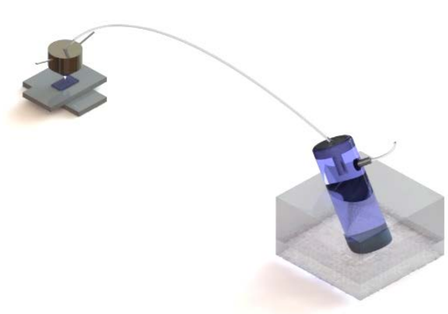
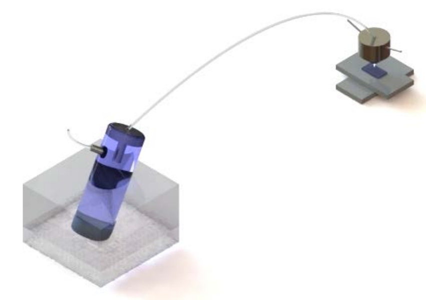
Low-Cost, Efficient and Durable High Temperature Wireless Sensors by Direct Write Additive Manufacturing for Application in Fossil Energy Systems





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Agenda

- Introduction and Background
  - Team
  - Project Goals and Objectives
  - Tasks and Timelines

- Research Accomplishments
  - Manufacturing: Additive Printing Technique
  - Material Selection and Sensor Characterization
  - Single Sensor Printing/Testing and High Temperature Test Set-up
  - Sensor Reliability
  - Student Training and Research Dissemination

- Summary of Research

- Future Direction

The Team

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Project Co-PI

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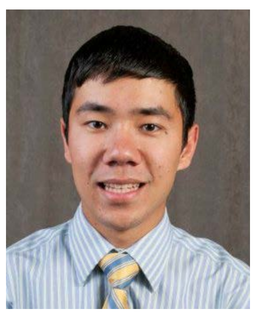

Russell Moser,
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M. Dessimie
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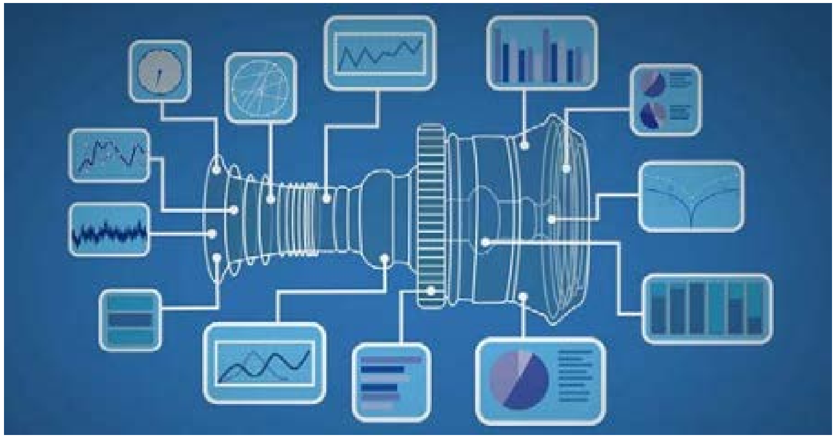
PI Prof. Rahul Panat moved from WSU to CMU last year (fall 2017)

