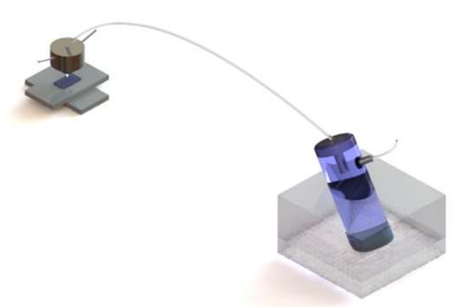
Low-Cost, Efficient and Durable High Temperature Wireless Sensors by Direct Write Additive Manufacturing for Application in Fossil Energy Systems



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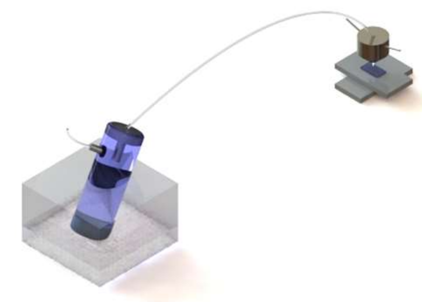

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Agenda

- Introduction and Background
  - Team
  - Project Goals and Objectives
  - Tasks and Timelines
- Research Accomplishments
  - Manufacturing: Additive Printing Technique
  - Material Selection
  - High Temperature Test Set-up
  - Single Sensor Printing, and Characterization
  - Sensor Reliability
  - Student Training and Research Dissemination
- Summary of Research
- Future Direction

The Team

Carnegie Mellon University

Rahul Panat
Project Lead PI

Taibur Rahman
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Post Doc)

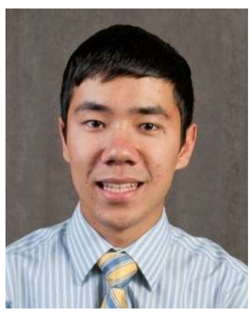
University of Texas, El Paso

C. V. Ramana
Project Co-PI

Juan Gomez
(PhD, expected ’18)

Postdocs: Dr. P. Dubey
Postdocs: Dr. M. Bandi

Washington State University



Russell Moser,
(MS), Janicki
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James Goding
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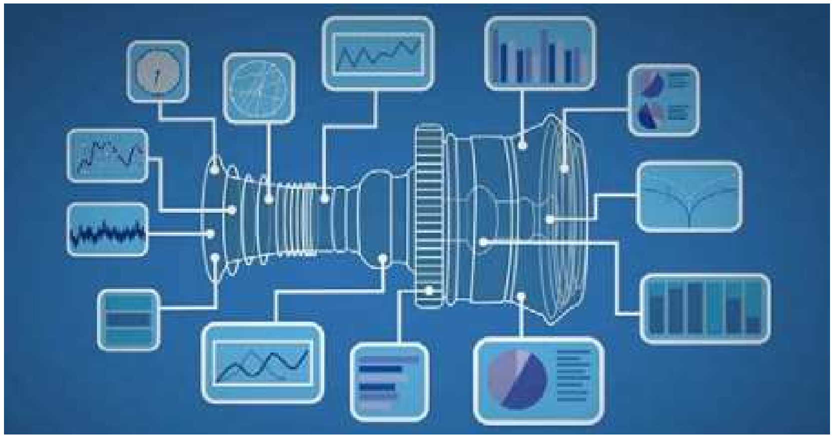
PI Prof. Rahul Panat moved from WSU to CMU last year (fall 2017)

Background

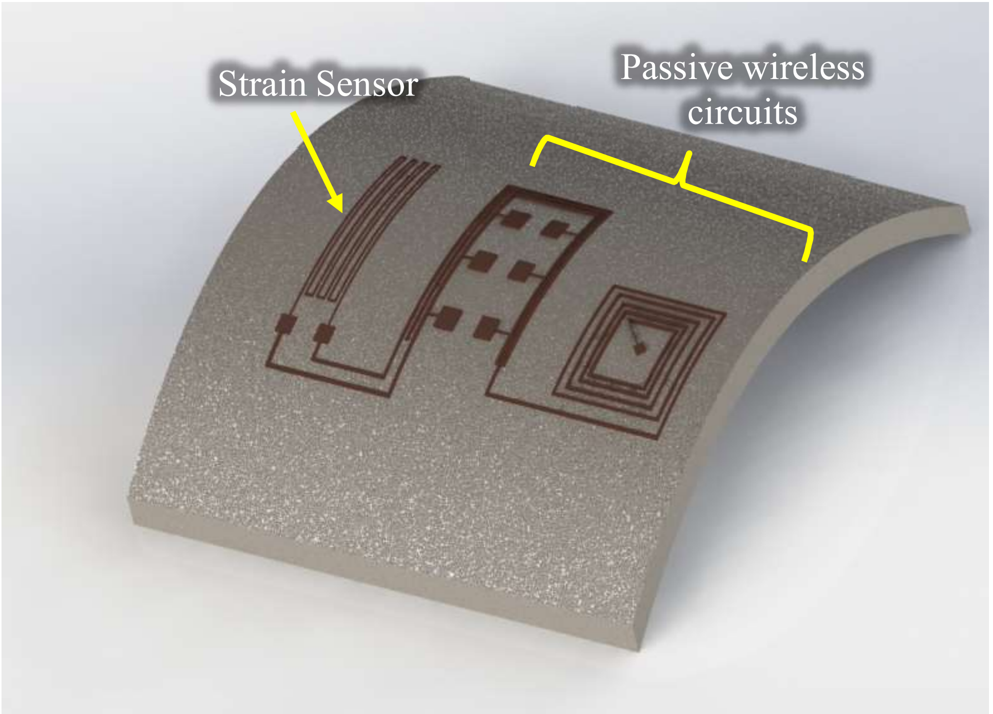


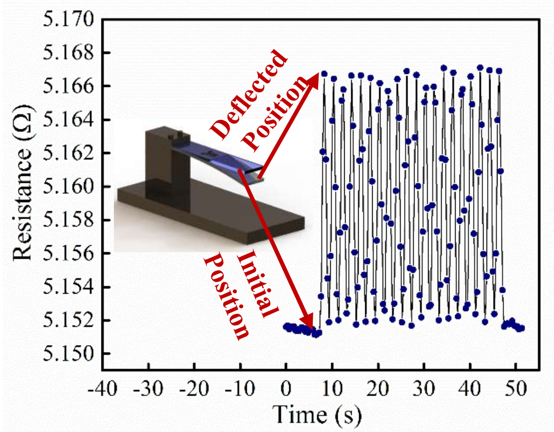

200 sensors across the turbine generate 300 data points per second

- ❑ 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

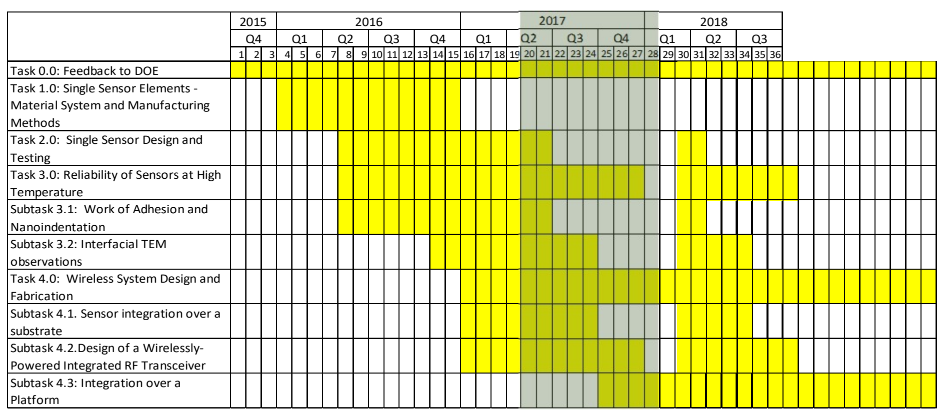
- Goals:
  - Demonstrate the feasibility of low-cost aerosol jet manufacturing for Fossil Energy (FE) systems and develop materials, next-generation sensors (**Strain and Pressure**) that can reliably operate at high temperatures (>350 ^{o}C up to 500 ^{o}C) with wireless transmission (**Passive electronics**)





Tasks and Timelines

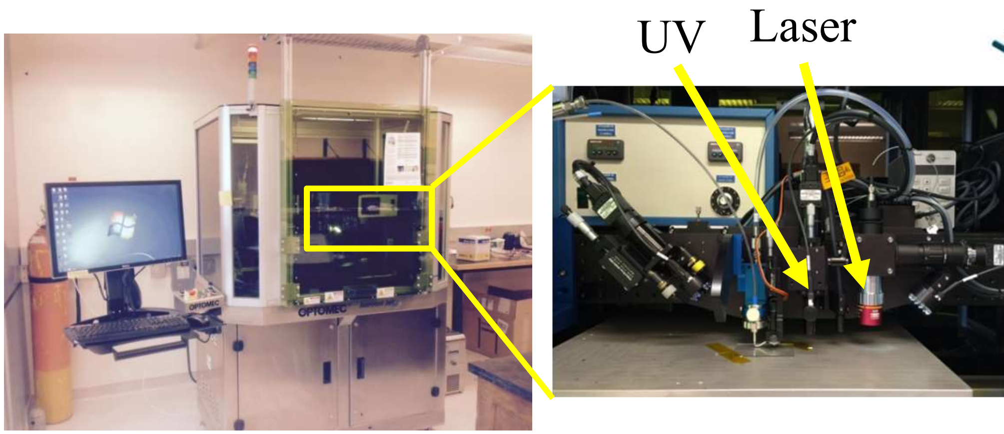
- Key Milestones:
  - Method Development and Sensor Material Characterizations (Impedance analysis, Oxidation study, Micro/Nano structure study): **Done**
  - Primary Material Selection: **Done**
  - Single Sensor Design, Fabrication and Testing: **Done**
  - Reliability Tests (adhesion, nanoindentation,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

