



Boise State University Supporting Activities

**Advanced Sensors and Instrumentation (ASI)
Annual Program Webinar**

October 24 – 27, 2022

PM for ASI Activities at BSU and Assistant Professor:
Brian J. Jaques, PhD

Boise State University, Micron School of Materials Science and Engineering

Program Overview: BSU Supporting Activities

To establish research capabilities, analysis methods, sensor and materials optimization, and provide sensor fabrication support with the INL. The research activities at BSU are continuing to use advanced manufacturing processes in combination with a foundational materials science and engineering approach that includes modeling and simulation. The research program is rapidly advancing the design and development of sensors at BSU in close collaboration with the INL. The program is significantly contributing to the DOE-NE mission. The sensors focused in this research are designed for *in-situ* and in-pile applications to be used in instrumented experiments.

SENSOR R&D THRUSTS	INL	BSU					Deliverables
	TPOC	TPOC	FACULTY	STAFF	GRA	UGRA	
NUCLEAR THERMOCOUPLES	Richard Skifton	Brian Jaques	1	-	1	1	Create a drift model on commercially available TCs, including types K, N, C, and B, then implement on HTIR-TC designs during AGR 5/6/7 test data. Develop a mechanistic understanding (Using MSE) of HTIR-TC stabilization heat treatment optimization and extended time, exposures at elevated temperatures.
LINEAR VARIABLE DIFFERENTIAL TRANSFORMERS (LVDT)	Kurt Davis	Zhangxian (Dan) Deng	1	-	-	2	Work with the INL to design and fabricate a testing assembly for LVDTs, optimize the testing procedures, and obtain results with the novel fixture.
ACOUSTIC SENSORS	Joshua Daw	Zhangxian (Dan) Deng	2	-	2	2	Develop magnetostrictive and piezoelectric ultrasonic waveguide thermometer (UT) that can measure in-pile temperature through speed of sound. Focus on Galfenol-based thermometers. Enhance the thermometer performance using FEA techniques. Validate the thermometer at elevated temperatures, and investigate new options of magnetostrictive waveguides
LINE SOURCE	Austin Fleming	David Estrada	1	-	1	-	Develop FEA and analytical model for the frequency response of an advanced (miniature, optimized material and fab) needle probe design. Assist in the fabrication and characterization of the design
PRINTED SENSORS FOR HARSH ENVIRONMENTS	Mike McMurtrey	Dave Estrada	3	1	3	2	Improve and optimize the printing process of piezoelectric inks (compatible with aerosol jet printing (AJP)) for AM of piezoelectric surface acoustic wave (SAW) devices for sensing in harsh environments. Develop heterogenous integration methods to printed sensors. Improve and optimize printed capacitive strain gauges (CSGs) and increase the robustness of printed sensors at elevated temperatures. Develop strain validation techniques (DIC, RSGs, etc). Quantify the adhesion strengths of printed films using destructive techniques
LINE SOURCE FOR THERMAL PROPERTIES	Austin Fleming	Dave Estrada	1	-	1	-	Develop an axisymmetric, multi-layered, finite element and analytical model for a novel needle probe design
NEUTRON GENERATOR FOR SENSOR DEVELOPMENT	Troy Unruh	Brian Jaques	1	1	-	-	Finalize documentation to install at BSU (ID suitable location and controls, modeling of appropriate shielding, Safety procedures, monitoring equipment, training documentation)

Project Overview: Nuclear Thermocouples

OVERVIEW

Purpose:

Real-time temperature measurement is arguably the most important operational parameter to measure for the characterization of irradiation experiments and the control of power plant systems. The high temperature irradiation resistant thermocouples (HTIR TCs) have been extensively researched over the last decade and models have been developed to predict their Seebeck Coefficient, Electromotive force, and decalibration.

Research will be completed to apply these models and applications to other prevalent commercially available thermocouples, including: type-K, N, and B. Appropriate data sets for each must be presented and compared to the HTIR-TC Drift Model found in INL/EXT-21-63346

Research will also focus on developing an understanding of the mechanistic behavior of HTIR-TCs resulting in decalibration when they are exposed to excessively high temperatures and neutron fluxes for extended periods of time.

Objectives:

- Use the HTIR-TC drift model on other commercially available thermocouples including Type-K, N, B
- Develop mechanistic understandings of HTIR-TC drift and stabilization

DETAILS

Principal Investigator: Brian Jaques (BSU)
Richard Skifton (INL)

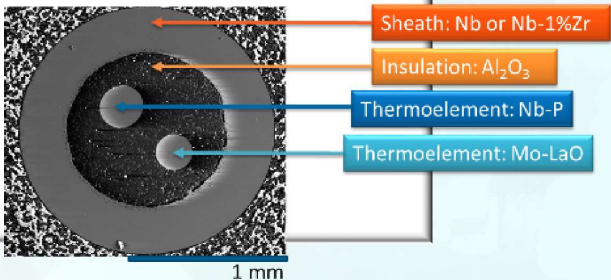
Institution: Boise State University

Collaborators: Idaho National Laboratory

TPOC (Technical Point of Contact): Troy Unruh (INL)

Federal Manager: Daniel Nichols

PICS:NE Workpackage: CT-22IN070204 – Thermocouples

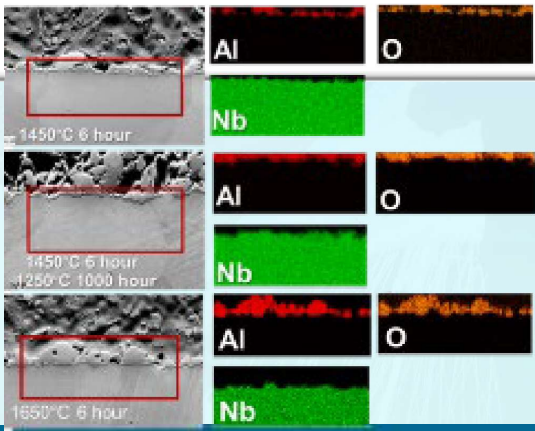


PATH

- Use the HTIR-TC drift model on other commercially available thermocouples including Type-K, N, B
 - From literature, compare drift model to drift data at high temperatures or during irradiation tests
 - Compare drift model to HTIR-TC performance in AGR 5/6/7
 - Perform thorough literature review of available thermocouple data
- Develop HTIR-TC mechanistic understanding to include reporting on:
 - DSC testing
 - Prolonged furnace testing
 - Separate effects testing
 - Microstructural and chemical characterization

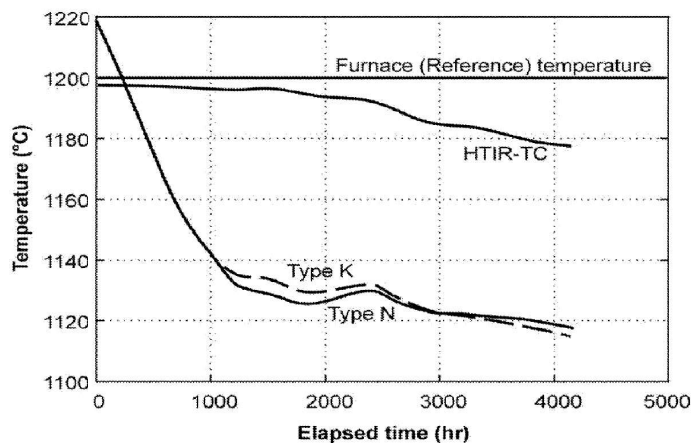
RESULTS

- Literature was compiled to develop a drift model ($f(\text{temp, time, flux})$)
- HTIR-TCs perform similar to N and K TCs for a neutron fluence of 10^{21} N/cm^2 up to 1200 °C.
- HTIR-TCs have minimal drift up to 1500 °C.
- Nb-P thermoelement is drift culprit – Mo-LaO appears stable
 - Exothermic reaction observed in Nb-P thermoelements
 - Alumina interactions between the thermoelement-insulation ($\text{Nb-Al}_2\text{O}_3$) interface were observed.
 - Formation of Nb_3P precipitates after 1450 °C heat treatment
 - Recrystallization was observed after heat treatment
 - Heat treatment results in nucleation of fine grains (Mo)
- Lit review identified alternative insulating ceramics to compare to Al_2O_3 :
 - SiC
 - HfO_2
 - BeO
 - MgO
 - ThO_2



How do HTIR-TCs Compare?

