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In-Pile Sensor Fabrication by Advanced Manufacturing

Advanced Sensors and Instrumentation Annual Webinar
October 30, 2019

PI Michael McMurtrey, **INL**
Presenter Kiyo Fujimoto, **INL, BSU**

INL/MIS-19-56308

Project Overview

- **Goal**: To enable the production of novel sensor designs for *in-pile sensors* and instrumentation by utilizing advanced manufacturing techniques.

- **FY19 objective**:
  - Develop new plasma printing technology to print a variety of sensor materials for test reactors
    - neutron flux, mechanical strain, thermal conductivity and peak temperatures
  - To use combinatorial materials science to expedite the development of radiation hardened sensor materials.

- **Participants (2019)**
  - Michael McMurtrey, INL
  - Kiyo Fujimoto, INL, BSU
  - Kunal Mondal, INL
  - Joseph Bass, INL
  - Dave Estrada, BSU
  - Austin Biaggne, BSU
  - Lan Li, BSU
  - Yanliang Zhang, ND
  - Nicholas Kempf, ND

Project Overview

Three activities working together to achieve project objectives:

- ***Process control and sensor fabrication (INL, BSU)***
  - *Refine advanced manufacturing processing parameters to create robust and reliable sensors (minimal voids/defects, good adhesion to the substrates).*
  - *Develop advanced manufacturing capabilities for sensor fabrication*
- ***Ink and feedstock synthesis (INL, BSU)***
  - *Ink/feedstock development and characterization to enable the fabrication sensors/instrumentation with advanced manufacturing techniques.*
- ***Combinatorial Materials Science (INL, ND)***
  - *Develop combinatorial materials science methods for the rapid screening of materials for use in instrumentation such as strain gauges and thermocouples.*

Idaho National Laboratory

BOISE STATE UNIVERSITY

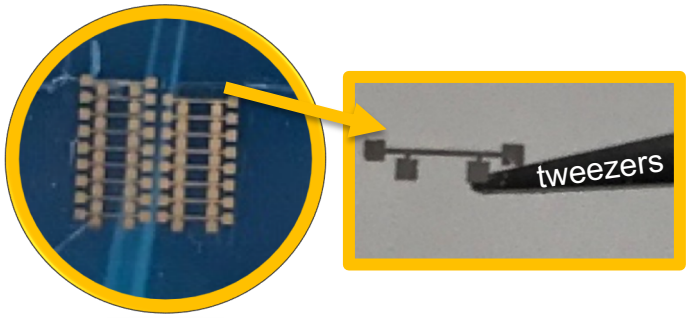
Accomplishments

- Plasma jet printer sensor fabrication with process parameters optimized by simulation input: *Report submitted 9/30/19*
- Demonstrate the fabrication of passive neutron dosimeters for in-pile applications using advanced manufacturing: *Report submitted 5/23/19*
- Finalize recipes and procedures for synthesis of iron, cobalt, zinc, tungsten and indium nano-particle inks: *Report submitted 6/20/19*
- Develop molybdenum, niobium and platinum inks compatible with plasma jet printing: *Report submitted 12/20/18*
- Examine radiation effects on sensor material composition using ion irradiations and high-throughput combinatorial material testing: *Report submitted 9/30/19*
- Complete scanning probe system to perform electrical conductivity and thermal conductivity combinatorial material studies: *Report submitted 1/31/19*

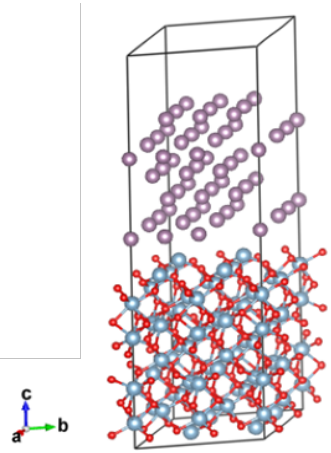
Accomplishments: Aerosol and Plasma Jet Printing

