



Advanced Sensors and Instrumentation

Printed Sensors for Harsh Environments:

- Ink Development
- Strain Gauges









Advanced Sensors and Instrumentation (ASI)

April 13, 2023

Associate Professor: David Estrada, PhD Assistant Professor: Brian Jaques, PhD Boise State University / Idaho National Laboratory





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- Digital Image Correlation



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Project Overview

The overall objective of this project is to leverage advanced manufacturing methods to develop novel sensors and instrumentation for in-pile monitoring and in-situ analysis of fuels and materials.



Project Overview – Additive Electronics Tool Sets





Precursor Gas With Particles

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N. McKibben, et al, Microsyst Nanoeng., in press 2023 C. Hollar, et al., Adv. Mat. Tech, 2020 T. Pandhi, et al, Sci Reps., 2018



Drop on demand

- 30 µm drop size (DMP 2850)
- Contact angle: 20 30°
- Viscosity: 2 12 cP
- Particle Size <50 nm
- Organic co-solvents, water based inks

Focused Aerosol

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- 10 200 µm feature size (Optomec AJ 200)
- Viscosity: 1 1000 cP (pneumatic atomizer)
 - < 10 cP (ultrasonic atomizer)
- Particle Size <300 nm
 - Organic co-solvents, water based inks

Focused Aerosol through CAP

- >100 µm feature size (Space Foundry)
- Viscosity: 1 5 cP (ultrasonic atomizer)
- Particle Size TBD, from 10 nm to 1 μm
- Water based inks
- Compatible with microgravity

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Project Overview – Ink Development







N. McKibben, et al, NPG Microsystems & Nanoengineering., in press 2023

Technology Impact

- Additive Manufacturing is expected to reach \$26.68B by 2027 with a ~14% growth (Reports and Data)
- This project impacts the Digital Ink Market valued at \$2.6B in 2018 which is expected to reach \$4.3B by 2026 with CAGR of 6.1% from 2019 – 2026
 - UV ink, Solvent ink, and water-based inks constitute over 60% of the market
 - Nuclear industry requires specialty inks for harsh environments
- Only handful of key players such as:
 - UTDots
 - NovaCentrix
 - Applied Nanotech
- Success in nuclear can translate to military, aerospace, petrochemical, medical, and corrosive environments





Materials and Technology for a Flexible World

Nickel Nanoparticle Ink



7

Nickel Nanoparticle Ink

AJP of Magnetic Patch Transducer on Steel



Flexibility of Design





40 µm



Surface Acoustic Wave Devices



Electrical Devices/Thermal Sintering



- Sintering studies have been performed under an argon environment
- 450°C is currently the best sintering condition for electrical applications
- Microstructural cracking occurs during sintering

Our current hypothesis: propagation of this cracking is responsible for the decrease in electrical performance beyond 450°C. This property may be thickness dependent and is currently being investigated

Reactive Piezoelectric Lithium Niobate Ink



Results and Accomplishments



- Scalable synthesis of the inks \succ
- Nanoparticles size ranging from 6nm to 60nm
- Variable viscosity 1-10cP \geq
- Low temperature sintering <300°C \succ
 - Photonic sintering on low temperature substrates
 - 4X Resistivity of the bulk after thermal and photonic sintering







Plasma Jet







350°C - 30 minutes

Printed sample



Concluding Remarks



- > ASI has been instrumental in advancing nanomaterial based ink technologies aligned to national interests.
- Harsh environment testing of printed materials and sensors with in-situ readout is the next frontier
- Advanced manufacturing + advanced materials = new capabilities in fundamental and applied sciences



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Thank You

David Estrada, PhD

Associate Director | Center for Advanced Energy Studies Advanced Manufacturing Deputy Director of Academic Research | Idaho National Laboratory Site Director | NSF IUCRC Center for Atomically Thin Multifunctional Coatings (ATOMIC) Site Director | NSF REU Site Advanced Manufacturing for a Sustainable Energy Future Associate Professor | Micron School of Materials Science and Engineering +01.208.426.2950 daveestrada@boisestate.edu







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- Digital Image Correlation



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Project Overview

<u>Goal</u>: To expand the capabilities for monitoring mechanical properties and the structural health of material specimens in test reactor experiments with strain sensing technologies.