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		1/10 th	1/4 th	1/100 th



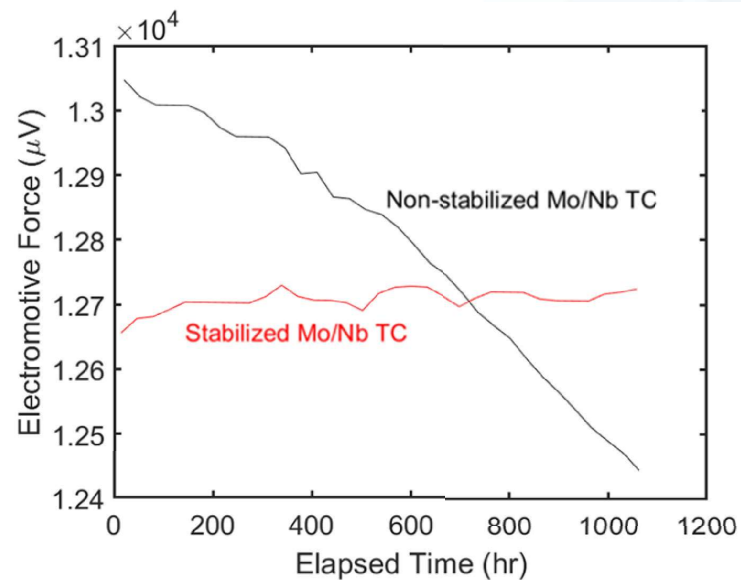
NOTE: Gen IV - Very high temperature reactor Core Temperature³: >1200°C

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

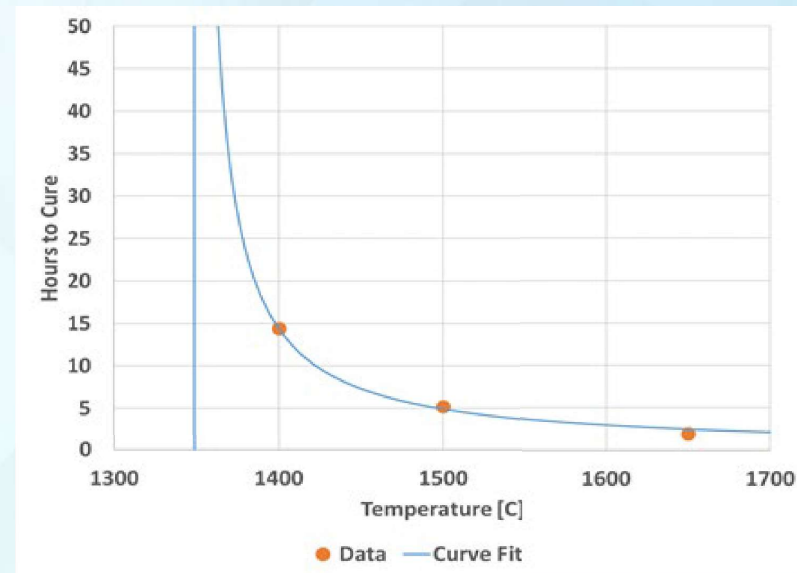
2. Data courtesy of Dr. Skifton, INL

3. Murty K., Charit I., An Introduction to Nuclear Materials. Vol. 1, Wiley-VCH, 2013, Weinheim, Germany.

Background and Motivation



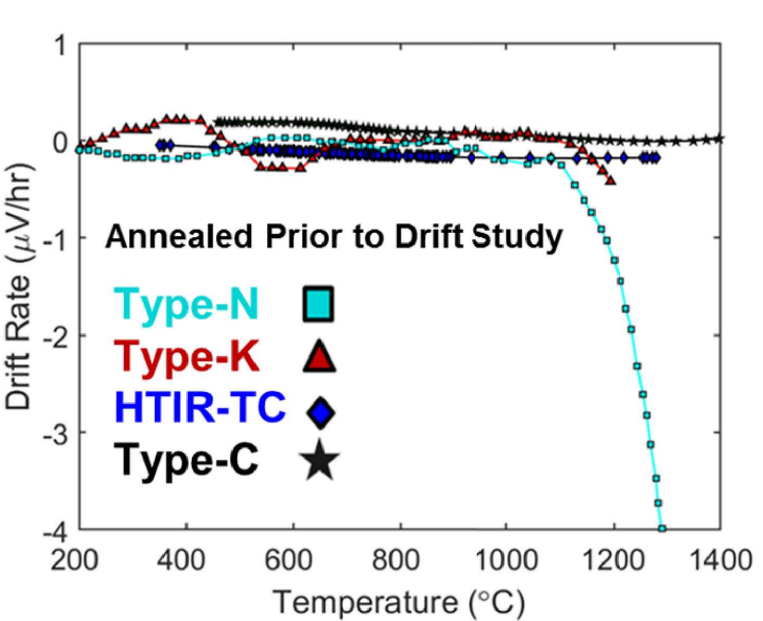
Signal measured by Mo/Nb thermocouples during 1100 °C 1000 hour test⁴.



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

4. Bong Goo Kim, Joy L. Rempé, 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](https://doi.org/10.13182/NT11-A13294)

Drift Model: Temperature Comparison

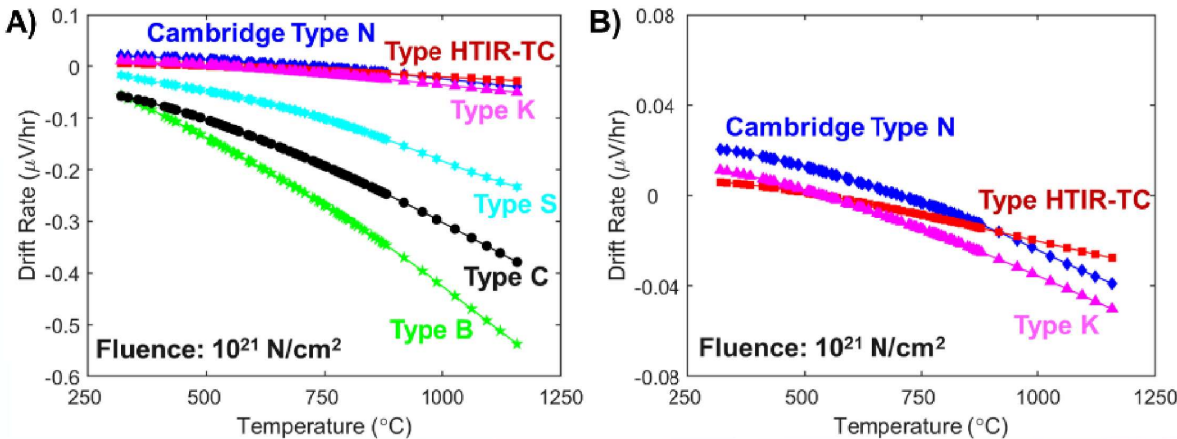


Comparison of the drift in the EmF signal normalized to the duration of the heat treatment for HTIR-TC, type-K, and type-N thermocouples

Thermocouple	Temperature Range ($^{\circ}\text{C}$)	Irradiation Drift (%)	High Temperature Drift (%)
C	0-2315 $^{\circ}\text{C}$	-18.99	-0.22
K	-270-1260 $^{\circ}\text{C}$	-0.02	0.12
N	-270-1260 $^{\circ}\text{C}$	-0.45	-0.66
HTIR-TC	0-1700 $^{\circ}\text{C}$	-0.46	Negligible

Comparison of % drift of type C, K, N, and HTIR-TC thermocouples using the drift model for a temperature of 1083 $^{\circ}\text{C}$ and a neutron fluence of 1.8×10^{-21} N/cm

Drift Model: Temperature and Fluence Comparison



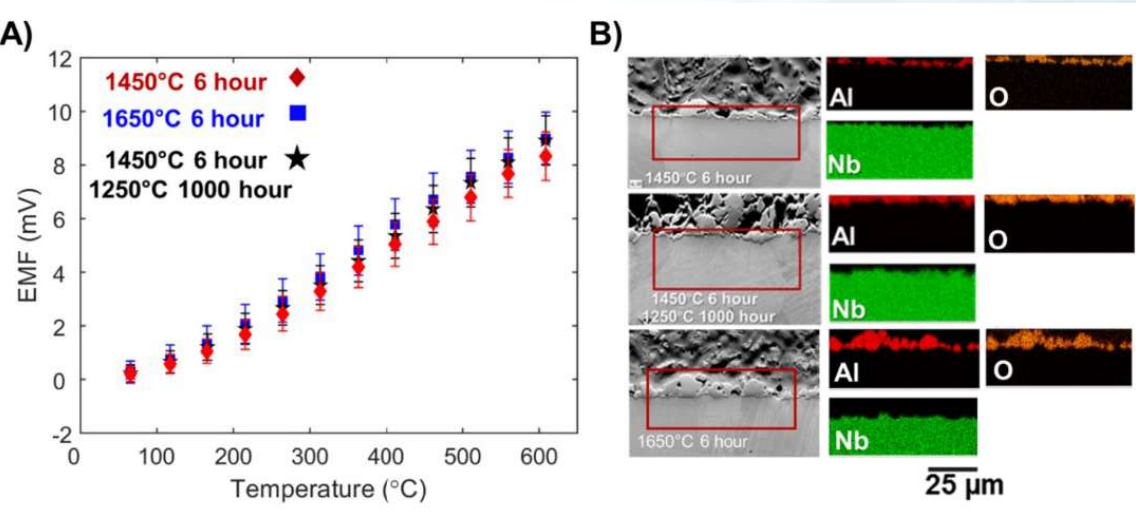
A) Irradiation induced drift in the EMF signal for commonly used thermocouples for temperature sensing in both irradiative and high temperature environments ^{4, 15}.

B) Zoomed in subplot emphasizing the drift due to irradiation for Cambridge type N, K, and HTIR-TC thermocouples.

Comparison of calculated and observed HTIR-TC drift in the AGR 5/6/7 test ⁴.