Process control

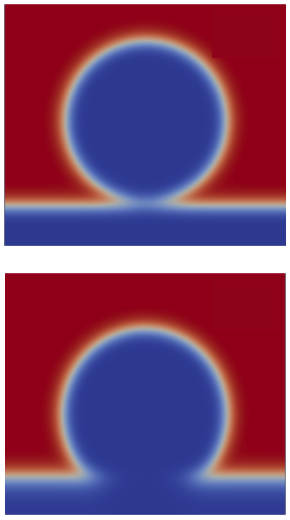
- *Density functional theory (DFT) and ab-initio molecular dynamics used for atomic scale, phase field used for actual scale*
- *Input from modelling/simulation to inform experimental process parameters to improve sensor robustness and materials studies*



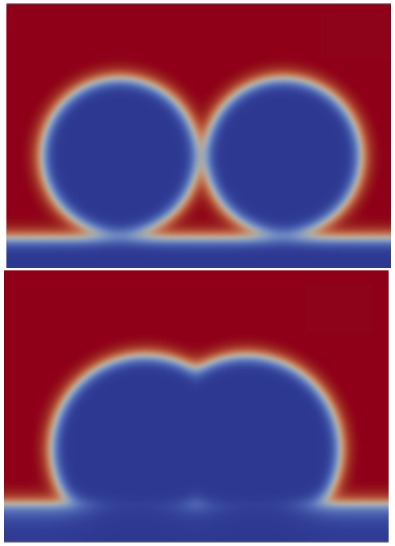
Molybdenum and alumina interface



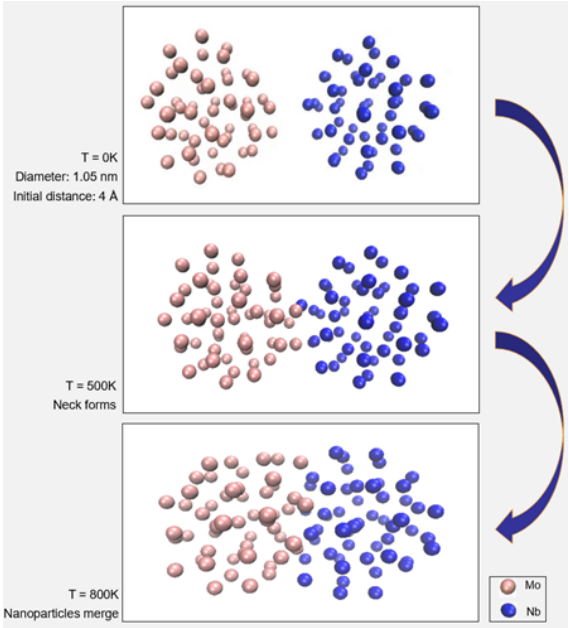
Initial (top) and equilibrium (bottom) nickel on alumina substrate at 1000 °C.



Initial (top) and equilibrium (bottom) nickel particles on alumina substrate at 1000 °C.



Sintering of two 1 nm nanoparticles (one Mo and one Nb) at 0K, 500K and 800K.

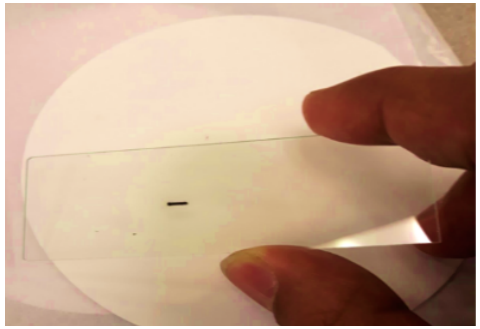


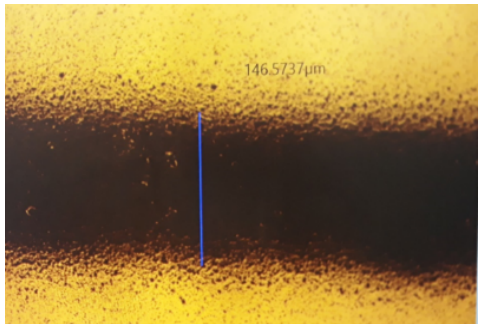
Accomplishments: Aerosol and Plasma Jet Printing