Background

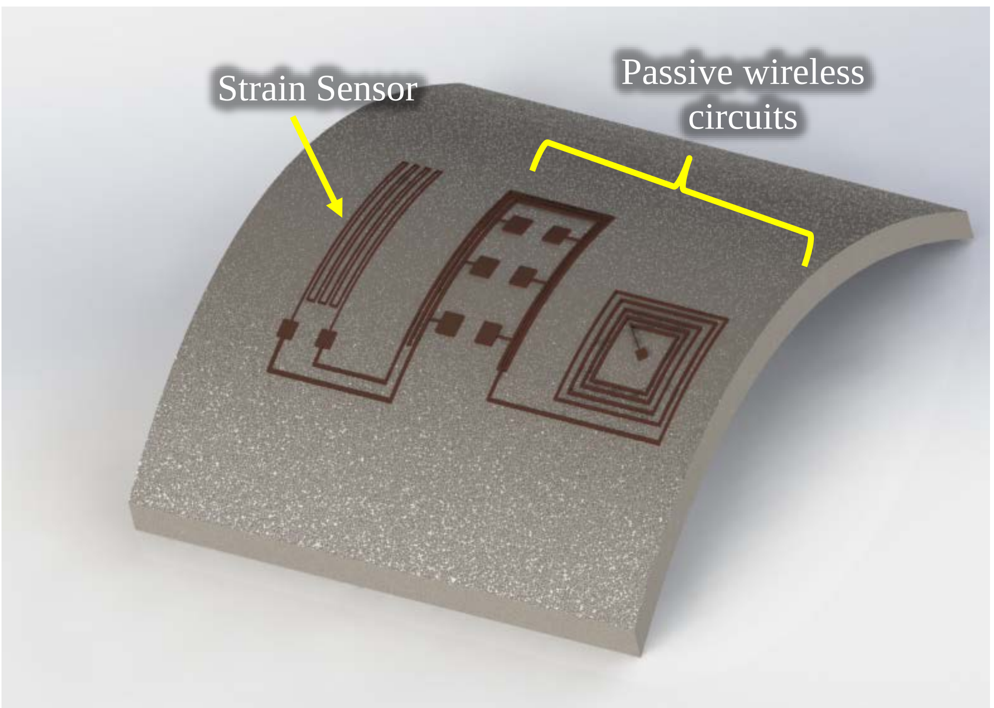


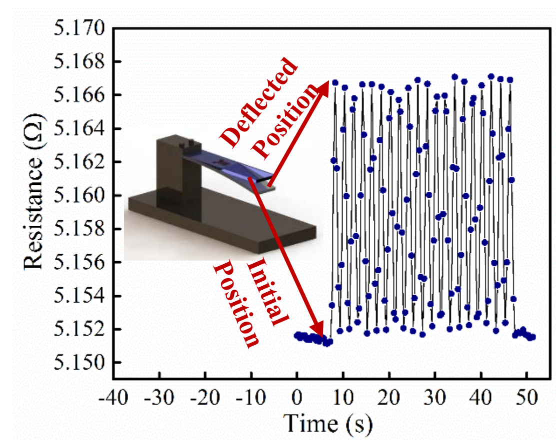
200 sensors across the turbine generate 300 data points per second

- 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

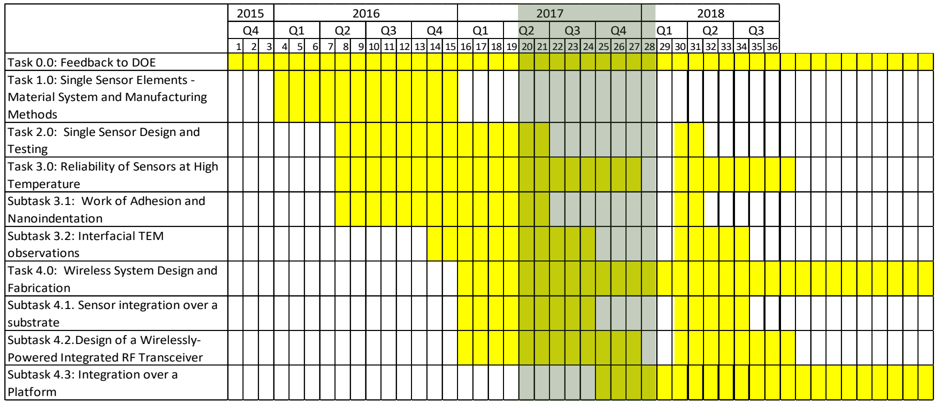
- Goals:
  - Demonstrate the feasibility of low-cost aerosol jet manufacturing for Fossil Energy (FE) systems and develop materials, next-generation sensors that can reliably operate at high temperatures (>350 ^{o}C up to 500 ^{o}C) with wireless transmission (**Passive electronics**)





