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

ASI | Advanced Sensors and Instrumentation

Passive Monitors: Silicon Carbide Temperature Monitors and Printed Melt Wires

Advanced Sensors and Instrumentation (ASI) Annual Program Webinar

October 29th – November 2nd , 2022

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Project Overview

- **Brief summary of research scope:**

Passive temperature monitors are needed for when real-time sensors are not practical or economical to install in an irradiation test. The main purpose is to provide a practical and reliable approach to estimate irradiation temperature during post-irradiation examination (PIE) for direct integration in irradiation test designs. Passive temperature monitors have been in use for many decades in irradiation testing experiments, but limited innovation has been applied to these technologies. This work package focuses on two activities: Silicon Carbide (SiC) Temperature Monitors (TMs) and Printed Melt Wires.

- **Project Schedule:**

Due October 30th, 2023 (delayed by 2 months): Complete level 3 milestone (M3CT-23IN0703026) report titled "Assessment of read-out techniques for passive monitors."

- **Participants:**

Malwina Wilding (WPM and PI) and Kiyo Fujimoto (co-PI) – Idaho National Laboratory

Collaborators: David Estrada and Josh Eixenberger – Boise State University

Austin Fleming (TPOC)

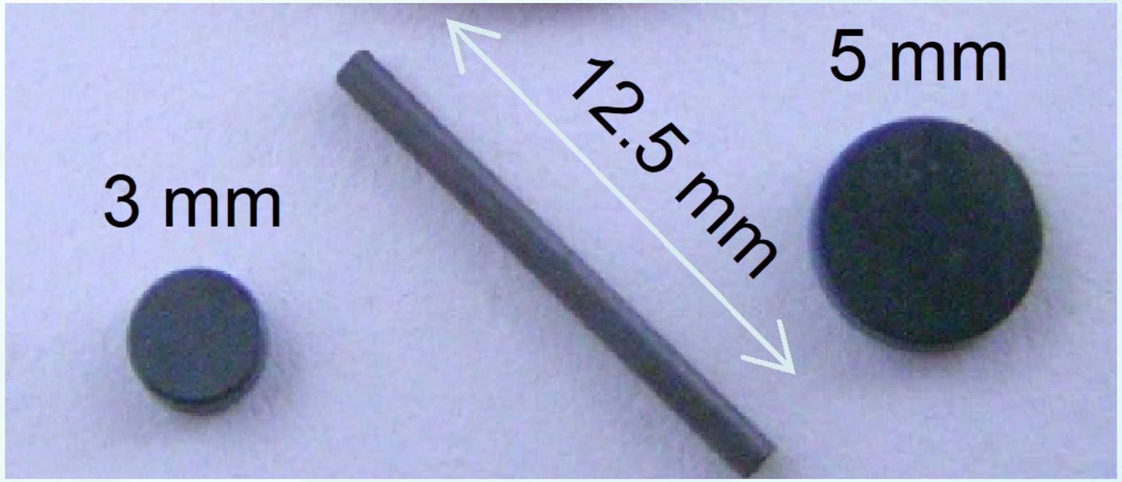
Idaho National Laboratory

Technology Impact

- Passive monitors provide a practical, reliable, and robust approach to measure irradiation temperature during post-irradiation examination while requiring no feedthroughs/leads comparable to current more-complex real-time temperature sensors
- They have been chosen because they have a proven history for use by stakeholders for deployment and require continued development and characterization to assure successful integration with program schedules and objectives
- Further develop the temperature passive monitor capability for wider rage of temperatures, geometries and neutron damage
- Facilitates the development of advanced sensors and instrumentation with cross-cutting technology development to support the existing fleet, advanced reactor technology and advancing fuel cycle technology development

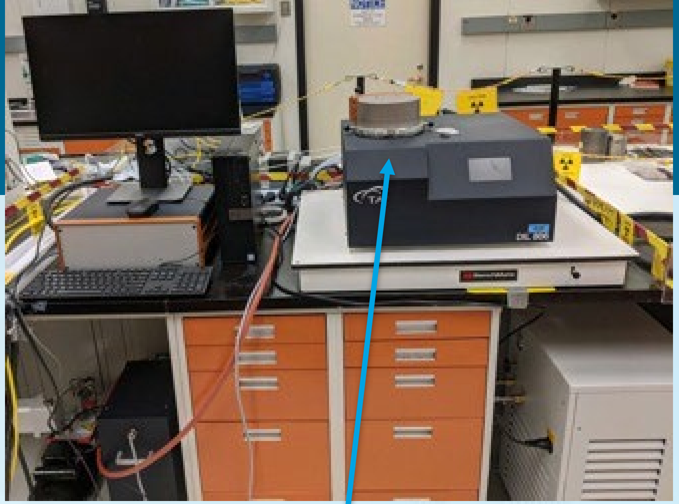
Silicon Carbide Temperature Monitors (SiC TMs)

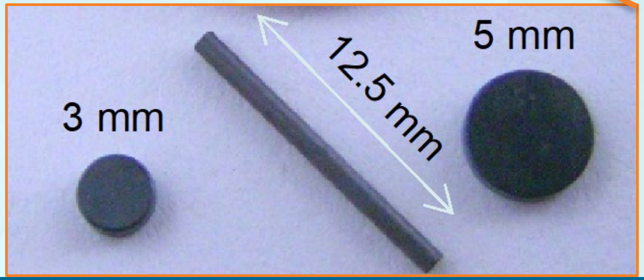
Since the early 1960s, SiC has been used as a post-irradiation temperature monitor. Researchers observed that SiC's neutron-irradiation-induced lattice expansion annealed out when the post-irradiation annealing temperature exceeded the peak irradiation temperature. FY23 work for SiC monitors conducted further verification testing of the optical dilatometry method by using eight 3-mm SiC disks provided by NSUF's N-SERT experiment. This will conclude continuous optical dilatometry to be a valid method for measuring peak irradiation temperatures of various shapes and sizes of SiC TMs (12.5 mm long sticks, 3 mm and 5 mm disks).



Dilatometry Method

- Automated process requiring minimal setup time
- Dilatometer runs under vacuum or inert gas
  - key requirement for avoiding any oxidization issues
- Max. operating temperatures of 1400ºC (resolution of 0.1ºC)
  - SiC TMs working temperature range of 200 – 1200ºC
- Reduced time expense
  - Time to process each sample 2 to 3 days (target temperature dependent)
- Contactless dilatometric measurement system
  - Allows samples to freely expand/shrink without any interference from mechanical contact
- Can process all SiC TMs (rod, 3-mm and 5-mm disks)
  - 0.3–30 mm in length with a maximum height of 10 mm





Issues (Schedule/Cost/Technical)

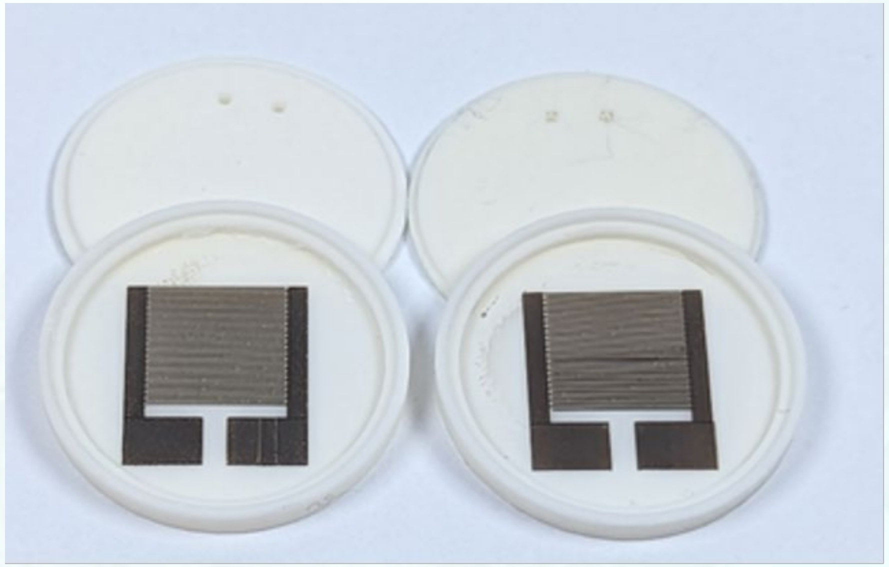
- NSUF provided SiC temperature monitors:
  - N-SERT Experiment with 8 SiC 3-mm disks
- Multiple Delays:
  - Acquiring irradiated SiC disks from previous experiments
  - Rad Material Shipping between MFC, ATR and IRC
  - Decontaminating twice (MFC and HF wash at ATR)
  - Dilatometer currently shipped to Germany for repairs (loose TC connection)
- Highlight:
  - All 8 NSUF SiC TMs successfully decontaminated and awaiting processing in the optical dilatometer at IRC