Sensor Fabrication

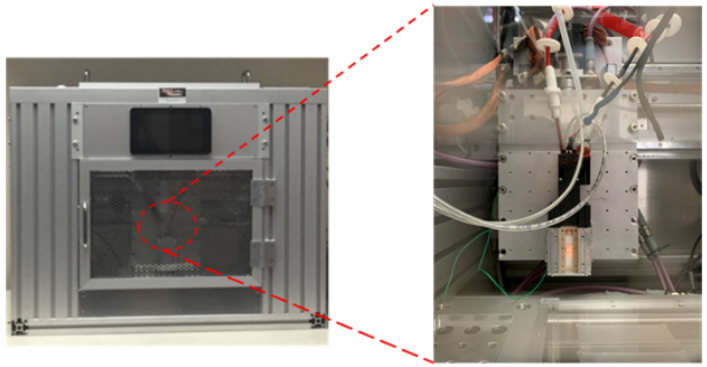
- *Focus has been on aerosol jet and plasma jet printing techniques*
- *Method developed for small melt-wire chip for use in limited space experiments*

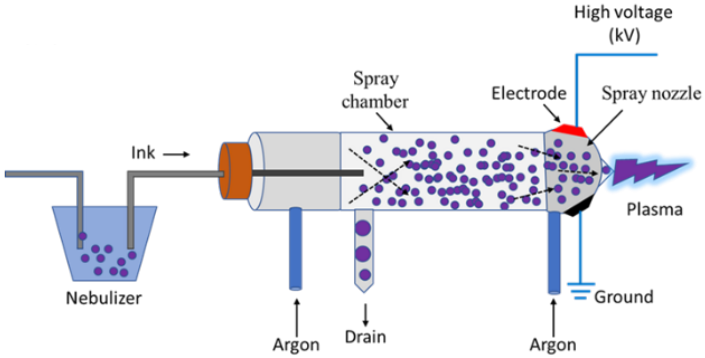
Plasma jet printed copper line

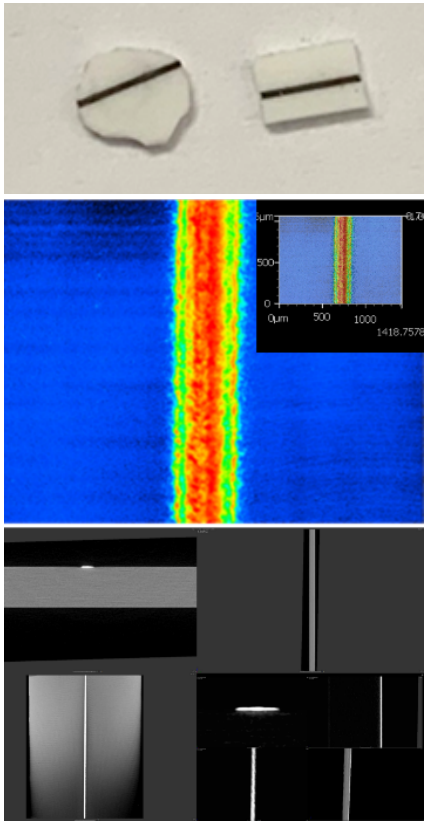




INL plasma jet printer





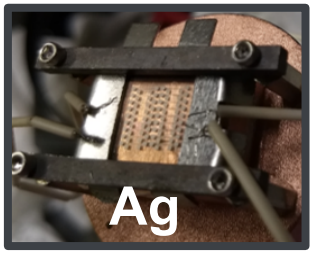


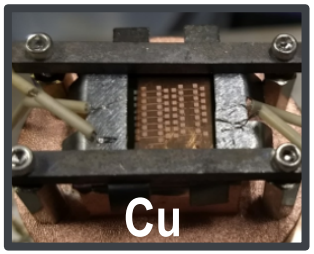
AJP copper line on alumina

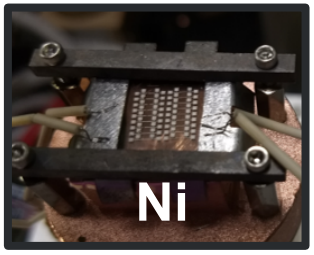
***3 RTE's (2-MIBL 1-MITR) have been leveraged to perform irradiation experiments on printed structures using commercially available inks**

