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

Advanced Sensors and Instrumentation

Testing and Development of Strain Sensors for Structural Materials Characterization

Advanced Sensors and Instrumentation (ASI) Annual Program Webinar
October 30 – November 2, 2023

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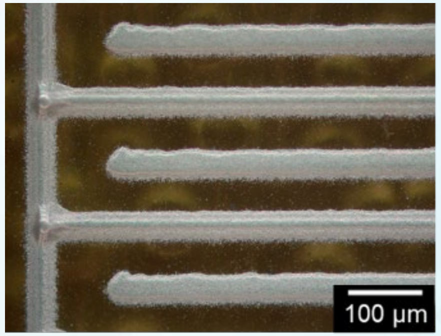
Project Overview

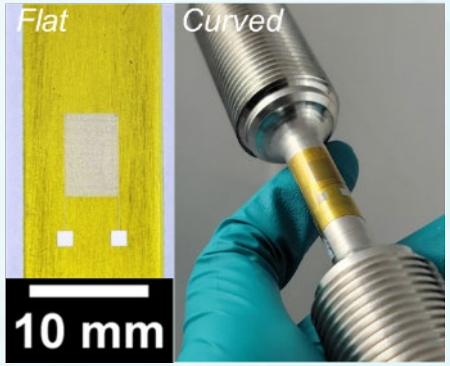
To expand the capabilities for monitoring mechanical properties and the structural health of material specimens in test reactor experiments with strain gauge technologies.

Research Scope/Objectives

Printed strain gauge development to ensure operation limits greater than 300 °C:

- Development of necessary materials for the multi-layered strain gauge system (i.e., insulation, encapsulation)
- Determine methods for sensor robustness and quality control
- Validate printed sensor operation up to 500°C





Participants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Brian Jaques (BSU)

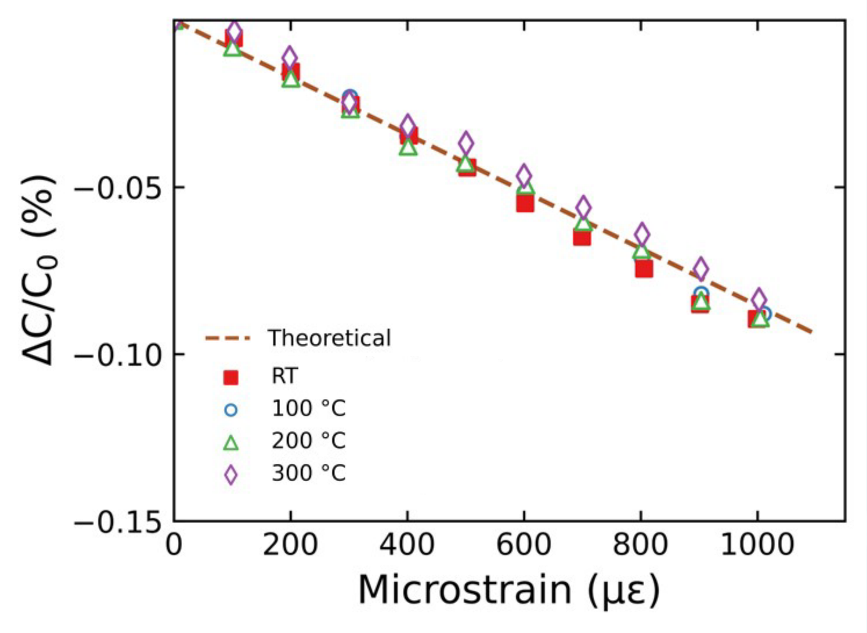

David Estrada (BSU)

Project Schedule for FY24

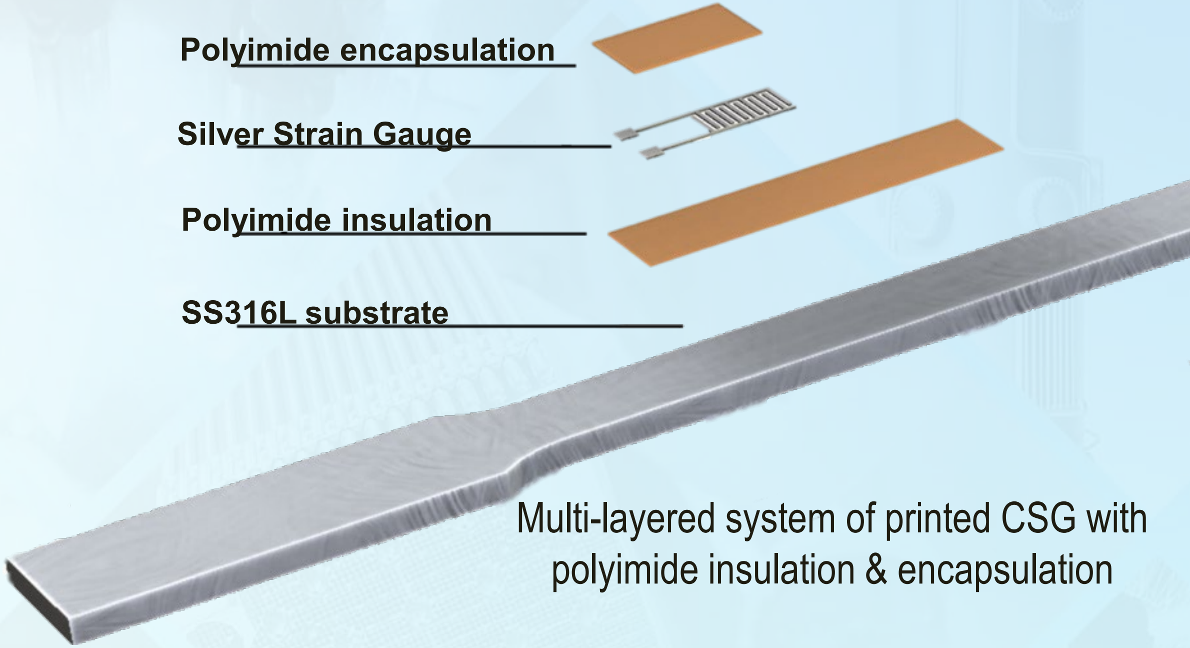
- Fabricate and test printed strain gauges that can measure mechanical strain up to 750°C
- Continue developing quality control metrics (adhesion) for additive manufactured sensors and instrumentation
- Test strain gauge in relevant harsh environments (i.e., irradiation)

Project Overview – FY22 Strain gauge on polymer; tested up to 300°C

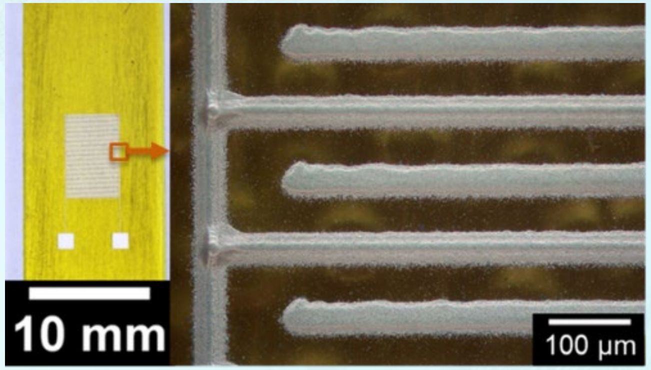
- Printed capacitive strain gauges (CSG) were printed and tested on a SS316 tensile specimen up to 300°C
- The CSGs with polyimide insulation/encapsulation matched well with the theoretical models that shows the sensitivity of the strain gauge on SS316L



Mechanical strain results from printed strain gauge



Multi-layered system of printed CSG with polyimide insulation & encapsulation



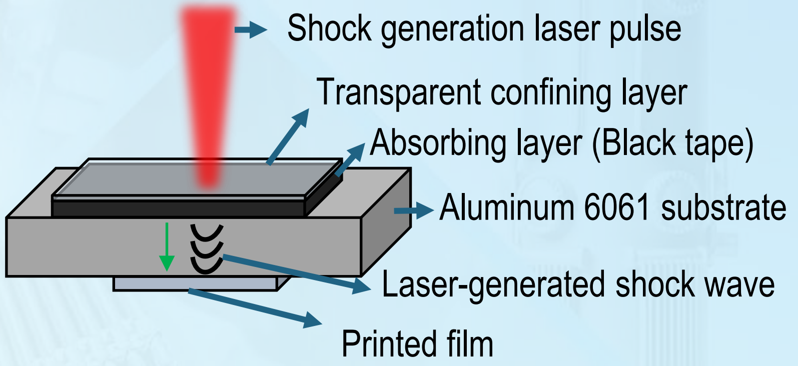
Representative image of CSG on polyimide insulation without the encapsulation

