Advanced Sensors and Instrumentation

Boise State University Research Overview:

Supporting Activities

Advanced Sensors and Instrumentation (ASI)
Boise State University (BSU) Research Overview
31 October, 2023

Brian J. Jaques, BSU, Program Manager for ASI at BSU
Boise State University (MSE) and Idaho National Laboratory

Focus Today

- Brief history of the ASI Program at BSU
- Highlight ASI Program impacts at BSU
- Overview of all Supporting activities
- More in-depth discussion of support for
  - Nuclear Thermocouples
  - Neutron Laboratory development

ASI at BSU Program History

ASI at BSU History

2017 – Program discussion and planning - **In-Pile Initiative (I^3)**

Research Thrusts identified:

- Nuclear Instrumentation
- Halden Capability recovery
- Irradiation deployment
- Materials Properties
- Structure and Chemistry
- Advanced Manufacturing

2018 – Research begins

Research Thrusts redefined:

- AM for Sensors
- HTIR-TC Sensors
- Sensors for Mechanical Properties
- Acoustic Sensors
- Line Source methods for Thermal Sensors
- Radiation Tolerant Fiber Sensors
- Electrochemical Sensors
- Neutron Generators for Sensor Development

2019 – Slight re-org and "Program" begins - **In-Pile Instrumentation Program (I^2)**

2020 – Program Alignment – **Advanced Sensors and Instrumentation Program (ASI)**

2020 – 3 year contract begins

Current Structure and BSU PIs:

- Nuclear Thermocouples – B. Jaques
- Linear Variable Differential Transformers – D. Deng
- Acoustic Sensors – D. Deng
- Printed Sensor Tech. for Harsh Environments – D. Estrada
- Line Source for Thermal Properties – D. Estrada

The program has resulted in nearly $4M of funding and supported more than:

10 faculty members
8 staff members
14 graduate research assistants
16 undergraduate research assistants
Several pieces of infrastructure within the COEN

ASI Program Impacts (In the past 5 years!)

Student Successes as a result of ASI

- 9+ INL Internships (Graduate and Undergraduate)
- 5 INL Grad Fellows
  - Kiyo Fujimoto, Timothy Phero, Sohel Rana, Corey Efaw, Joy Morin (soon!)
- 5 Graduate fellowships
  - Kati Wada, Kaelee Novich (UNLP and NNSA),Kiyo Fujimoto, Addie Lupercio, Courtney Hollar
- > 30 graduate and undergraduate Scholarships
- 5 National Awards/recognitions
- Formed the Nuclear Energy Club at BSU –recognized by the ANS (Spring 2023) as an ANS Boise Chapter Student Section

Talent Pipeline to INL

- Al-Amin Ahmed Simon
- Kiyo Fujimoto
- Corey Efaw
- Arvin Cunningham
- Jennifer Watkins
- Sohel Rana (Industry)
- Ember Sikorski (SNL)

INL Researcher support

- ≈ 20 Researchers involved

Nuclear Science User Facility impact

- 3 RTEs
- Neutron Generator Lab at BSU
- 3 Infrastructure awards
  - AJP, Ink lab, Panda 3D Printer

4 Patents (2 awarded, 1 pending, HTIR-TC patent support)

> 20 Publications (Accepted or submitted)

> 60 Research Conference presentations and seminars (Including >25 invited presentations)

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

ASI Advanced Sensors and Instrumentation

Boise State University Overview of Supporting Activities

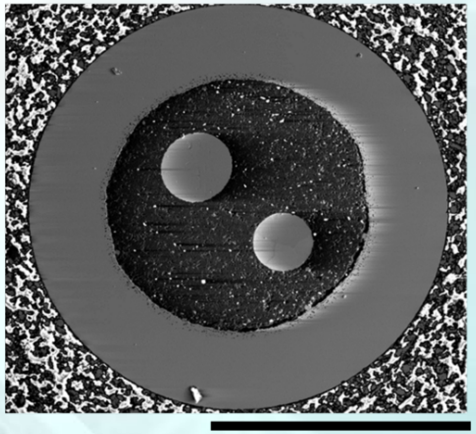
Several research support activities presented during this webinar:

- Nuclear Thermocouples
  - R. Skifton @ 10:30 am
- Acoustic Sensors (UTs, UWTs)
  - J. Daw @ 10:50 am
- Linear Variable Differential Transformers
  - K. Davis @ 1:20 pm
- Printed Sensor Tech. for Harsh Environments
  - M. Wilding @ 2:30 pm
  - M. McMurtrey @ 2:50 pm

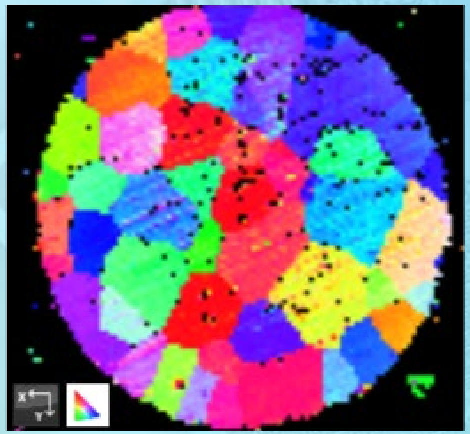
BSU has supported several additional research activities over the years:

- Radiation tolerant fibers
- Mechanical properties
  - CSG, DIC, CTE mismatch, LVDTs
- Neutron detectors
- Electrochemical sensors
- IR thermography
- Fuel sample development
- Line Source for Thermal Properties

Nuclear Thermocouples



1 mm



Advanced Sensors and Instrumentation (ASI)
Boise State University (BSU) Research Overview
31 October, 2023

BSU: Brian J. Jaques (PI) and Scott Riley (GRA)
INL: Richard Skifton
Boise State University (MSE) and Idaho National Laboratory

Project Overview

OVERVIEW

Purpose: The thermocouple element implements R&D activities to develop nuclear instrumentation that address critical technology gaps for monitoring and controlling existing and advanced reactors and supporting fuel cycle development. For temperature measurements, thermocouple instrumentation is typically composed of one or more sensing element, interrogation systems, data acquisition system as well as processes and procedures to collect, analyze and calibrate data. Instrumentation is used to measure process parameters, such as temperature, independently of the experiment, component or process in which it is deployed.

Objectives: The thermocouple activities:
- Characterize influence of traditional stabilization heat-treatment on HTIR-TCs
- Performance assessment of commercial TCs for nuclear applications
- Characterize influence of alternative heat-treating methods, Joule heating, on stabilization of HTIR-TCs

DETAILS

Principal Investigator: Brian Jaques (BSU), BrianJaques@BoiseState.edu
Richard Skifton (INL), richard.skifton@inl.gov

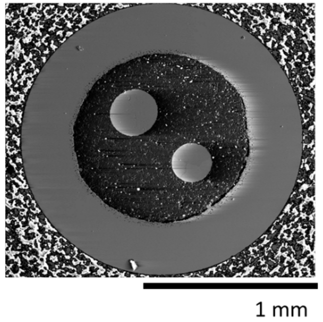
Institution: Boise State University

Collaborators: Idaho National Laboratory

Duration: FY2021-23

TPOC (Technical Point of Contact): Kort Bowman, kort.bowman@inl.gov

Federal Manager: Daniel Nichols



SEM micrograph of HTIR-TC cross-section

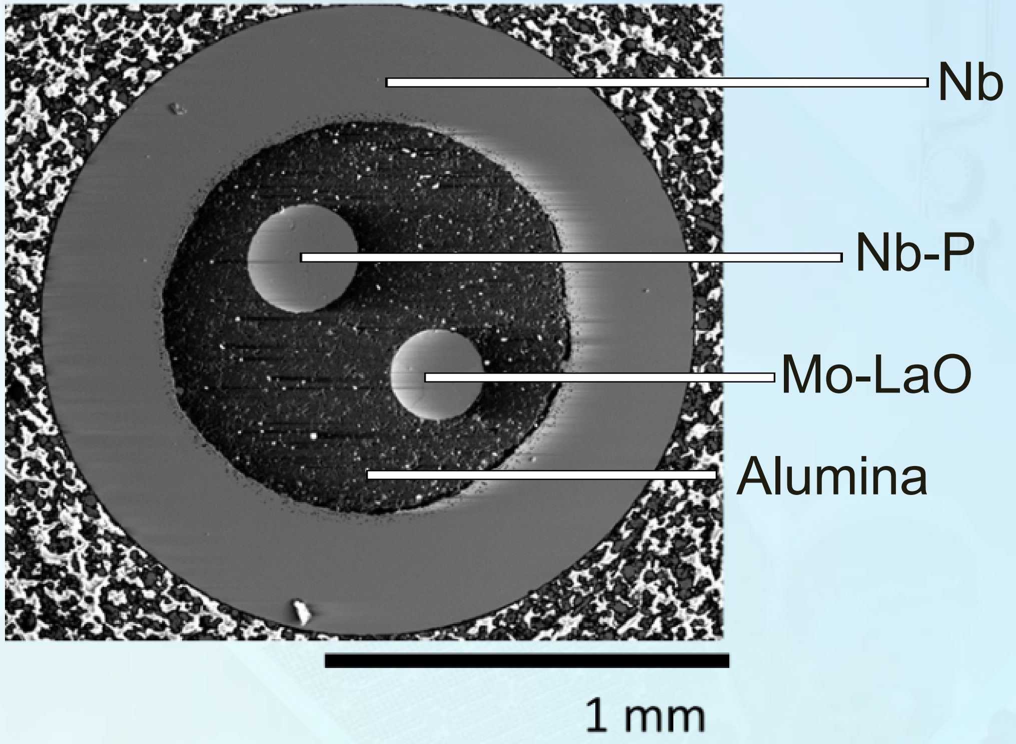
Schedule: The thermocouple activities:
- ***Characterize influence of traditional heat treatments and Joule heating on the stabilization of the HTIR-TCs***
  - During the stabilization heat-treatment a secondary Nb_3P phase along with an alumina/Nb-P interaction region forms. With Joule heating an interaction forms between the alumina/Mo-LaO.
- ***Performance assessment of commercial TCs for nuclear applications***
  - A Halden-Conroy model was used to model the empirical drift due to transmutation for type C, S, and K.
- ***Characterize influence of alternative heat-treating methods, Joule heating, on stabilization of HTIR-TCs.***
  - A model using COMSOL was made to evaluate the temperature within the thermoelements during joule heating.