Accomplishments: Ink and Feedstock Fabrication

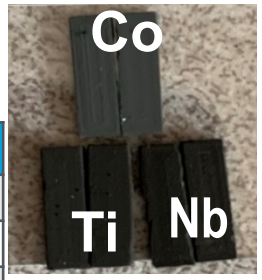
- *Novel material inks are needed for printing techniques used in advanced manufacturing techniques for sensor fabrication*
- *Platinum, molybdenum, niobium, iron, cobalt, zinc, tungsten and indium nanoparticle inks were developed in FY19 (highlighted in yellow)*
- *Two general methods of synthesis: Top-down and bottom-up*

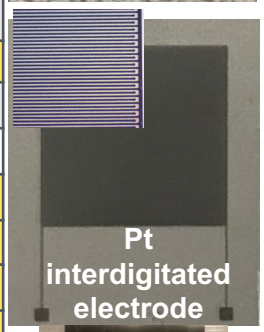


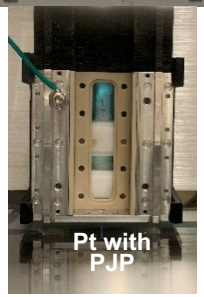




Material	Ink Status	Sensor
Ag	Commercial	Melt wires, dosimeters
Cu	Commercial	Melt wires
Ni	Commercial	Melt wires
Pt	BSU/INL	Melt wires, 3-omega, Strain gauge
Ti	BSU/INL	Melt wires, dosimeters
Nb	BSU/INL	Dosimeters
Mo	BSU/INL	Dosimeters
Co	BSU/INL	Melt wires, dosimeters
Fe	BSU/INL	Melt wires, dosimeters
W	BSU/INL	Melt wires, dosimeters
Zn	BSU/INL	dosimeters
In	BSU/INL	dosimeters
Al2O3	BSU/INL	Insulator
CeO2	BSU/INL	Insulator