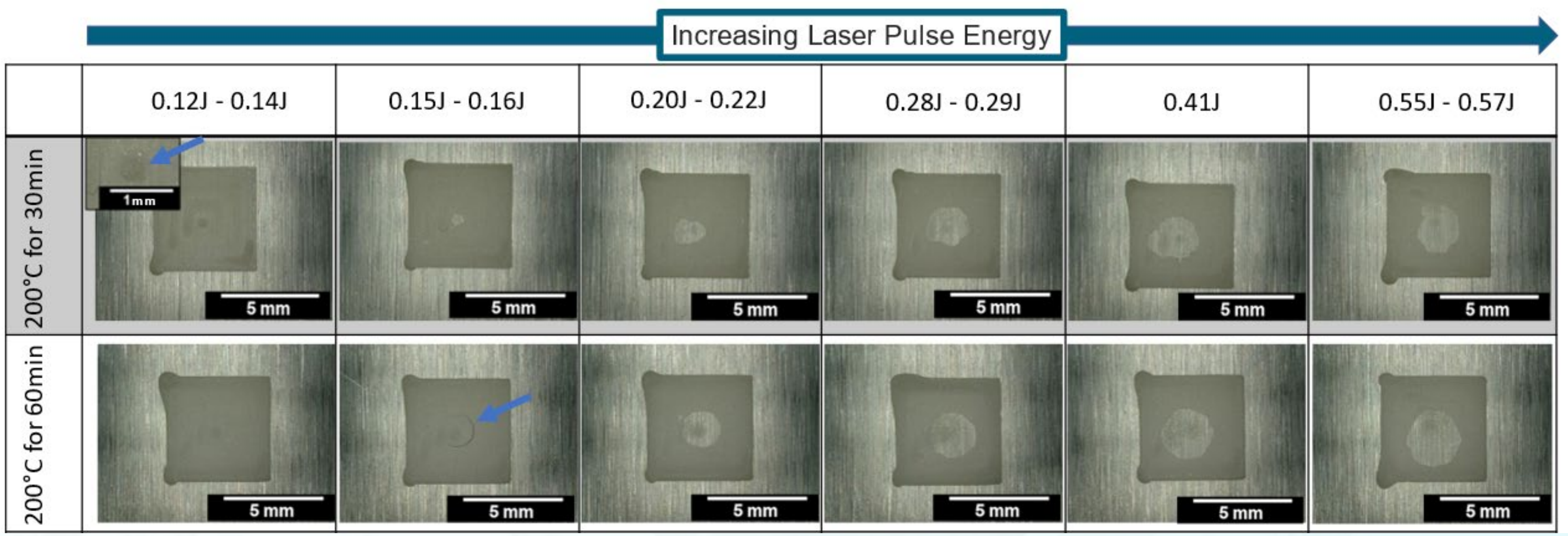
***<u>Outcome for FY23</u>**: Use ceramic insulation/encapsulation to increase operating temperature of CSG*

[1] Phero, T.L., et al., *Additively manufactured strain sensors for in-pile applications.* Sensors and Actuators A: Physical, 2022: p. 113691.
[2] Kim, S.R., et al., *Wearable and transparent capacitive strain sensor with high sensitivity based on patterned Ag nanowire networks*, ACS Appl. Mater. Interfaces 9 (2017) 26407–26416.

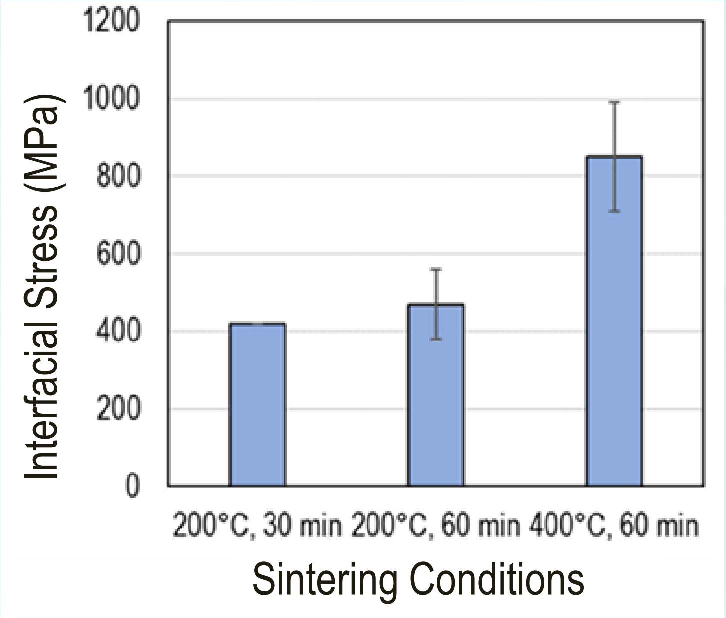
Project Overview – FY22 Quality control methods for reliable design

- A laser-induced spallation and baseline pull-off adhesion measurements were used to quantify the adhesion strength at the interface of the print and substrate
- Laser spallation is non-contact, has a quicker preparation time, and requires smaller "sacrificial" samples since it's spatially localized to less than 25 mm^2





Results from monotonically increasing the laser pulse energy for samples sintered at three different conditions; arrows indicate onset of failure



Interfacial stress induced by laser spallation at the onset of interfacial failure at the print/substrate interface

Outcome for FY23: *Incorporate developmental laser spallation technique to the substrate/insulation interface*

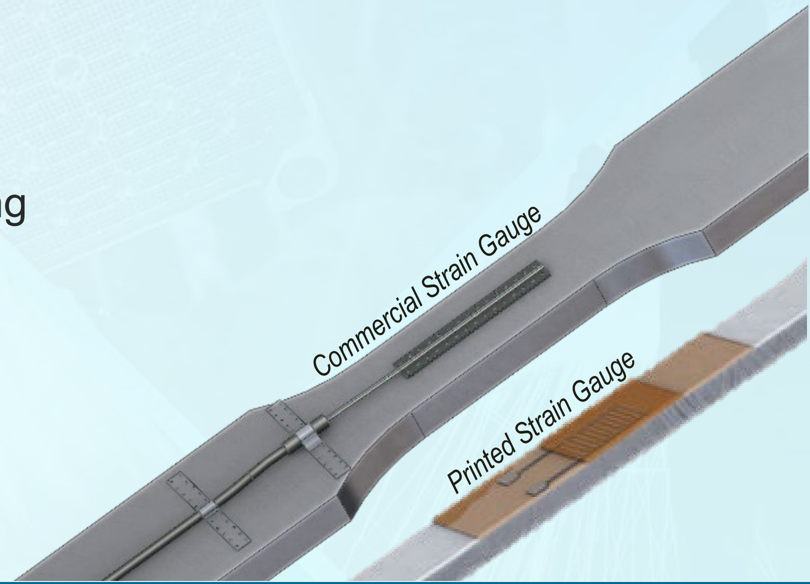
Technology Impact

Technology application

- **Ubiquitous requirement**: Harsh environment strain sensing is needed in many industries as it provides crucial, real-time data on expansion and swelling of materials
- **Baseline strain measurement**: Commercial strain gauges provides validation metrics for both developmental strain sensing technologies and modeling/simulation efforts
- **Expands strain sensing capabilities**: Printed strain gauges are not looking to replace traditional high-temperature strain gauges, however, allow strain gauges to be applied in areas where specialized requirements are required (e.g., materials restriction, attachment limitations, miniaturized specimen)
- **Sensor Qualification Methods**: Inspection, tests, and quantifiable assessments ensure that the printed sensors are compliant and reliable prior to its deployment

