

Advanced Materials and Manufacturing for Extreme Environments

Dewen Yushu	Artificial Intelligence Based Process Control and Optimization for Advanced Manufacturing
Hypo Chen	Synthesizing thin, dense ion conductive layers via digital light processing assisted electro sinter forging for energy storage devices
Jorgen Rufner	Large Scale Spark Plasma Sintering Process and Die Design
Nathan Jerred	Nanostructuring of Uranium Based Metallic Fuels via Spark Plasma Sintering
Ryan Bratton	Shock Wave Mitigation in Metal Materials Through Advanced Manufacturing Processes
Tiankai Yao	In-situ Probing of Temperature, Strain, and Phase Change in Spark Plasma Sintering
Xinchang Zhang	Electric Current Enhanced Diffusion Welding to Fabricate Compact Heat Exchangers for Nuclear Applications
Xinchang Zhang	Embedded Fiber Optic Sensors for Real Time in situ Sensing in Extreme Environments
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Artificial Intelligence-Based Process Control and Optimization for Advanced Manufacturing

Dewen Yushu, Peter German, Asa Monson, Michael McMurtrey (INL), Xu Wu, Mahmoud Yaseen (NCSU)

Background

- Process qualification, design optimization, and material discovery are among the main challenges in advanced manufacturing (AM).
- This project aims to create an intelligent AM system that can minimize human inputs in the optimization process while relying on an **automated process-level control mechanism** to generate **optimal design variables** and **adaptive system settings** for improved end-product properties.

ML is Enabled within MOOSE

- MOOSE-based machine learning (ML) capability is enabled by linking MOOSE with LibTorch (C++ front end of PyTorch).
- The linkage enables neural network (NN)-based controller, surrogate model, and reduced order model training and deployment on the fly.



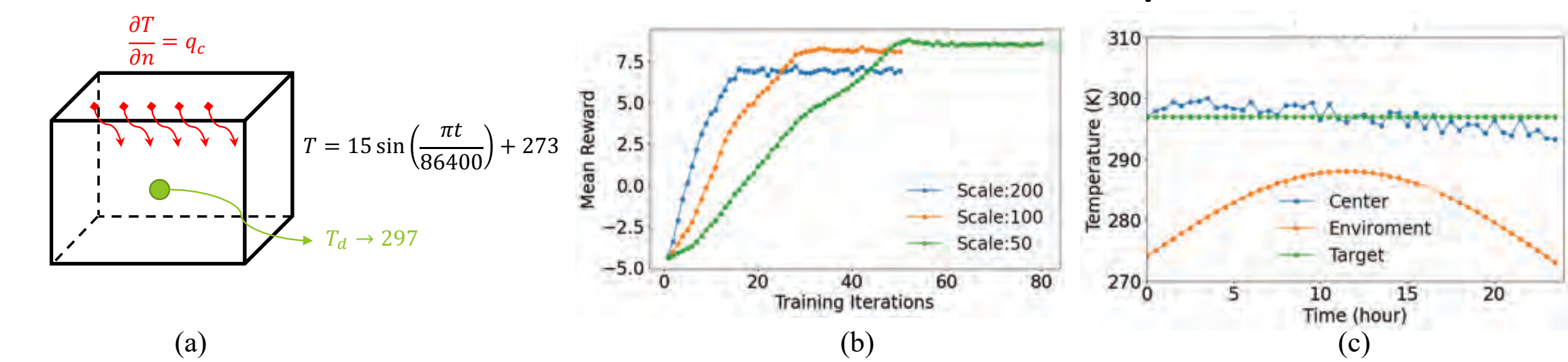
Multiphysics Object-Oriented Simulation Environment



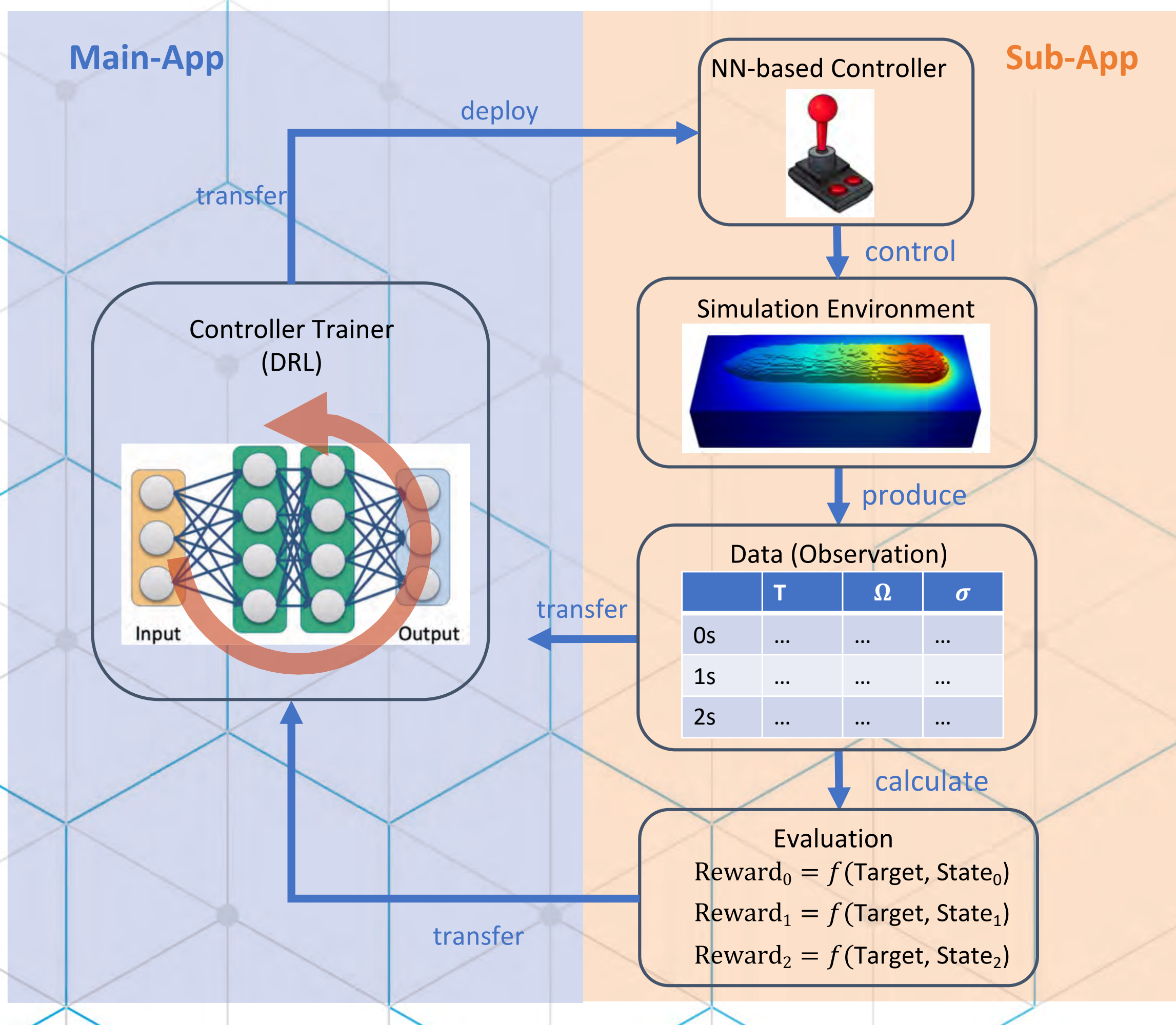
Machine learning library

DRL-based Controller

- Deep reinforcement learning (DRL)-based controller is implemented in MOOSE using the proximal policy optimization (PPO) algorithm.
- Effectiveness of the DRL-based controller is demonstrated on a 3D heat conduction problem.

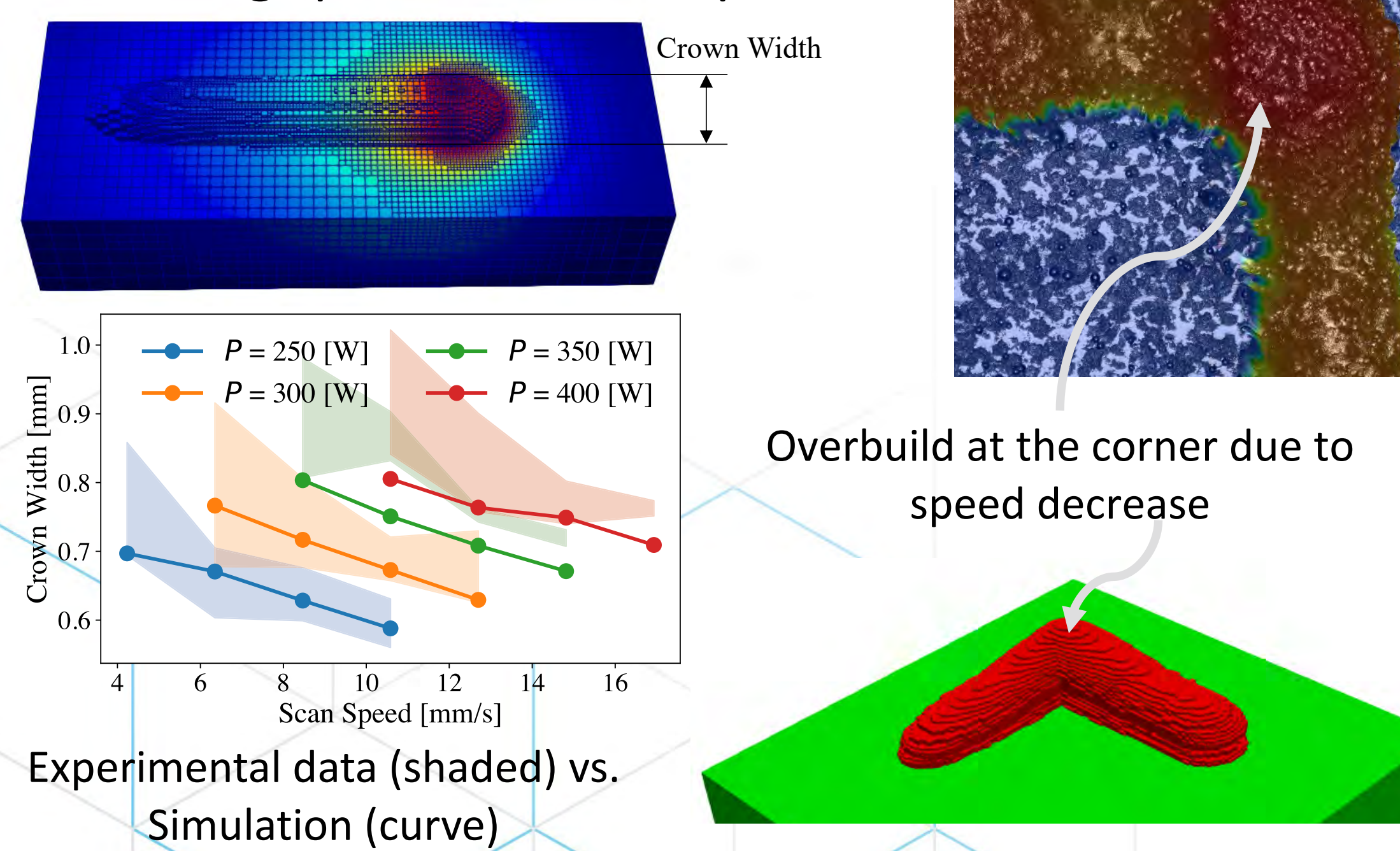


AI-Based Controller Training



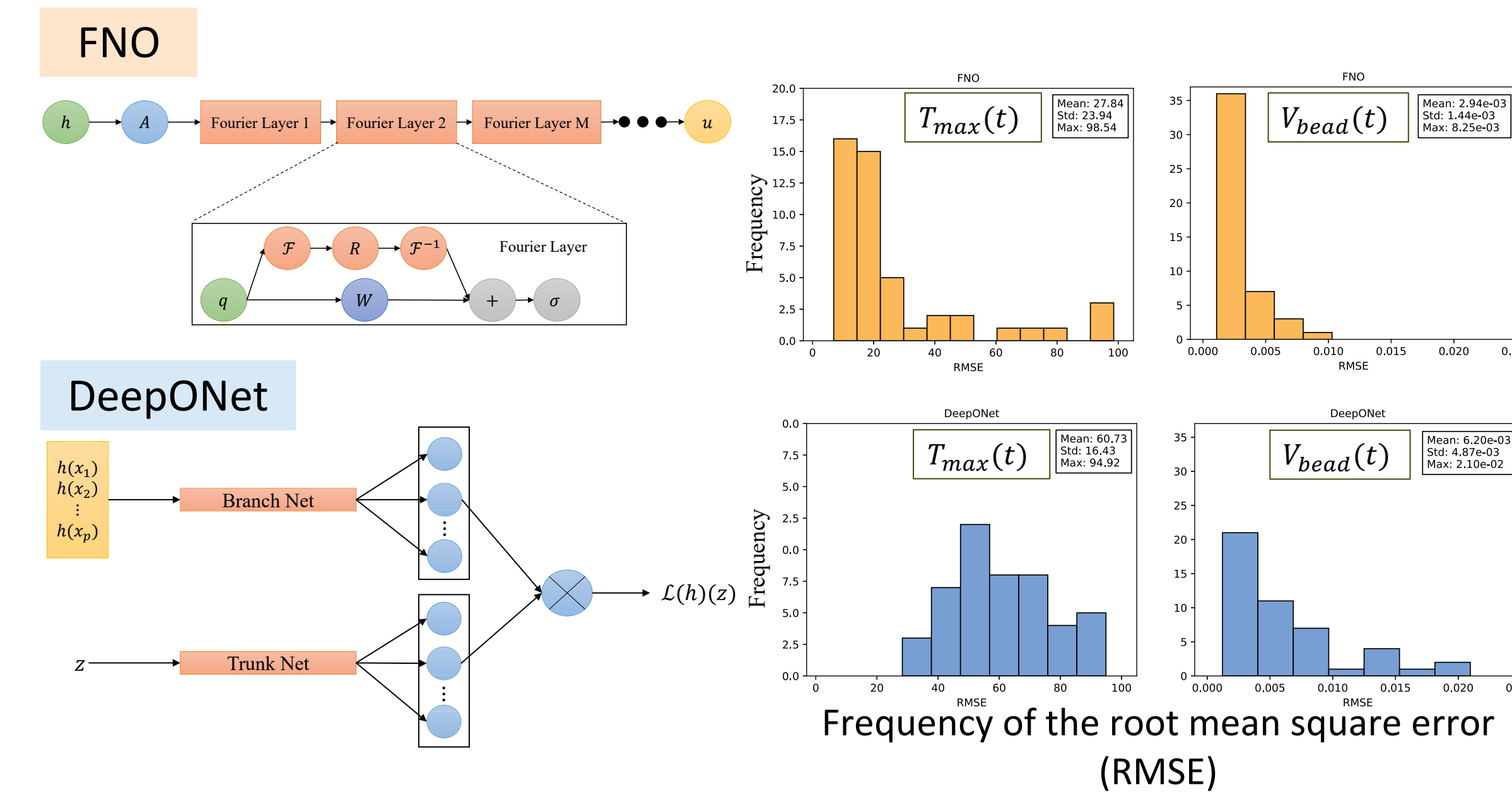
DED Modeling and Validation

- Directed energy deposition (DED) process model is improved by enabling **adaptive mesh refinement** and taking **feed-rate and machine uncertainty** into account.
- Model validation is carried out by comparing geometrical features for single-bead scans using various scanning speeds and laser powers.



ROM Development

- Reduced order model (ROM) is developed using Fourier neural operator (FNO) and deep operator network (DeepONet) based on the high-fidelity DED model.
- Both FNO and DeepONet accurately predict time-dependent temperature and bead volume with minimal computational cost.



Project Number: 22A1059-047FP

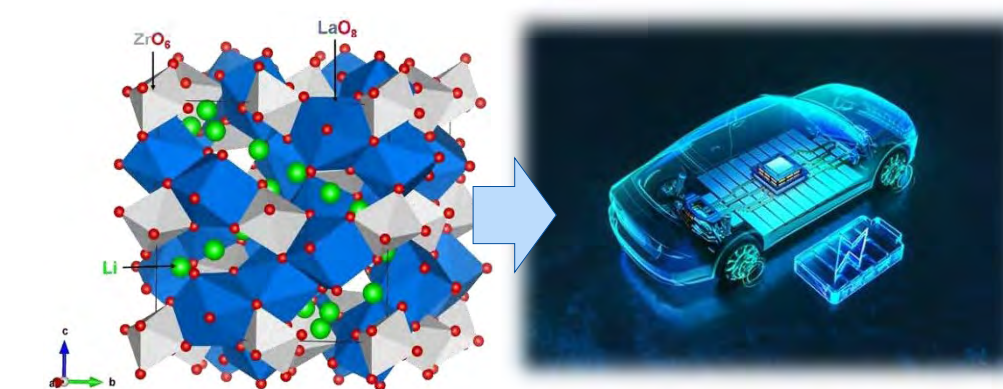
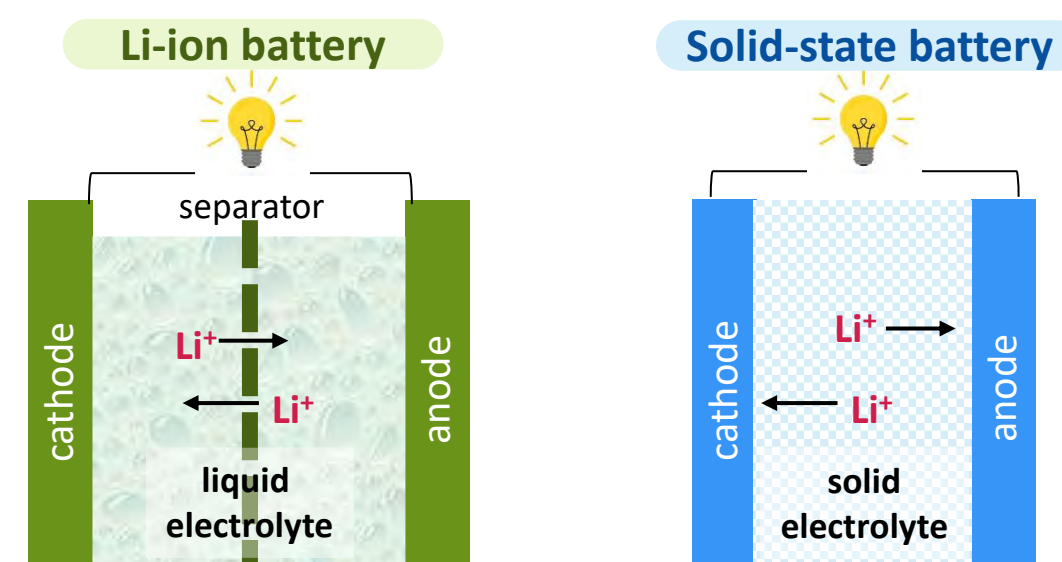
LRS Number: INL/RPT-23-74171

Advanced manufacturing of solid-state electrolytes

Lead PI: Bor-Rong 'Hypo' Chen

Energy Storage & Electric Transportation Department
Energy Environment Science & Technology (EES&T), Idaho National Laboratory

Solid-state batteries: A revolution in the next-generation electric vehicles



$\text{Li}_7\text{La}_3\text{Zr}_2\text{O}_{12}$ (LLZO) is a promising SE material

- High Li conductivity (10^{-4} S cm^{-1})
- Chemical stability against Li metal anode

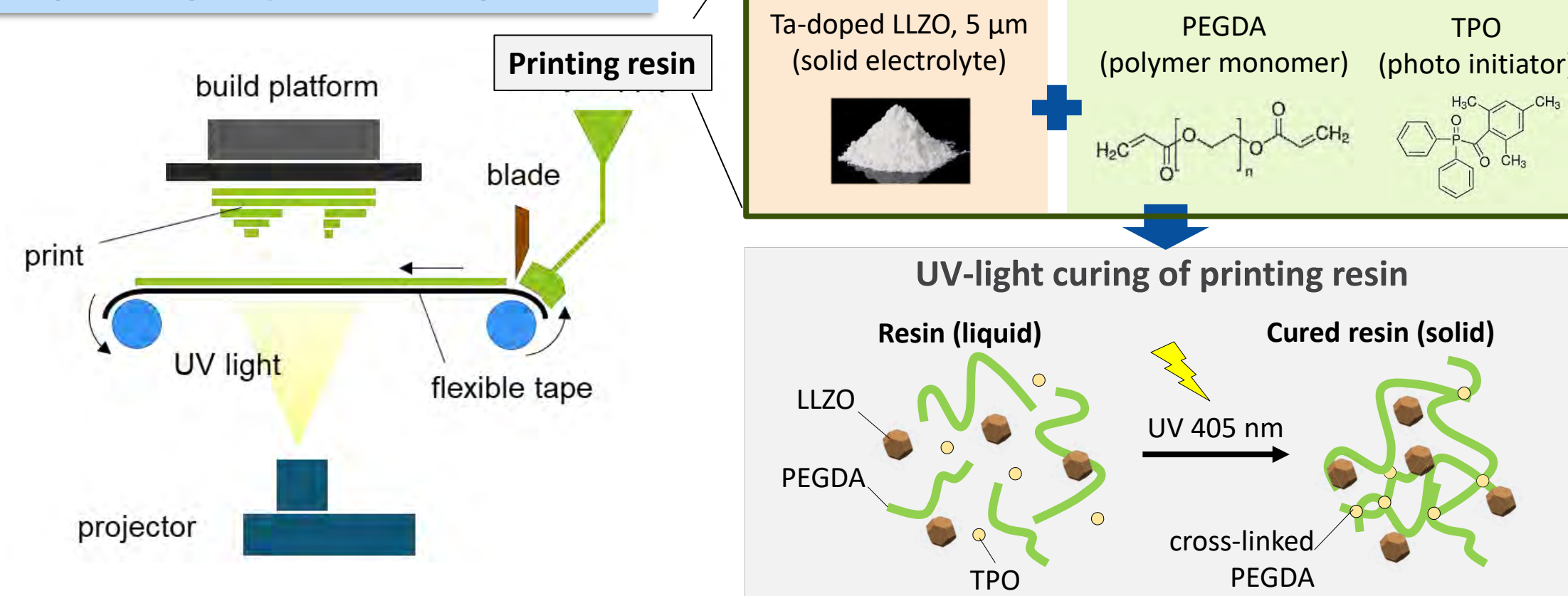
But challenged by....

- Processibility into thin (< 100 μm) & dense (> 95% density) layers
- Energy-consuming processing conditions (> 1000 $^\circ\text{C}$, 12-18 hr)

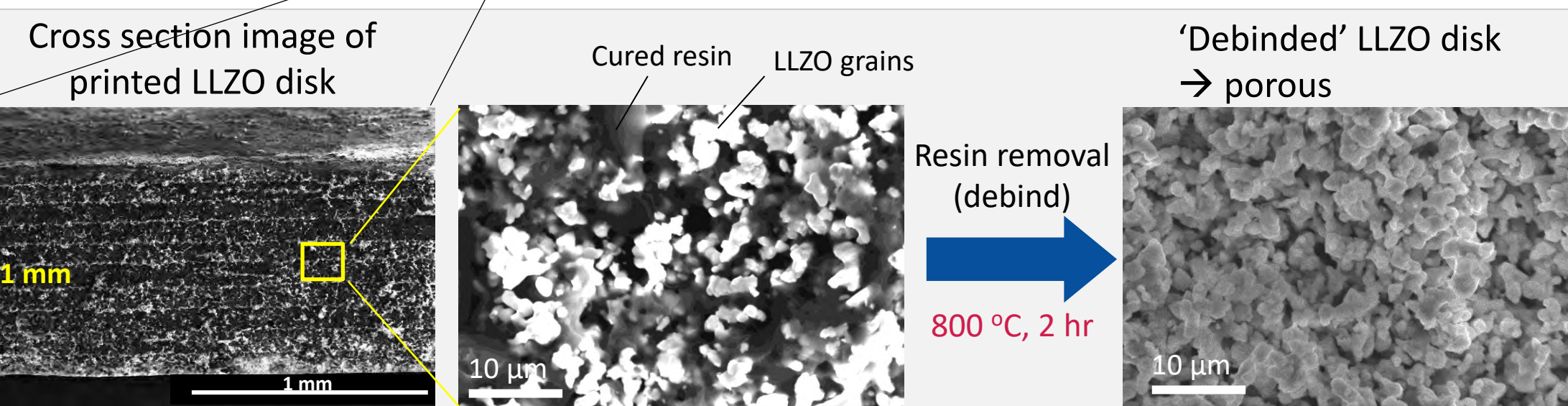
Electrolyte allows lithium ion (Li^+) to cycle between electrodes during battery charging & discharging. A solid-state battery replaces conventional liquid electrolyte with **solid electrolyte (SE)**, which is the key to higher energy density and improved safety for future electric vehicles.

3D printing of LLZO electrolytes

Digital light processing (DLP)



INL's Admaflex 130 DLP printer and demonstrations



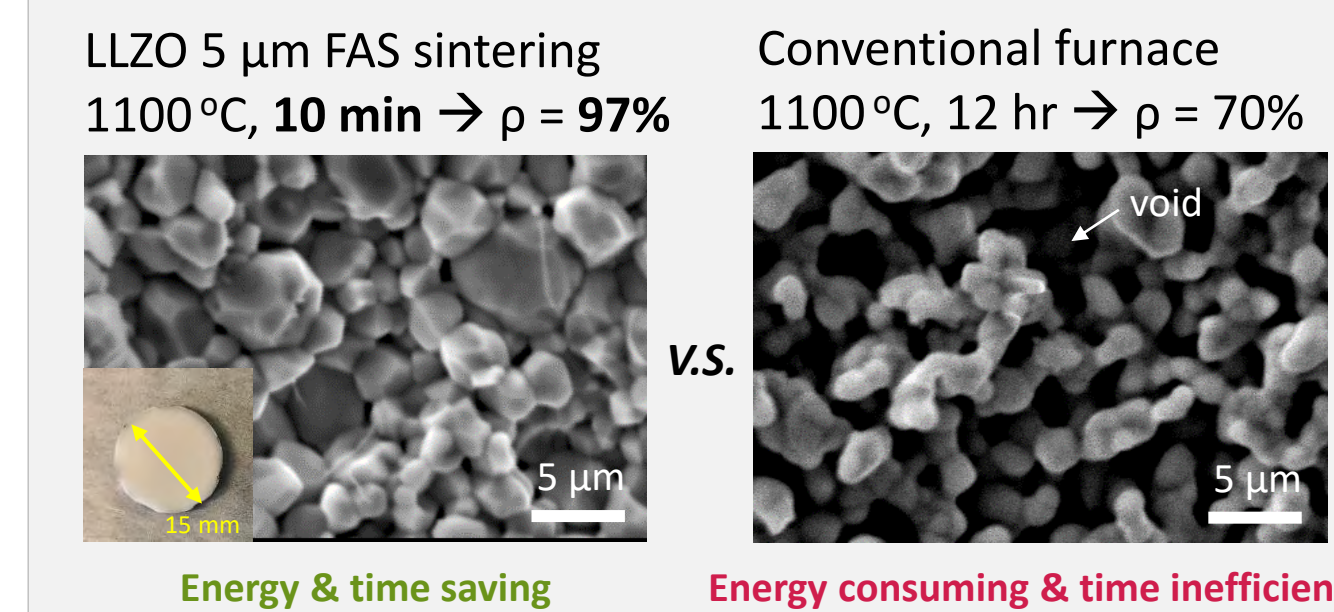
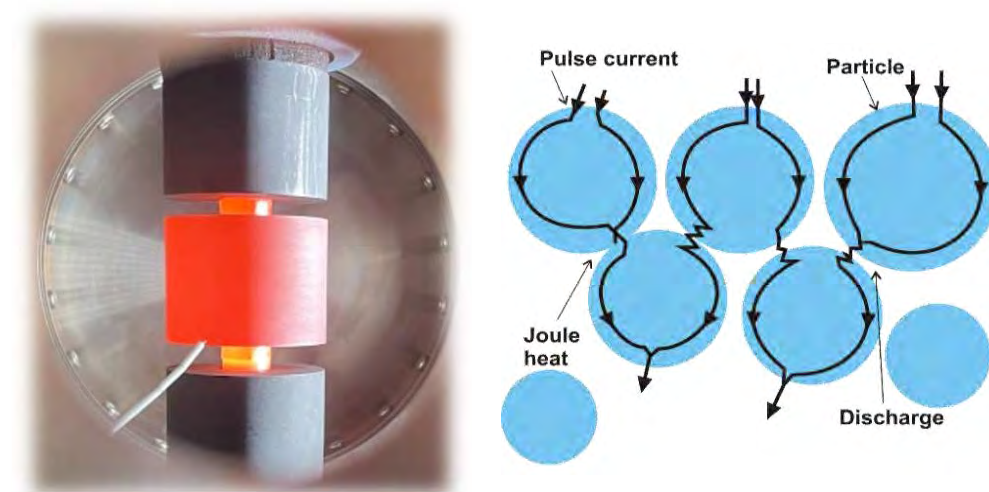
How to make the printed LLZO structure function as solid electrolyte?