Technical Impact

- In order to decrease nuclear innovation time, robust, in-pile measurement techniques and sensors must be developed.

Gen IV: Very High Temperature Reactor
Core Outlet Temperature[3]: >1000°C

Thermocouple[2]	HTIR-TC	Type K	Type N	Type B
Materials	Mo vs. Nb	Chromel vs. Alumel	Nicrosil vs. Nisil	Pt – 30%Rh vs. Pt – 6%Rh
Temp Range	0 – 1700 °C	-270 – 1260 °C	-270 – 1260 °C	0 – 1700 °C
Cost	~$250/ft	~$30/ft	~$50/ft	~$250/ft
Radiation Tolerance as compared to HTIR-TC		1/10th	1/4th	1/100th

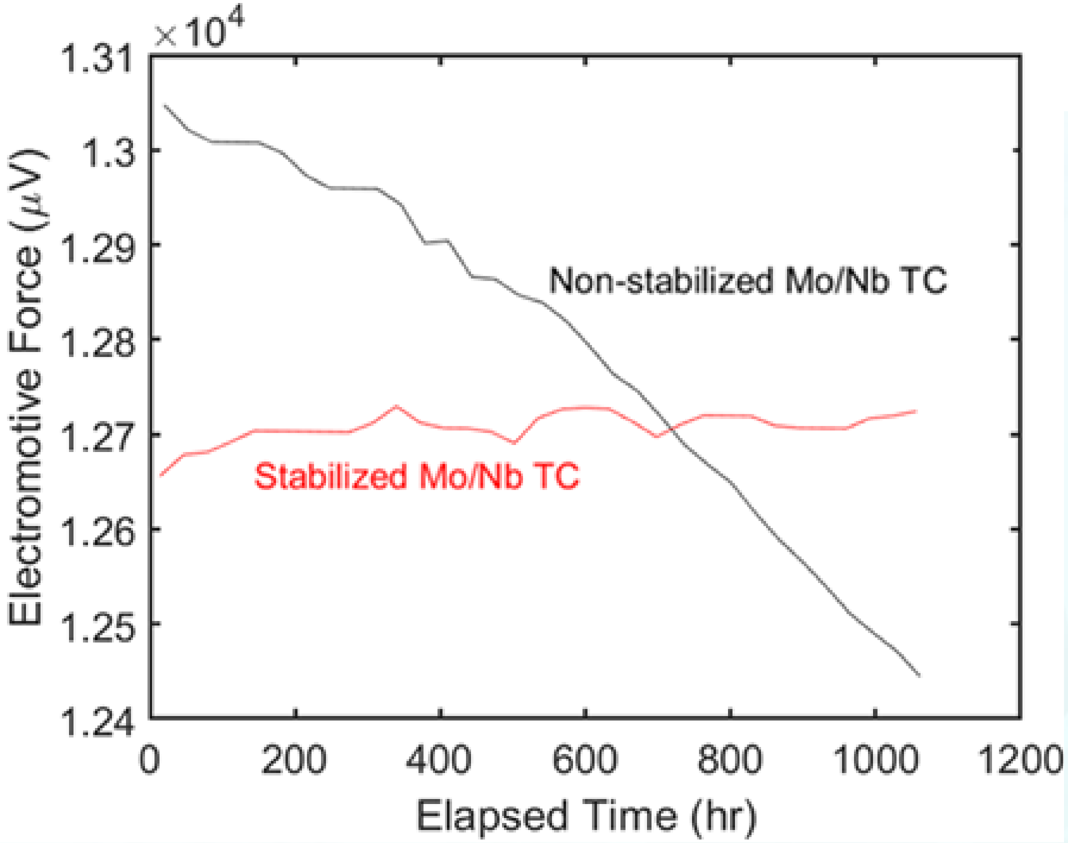


SEM micrograph of HTIR-TC cross-section

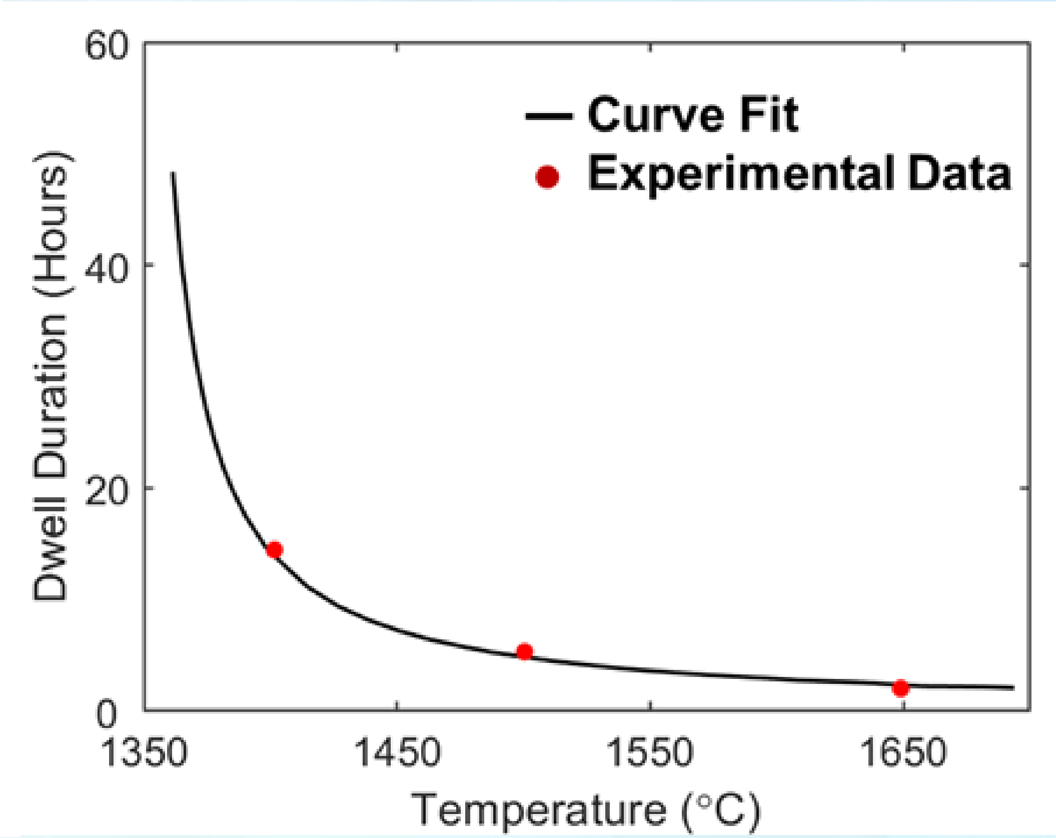
HTIR-TC combines the high temperature of the Type B thermocouple with the irradiation tolerance of Type N & K.

2. Data courtesy of Dr. Skifton, INL
3. Murty K., Charit I., An Introduction to Nuclear Materials. Vol 1, Wiley-VCH, 2013, Weinheim, Germany.

Technical Impact



Signal measured by Mo/Nb thermocouples during 1000-hour 1100 °C test [4].



Hours to stabilize as a function of heat treatment temperature for HTIR-TCs.

4. Bong Goo Kim, Joy L. Rempe, Jean-François Villard & Steinar Solstad (2011) Review Paper: Review of Instrumentation for Irradiation Testing of Nuclear Fuels and Materials, Nuclear Technology, 176:2, 155-187, DOI: 10.13182/NT11-A13294

Results and Accomplishments

Experimental:

- Investigated and compared the effects of Joule heat treatment vs. traditional heat treatment on HTIR-TCs through:
  - Mechanical properties
  - Electrical properties
  - Microstructural evolution and chemical stability
- Provided optimization procedures and suggestions for path forward for stabilization heat treatment strategies.
- Developed a thermocouple drift model based on the empirical data in open literature.