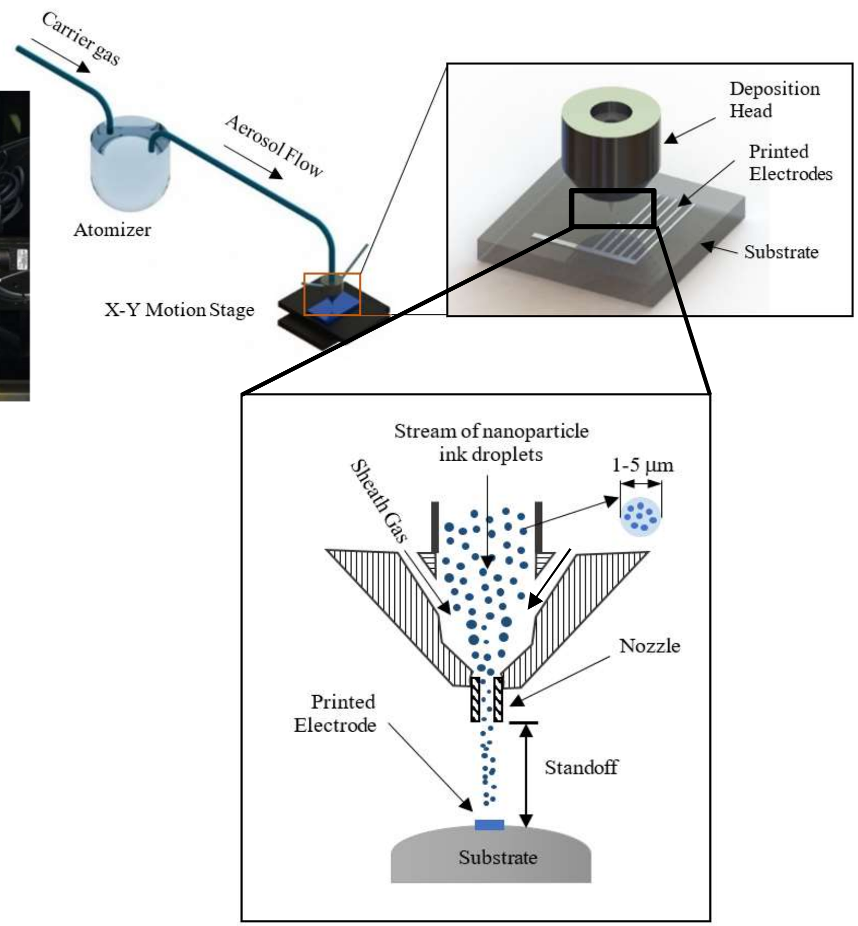


Task 1: Manufacturing Method and Material System

- 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

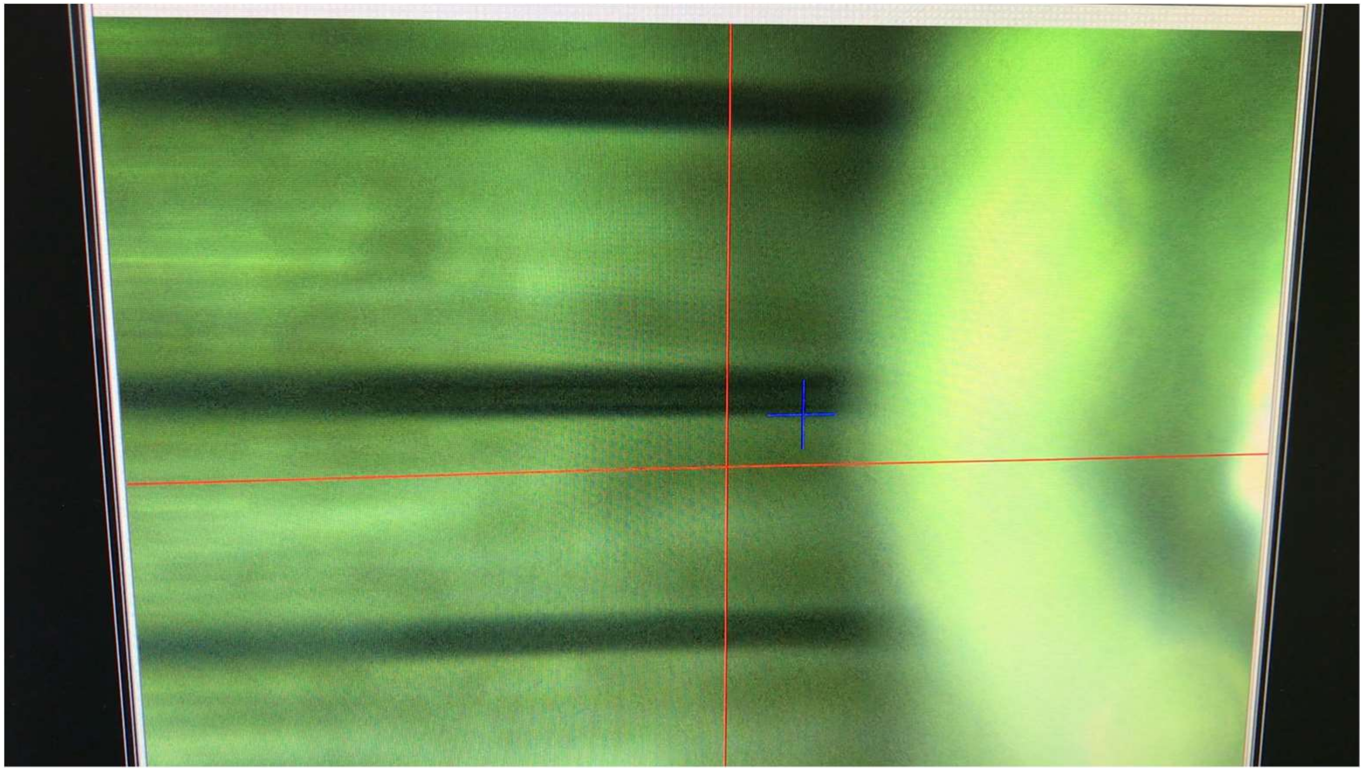
Approach: Aerosol-Jet Direct-write Printing



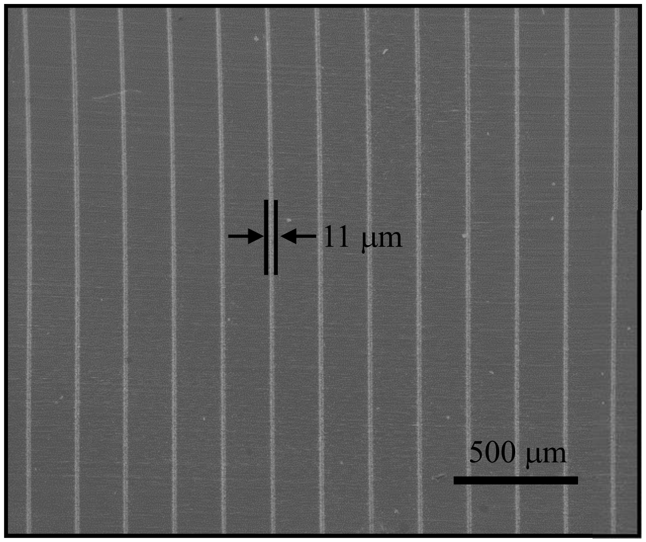


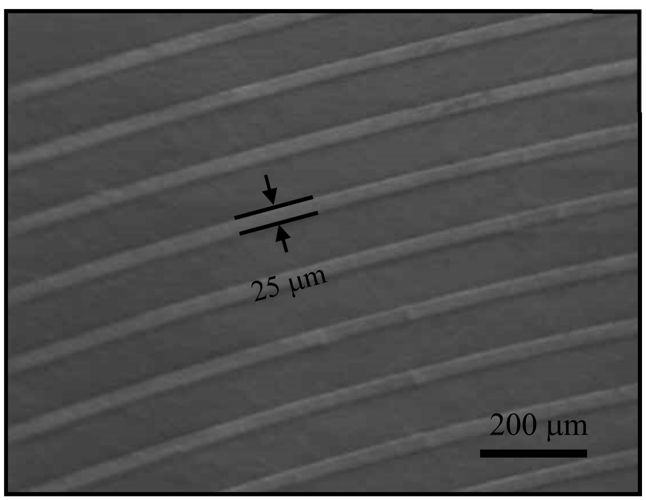
- ❑ Operates by creating aerosol mist of 1-5 µm droplets, each containing nanoparticles
  - Stand off height about 5mm
  - No restriction on substrate type
  - Minimum feature size down to 10 µm
  - Printing accuracy of ± 2 µm stdev
- ❑ Clog resistant nozzles
- ❑ Ink viscosity up to 1000 cP

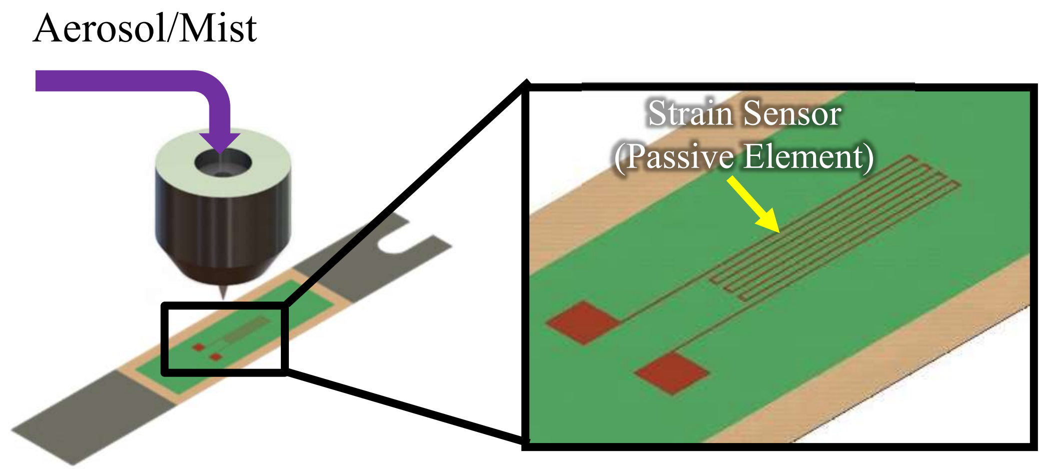
Aerosol-Jet Printing Video



High Resolution Printing







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

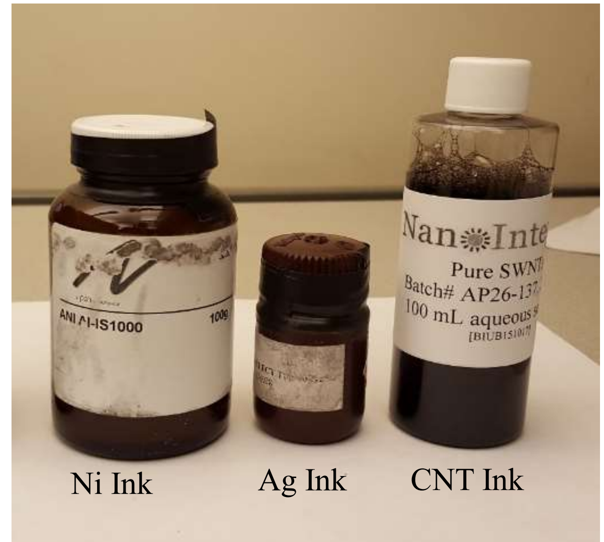
Material Systems

- 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

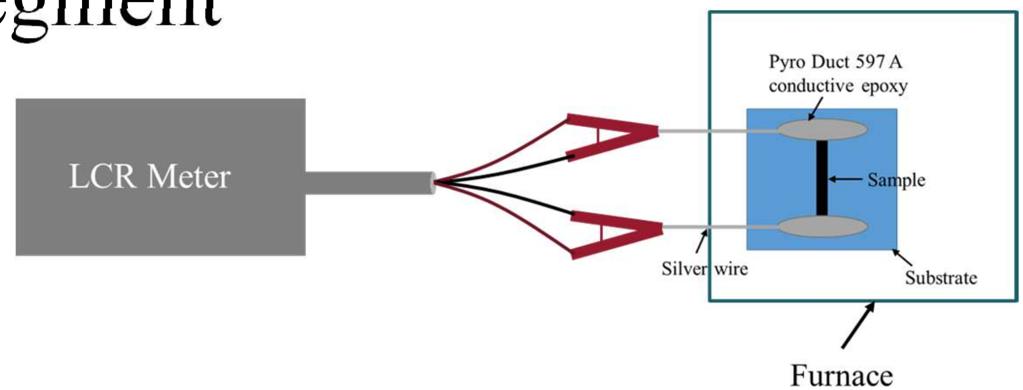


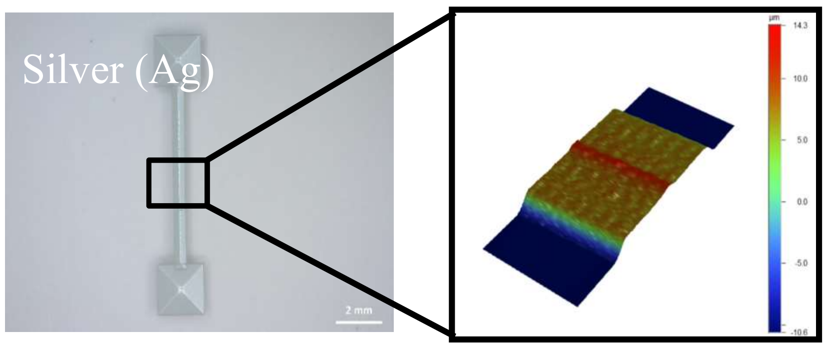
Electrical Characterization on Sensor Segment

Impedance Spectroscopy

$Z = R + jX$

- R → Real Part, Resistance
- X → Imaginary Part, Reactance (Inductive and Capacitive)



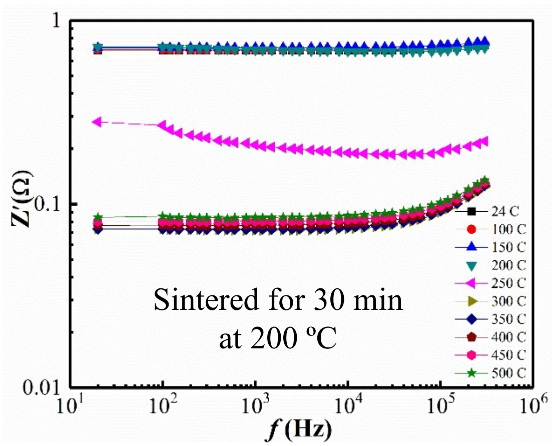


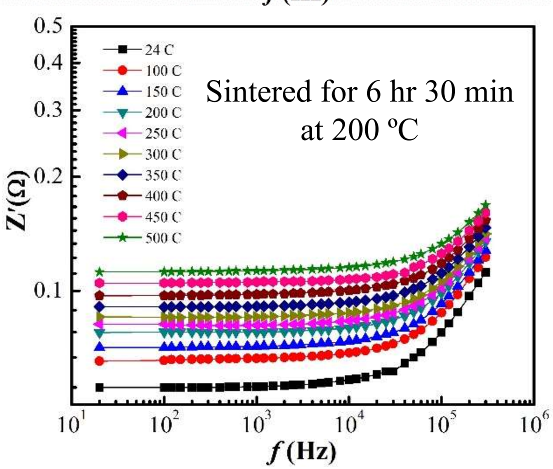
- ❑ Target is to evaluate temperature and frequency dependent impedance/electrical property change on sensor segment (i.e. passive element)