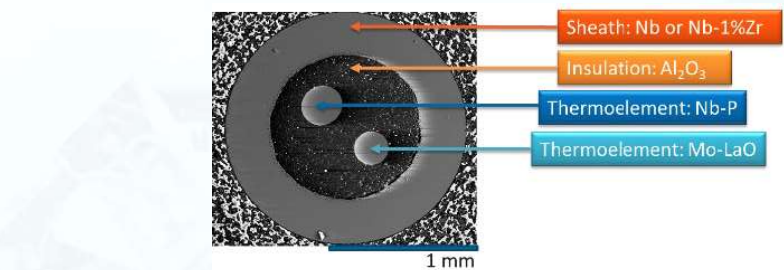
HTIR-TC #	Time at High Temperature (EFPD)	Operating Temperature [$^{\circ}\text{C}$]	Calculated Drift by HTIR-TC Drift Model [%]	Observed Drift of HTIR-TC in ATR Test [%]	Difference between Calculated and Observed Drift [%]
1-12	125	1293	-3.29	-3.33	~ -0.03
1-13	125	1293	-3.29	-3.33	~ -0.03
1-14	125	1381	-3.50	-3.48	~ -0.02
3-5	125	1500	-8.65	-8.67	~ -0.02

Nuclear Thermocouples: Seebeck Coefficient



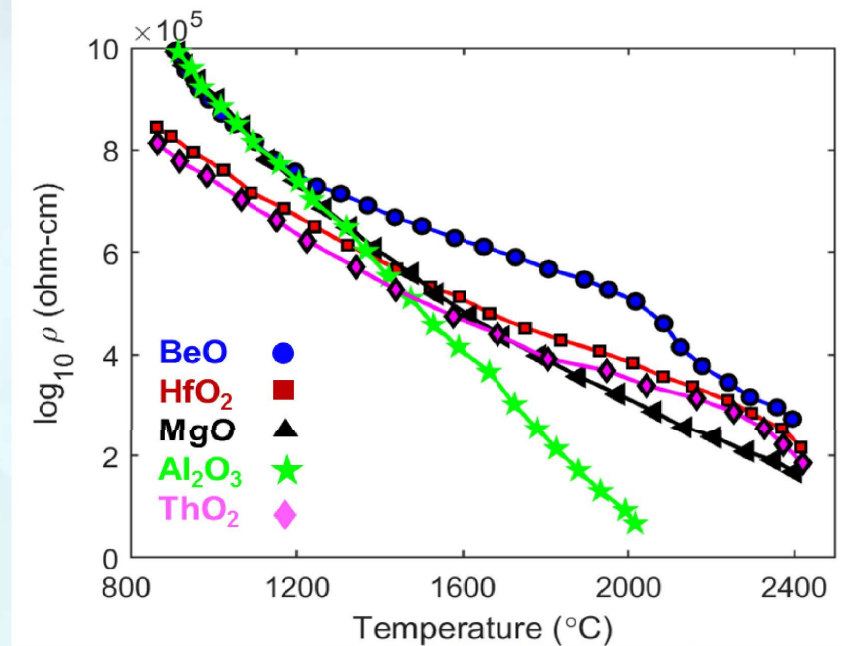
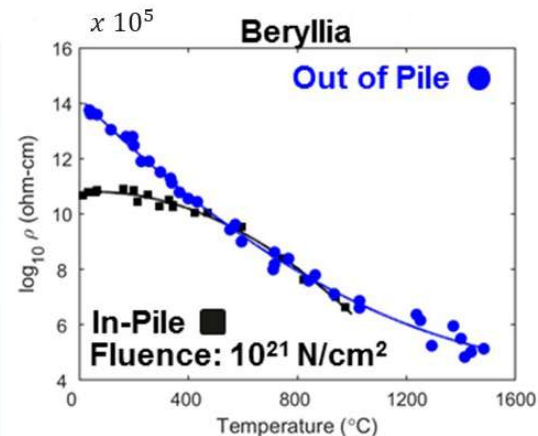
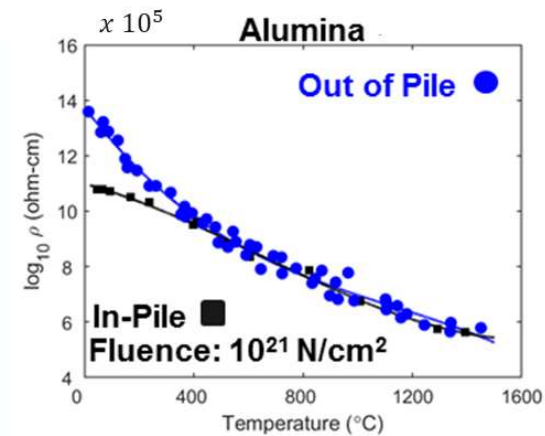
A) The EMF signal does not vary with increasing stabilization heat treatment temperature. After the stabilization heat treatment, prolonged exposure to temperatures below the stabilization heat treatment temperature did not impact the EMF signal.

B) An interaction region is observed between the interface of the Nb-P thermoelement and the alumina insulation during the stabilization heat treatment.



Insulator	Interaction with Niobium Onset Temperature (°C)	References	Interaction with Molybdenum Onset Temperature (°C)	References	Melting point (°C)	Thermal adsorption cross-section $\nu_0 = 2200 \text{ m/sec}$ (Barn)
SiC	1300	39	1500	40,43	2730	3.86
HfO ₂	1300	38,41			2758	104.10
Al ₂ O ₃	1450	37, 38, 42			2072	0.23
BeO	1600	38, 42			2578	0.01
MgO	1800	42			2852	0.06
ThO ₂	1800	42			3390	7.37

Nuclear Thermocouples: Insulation Considerations



MgO's chemical stability with the HTIR-TC thermoelements, low thermal adsorption cross-section, and high resistivity above 1500 $^{\circ}\text{C}$ make it an apparent candidate for the HTIR-TC insulator.

- Davis et. al. The effect of environment on ceramic insulators for nuclear thermionic applications. Nuclear Applications of Nonfissionable Ceramics. Alvin Boltax and J.H. Handwerk, eds., American Nucl. Soc. 1966, pp229-246.
- John Mayer. Summary of radiation effects on thermionic insulator materials. NASA technical note, 1968.
- John Boland, Nuclear reactor instrumentation (in-core), 150 Fifth Avenue, New York, N.Y. 10011: Cordon and Breach 1970.

Project Overview: Acoustic Sensors

Research goal: Develop magnetostrictive and piezoelectric ultrasonic waveguide thermometer (UT) that can measure in-pile temperature through speed of sound

Point of Contact: Joshua Daw (INL); Zhangxian (Dan) Deng (Boise State University)

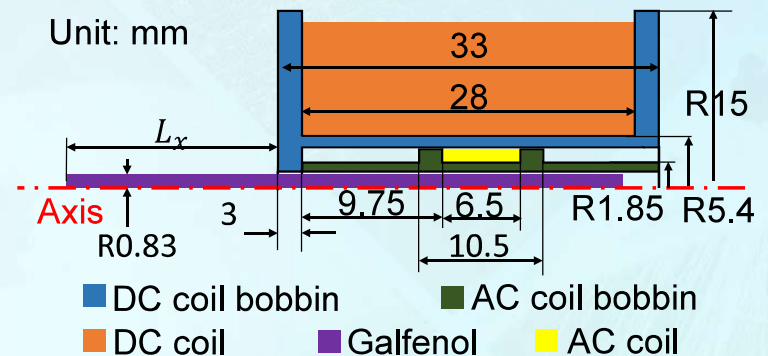
Students: Joy Morin (PhD student), Drew Keller (MS student), Alex Draper (Undergraduate), Ashton Enriques (Undergraduate)

Research Scope (FY22):

Enhance the thermometer performance based on finite element modeling

Validate the thermometer at elevated temperatures

Investigate new options of magnetostrictive waveguides



Technology Impact

Motivation:

- The number of worldwide operational nuclear reactors increased from 230 to 443 from 2009 to 2019
- A total of 11 core melt accidents have occurred worldwide since 1952, including the Chernobyl and Fukushima disasters

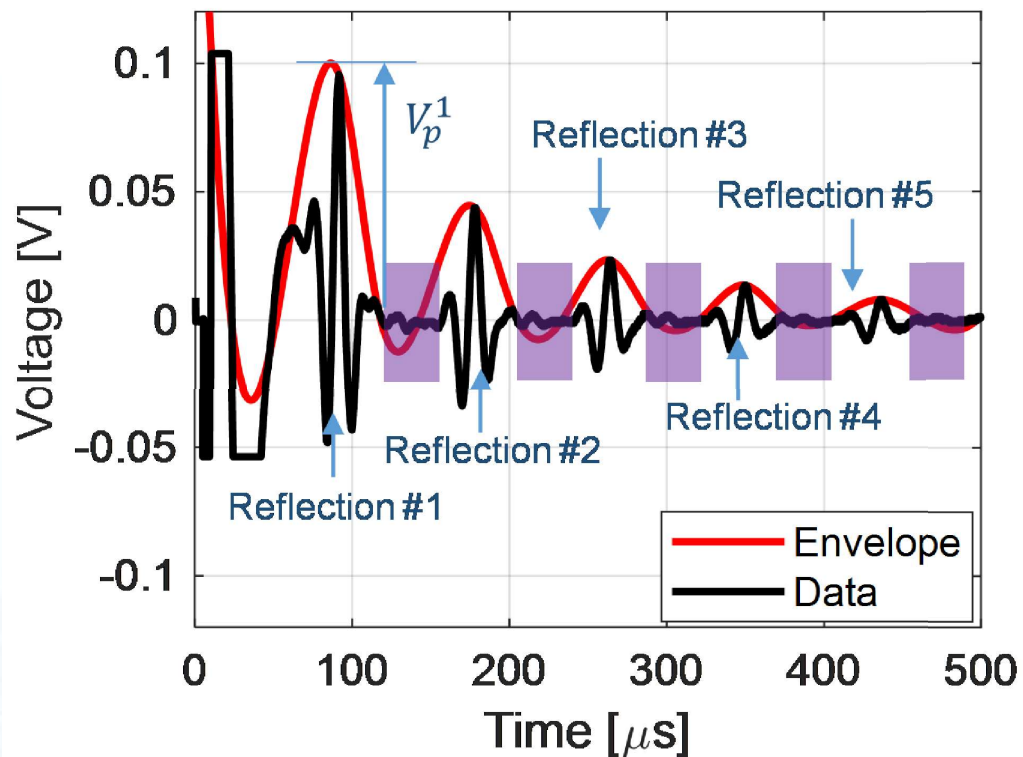
Needs:

