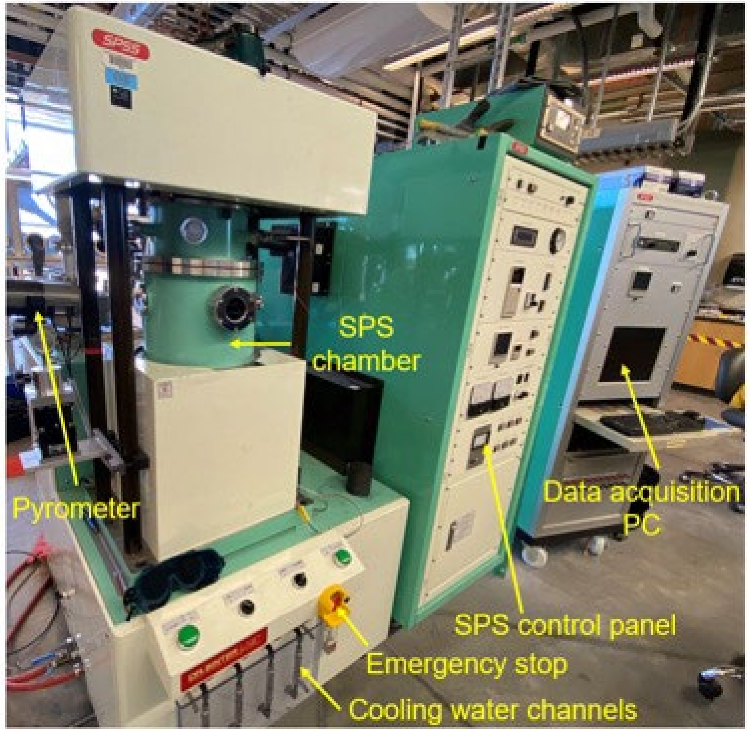
- Exceptional at consolidating difficult to process materials.
- 60-70% faster than closest technological “cousin”.
- Direct heating of sample/mold ensemble.
- Proven at small/research scale → scale up @ INL (DCS-800).

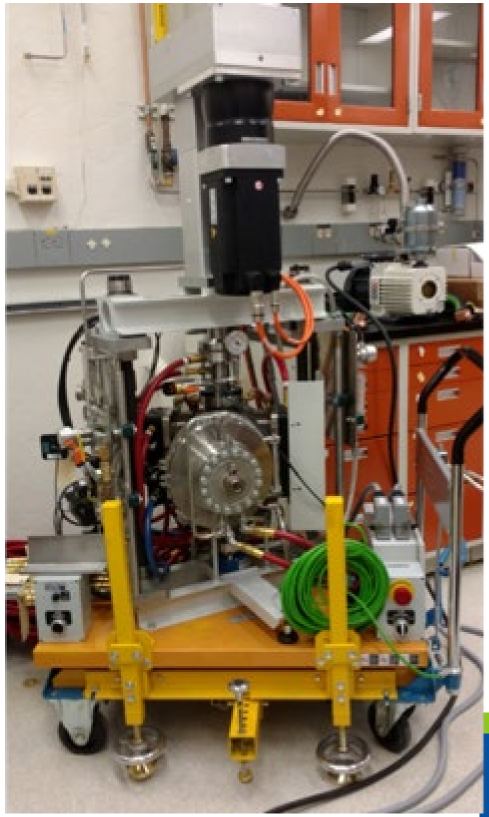
Thermal Technology "MicroSPS"

- 5 ton / 2,000 Amp
- 5-30mm sample size
- Custom built system for use in x-ray radiography cave at INL
- In-situ analysis of microstructural evolution during material processing

Fuji Dr. Sinter 515

- 5 ton / 1,500 Amp
- 5-20 sample size
- 2300°C Continuous
- 2500°C Peak Temp





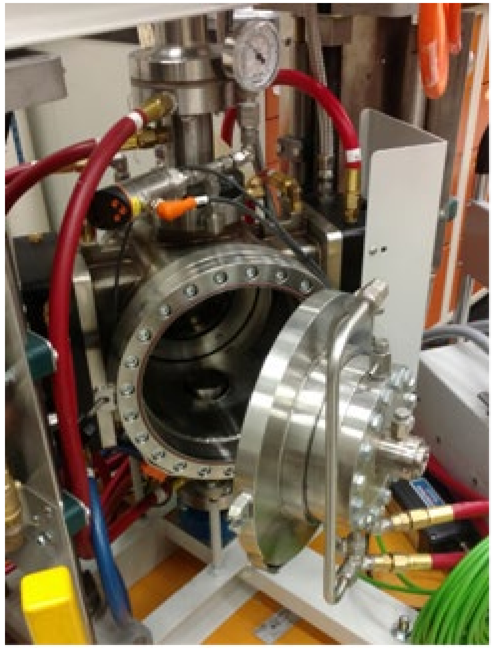


Figure 2. Thermal Chamber

INL EFAS Capabilities "NanoSPS"

- Experimental setup for real time direct observation of chemical and structural evolution between single particles during EFAS, along with the generation of 3D tomograms

- Use at any of our DOE beam line facilities