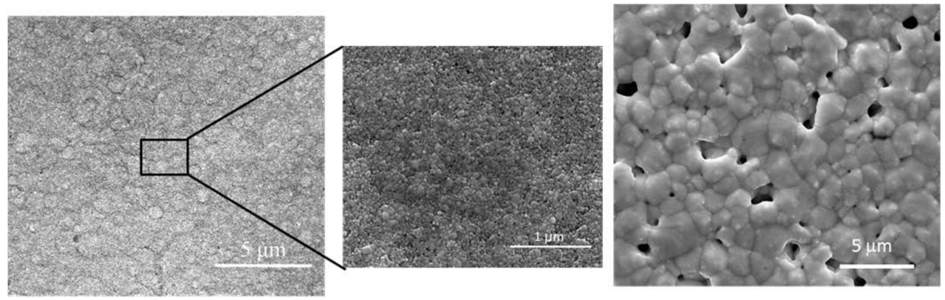
- Temperature range: 24-500 ºC
- Frequency range: 0.020-300 kHz
- Temperature interval: 50 ºC

High Temperature Stability of Silver (Ag)NP Film

Impedance Spectroscopy



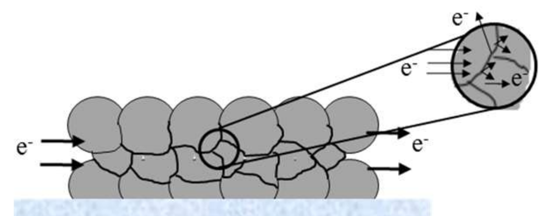




As Sintered – 200 °C/ 30 min

After impedance tests up to 500 °C

$$\rho = \rho_b + \frac{\rho_{gb}\delta_{gb}}{d}$$



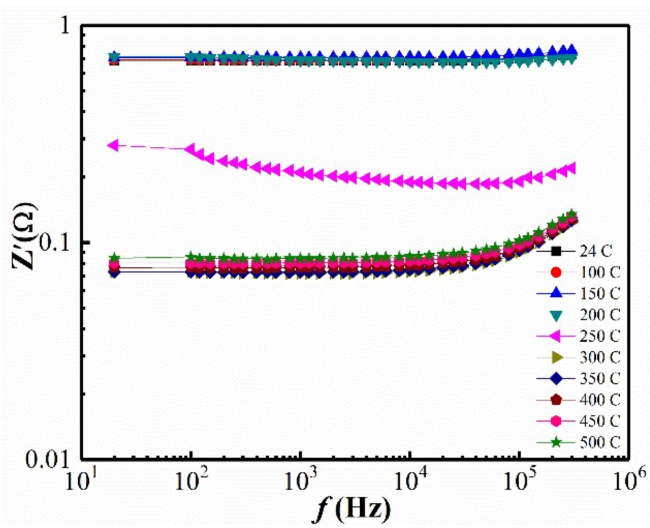
We observed instability in the impedance data. The instability was traced to grain growth, which increases conductivity

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

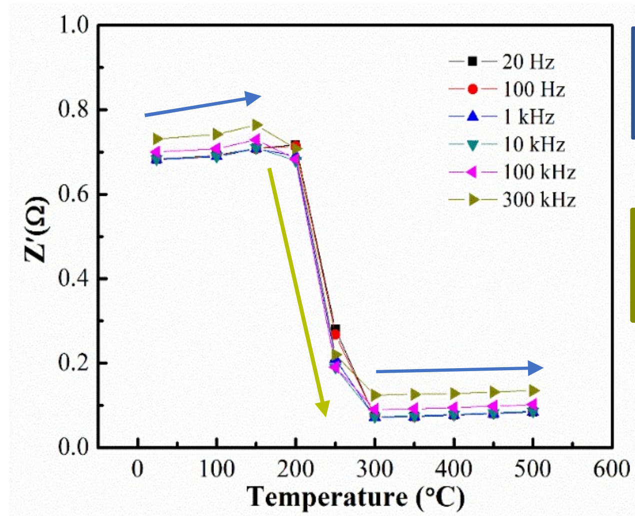
* Z′ real part of impedance

Impedance Spectroscopic Characterization of Ag

- Sintered for 30 min at 200 ºC

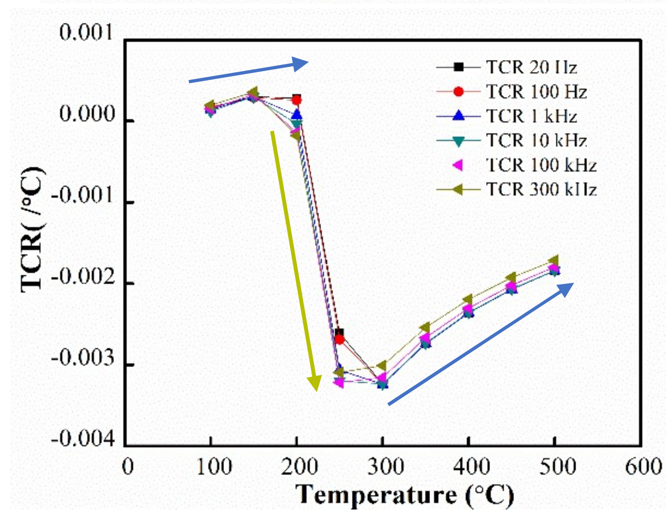


Unstable impedance behavior



Increase in Z' up to 150 °C

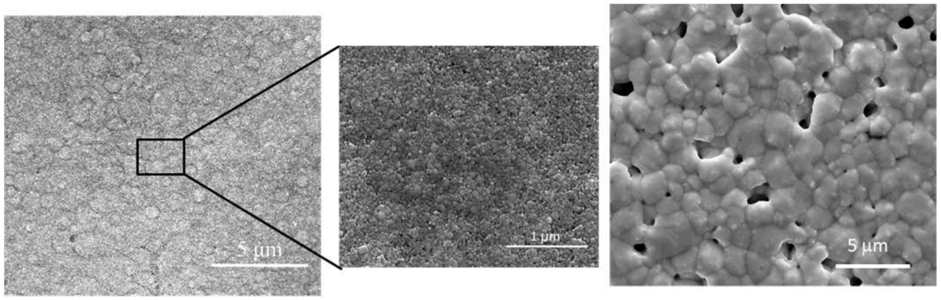
Z' drops beyond 150 °C



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

TCR for 150-300° C: -0.0059 /°C, at 20 Hz

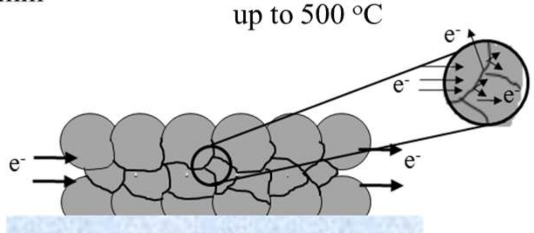
Impedance Spectroscopy: Effect of long sintering time



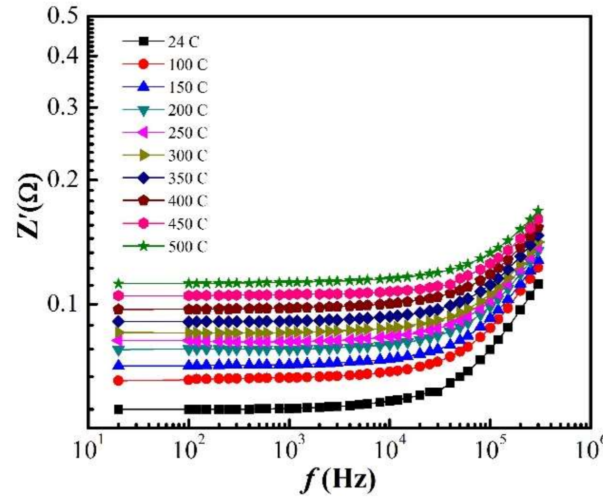
As Sintered – 200 °C/ 30 min

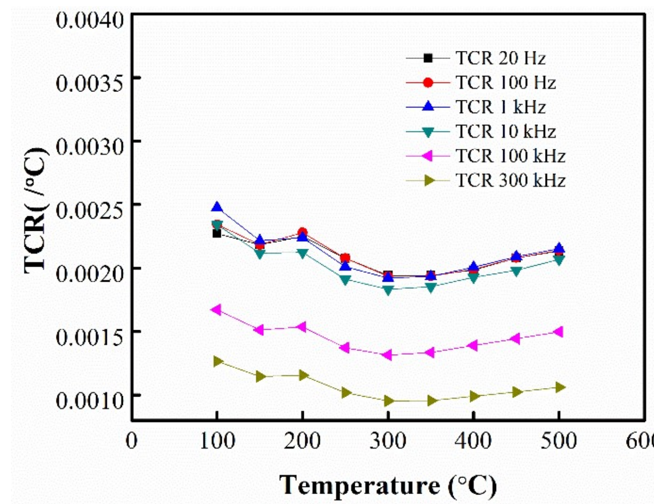
After impedance tests up to 500 °C

$$\rho = \rho_b + \frac{\rho_{gb}\delta_{gb}}{d}$$



Sintered for 30 min at 200 ºC, post processed 6 hr at 200 ºC



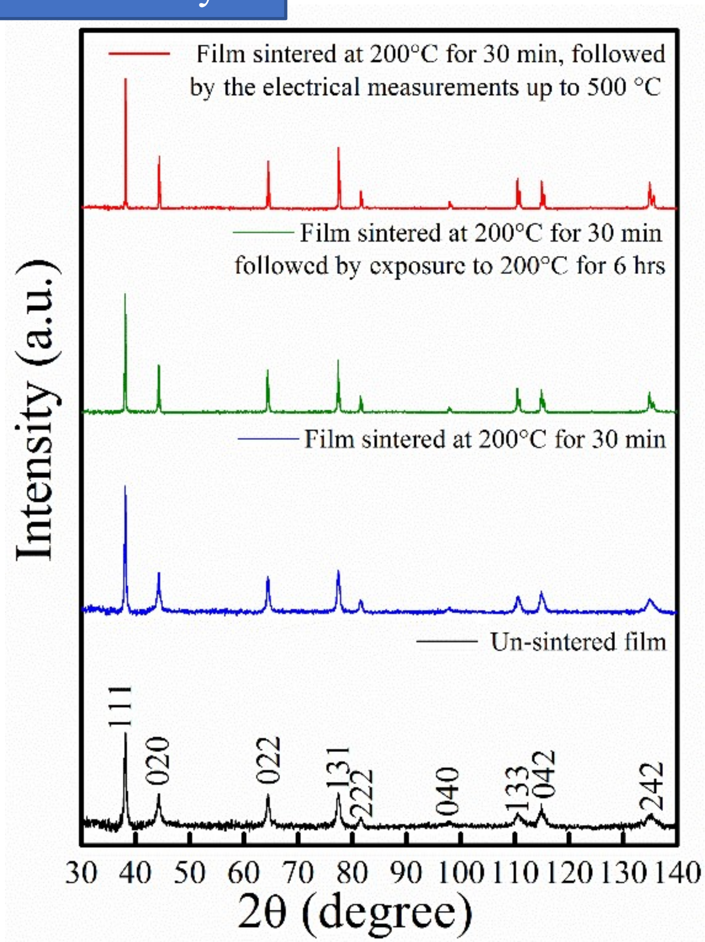


We observed instability in the impedance data. The instability was traced to grain growth, which increases conductivity

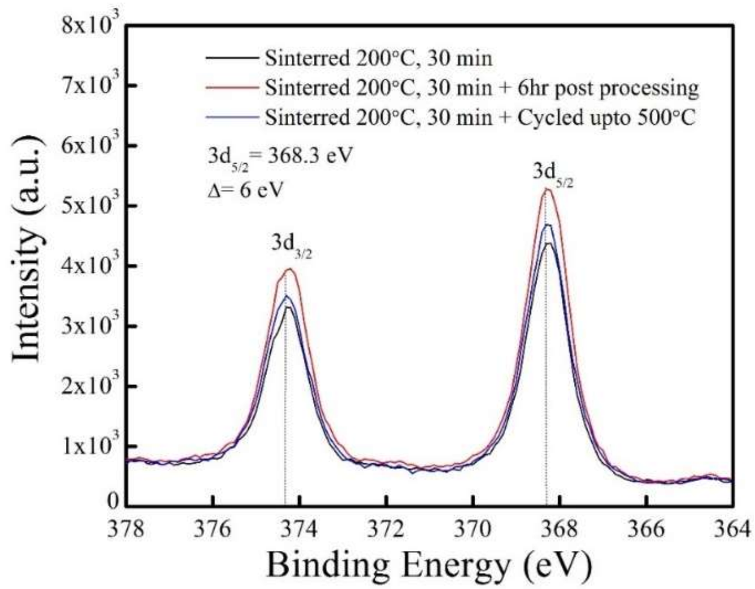
Issue solved by increasing the sintering time at the beginning of the sensor test to stabilize (increase) the grain size