Research Scope/Objectives

- Develop and test both printed strain gauges and printed digital image correlation patterns using advanced manufacturing techniques and compare them against conventional and/or commercial techniques
- Develop quality control methods for the printed sensors

Project Schedule for FY23

- Continue increasing the robustness of the printed strain gauge to allow for applications up to 650 °C
- Continue developing laser-based methods for interrogating the quality of printed sensors
- Continue developing heterogeneous integration strategies for printed sensors
- Continue to correlate the laser-induced spallation technique to quantitative print adhesion properties



Strain gauge fabricated using aerosol jet printing

Printed Digital Image Correlation Patterns







Technology Impact

Technology application

- Ubiquitous requirement: Harsh environment strain sensing is needed in many industries as it provides crucial, realtime 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 sensors are not replacing traditional high-temperature strain gauges, but 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 out- and in-core experiments

Support the DOE-NE research mission

 These efforts provide additional support to programs such as the Advanced Reactors Technologies, Microreactor, and Advanced Materials and Manufacturing Technologies programs through its implementation of advanced manufacturing techniques in sensor fabrication and deployment. Printed Strain Gauge

Overview of Accomplishments

1) Printed Digital Image Correlation

- Small scale periodic patterns were printed using aerosol jet printing for digital image correlation.
- Digital image correlation was used to measure strain (up to 1100 με) during cyclic tests at 23-600 °C.
- 2) Printed Capacitive Strain Gauge
- Aerosol jet printing was optimized to allow for the fabrication of repeatable and predictable performance capacitive strain gauges on polyimide insulation/encapsulation up to 300 °C
- High temperature ceramic insulation/encapsulation was formulated and successfully printed using INL capabilities
- 3) Commercial Weldable Strain Gauge
- A uniaxial test frame was set-up and used to test a welded a strain gauge on a SS316 tensile specimen up to 300 °C
- A strain gauge was welded onto a SS304 prototypical microreactor core block to support the test of embedded thermocouples and strain sensing optical fibers in the Single Primary Heat Extraction and Removal Emulator (SPHERE)
- 4) Additive Printing Qualification Testing
- Laser-induced spallation and a standardized pull-off test was compared and used to quantify the adhesion of printed films







Shock generation laser pulse
 Transparent confining layer
 Absorbing layer (Black tape)
 Aluminum 6061 substrate

Printed film



Laser-generated shock wave

Other Accomplishments

General accomplishments

- Timothy Phero (Strain gauges):
 - CAES Student of the year, 2022
 - ANS Graduate Scholarship, 2022
 - MSE Graduate Service award at BSU, 2021
 - INL Graduate Fellowship, 2020

- Kaelee Novich (Digital image correlation):
 - NNSA Fellowship, 2023
 - ANS Graduate Scholarship, 2022
 - Internship at INL under UNLP program, 2022-2023
 - ANS Michael J. Linebeberry Scholarship, 2021
 - DOE UNLP (formerly NEUP) Fellowship, 2020

- **Publications**
- 1. "Additively manufactured patterns on structural nuclear materials for high temperature applications using digital image correlation." Novich, K.A., Phero, T.L., Cole, S.E., McMurtrey, M.D., Estrada, D., Jaques, B.J. In Draft. 2023
- 2. "Additively Manufactured Strain Sensing for Nuclear Reactor Applications." T.L. Phero and K.A. Novich, K.T. Fujimoto, A.R. Khanolkar, B.C. Johnson, M.D. McMurtrey, D, Estrada, B.J. Jaques. Nuclear Plant Instrumentation, Control, and Human Interface Technologies (NPIC-HMIT) 2023 conference proceedings. 2023.
- 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[†]. Experimental Mechanics. Submitted. October 2022.
- 4. "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
- 5. "A wireless, multi-channel printed capacitive strain gauge system for structural health monitoring." K.L. Ranganatha, K. Novich, T. Phero, B.J. Jaques, D. Litteken, D. Estrada, B.C. Johnson[†]. IEEE Sensors. pp. 1-4. 2021. DOI: 10.1109/SENSORS47087.2021.9639749.
- 6. "Aerosol jet printed capacitive strain gauges for soft structural materials." K. Fujimoto, J. Watkins, T. Phero, D. Litteken, K. Tsai, T. Bingham, K.L. Ranganatha, B.C. Johnson, Z. Deng, B.J. Jaques, D. Estrada[†]. Nature Partner Journal: Flexible Electronics. Vol. 4, Issue 32, 2020. DOI: 10.1038/s41528-020-00095-4