Thin and dense LLZO layers: Combining 3D printing and field-assisted sintering



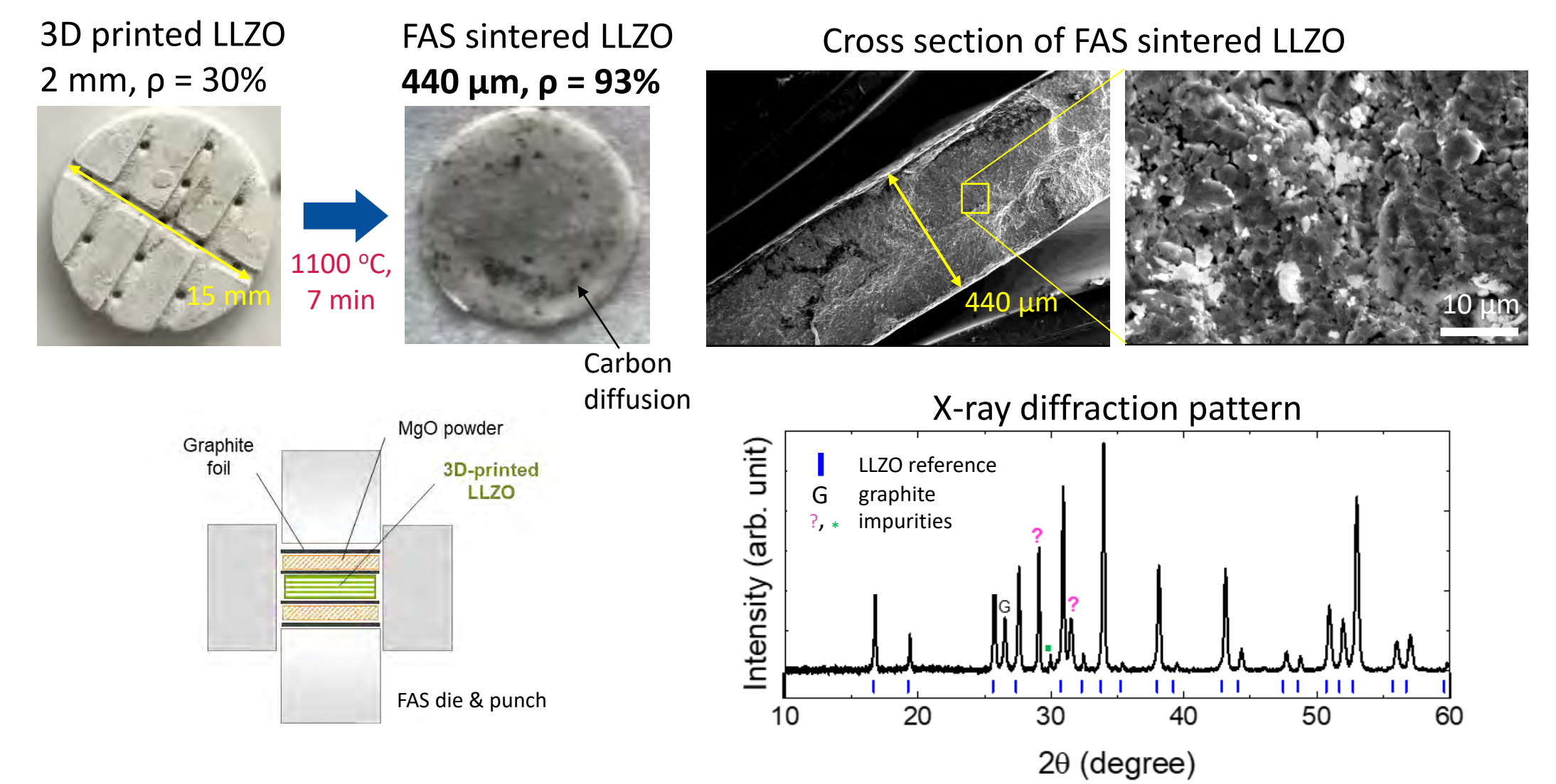
Jorgen Rufner¹, Arin Preston¹, Spencer Doran², Asa Monson¹, Donna Guillen¹
1. Idaho National Laboratory 2. Oregon State University

Field-assisted sintering (FAS)



- FAS uses joule heating created by electric current to densify ceramic or metal powders
- Using FAS, LLZO powders can be densified to 97% density within 10 min
- However, the typical processible scale for FAS is ≥ 1 mm \rightarrow too thick for practical SEs

Printing \rightarrow FAS sintering \rightarrow Thin & dense LLZO SE

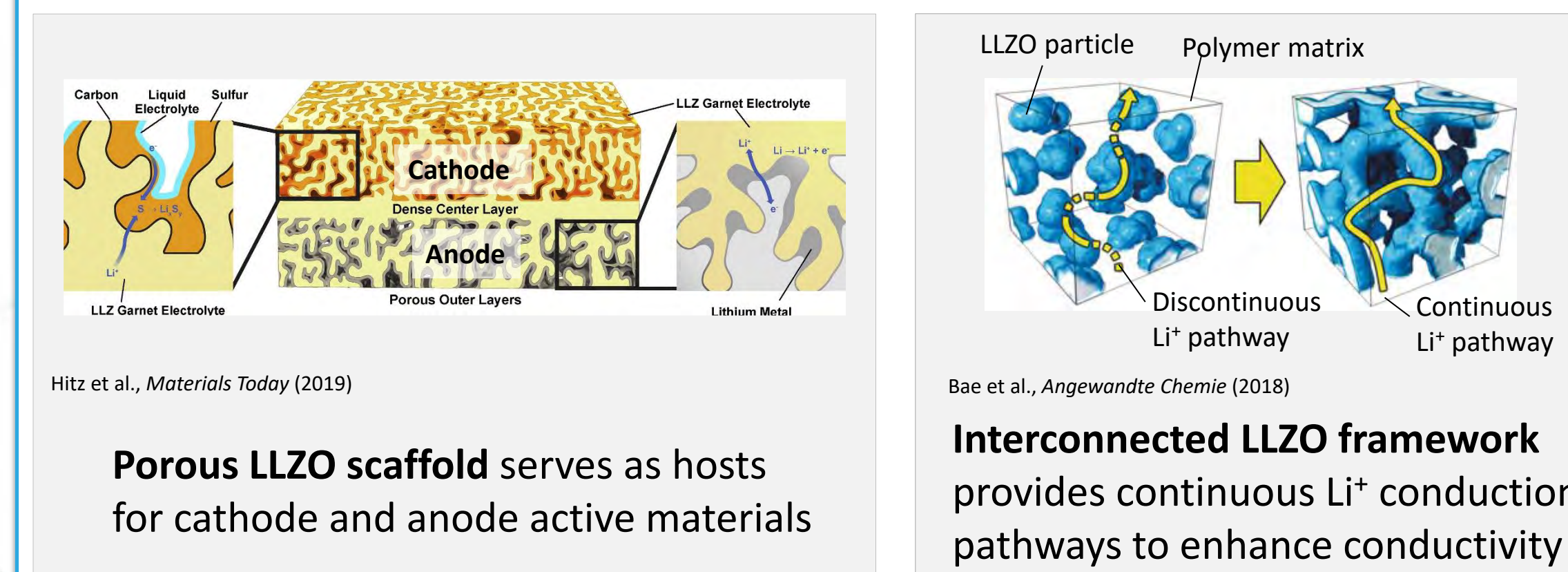


- 3D printing-assisted FAS allows workpieces with controlled mass and dimension to be loaded into FAS \rightarrow sub-mm thickness & dense LLZO layers achieved

Porous LLZO scaffold: Interconnected pores created by partial sintering

Asa Monson¹, Pete Barnes¹, Corey Efaw¹, Eric Dufek¹
1. Idaho National Laboratory

Application of porous structures in solid-state electrolytes

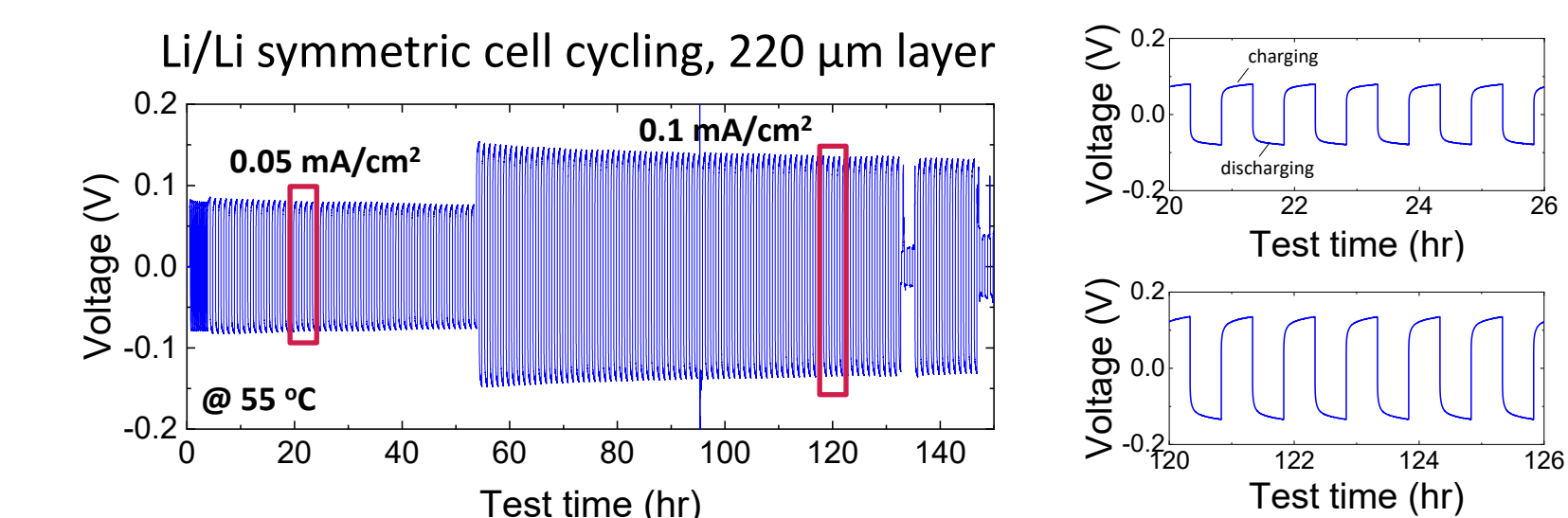
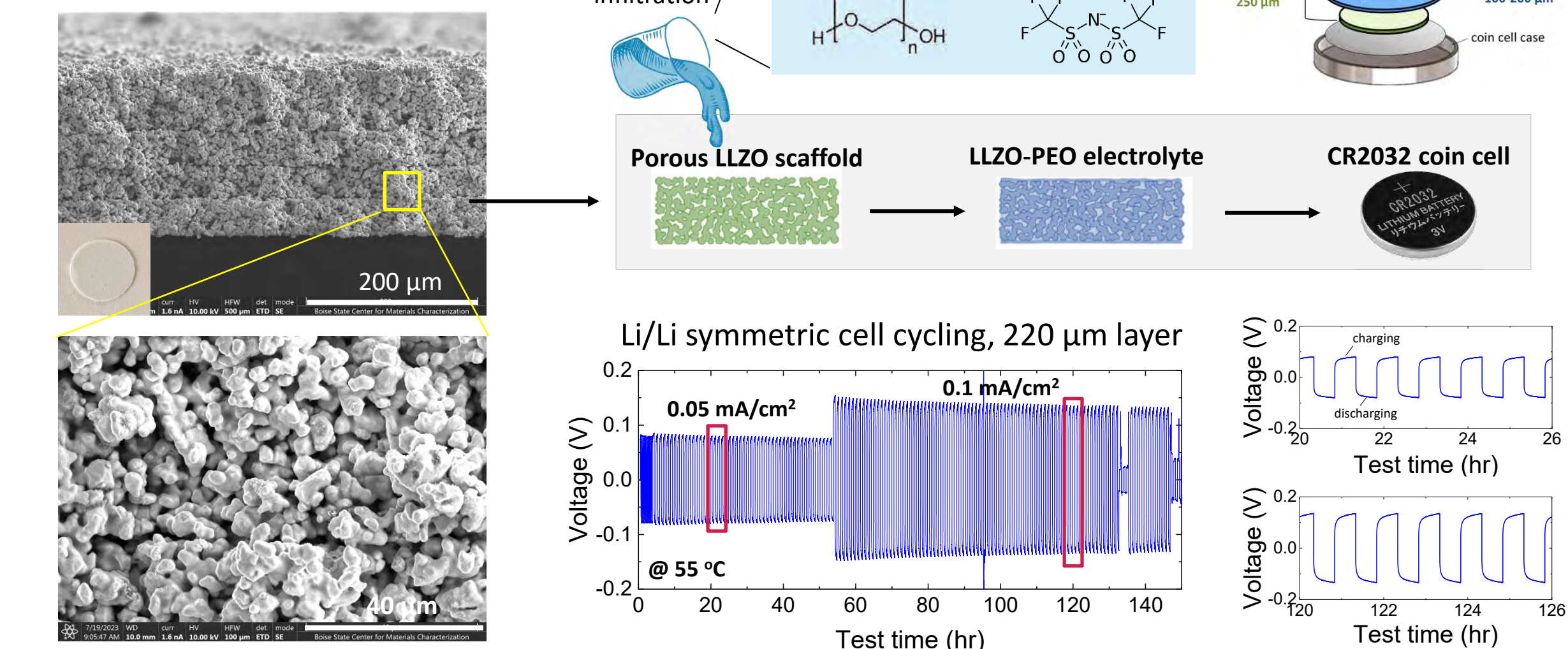


Porous LLZO scaffold serves as hosts for cathode and anode active materials

Interconnected LLZO framework provides continuous Li^+ conduction pathways to enhance conductivity

Printing \rightarrow Partial sintering \rightarrow Porous LLZO scaffold SE

Porous LLZO scaffold created by sintering printed LLZO at 1100 $^\circ\text{C}$, 10 min in conventional furnace

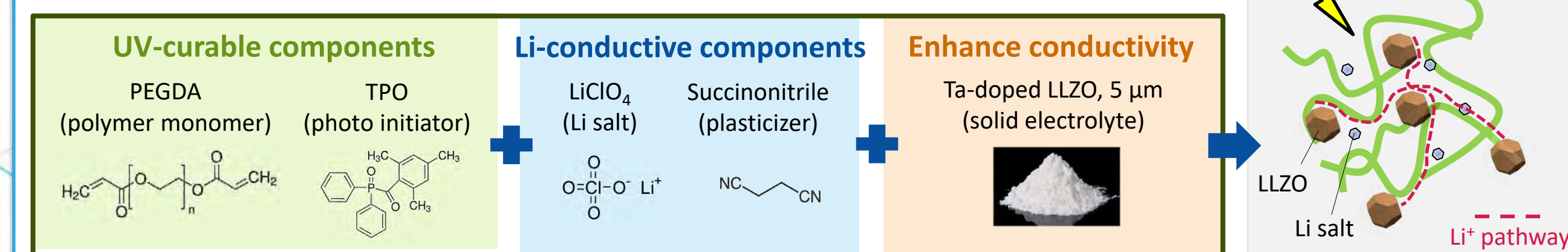


3D printable solid polymer electrolyte: Without the need of heat treatment after printing



Asa Monson¹, Alexis Maurel², Ana Martinez²
1. Idaho National Laboratory 2. The University of Texas at El Paso

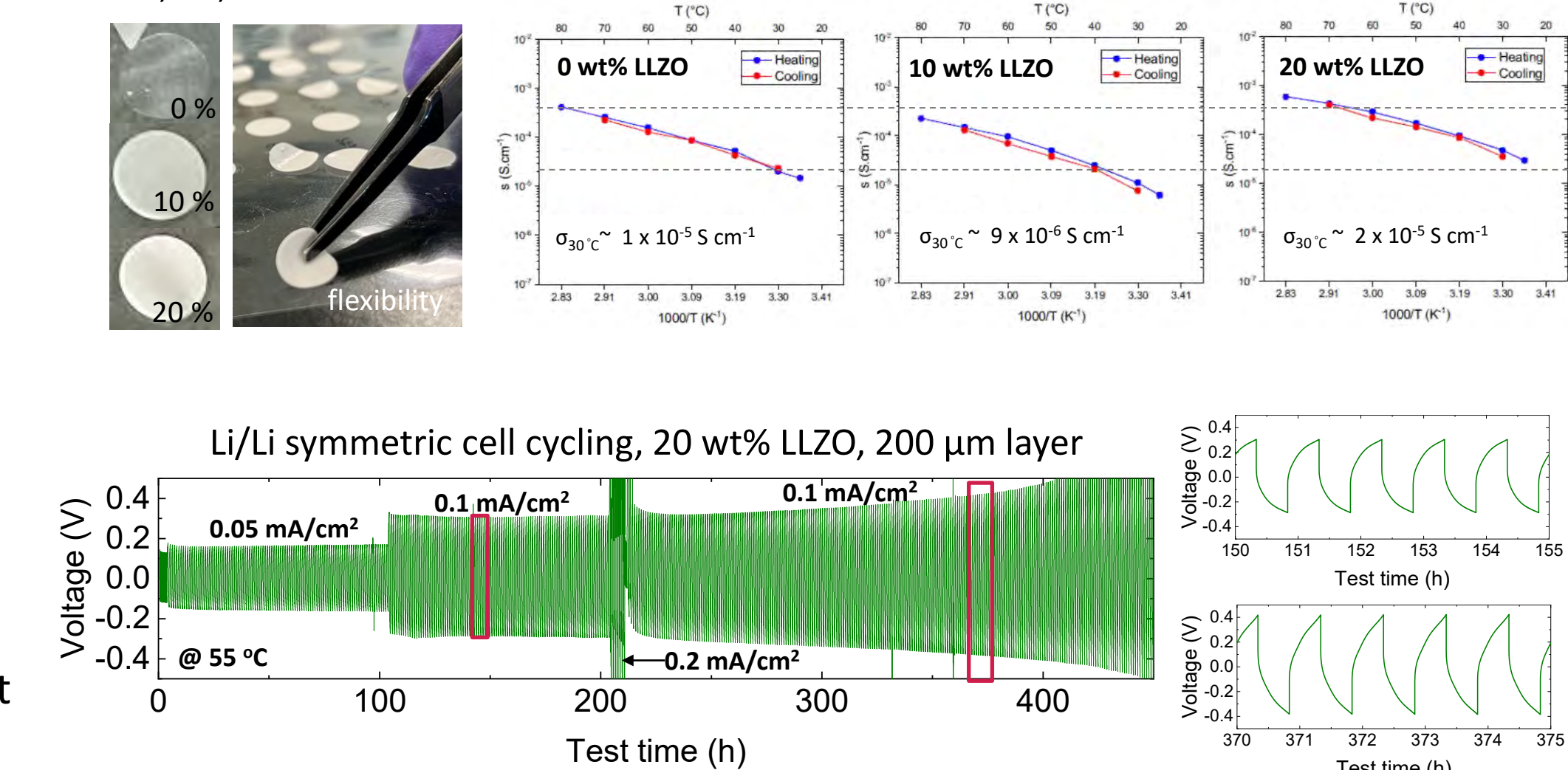
UV-curable & Li-conductive printing resin



- Adding Li salt to allow the printing resin to have both Li conductivity and UV curability
- Prints directly serve as SE, without the need of resin removal or high temperature heat treatment
- Polymer-base offers flexibility; LLZO enhances the conductivity; 3D printing allows freedom in shape design

Printing \rightarrow Solid polymer LLZO SE

SE layers 100-200 μm thick with 0, 10, and 20 wt% LLZO



Publications: 1 review article (*Energy Storage Materials*, 2022 (IF=20.8)), 2-3 journal articles (*in prep*)
Conferences: 1 oral presentation (*MRS Spring 2023*), 1 accepted abstract (*MRS Fall 2023*)

Project Number: 22A1059-039FP

LRS Number: INL/CON-23-74193

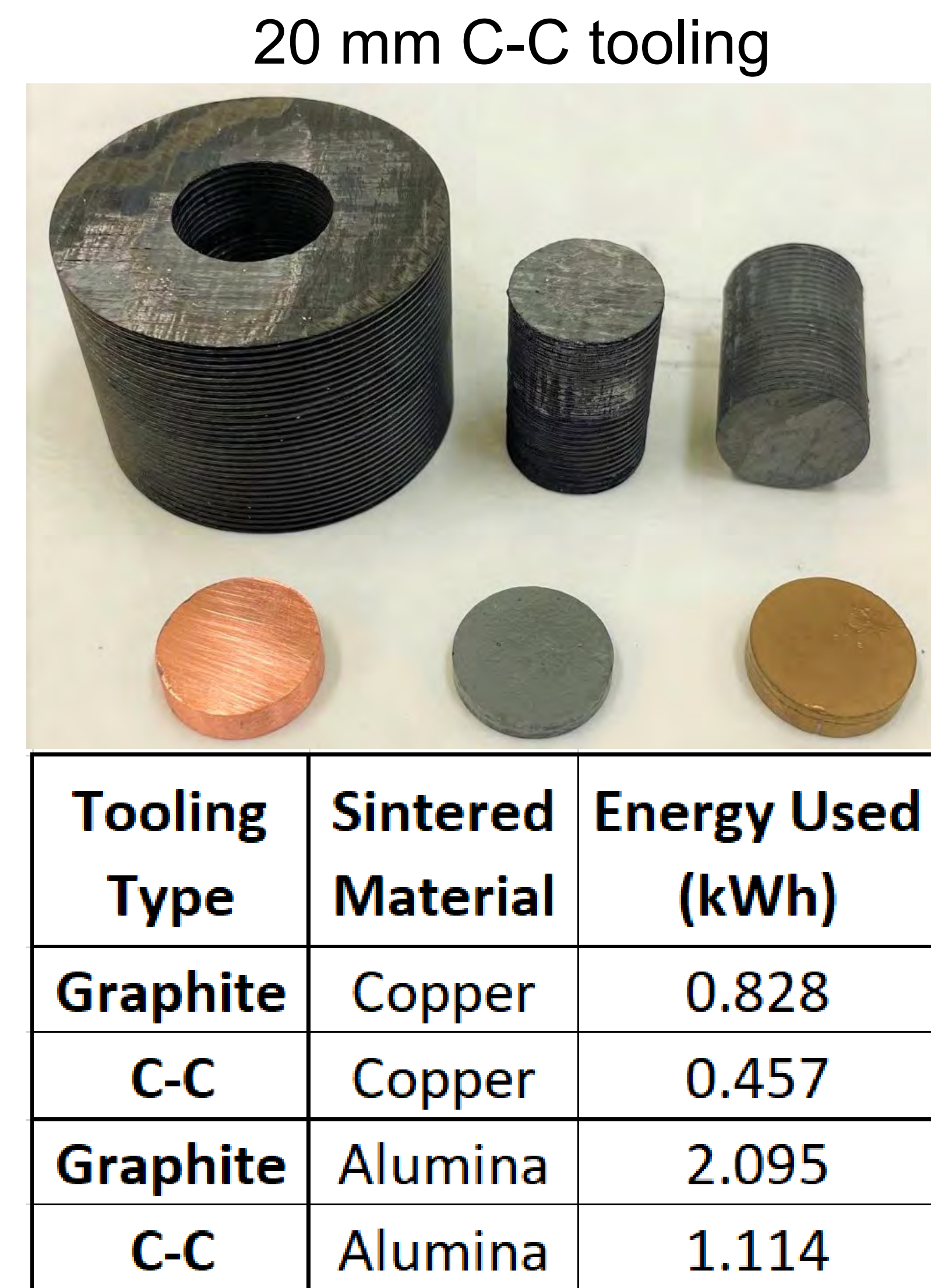
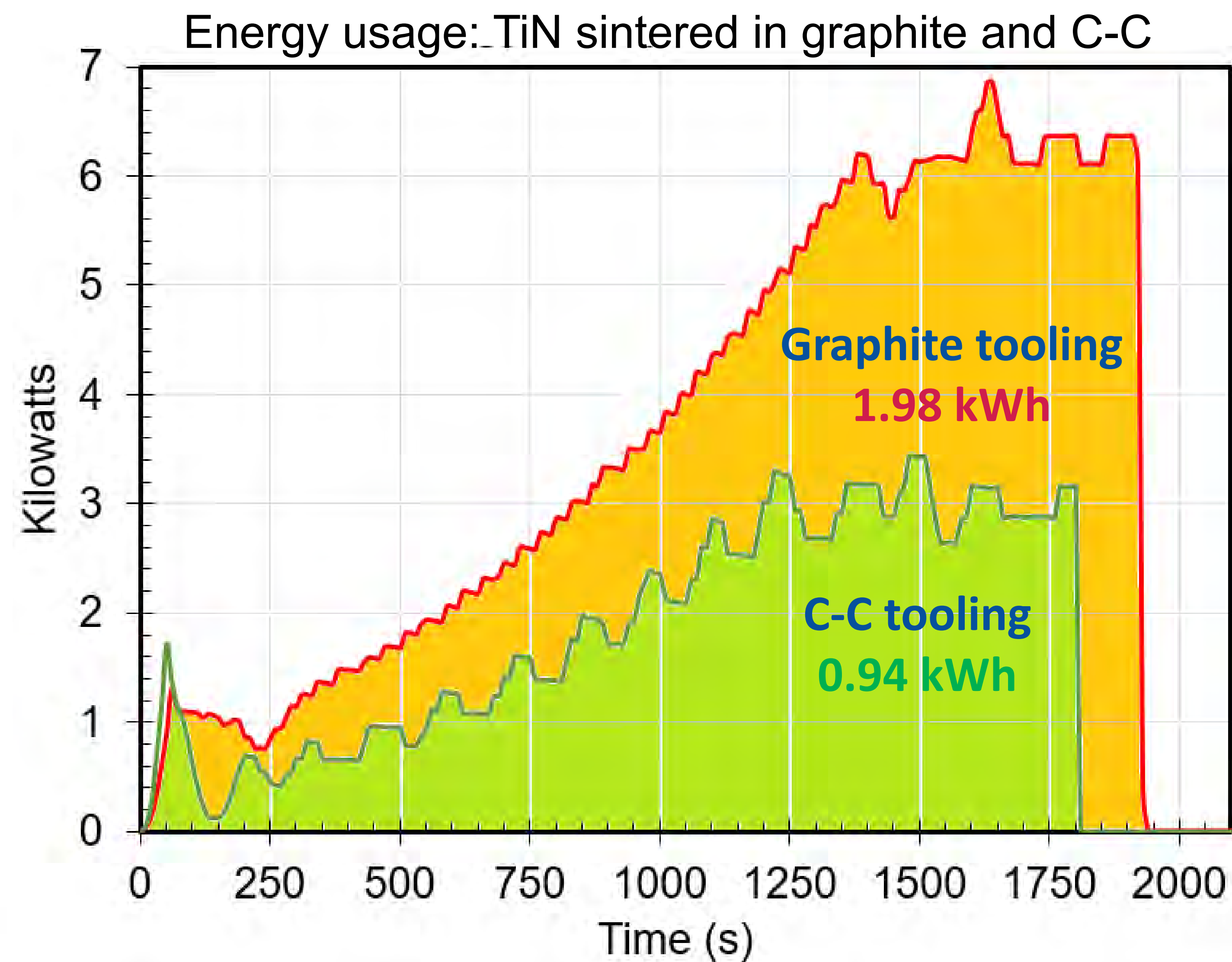
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Work supported through the INL Laboratory Directed Research & Development (LDRD) Program under DOE Idaho Operations Office Contract DE-AC07-05ID14517

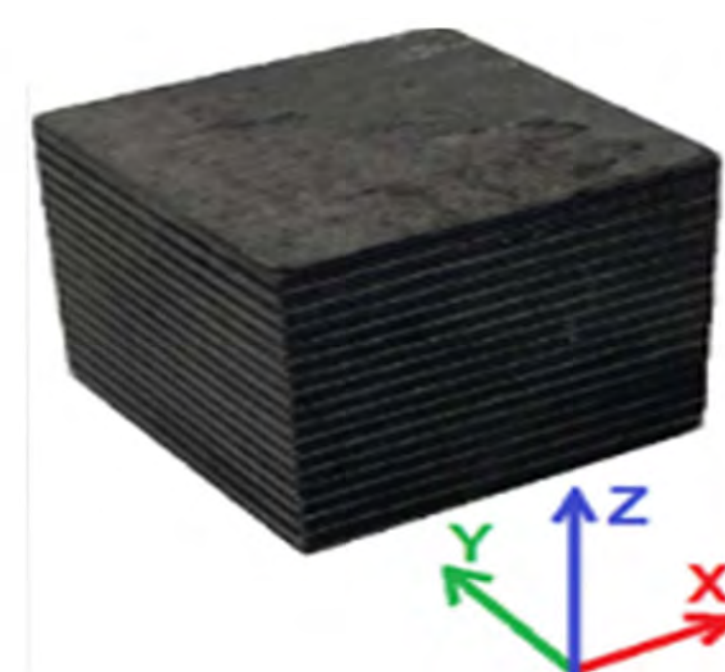
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Carbon-Carbon composites: stronger and more energy efficient EFAS tooling.