Impacts on the nuclear energy industry

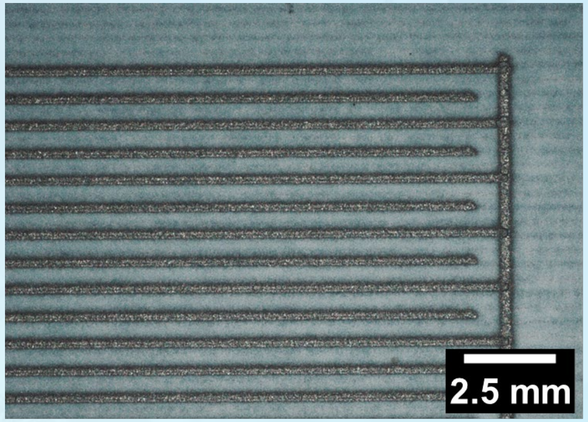
- These sensors enable data acquisition for improved material testing and validating modeling and simulation efforts to support the development, testing, and qualification of new nuclear materials during in- and out-of-core experiments

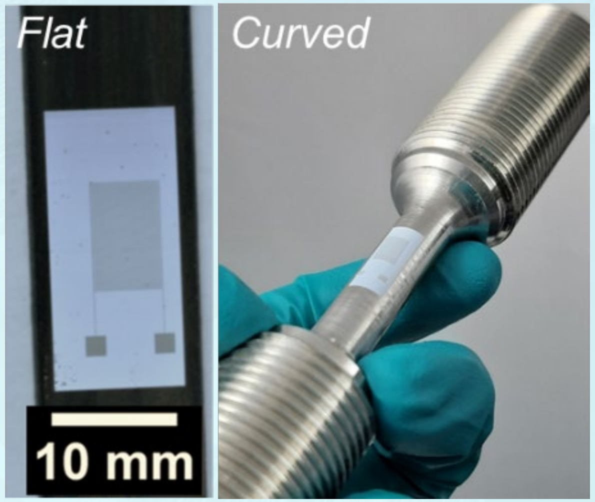


Summary of Accomplishments

Milestones

- Validation of printed strain gauges at moderate temperatures (up to 300 °C) (Complete)
- Printed strain gauges for high temperature applications (>300 °C) (Submitted)





FY23 Accomplishments

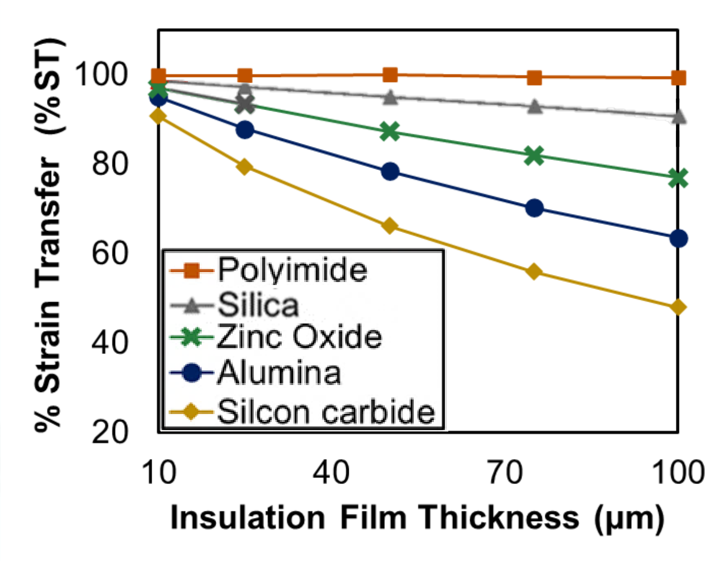
- Initial formulation of the high temperature ceramic insulation/encapsulation ink was successfully printed at using aerosol jet printing on both flat and conformal substrates
- The effective dielectric constant of the printed barium strontium titanate film was measured in thermal transients and dwells up to 500 °C
- A capacitive strain gauge was printed on barium strontium titanate and mechanically tested on a uniaxial test frame up to 0.11% strain
- Laser-induced spallation and ablation was used to qualify the adhesion of barium strontium titanate printed films
- Presented work at The Minerals, Metals & Materials Society (TMS) 2023 and Nuclear Plant Instrumentation, Control, and Human Interface Technologies (NPIC-HMIT) 2023 conferences

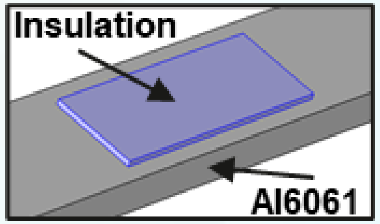
Results and Accomplishments (1/5):
Modeling to guide materials selection

For the interdigitated electrode (IDE) capacitive strain gauge (CSG), two mechanisms that can result in the variation in the capacitance with mechanical strain are:

1. Changes in electric field near the electrodes due to geometric changes with stress, $\Delta E/E$
2. Changes in dielectric properties of the insulation/encapsulation material with stress, $\Delta\varepsilon/\varepsilon$

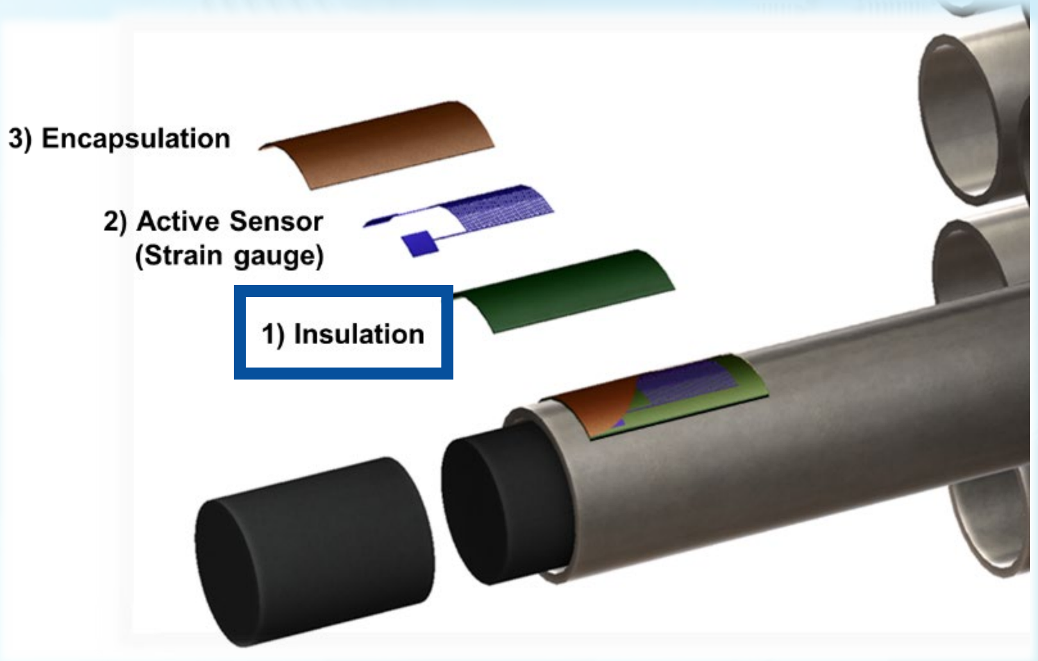
Finite Element Analysis (FEA)





$$\varepsilon = strain$$

$$\%\mathrm{ST} = \frac{\varepsilon_{insulation}}{\varepsilon_{Al6061}} \times 100$$