Presentations

- 1. "Additively Manufactured Strain Sensing for Nuclear Reactor Applications." T.L. Phero and K.A. Novich, K.T. Fujimoto, A.R. Khanolkar, B.C. Johnson, M.D. McMurtrey, D, Estrada, B.J. Jaques. Accepted for presentation at the Nuclear Plant Instrumentation, Control, and Human Interface Technologies (NPIC-HMIT) 2023 conference. Knoxville, TN. July 15-21, 2023.
- 2. "Additively Manufactured Digital Image Correlation for Nuclear Materials." K. Novich, T. Phero, S. Cole, M. McMurtrey, D. Estrada, B.J. Jaques. Presented at TMS 2023. March 19-23, 2023.
- 3. "Additive manufactured strain sensors for nuclear applications." T.L. Phero, K.A. Novich, B.C. Johnson, M.D. McMurtrey, D. Estrada, B.J. Jaques. Presented at TMS 2023. March 19-23, 2023.
- 4. "Additive Manufactured Strain Gauges for Structural Health Monitoring." T.L. Phero, K.A. Novich, B.C. Johnson, M.D. McMurtrey, D. Estrada, B.J. Jaques. Presented at the University of Idaho American Nuclear Society student section virtual symposium. October 21, 2022
- 5. "Development of Additively Manufactured Strain Gauges." Phero, T., Novich, K., Johnson, B., McMurtrey, M., Estrada, D., Jaques, B., CAES Ribbon-Cutting event. Poster Session, Nov. 2022.
- 6. "Additively Manufactured Strain Gauges for Structural Health Monitoring." Phero, T. L., ID-UT Symposium: Nuclear in the Mountain West, Virtual Conference; Oct. 2022.
- 7. "A Wireless, Multi-Channel Printed Capacitive Strain Gauge System for Structural Health Monitoring." K. L. Ranganatha, K. Novich, T. Phero, K. Fujimoto, D. Litteken, D. Estrada, B.J. Jaques, B. Johnson. Presented at the IEEE Sensors 2021 virtual conference. October 31-November 4, 2021.
- 8. "Additive manufactured in-pile strain sensors." T.L. Phero, K.A. Novich, B. Gougar, S. Cutler, K. Fujimoto, R. Skifton, D. Estrada, B.J. Jaques. Presented at the Materials Science and Technology 2020 Conference. Virtual Event. November 2-6, 2020.

Results and Accomplishments (1/4) Printed Digital Image Correlation: Pattern Analysis

- Aerosol jet printer was used to print two types of DIC patterns on SS316L and Al6061 with a variety of pitch sizes
- Patterns were verified at room temperature with an extensometer and resistive strain gauges



Results and Accomplishments (1/4) Printed Digital Image Correlation: Mechanical test up to 600 °C

 SS316L samples with the printed 150 µm pitch pattern were ramped from 23 – 600 °C and compared to the data collected for the spray-painted pattern and extensometer



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2



- At 100 °C, 200 °C, 300 °C, and 600 °C, SS316L samples with printed and spray-painted patterns were tensile tested
- The printed DIC patterns performed well when compared to data from the extensometer – 150 µm pitch dot pattern correlated to highest accuracy

Results and Accomplishments (2/4) Printed Strain Gauge: Elevated temperature mechanical Test

- Polyimide insulation/encapsulation was selected as it is commercially available, stable up to 400 °C, and sufficiently elastic
- Printed capacitive strain gauges (CSG) were printed and demonstrated on SS316 up to 300 °C
- A printable ceramic material was formulated and currently being developed for higher temperature printed strain gauged



Formulation and printing of ceramic film

[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.



Printed Strain Gauge

Results and Accomplishments (2/4) Printed Strain Gauge: Compressive and thermal cycling test

- The CSG's response to compression was measured for 10 cycles from $-1,100 1,100 \mu\epsilon$
- The performance of the CSG was measured after a multiple thermal cycles (22 300 °C and then cooled down to 22 °C for mechanical testing)



^[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.