Presentations:

1. "Effect of Processing on Nuclear Thermocouple Stability for In-Pile Temperature Sensing." S. Riley, K. Holloway, R. Skifton, B.J. Jaques. Advancements in Nuclear Instrumentation Measurement Methods and their Applications (ANIMMA) presentation. Real Collegio, Italy. June, 2023.
2. The Influence of Heat Treatment on the Stability of High Temperature Irradiation Resistant Thermocouples." S. Riley, K. Holloway, A. Bateman, R. Skifton, B.J. Jaques. Presented at the FuNZI 2023 Conference. March 30-31, 2023.

Other:

- MaCS Seed Grant ($5k), "Influence of heat treatment on the grain stability of Mo-LaO" in HTIR-TCs
- Scott Riley (GRA) interned at INL for 3 months to optimize HTIR-TC builds and stabilization heat treatments via Joule heating
- Scott Riley presented his dissertation proposal on the Stability of Nuclear Thermocouples.

Publications:

1. "Influence of Microstructure and Phase Morphology on the Stability of High temperature Irradiation Resistant Thermocouples." S. Riley, K. Holloway, A. Bateman, R. Skifton, B.J. Jaques. Materials Today Communications. 2023. *In Press*.
2. "Nuclear Thermocouple Drift models." S. Riley, R. Skifton, B.J. Jaques. *In Draft*.
3. "Methods for Temperature and Thermal Conductivity Measurements in Extreme Environments." K. Wada, S. Riley, B.J. Jaques, D. Estrada. *In Draft*.
4. "Influence of Joule heating on the Stability of High temperature Irradiation Resistant Thermocouples." S. Riley, K. Holloway, A. Bateman, R. Skifton, B.J. Jaques. *In Draft*.

Why Joule Heating?

Challenges of the traditional stabilization heat treatments of HTIR-TCs:

>24 hours of furnace time

>1400 °C

Limited to hot zone length of furnace

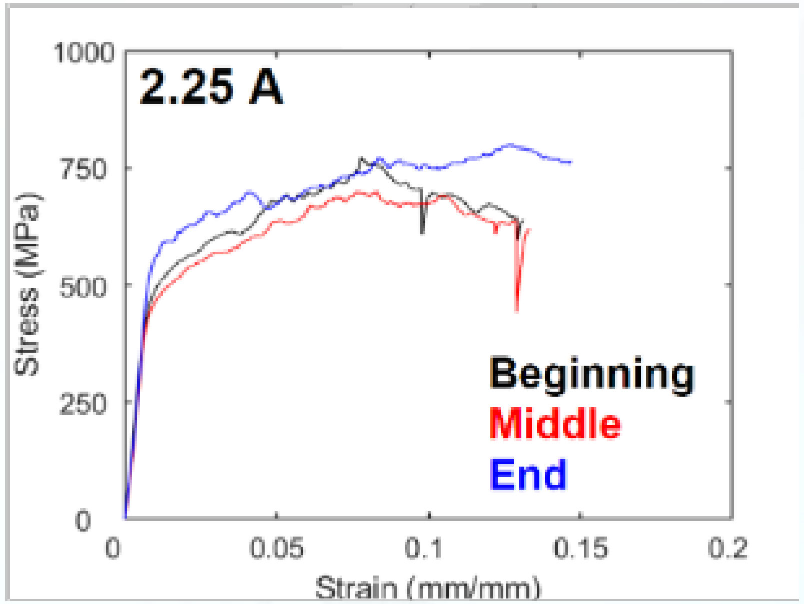
Longer times at elevated temps increases oxygen uptake resulting in reduced ductility

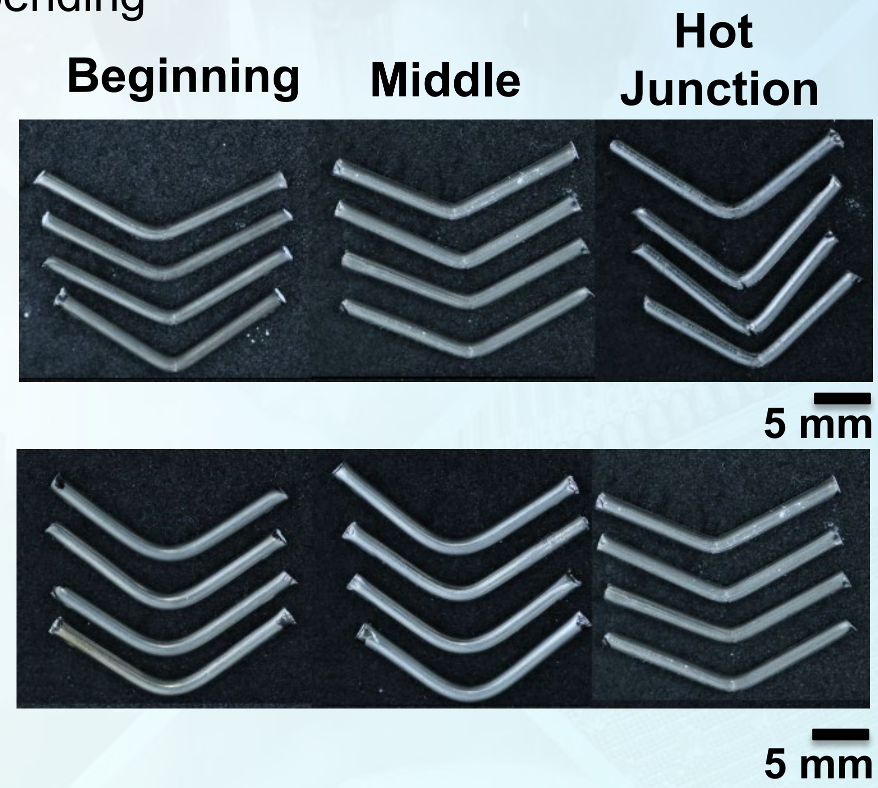
Requires expensive processing equipment and large footprint

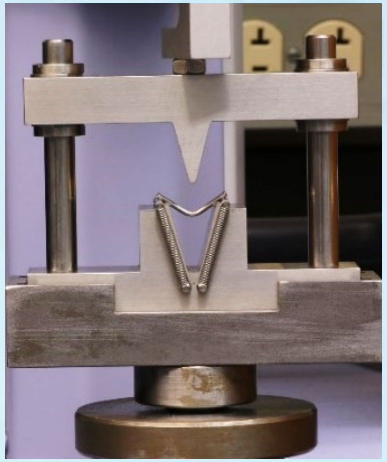
Difficult to industrialize

Results and Accomplishments: Joule heating-Ductility

Effect of current on HTIR-TC ductility via 3-point bending





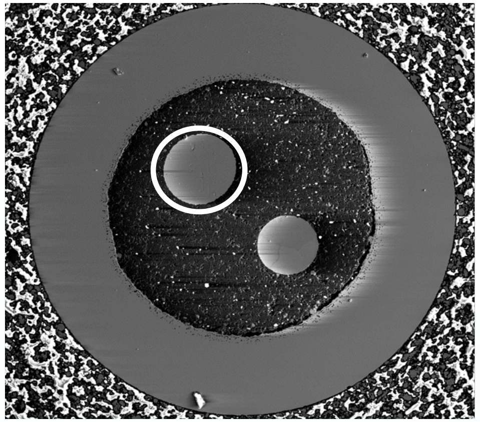


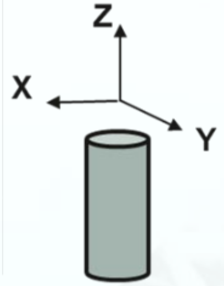
Sample	Average Yield Stress (MPa)	Average Modulus (Gpa)
2 A Beginning	455 ± 27	82 ± 4.9
2 A Middle	405 ± 24	81.5 ± 4.8
2 A Hot Junction	316 ± 18	77.5 ± 4.6
2.25 A Beginning	494 ± 29	72.0 ± 4.3
2.25 A Middle	473 ± 28	75.8 ± 4.5
2.25 A Hot Junction	505 ± 30	81.7 ± 4.9

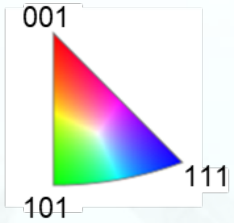
Finding: Joule heated HTIR-TCs maintained ductility after heat treatment under vacuum using 2 and 2.25 A.