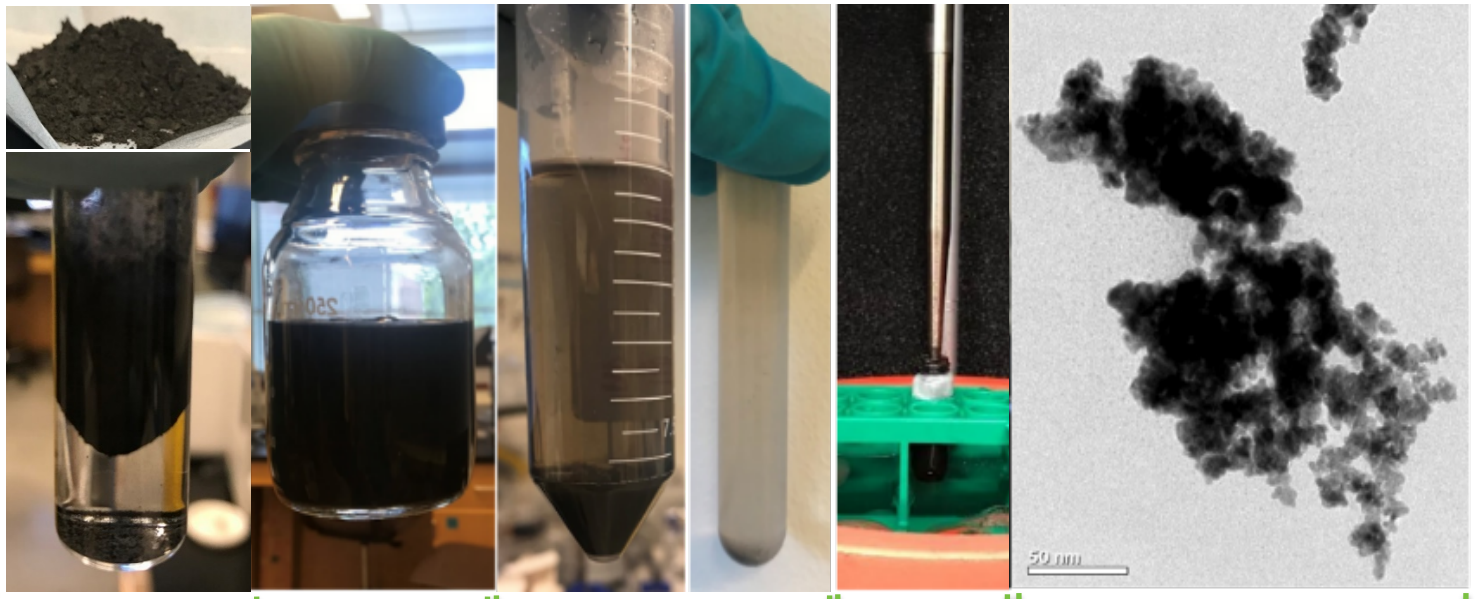


Accomplishments: Ink and Feedstock Fabrication

Top-Down Methods

- *All developed inks were synthesized via top-down methods*

Top down platinum ink synthesis for AJP/PJP

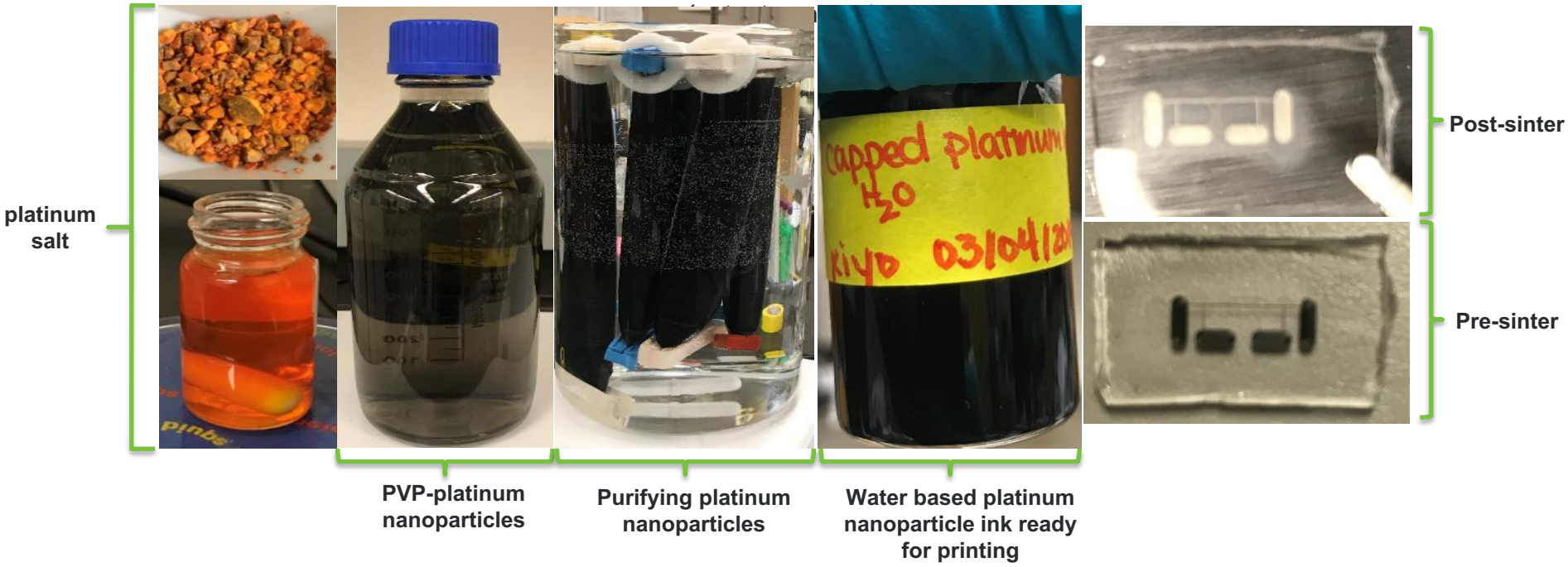


Accomplishments: Ink and Feedstock Fabrication

Bottom-Up Methods

- *Bottom-up synthesis allows for tighter nano-particle size distribution and smaller sizes*
- *These methods are being published and may be used by commercial companies to expand the commercially available materials*