Oxidation Analysis of Ag NP Film

XRD Analysis



XPS Analysis

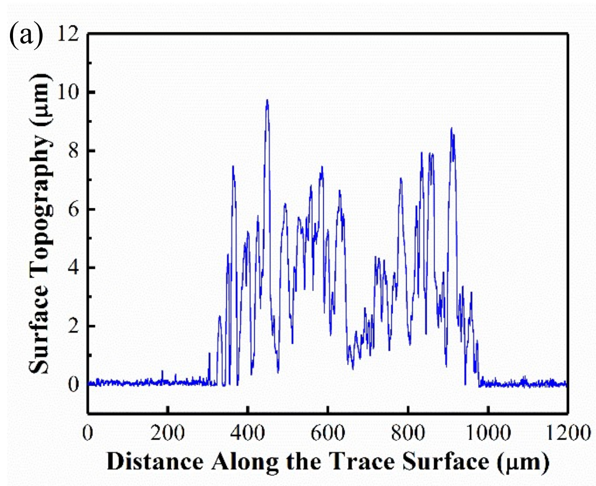


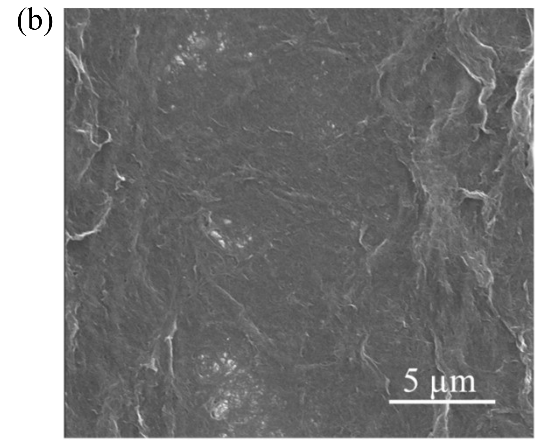
- XRD results did not show any oxide phases at high temperatures
- XPS: No peak shift or change in peak shape due to heating
- Ag did not oxidize when exposed to 500 °C

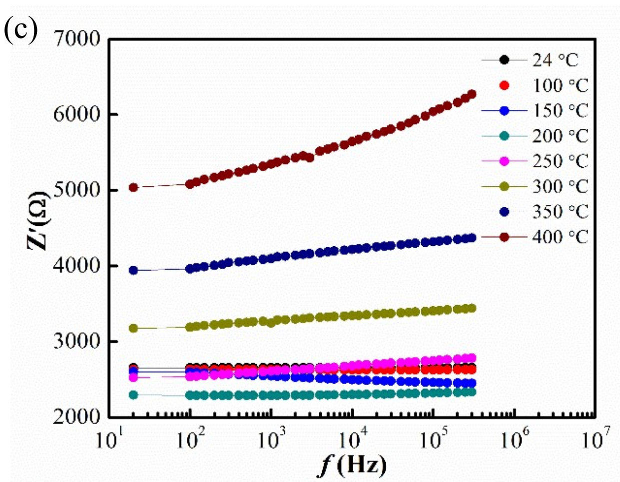
Based on this study we conclude that Ag films can be used as sensor elements up to 500 °C

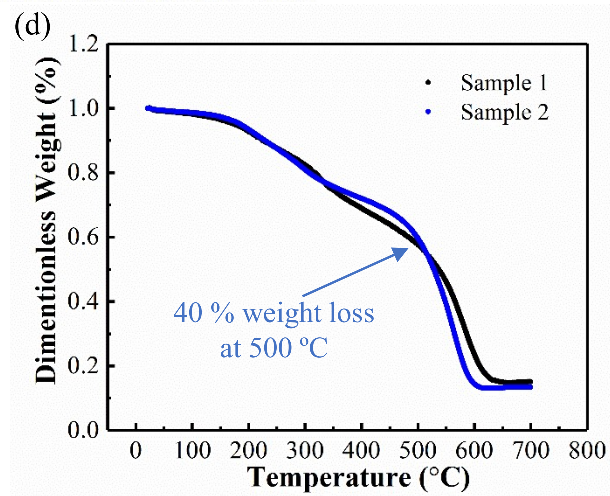
High Temperature Stability of CNTs

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



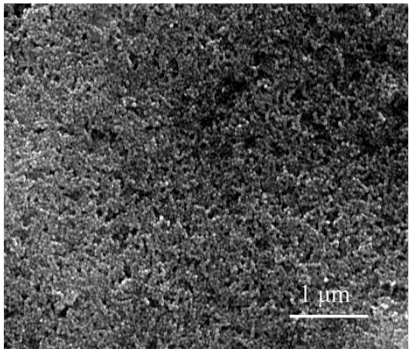


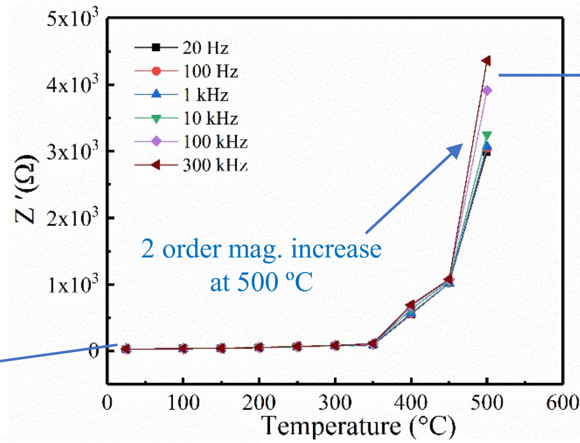


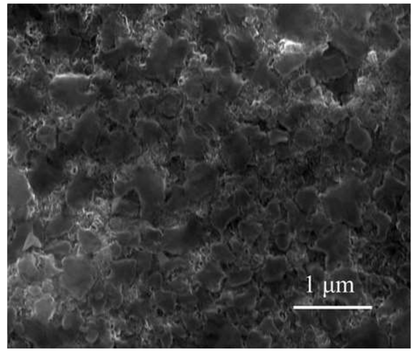


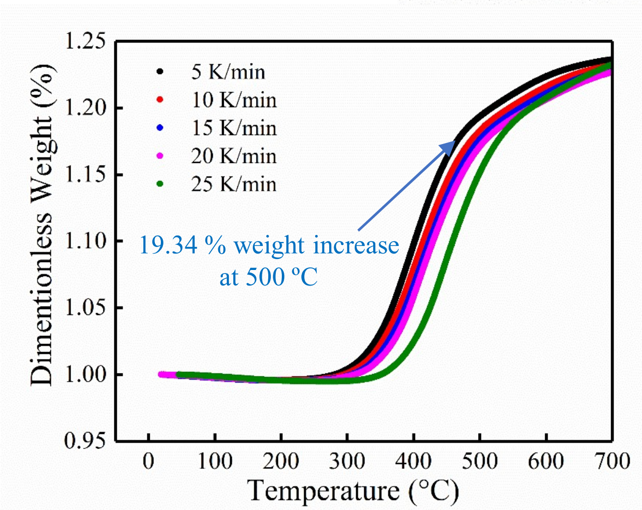
Based on this study we conclude that use of CNT films is not feasible at high temperatures (above 200-300 °C)

High Temperature Behavior of Ni NP Films







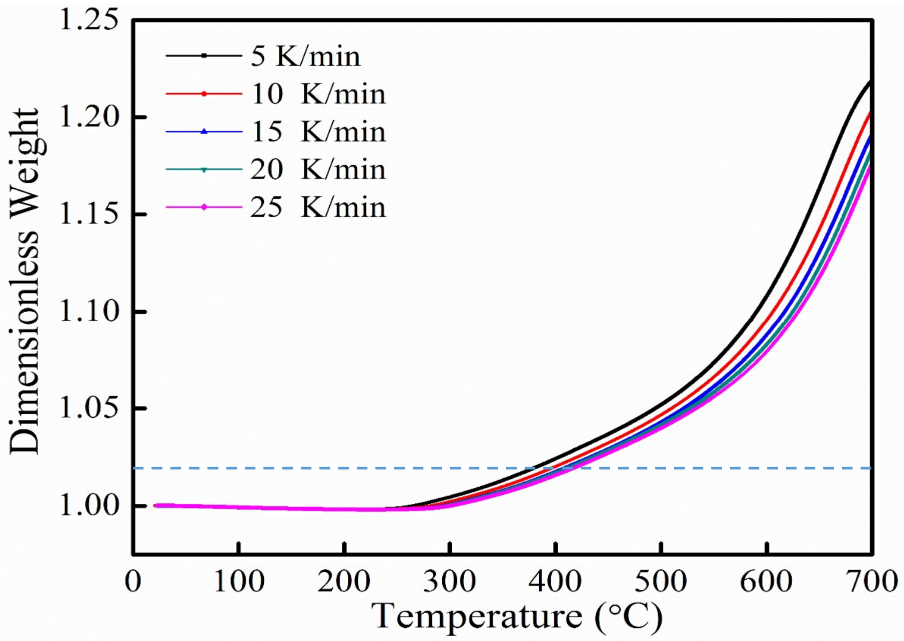


- ❑ Impedance increases by 2 order of mag. compared to RT beyond 350 ºC indicates an onset of oxidation
- ❑ TGA analysis shows high weight gain about 19.34 % at 500 ºC indicating heavy oxidation of Ni NPs

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
- Thermogravemetric 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