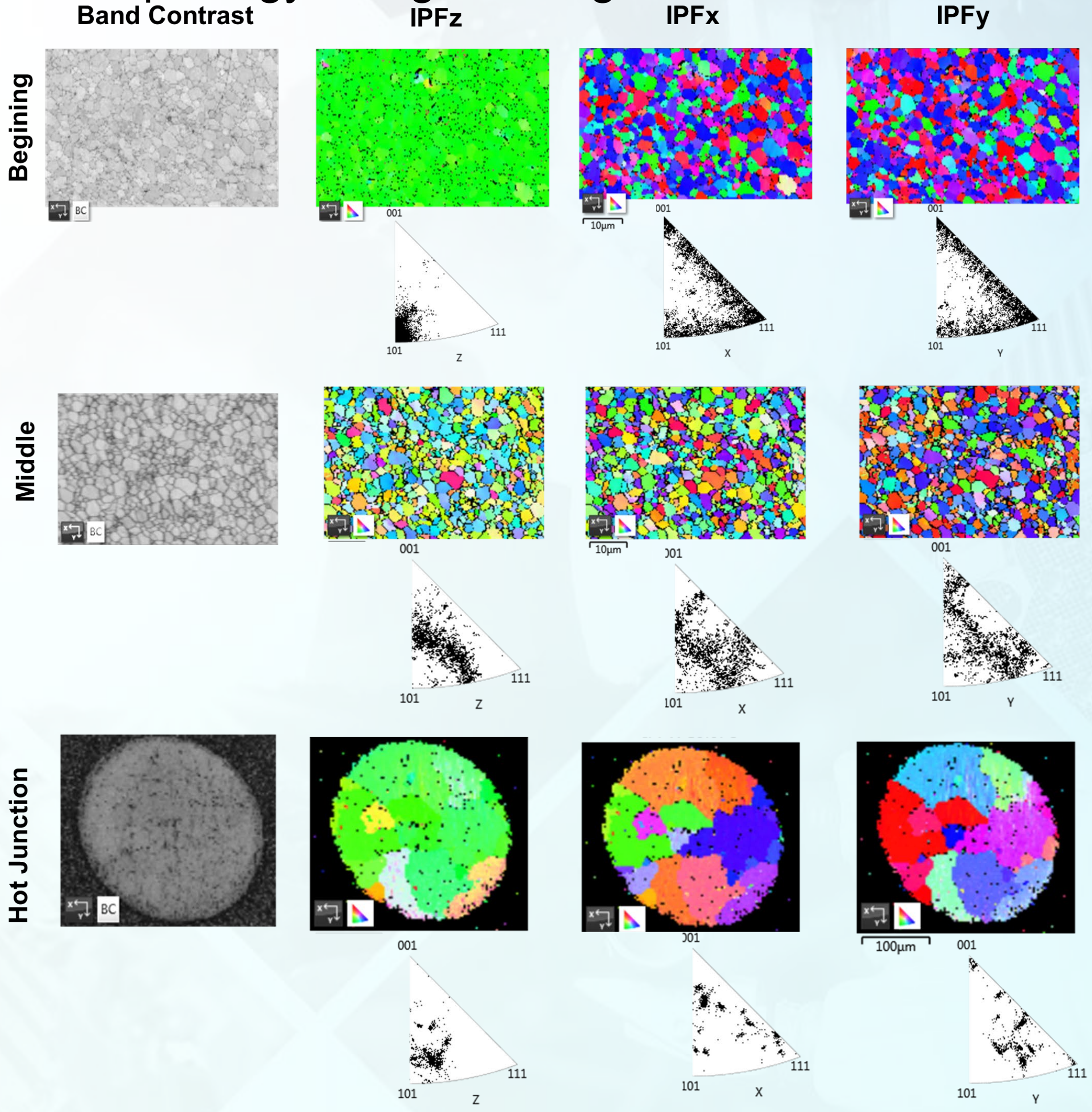
Results and Accomplishments: Joule heating-Crystal texture

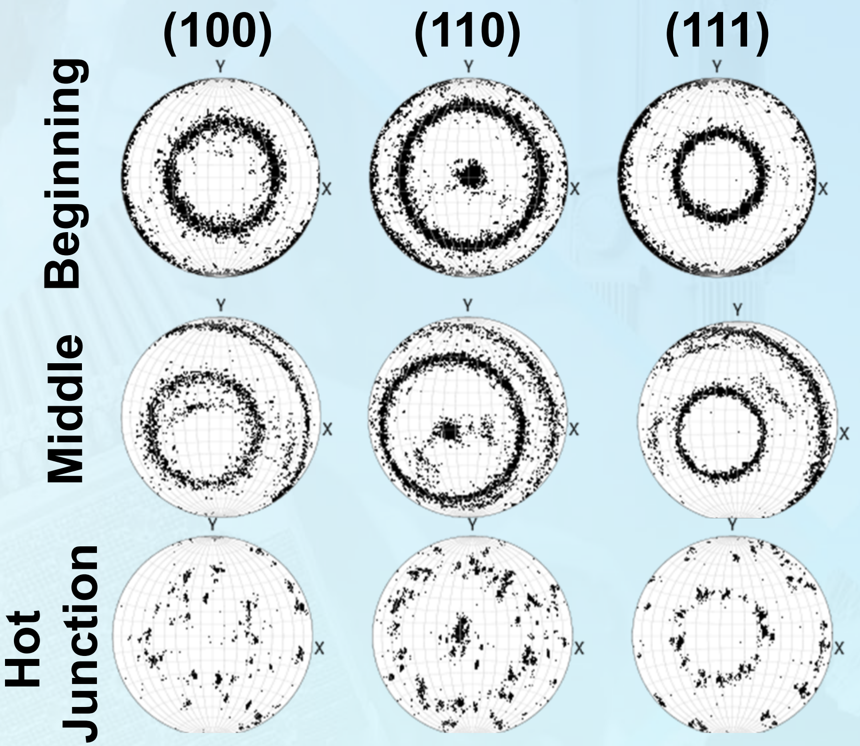
Effect of 2 A current on grain morphology along the length of a HTIR-TC.