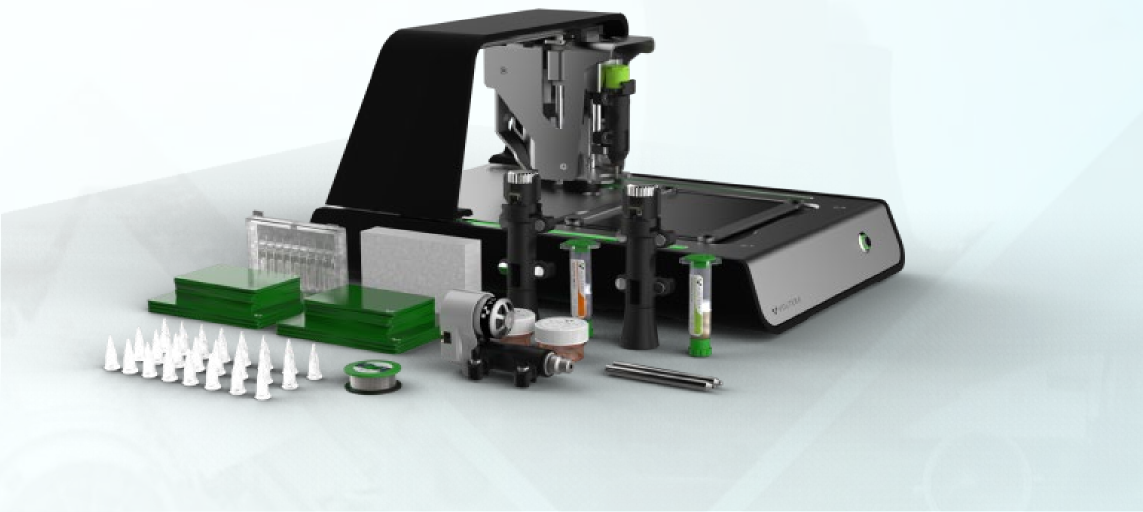


Multi-layered system of the printed CSG

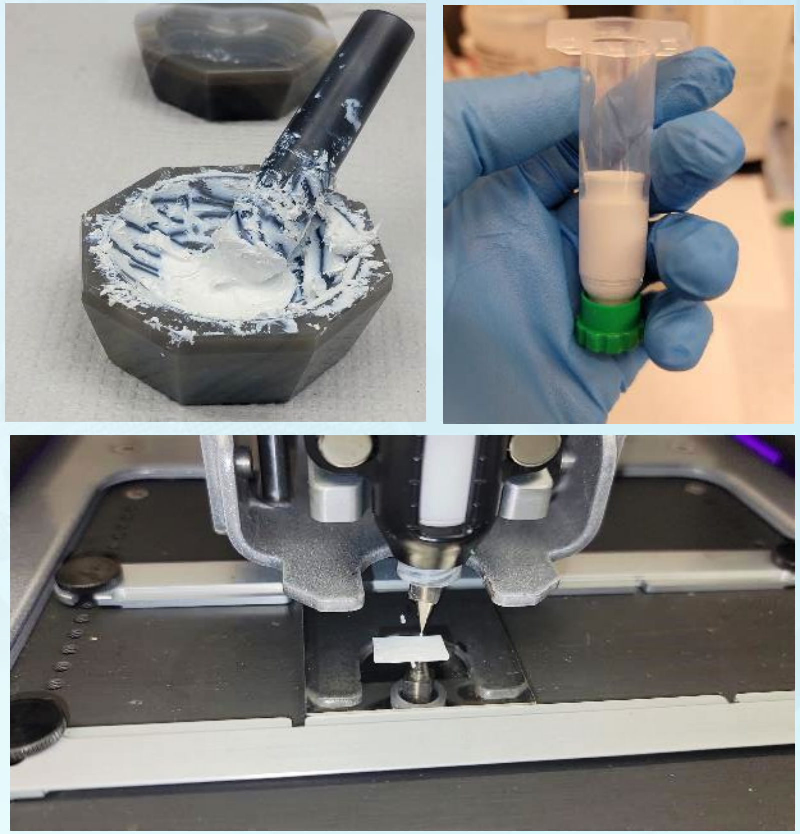
- Finite element analysis was used to investigate the efficacy of potential ceramic insulators
- The inherent stiffness and reduction in %STR of ceramic materials requires a material with dielectrostriction effects (i.e., change in dielectric constant with an induced stress)

Results and Accomplishments (1/5):
Barium strontium titanate ink formulation

- Barium strontium titanate was observed to have a changing dielectric constant with an induced mechanical stress
- Printable BST ink was formulated to be compatible with current direct-write additive technologies available at INL facilities



Printed Sensor Fabrication using Voltera V-One (Extrusion PCB Printer)