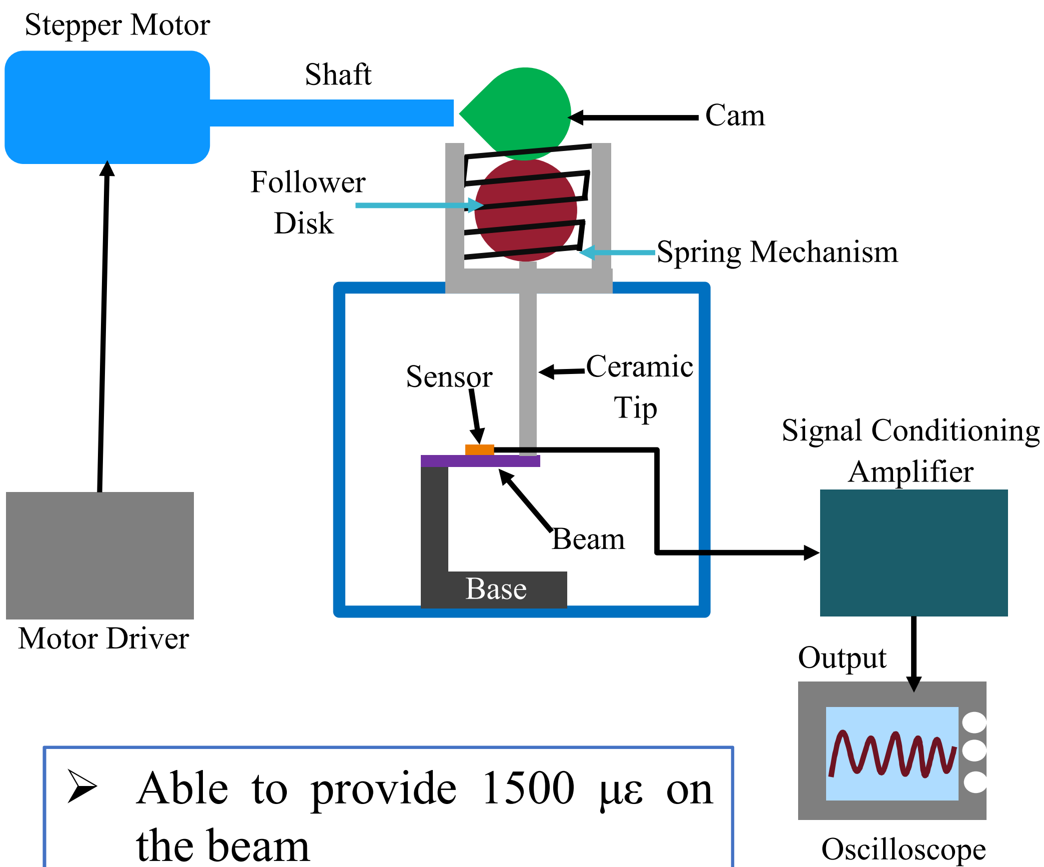
Materials Characterization

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

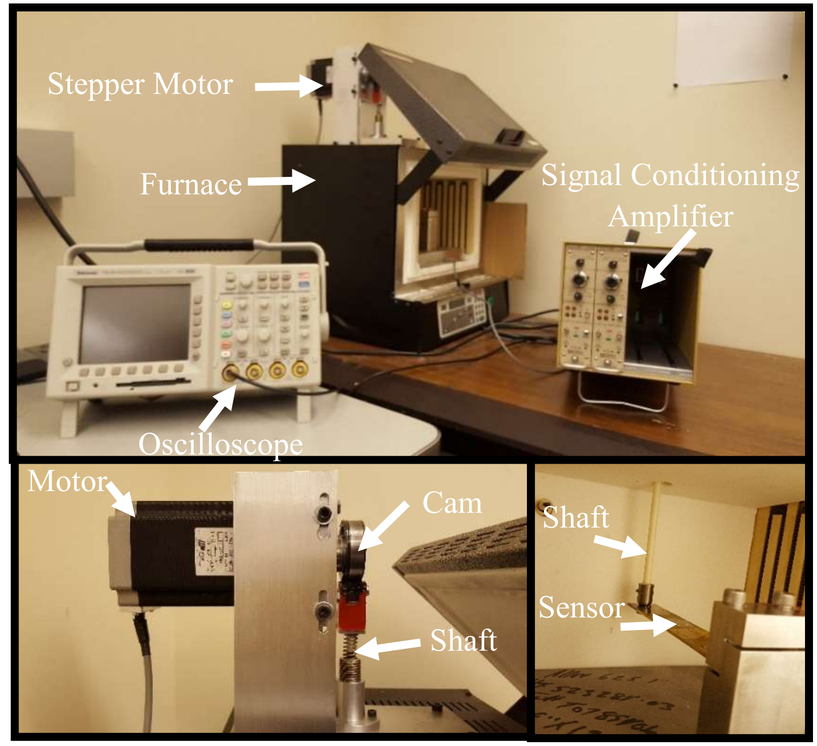
Silver NP film is a suitable candidate for sensor applications in the temp. range of RT-500 ºC

Task 2: Single Sensor Design and Testing

High Temperature Sensor Set Up

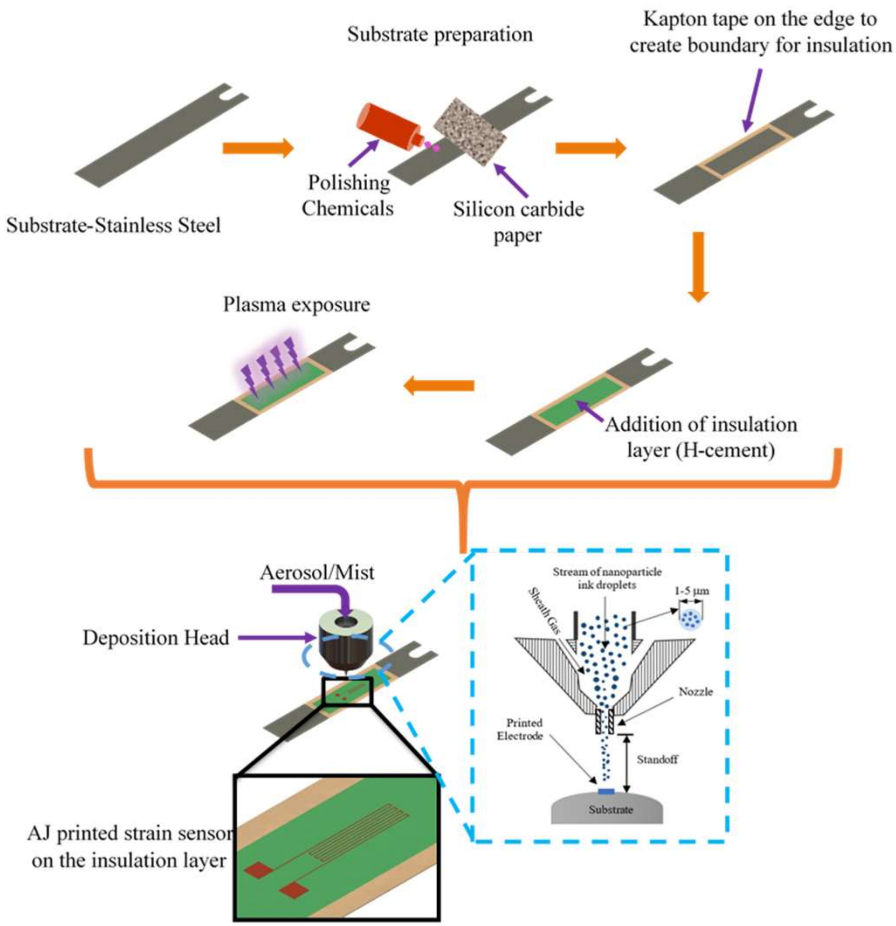


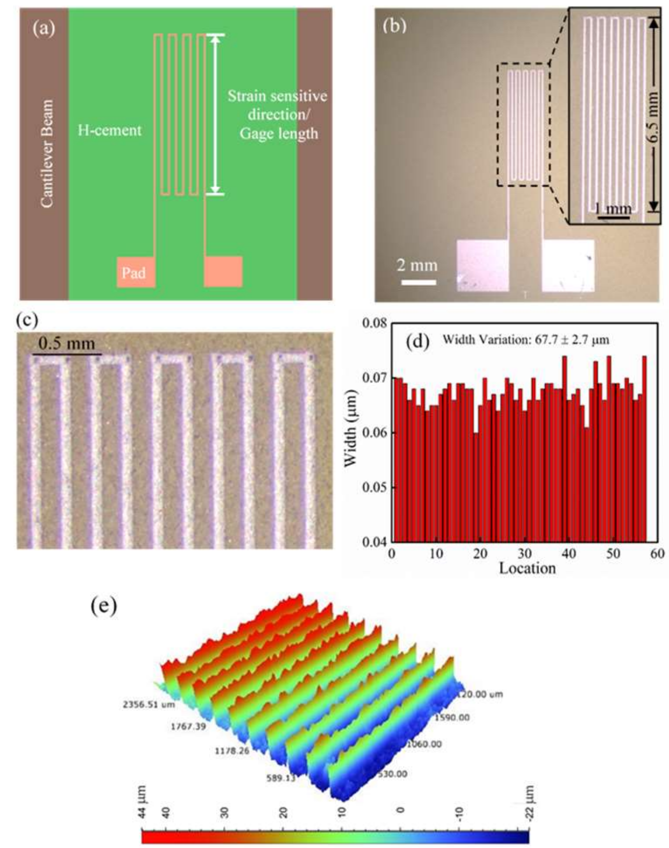
- Able to provide 1500 με on the beam
- Deflection frequency: up to 10 Hz



Actual Strain Sensor Test Set up

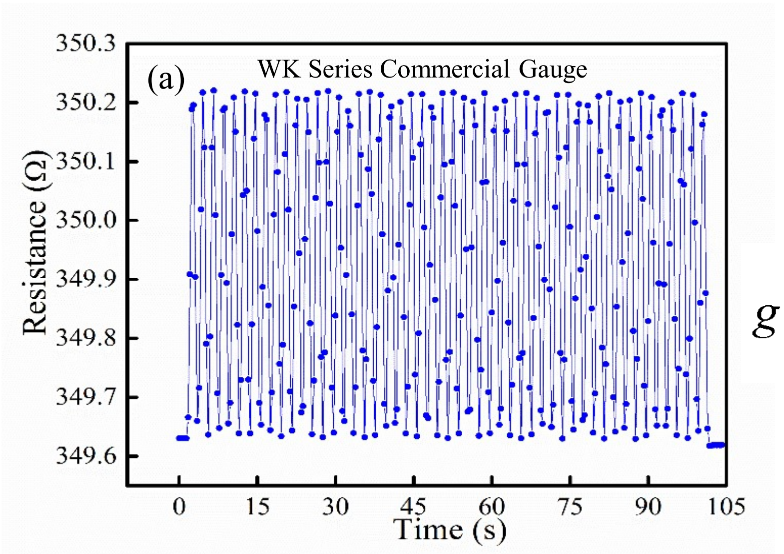
Sensor Fabrication





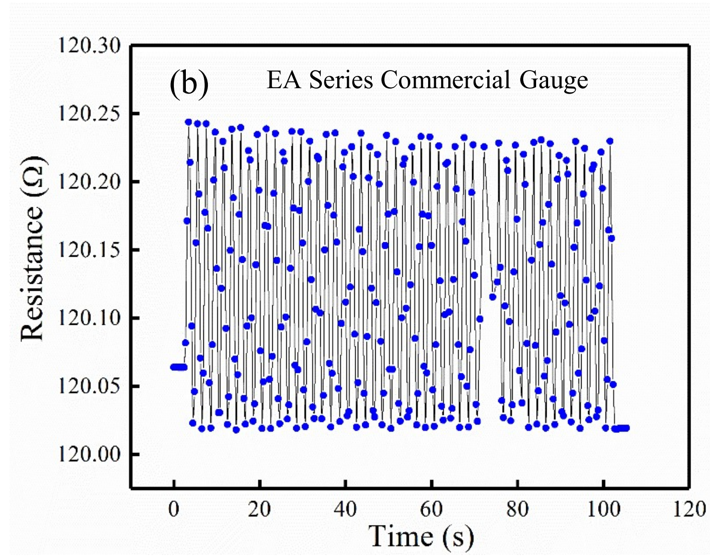
We have developed a robust protocol for sensor design and fabrication which give rise to repeated properties

Calibration of the High Temperature Sensing Test Set up



- 4-wire probing was used to measure the R change using $7_{1/2}$-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: 862 με using the following equation:

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



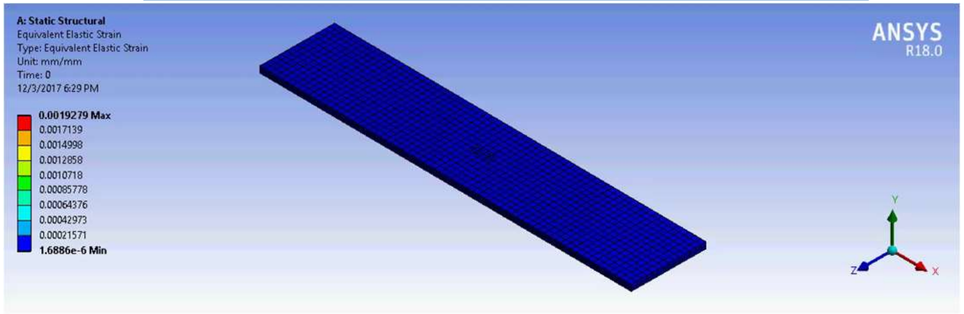
- 4-wire probing was used to measure the R change using $7_{1/2}$-digit precision multimeter
- Sensor was installed in a SS substrate
- Resistance of the Sensor: 120 Ω
- Given GF: 2.0 (from manufacturer)
- Measured strain in the beam: 878 με using the following equation:

Both the commercial gages showed similar strain in the beam, which indicates a proper calibration of the strain measurement system