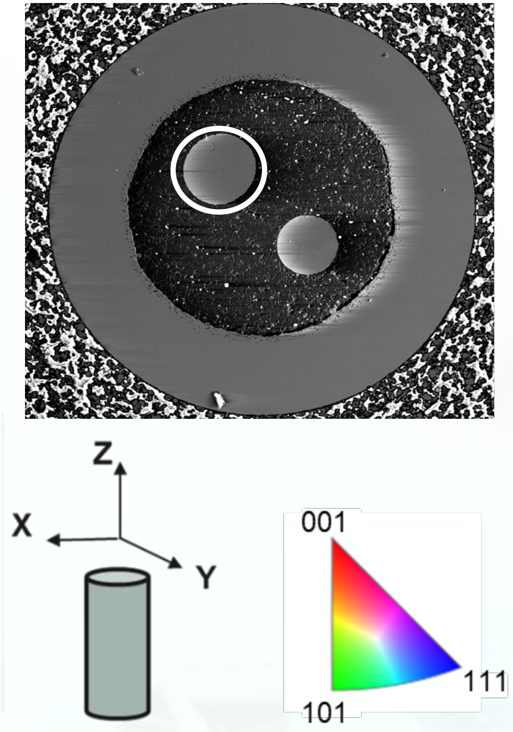


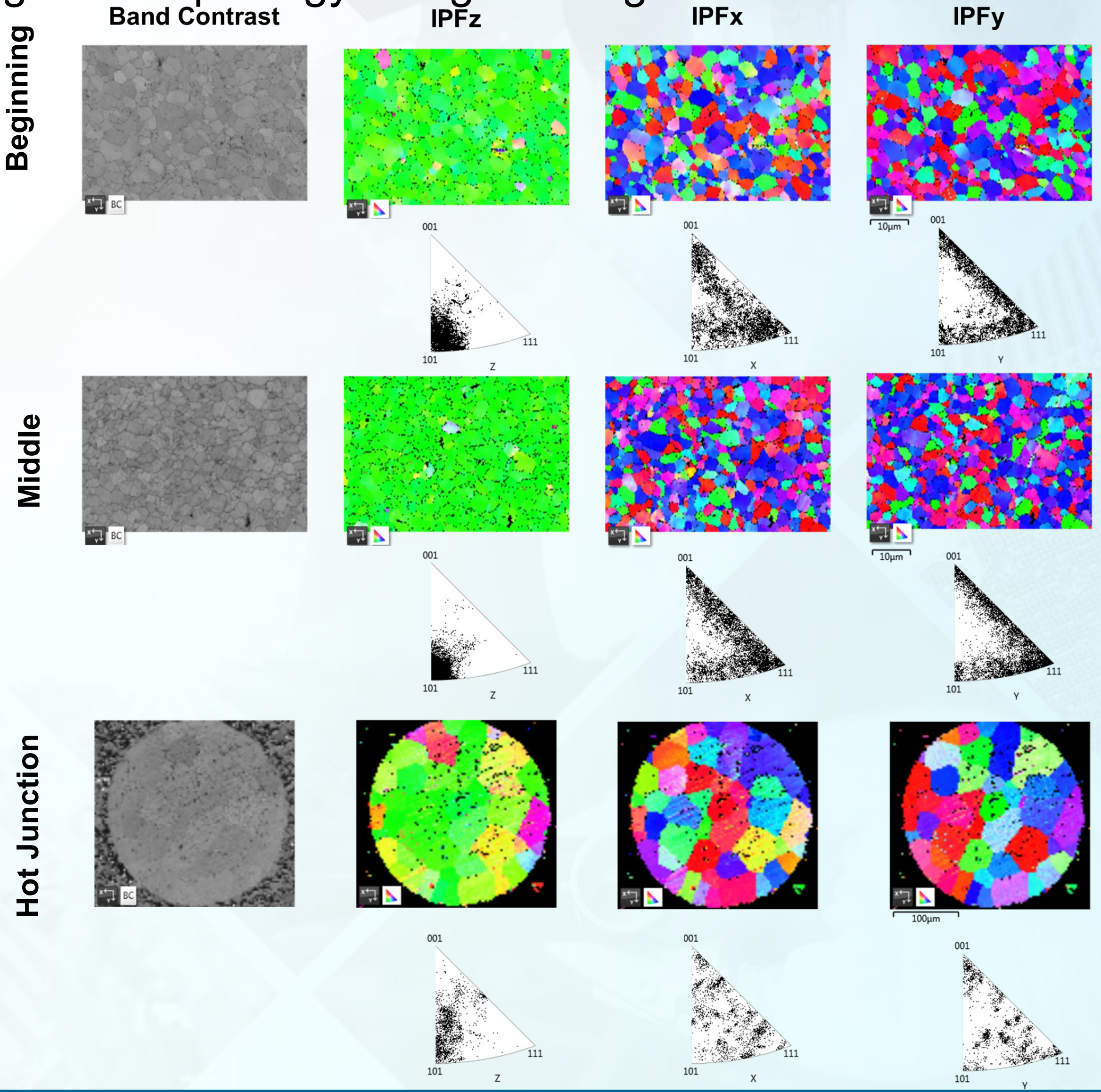


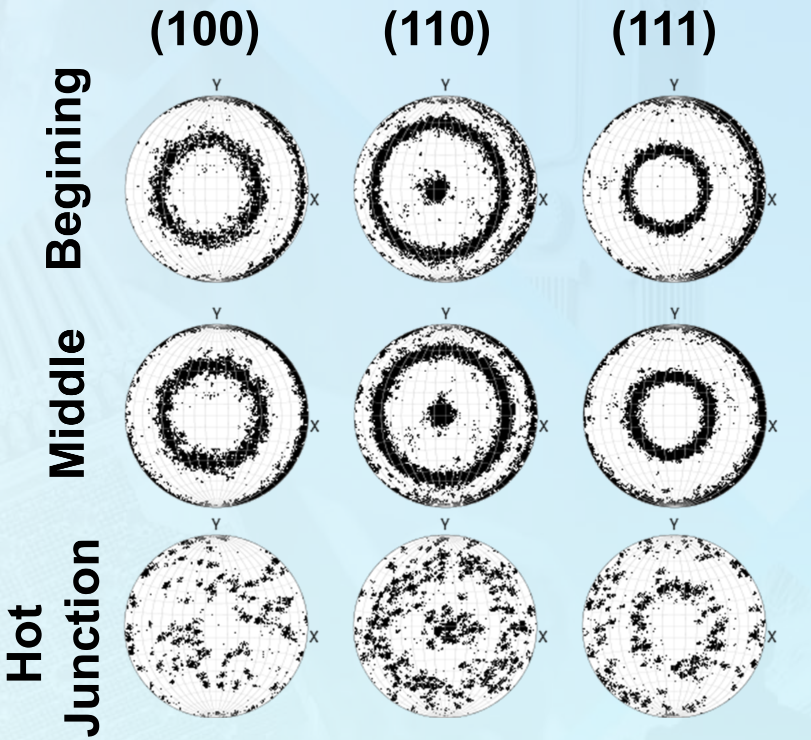
Findings: The HTIR-TCs maintained a 110//drawing axis fiber texture after Joule heating and a subsequent hold for 50 hours at both 1250 and 1450 °C.

Results and Accomplishments: Joule heating-Crystal texture

Effect of 2.25 A current on grain morphology along the length of a HTIR-TC.



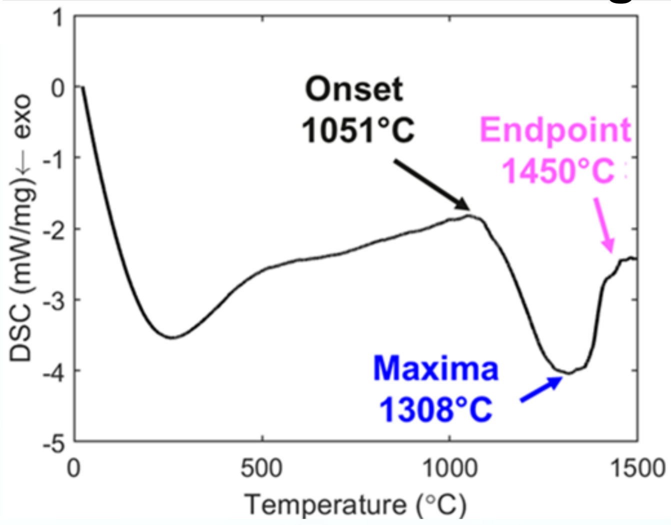


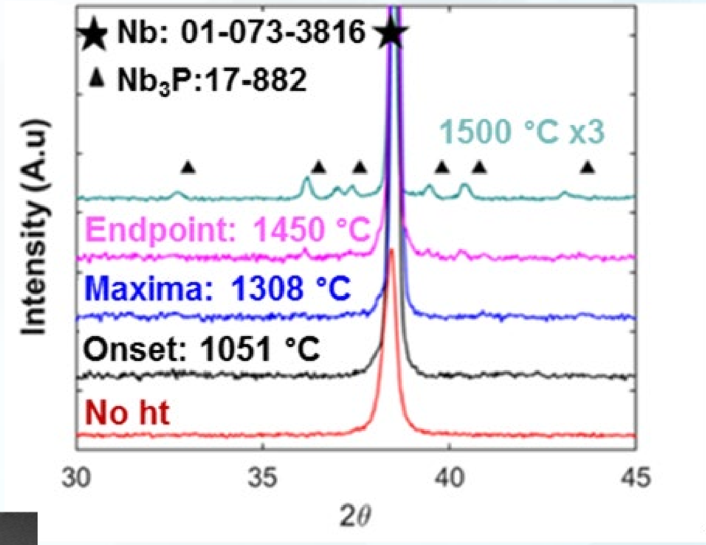


Findings: The HTIR-TCs maintained a 110//drawing axis fiber texture after Joule heating and a subsequent hold for 50 hours at both 1250 and 1450 °C.

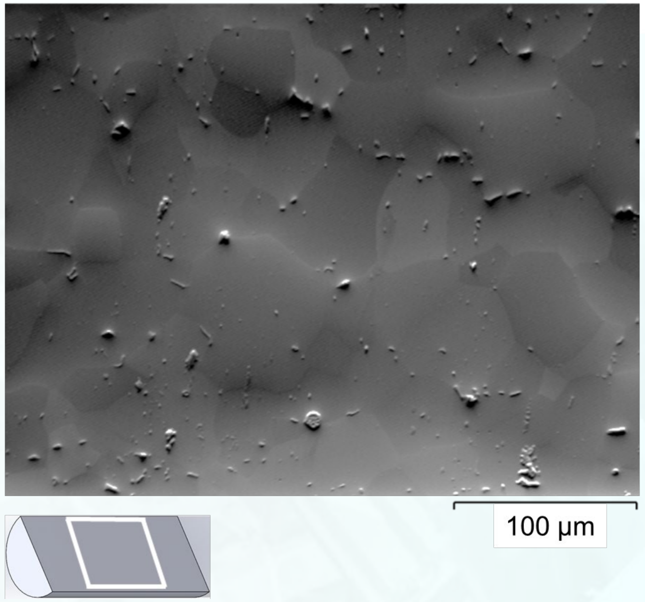
Results and Accomplishments: Joule heating-precipitation

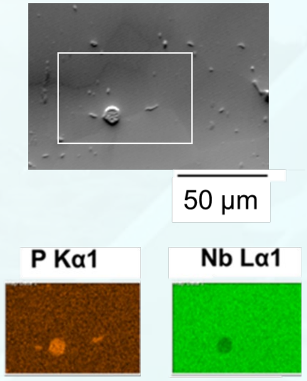
Influence of Joule heating on chemical stability of Nb-P