Tasks and Timelines

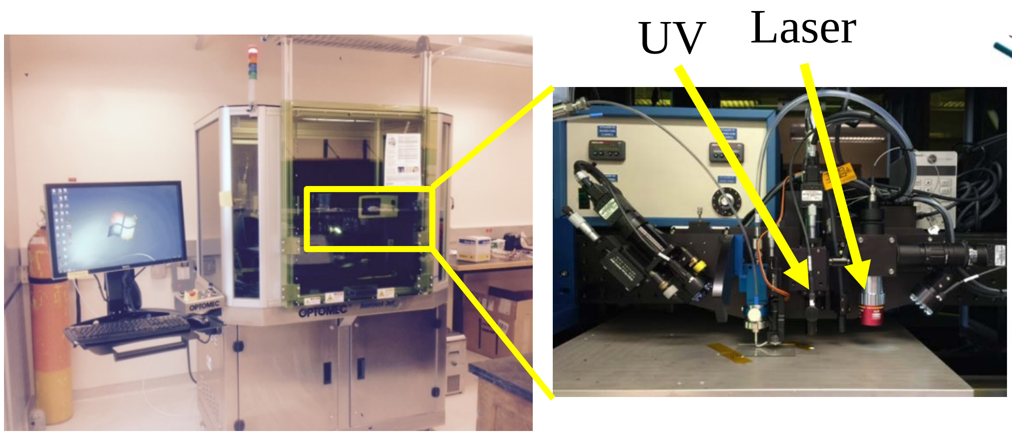
- Key Milestones:
  - Method Development and Sensor Material Characterizations (Impedance analysis, Oxidation study, Micro/Nano structure study): **Done**
  - Primary Material Selection: **Done**
  - Single Sensor Design, Fabrication and Testing: **Done**
  - Reliability Tests (adhesion, nanoindentation,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

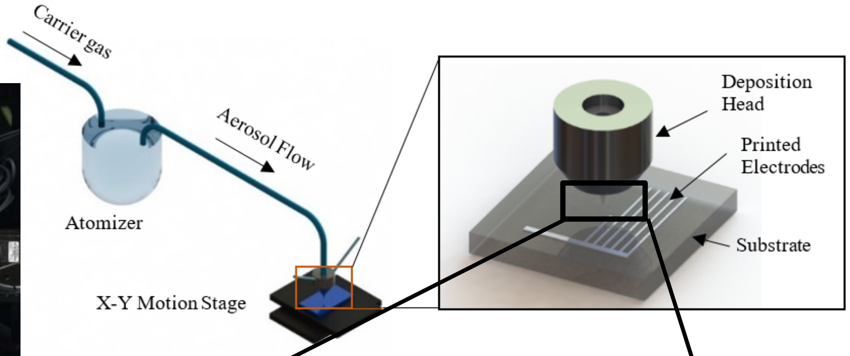


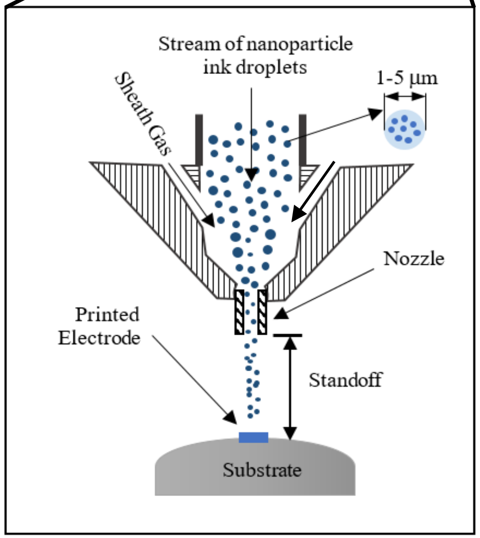
Task 1: Manufacturing Method and Material System

