Low-Cost, Efficient and Durable High Temperature Wireless Sensors by Direct Write Additive Manufacturing for Application in Fossil Energy Systems

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Agenda

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The Team

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PI Prof. Rahul Panat moved from WSU to CMU last year (fall 2017)
Background

- In-situ monitoring can lead to
  - Improved safety
  - Increased fuel efficiency
  - Improved system design

- Monitoring is challenging due to
  - Manufacturing limitation (due to complex surfaces)
  - Materials limitations (harsh operating conditions and high temperature)

- We are exploring nanoparticle based additive printing for sensor fabrication and high temperature electronics with wireless transmission
Project Goals and Objectives

☐ Goals:
  - Demonstrate the feasibility of low-cost aerosol jet manufacturing for Fossil Energy (FE) systems and develop materials, next-generation sensors (Strain and Pressure) that can reliably operate at high temperatures (>350 °C up to 500 °C) with wireless transmission (Passive electronics)
Tasks and Timelines

Key Milestones:
- Method Development and Sensor Material Characterizations (Impedance analysis, Oxidation study, Micro/Nano structure study): **Done**
- Primary Material Selection: **Done**
- Single Sensor Design, Fabrication and Testing: **Done**
- Reliability Tests (adhesion, nanoindentation, TEM): **Ongoing**
- Wireless system design and fabrication: **Ongoing**
Task 1: Manufacturing Method and Material System

- Manufacturing Method: Additive Printing
- Material Selection:
  - Study of electrical characterization by impedance spectroscopy
  - Microstructuctural observation through SEM, TEM, XRD, AFM
  - Study of oxidation resistance by TEM/SAED, XRD, XPS, TGA
Approach: Aerosol-Jet Direct-write Printing

- Operates by creating aerosol mist of 1-5 μm droplets, each containing nanoparticles
  - Stand off height about 5mm
  - No restriction on substrate type
  - Minimum feature size down to 10 μm
  - Printing accuracy of ± 2 μm stdev
- Clog resistant nozzles
- Ink viscosity up to 1000 cP
Aerosol-Jet Printing Video
High Resolution Printing

- High spatial resolution
- Feature size down to 11 μm
- High consistency in width
Material Systems

- Printed and sintered Silver (Ag) films
  - Viscosity: 1cP
  - Particle Size: 20-30 nm

- Printed Dispersed Carbon Nanotubes (CNTs) films
  - Viscosity: 1cP
  - Diameter: 100 nm

- Printed and sintered Nickel (Ni) films
  - Viscosity: 16-25cP
  - Particle Size: 20-100 nm

- Nichrome (NiCr) Alloy Nanoparticles
  - Viscosity: 1-5cP
  - Particle Size: 100 nm
Electrical Characterization on Sensor Segment

Impedance Spectroscopy

\[ Z = R + jX \]

Real Part, Resistance
Imaginary Part, Reactance (Inductive and Capacitive)

Target is to evaluate temperature and frequency dependent impedance/electrical property change on sensor segment (i.e. passive element)

- Temperature range: 24-500 °C
- Frequency range: 0.020-300 kHz
- Temperature interval: 50 °C
High Temperature Stability of Silver (Ag)NP Film

We observed instability in the impedance data. The instability was traced to grain growth, which increases conductivity.

* Issue solved by increasing the sintering time at the beginning of the sensor test to stabilize (increase) the grain size

* $Z'$ real part of impedance
Impedance Spectroscopic Characterization of Ag

- Sintered for 30 min at 200 °C

Increase in $Z'$ up to 150 °C

$Z'$ drops beyond 150 °C

Unstable impedance behavior

TCR for 24-150°: 0.000302/°C (at 20 Hz)

TCR for 150-300° C: -0.0059 /°C, at 20 Hz
Impedance Spectroscopy: Effect of long sintering time

We observed instability in the impedance data. The instability was traced to grain growth, which increases conductivity.

Issue solved by increasing the sintering time at the beginning of the sensor test to stabilize (increase) the grain size.
Oxidation Analysis of Ag NP Film

- XRD results did not show any oxide phases at high temperatures.
- XPS: No peak shift or change in peak shape due to heating.
- Ag did not oxidize when exposed to 500 °C.

Based on this study we conclude that Ag films can be used as sensor elements up to 500 °C.
High Temperature Stability of CNTs

- CNT dispersion were printed and then washed with DI water to make the film conductive.
- EIS analysis shows high impedance gain at 500 °C.
- Thermogravimetric analysis was performed up to 700 °C, shows about 40 % weight loss at 500 °C.