Ultrasonic transducers that can

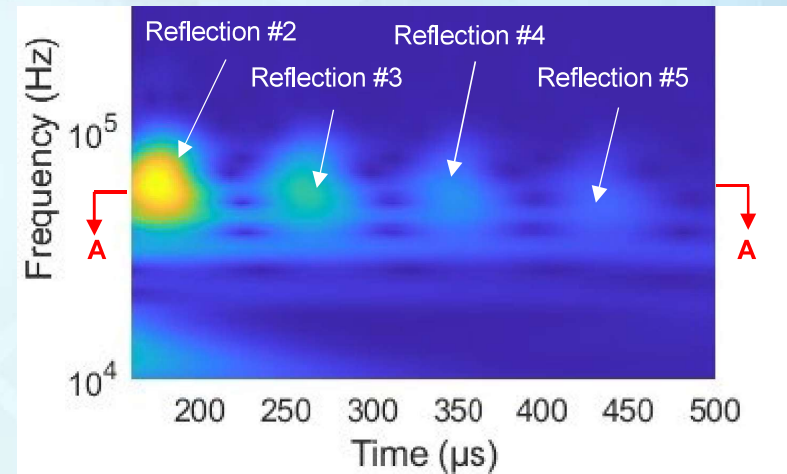
- Fit in tight spaces inside fuel claddings (Small form factor)
- Withstand gamma and neutron flux radiation
- Detect centerline temperatures exceeding 1500 °C

Signal Processing

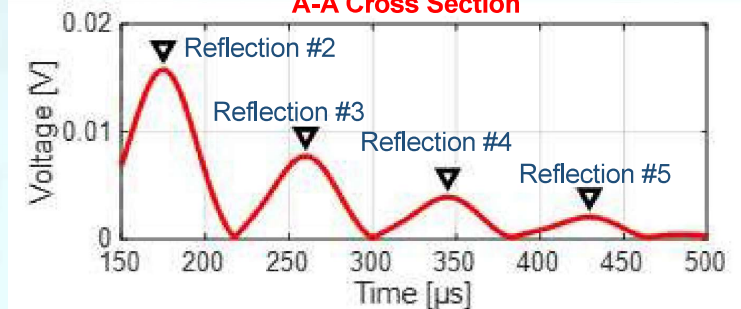
Hilbert Transform (Time Domain)



Wavelet Analysis (Frequency Domain)



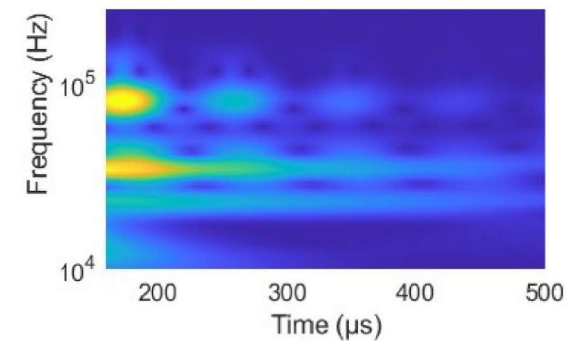
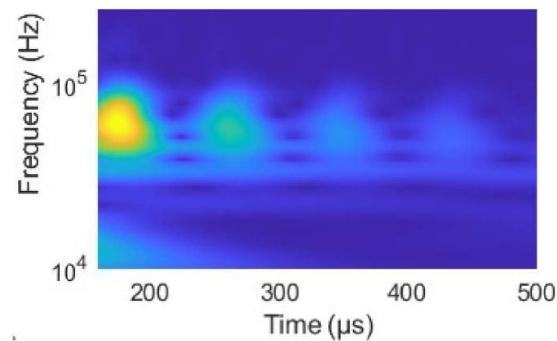
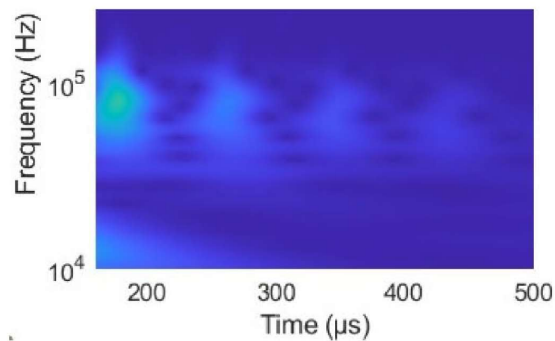
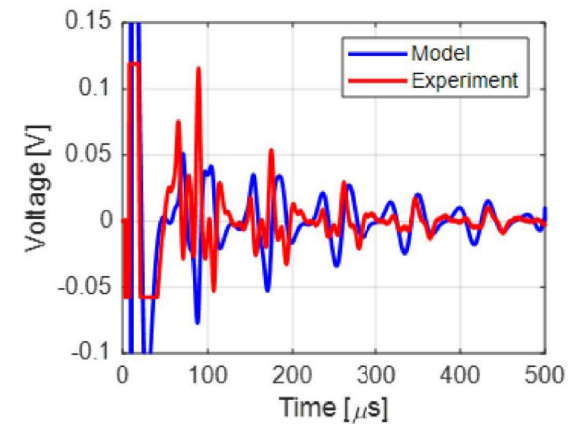
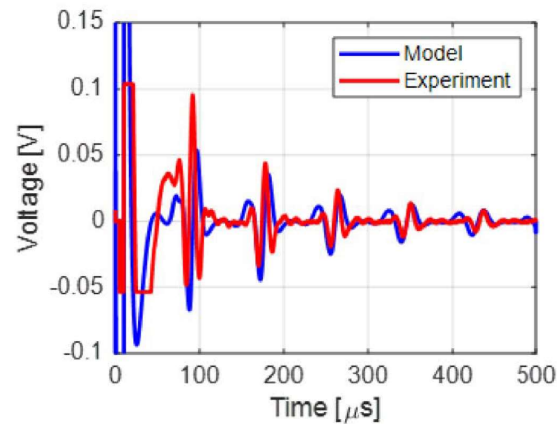
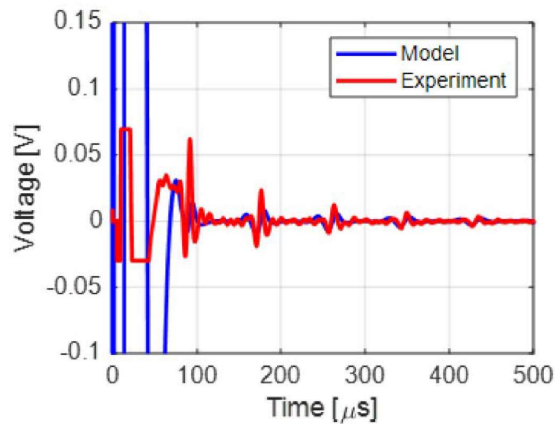
A-A Cross Section



Findings: Wavelet analysis is more reliable.

Optimize Waveguide Location L_x

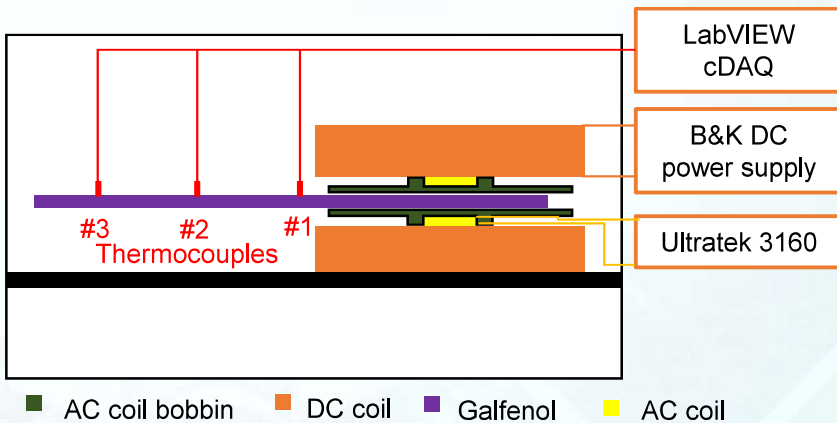
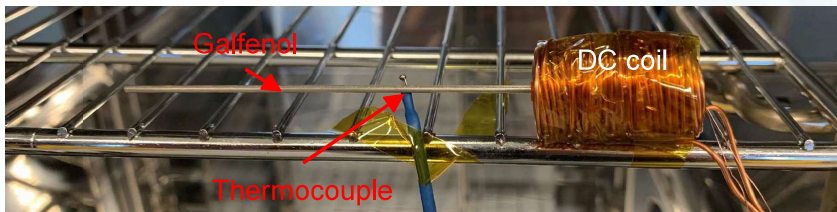
Decreasing L_x



Findings: Optimal $L_x = 82.5$ mm (from both finite element modeling and experimental trial and error)

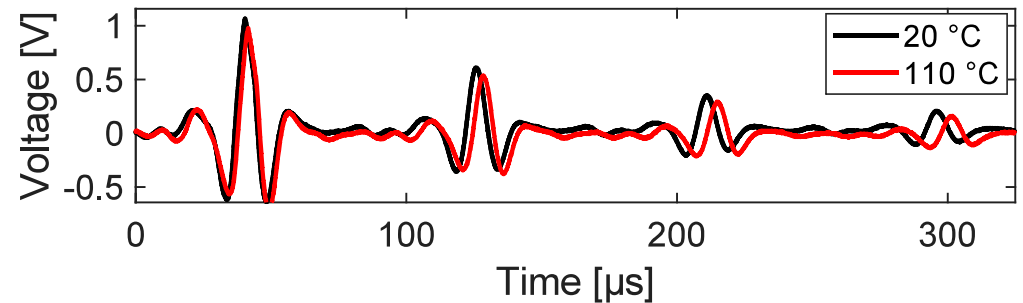
High-temperature Test

Experimental Setup

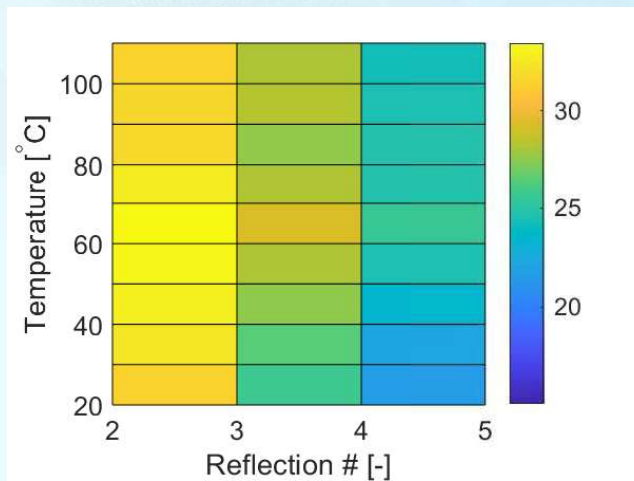


Findings: The signal to noise ratio is >30 dB up to 110 °C if the first two reflections were selected in calculation