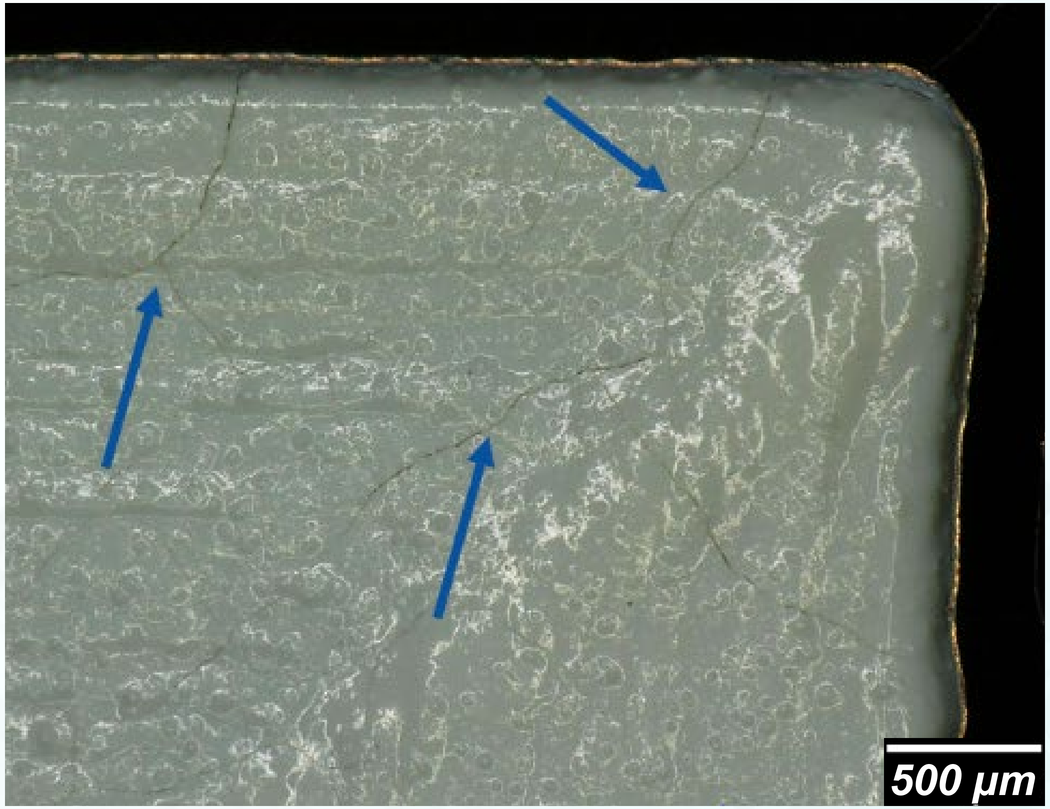


Formulation of printable BST Ink

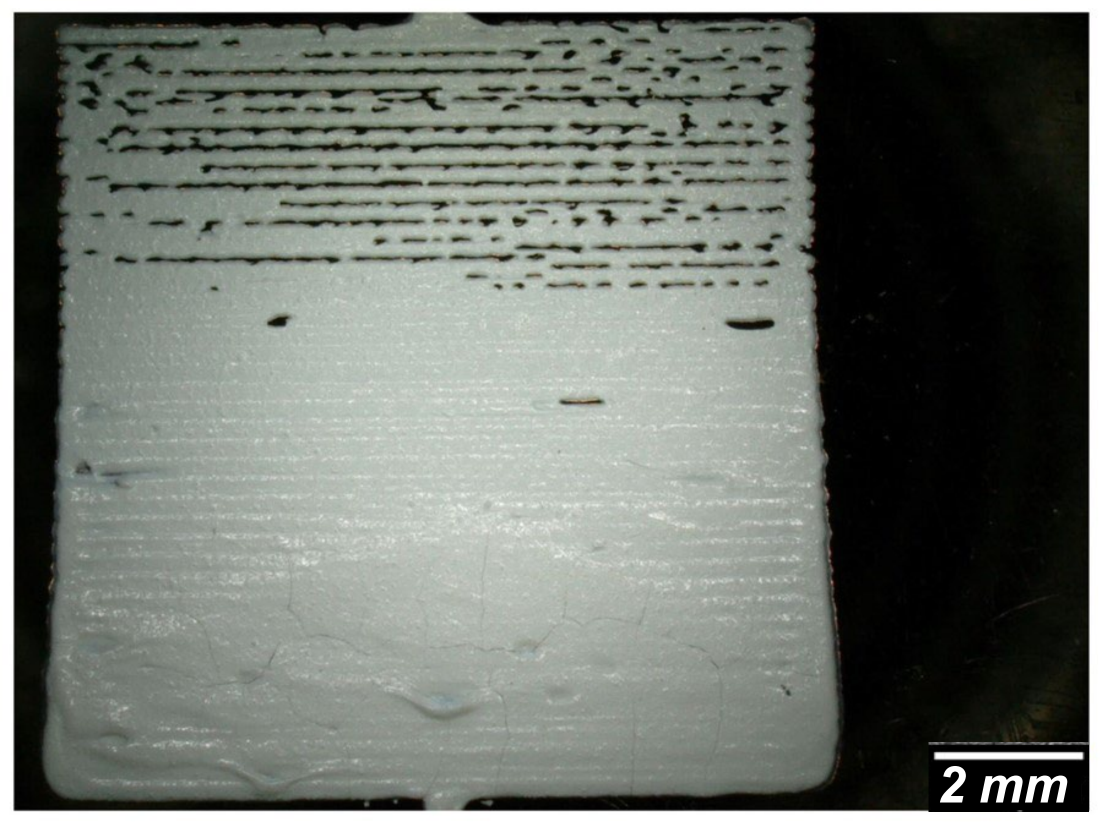
[1] https://www.voltera.io/store/v-one
[2] T. Shaw, Z. Suo, M. Huang, E. Liniger, R. Laibowitz, J. Baniecki, The effect of stress on the dielectric properties of barium strontium titanate thin films, Appl Phys Lett, 75(1999) 2129-31.

Results and Accomplishments (1/5):
Challenges with BST printed with Voltera V-One

- The films printed with the Voltera were relatively thick (i.e., > 100 µm), which caused the film to have excess residual stresses upon cooling on SS316 and crack formation
- The deposition of the BST paste with the Voltera was also inconsistent causing challenges for reliable CSG fabrication



Optically visible Cracking of film after sinter the printed BST film; caused by coefficient of thermal mismatch between print and substrate



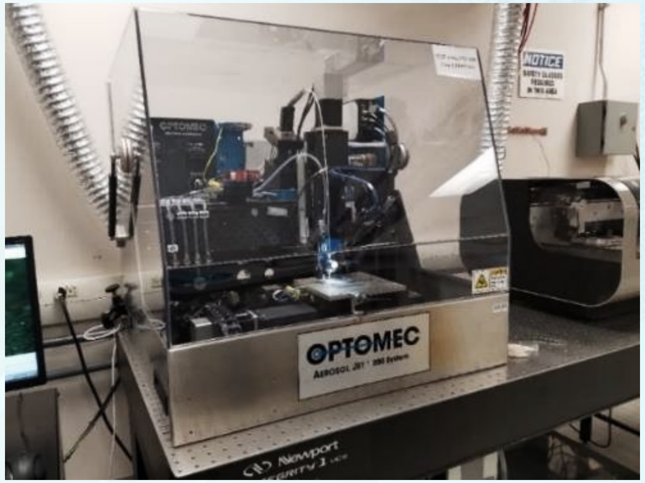
Inconsistent and nonuniform deposition of BST with the Voltera made it a challenge to reliably fabricate CSGs

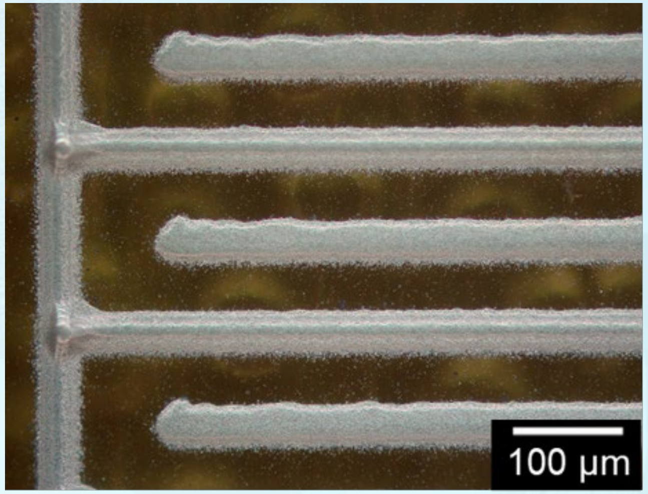
Results and Accomplishments (2/5):
Aerosol Jet Printing at Boise State

- Starting in mid-FY23, the BST ink was reformulated and printed using the aerosol jet printer at Boise State University
- Simplifies the fabrication process from a multi-modal procedure to a singular technology

<u>Advantages to Aerosol Jet Printing</u>

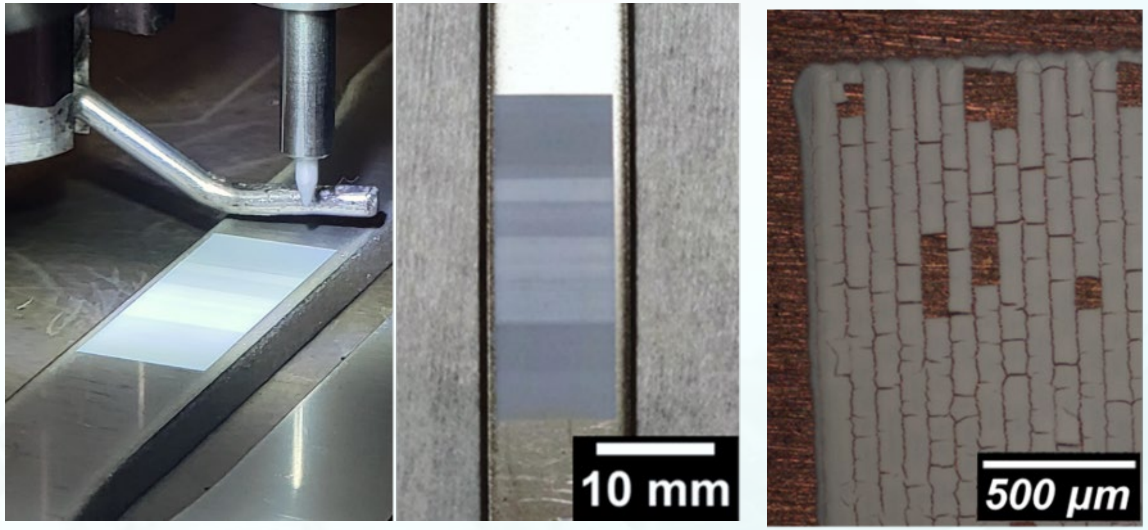
1) Reduce Invasiveness
   - Fine, complex features down to 10μm resolution
2) Direct fabrication on cladding
   - Conformal deposition on 3-D geometries
3) Nuclear relevant materials
   - Compatibility with variety of materials



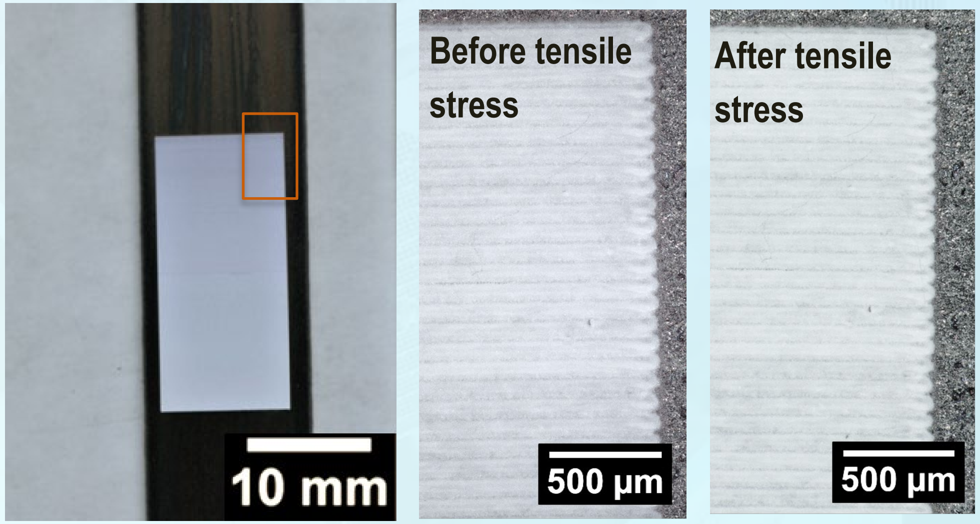


Results and Accomplishments (2/5):
Preliminary successes to BST printing on AJP

- Successfully demonstrated ability to deposit BST using the aerosol jet printing AM technology in June 2023
- The film was thinner (i.e., < 20 μm) than the Voltera films, however still had non-uniform deposition and film cracking
- Addition of immiscible co-solvents and improved AJP process parameters (i.e., continuous stirring of ink and saturated gas flow) helped improved uniformity of film and reduced optically visible cracking.