Based on this study we conclude that use of CNT films is not feasible at high temperatures (above 200-300 °C).
High Temperature Behavior of Ni NP Films

- Impedance increases by 2 order of mag. compared to RT beyond 350 °C indicates an onset of oxidation.
- TGA analysis shows high weight gain about 19.34 % at 500 °C indicating heavy oxidation of Ni NPs.

Use of Nickel NP films challenging beyond 300 °C due to oxidation.
High Temperature Behavior of NiCr Nanoparticles (80:20 wt%)

- NiCr NPs as potential materials for additive printing of high T sensors
- Thermogravimetric analysis was performed on the NPs at different heating rates up to 973K – **Minimal oxidation up to 400-450 °C**
- For films with sintered nanoparticles, the oxidation behavior is expected to be significantly better
Materials Characterization

- Ag undergoes no/minimal oxidation up to 500 °C
- Use of CNTs is challenging at above 200 °C-300 °C
- Ni is a suitable candidate for sensors application up to 350 °C
- NiCr alloy can be useful in the temperature of RT-450 °C

Silver NP film is a suitable candidate for sensor applications in the temp. range of RT-500 °C
Task 2: Single Sensor Design and Testing
High Temperature Sensor Set Up

- Able to provide 1500 με on the beam
- Deflection frequency: up to 10 Hz
Sensor Fabrication

We have developed a robust protocol for sensor design and fabrication which give rise to repeated properties.
Calibration of the High Temperature Sensing Test Set up

WK Series Commercial Gauge

(a) 4-wire probing was used to measure the R change using 7½-digit precision multimeter
• Sensor was installed in a SS substrate
• Resistance of the Sensor: 120 Ω
• Given GF: 2.0 (from manufacturer)
• Measured strain in the beam: 862 με using the following equation:

\[ g = \frac{\Delta R}{R} \frac{1}{\varepsilon} \]

EA Series Commercial Gauge

(b) 4-wire probing was used to measure the R change using 7½-digit precision multimeter
• Sensor was installed in a SS substrate
• Resistance of the Sensor: 350 Ω
• Given GF: 2.0 (from manufacturer)
• Measured strain in the beam: 878 με using the following equation:

Both the commercial gages showed similar strain in the beam, which indicates a proper calibration of the strain measurement system.
FEM Analysis to Understand the Effect of Insulation Layer on Beam Strain

Without Insulation layer

With Insulation layer

The variation in $\varepsilon$ was found to be only about 1%
Sensitivity/Gauge Factor of Printed Strain Sensor

- Four-wire probing was used to measure the R change using $7_{1/2}$-digit precision multimeter

$$g = \frac{\Delta R / R}{\epsilon}$$

- Sensitivity of Strain Sensor is given by Gauge Factor (GF)
- GF: $3.15 \pm 0.086$, with standard deviation within 2.7% of the mean
  - Commercial sensor has a GF of 2.0

- High measurement repeatability during cycling
- Additively manufactured sensors shows a highly repeatable performance along with higher GF than commercial gages
Why Gauge Factor is High?

- Gauge Factor/ Sensitivity of the Sensor,

\[
GF = 1 + 2\nu + \left(\frac{d\rho}{\rho}\right)/\left(\frac{dl}{l}\right)
\]

- Contribution due to shape change
- Contribution from piezo resistivity

- GF: 3.15 ± 0.086,
- Commercial sensor has a GF of 2.0

- Piezo-resistive effect could be positive or negative, generally small, compared to other materials such as semi-conductors

- For Ag this effect is about less than 20% of the total GF which indicates in our case Poisson ratio must be close to 0.7, which is higher than what solids can achieve

Porosity of the film increases Poisson ratio, hence GF
Analytical Model for Effective Poisson Ratio

- We developed a model by considering our film as a cellular material.
- For cellular materials, geometric effects can give rise to superior mechanical properties.
- $L_{AD}$ extends by $2\delta$ with consequent change in $L_{AB}$ by $\delta_{AB}$, the relation between $\delta$ and $\delta_{AB}$ can be shown as,

$$\delta_{AB} = \delta \sin \theta$$

Where, $L = \sqrt{L_y^2 + L_x^2}$, $\sin \theta = \frac{L_y}{L}$, $\cos \theta = \frac{L_x}{L}$,

So, $\delta_{AB} = \frac{fL}{AE} = \left(\frac{FL}{AE}\right) \times \left(\frac{1}{2 \sin \theta}\right)$

Hence, $\delta = \left(\frac{F}{2AE}\right) \times \left(\frac{L_y^2}{L_x}\right)$

$$\delta_{BC} = \frac{P(2\times L_x)}{AE} = \left(\frac{2F}{AE}\right) \times \left(\frac{L_x^2}{L_y}\right)$$