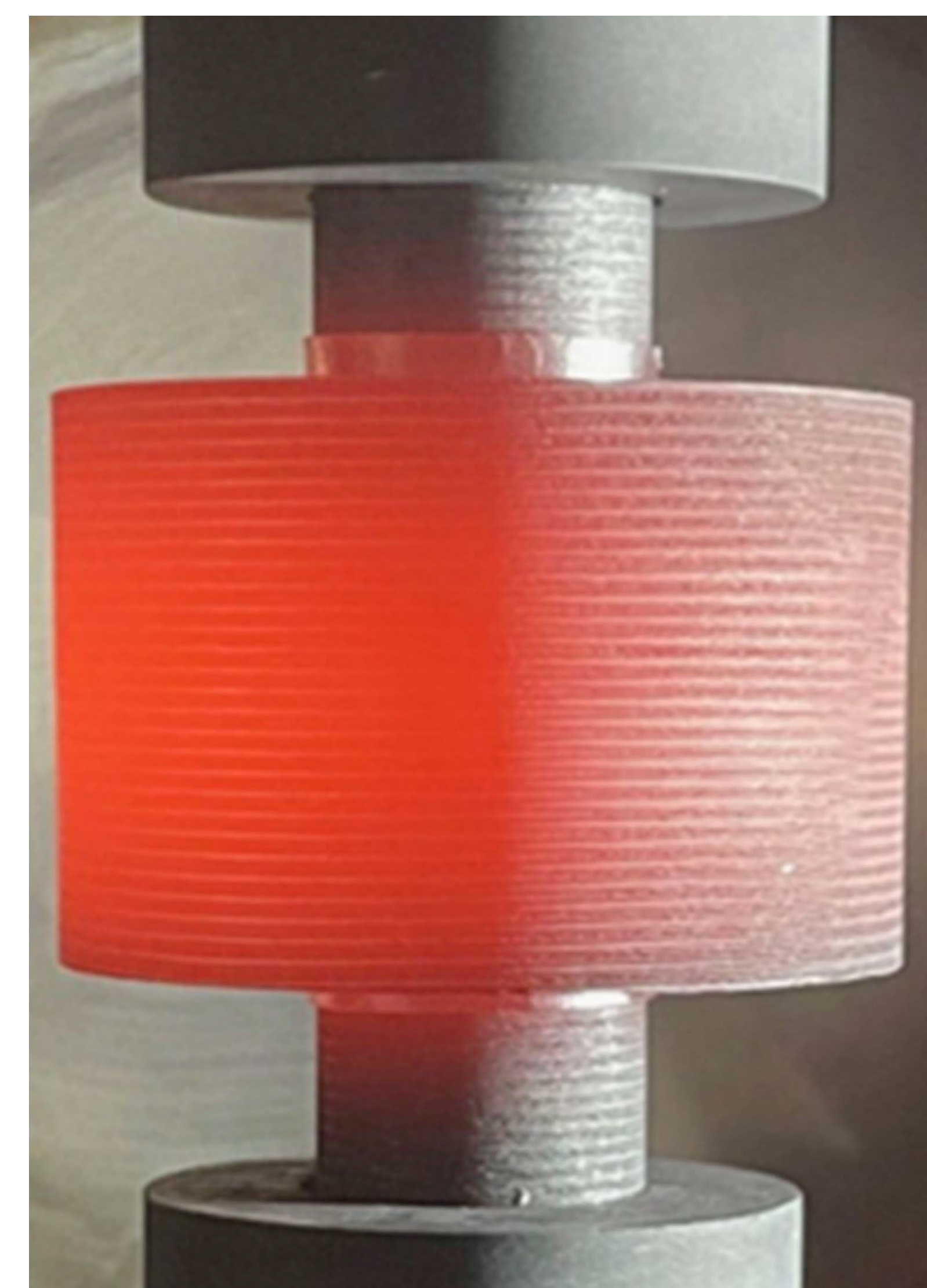


- Carbon-carbon composites were manufactured at INL from 3D printed carbon fiber preforms using a proprietary process.
- High density C-C, comparable or better than “premium” commercially available material, was produced.
- Continuous fiber printing, which is novel to INL’s method, enables tailorable anisotropic material properties.
- Tooling for electric field assisted sintering (EFAS) was made from the anisotropic C-C material and evaluated as an energy efficient alternative to traditional graphite tooling.



Anisotropic Properties:	X/Y	Z
Electrical Resistivity ($\mu\Omega\text{m}$)	16.5	121.2
Thermal Diffusivity (mm^2/s)	75	5

- High resistivity in the Z-direction enables **more efficient Joule heating**.
- Low thermal diffusivity in the Z-direction means the **heat does not conduct away** as quickly; heat is effectively "trapped" where it is needed.
- 3D printed **C-C tooling is stronger**, and **more energy efficient** than identical graphite tooling.
- Compared to Tokai G535 graphite:
 - At least **2x stronger** in tension.
 - At least **3x stronger** in compression.
 - Uses **48% less energy**, 35% lower ram temps



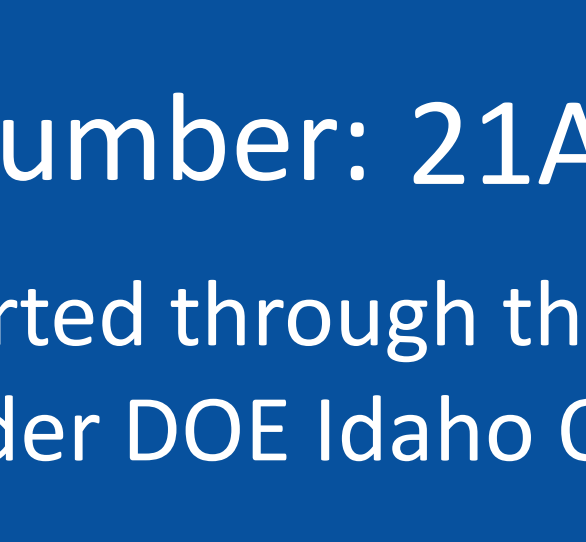
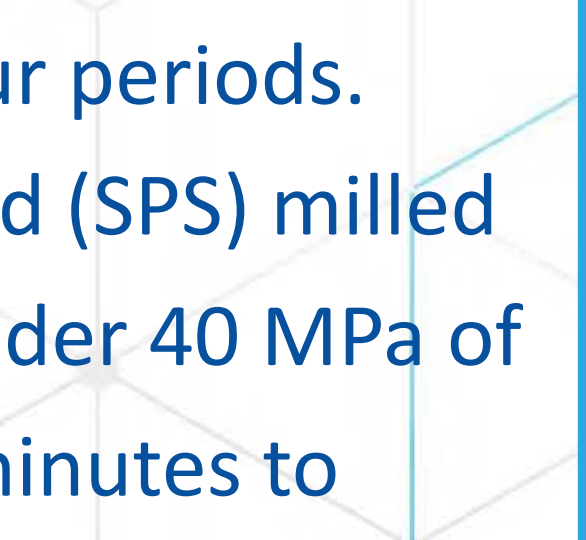
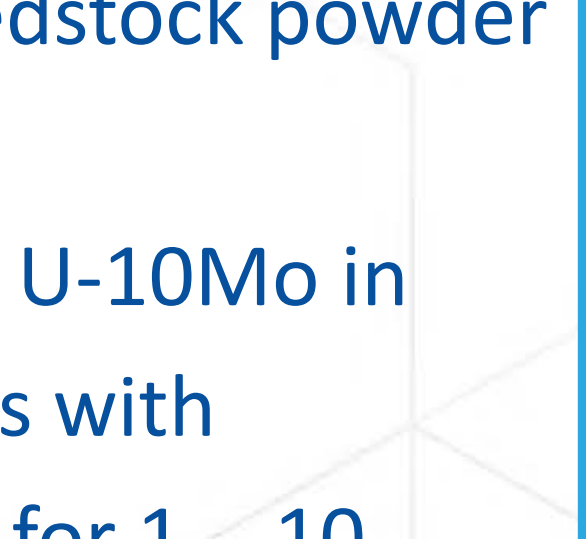
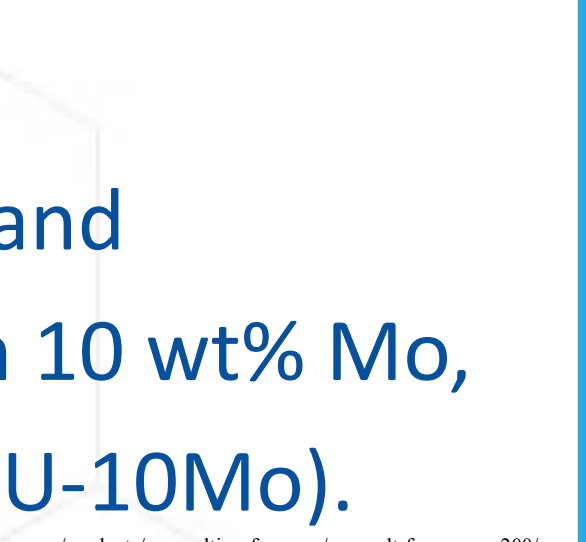
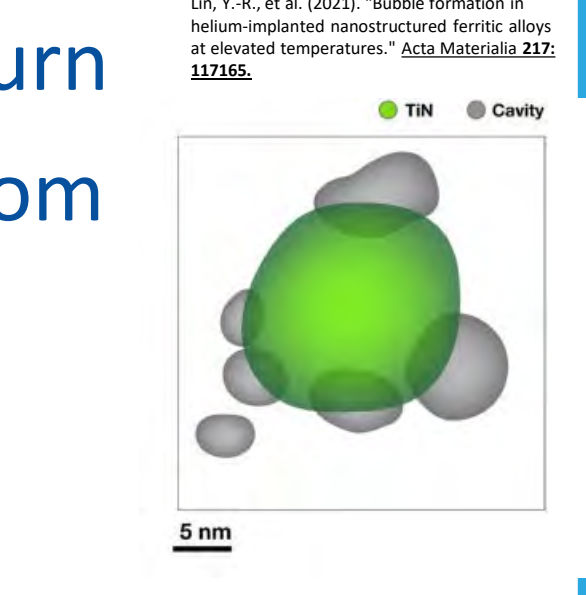
Jorgen Rufner, Arin Preston, Robert Fox, Josh Kane, Troy Holland.

Project Number: 21A1050-096FP

LRS Number: INL/MIS-23-74205

Nanostructuring of Uranium Based Metallic Fuels via Spark Plasma Sintering

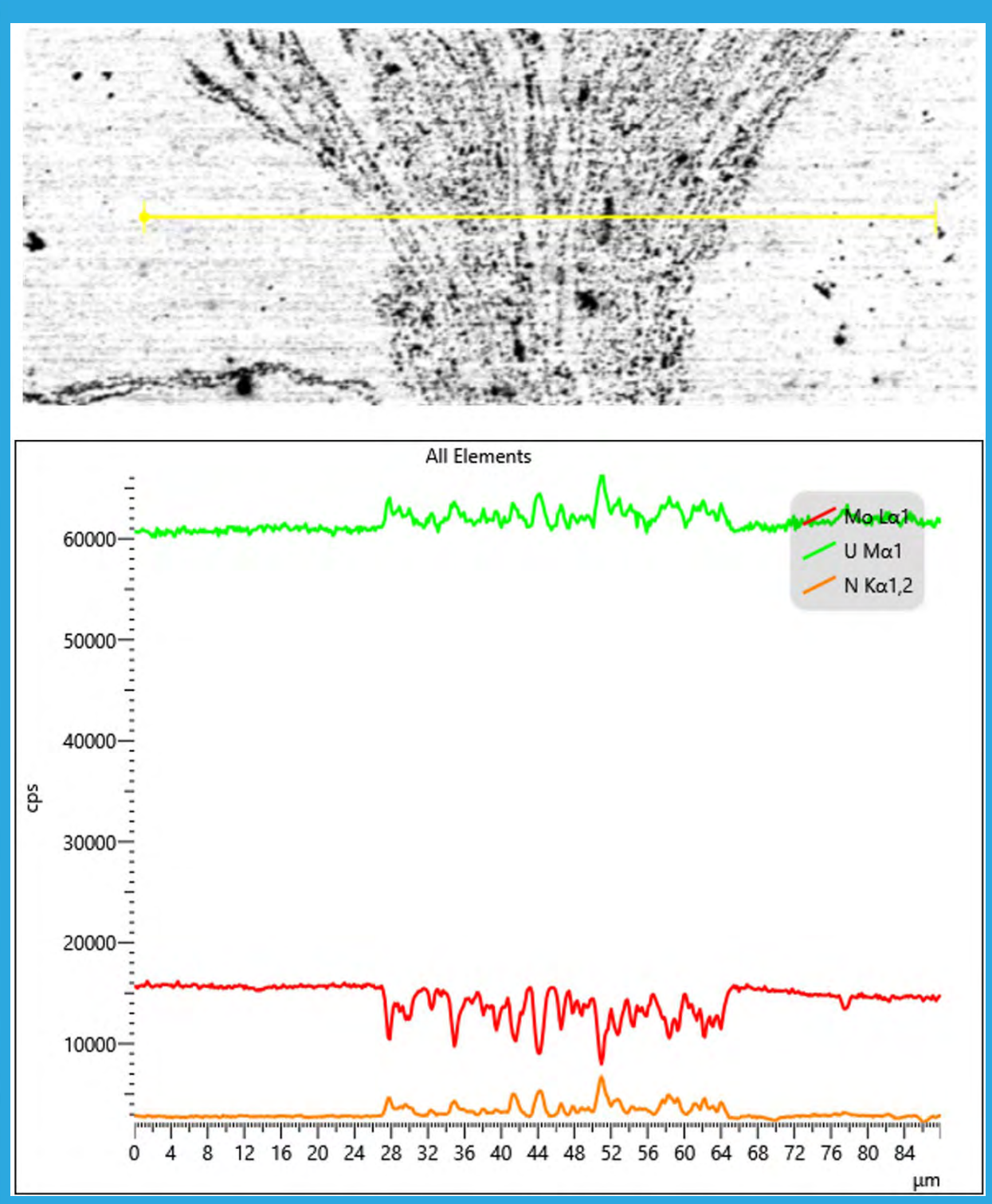
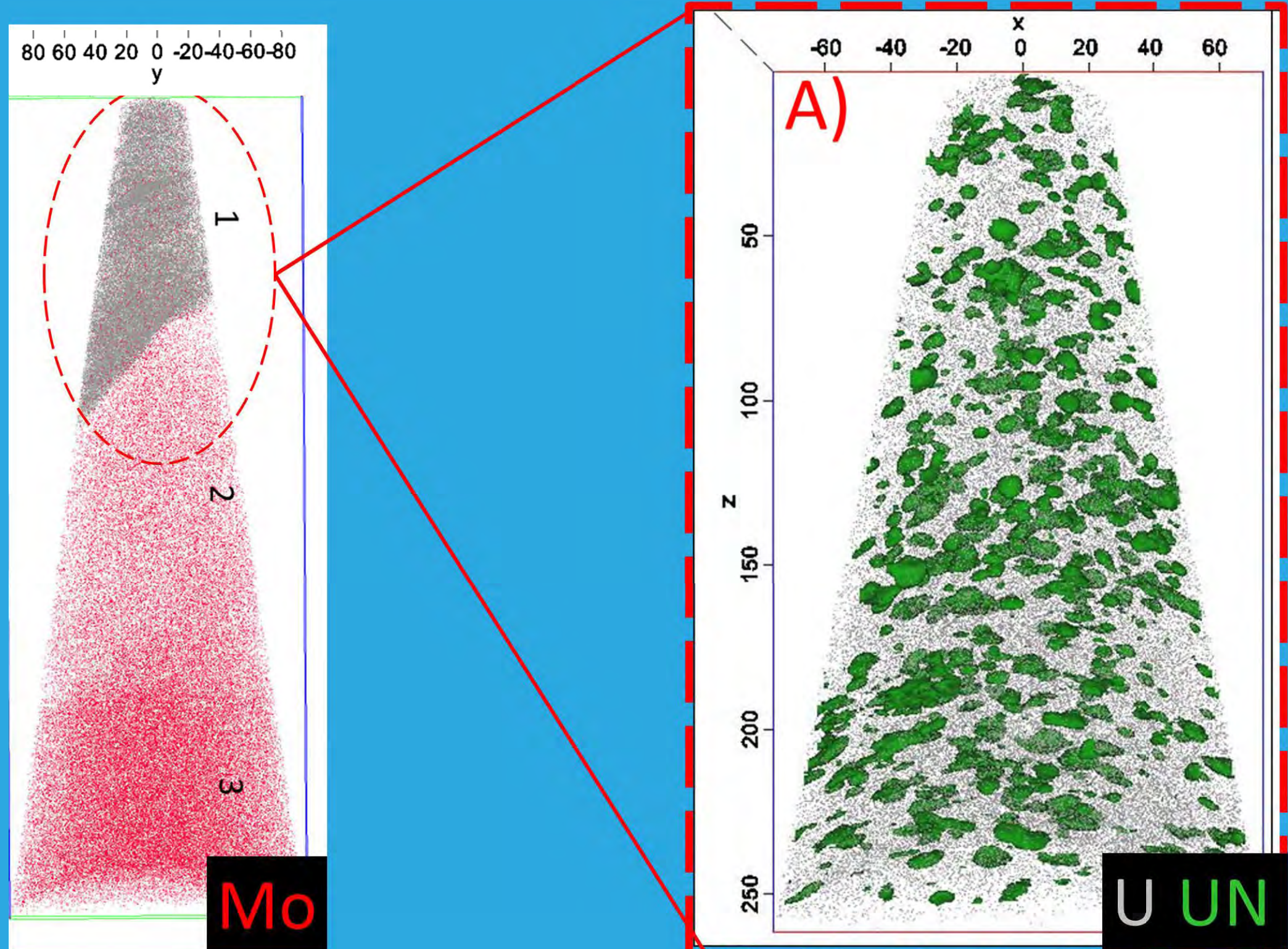
Nathan Jerred
James Zillinger



UN NANOSTRUCTURES (1-5 nm) formed in U-10Mo via mechanical alloying could GREATLY EXTEND FUEL LIFE by inhibiting fuel/cladding interactions!

BACKGROUND: Metallic fuels struggle with irradiation induced swelling and chemical interaction at fuel/cladding interfaces. Our study aimed at forming UN nanostructures homogenously across the fuel volume that would act as defect sinks for fission products, in turn reducing fuel/cladding interaction from solid fission products and reduce swelling due to void formation from gaseous fission products.

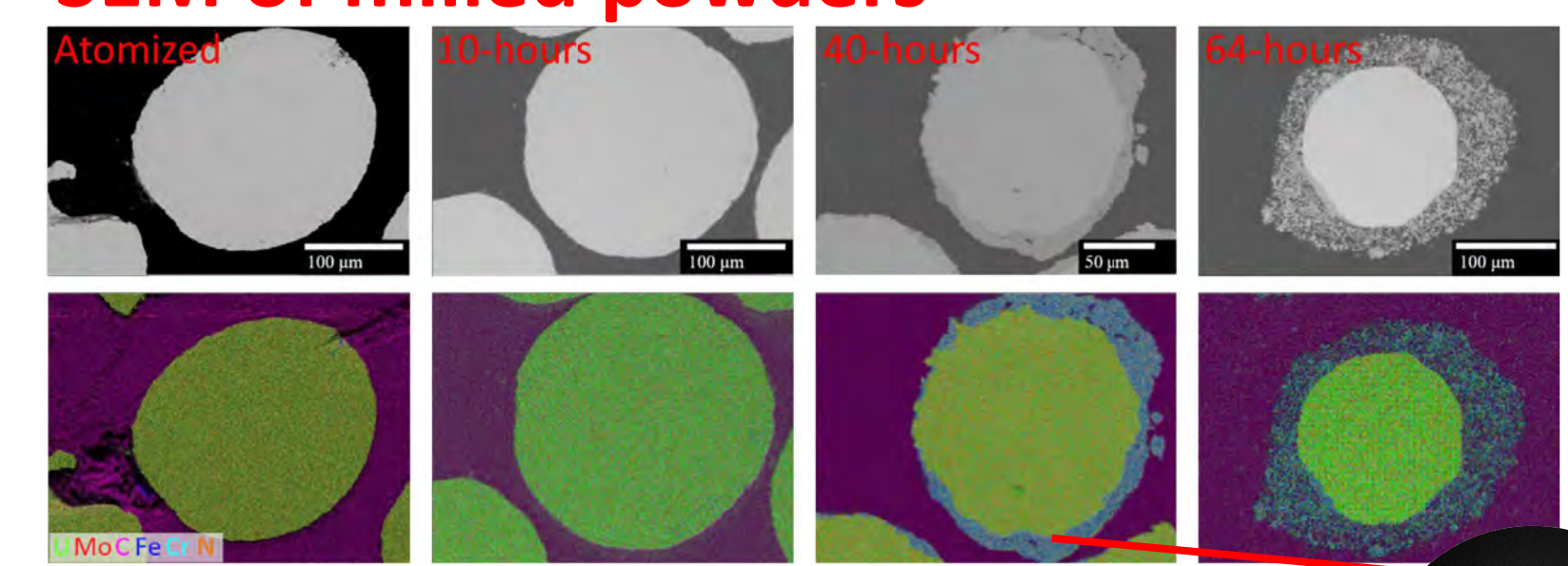
- METHODS:**
1. Arc-melted uranium and molybdenum to form 10 wt% Mo, 90 wt% U feedstock (U-10Mo).
 2. Atomized to form feedstock powder (~190 μm diameter).
 3. Mechanically alloyed U-10Mo in 99.9995% pure N₂ gas with stainless-steel media for 1-, 10-, 20-, 40-, and 64-hour periods.
 4. Spark-plasma sintered (SPS) milled powder at 900 °C, under 40 MPa of axial pressure for 5 minutes to solidify.
 5. Analyzed alloyed powder and sintered compacts and compared to first principle simulations.



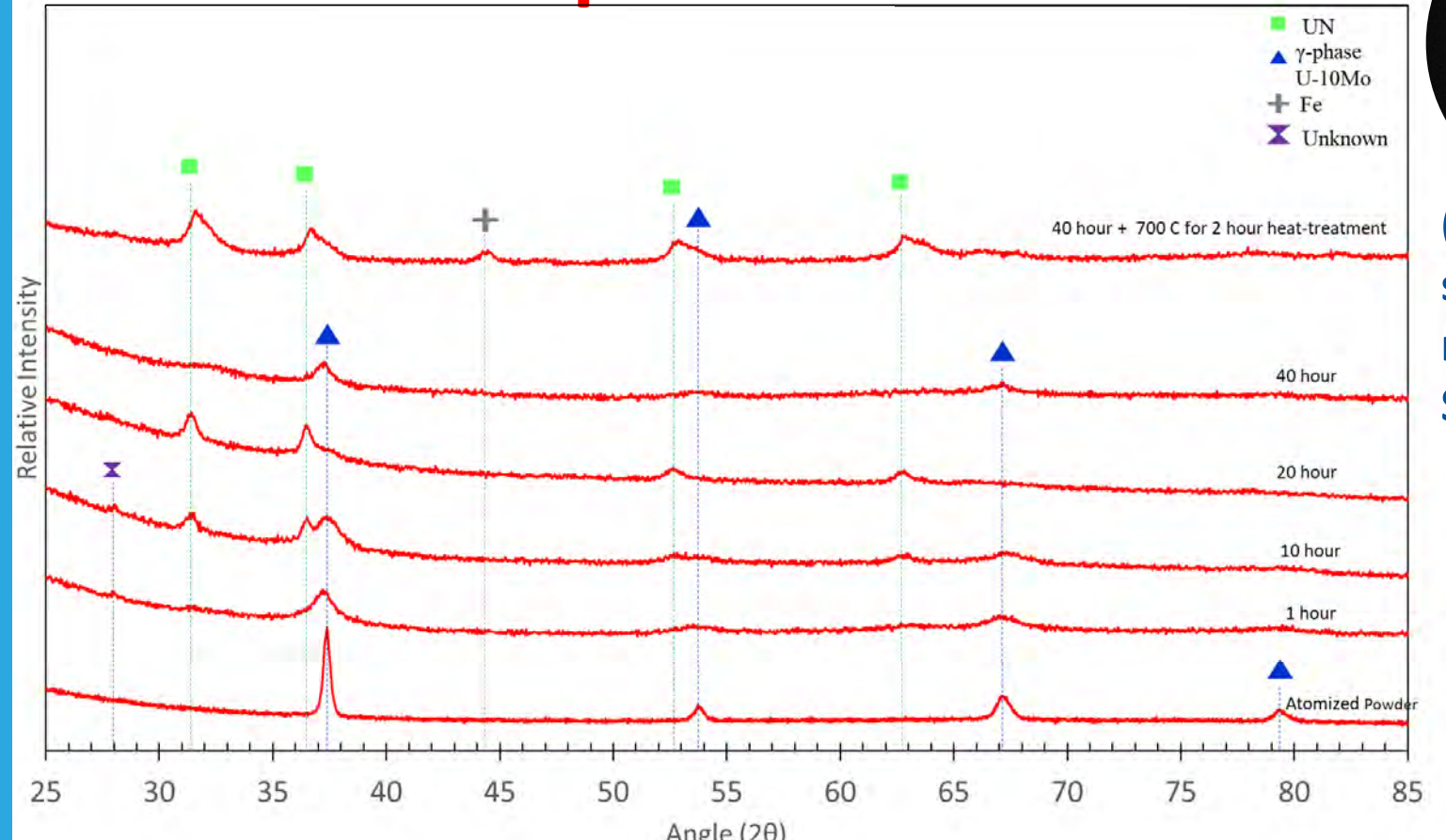
RESULTS AND DISCUSSION:

- Iron deposited ~40 hours of milling

SEM of milled powders



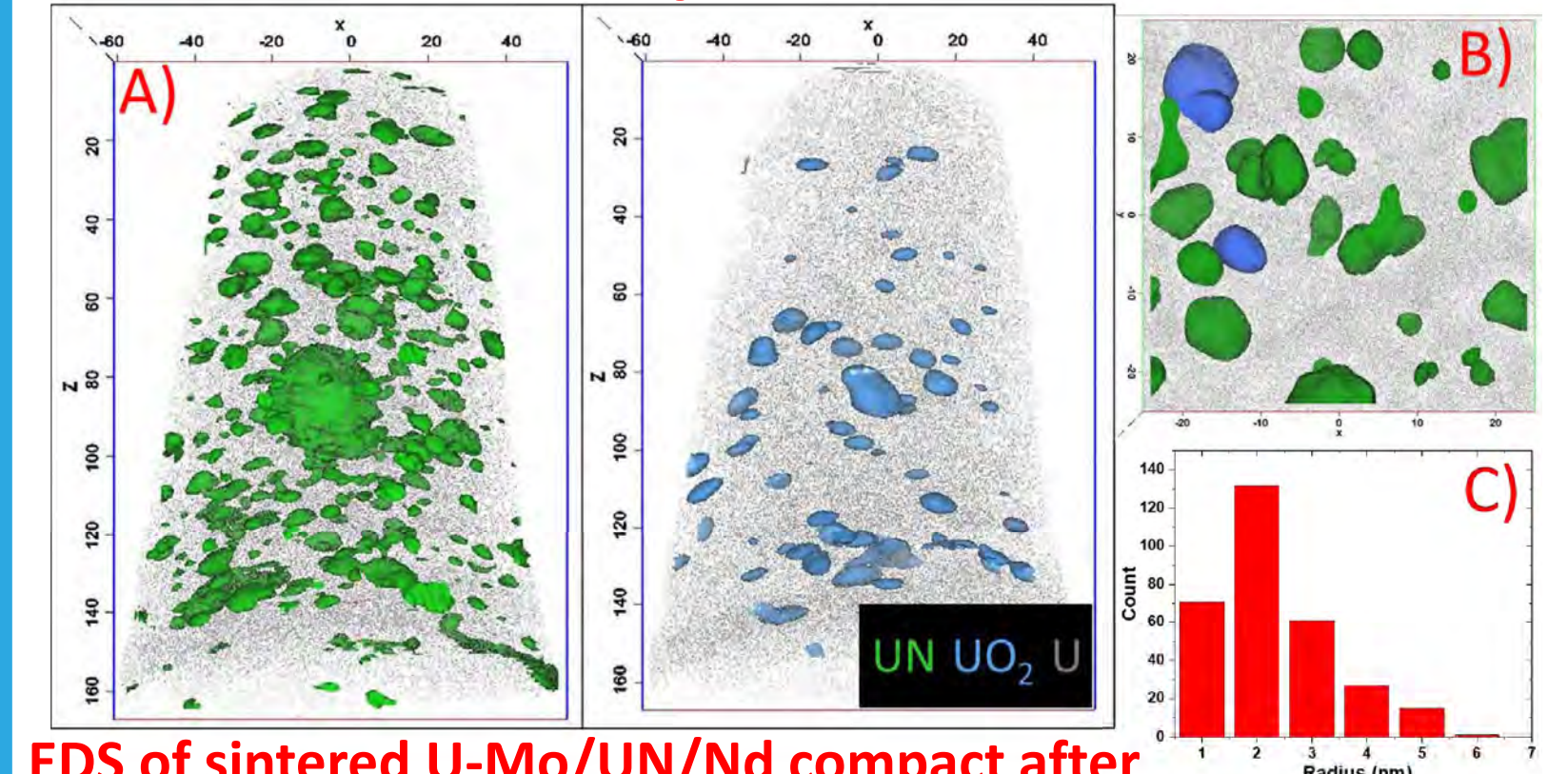
XRD of milled powders



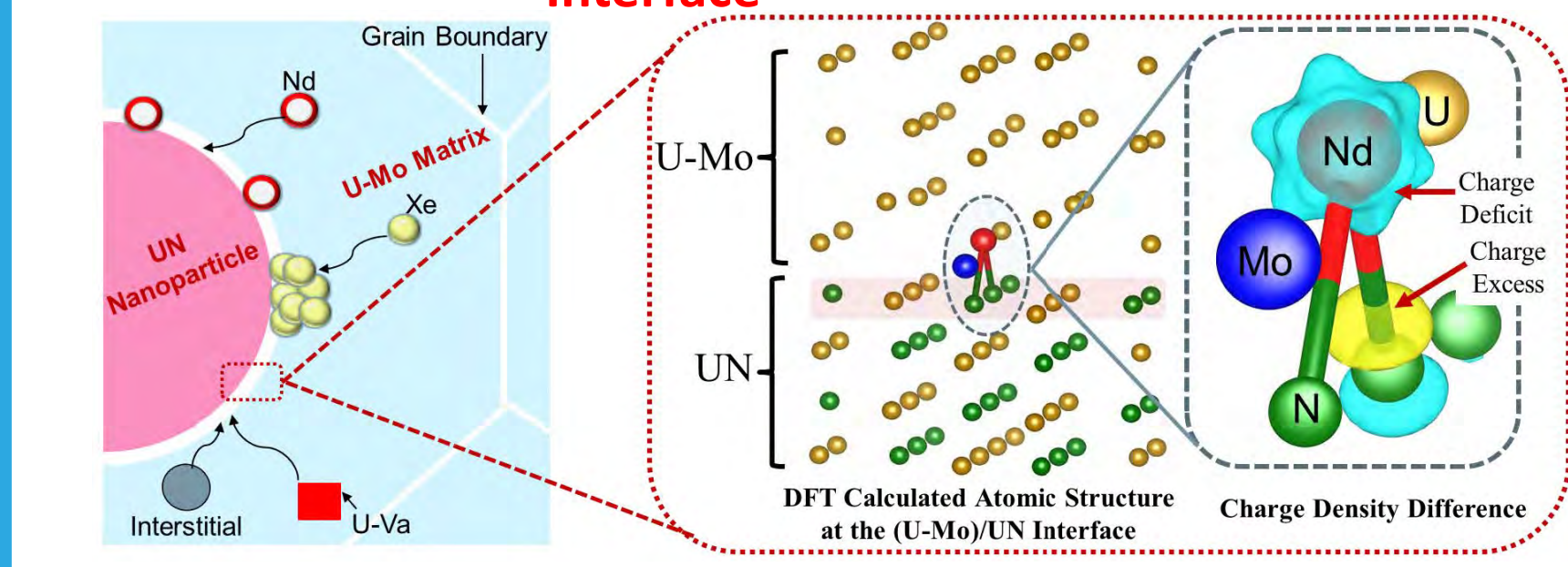
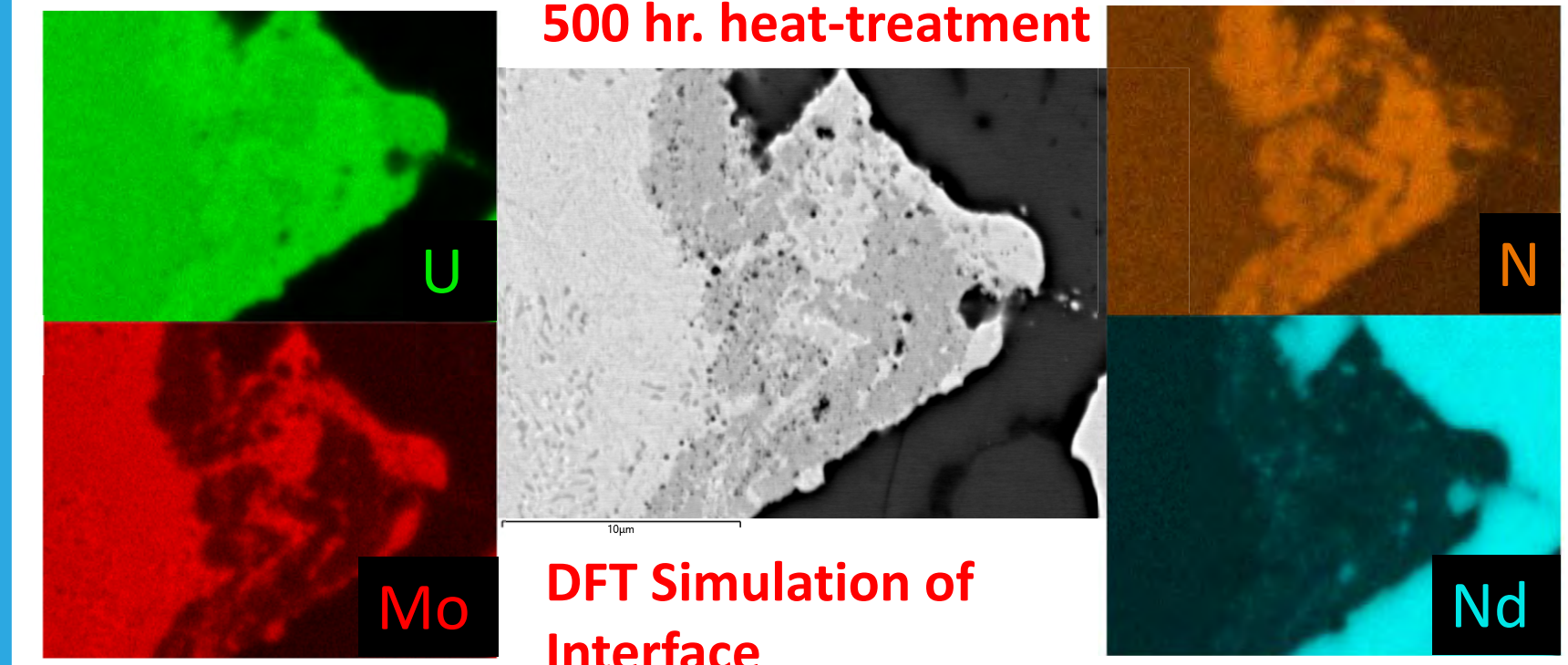
(Amorphous structure revealed by SAED)

- UN observed forming after 10 hours via XRD

APT on 1-hr milled powder



EDS of sintered U-Mo/UN/Nd compact after 500 hr. heat-treatment



James Zillinger, Nathan Jerred, Mukesh Bachhav, Samrat Choudhury, Indrajit Charit

Project Number: 21A1050-128FP

LRS Number: INL/EXP-23-74220

www.inl.gov

Work supported through the INL Laboratory Directed Research & Development (LDRD) Program under DOE Idaho Operations Office Contract DE-AC07-05ID14517."

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Shock Wave Mitigation in Metal Materials Through Advanced Manufacturing Processes

A material texture study

PRESENTER: **Kenneth Bratton**

BACKGROUND: Large-impulse tolerant and shock mitigating materials have many potential applications including armor, structures, space-faring asset protection and vibration damping in heavy industry vehicles. This project will create strategically oriented microstructures allowing the attenuation/dissipation of shock waves in the material. The key objective is to understand what forming processes, and associated processing parameters influence microstructure, specifically the crystallographic orientation, to become oriented favorably to dissipate shock wave energy or guide shock wave propagation in a harmless direction through the material.

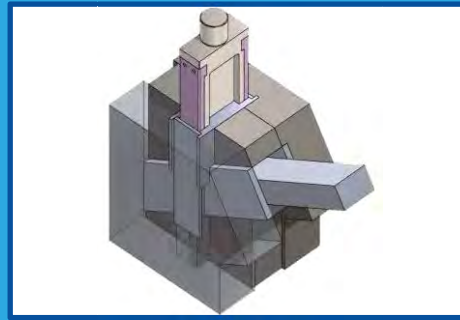
METHODS

1. Process Stock Oxygen Free High Conductivity Copper and 304 stainless steel through Equal Channel Angular Extrusion
2. Section processed bars for Orientation Imaging Microscopy and artificial intelligence image segmentation
3. Section processed and stock bars for Split Hopkinson Pressure Bar dynamic testing

RESULTS

- A new die design has been developed that can process 2- and 3-inch-wide tiles.
- An in-house artificial intelligence texture analysis tool has been developed
- A novel way of testing samples utilizing a Split Hopkinson Pressure Bar and Photon Doppler Velocimetry has been tested.

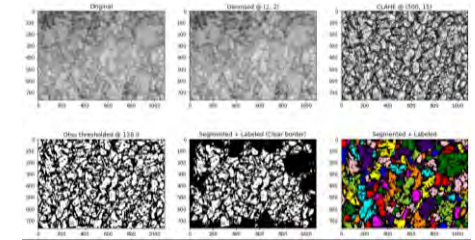
A key accomplishment of this project was the development of the new plate die design. The new design allows for the fabrication of a new die that will produce ECAE processed tiles as opposed to bars.



At the current processing level, 2A, there was no discernible difference in wave speed in stock and processed materials.

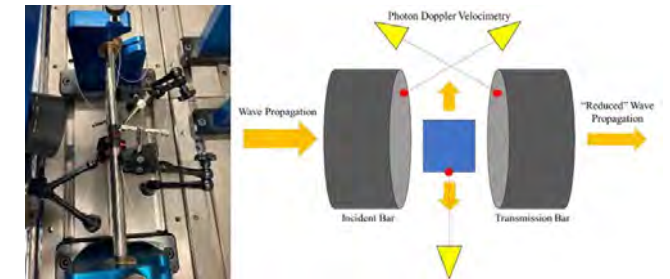
Artificial Intelligence Texture Tool

An in-house artificial intelligence algorithm was created to distinguish levels of texture disruption as well as count the number of grains present within each sample. This tool uses algorithms to deduce texture boundaries within a discretized image to isolate and identify grains within the image. The colored image is the final output of the tool culminating the processing of the tool to ultimately identify the grains located in the samples.



Split Hopkinson Pressure Bar and Photon Doppler Velocimetry Combined Testing

A novel method of testing the dynamic response on a material was developed during this project. This methodology can measure incoming, exiting, and normal velocities coming out of the sample. This is crucial for measuring these parameters



• Kenneth Bratton, Brady Aydelotte, Thomas Lillo, Zherui Guo

Project Number: 21A1050-101FP

LRS Number: INL/MIS-23-74645

Understanding of Spark Plasma Sintering at Different Length Scale

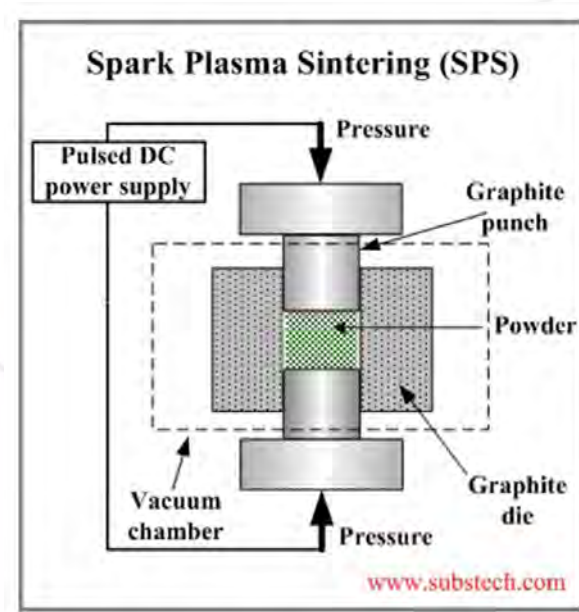
PRESENTER

Tiankai (TK) Yao

Background

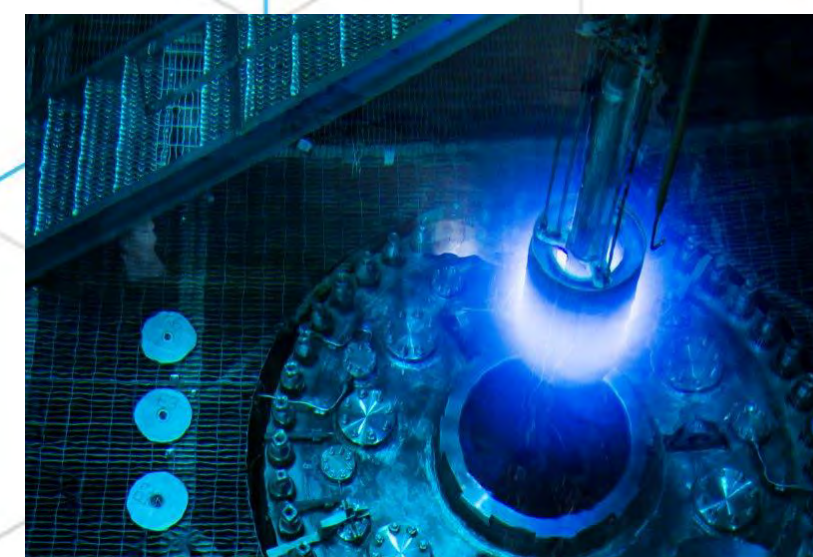
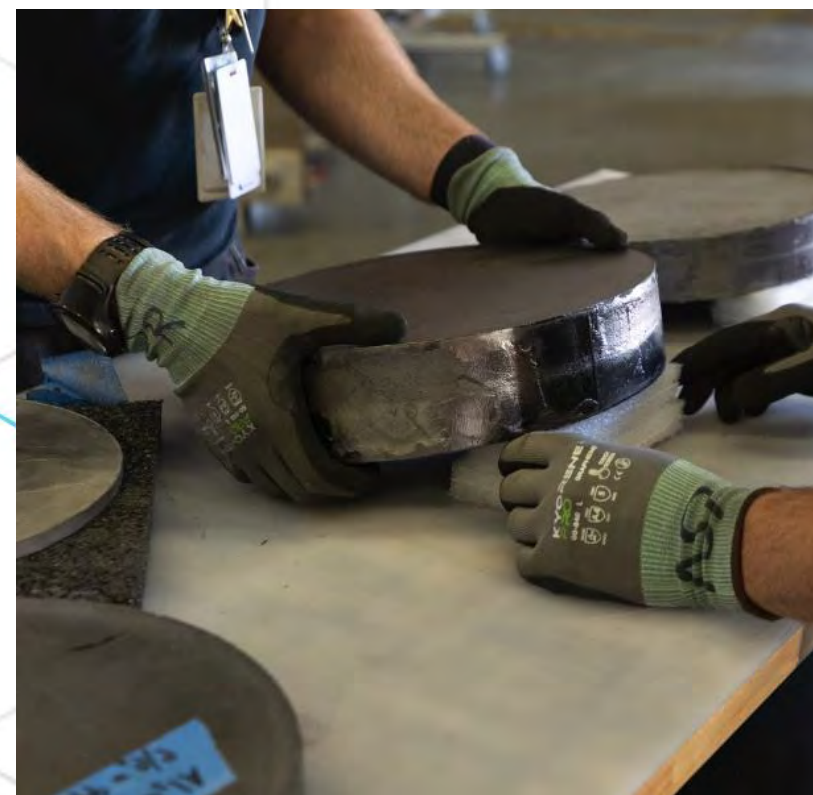
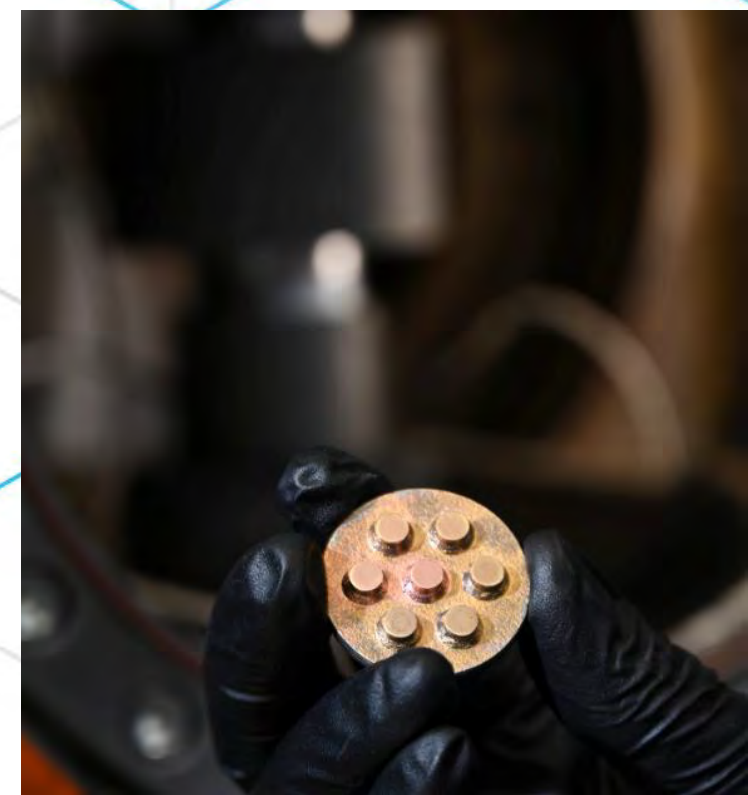
Spark plasma sintering uses a high-intensity, low voltage, pulsed current simultaneously with uniaxial pressure to achieve a fast consolidation of powder into solid component in seconds and minutes. The rapid densification can lead to high levels of residual stress during part scale up if appropriate processing methods are not maintained. This project uses a combination of synchrotron and neutron beam imaging and diffraction technique to provide knowledge and data for MALAMUTE, a modeling application for advanced manufacturing process.

Methods

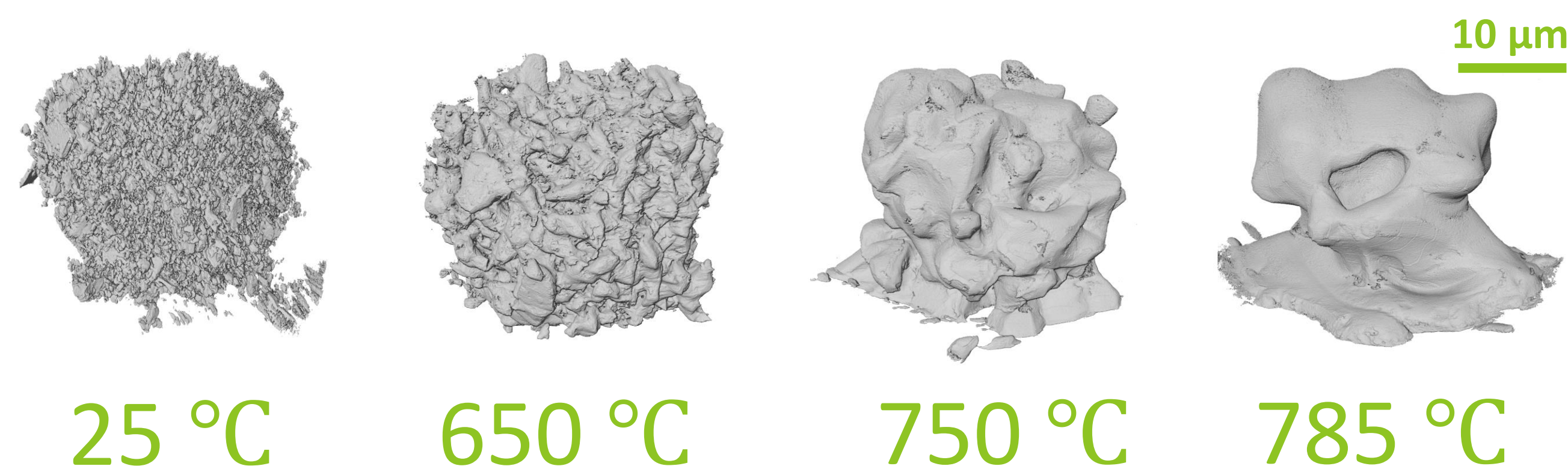


$\mu\text{m-mm}$

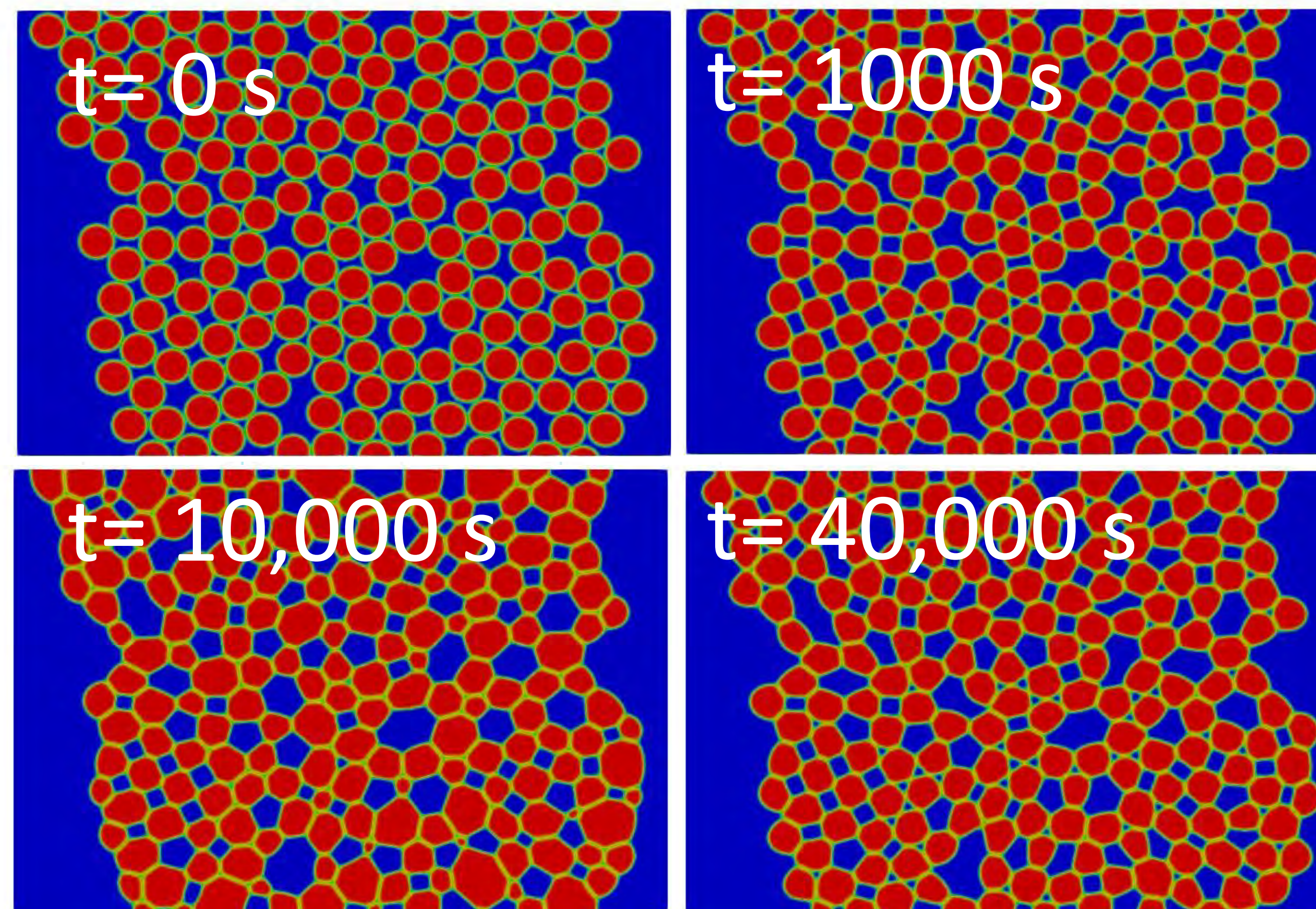
Inch



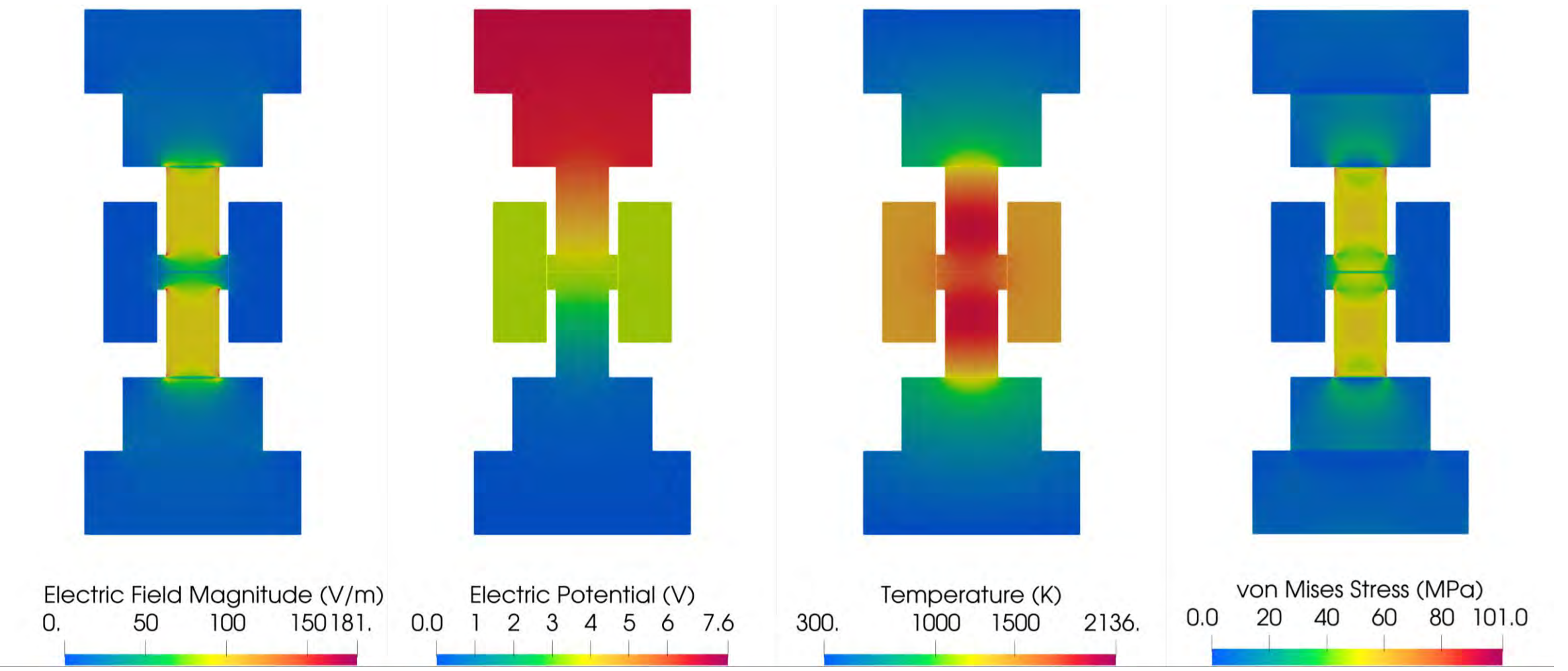
Sintering and Densification for Ceramic Apatite is revealed by in-situ nano X-ray Computed Tomography
(POC: rahulreddy.kancharla@inl.gov)