Nonuniform deposition of BST with thickness variation between 3-20 μm; cracking of the BST film



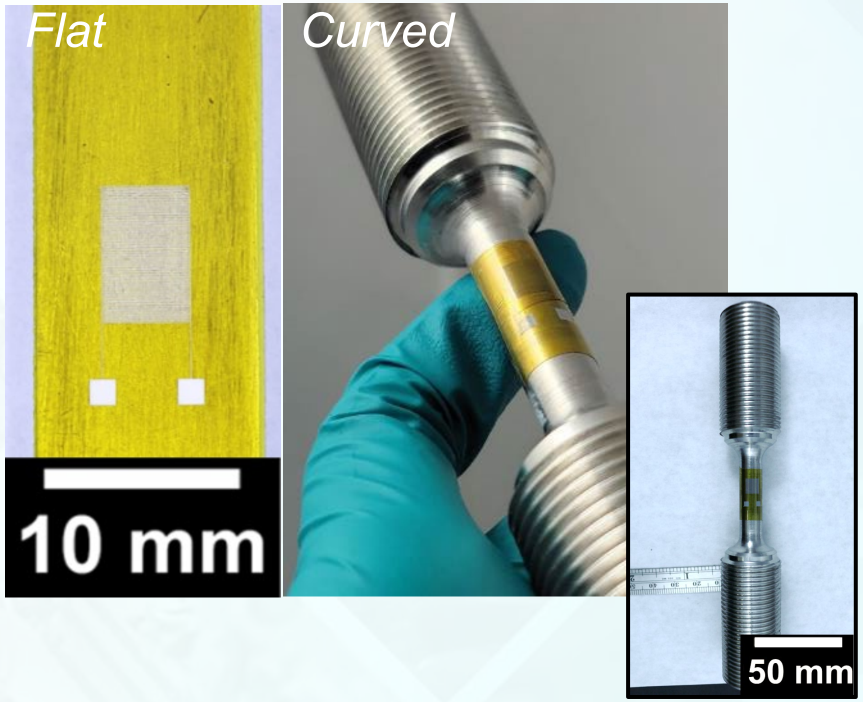
Improved AJP process controls enabled uniform film thickness of 7 μm across the whole printed film; BST film before and after mechanically strained in tension up to 0.11%

[1] M. Schneider, J. Maurath, S.B. Fischer, M. Weiß, N. Willenbacher, E. Koos, Suppressing crack formation in particulate systems by utilizing capillary forces, Acs Appl Mater Inter, 9(2017) 11095-105.

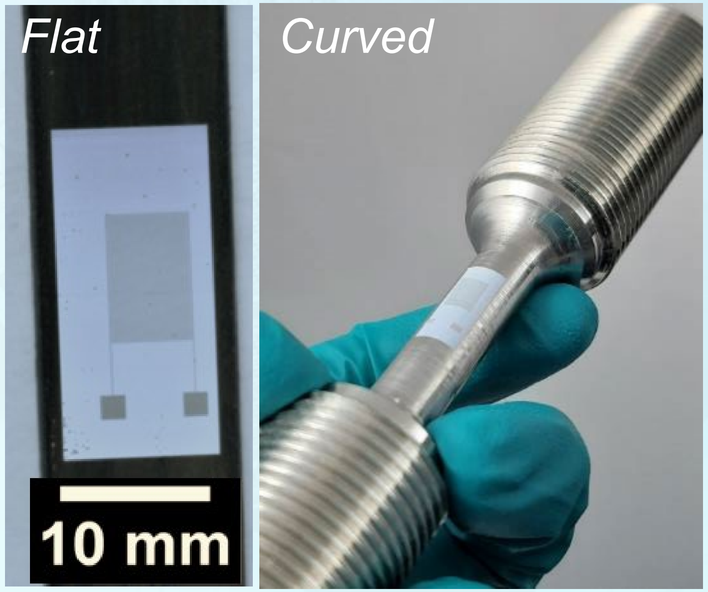
Results and Accomplishments (2/5):
Conformal printing with aerosol jetprinting

- The multi-axis AJP allows for fabrication on both planar and conformal substrates (i.e., 9.3 mm diameter tensile specimen)
- Demonstrated fabrication of CSG on polyimide system application up to 300 °C and BST for applications above 300 °C

Polyimide Insulation & Encapsulation



BST Insulation

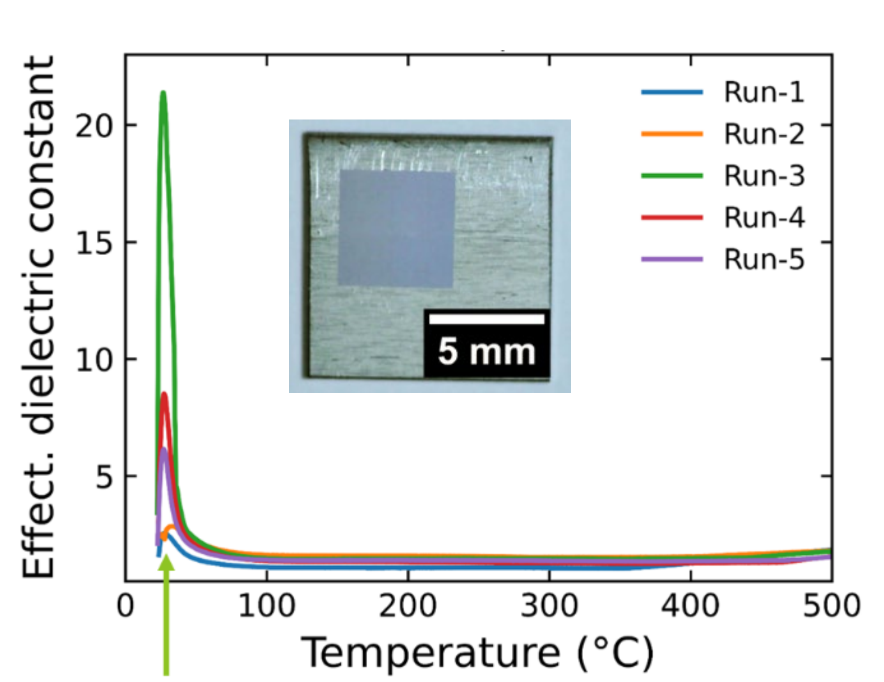


Results and Accomplishments (3/5)

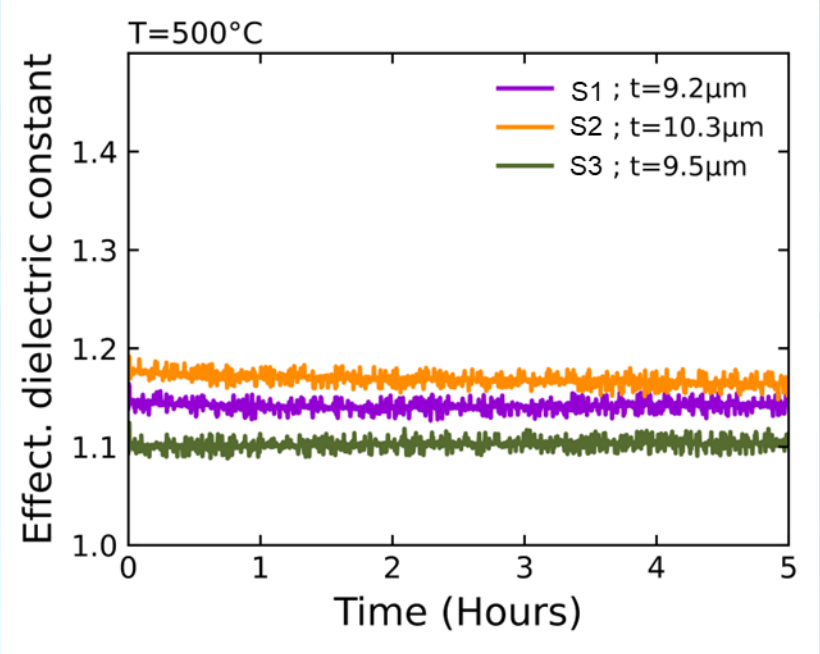
Dielectric constant measurement of BST film

- An effective dielectric constant (i.e., dielectric constant of both SS316 and BST) was measured in a dielectric tester that can capture in-situ dielectric constant measurements up to 800 °C
- Curie temperature (T_c) is 24 °C causes a polymorphic transformation from a tetragonal (ferroelectric) to cubic (paraelectric) unit cell material.