Acoustic Waves at Various Temperatures

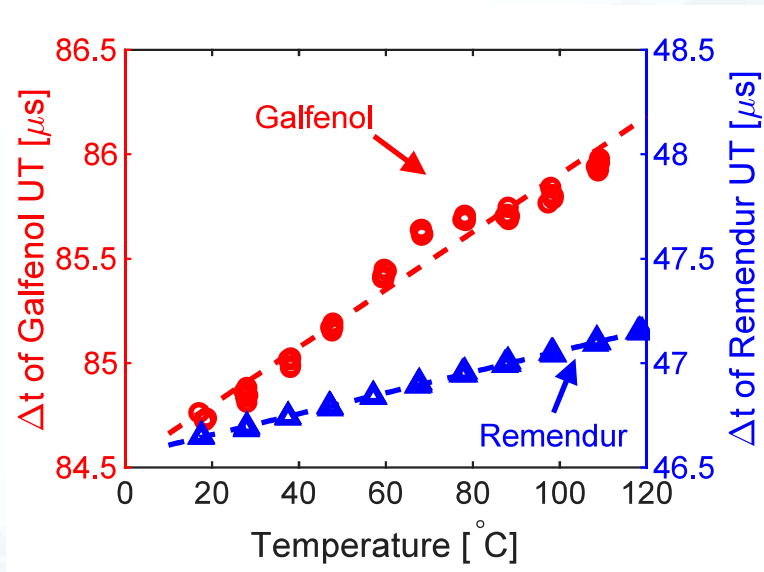


Signal-to-noise Ratio (in dB)

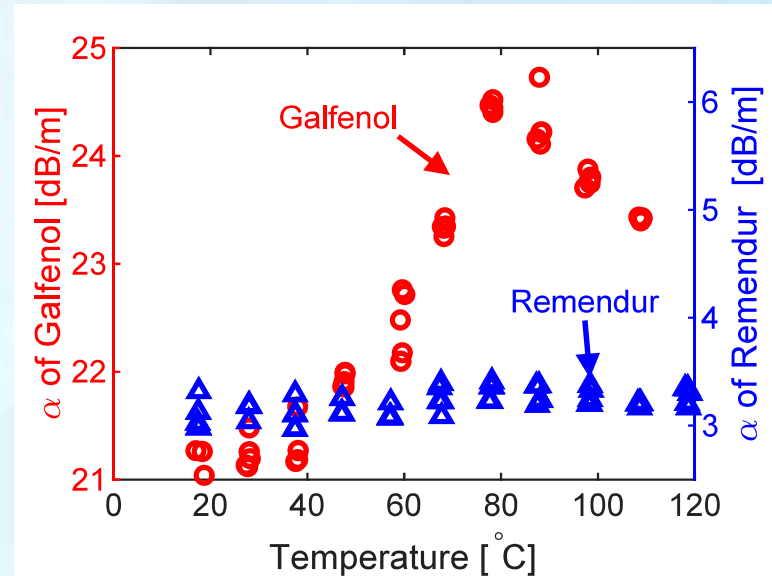


High-temperature Test

Sensitivity



Acoustic Attenuation

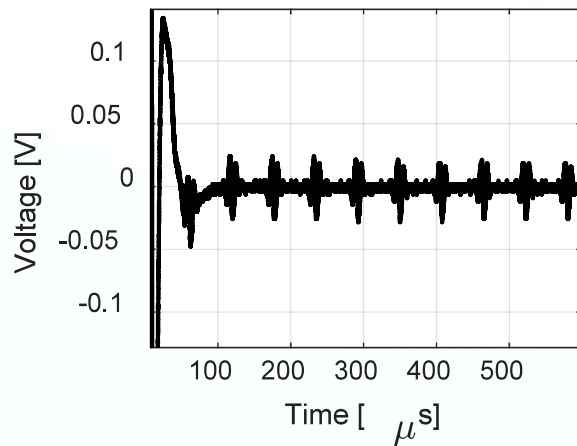


Findings:

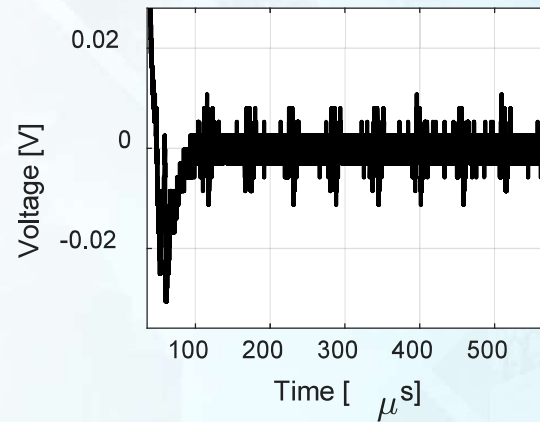
- Sensitivities of Galfenol (FeGa) UT and Remendur (FeCoV) UT are $162.8 \text{ ppm}/^{\circ}C$ and $107.3 \text{ ppm}/^{\circ}C$, respectively
- The resolution of the Galfenol UT is 64% smaller than that of the Remendur UT at the same frequency
- Galfenol UT exhibits nonlinear sensitivity and acoustic attenuation

New Magnetostrictive Waveguides

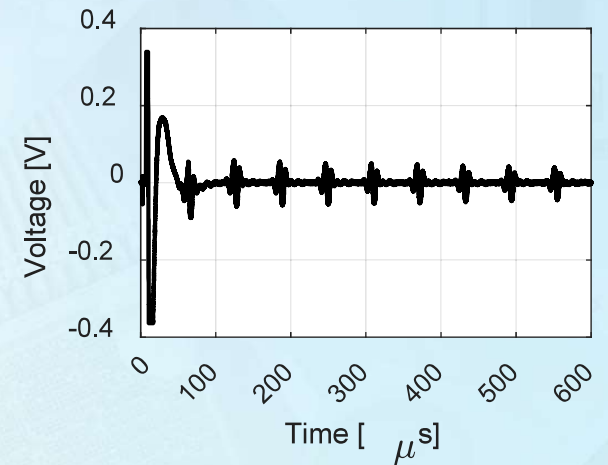
$\Phi 0.8$ mm FeNi



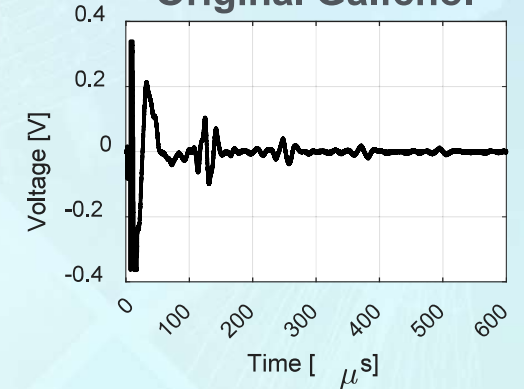
$\Phi 0.5$ mm Galfenol (17 wt.% Ga)



$\Phi 0.8$ mm Galfenol (17 wt.% Ga)



Original Galfenol



Findings:

- Thin and flexible Galfenol waveguides are available from a new vendor; preliminary results have confirmed their functionality
- Thinner waveguide helps to reduce acoustic attenuation
- Coils need to be re-designed to minimize magnetic flux leakage and enhance the signal-to-noise ratio.

Printed Sensor Technologies for Harsh Environments

OVERVIEW

Purpose: Enable novel sensor designs through advanced manufacturing. Once the feasibility of the fabrication process is validated, advanced manufactured sensors will be deployed in relevant irradiation tests within NEET ASI activities as well as through other awarded irradiation testing proposals. Focus is on sensors for advanced structural health monitoring.

Objectives:

- Improve and optimize the printing process of piezoelectric inks
- Additively manufacture piezoelectric surface acoustic wave (SAW) devices for sensing in harsh environments
- Develop novel nanoparticle inks with improved materials for harsh environments that are compatible with AJP.
- Develop heterogenous integration methods to printed sensors
- Improve and optimize printed capacitive strain gauges (CSGs)
 - Develop strain validation techniques (DIC, RSGs, etc)
 - Increase the robustness of printed sensors at elevated temperatures (650 °C)
- Quantify the adhesion strengths of printed films using destructive techniques

Outcomes: Development of advanced manufacturing methods and capabilities to enable transformative sensor technology for in-pile monitoring and in-situ analysis of fuels and materials that are not otherwise achievable through classical fabrication techniques.