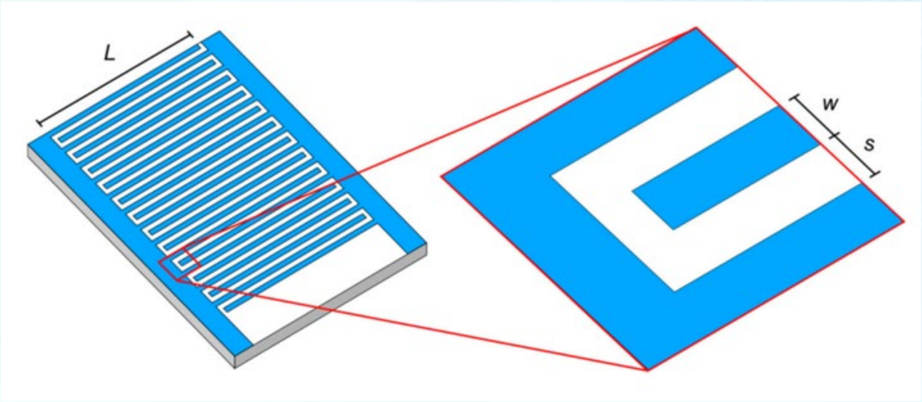
Remaining Work for FY23

- Anneal all 8 SiC 3-mm disks in the optical dilatometer
- Analyze each disk for its irradiation temperature
- Write Summary Report
  - Level 3 milestone report titled “Assessment of read-out techniques for passive monitors.”

Printed Melt Wires

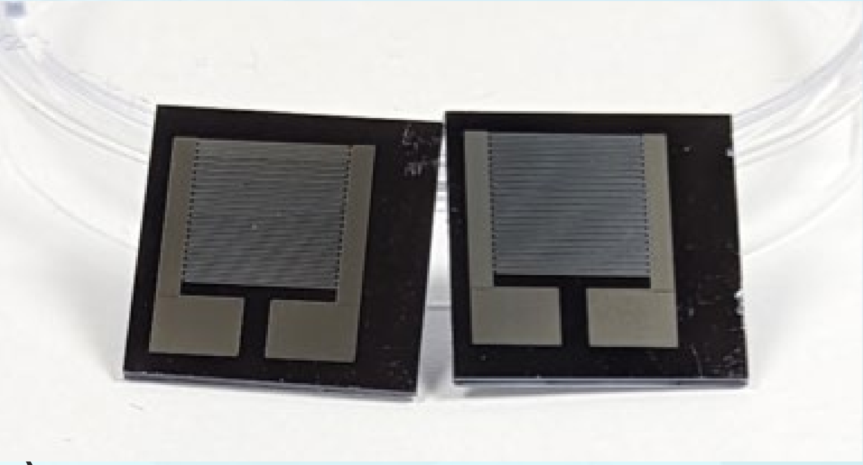
One method to determine irradiation temperature involves placing material wires of known composition and melting temperature in an irradiation test. In this method post-irradiation examination of each melt wire is required to determine if the melting occurred, indicating that the corresponding melting temperature was reached or exceeded. Sensor development using Advanced Manufacturing methods enables the production of robust and miniaturized sensors for nuclear application and expands the current capability of using passive melt wires.