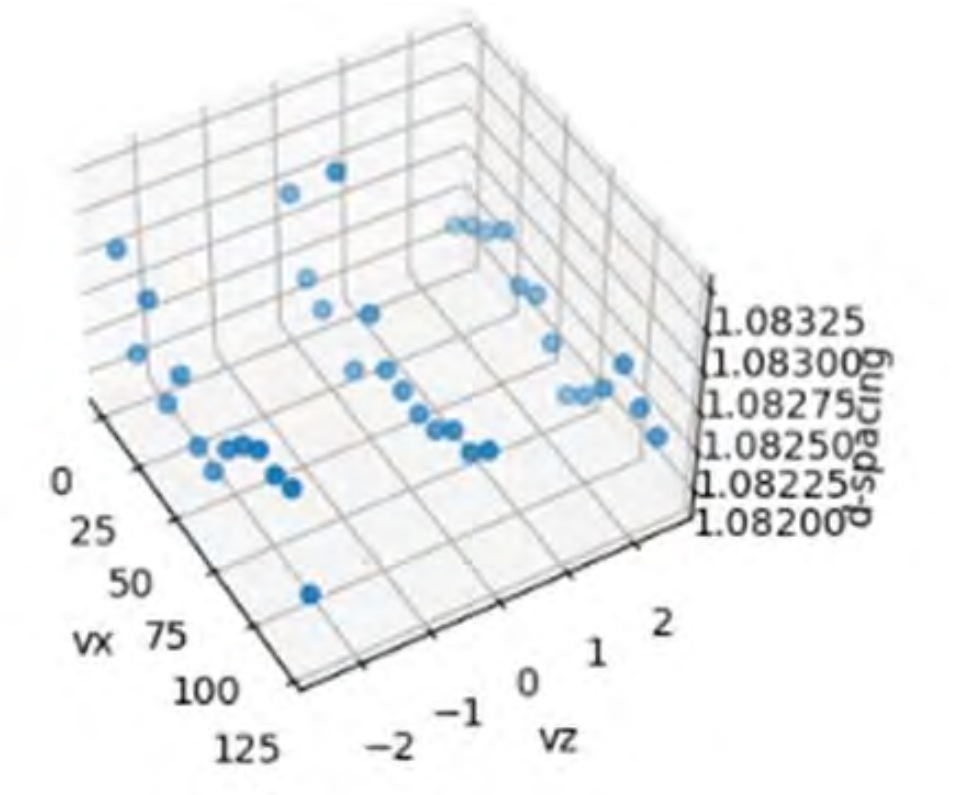
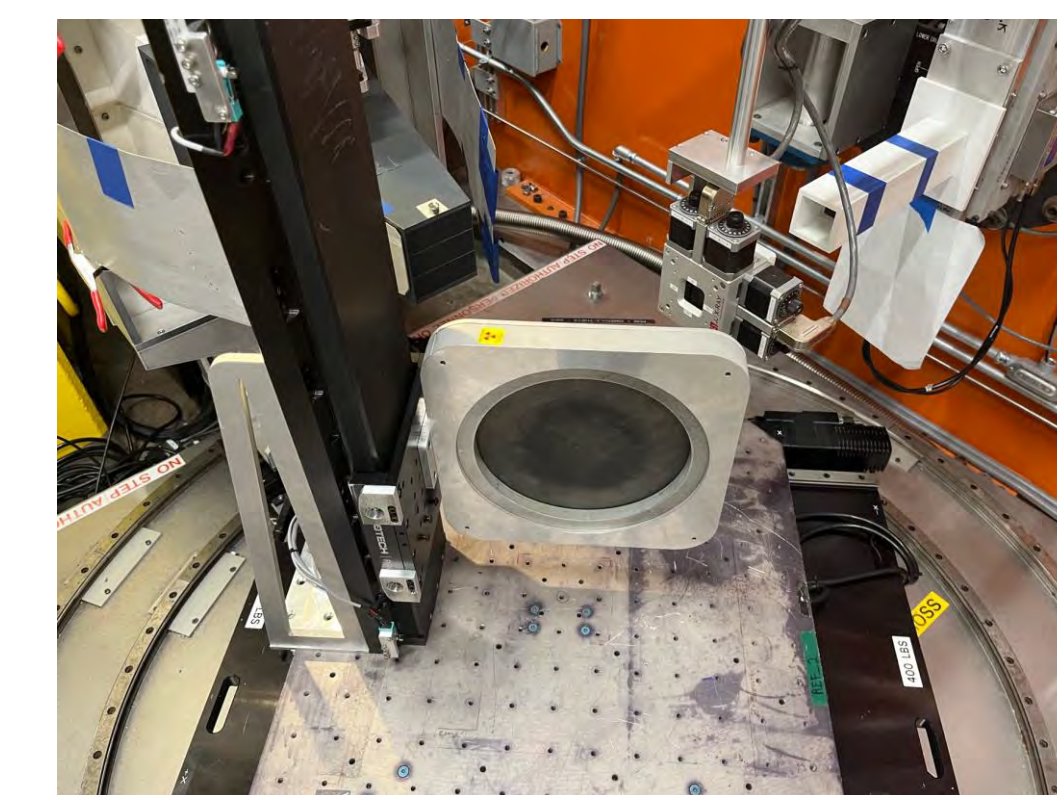
Phase-field simulation of microstructural evolution of many-particle system with applied electric field of 1000 V/m
(POC: larry.aagesen@inl.gov)



Spark Plasma Sintering is simulated by engineering scale by MALAMUTE
(POC: stephanie.pitts@inl.gov)

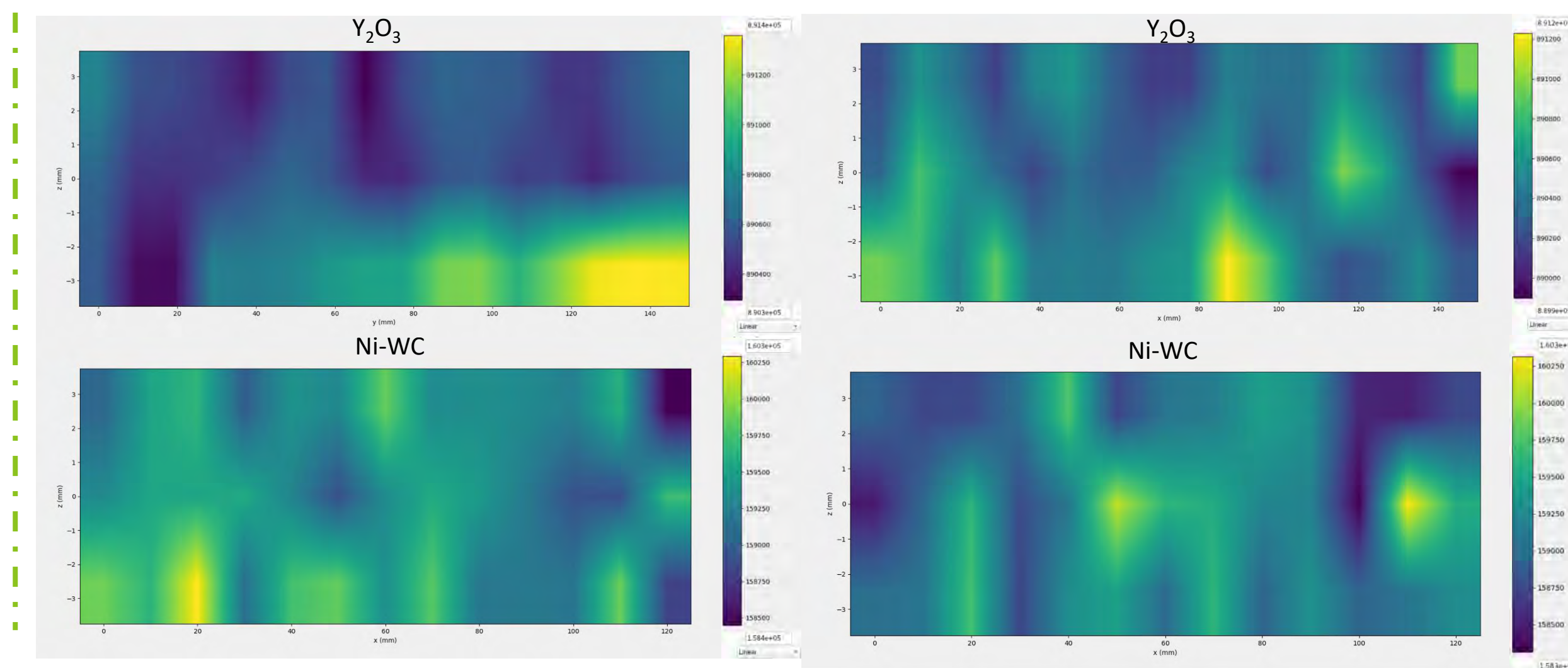


Residual strain measurement on the 12-inch samples at HFIR
(POC:jorgen.rufner@inl.gov)



Hoop

Radial



Project Number: 21A1050-075FP

LRS Number: INL/CON-23-74281

www.inl.gov

Work supported through the INL Laboratory Directed Research & Development (LDRD) Program under DOE Idaho Operations Office Contract DE-AC07-05ID14517."

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Enhanced Diffusion Welding via EFAS to Fabricate Compact Heat Exchangers

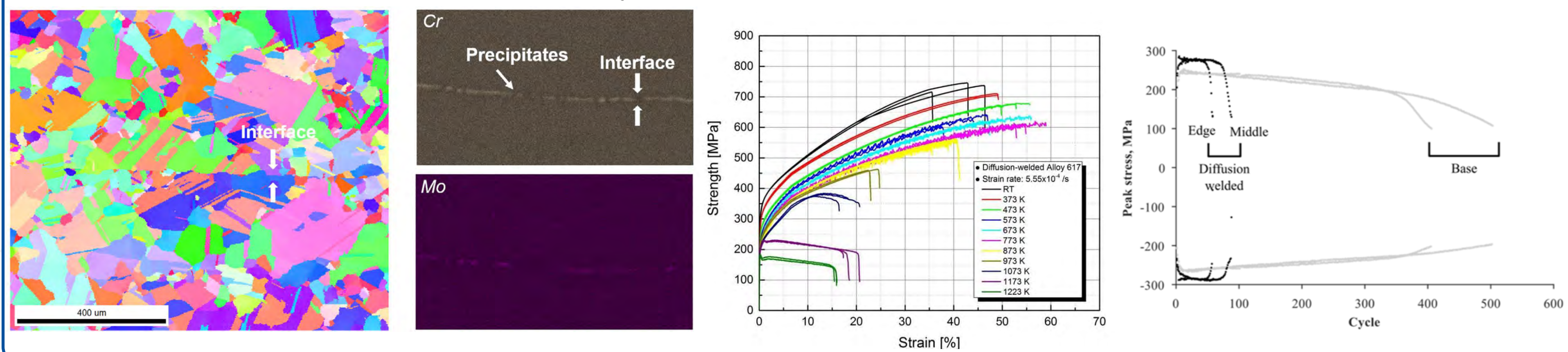
Xinchang Zhang, Michael D. McMurtrey, Tate Patterson, Andrew Gorman, Ryann Rupp, Jorgen Rufner

Objective

- To fabricate Alloy 617 diffusion-welded plates that meet ASME BPVC Section IX requirements and have the same elevated-temperature mechanical properties as the base metal found in the ASME BPVC Section III, Division 5 Code Case

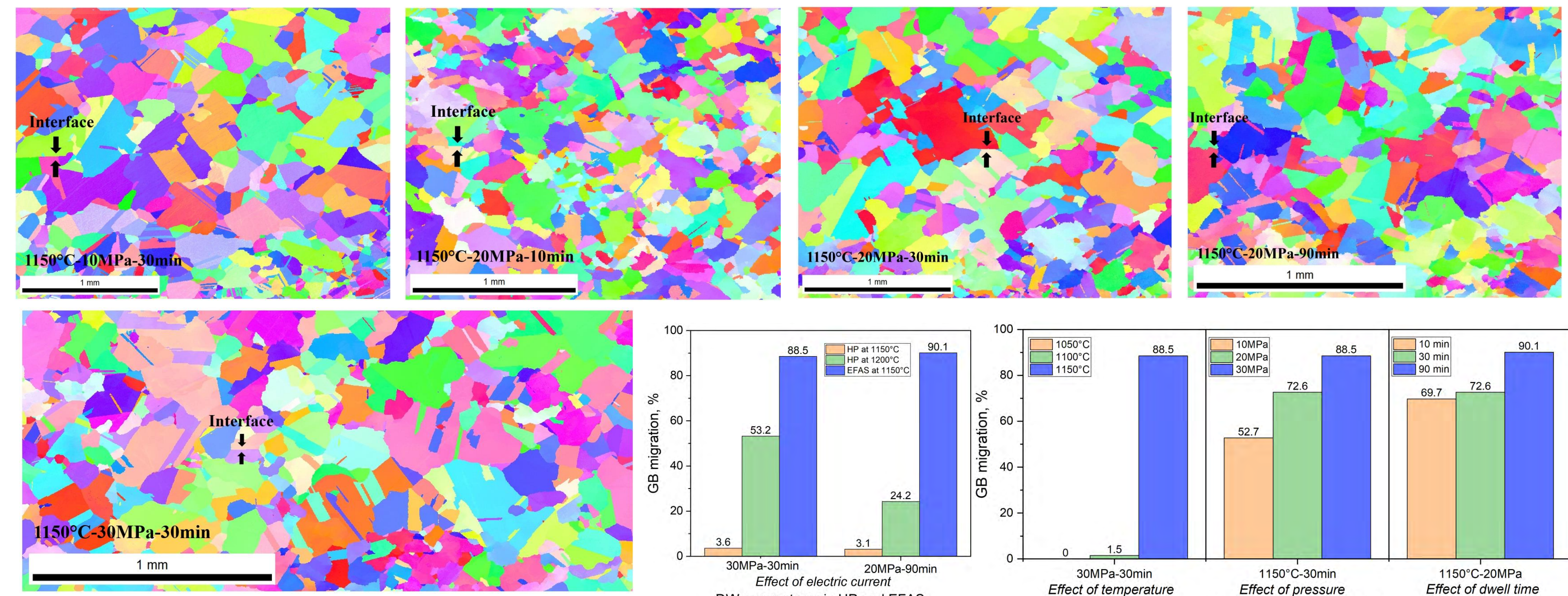
Prior Literature

- Endeavors to diffusion-weld Alloy 617 by hot pressing have been hindered by precipitation at the interface of the contacting surfaces
- Limited grain boundary migration across the interface significantly reduced mechanical properties of diffusion-welded Alloy 617 at elevated temperatures

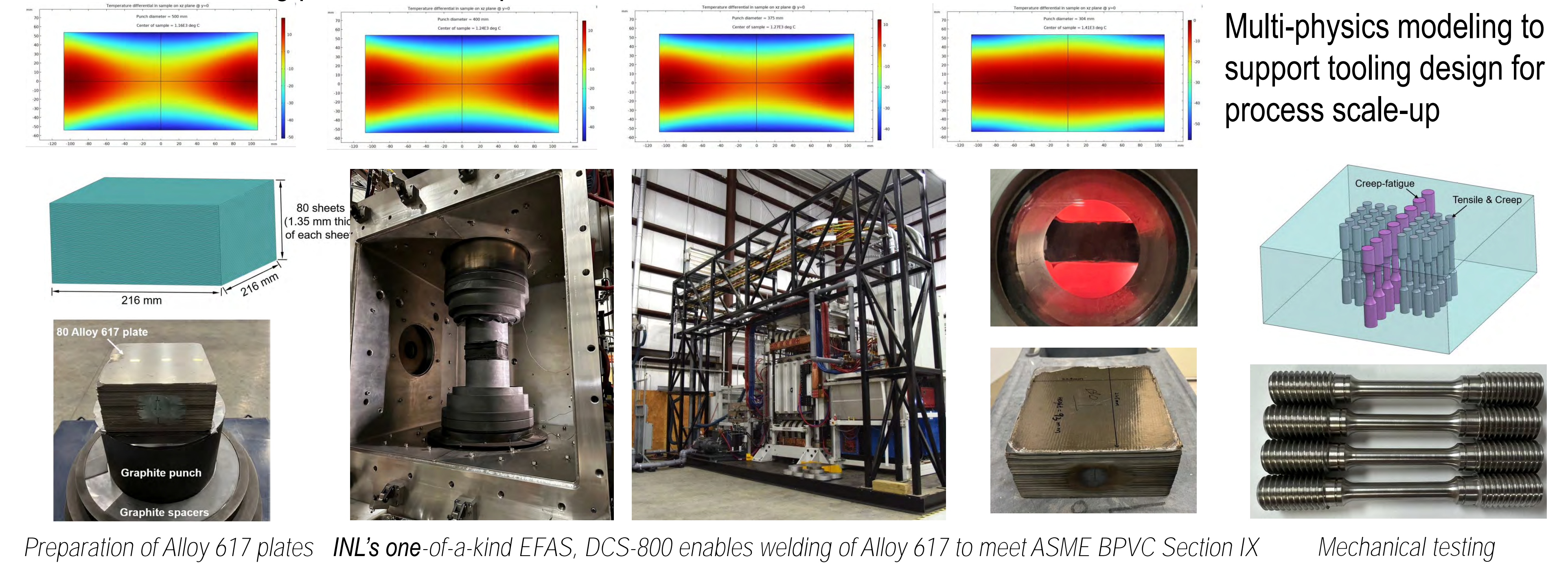


Results

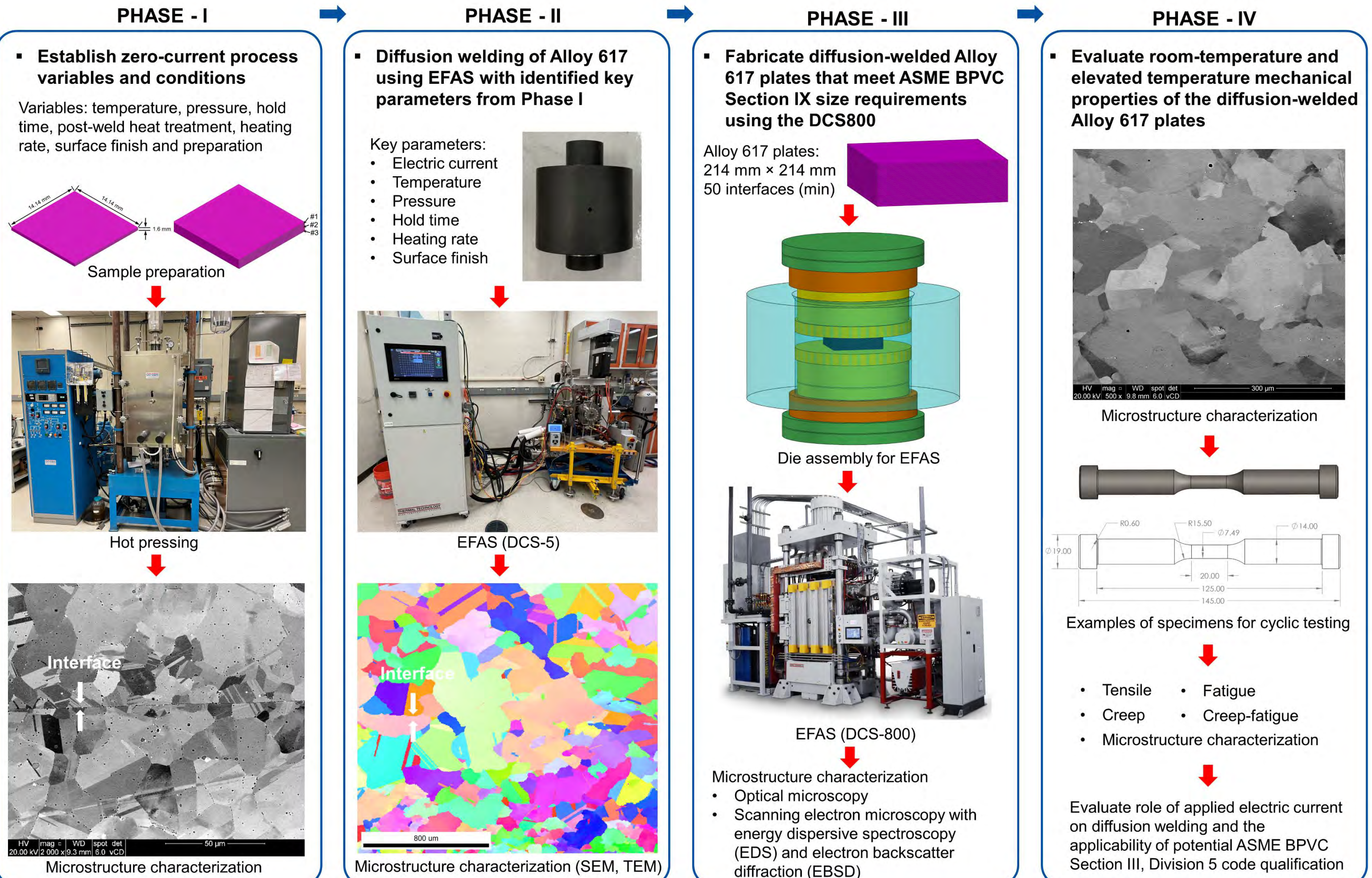
- The intense current during EFAS has a significant influence on precipitation and grain boundary migration. Coupled with optimized temperatures, the microstructure produced using EFAS is superior to that produced using hot pressing



- Diffusion welding process scale-up



Method



Deliverables

- U.S. patent application: No.63/269,302
- U.S. patent application: No.18/183,513
- Zhang et al. PVP2022-83842, 2022
- ASME PVP Conference, Las Vegas, NV, 2022
- MS&T Technical Meeting, Pittsburgh, PA, 2022
- ASME PVP Conference, Atlanta, GA, 2023
- Zhang et al. J. Mater. Res. (under review)
- Zhang et al. Mater. Sci. Eng. A (in preparation)
- Zhang et al. J. Alloys Compd (in preparation)
- AMMTO 2864-1774 & CINR proposals

Project Number: 21A1050-120FP

LRS Number: INL/EXP-23-74243

www.inl.gov

Work supported through the INL Laboratory Directed Research & Development (LDRD) Program under DOE Idaho Operations Office Contract DE-AC07-05ID14517."

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INL Idaho National Laboratory

Embedded Fiber Optic Sensors for Sensing in Extreme Environments

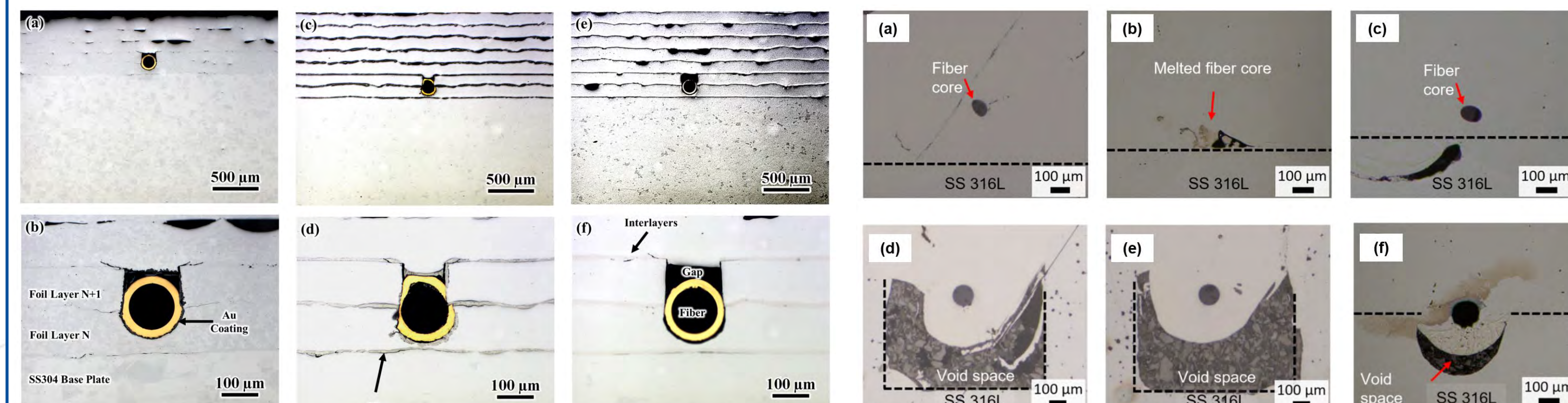
Xinchang Zhang, Zilong Hua, Jorgen Rufner

Objective

- Integrate fiber optic sensors into high-temperature high-strength materials to measure real-time critical information (e.g., temperature, strain) for structural health monitoring to enhance the performance of critical components operating in harsh environments

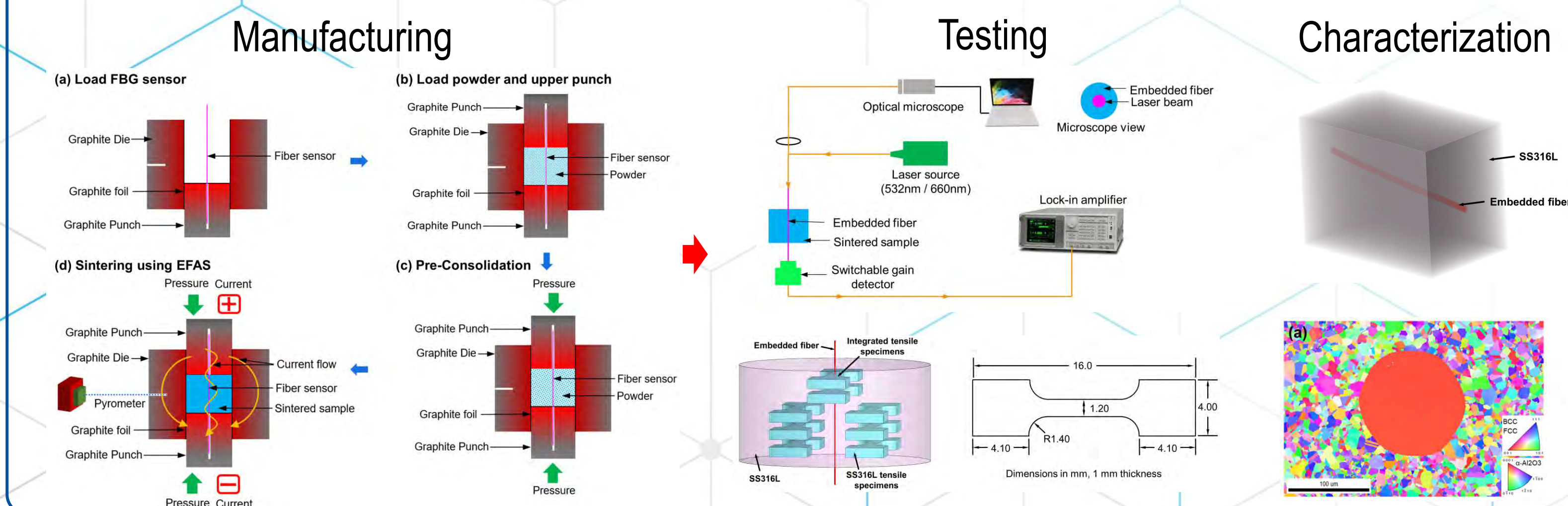
Prior Literature and Challenges

- Ultrasonic additive manufacturing (UAM) and laser-based AM techniques have challenges in achieving good integration between embedded fiber optic sensors and matrix