DETAILS

Principal Investigators: David Estrada (BSU)
Michael McMurtrey (INL)

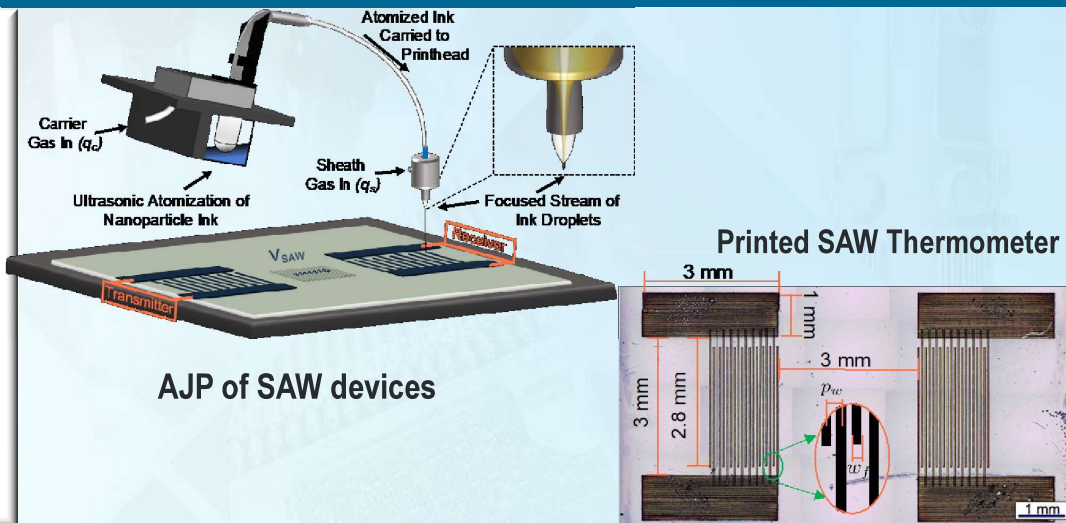
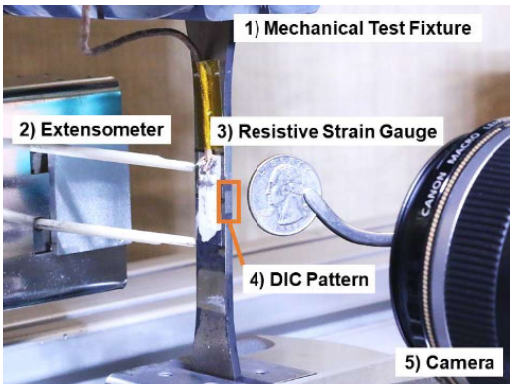
Institution: Boise State University

Collaborators: Idaho National Laboratory

TPOC (Technical Point of Contact): Troy Unruh (INL)

Federal Manager: Daniel Nichols

PICS:NE Workpackage: CT-22IN070205 –
Printed Sensors Technologies for Harsh Environments



RESULTS

Results:

- Digital image correlation was used to measure strain (up to 1100 $\mu\epsilon$) during cyclic tests at 23-600 °C.
- Small scale periodic patterns were printed using aerosol jet printing for digital image correlation.
- CSG, RSG, and adhesion results will be presented in the next presentation (Mechanical Properties Characterization) by Michael McMurtrey and Tim Phero.
- Novel synthesis techniques were developed for nanoparticle LiNbO_3 ink formulations
 - Controlled stoichiometry
 - Lowers the reaction temperature of calcination
 - Produces greener biproducts
 - Increases phase purity of final product
- Aerosol jet printing (AJP) of the reactive LiNbO_3 ink was performed for the first time.

Technology Impact

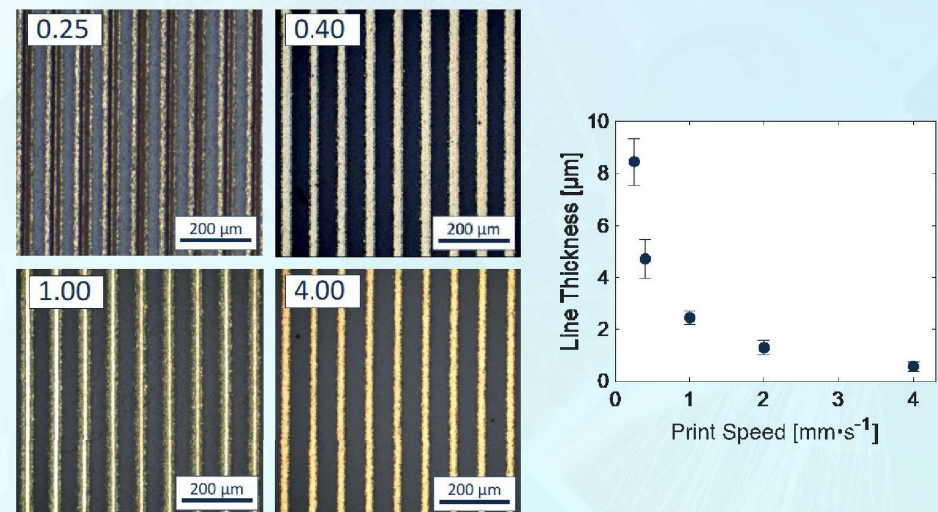
Motivation:

- R&D of novel in-pile sensors capable of thermometry and structural health monitoring can reduce O&M costs of reactors and improve safety through live status monitoring.
- Utilize advanced manufacturing techniques for rapid prototyping of sensors for harsh environments.
- Expand the library of materials compatible with AJP, including high temperature conductive nanoparticle inks and piezoelectric nanoparticle inks.

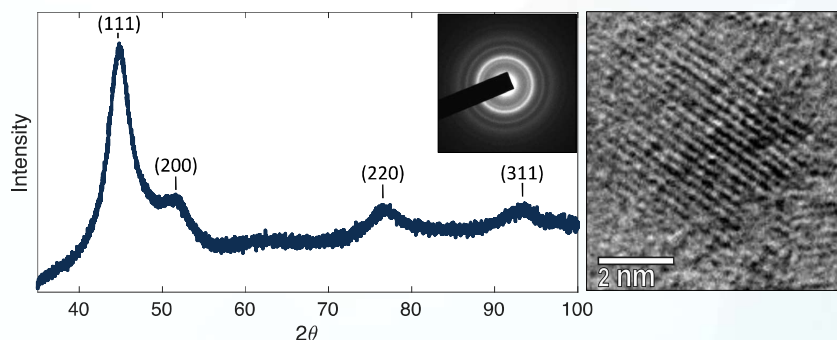
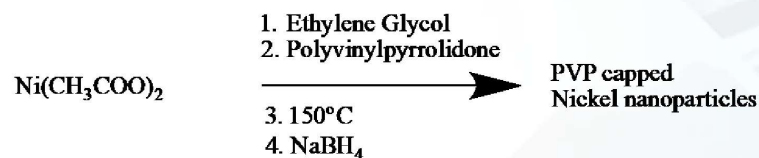
Needs:

- Process improvements to increase consistency and predictability of outcome for AJP
- Conductive material inks for AJP that are resistant to thermal and nuclear radiation
- Piezoelectric nanoparticle inks that can be deposited directly onto host structures like steel, allowing for integration of SAW devices directly onto reactor components

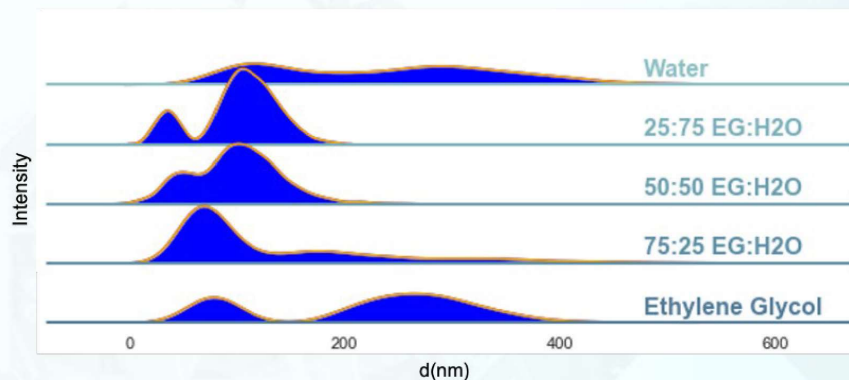
Coating Thickness vs. Print Speed with AJP Silver



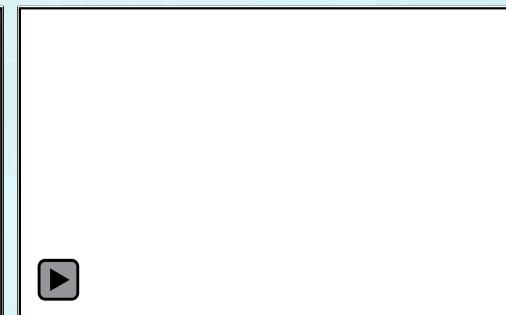
High Temperature Nickel Nanoparticle Ink



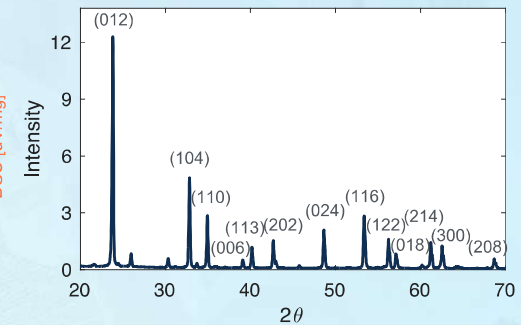
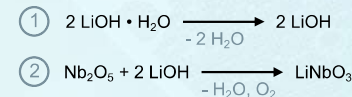
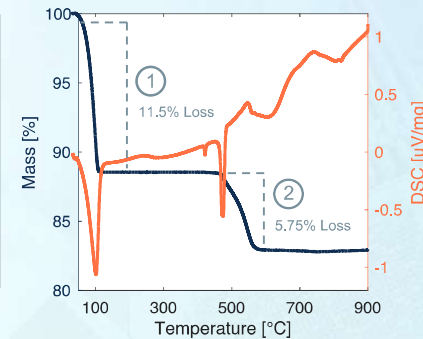
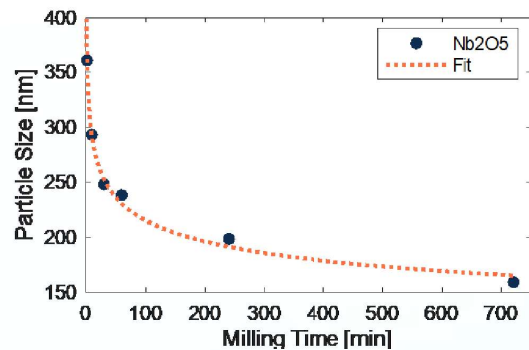
Impact of Co-Solvent Ratio on Hydrodynamic Particle Size
Nickel Nanoparticle ink Rheology