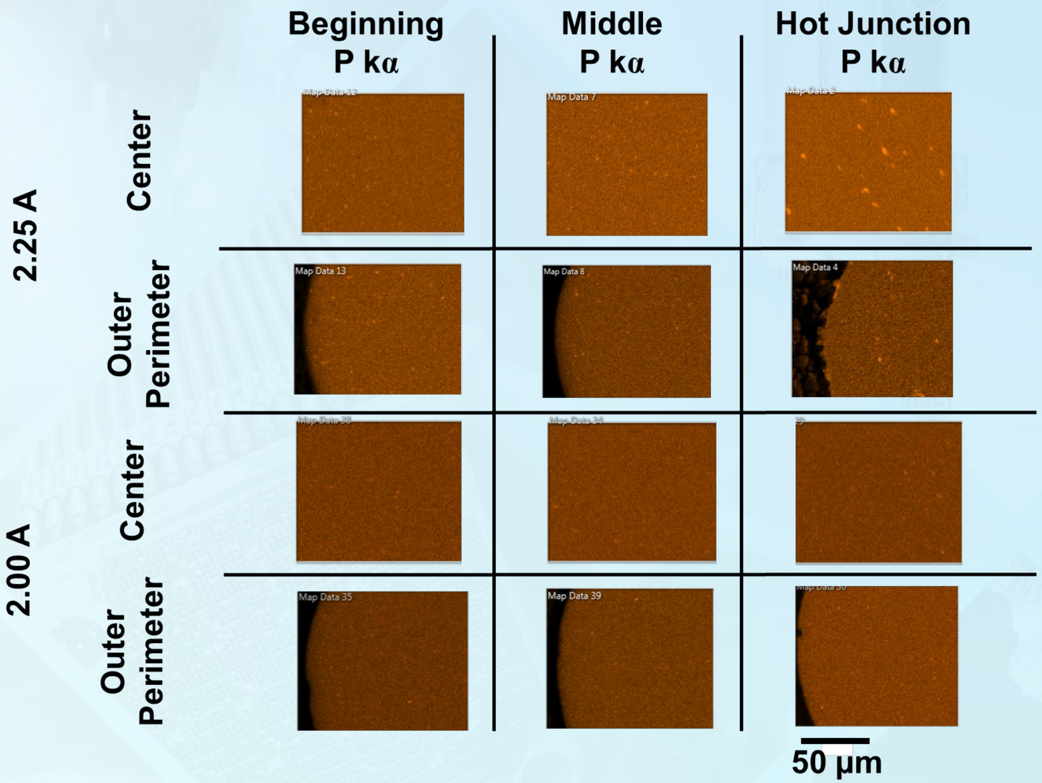




1500 °C
1 hr





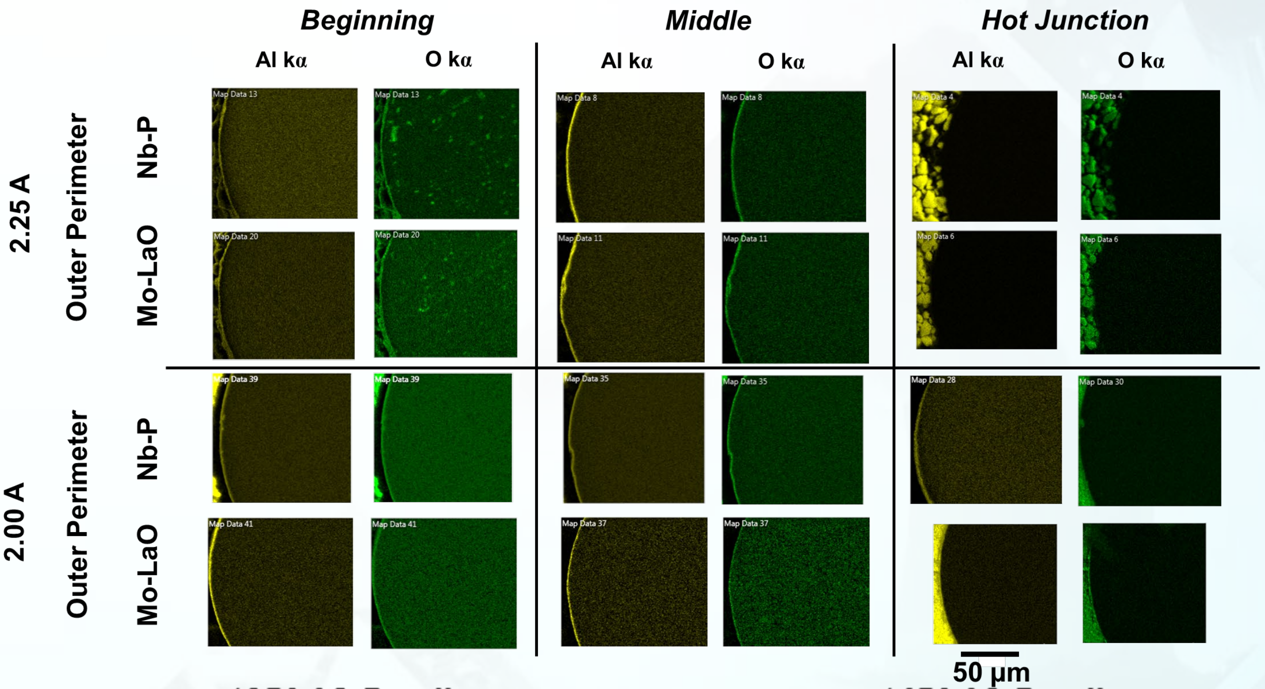


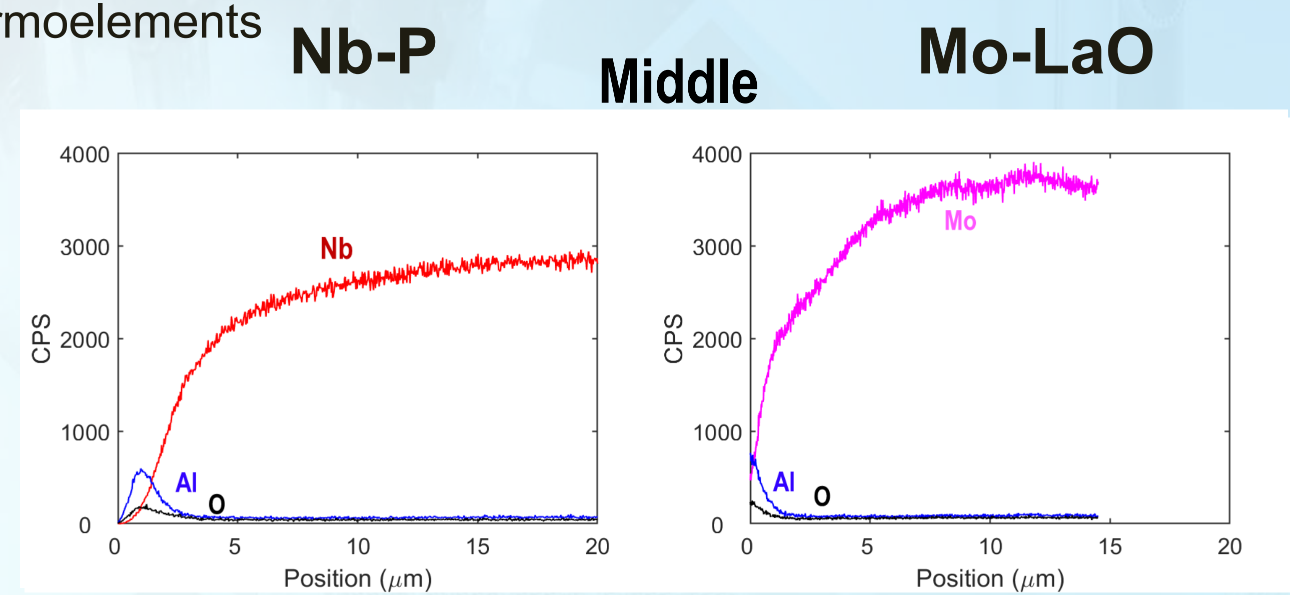
Findings: Joule heating resulted in coarsening of the Nb_3P phase within the Nb-P thermoelement.

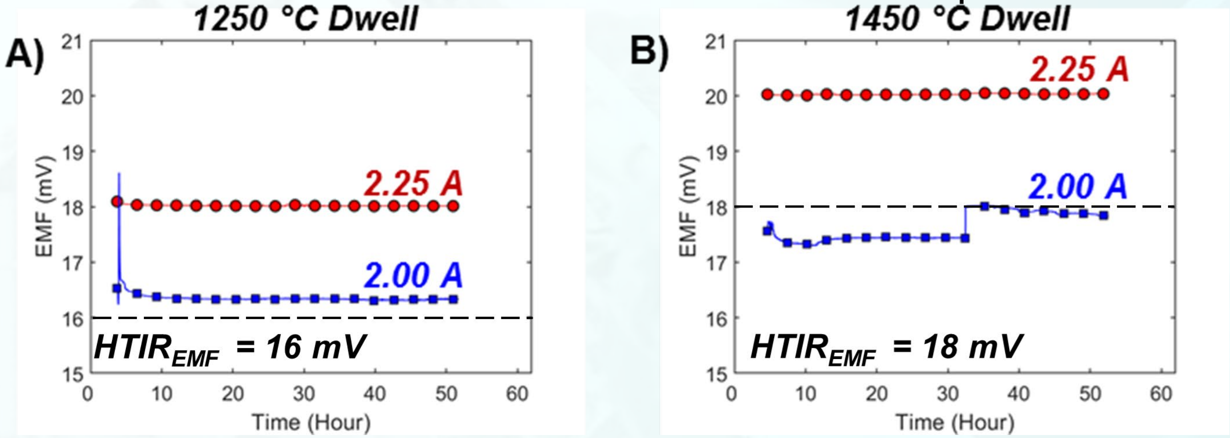
Impact: A stabilization heat treatment is necessary to reduce Nb-P thermoelement drift during operation by stabilizing the formation of the Nb_3P phase.