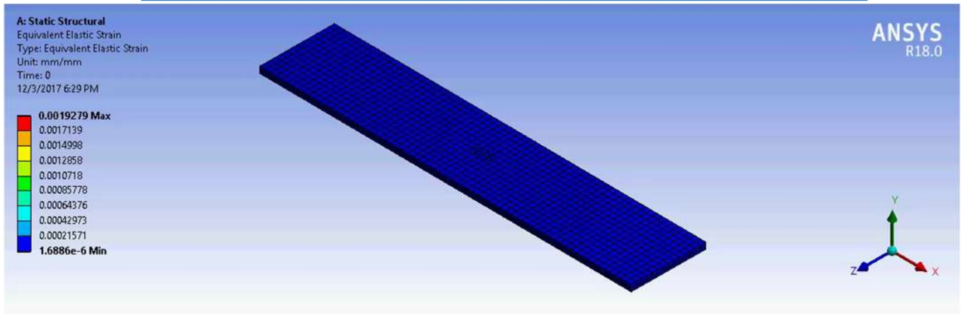
Video

FEM Analysis to Understand the Effect of Insulation Layer on Beam Strain

Without Insulation layer



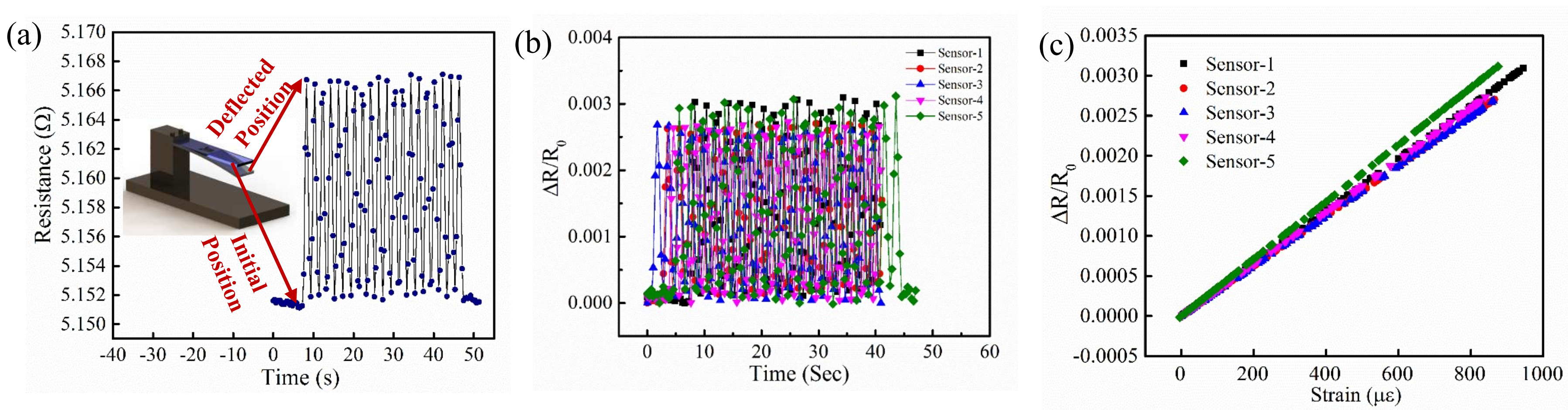
With Insulation layer



The variation in ε 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



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

- Sensitivity of Strain Sensor is given by Gauge Factor (GF)
- GF: 3.15 ± 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,

$$GF = 1 + 2\upsilon + (d\rho/\rho)/(dl/l)$$

Contribution due to shape change

Contribution from piezo resistivity

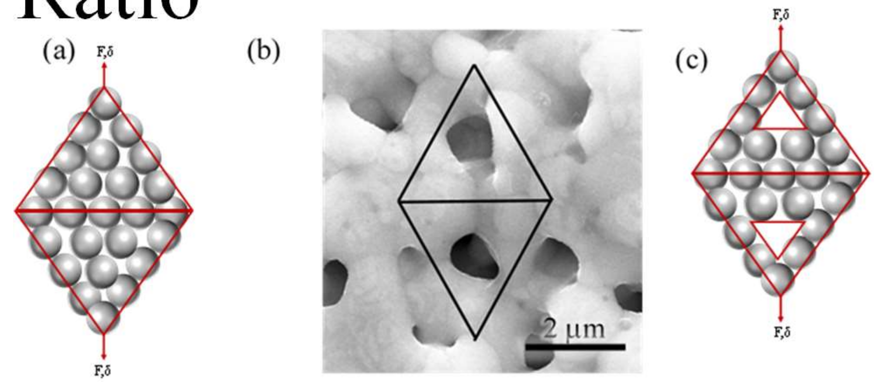
- GF: 3.15 ± 0.086,
- Commercial sensor has a GF of 2.0

- 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 2δ with consequent change in L_{AB} by δ_{AB}, the relation between δ and δ_{AB} can be shown as,



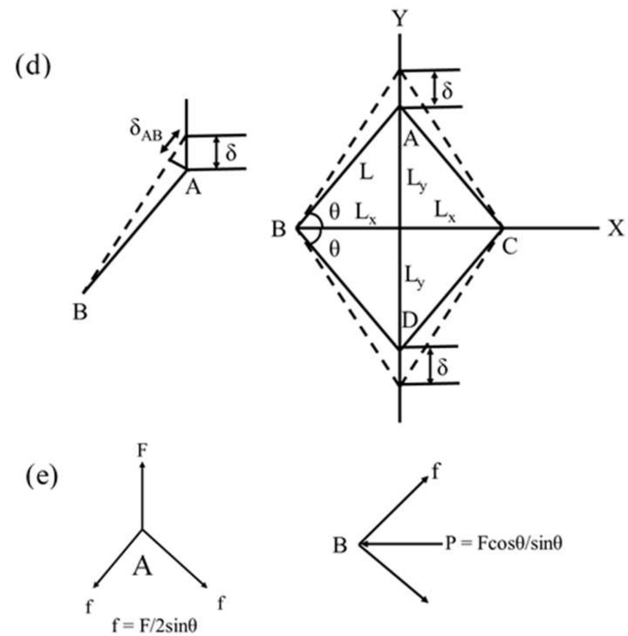
$$\delta_{AB} = \delta \sin\theta$$

Where, $L = \sqrt{\left(L_y^2 + L_x^2\right)}$, $\quad \sin\theta = \dfrac{L_y}{L}$, $\quad \cos\theta = \dfrac{L_x}{L}$,

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

Hence, $\delta = \left(\dfrac{F}{2AE}\right)\times\left(\dfrac{L^3}{L_y^2}\right)$

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



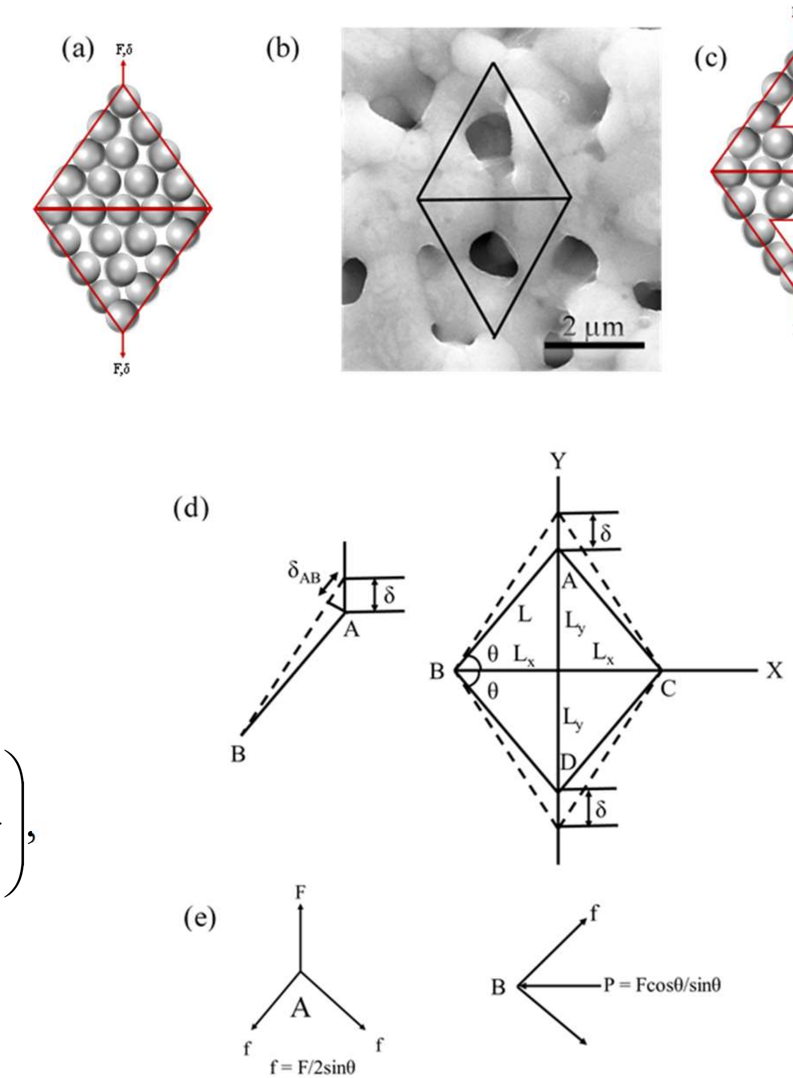
Analytical Model for Effective Poisson Ratio

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

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

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

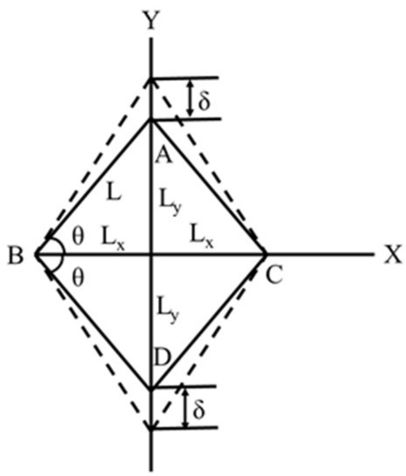
Poisson's ratio: $\upsilon^* = \upsilon^*_{12} = \left(\frac{\varepsilon_{BC}}{\varepsilon_{AD}}\right) = \left(\frac{2L_x L_y^2}{L^3}\right),$

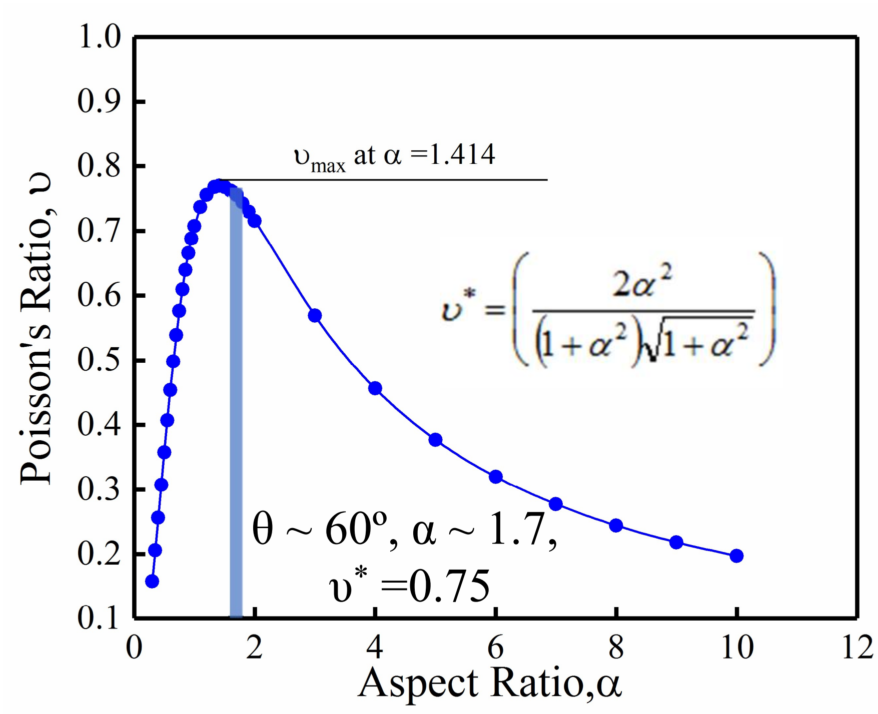


Analytical Model for Effective Poisson Ratio

Aspect ratio: $\alpha = {L_y}/{L_x}$

$$\upsilon^* = \left(\frac{2\alpha^2}{(1+\alpha^2)\sqrt{1+\alpha^2}} \right)$$

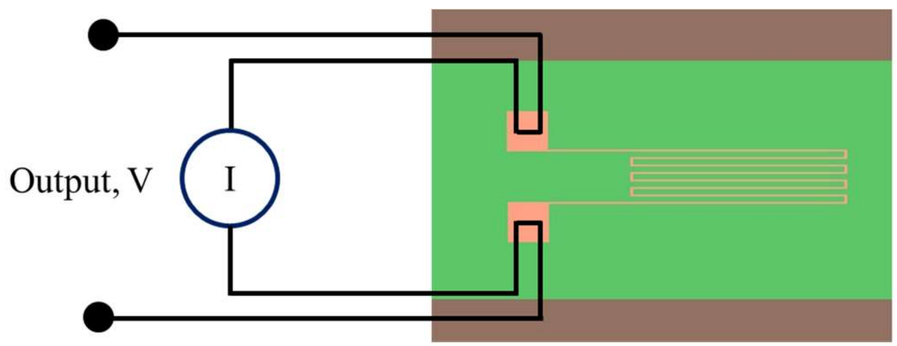




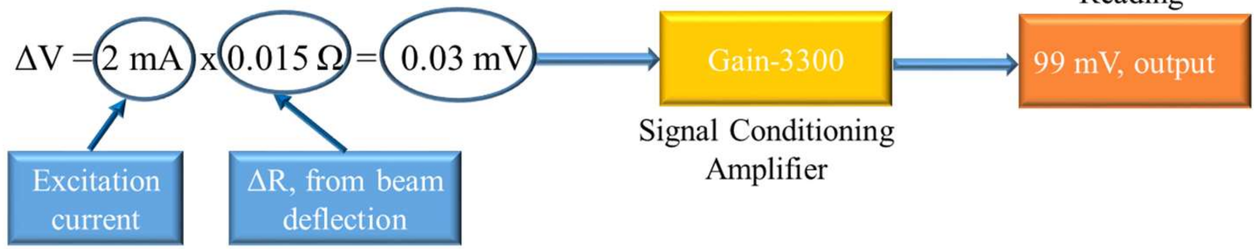
Porosity can result in higher Poisson ratio for 3-D printed films