- The polyol synthesis was used to generate nickel nanoparticles
- TEM revealed grain sizes up to 5 nm, and aggregates of ~200 nm
- Rheological studies were performed to develop the ink system showing an average hydrodynamic particle size of ~80 nm for the selected co-solvent system
- Nick's nickel nanoparticle ink was used to print various structures including piezoelectric SAW devices
- Sintering conditions have been determined and ink has been nearly fully optimized



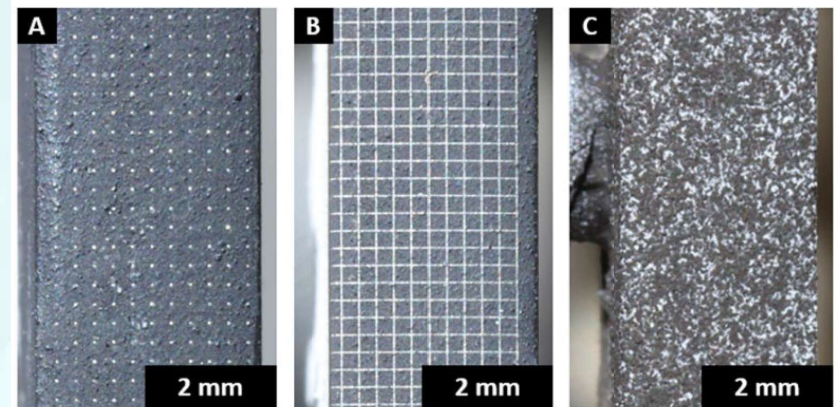
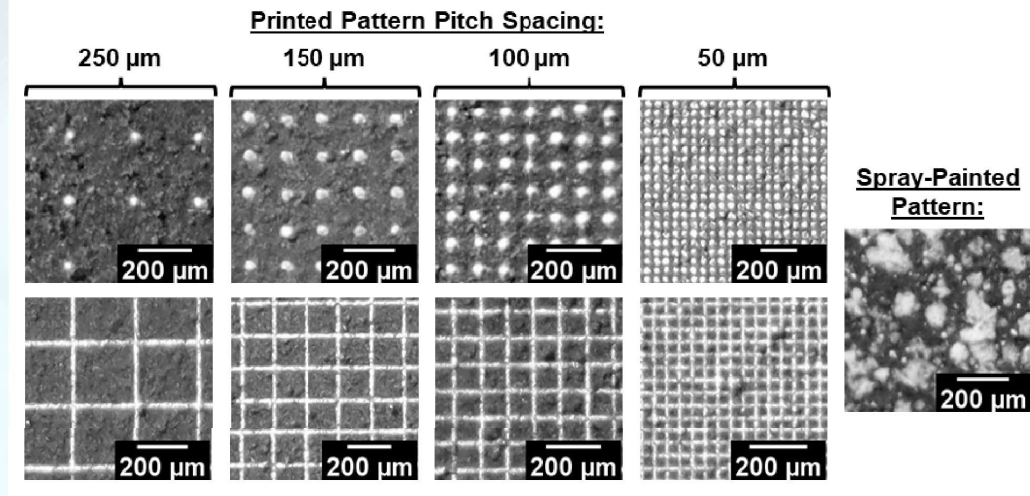
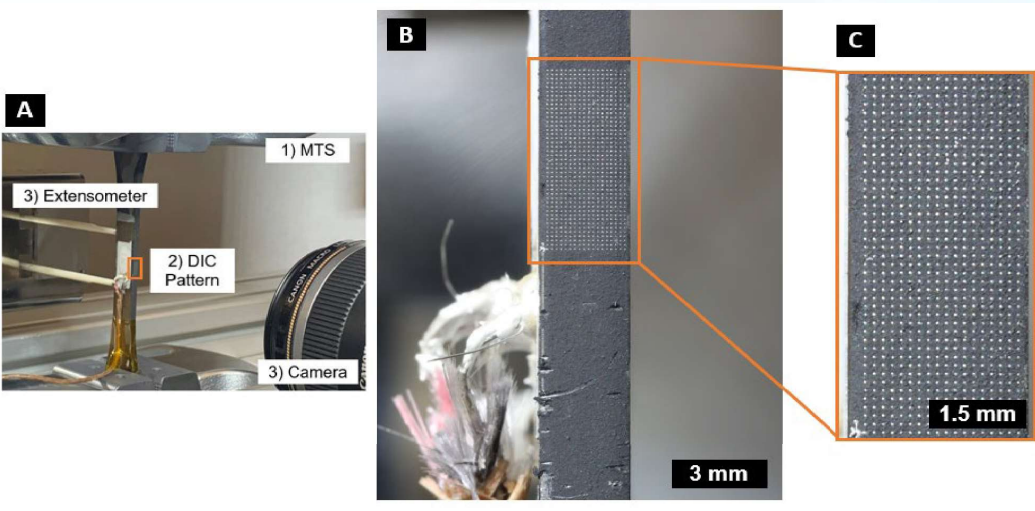
Reactive Piezoelectric Lithium Niobate Ink



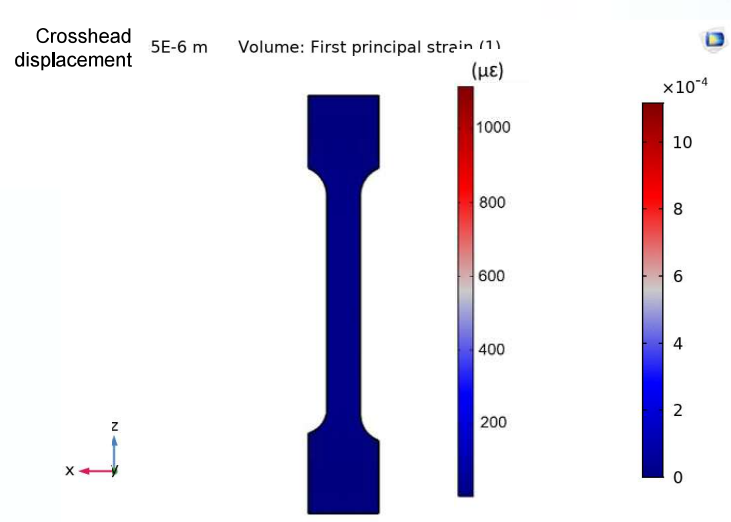
- Niobium oxide particles were ball milled to produce nanoparticles <100 nm after 24 hours mill time
- An AJP ink was synthesized by suspending Nb_2O_5 in an aqueous lithium hydroxide solution
- Preliminary printing studies have been performed to some degree of success
- Reaction conditions have been determined for thermal combustion and product synthesis
- Phase pure product formation has been observed after deposition onto surface
- Future directions
 - New ink formulation with improved particles and ink stability
 - Piezoelectric testing of AJP fabricated LiNbO_3 films

Printed Patterns for Digital Image Correlation

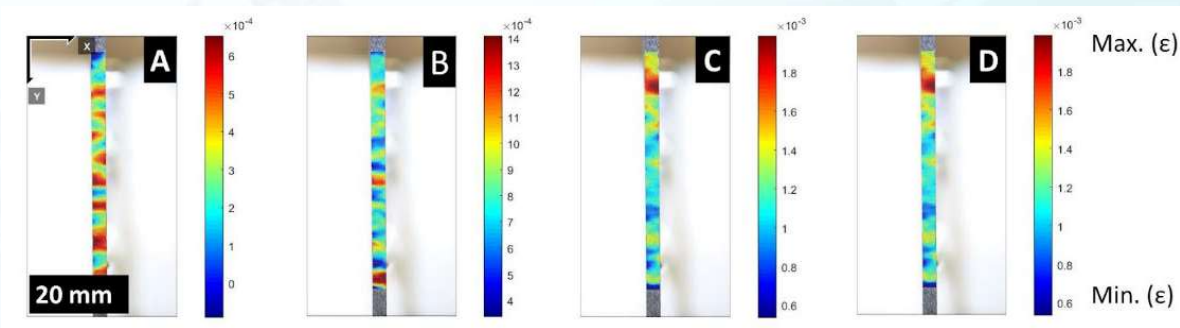
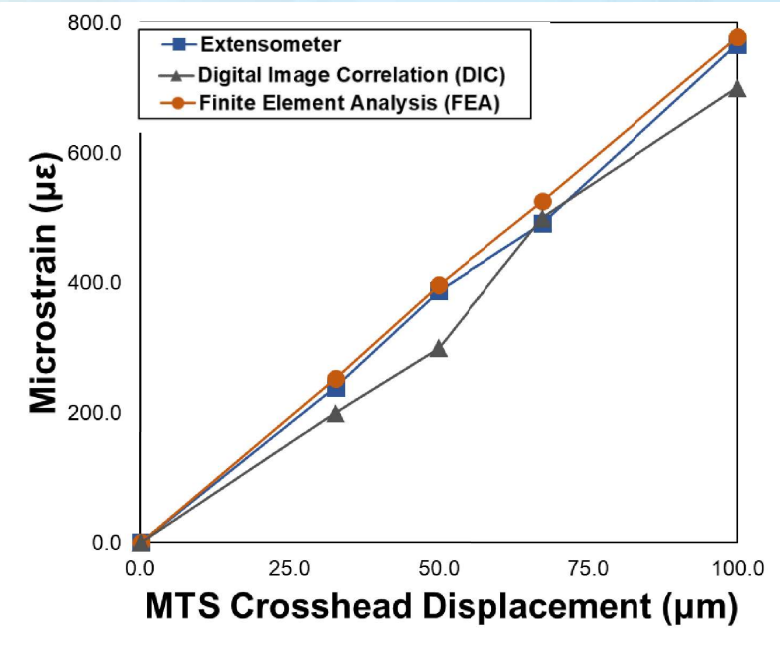
Digital Image Correlation (DIC) is a non-contact process to measure strain with the use of a camera and a speckle pattern in the region of interest