Results and Accomplishments: Joule heating-Al diffusion

Chemical Stability of insulation with Mo-LaO and Nb-P Thermoelements





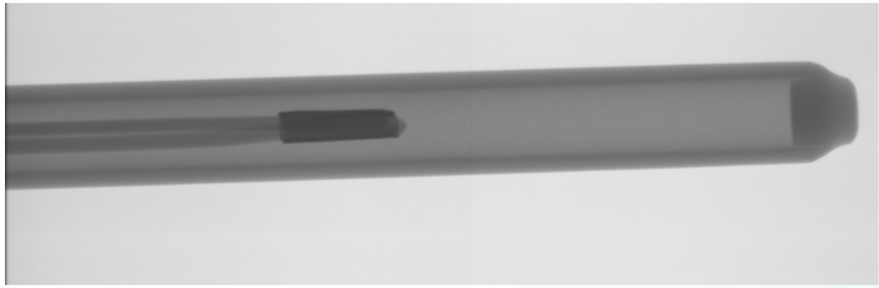


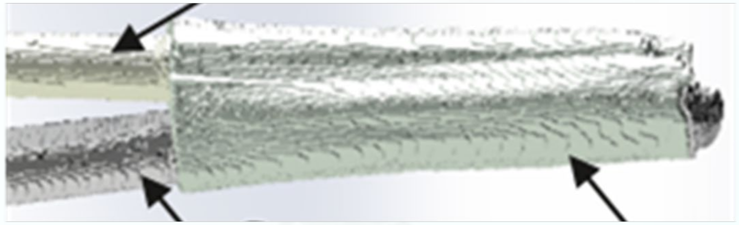
Findings: During joule heating alumina reacts with both the Nb-P and Mo-LaO where it dissociates and diffuses through the thermoelements; however, once it is formed, it appears to be stable.

Impact: Stabilization heat treatment induces mass diffusion and therefore reduces driving force for continued diffusion (contributing to thermal drift) especially when operated at lower than heat treatment temperatures.

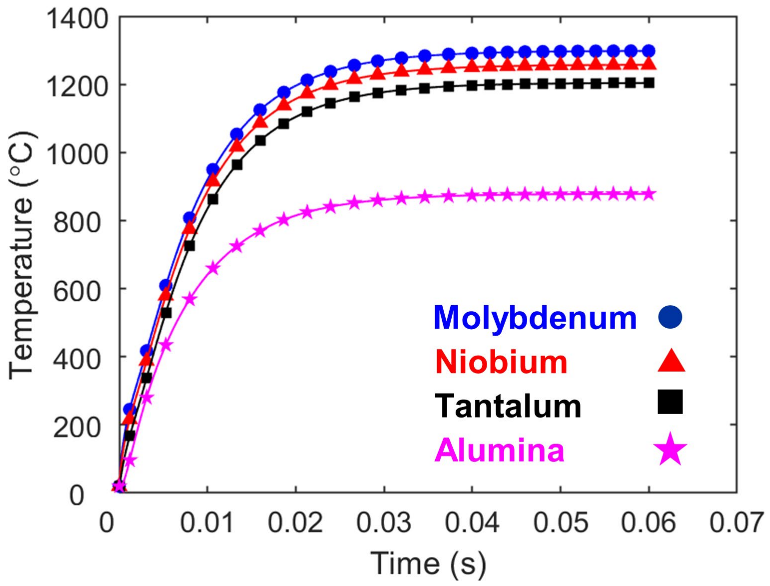
Results and Accomplishments: Joule heating-Modeling via COMSOL

Modeling of Alternative Heat Treatment Methods of HTIR-TCs





Joule heating model of HTIR-TC hot junction cross-section in COMSOL[1]



Preliminary process:

Applied current: 2 A

Surface Temperature of sheath: 600 °C

Under vacuum: Radiative heat loss

Physical properties of HTIR-TC components

HTIR-TC Component	Average Temperature (°C)	
	Junction	Bulk
Mo-LaO	1260	1063
Nb-P	1257	1136
Ta	1204	N/A
Al_2O_3	878	873
Nb sheath	645	651

Findings: Joule heating using a small current can stabilize the entire length of the thermoelements in a short duration

Impact: Reduced time to market, reduced processing uncertainties, and significant reduction in processing footprint

1. Multiphysics, C., 1998. Introduction to COMSOL multiphysics extregistered. COMSOL Multiphysics, Burlington, MA, accessed Feb, 9, p.

Concluding Remarks

HTIR-TCs marry the high temperature capability with the stability necessary for nuclear applications.

- Stabilization heat treatments are necessary to reduce (not quite eliminate) thermal drift
- The ductility of the HTIR-TC build can be retained by:
  - Joule heating resulting in rapid heat treatments and low sheath surface temperatures
- Nb-P thermoelement is the primary culprit in HTIR-TC drift due to:
  - Precipitation of Nb_3P
  - Nb – Alumina interaction region that results in dissociation and diffusion of aluminum throughout the thermoelement.
- Joule heating induced an interaction between the alumina insulation and the Mo-LaO thermoelement.
- However, these phenomena can be stabilized by a heat treatment >100 °C above the max service temperature

- This work was/will be disseminated through:
  - 2 journal publications
  - 2 manuscripts that are in draft
  - 4 conference presentations
  - 1 upcoming conference presentation
  - 1 ASI newsletter
  - Scott Riley's PhD Dissertation

Future Work

- Optimize the stabilization heat treatment via Joule heating through mechanical and electrical properties, chemical stability, and microstructural evolution.
- Evaluate the influence of forced convective cooling on the mechanical, chemical, and microstructure of the HTIR-TCs during Joule heating.
- Extended period high temperature drift

Neutron Generators and the Development of a Neutron Sciences Laboratory

Advanced Sensors and Instrumentation (ASI)

31 October, 2023

BSU: Allyssa Bateman (Research Associate) and Brian J. Jaques (PhD, PE)
INL: Pattrick Calderoni, Kort Bowman, Troy Unruh, Brenden Heidrich
Boise State University (MSE) and Idaho National Laboratory

Project Overview

Through the DOE-NE University Infrastructure program, Boise State University has access to two Thermo-Scientific P385 neutron generators.

- Valued at $230k
- Deuterium-Deuterium (D-D) head with 2.5 MeV neutrons
- Deuterium-Tritium (D-T) head with 14.1 MeV neutrons

End Goal: Operate neutron generators at BSU

Intermediate Goals:

- ✓ Understand the facility, licensing, and safety requirements for operating neutron generators on campus
- ✓ Amend BSU’s current NRC license to include neutron generators
- ✓ Identify space on campus and funding for a neutron science lab
- ✓ Design shielding and safety framework to operate neutron generators
- Build a neutron science laboratory
- Add analytical equipment
- Do neutron science!

Technology Impact

- Support programs such as ASI and AMMT by providing neutron irradiation to screen nuclear-relevant materials, sensors and equipment.
    - Rapid, low-cost testing allows for design iteration before reactor studies
    - Flexible experimental set up for in-situ testing and post-testing characterization

- Grow the next generation of nuclear scientists by providing hands-on experiences with neutron physics, health physics, radiological materials handling, etc.
    - Enhance coursework across multiple disciplines
    - Enhance research for students across Idaho and the NSUF network

- Stakeholders: DOE, INL, NEUP, NSUF, BSU

Project Timeline

2018

- Neutron generators are available to BSU through the DOE-NE University Infrastructure program
- Neutron Detectors is written into the Nuclear Instrumentation work package of I2

2019

- BSU researchers travel to University of Michigan's Neutron Science Lab to discuss facility design
- Campus leadership identifies a potential location for a Neutron Sciences Lab at BSU
- Initial shielding modeling via MCNP
- BSU submits initial NRC license amendment request for storing neutron generators

2020

- Continued efforts to identify a lab location on campus and model effective shielding
- BSU submits NEUP CINR GSI proposal to build shielding for neutron lab – *not awarded*

2021

- NRC approves license amendment for storing neutron generators
- BSU agrees to fund lab build out and shielding in Micron Center for Materials Research