- 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

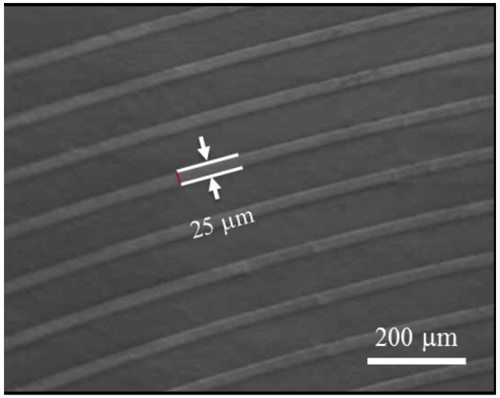


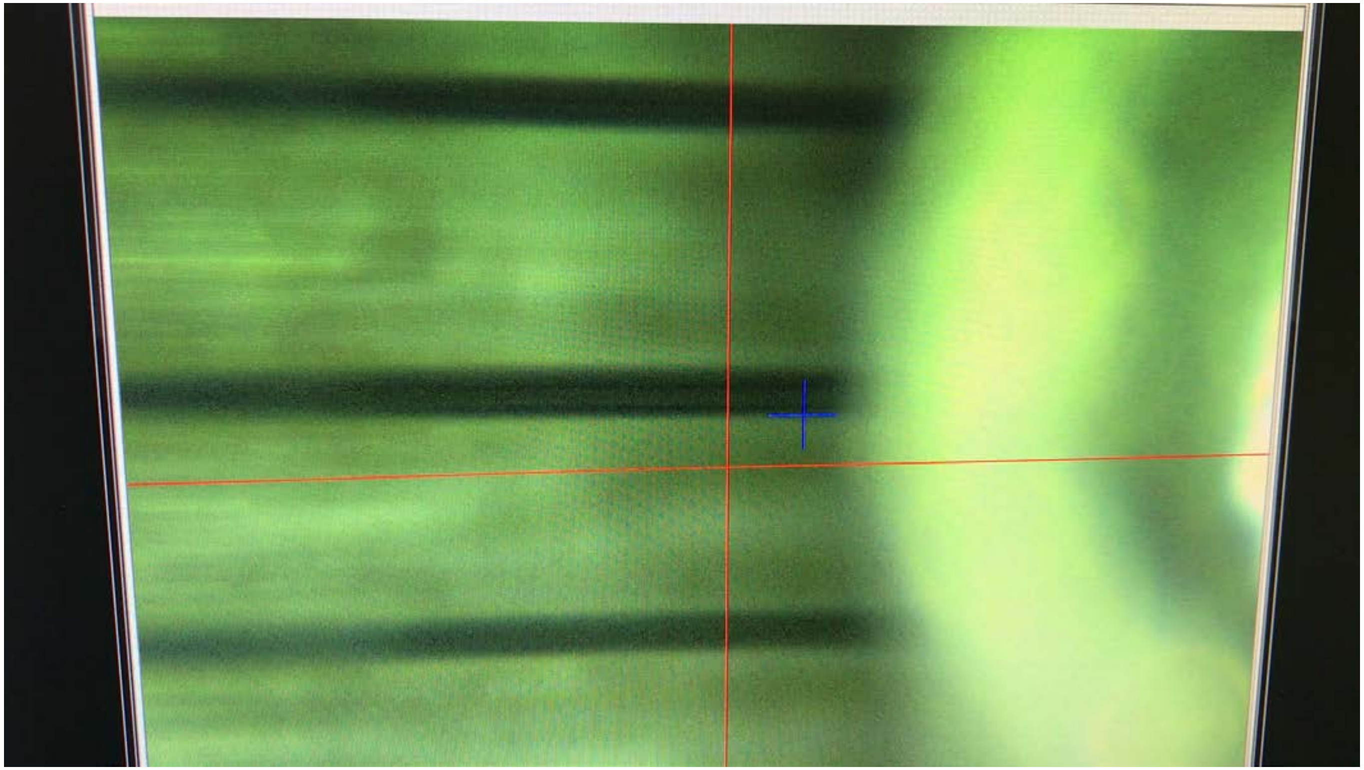




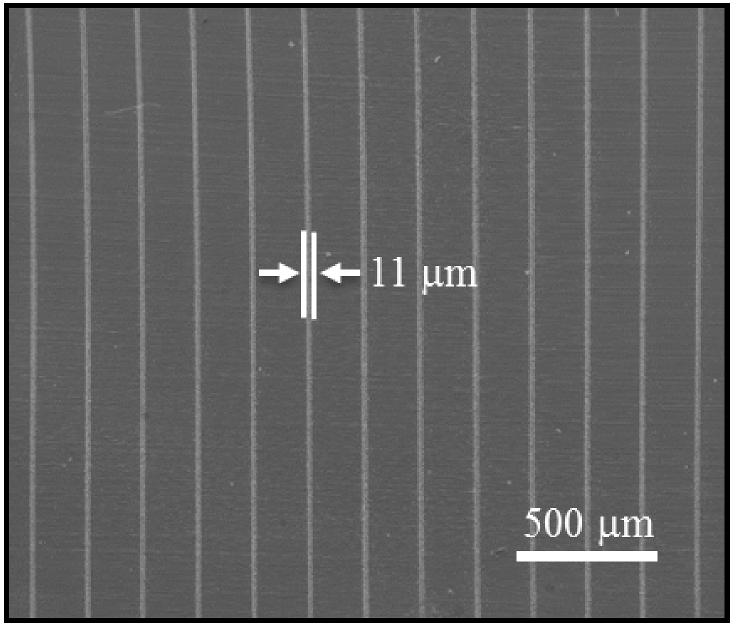
- ❑ Operates by creating aerosol mist of 1-5 µm droplets, each containing nanoparticles
  - Stand off height about 5mm
  - No restriction on substrate type
  - Minimum feature size down to 10 µm
  - Printing accuracy of ± 2 µm stdev
- ❑ Clog resistant nozzles
- ❑ Ink viscosity up to 1000 cP