Modelling was performed by Prof. H. Zbib
Analytical Model for Effective Poisson Ratio

• Now due to the deformation, strain along X and Y direction becomes,

\[
\varepsilon_{AD} = \left( \frac{2\delta}{L_{AD}} \right) = \left( \frac{F}{2AE} \right) \times \left( \frac{L}{L_y} \right)^3,
\]

\[
-\varepsilon_{BC} = \left( \frac{\delta_{BC}}{2L_x} \right) = \left( \frac{F}{AE} \right) \times \left( \frac{L_x}{L_y} \right),
\]

Poisson’s ratio: \( \nu^* = \nu_{12}^* = \left( \frac{\varepsilon_{BC}}{\varepsilon_{AD}} \right) = \left( \frac{2L_x L_y^2}{L^3} \right), \)
Porosity can result in higher Poisson ratio for 3-D printed films

Analytical Model for Effective Poisson Ratio

Aspect ratio: \( \alpha = \frac{L_y}{L_x} \)

\[ \nu^* = \left( \frac{2\alpha^2}{(1+\alpha^2)\sqrt{1+\alpha^2}} \right) \]

\( \theta \approx 60^\circ, \alpha \approx 1.7, \nu^* = 0.75 \)

\( \nu_{\text{max}} \) at \( \alpha = 1.414 \)
High Temperature Sensor Measurement Overview

- Four-wire constant current measurement method was used for HT measurement

\[ V = I \times R_g \]
\[ V = 2 \text{ mA} \times 5 \Omega = 10 \text{ mV} \]
\[ \Delta V = I \times \Delta R = \Delta V \text{ mV} \]
\[ \Delta V = 2 \text{ mA} \times 0.015 \Omega = 0.03 \text{ mV} \]
High Temperature Sensor Output As-measured through Oscilloscope

- Representative HT sensor output for a sensor with $R = 5.15 \, \Omega$
- Excitation Current $= 2.0 \, mA$
- Amplifier Gain $= 3300$
- Stable Signal at High Temperatures
Thermal Output of the HT Sensor as a Function of Temperature

\[ \varepsilon_{thermal} = \varepsilon_{total} - \varepsilon_{stress} \]

3-D printed NP-based films show superior performance both in sensitivity and at high temperatures

M. T. Rahman et al., J. Appl Phys: Vol. 123 (2), 024501, 2018

Strain Sensor Work Caught Media Attention

Research on high temperature strain sensor was highlighted in several tech magazines:

**CMU News:** 3-D printing remakes the strain gauge

**Physics.org:** 3-D printing remakes the strain gauge

**3Dprint.com:** 3D Printing Put to Use to Create More Sensitive Strain Gauges for High-Temperature Applications

**India today:** New 3D printing method for more sensitive strain gauges

**3dprintingprogress.com:** New 3-D printing technique for manufacturing strain gauges
Task 3. Reliability Study of the Sensor
Films were created on 2 mm thick alumina substrate. Sample were then cured for 3 hours at 60°C followed by sintering at 200°C for ½ Hour. Initial results show that thermal cycling improves strength of the film!
Interfacial Reliability Study

- Focused ion beam was used to observe cross section of the isothermal samples
- Existence of intermediate phase was observed
- TEM analysis will be performed to understand the intermediate phase
Summary of Research

Deliverables of year 1-2:

- Manufacturing Process Selection ✓
- Material characterization and selection (lead and backup) ✓
- High temperature testing set up ✓
- Strain sensor response at high temp ✓
- Reliability Study ✓
  - Interfacial TEM observation
- Wireless design and fabrication
Year-1-2: Student Training and Research Outcomes

Student Training
1. 1 PhD student graduated, 1 PhD student (minority) working on the project
2. 2 students pursuing MS, 1 MS student graduated in March 2018
3. 3 Undergraduate researchers (1 minority through Louis Stokes Alliance for Minority Participation), 1 Postdoc

Journal Papers

Additional journal papers are in preparation

Conference Presentations:
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2018 Deliverables

- Completion of reliability work
- Fabrication of workable antenna at high temperatures
- Wireless system design and fabrication
Wireless Antenna Work: (with WVU)

- Encouraging preliminary results, where antennas could ‘talk with each other
- Change in response as a function of temperature
- Further work will involve EM simulations and transmission…….
High Temperature Pressure Sensor Testing Protocols
-UTEP

Pressure Sensor testing

[Diagram showing a setup with labeled parts such as Dual gauge, Sample holder, Chamber, Heater, Valve for gas filling, Valve for gas release, Thermocouple, 4-probe contact, Spring for slight pressure, 4-probe assemble inside the chamber, Wire, Ceramics, Wafer film]
Thank You