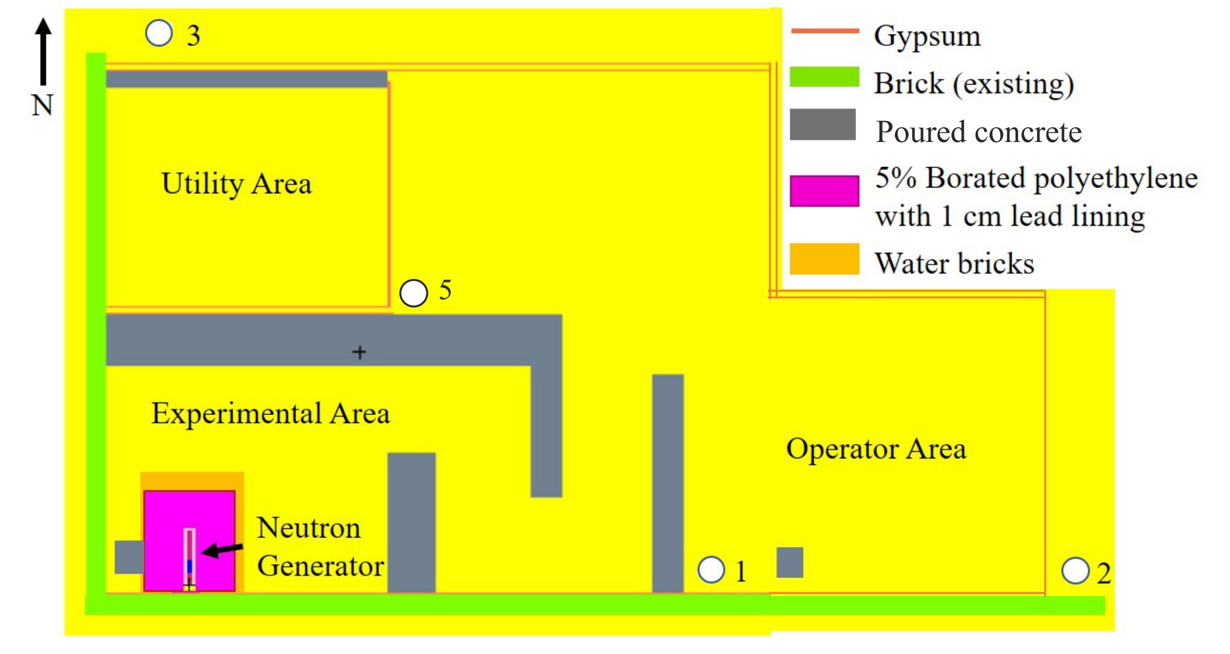
2022 -

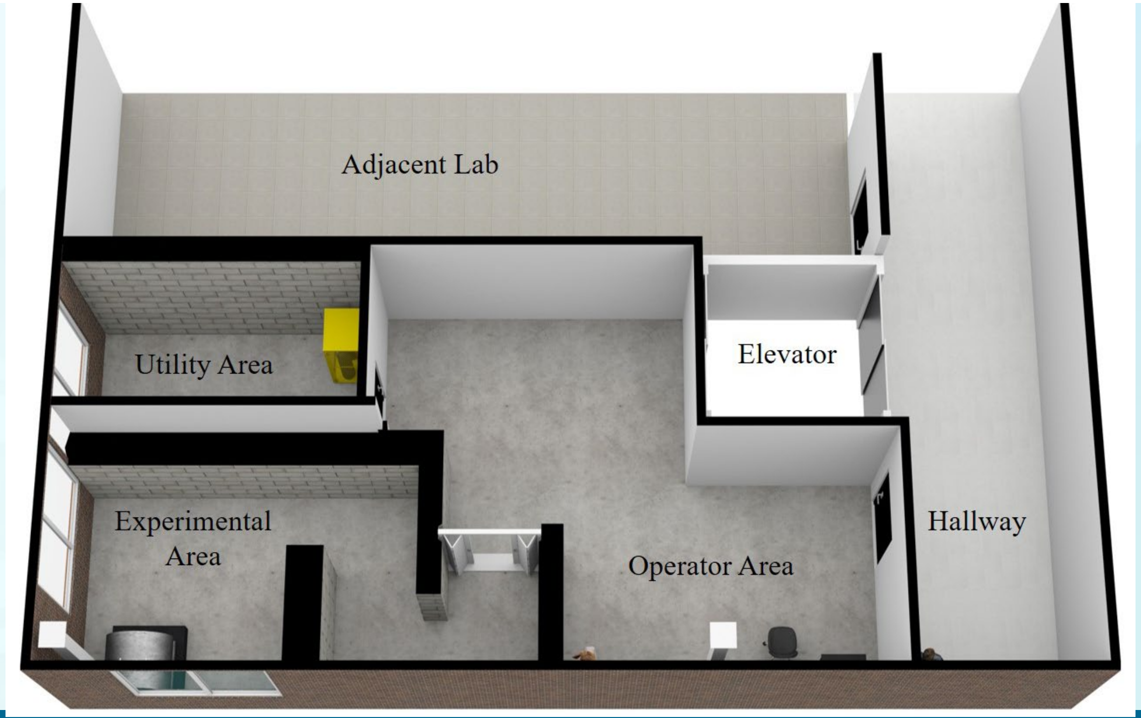
- BSU submitted NEUP CINR GSI proposal to equip the Neutron Sciences Laboratory – *not awarded*

Results and Accomplishments

- BSU designated lab space for the Neutron Sciences Lab
- MCNP used for exposure calculations and lab design
- BSU committed $879k to finish the lab space and build shielding.
- Neutron Generators arrived to BSU in August, 2023
- Construction began in October, 2023
- Lab opening in August or 2024

Tally location	Dosage rate (mrem/hr)	NRC limit (mrem)	Allowable operator time (hrs/year)
1. Operator 1	5.792	5000	863
2. Non-worker hallway	0.234	100	427
3. Non-worker adjacent	1.068	100	94
4. Non-worker below	0.794	100	126
5. Operator 2	34.979	5000	143





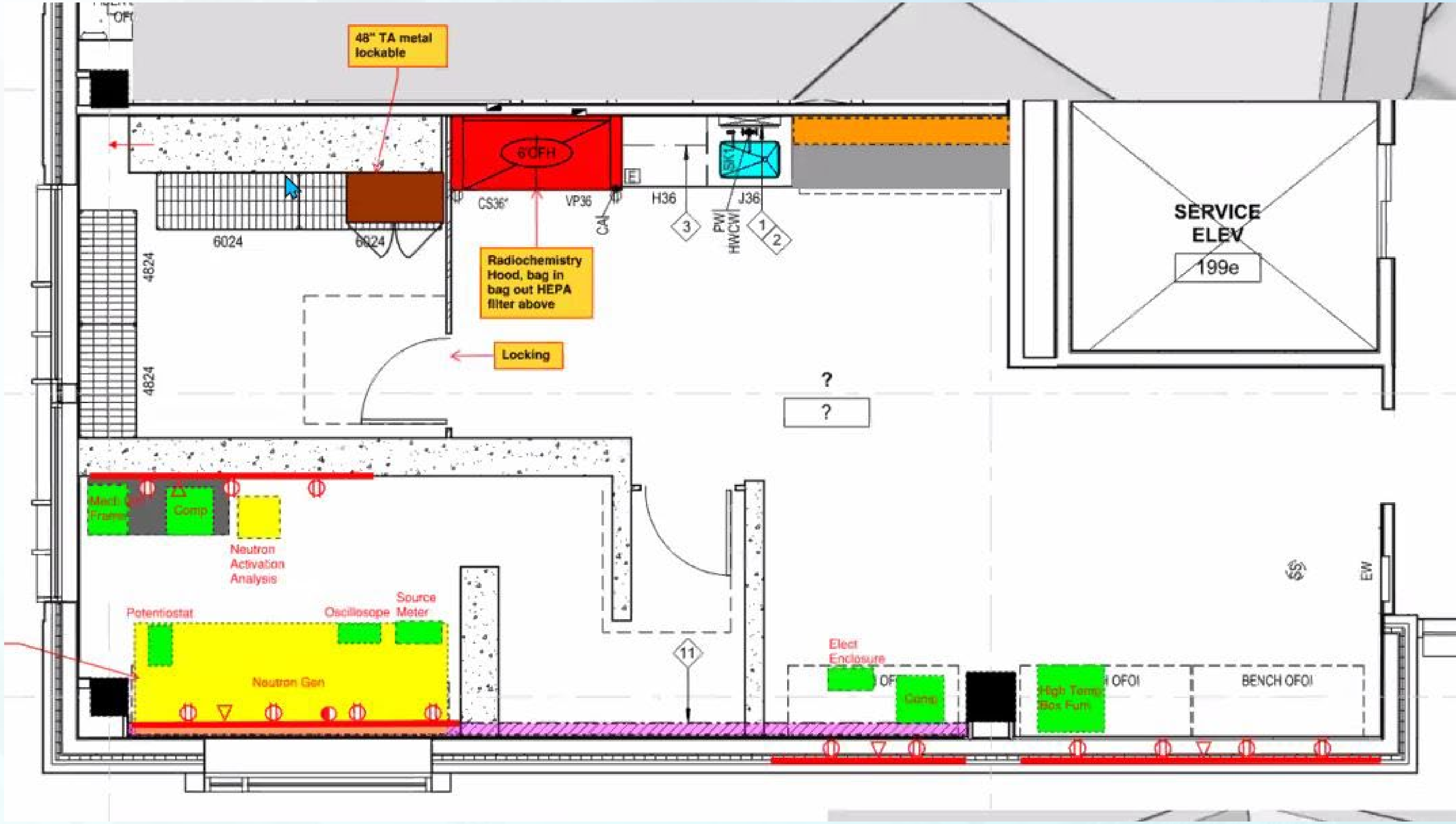
Results and Accomplishments

Final construction drawings

- Expected construction completion: Summer/Fall 2024

Included equipment:

- Secure storage and "cooling" area for neutron generators and activated samples
- Radiological fume hood
- Security equipment including locks and cameras



Concluding Remarks

- DOE-NE has donated a neutron generator with both D-D and D-T heads (valued at $230k) to Boise State.
- NRC license amended and generators are currently on campus
- Boise State University has committed $879k toward a neutron science laboratory, with construction completion in Fall of 2024.

AML Advanced Materials Laboratory

Thank You

Brian J. Jaques, PhD, PE
Assistant Professor | Micron School of Materials Science and Engineering, BSU
Director | Advanced Materials Laboratory, BSU
Program Manager | Advanced Sensor and Instrumentation at BSU
Joint Appointment | Idaho National Laboratory
Fellow | Center for Advanced Energy Studies
+01.208.484.0597
BrianJaques@BoiseState.edu

AML Advanced Materials Laboratory