Dielectric tester with furnace



Effective dielectric constant of barium strontium titanate on SS316L as temperature is ramped up to 500°C for five thermal cycles



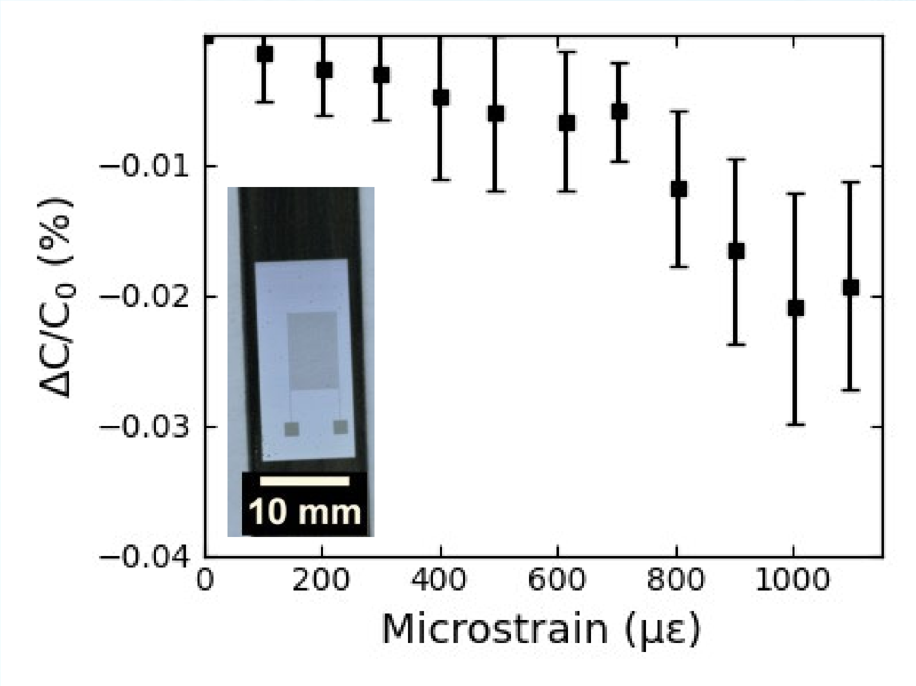
Effective dielectric constant of barium strontium titanate on SS316L during a 5-hour dwell at 500°C

Results and Accomplishments (4/5)

Testing CSG fabricated on BST insulation

- In-situ strain sensitivity measurements of AJP printed CSGs with BST insulation tested at room temperature
- Initial capacitance of the CSG with BST insulation was 445 pF; CSGs with polyimide insulation/encapsulation is ≈10 pF
- Gauge factor of the CSG on BST 0.2 which is lower than the gauge factor of the IDE CSGs

CSG is mechanically loaded in tension in an Instron uniaxial test-frame with an external extensometer

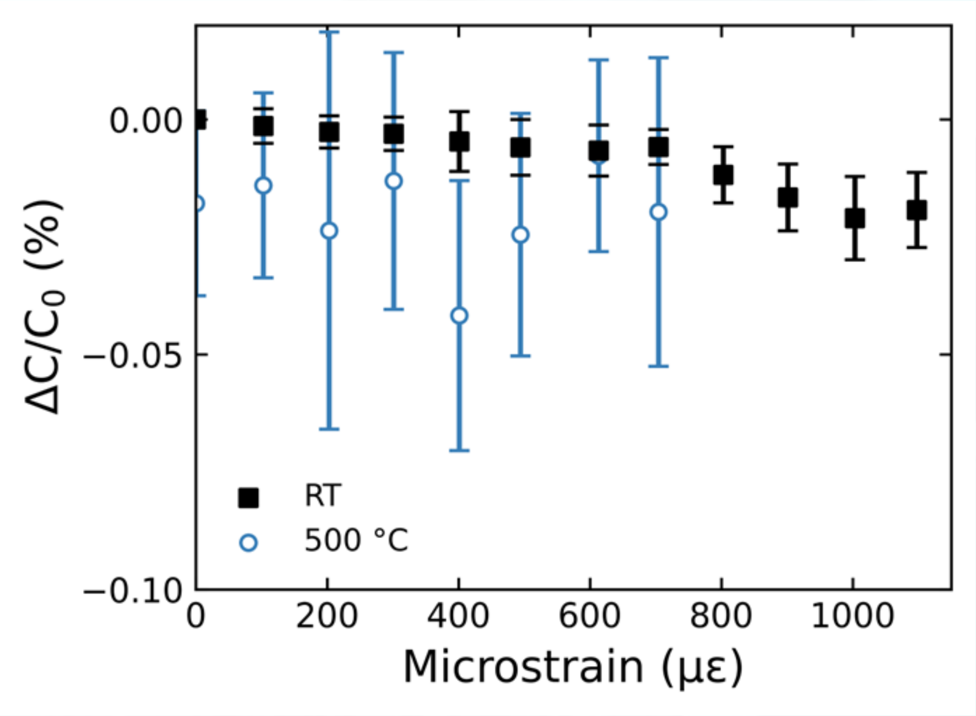


Mechanical strain results from printed strain gauge on BST insulation

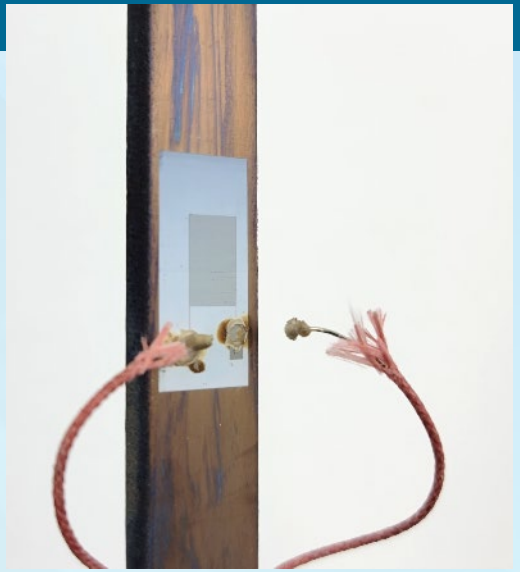
Results and Accomplishments (4/5)