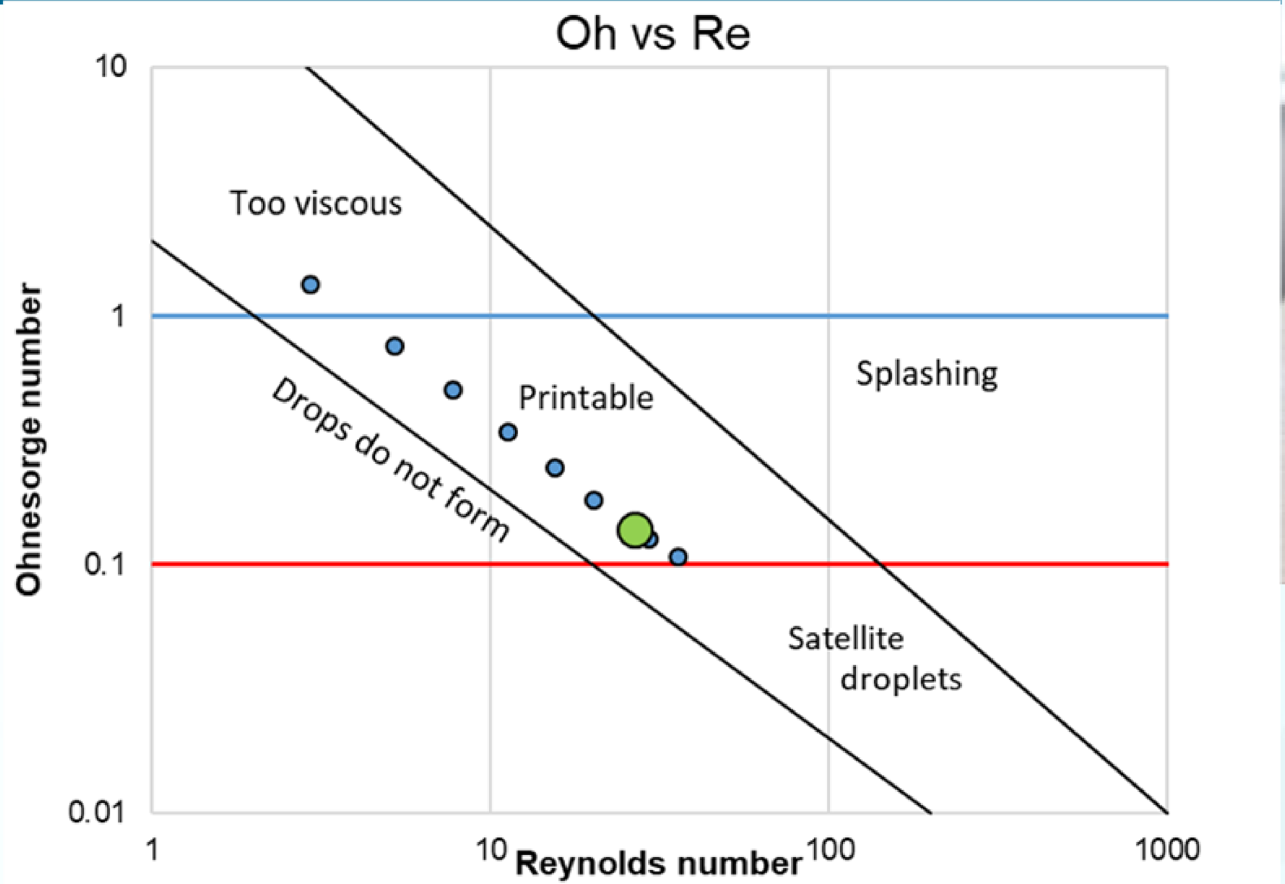


Progress on Printed InterDigitated Electrodes (IDE) Melt Wires

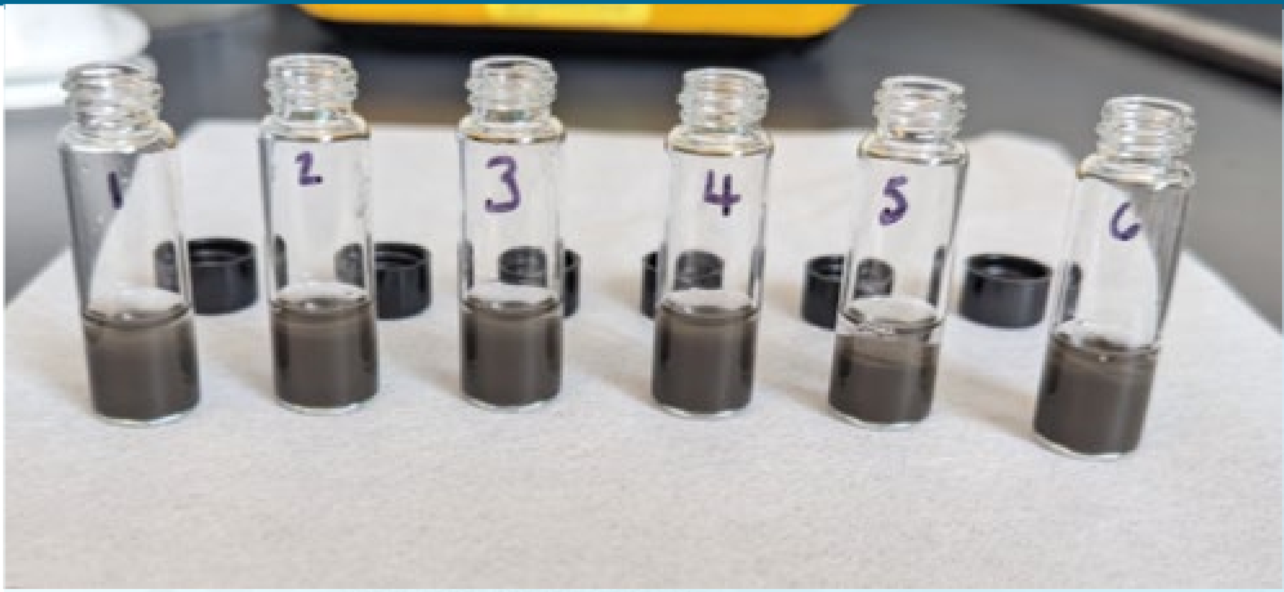
- Obtained: PVP (Polyvinylpyrrolidone) coated Tin, Zinc Nanoparticles (US NM Research), and Indium Ink (Applied Nanotech)
- New Hires:
  - Brandon Ortiz (Physics major- undergraduate student)
  - Dr. Nick McKibben (PhD, Materials Science and Engineering)
- Stability test of Tin NPs in various co-solvents
- Rheological properties of co-solvent system
- Characterization of nanomaterials
- Size separation
- Ink formulation
- Printing of Tin interdigitated electrodes (planar capacitors)



Stability Study



Rheological properties of different ratios of co-solvent systems.
For Ink Jet Printer, comparison of the Reynolds number vs. Ohnesorge number indicates if the solvent combinations fall within the printable window. Green dot- Includes NPs with co-solvent, demonstrating at 10% loading, minimal effects on rheological properties (overlapping with blue dot that utilizes the same co-solvent system).



Rheological study with different ratios of co-solvents

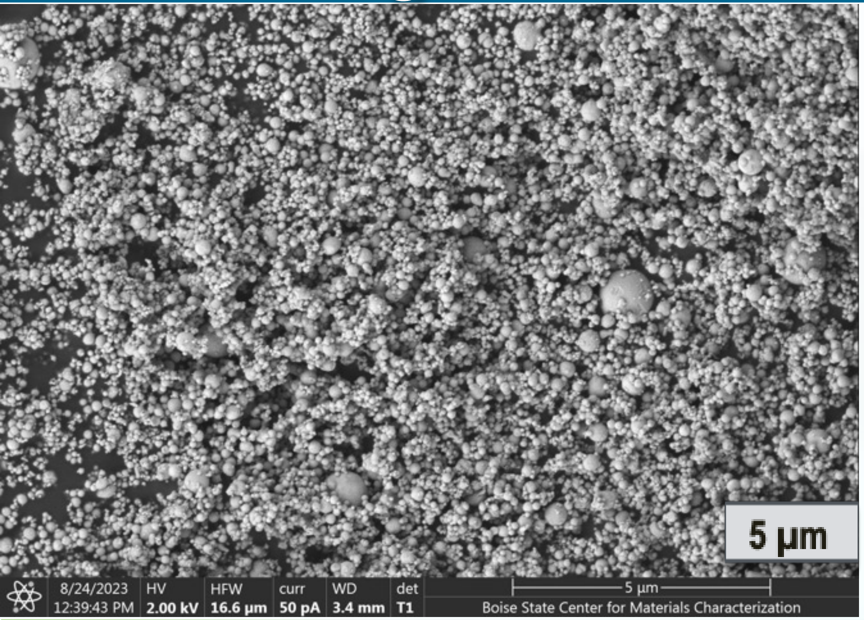
- Conducted nanoparticle stability study with different ratios of co-solvents
- 1-5: Glycol- Isopropyl Alcohol- Ethanol at different ratios
- 6: Tin NPs dispersed in water- most stable
- Improved stability in water likely due to PVP (hydrophilic) capping of nanoparticles

Characterization of Nanoparticles

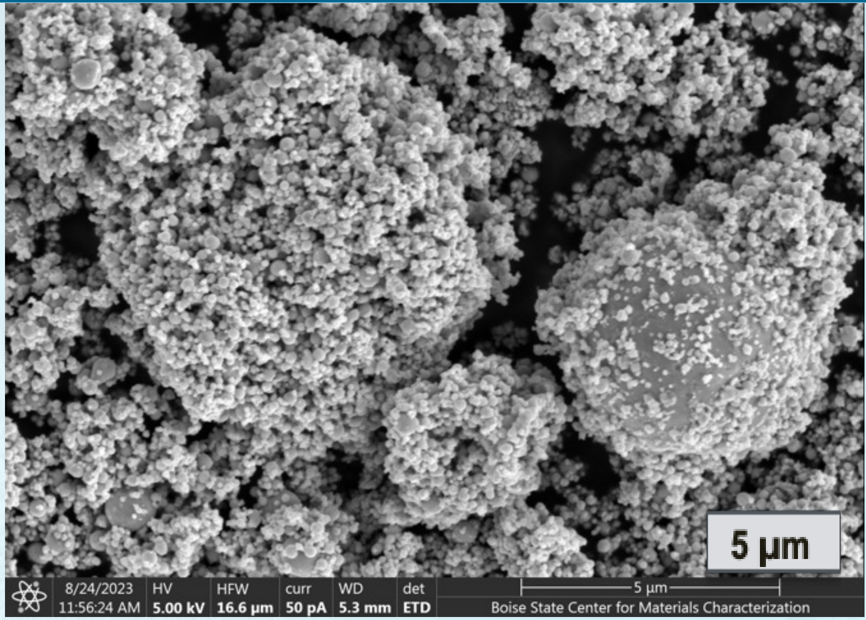
- SEM Images for morphology and size distribution
  - Spherical
  - Large size distribution (some >5 um)
  - Requires size separation to be compatible with printers
- TGA/DSC- PVP content & sintering/melting characteristics
  - A small mass loss observed up to ~215 °C but mass gained at higher temperatures
  - Possible oxidation or formation of tin nitrate- Needs verification
  - Will do TGA/DSC in Argan atmosphere and perform X-Ray Photoelectron Spectroscopy (XPS) analysis