Validating DIC Results



STRAIN VALIDATION METHODS – FEA With COMSOL

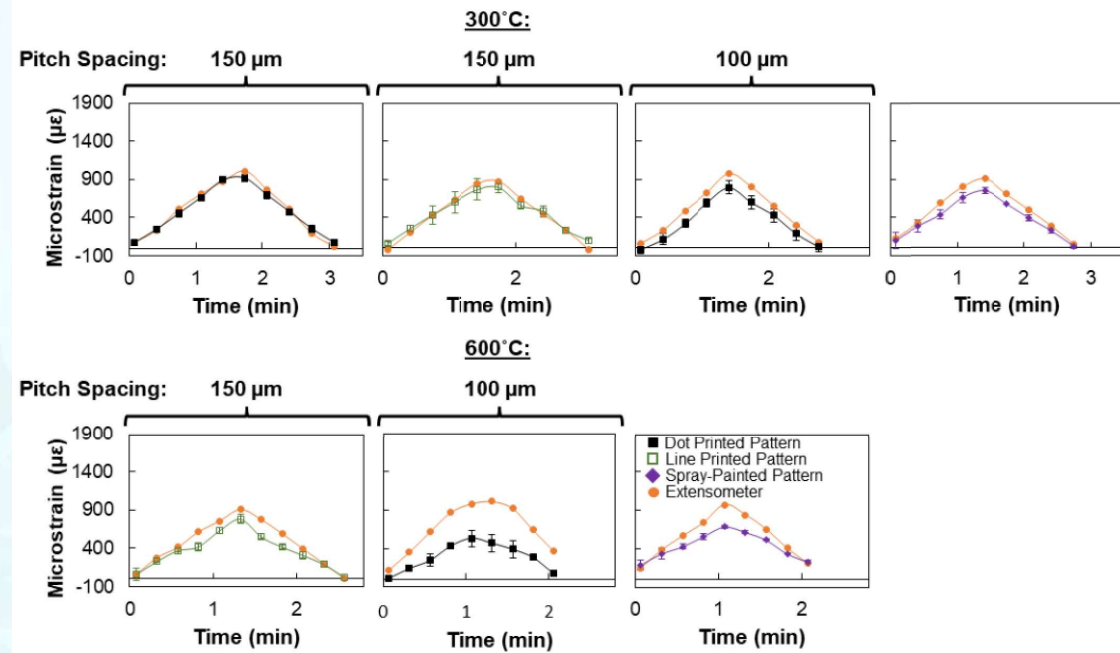
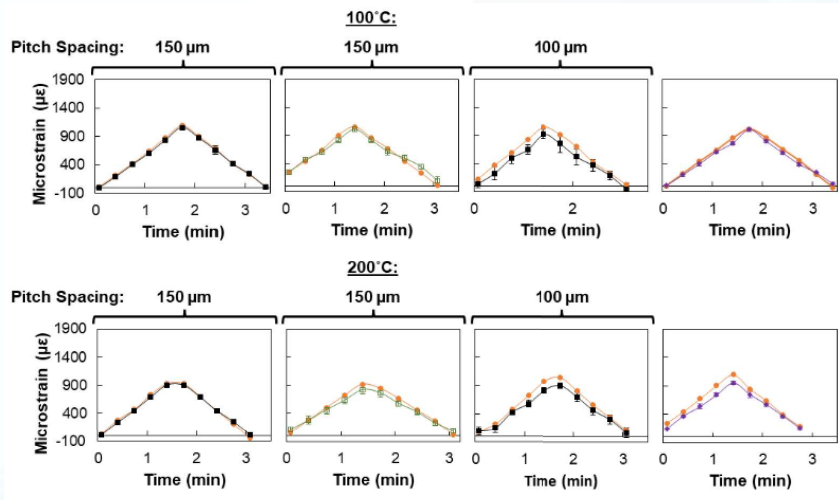


	Average DIC Strain (ϵ)	Extensometer Strain (ϵ)
A	0.0003	0.0006
B	0.0008	0.0013
C	0.0012	0.0020
D	0.0007	0.0011

Printed Sensor Technologies for Harsh Environments

Mechanical tests were conducted with digital image correlation (DIC) at 100°C, 200°C, 300°C, and 600°C on SS316L substrates

The printed 150 μm spaced line pattern tabulated similar strain values to the data read by the extensometer.





BSU Supporting Activities Outputs

Student successes:

- Nick McKibben – Successfully defended his dissertation proposal and advanced to candidacy in April
- Kiyo Fujimoto – NEUP fellow, INL Grad Fellow at INL HTTL, Successfully defended dissertation in March
- Timothy Phero – INL Grad Fellow at INL HTTL
- Kaelee Novich – NEUP Fellow, INL internship (currently)
- Kati Wada – NEUP Fellow, internship at INL HTTL for summer 2022
- American Nuclear Society formally recognized the BSU Nuclear Energy Club as an ANS Student Section in May of 2022
- Timothy Phero and Kaelee Novich were awarded Graduate Scholarships from the American Nuclear Society

Publications

1. "Additively manufactured strain sensors for in-pile applications." *T.L. Phero, K.A. Novich, B.C. Johnson, M.D. McMurtrey, D. Estrada, and B.J. Jaques*[†]. *Sensors and Actuators A: Physical*. Vol. 344, pp. 113691, 2022. DOI: 10.1016/j.sna.2022.113691

Submitted Publications

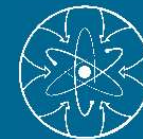
1. "Magnetostrictive Ultrasonic Waveguide Transducer for In-pile Thermometry." D. Keller, B. Robinson, A. Draper, A. White, J. Daw, and Z. Deng, *IEEE/ASME Transactions on Mechatronics* (in print)
2. "Multi-Modal Printed Interconnects for Flexible Hybrid Electronic Applications," S. Seva, *T. L. Phero*, A. Olivas, J. Manzi, T. Gabel, T. Varghese, T. R. Dabrowski, J. D. Williams, D. Estrada, H. Subbaraman, *Flex. Print. Electron.* Submitted.
3. "Experimental examination of additively manufactured patterns on nuclear materials for digital image correlation." K.A. Novich, T.L. Phero, S.E. Cole, C.M. Greseth, M.D. McMurtrey, D. Estrada, B.J. Jaques, *Measurement Science and Technology*. Submitted October, 2022
4. "Aerosol Jet Printing of Piezoelectric Surface Acoustic Wave Thermometer," to *NPG journal, Microsystems and Nanoengineering*. Revisions submitted in October, 2022.
5. "Multiphysics Modeling of Printed Surface Acoustic Wave Thermometers." *Journal of Intelligent Material Systems and Structures*. Submitted September, 2022

Conferences:

1. K. Fujimoto, M. McMurtrey, T. Unruh, T. Holschuh, L. Hone, P. Moo, D. Estrada, "Advanced Manufacturing for the Development of Advanced In-Pile Sensors and Instrumentation", The Minerals, Metals, and Materials Society (TMS) Annual Meeting & Exhibition, (Anaheim, CA; Feb. 2022).
2. N. McKibben, B. Ryel, A. Draper, D. Estrada, Z. Deng, "Additive Manufacturing and Characterization of Surface Acoustic Wave Devices", The Minerals, Metals, and Materials Society (TMS) Annual Meeting & Exhibition, (Anaheim, CA; Feb. 2022).
3. "Simulation of printed surface acoustic wave thermometer." Presented in the SPIE Smart Structures+NDE conference (online), 2022 and published a conference proceeding
4. Poster Presentation, TMS 2022 Annual Meeting & Exhibition
5. Poster Presentation, PNW AVS 2022 Annual Symposium
6. Additively Manufactured Digital Image Correlation for Nuclear Materials. K. Novich, T. Phero, S. Cole, M. McMurtrey, D. Estrada, B.J. Jaques. TMS 2023 (March 19-23, San Diego, CA).
7. Additively manufactured strain sensors for nuclear applications. T.L. Phero, K.A. Novich, B.C. Johnson, M.D. McMurtrey, D. Estrada, B.J. Jaques. TMS 2023 (March 19-23, San Diego, CA).

Patent:

- Joshua Daw, Troy C Unruh, Brenden J Heidrich, David H Hurley, Kiyo Tiffany Fujimoto, David Estrada, Michael McMurtrey, Kunal Mondal, Lance Hone, Robert D Seifert, "Sensors for passively measuring a maximum temperature of a nuclear reactor, and related methods," US Patent App. 17/303,633



Thank You

Brian J. Jaques

Assistant Professor, Micron School of Materials Science and Engineering
Boise State University

BrianJaques@BoiseState.edu

W (208)-426-5376 | C (208)-484-0597

ORCID: 0000-0002-5324-555X