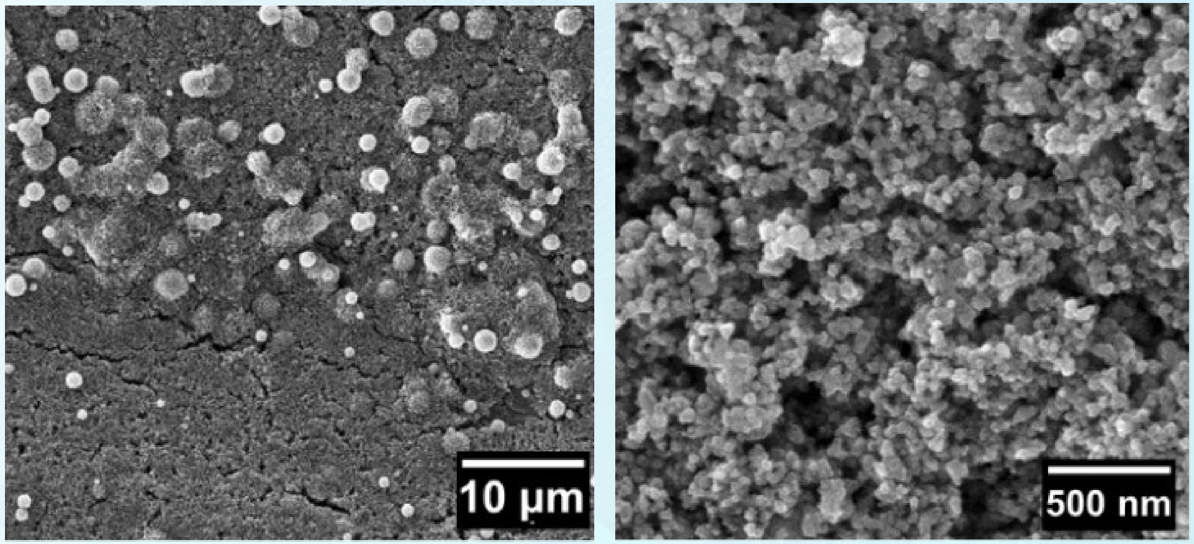
Testing CSG fabricated on BST insulation

- Silver epoxy is used to bond individual lead wires to the printed IDE CSG since it is easiest to implement and the most common technique to integrate to AM sensors
- Porosity and the micron-scale cracking in the BST film creates challenges to reliably measure strain and maintain continuity at 500 °C



Mechanical strain results from printed strain gauge



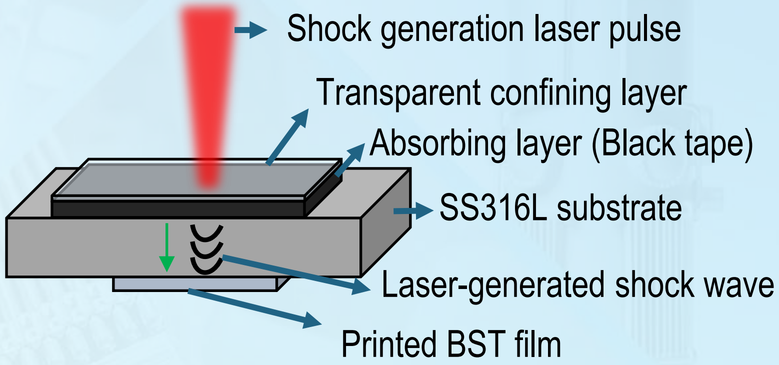


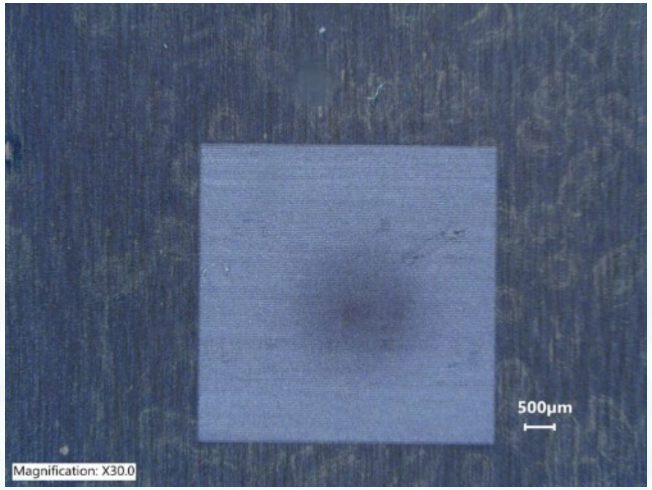
Lead-wire detaching from CSG during test and SEM micrograph of BST film

Results and Accomplishments (5/5)

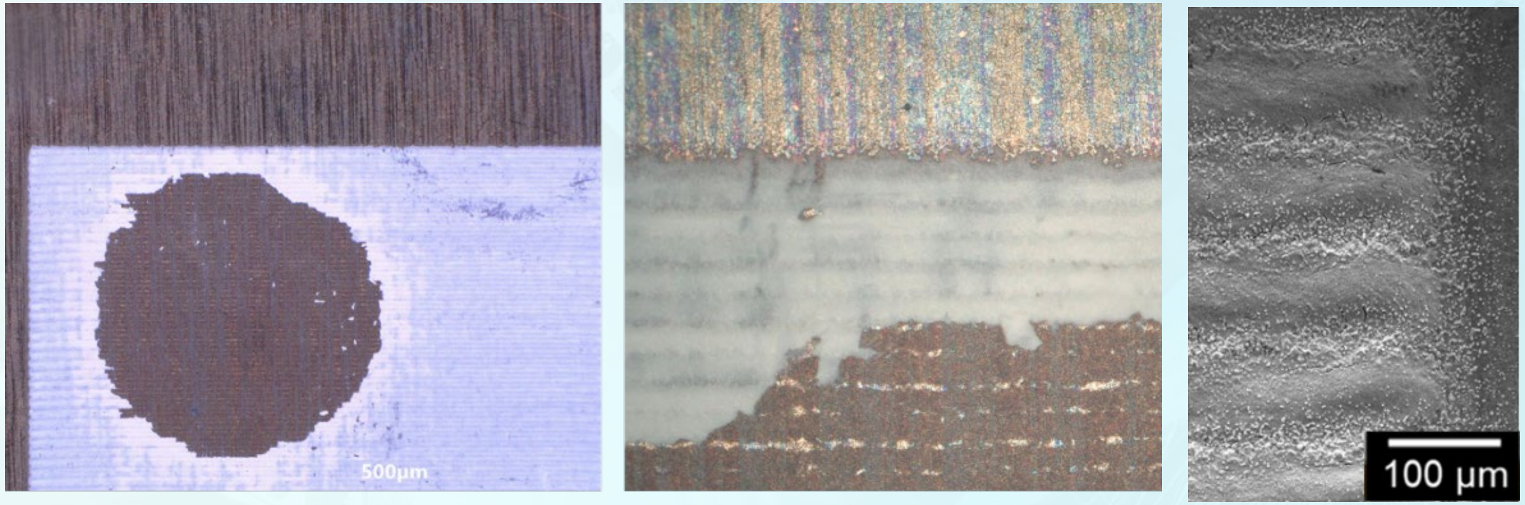
Adhesion testing of BST

- Laser spallation tests were performed at only the lowest laser pulse energy of 0.14 – 0.15 J for the BST insulation/SS316L substrate interface
- Thinner BST prints (thickness < 8 μm) showed an almost complete cohesive failure
- Thicker prints (thickness > 8 μm), failure occurred at the film/substrate in the overspray regions at the periphery of each print pass and remained adhered at the center of each print pass
- A reduction the overspray region and keep the thickness to less than 8 μm can result in consistent and robust printed insulation layers for IDE CSGs





Spalled region of a BST printed film with an average thickness of 5.2 μm



Spalled region of a BST printed film with an average thickness of 10.3 μm

Concluding Remarks

- FY23 accomplishments
  - Initial formulation of the high temperature ceramic insulation/encapsulation ink was successfully using aerosol jet printing on both flat and conformal substrates
  - The effective dielectric constant of the printed barium strontium titanate film was measured in thermal transients and dwells up to 500 °C
  - A capacitive strain gauge was printed on barium strontium titanate and mechanically tested on a uniaxial test frame up to 0.11% strain
  - Laser-induced spallation was used to qualify the adhesion of barium strontium titanate printed films
  - Presented work at The Minerals, Metals & Materials Society (TMS) 2023 and Nuclear Plant Instrumentation, Control, and Human Interface Technologies (NPIC-HMIT) 2023 conferences

- Upcoming FY24 work:
  - Continue increasing the robustness of the printed strain gauge to allow for applications up to 750 °C
  - Continue developing surface acoustic wave sensors for interrogating the quality of printed sensors
  - Test strain gauge in relevant harsh environments (i.e., irradiation)

Questions?

Contact Michael McMurtrey at: michael.mcmurtrey@inl.gov

Thank You