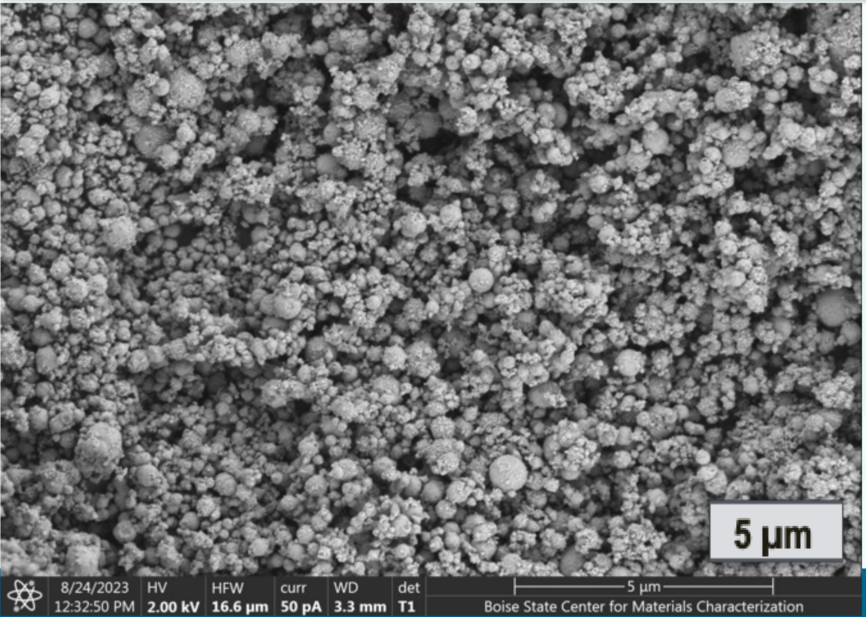
SEM Images

As-Received nanoparticles show a big size distribution but the majority are in the nanometer scale. The larger particles can potentially clog printer nozzles and/or may not aerosolize requiring separation by size.