Bottom-up platinum ink synthesis for AJP/PJP

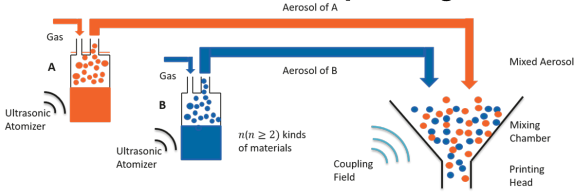


Accomplishments: Combinatorial Materials Science

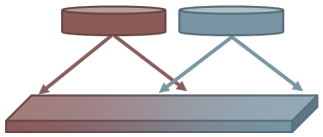
Combinatorial Materials Science

- *Combinatorial materials science combined with high-throughput screening methodology offers potential for rapid discovery and development of radiation-resistant sensor materials*
- *Two major challenges associated with combinatorial materials science that must be overcome for valid high-throughput testing*
  - *Creation of the combinatorial material – A design has been created and components purchased for a combinatorial printer. Multi-target sputtering has also been explored for created graded compositions*
  - *Localized measurements – A high resolution thermal microprobe capable of both thermal conductivity and Seebeck coefficient measurements* has been developed, along with an electrical conductivity microprobe

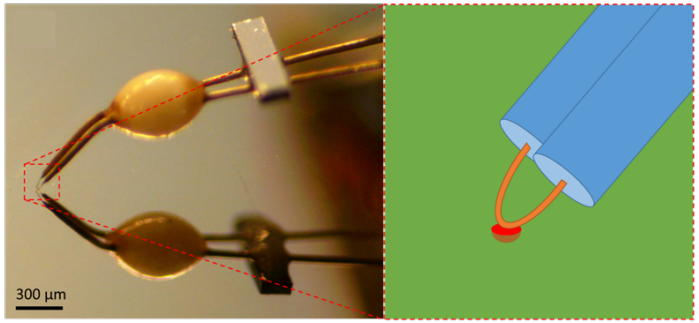
Multi-material printing



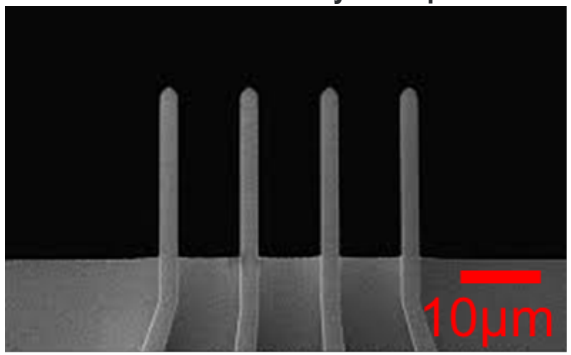
Sputtering



Thermal conductivity and Seebeck coefficient microprobe



Electrical conductivity microprobe

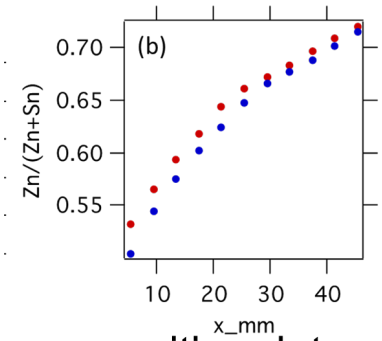


Accomplishments: Combinatorial Materials Science

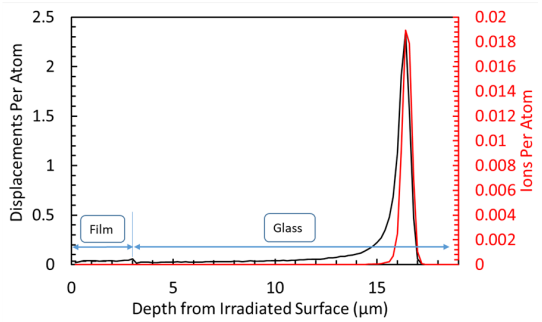
Combinatorial materials science

- *A trial run was performed this year to demonstrate this technique on a combinatorial $ZnSnN_2$ film manufactured via sputtering.*
- *This will be expanded to other material of interest to improve sensor base properties and irradiation resistance*