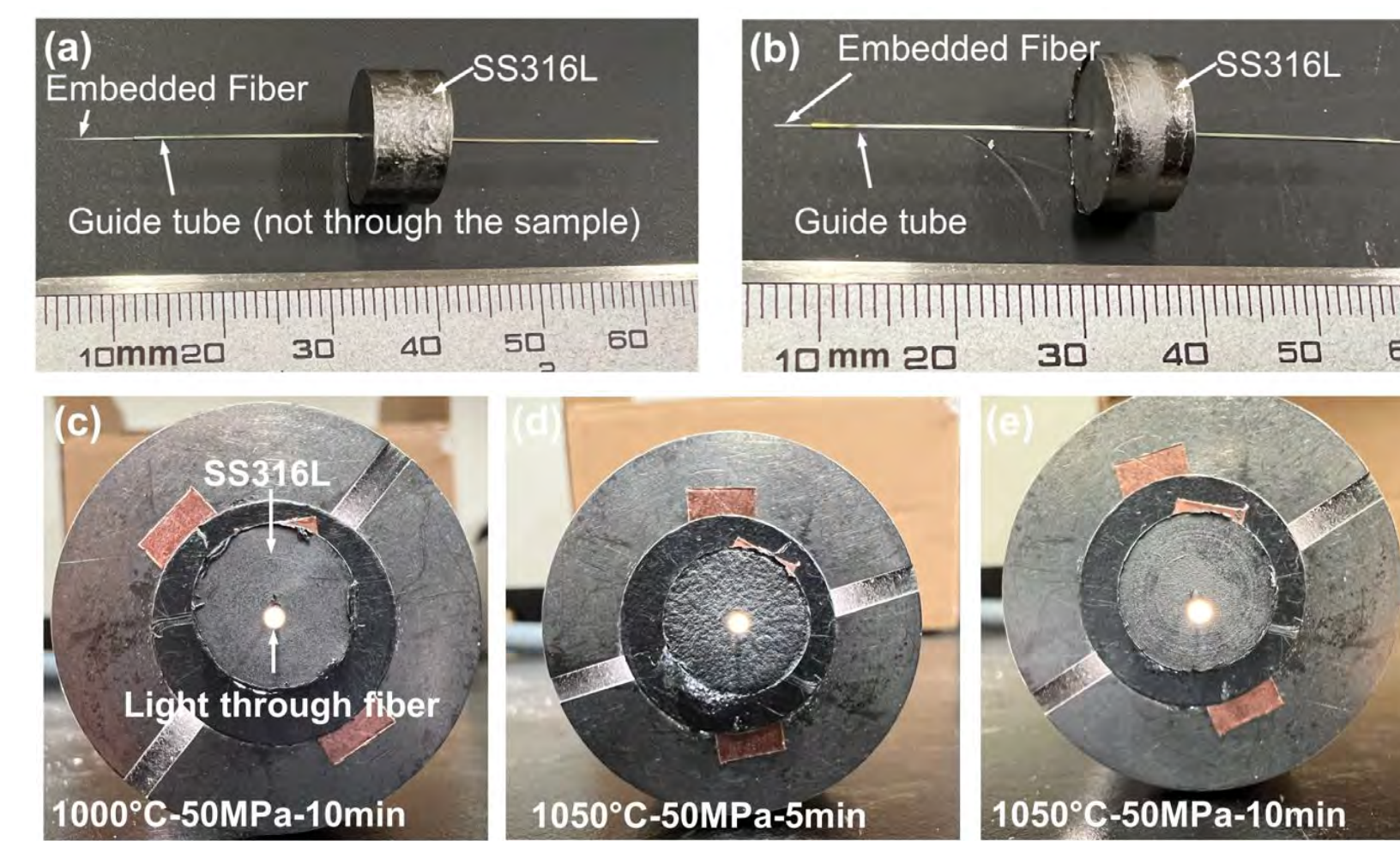
Approach

- Embed fiber optic sensors in materials via electric-field assisted sintering (EFAS)
- Investigate the sensor functionality, fiber-matrix bonding quality, and properties of the integrated materials through optical properties measurement, characterization, and mechanical testing
- Study the relationship between embedding parameters and embedding quality



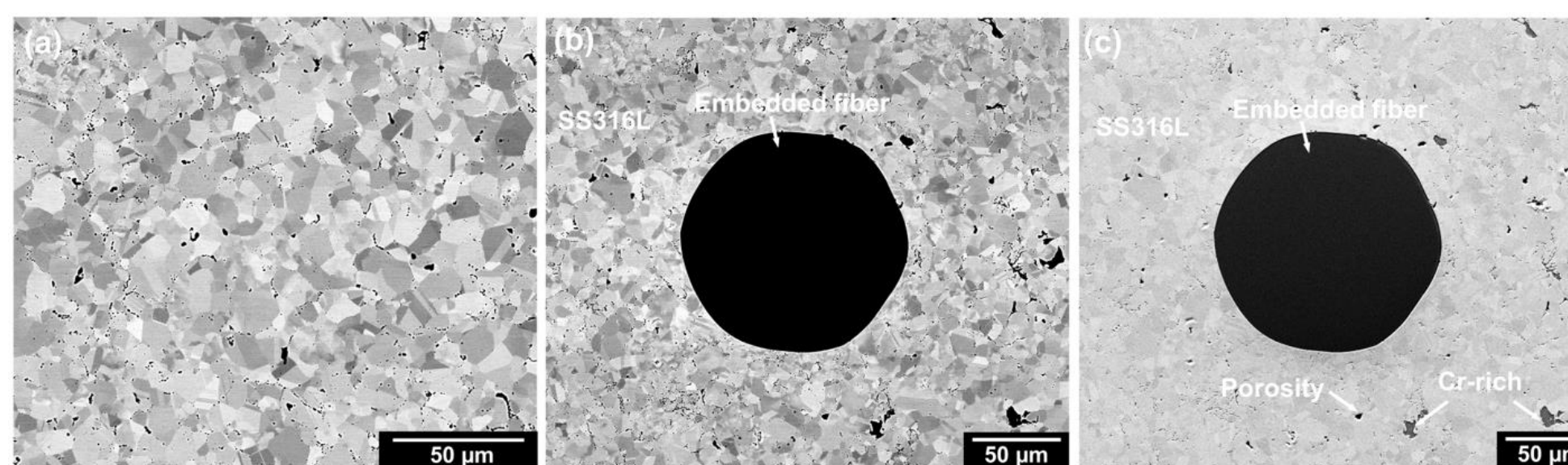
Results

- Fiber optic sensors were successfully encapsulated in stainless steel 316L matrix via EFAS
- Optical attenuation measurement of the sensors before and after encapsulation in SS316L evidenced good functionality of the sensors after embedding

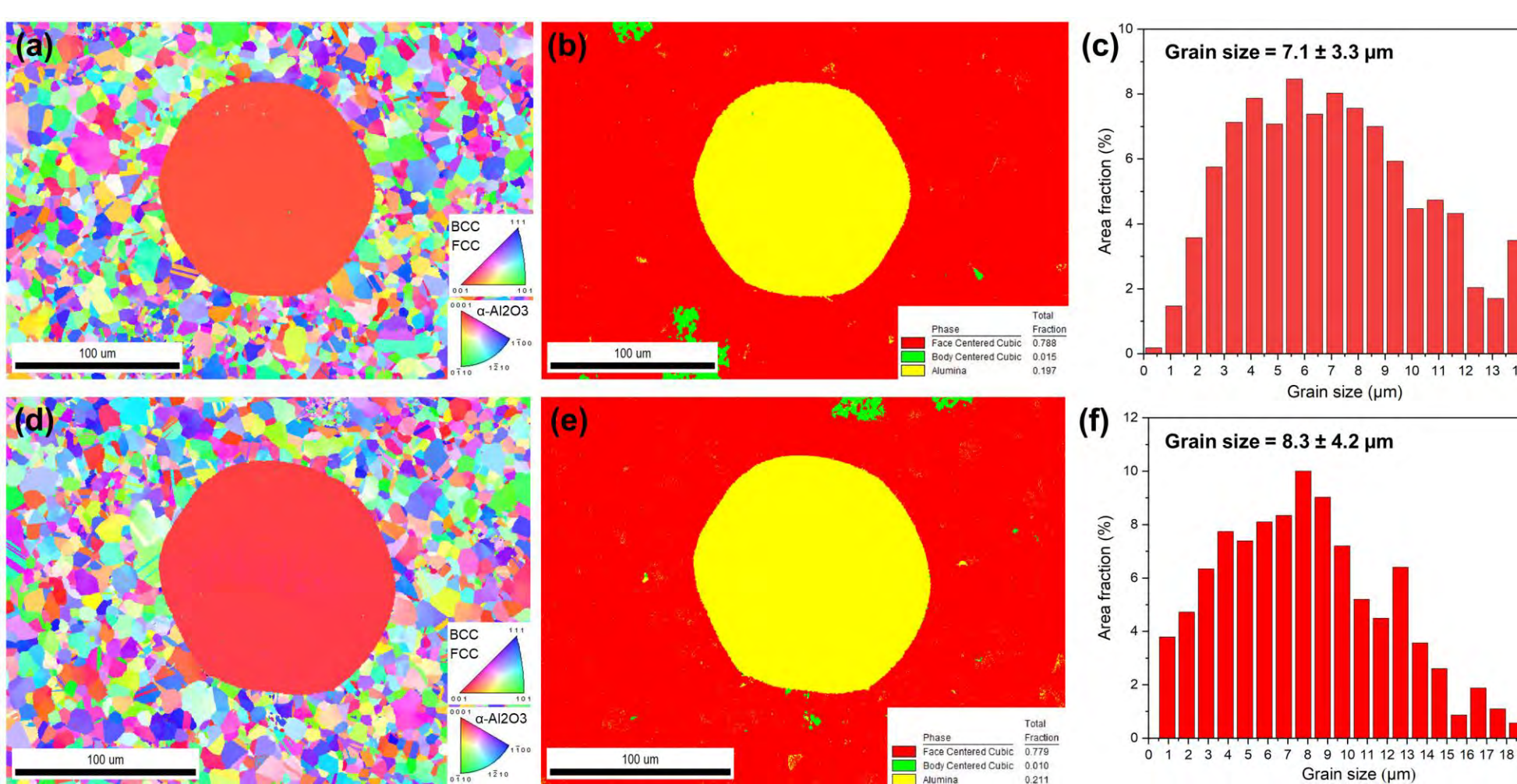


SS316L parts with integrated fiber optic sensors

- Good bonding quality was achieved between the embedded fiber optic sensors and SS316L
- Superior tensile properties were obtained in the EFAS SS316L (UTS=670MPa, YS=331MPa) and integrated materials with sensors (UTS=596MPa, YS=323MPa).



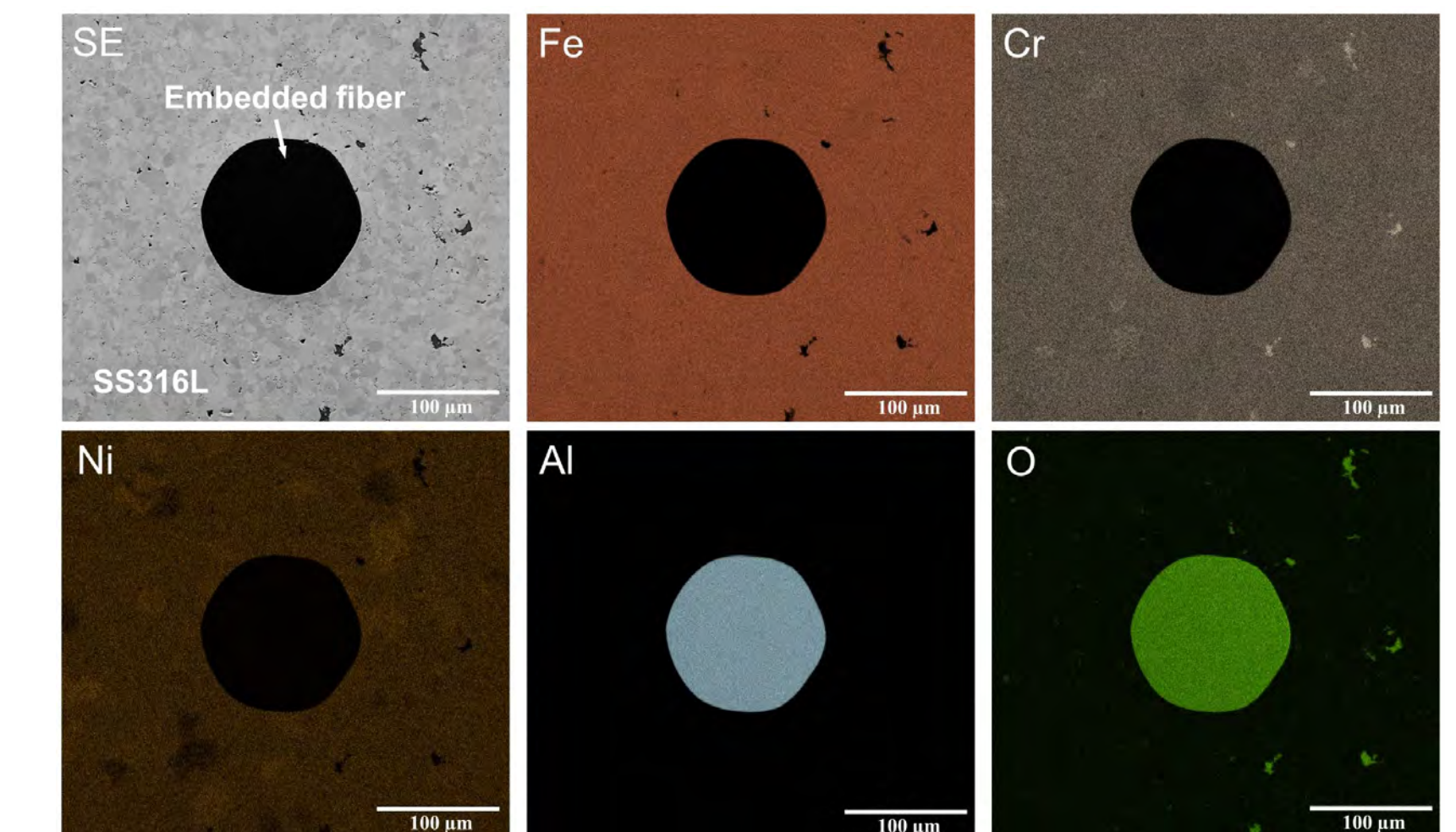
SEM micrographs of the embedded sensors



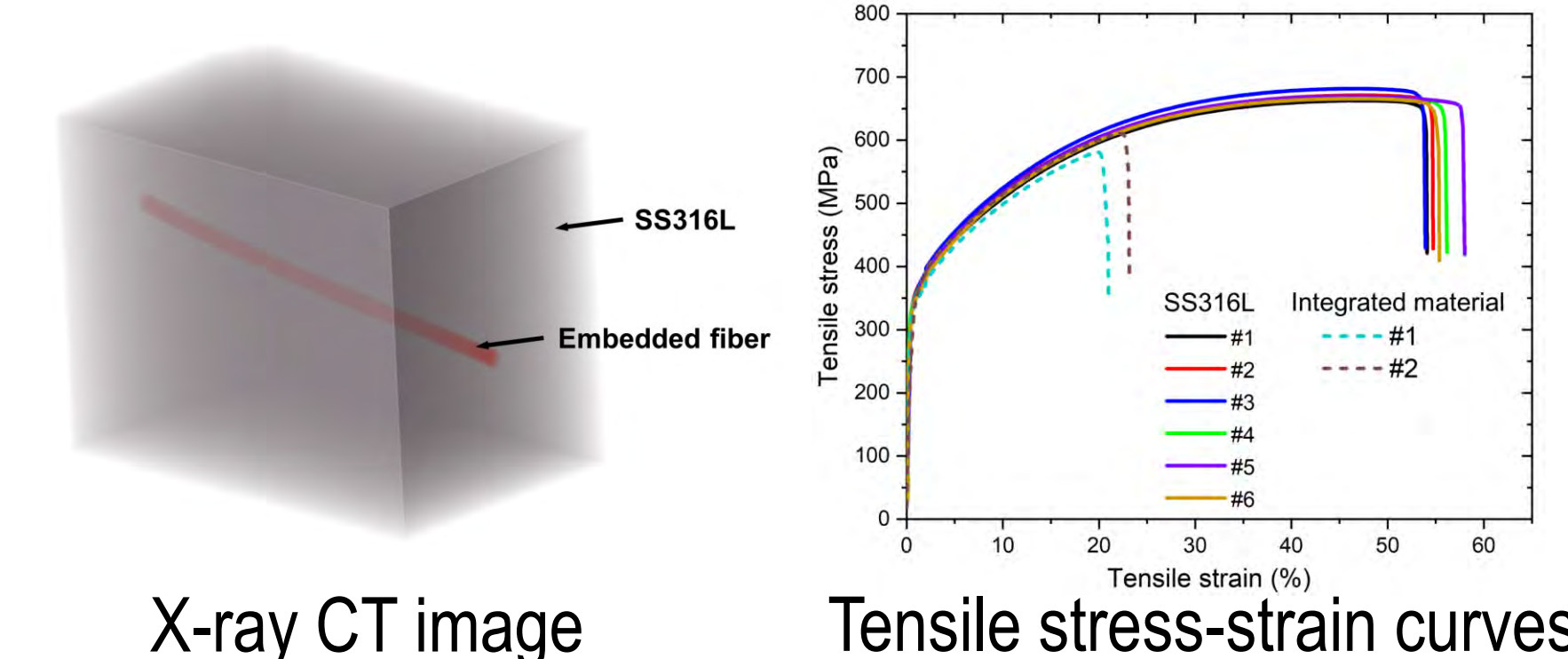
EBSD grain orientation maps, phase maps, and grain size

Optical attenuation of fiber optic sensors post-integration

Laser wavelength	As-received	Sample #2 (1000°C-20MPa-5min)	Sample #5 (1050°C-50MPa-5min)	Sample #9 (1100°C-50MPa-5min)
Relative to input signal power (dB)				
532 nm	1.57	1.87	3.02	3.31
660 nm	1.43	1.97	2.71	3.29
Relative to as-received fiber (dB)				
532 nm	-	0.30	1.45	1.74
660 nm	-	0.54	1.28	1.86



Elemental distribution across the embedded sensors



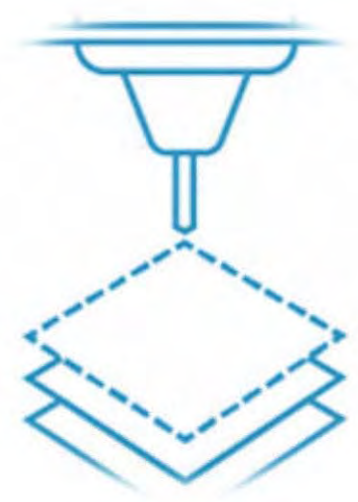
X-ray CT image

Tensile stress-strain curves

Deliverables

- U.S. patent application, "Embedded fiber optic sensors for real-time in-situ sensing in extreme environments", No.63/487,327
- Zhang et al. "Integrating fiber optic sensors into metallic components for sensing in harsh environments", Optics and Laser Technology (under review, submitted in June 2023)
- Zhang et al. "Smart structural materials with embedded fiber optic sensors for health monitoring in harsh environments", Proceedings of the ASME 2023 Smart Materials, Adaptive Structures and Intelligent Systems, SMASIS2023-117419, 2023
- Presentation at ASME 2023 Conference on Smart Materials, Adaptive Structures and Intelligent Systems, Austin, TX, 2023

Project Number: 22P1074-015FP (Seed Fund) LRS Number: INL/EXP-23-74269



PRESENTER

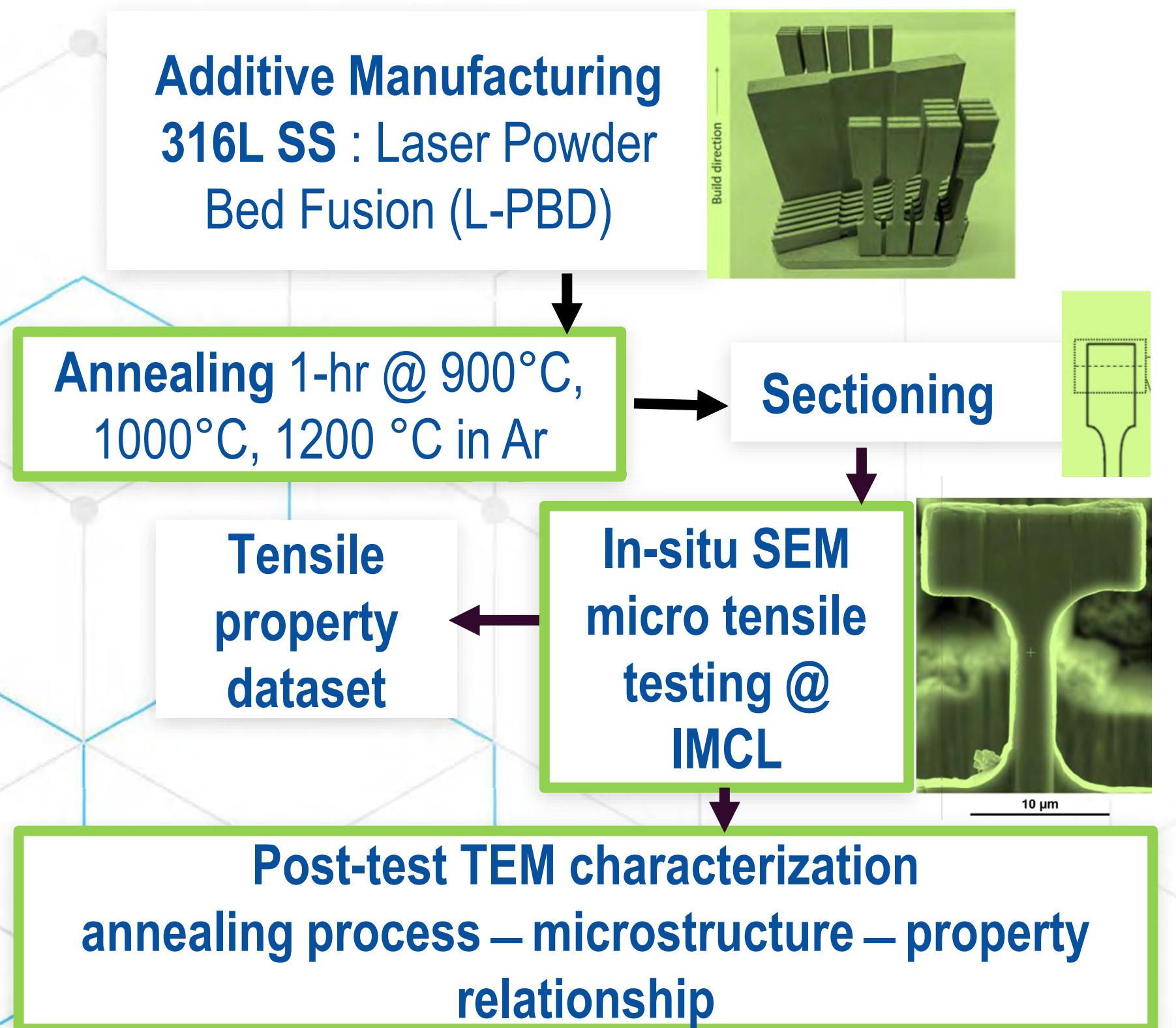
Yachun Wang
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Understanding how Annealing Affects Microstructure & Micromechanical Properties of AM 316L SS

Background

- AM 316L holds promise for application in advanced nuclear reactors
- Oxide inclusions in AM 316L are metastable
- It is important to understand how oxide inclusions evolve upon annealing and affect the local mechanical property of AM 316L