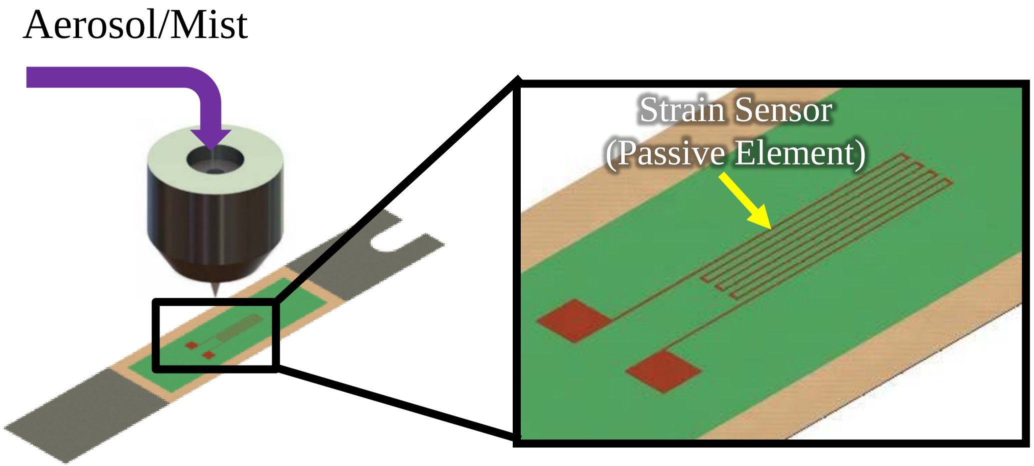
Aerosol-Jet Printing Video





High Resolution Printing





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

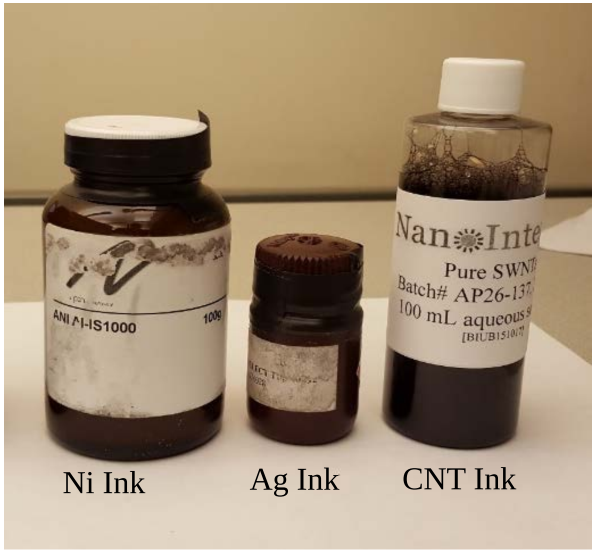
Material Systems

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

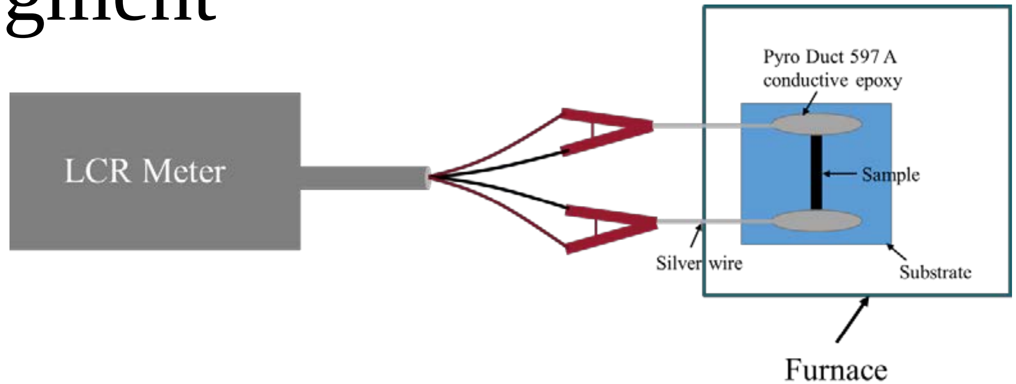
- Printed Dispersed Carbon Nanotubes (CNTs) films
  - Viscosity: 1cP
  - Diameter : 100 nm

- 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



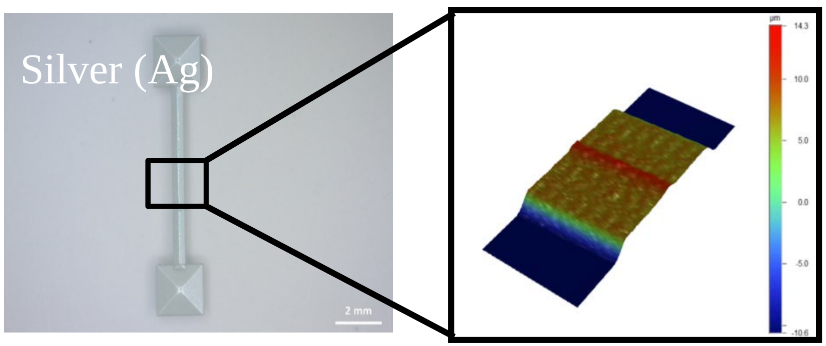
Electrical Characterization on Sensor Segment