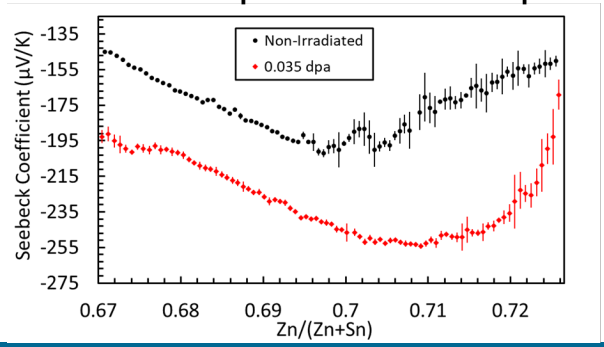
Graded composition create by sputtering

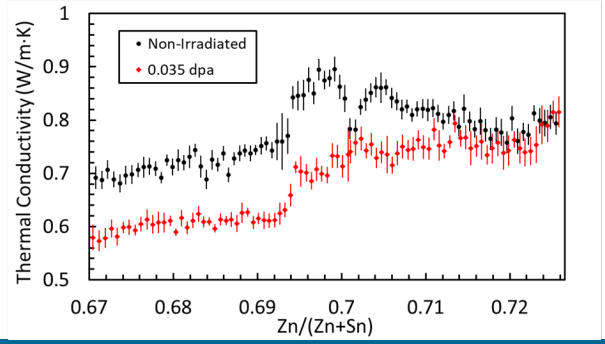


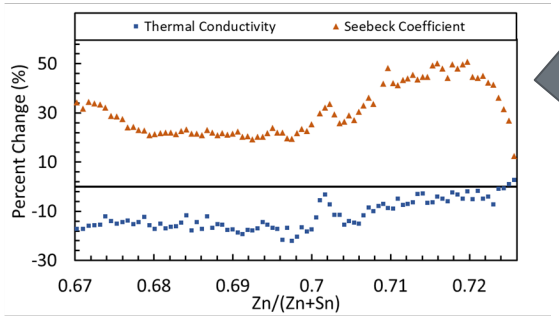
Irradiated with He ions



Properties vs composition determined for unirradiated and irradiated material







Technology Impact

- Our work is focused towards advanced manufacturing for in-pile applications
- We currently have a program interested in implementing advanced manufactured sensors (AFC-FAST)
- **InFlex, LLC –** Spin off from ink synthesis developments.

INFlex Labs, LLC.
Materials and Technology for a Flexible World

- A provisional patent has been assigned for the work on aqueous based inks
  - "Aqueous Based Nanoparticle Ink For Aerosol Jet Printing-Ultrasonic Atomizer and Plasma Jet Printing and Other Printed Electronic Direct Write Methods"

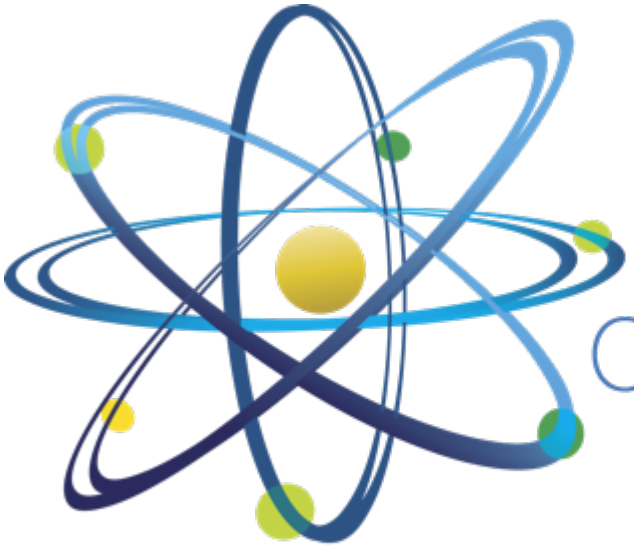
Conclusion

Future Work

- One of the primary focuses of FY20 will be thermocouples, examining printed thermocouples and using combinatorial material studies to improve the thermocouple material.
- Ink synthesis will shift to piezo-electric and magnetostrictive inks for use with the ultrasonic thermometer.
- Additional work will examine additional advanced manufacturing techniques for cases where inkjet printing is not adequate

Questions?

Follow up questions after the webinar can be sent to:
michael.mcmurtrey@inl.gov



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