Results and Accomplishments (3/4): Commercial Weldable strain gauge

- High temperature weldable strain gauges were tested on SS316 tensile specimens up to 300 °C for multiple cyclic tensile cycles
- Strain gauge output matched the output of the calibrated high temperature extensometer





Thermocouples

Strain gauge loaded in axial mechanical test fixture

Results and Accomplishments (4/4) Adhesion testing of AM printed structure

- Standardized pull-off adhesion measurements were used to develop a laserinduced spallation technique to quantify the adhesion strength of printed structures cured at different conditions
- Pull off adhesion requires large print and extensive setup for testing
- Laser spallation is non-contact, has a quicker preparation time, and requires smaller "sacrificial" samples since it's spatially localized to less than 25 mm²



Example of pull-off adhesion samples that failed at the substrate/film interface for each sintering condition



Shock generation laser pulse

Transparent confining layer

Absorbing layer (Black tape)

Aluminum 6061 substrate

Laser-generated shock wave

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

Laser-induced Spallation

Multi-program Collaboration and Interest

dvanced Sensors



Fabricating Printed Strain Sensors

Objective: Fabricate strain gauges that can fit within a small footprint (i.e., 1x10mm) and withstand elevated temperatures (above 300°C)

Printed Capacitive Strain Gauge



Printed Digital Image Correlation Pattern

250 µm	150 µm	<u>Pitch Size:</u> 100 µm	50 µm
200 µm	2 <u>00 µ</u> m	<u>200 µ</u> m	200 µm
200 µm	200 µm	2 <u>00 µ</u> m	200 µm

Strain Gauge Mechanical Testing

Objective: To test the response of the resistive strain gauge to mechanical strain and validate it against additively printed strain gauges at moderate temperatures (i.e., up to 300 °C)

Uniaxial Mechanical Testing





Deployment of Commercial Strain Gauges

Objective: Use strain gauges as baseline measurements for other novel distributed sensors (i.e., fiber optics)

Prototypic Hexagonal Core Block





Deployment of Printed DIC Patterns

Objective: Use digital image correlation to measure strain during the thermal cycling (500 °C to 700 °C) of miniature surveillance specimens.



Concluding Remarks

Several methods of strain monitoring were developed and tested at elevated temperatures

- The "best" solution is dependent on the application, but AM sensors provide a versatile (geometry, materials, non-invasive, etc.) solution with small form factors
- The quality of an AM print can be quantified by adhesion properties, and a laser-induced spallation technique has been developed
- This work was/will be disseminated through:
 - 5 journal/conference publications
 - 2 manuscripts that are in draft
 - 7 conference presentations
 - 1 upcoming conference presentation
 - Timothy Phero's PhD Dissertation (TBD)
 - Kaelee Novich's PhD Dissertation

• Future work:

- Continue increasing the robustness of the printed strain gauge to allow for applications up to 650 °C
- Continue developing laser-based methods for interrogating the quality of printed sensors
- Continue developing heterogeneous integration strategies for printed sensors
- Continue to correlate the laser-induced spallation technique to quantitative print adhesion properties



Timothy Phero

Graduate Research Assistant at BSU and INL Graduate Fellow

Kaelee Novich Graduate Research Assistant at BSU and INL Intern



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Advanced Sensors and Instrumentation

Thank You

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energy.gov/ne

Past Accomplishments



Optimized AJP Process Parameters

Print drift analysis



100 µm

100 µm

Mechanical Testing of CSG



2 mm

Overview of Technology

Capacitive Strain Gauge (CSG)

Extrusion Printing

Aerosol Jet Printing

10 mm

- Interdigitated CSGs have a low profile and allow for high in-plane strain sensitivity
- Interdigitated CSGs were fabricated using optimized aerosol jet printing parameters and then mechanically tested up to 300°C

500 µm

Digital Image Correlation (DIC)



DIC Additional Slides: Experimental Methods - Ncorr

- Ncorr, an open-source DIC software, was used to process images taken during tests
- The software relies on subset based matching to compare deformed images to the reference image

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Set Seeds

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Seed(s) Set: 4 of 4

Set Seeds

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DIC Additional Slides: Experimental Methods – Testing Parameters

Property	Value (Units)
Resolution Width	6960 pixels
Resolution Height	4640 pixels
Aperture Size	f/2.8
Exposure Time	1/125 sec
ISO	2500

- At room temperature, specimens were pulled to 1100
 microstrain
- With higher temperatures, the tensile load was decreased to keep specimens within the elastic region [#]



[#] Materials Properties Database, JAHM Software, v8.97.