High Temperature Sensor Measurement Overview

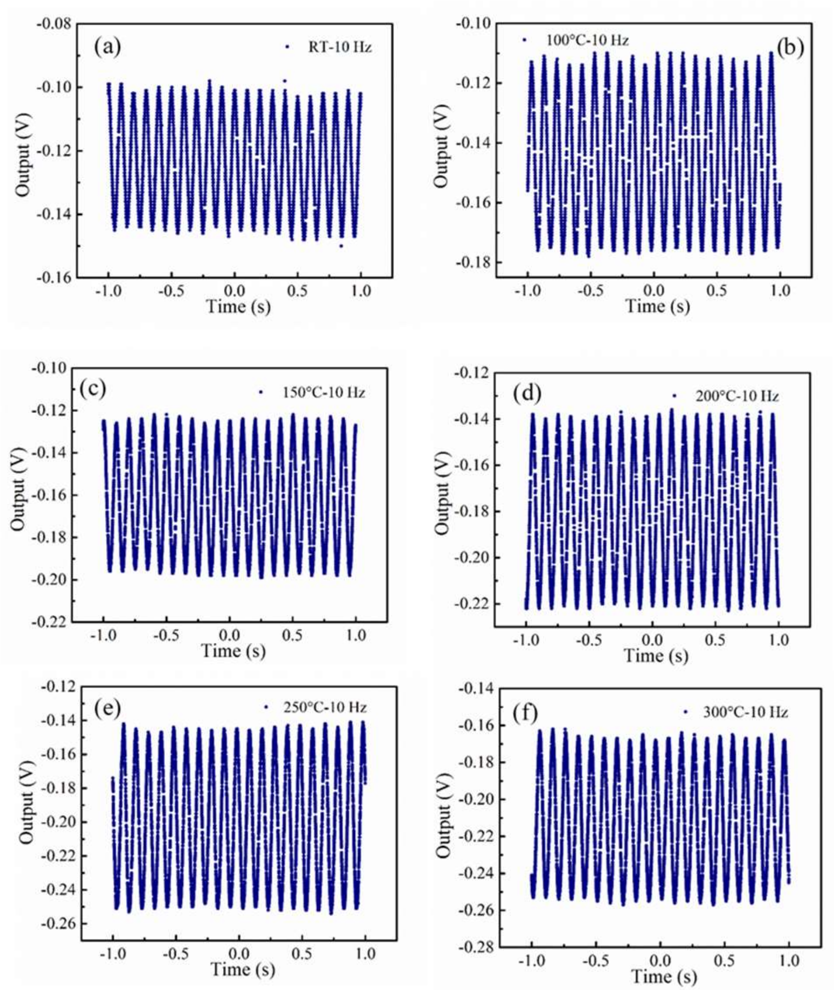
- Four-wire constant current measurement method was used for HT measurement

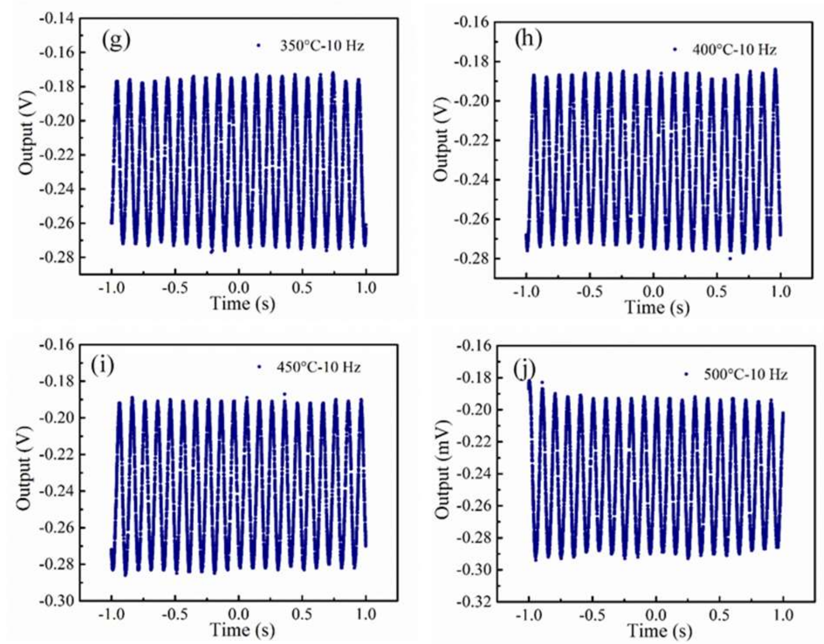


- $V = I \times R_g$
- $V = 2\ mA \times 5\ \Omega = 10\ mV$
- $\Delta V = I \times \Delta R = \Delta V\ mV$
- $\Delta V = 2\ mA \times 0.015\ \Omega = 0.03\ mV$



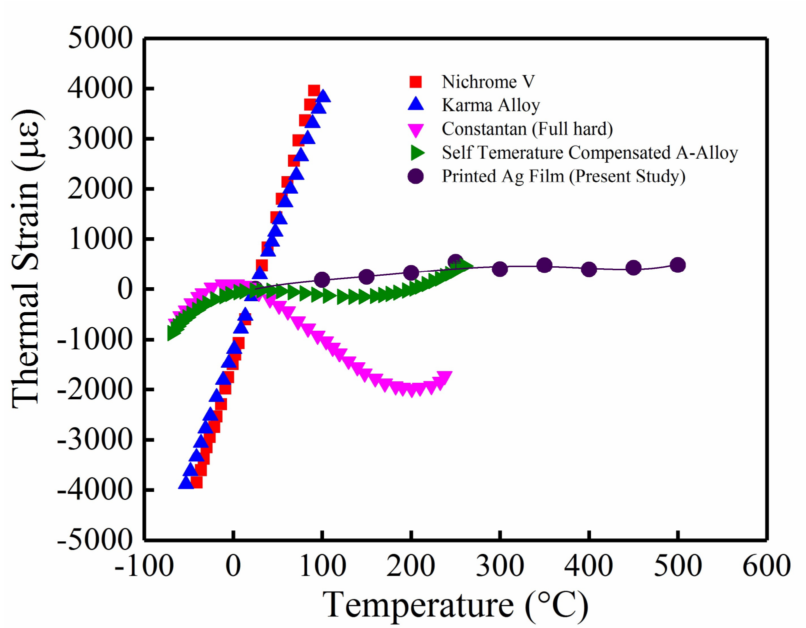
High Temperature Sensor Output As-measured through Oscilloscope





- Representative HT sensor output for a sensor with R-5.15 Ω
- 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

Ref 1: Strain Gage Thermal Output and Gage Factor Variation with Temperature; MICRO-MEASUREMENTS, A VPG Brand: Raleigh, NC, USA, 2014; pp 1-13.

Strain Sensor Work Caught Media Attention

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

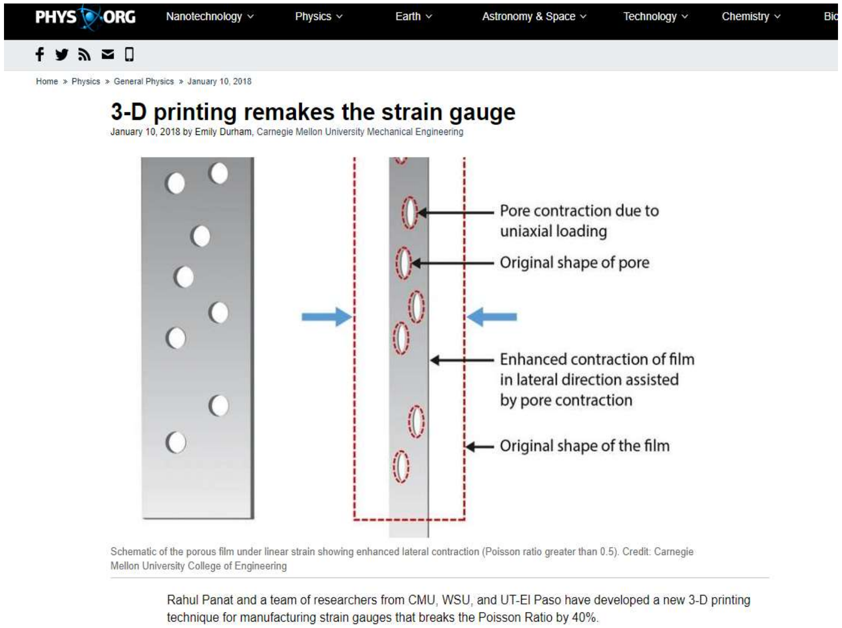
CMU News: 3-D printing remakes the strain gauge

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

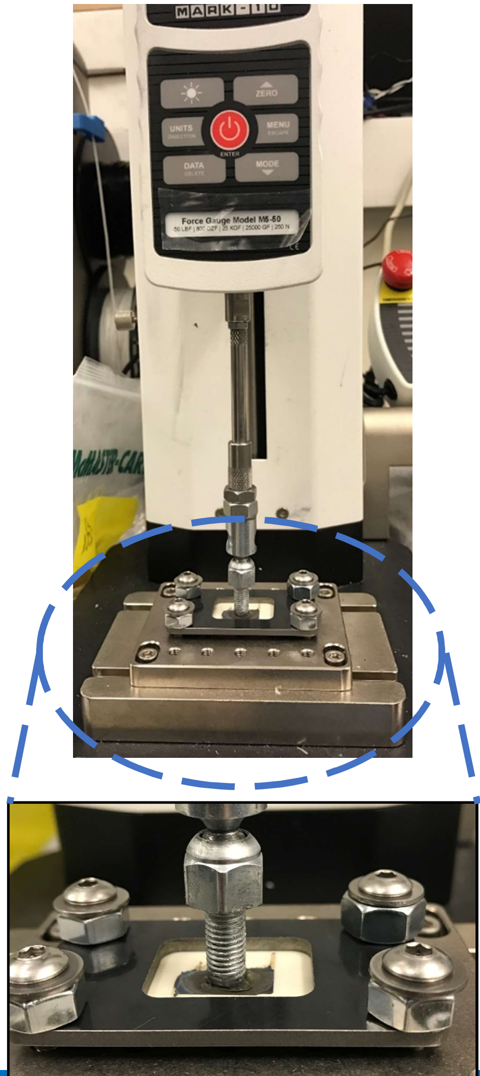
India today: New 3D printing method for more sensitive strain gauges

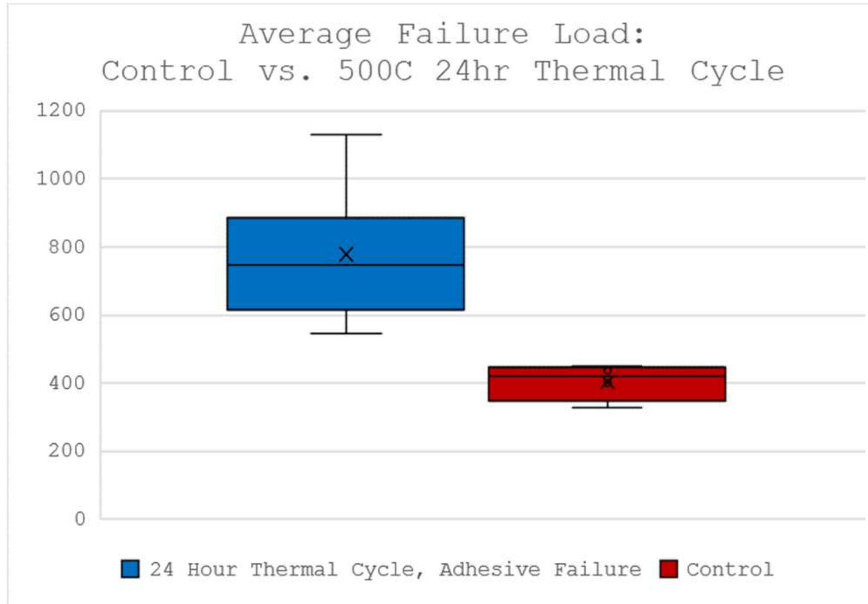
3dprintingprogress.com: New 3-D printing technique for manufacturing strain gauges