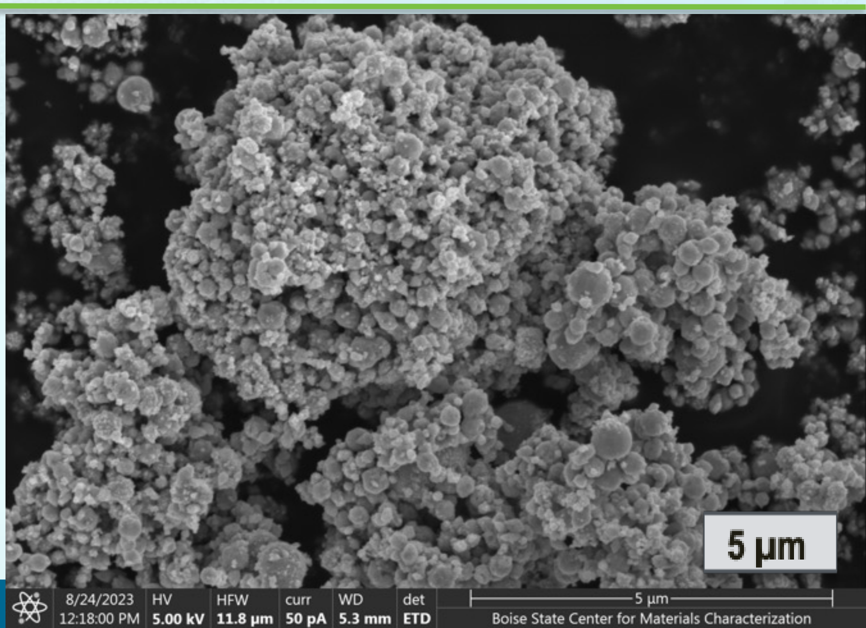


As-Received PVP capped Tin Nanoparticles

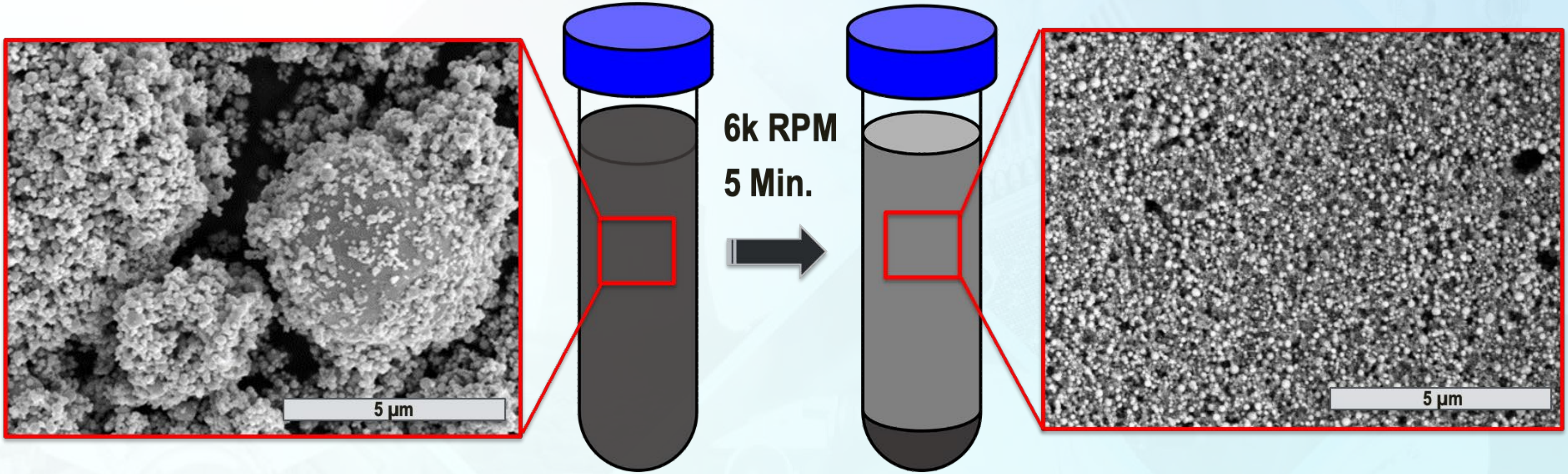




PVP capped Zinc Nanoparticles As-Received

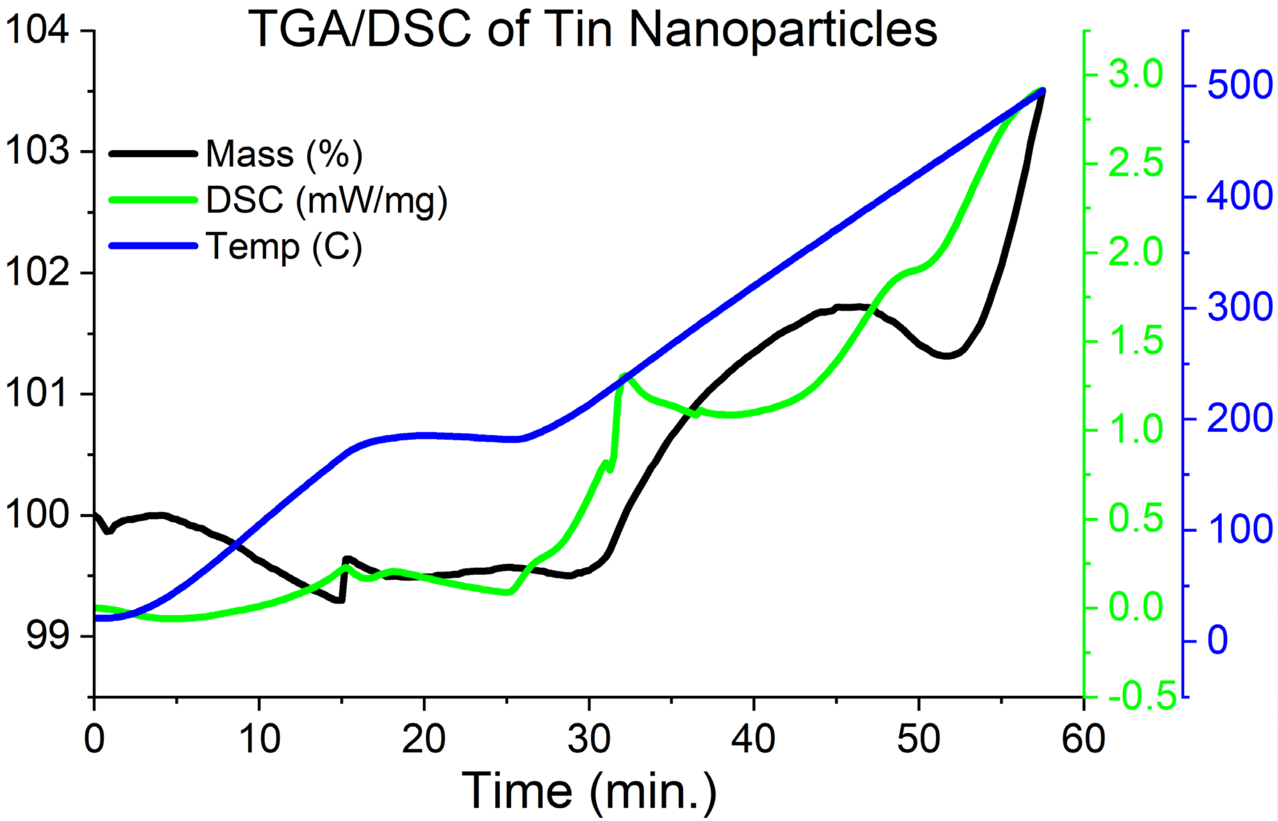


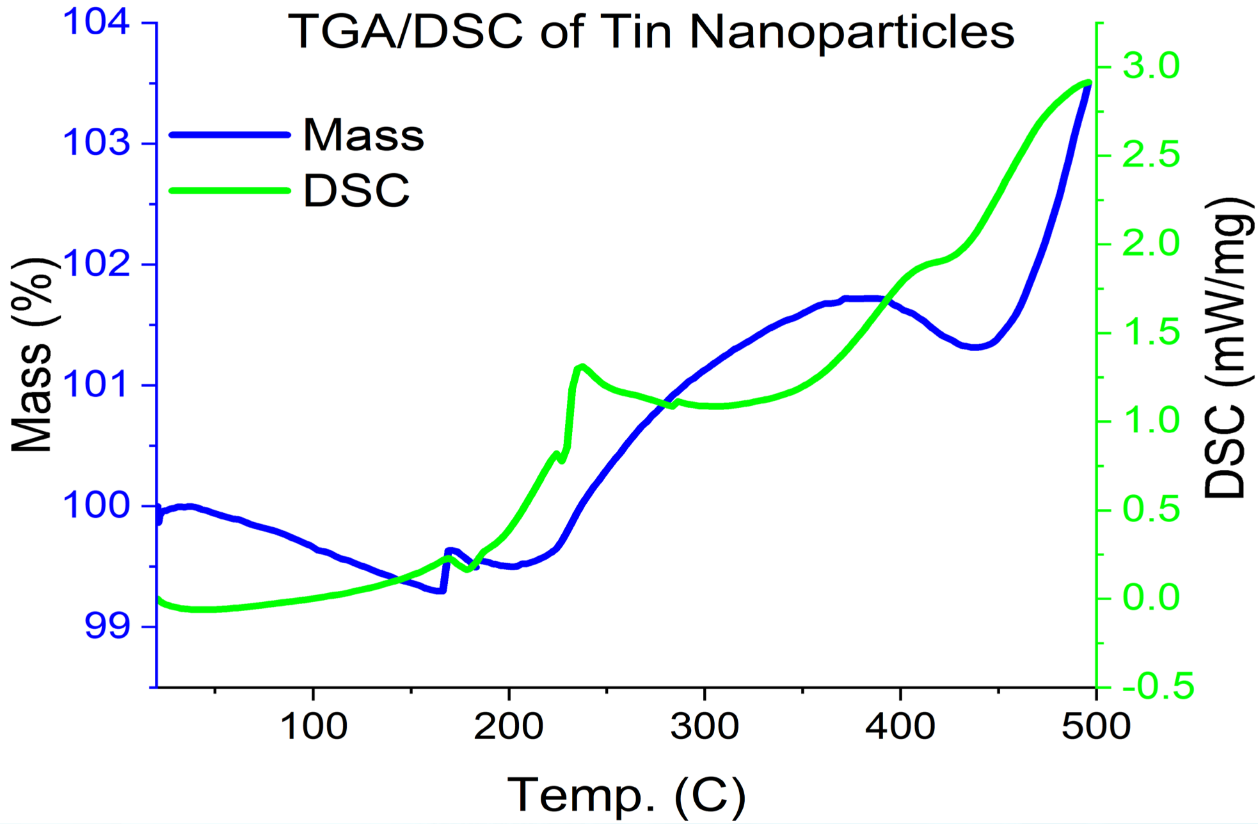
Nanoparticle Size Separation- Printer Compatibility



As-received Tin nanoparticles had a large size distribution with many particles > 1 µm. Large particles can clog printer nozzles and for AJP, likely will not aerosolize. The large particles were separated from the small particles via centrifugation at 6k RPM for 5 minutes. The small particles remained suspended, were collected, and dispersed into a co-solvent system of ethylene glycol, Isopropyl Alcohol, and water to formulate a printable ink. SEM images were collected before and after separation to confirm removal of the large particles.

Thermogravimetric Analysis/Differential Scanning Calorimetry of Tin Nanoparticles in Nitrogen Atmosphere





TGA/DSC measurement conducted with an isotherm at 175 °C for 10 minutes
(Melting Point at ~150 °C)

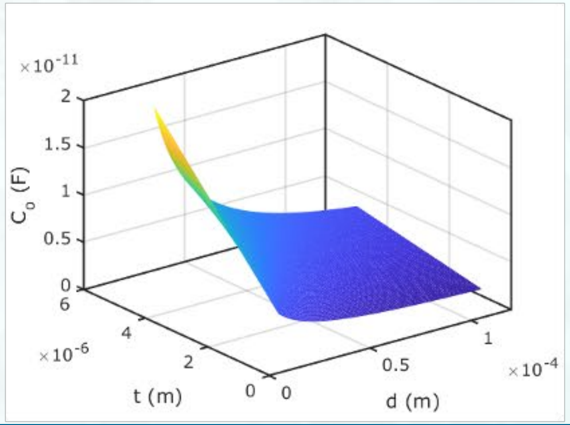
TGA/DSC measurement conducted without an isotherm to estimate PVP content from decomposition. Note, the sample gained mass as the sample was heated to 500°C.