[#] https://www.amazon.com/Canon-Digital-Camera-Black-3616C002/dp/B07WFQYDD5[#] https://www.canoncamerageek.com/best-lenses-for-a-canon-90d.html

EFSU

DIC Additional Slides: Al6061 Results – room temperature

• The same testing procedure was performed on Al6061 to compare results on materials with different mechanical properties



- The strain tabulated from the DIC patterns followed the cyclic tensile test profile
- All strain measurement methods produce similar results, except for the strain calculated from images of the printed 50 μm pitch dot pattern (400 με difference compared to extensometer data at maximum load)

Strain Gauge
Extensometer
Dot Printed Pattern
Line Printed Pattern
Spray-Painted Pattern

Results and Accomplishments Printed Digital Image Correlation

- Statistical analysis was performed for all experiments at room temperature to better understand accuracy between DIC calculated strain and strain measured by extensometer
- There no statistical significance between the extensometer data and the strain computed by Ncorr
- Patterns that were majority black or white on the pixel intensity spectrum had higher percent errors

Material Type	Printed Pattern Type	Pitch Spacing (μm)	Percent Error (%)	P-value
SS316L	Dots	50	32	0.37
		100	9	0.78
		150	27	0.44
		250	29	0.58
	Lines	50	27	0.42
		100	20	0.57
		150	11	0.61
		250	23	0.53
	Spray-Painted Pattern		20	0.48
Al6061	Dots	50	43	0.26
		100	2	0.99
		150	7	0.77
		250	13	0.73
	Lines	50	9	0.63
		100	5	0.95
		150	4	0.81
		250	8	0.81
	Spray-Painted Pattern		4	0.87

Results and Accomplishments Printed Digital Image Correlation

- DIC pattern analysis was used to predict computational feasibility
 - Grayscale intensity analysis and mean intensity gradient (MIG)
- ImageJ was used to process images for grayscale intensity analysis
- Matlab was used to calculate MIG
- Resolution of images: 1548 x 810 pixels
- A higher MIG = lower mean bias and standard deviation errors
- MIG above 20 was desired

Printed Pattern Type	Pitch Sizes (µm)	Mean Intensity Gradient
	50	87
Dete	100	47
Dots	150	46
	250	24
Lines	50	98
	100	75
	150	71
	250	51
Spray-Painted Pattern		42

Grayscale Intensity Analysis



[#] Park, J., et al. "Assessment of speckle-pattern quality in digital image correlation based on gray intensity and speckle morphology," Optics and Lasers in Engineering, 2017. 91: 62-72.
[#] Wang, H., et al., Fabrication of micro-scale speckle pattern and its applications for deformation measurement. Measurement Science and Technology, 2012. 23(3): p. 035402.
[#] Pan, B., Z. Lu, and H. Xie, "Mean intensity gradient: An effective global parameter for quality assessment of the speckle patterns used in digital image correlation." Optics and Lasers in Engineering, 2010. 48(4): p. 469-477.

[#] Schneider, C.A., W.S. Rasband, and K.W. Eliceiri, NIH Image to ImageJ: 25 years of image analysis. Nature Methods, 2012. 9(7): p. 671-675.

Results and Accomplishments Printed Digital Image Correlation

- Statistical analysis was performed for all high temperature tests
- There was no statistical significance between the strain measured by the extensometer and DIC
- There was a high linear relationship ($R^2 = 0.973$ or higher) between data from the extensometer and DIC

Printed	Pitch Size	P-Values, R ²			
Pattern Type	(µm)	100 °C	200 °C	300 °C	600 °C
Dots	150	0.640,	0.546,	0.526,	0.542,
		0.998	0.995	0.981	0.973
	100	0.422,	0.363,	0.293,	0.213,
		0.990	0.983	0.980	0.975
Lines	Lines 150 0.69	0.690,	0.450,	0.430,	0.297,
		0.996	0.998	0.994	0.984
Spray-Painted Pattern		0.798,	0.529,	0.564,	0.325,
		0.997	0.988	0.981	0.992

Results and Accomplishments (3/5) Printed Digital Image Correlation

- 2D DIC depends on the camera being perpendicular to the test specimen
- Any out-of-plane displacement will cause error in strain computation
- Different distances, angles, and tilts were explored