Methods



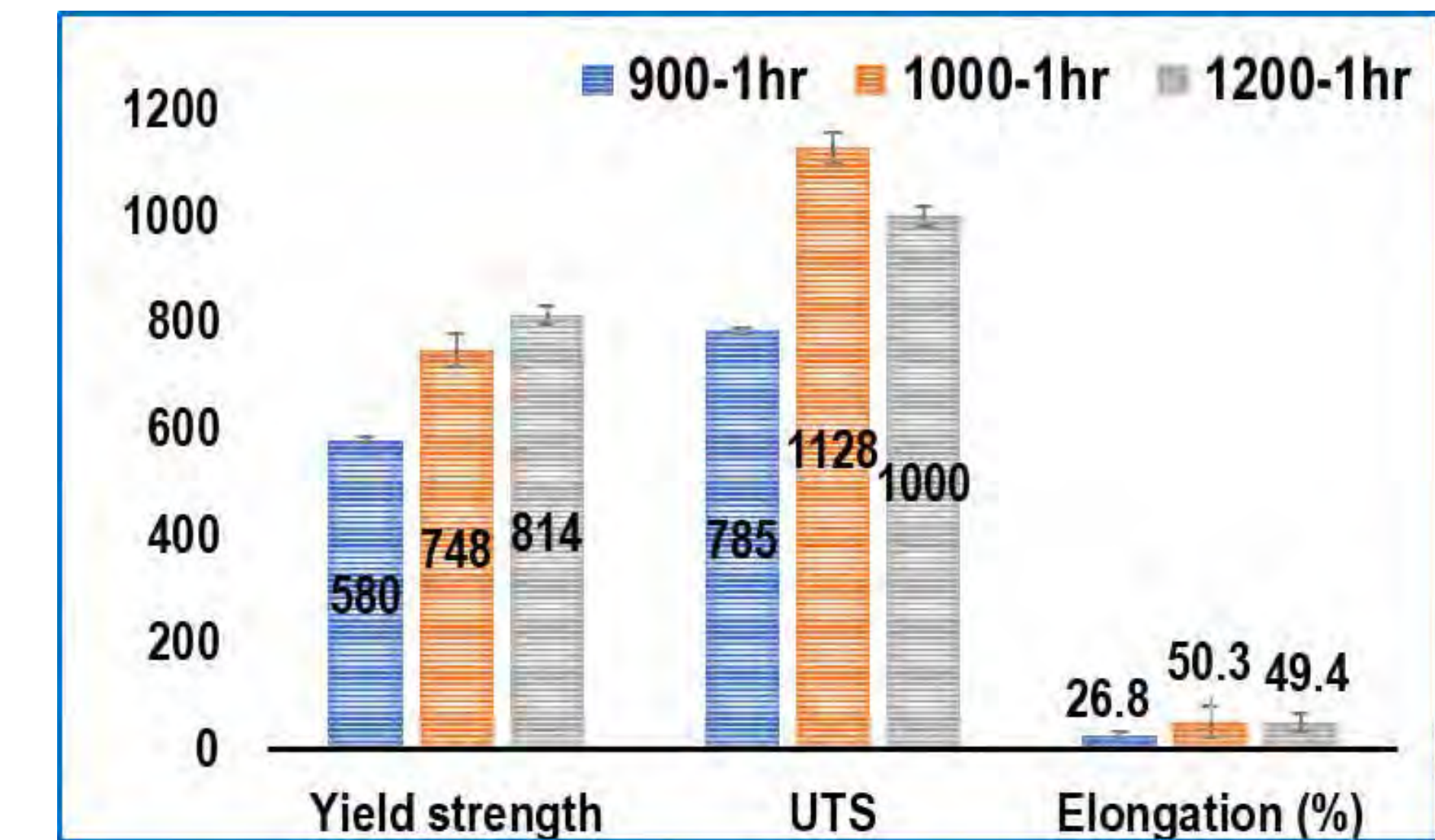
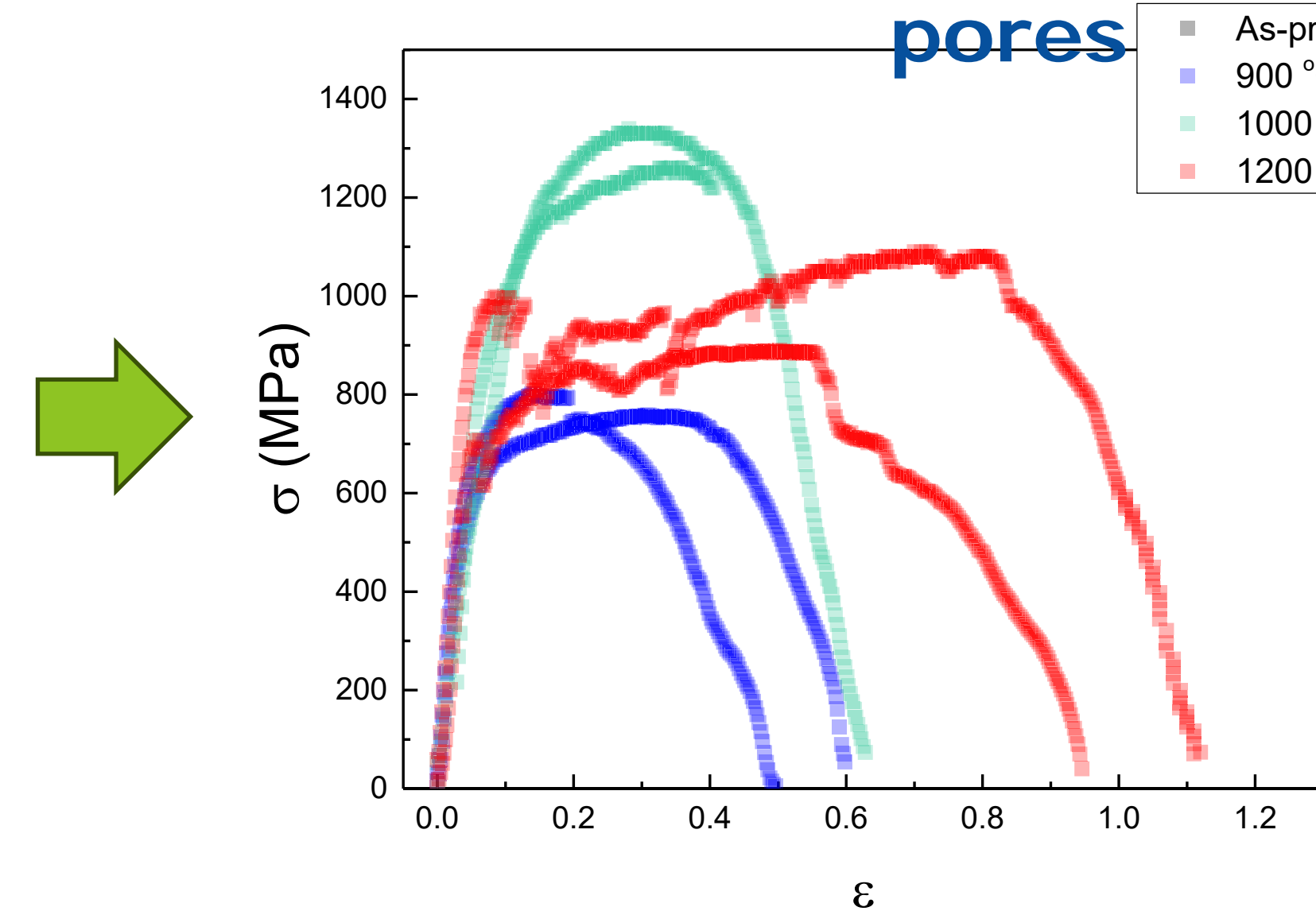
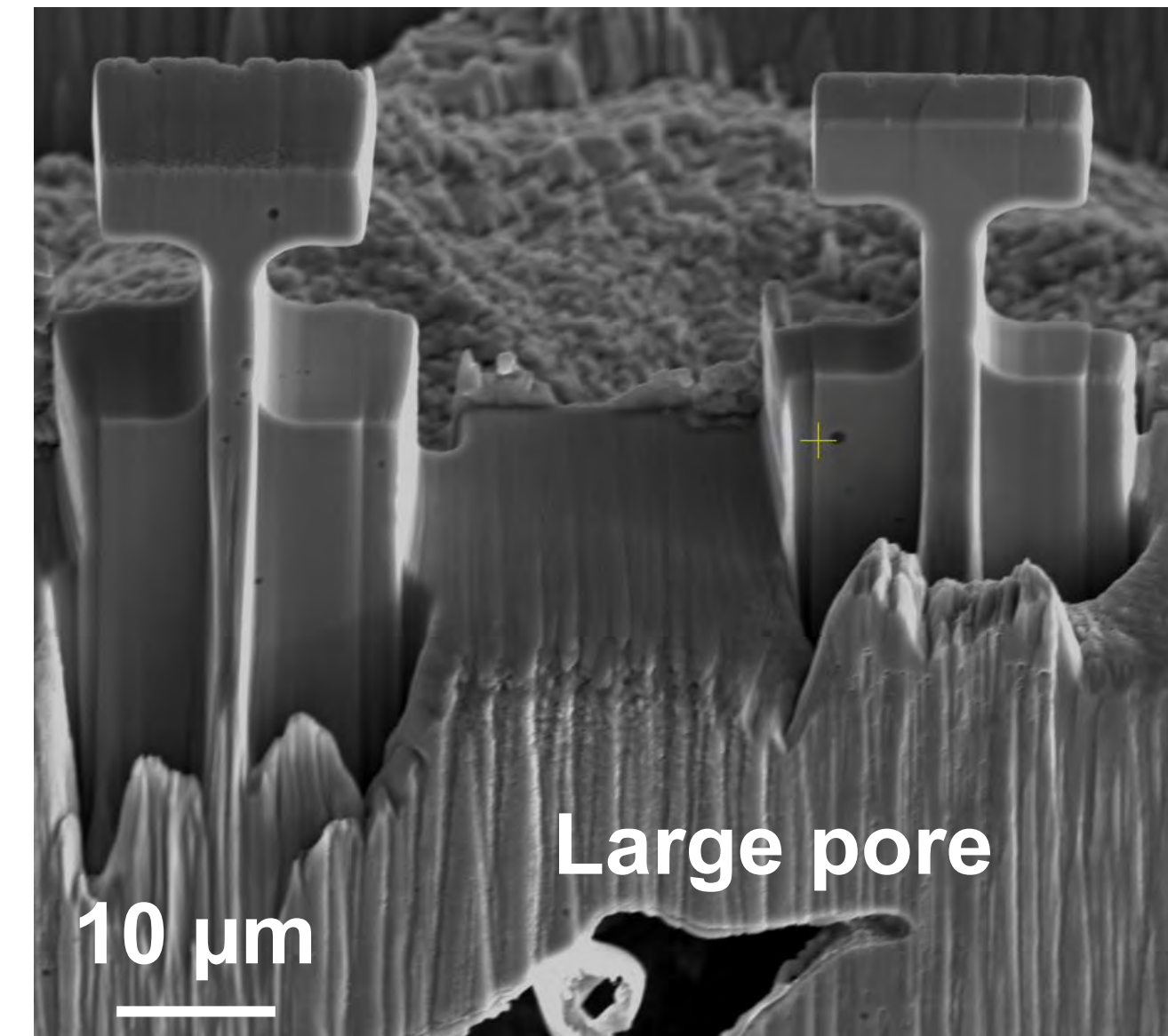
Presentation & Publication

- TMS 2023, Small-Scale Mechanical and Corrosion Properties of Additively Manufactured Stainless Steel
- MRF-FaSCINATe (UK), Testing Nuclear-Structural relevant Stress States and Reactor Operation Temperatures
- A journal manuscript is undergoing

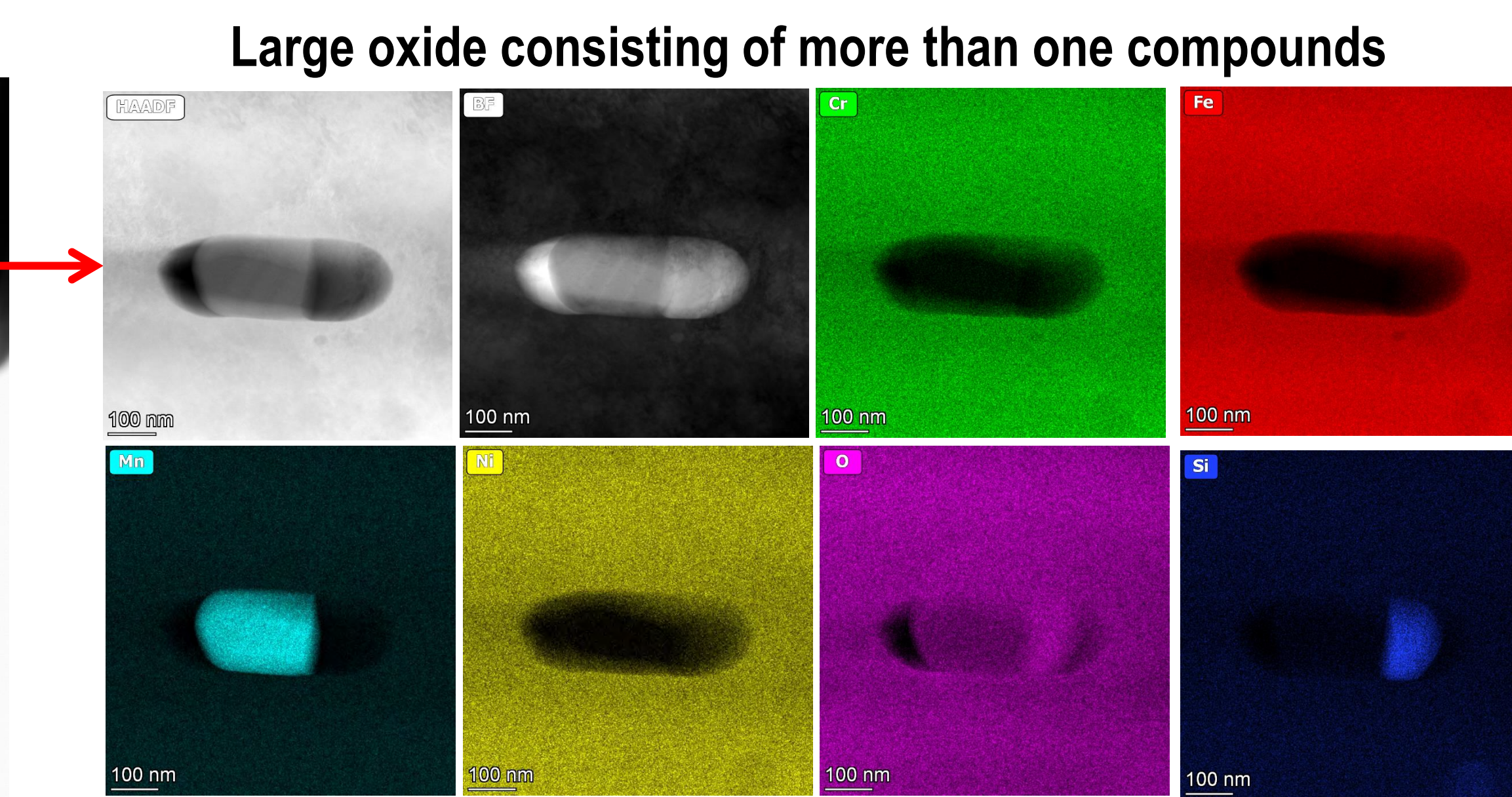
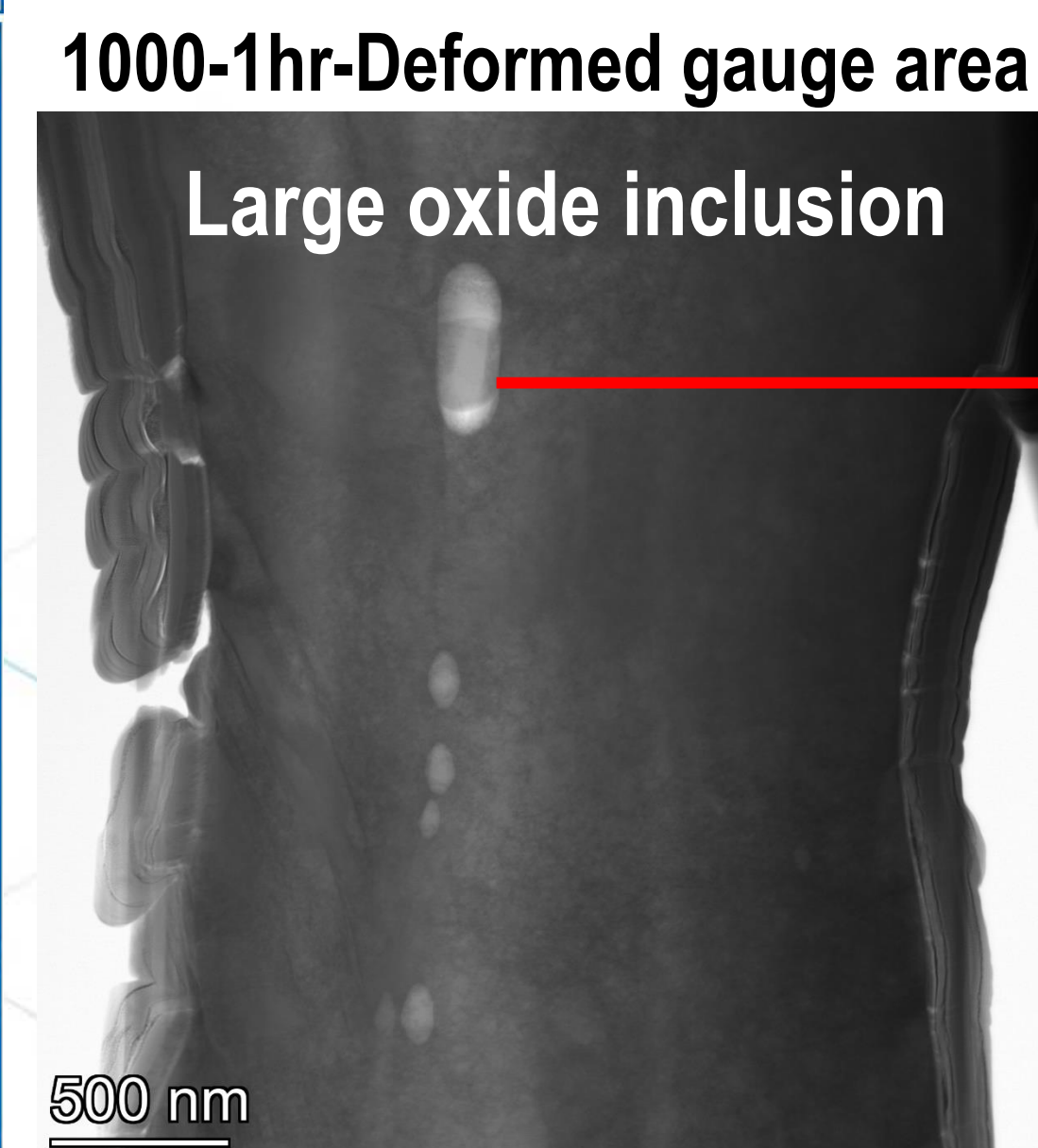
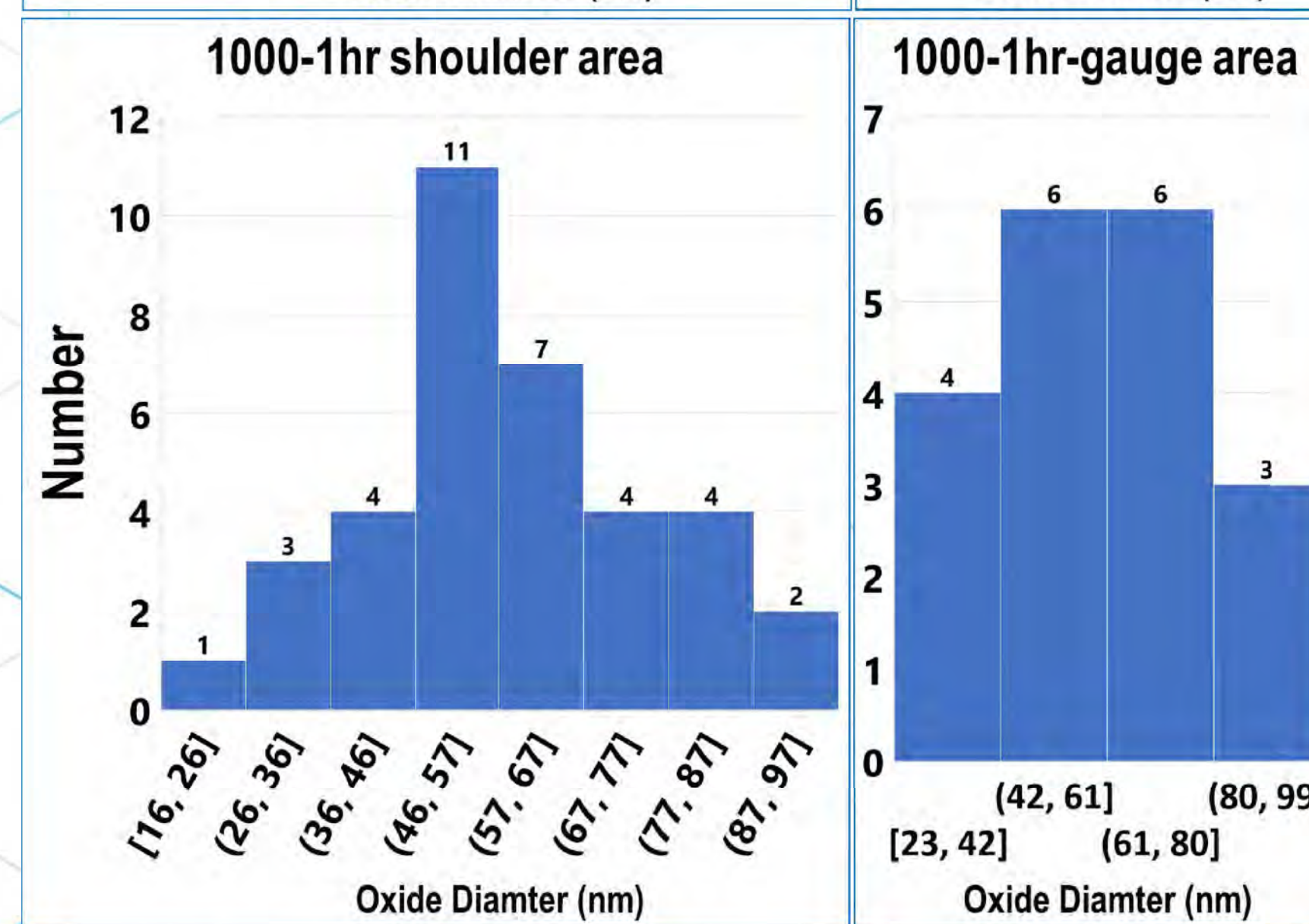
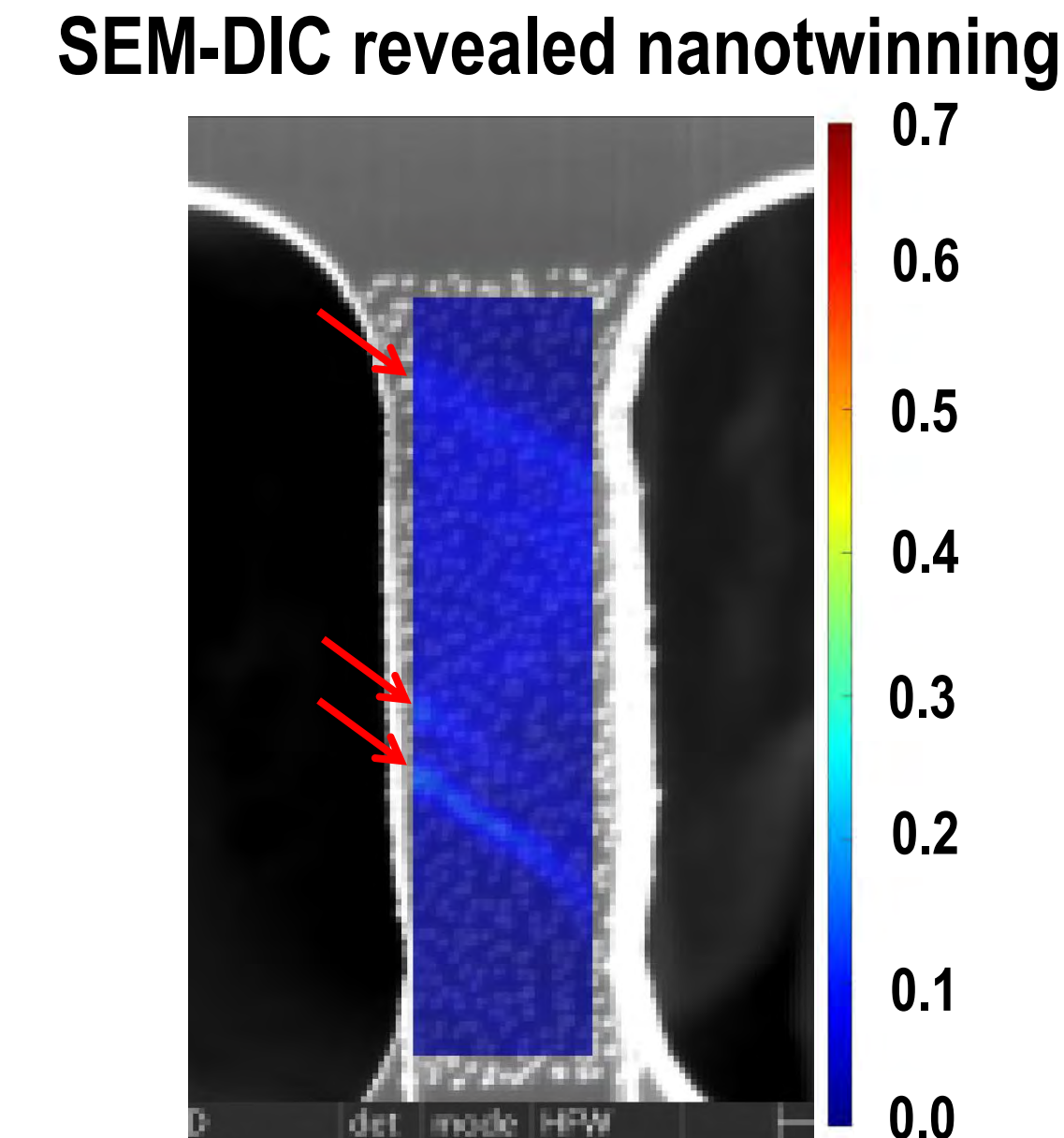
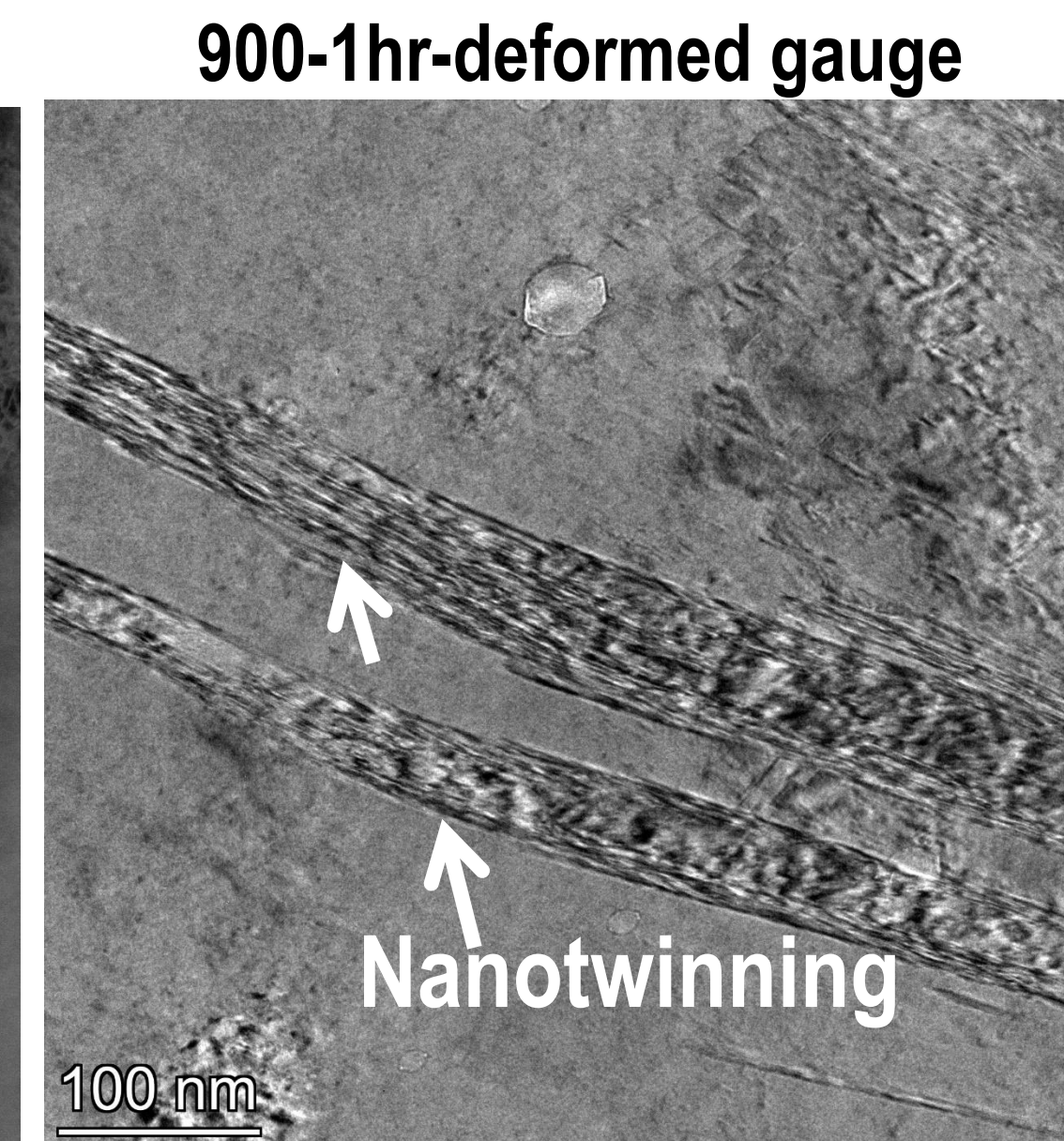
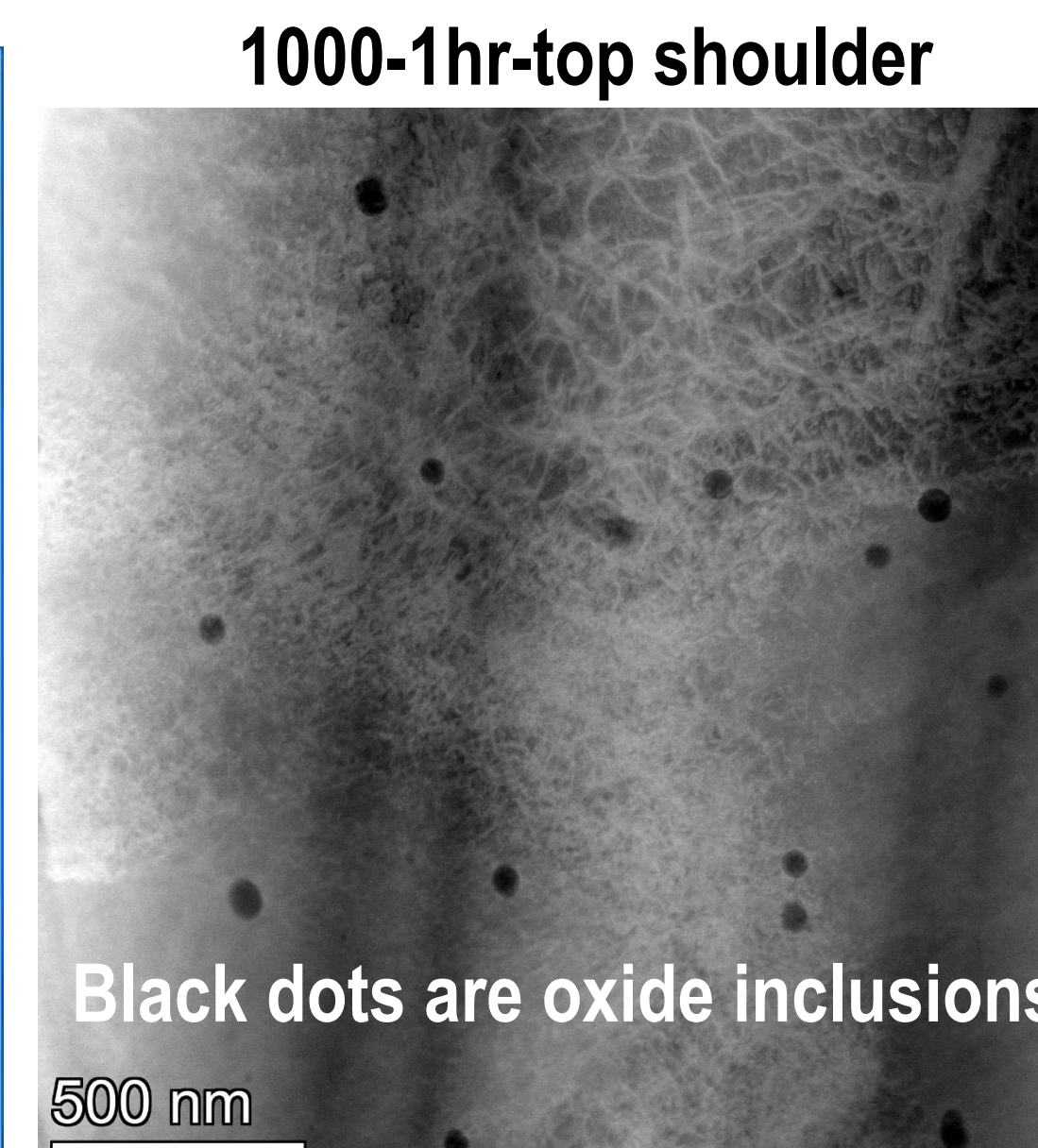
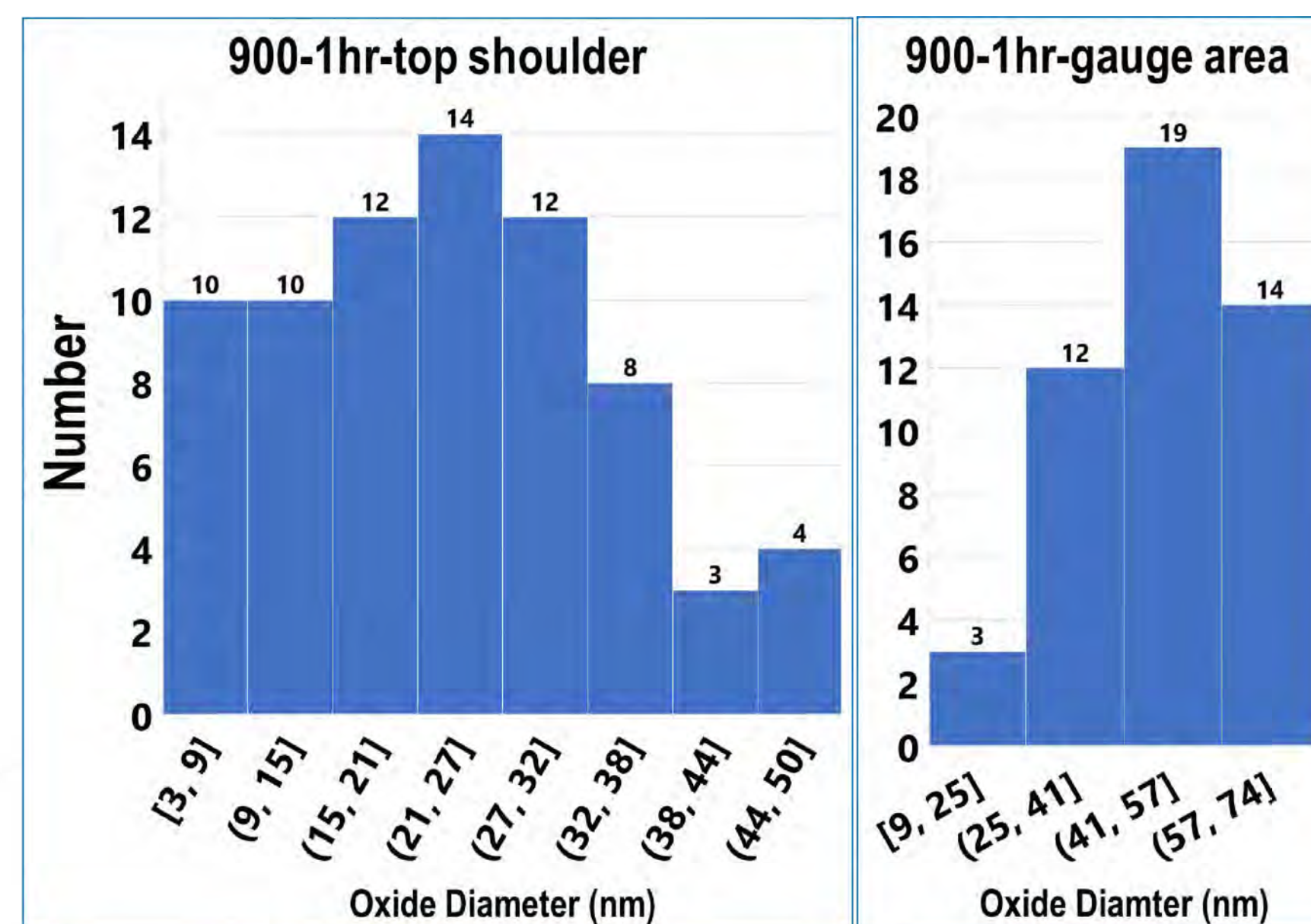
Acknowledgement:

Xiaolei Guo, Gerald S. Frankel (Ohio State University); Cameron Howard, Daniel Murray, Laura R. Hawkins, Fei Xu, Tiankai Yao, and IMCL facility operation team (MFC, INL)

Micro-tensile testing allows to probe micromechanical properties without interference of



Oxide inclusions & microstructure characterization



Project Number: 22P1071-022FP

LRS Number: INL/EXP-23-74097

Advanced Materials & Manufacturing for Extreme Environments: Multi-role and Integrated Material Systems

Computer-aided knitting for extreme scenarios using high-performance polymer fibers as constituent material

Zherui Guo*, John Klaehn

Objectives

- Increase pressure capacity of hydrogen cylinders by using knitted high-strength polymer fibers
- Optimize knitting pattern using computer-aided algorithms

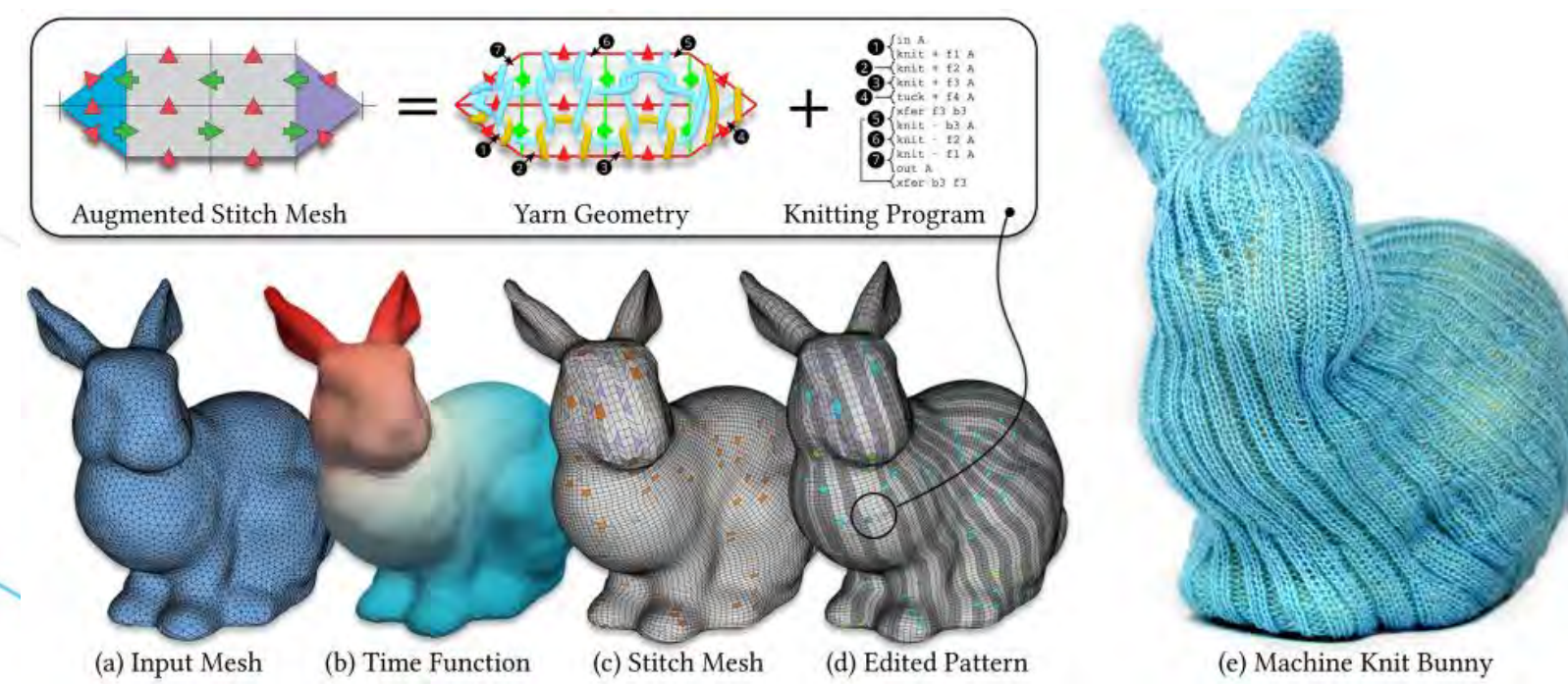


Fig. 1: Computer-aided knitting algorithms allow for complex geometries to be automatically translated into machine-knitting instructions¹.

Methodology

- Proposed workflow from tank geometry to full knitted system

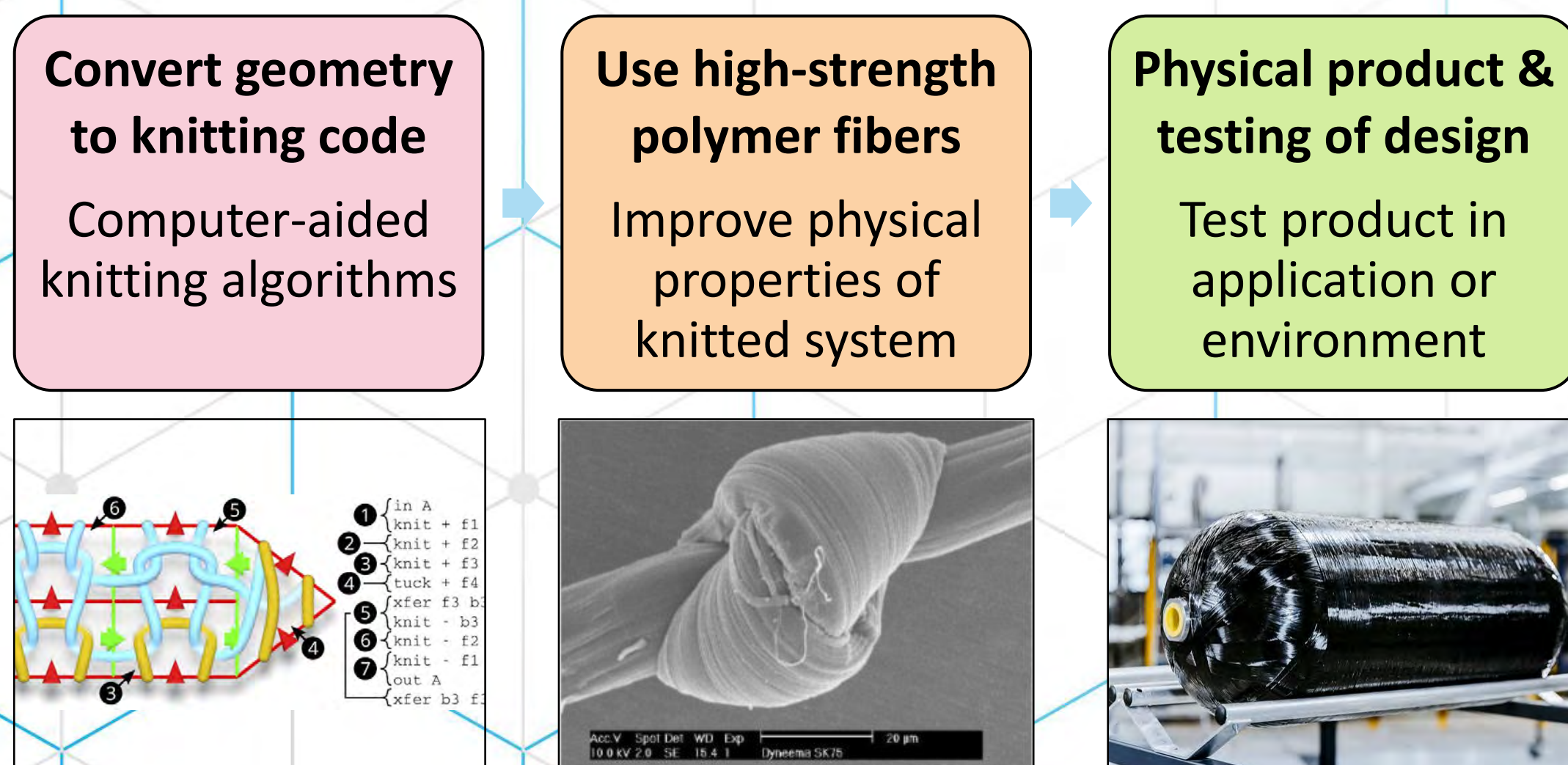


Fig. 2: Proposed workflow from complex geometry to complex environment applications. The ultimate goal is to have tailored strengths for knitted structures. Images from Refs. 1-3.

Results

- Novel in-situ setup for yarn elastica loop test under microscope
- Several polymer fibers and yarns tested
 1. Kevlar® – para-aramid
 2. Ultra high molecular weight polyethylene (UHMWPE)
 3. Vectran® HT – liquid crystal polymer
 4. SpiderWire® – braided fishing line

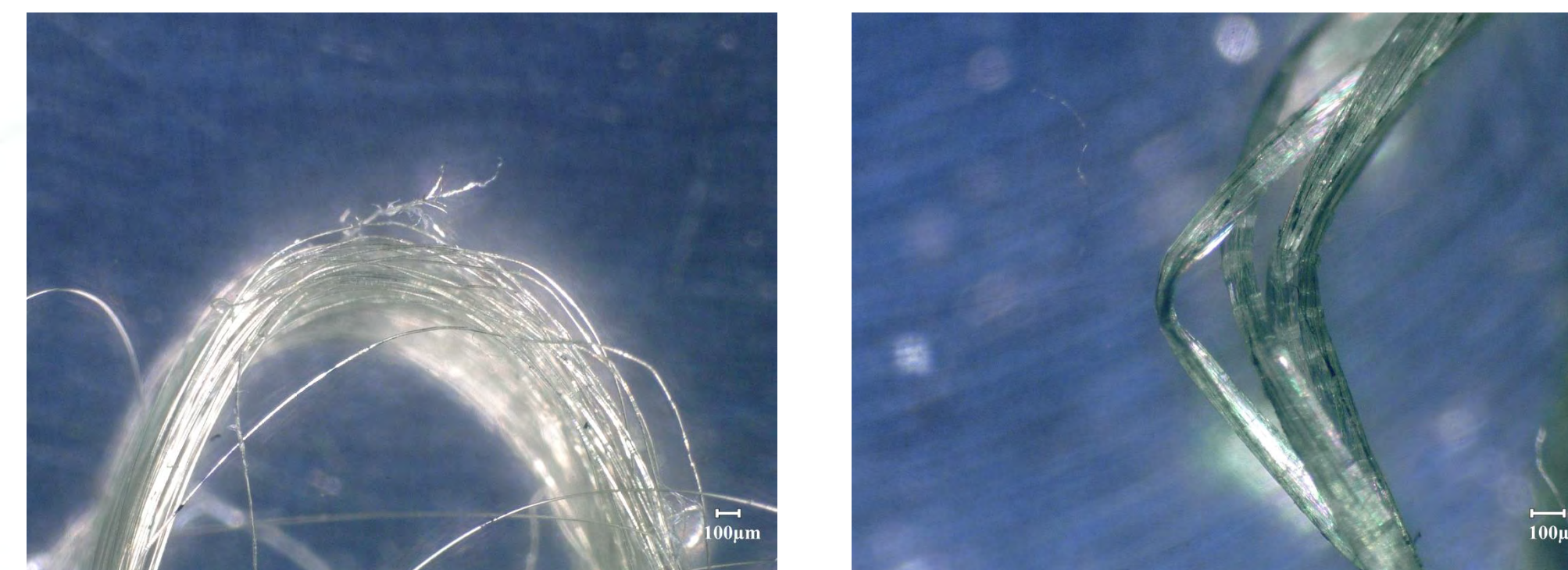


Fig. 3: Vectran® HT (left) and SpiderWire® (right) under severe loop bending. Braided structures tend to fray and split under complex mechanical loading.

- Twin-fiber transverse compression – obtain mechanical properties
- SwiftComp® – Homogenization algo. for yarn mesoscale properties

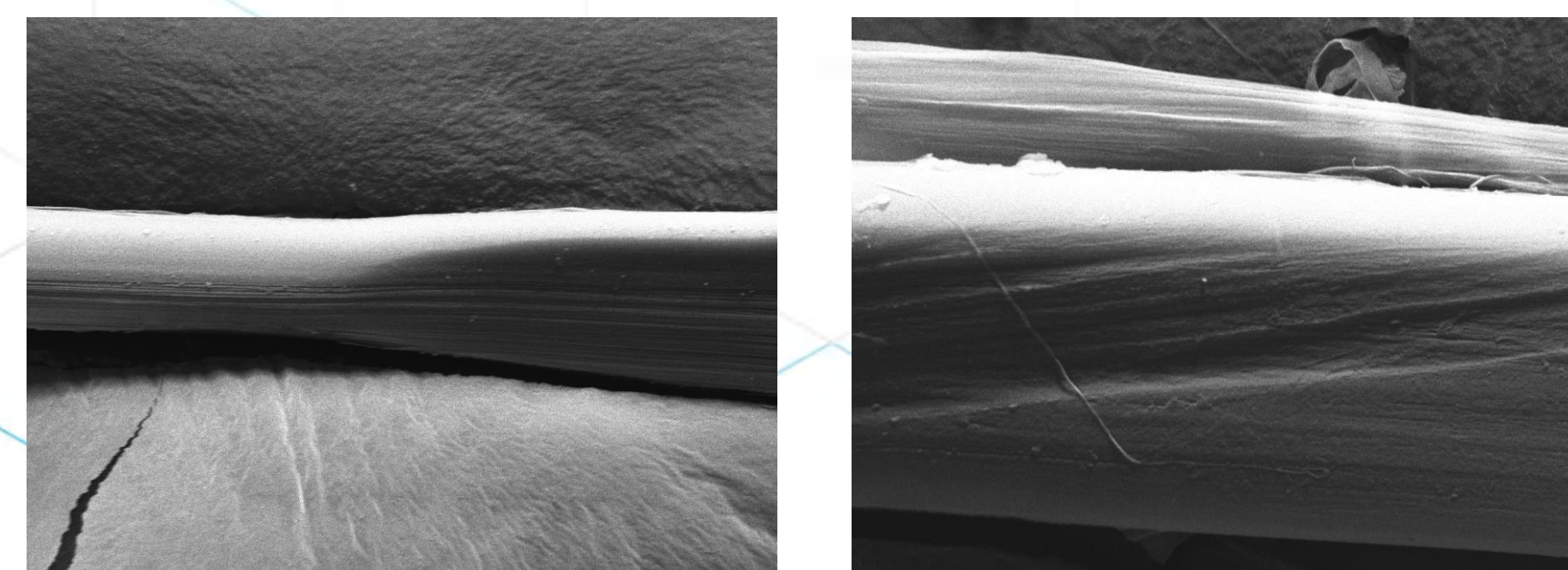


Fig. 4: Post-compression micrographs of Vectran® HT fibers exhibiting plastic behavior (left) and severe defibrillation (right).

- Finite element modeling
 - TexGen – yarn and fabric level mesh generation
 - ABAQUS/Explicit – simulation of fabric tensile test

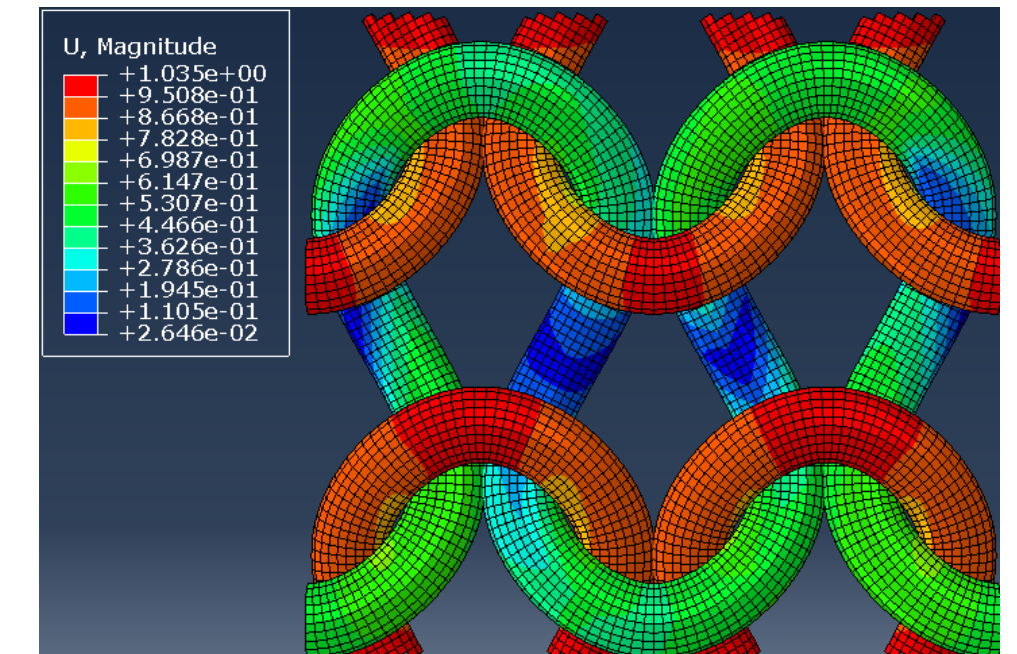


Fig. 5: ABAQUS/Explicit simulation of Vectran® HT fabric under tension.

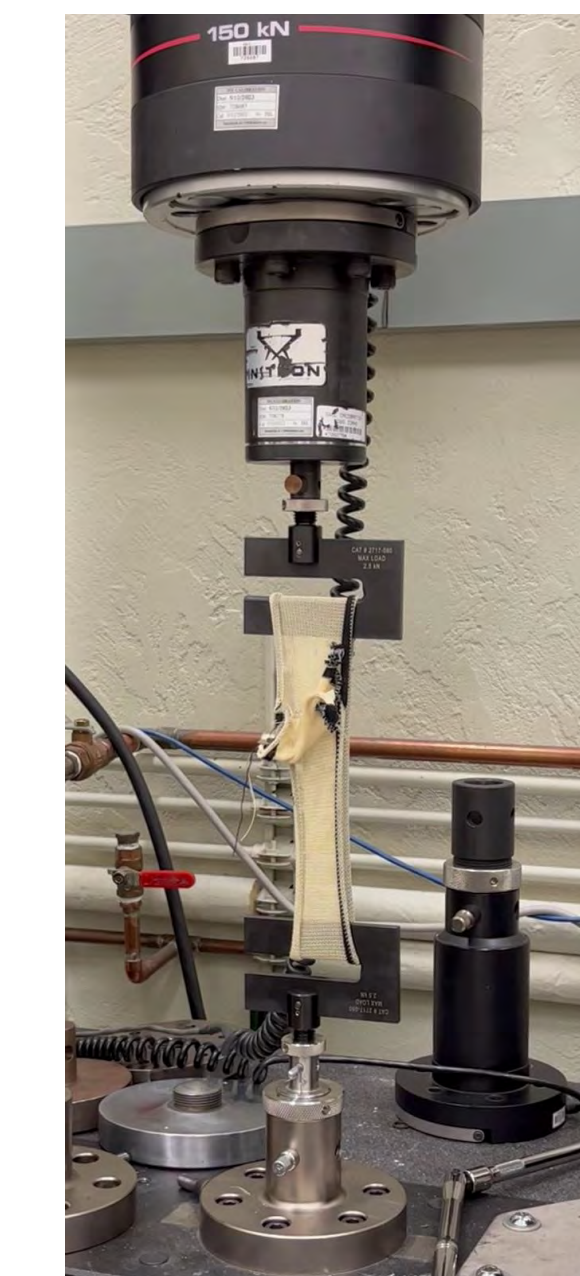
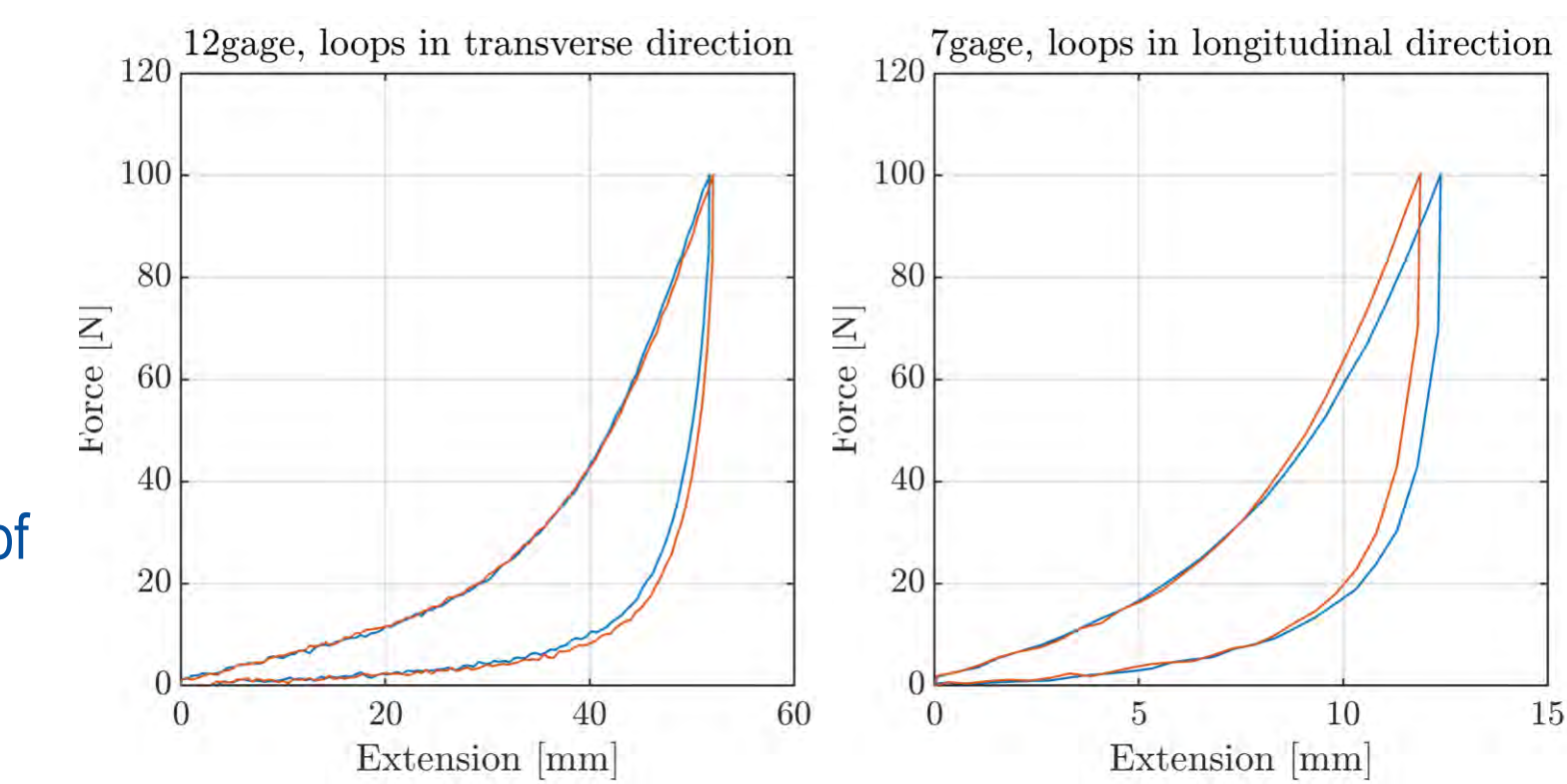


Fig. 6: Quasi-static tensile test of Vectran® HT fabric swatch (top) and measured force-extension curves (right).

- Quasi-static tensile tests
 - Computer-aided knitting of fabric using KnitOut algorithm
 - Comparison of knit patterns and corresponding tensile strengths



Conclusions

- Computer-aided knitting can generate custom geometry patterns
- Developed workflow to optimize knitted fabrics from constituent fibers

References

1. Narayanan, V., et al., *Automatic Machine Knitting of 3D Meshes*. ACM Transactions on Graphics, 2018. 37(3): p. 1-15.
2. Marissen, R., *Design with Ultra Strong Polyethylene Fibers*. Materials Sciences and Applications, 2011. 02(05): p. 319-330.
3. Nehls, G. Hexagon Purus signs multi-year global agreement for type IV composite hydrogen cylinders. Composites World, 2021.

Project Number: 22P1065-006FP

LRS Number: INL/MIS-23-74195