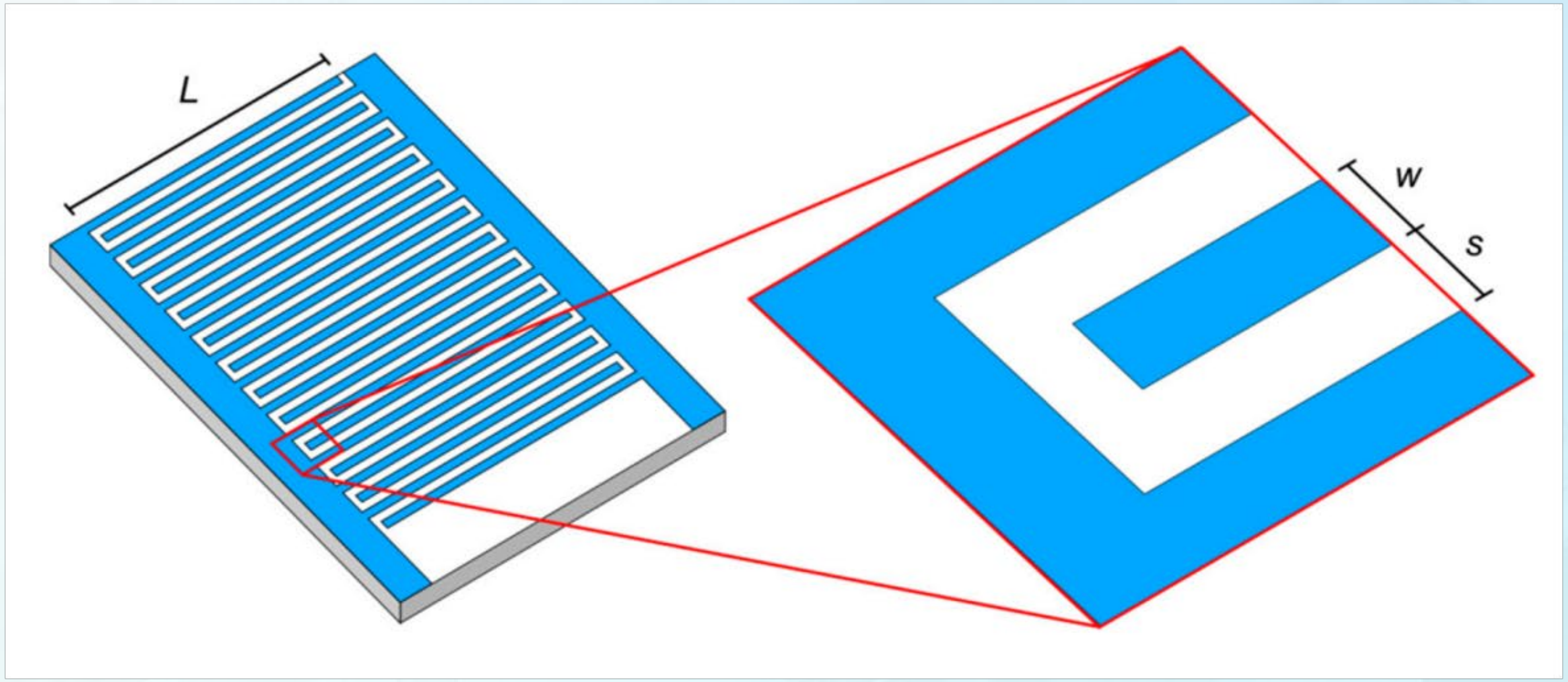
Capacitance of Interdigitated Electrodes (Planar Capacitors)

Analytical Model of IDE Capacitor

$$C_o = (k_r \times k_0)(2l - a + 2w)\left(\frac{2n-1}{s}\right)$$

Symbol	Definition
k_r	Relative permittivity
k_o	Free space permittivity
l	Length of an IDE digit
a	Width of capacitor
w	Width of an IDE digit
s	Distance between digits
n	Number of digit pairs
t	Thickness of electrode

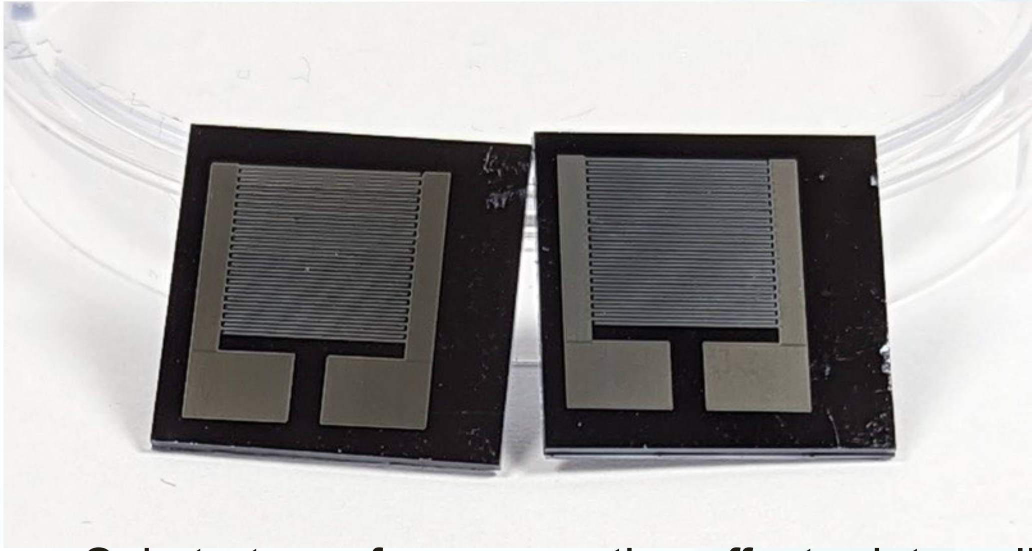




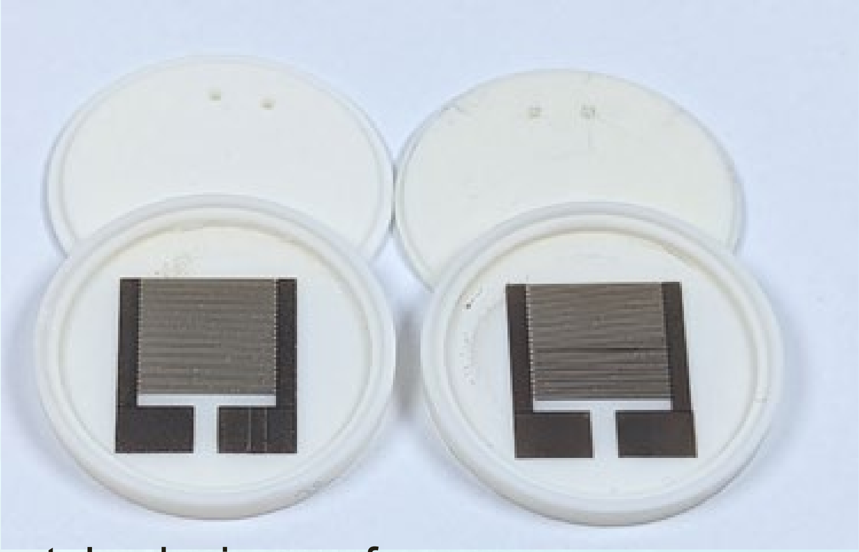
Study of Interdigitated Electrode Arrays Using Experiments and Finite Element Models for the Evaluation of Sterilization Processes, Oberlander et. al, *Sensors*, 2015, 15, 26115-26127; doi: 10.3390/s151026115

Printed Tin Interdigitated Electrodes (In-plane Capacitors)

IDEs printed with Tin nanoparticle ink on SiO_2.