Task 3. Reliability Study of the Sensor

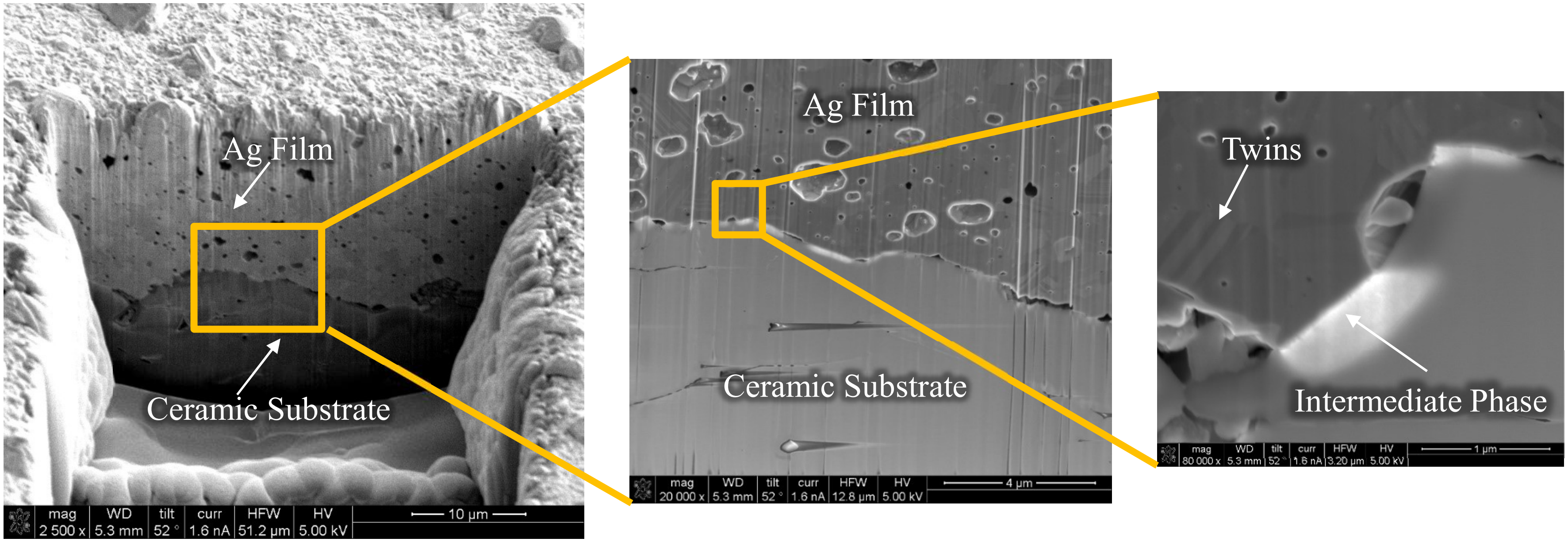
Work of Adhesion Test Setup





- 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

1. M. T. Rahman, J. McCloy, C. V. Ramana, and R. Panat, "Structure, electrical characteristics and high-temperature stability of aerosol jet printed silver nanoparticle films", ***Journal of Applied Physics, Vol. 120, Issue 7, pp. 075305-1 to 11, 2016***. **(Impact Factor: 2.1)**
2. M. T. Rahman, Kathryn Mireles, Juan J. Gomez Chavez, Pui Ching Wo, José Marcial, M. R. Kessler, John McCloy, C. V. Ramana, and Rahul Panat, "High Temperature Physical and Chemical Stability and Oxidation Reaction Kinetics of Ni–Cr Nanoparticles", ***J. Phys. Chem. C (ACS), 2017, 121 (7), pp 4018–4028***. **(Impact Factor: 4.5)**
3. M. T. Rahman, Juan J. Gomez Chavez, C. V. Ramana, and Rahul Panat, "3D Printed High Performance Sensors for High Temperature Applications", ***Journal of Applied Physics, Vol. 123, pp. 024501, 2018***. **(Impact Factor: 2.1)**

Additional journal papers are in preparation

Conference Presentations:

1. Md Taibur Rahman, Amy Wo, C. V. Ramana, Rahul Panat, "High Temperature Mechanical and Electrical Properties of Additively Manufactured Metal Nanoparticle Films", TMS, Nashville TN (2016)
2. Md Taibur Rahman, C. V. Ramana, Rahul Panat, "High Temperature Mechanical and Electrical Properties of Additively Manufactured Metal Nanoparticle Films", ICMCTF, San Diego CA (2016)
3. Md Taibur Rahman, C. V. Ramana, others, R. Panat, "Printed Nanoparticle Films for Electronic Applications", TMS, San Diego CA (2017)
4. Md Taibur Rahman, C. V. Ramana, others, R. Panat, "High Performance Sensors and Antennas by 2D and 3D Printing of Nanoparticles", TMS, Phoenix, AZ, Diego 2018)

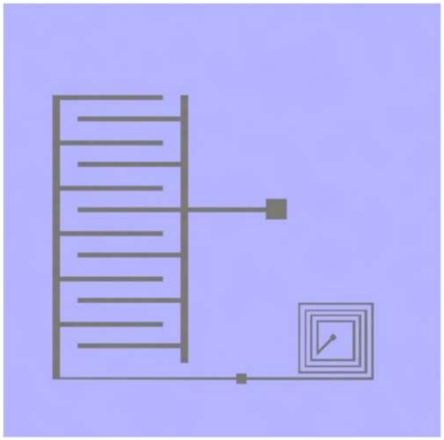
Acknowledgement

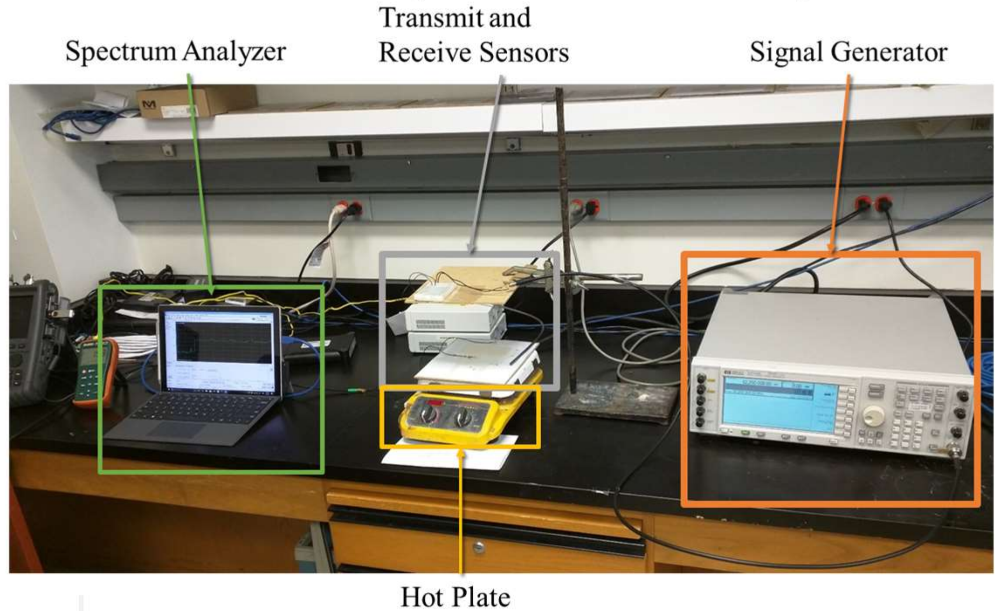
- DOE/NETL through grant # DE-FE0026170
- Barbara Carney
- NextManufacturing Center, CMU

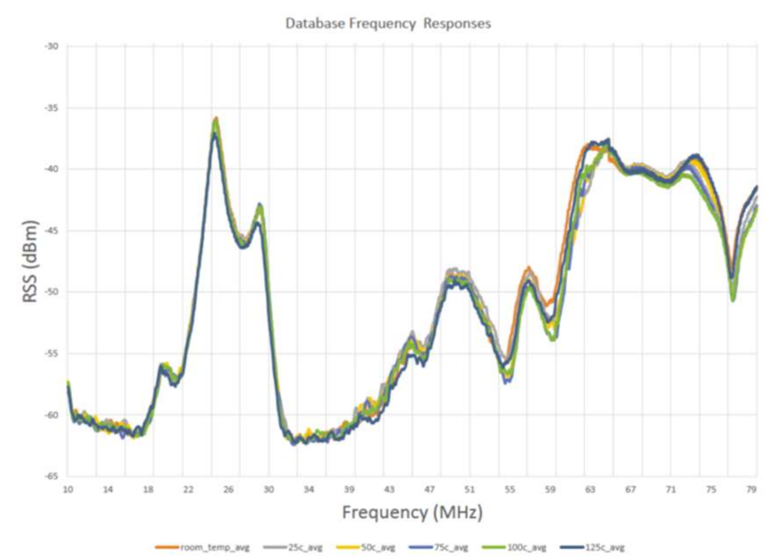
2018 Deliverables

- Completion of reliability work
- Fabrication of workable antenna at high temperatures
- Wireless system design and fabrication

Wireless Antenna Work: (with WVU)



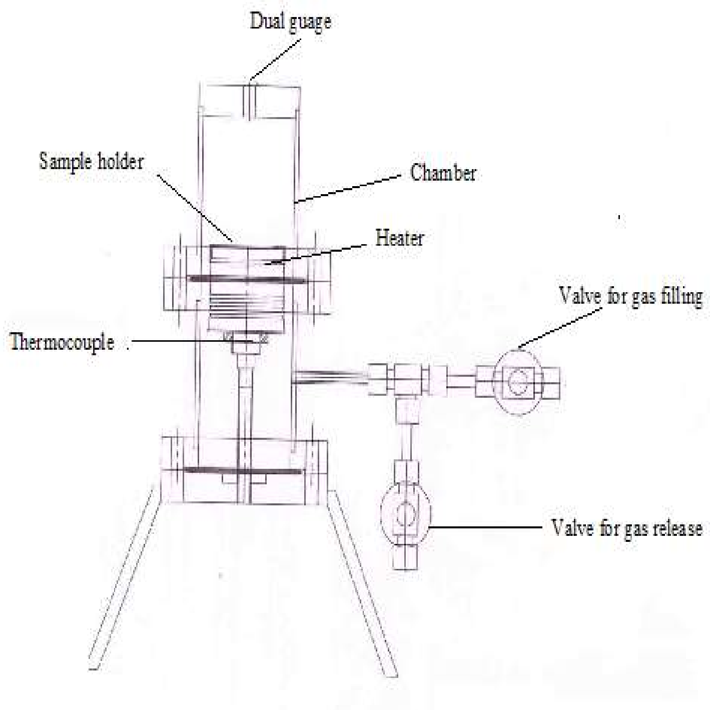


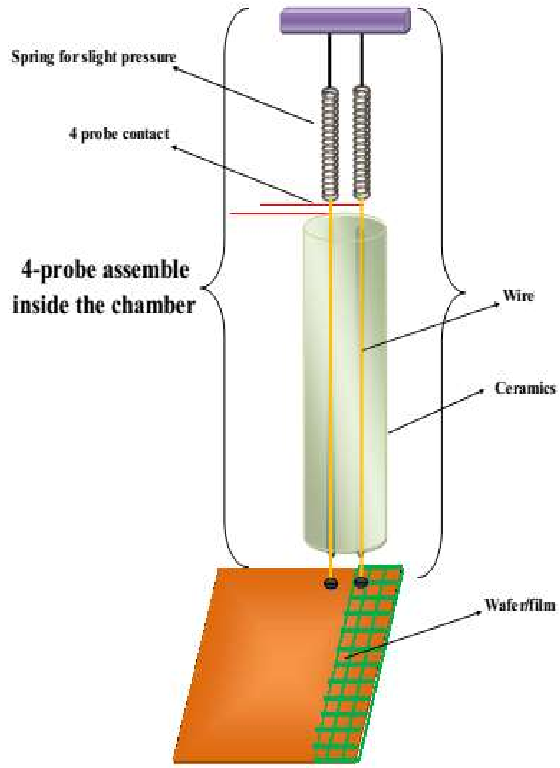


- Encouraging preliminary results, where antennas could ‘talk with each other
- Change in response as a function of temperature
- Further work will involve EM simulations and transmission…….

High Temperature Pressure Sensor Testing Protocols -UTEP

Pressure Sensor testing





Thank You