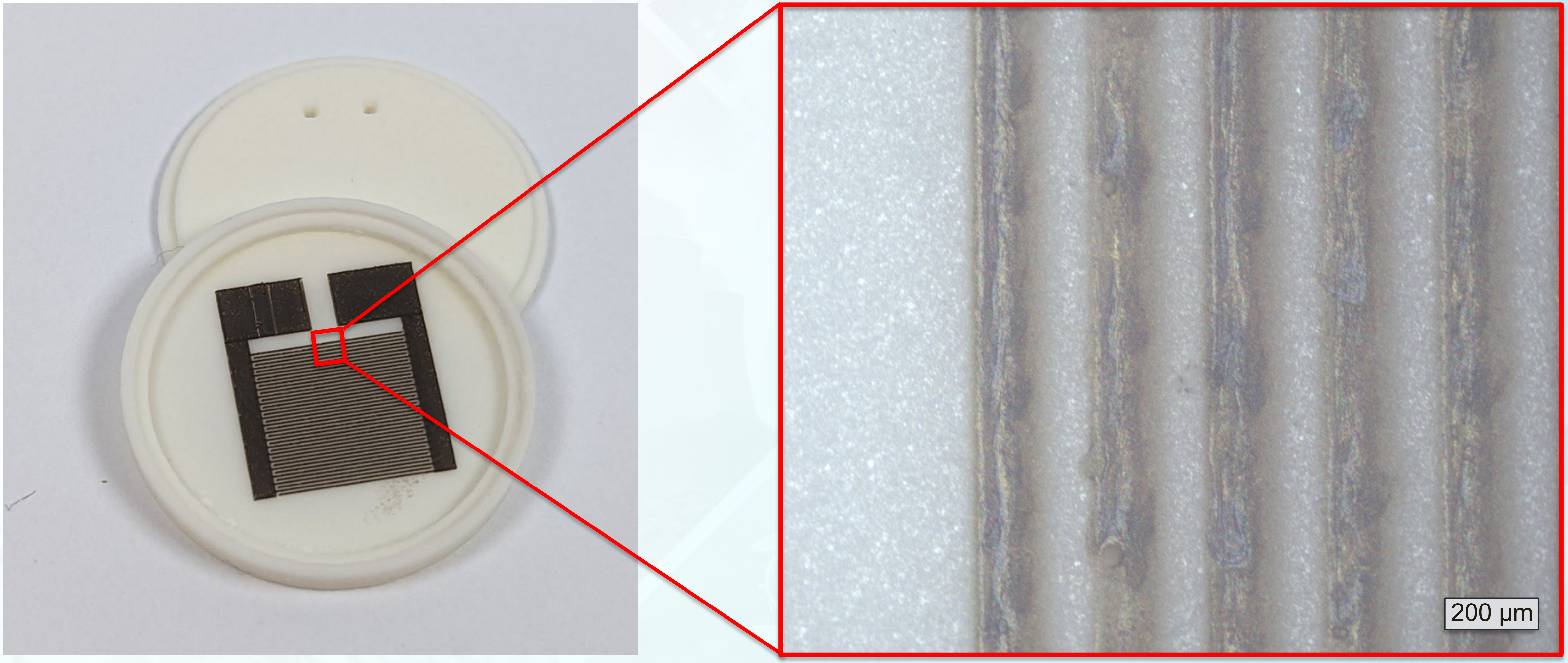
IDEs printed with Tin nanoparticle ink on additively manufactured alumina container.



- Substrate surface properties affect print quality and ultimately, device performance
- Exploring direct print of IDEs on 3D printed alumina container vs. prints on coupons that can be enclosed within the container.
- Electrical feedthroughs were fabricated in lids for real-time monitoring of devices and/or for evaluations post thermal treatment, removing the need for visual inspection
- Devices on all substrates will be thermally cycled with evaluations done through capacitance measurements and visual inspection
- Comparative analysis will be performed between substrates to understand effects on device performance and inform final designs

Printed Tin Interdigitated Electrodes (In-plane Capacitors)

IDEs printed with Tin nanoparticle ink directly on alumina container.



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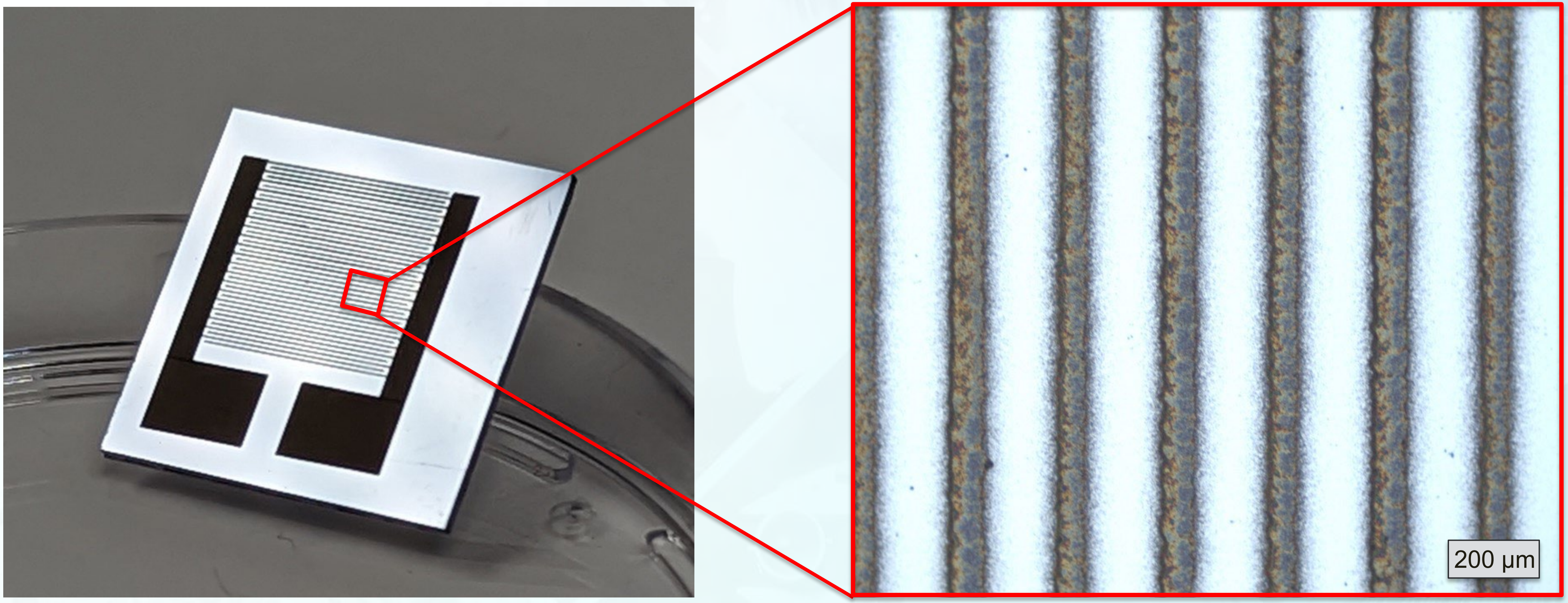
IDEs printed with Tin nanoparticle ink on SiO_2.



Results and Accomplishments – Ceramic Container

<u>Printing technology used:</u>

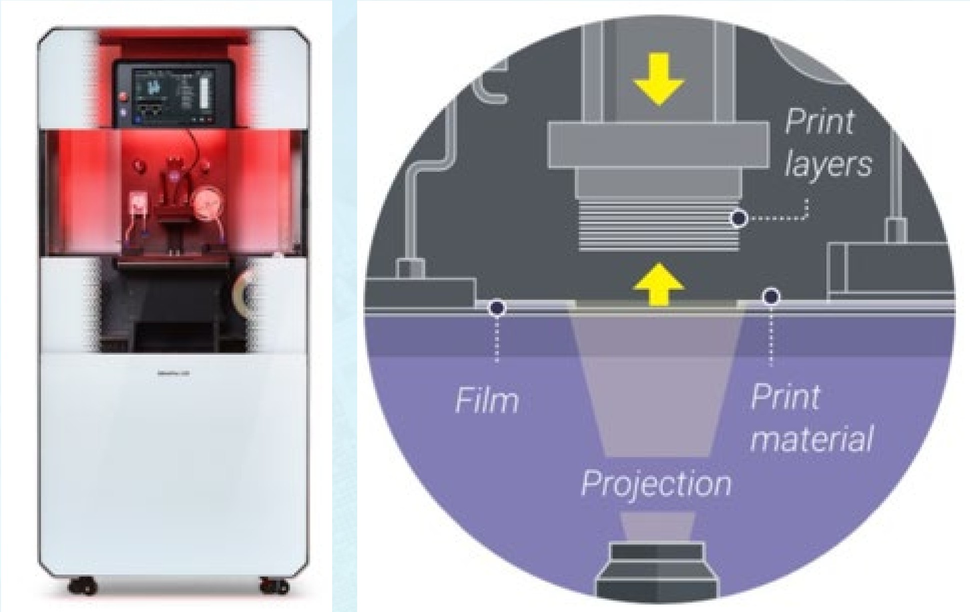
Digital Light Processing or Vat Photo Polymerization

- Uses a projected light source to cure an entire layer at once
- Layer Thickness: 10 - 200 μm
- Wall thickness: 0.1 – 10 mm (alumina)
- Capable of printing ceramics and metals:
  - alumina, zirconia, fused silica, and hydroxyapatite, stainless steel 316L, stainless steel 17-4, inconel 625, and copper

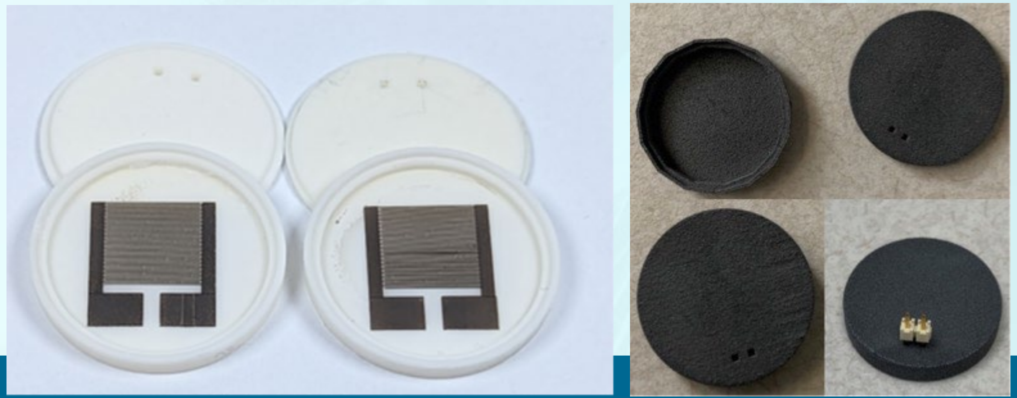
<u>Ceramic Container:</u>

- Alumina and Polymer
- Insulation layer to minimize premature melting

Admaflex 130



DLP alumina container



Results and Accomplishments – Printed Melt Wires

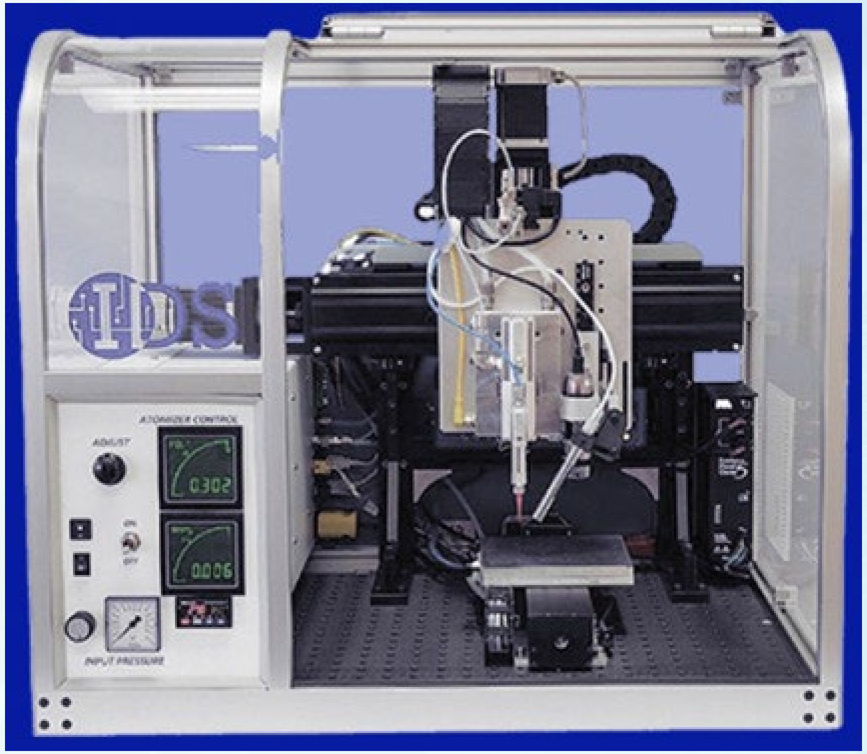
Aerosol Jet Printer – IDS Nanojet

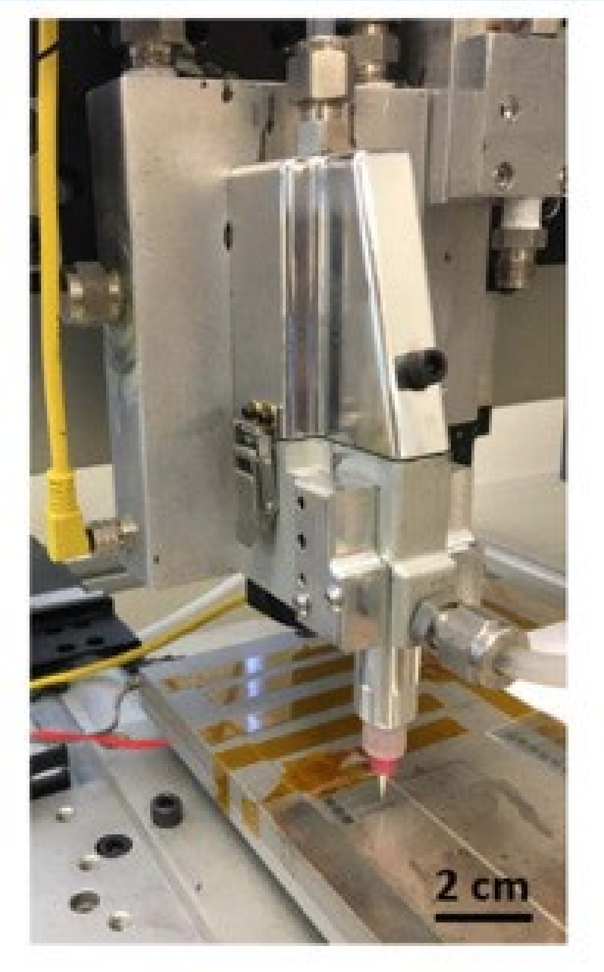
Aerosol Jet Printing:

- Creates an aerosol of an “ink” to deposit directly onto a substrate
- 10 µm – 5 cm linewidths
- 0.7 – 5000 cP ink viscosity
- 2 – 10mm working distance
- Can print a wide variety of materials
- Maximum particle size = 200 nm
- 3-axis motion control

Printed Melt Wires:

- Indium, tin and zinc melt wires were fabricated with AJP
- The entire capsule was sintered at 100 °C





Issues (Schedule/Cost/Technical)

- Delay in receiving BSU contract (awarded in April of 2023)
- Assessing material availability and receiving initial quotes
- Delays in receiving materials and fabrication times
- Ceramic Printer issues (MFC and IF printers broke at the same time)

Remaining Work

- Remaining Work:
  - Perform temperature testing on printed IDE devices to show passive melt behavior and effect on capacitor.
  - Report on integrated sensor package
  - Write Summary Report
    - Level 3 milestone report titled “Assessment of read-out techniques for passive monitors.”

Concluding Remarks

- SiC TMs:
  - Received eight 3-mm SiC disks from NSUF's N-SERT experiment
  - Fully decontaminated all SiC TMs
  - Awaiting to anneal and analyze in the optical dilatometer
- Printed Melt Wires:
  - Received all nanoparticles (Indium, Tin, and Zinc)
  - Fully characterized all nanoparticles
  - Printed ceramic containers with electrical feedthroughs
  - Printed Tin circuit on ceramic containers
  - Awaiting to test printed melt wires via capacitance readout technique
- Finish Level 3 Milestone Report
  - Assessment of read-out techniques for passive monitors

Contact information ->

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Thank You