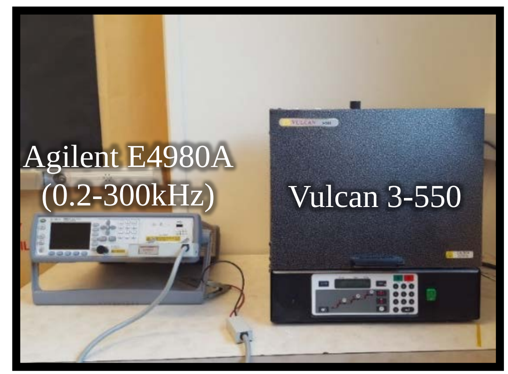


Impedance Spectroscopy

$Z = R + jX$

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



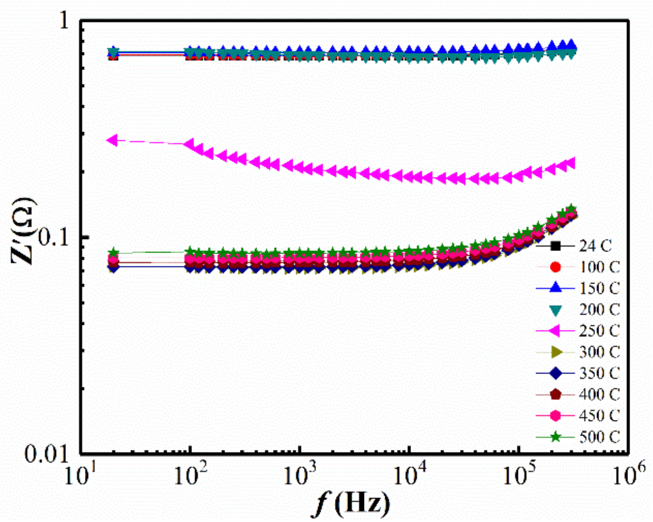


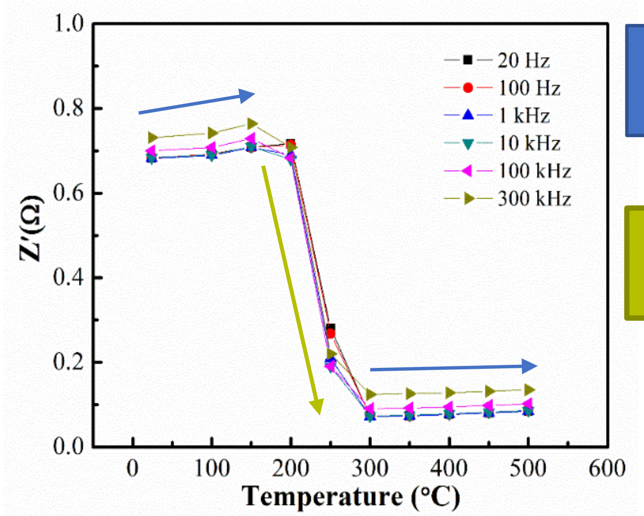
- ❑ Target is to evaluate temperature and frequency dependent impedance/electrical property change on sensor segment (i.e. passive element)

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

Impedance Spectroscopic Characterization of Ag

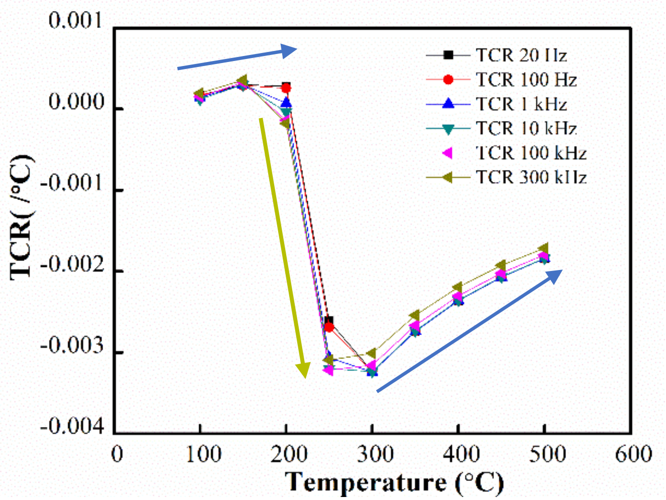
❑ Sintered for 30 min at 200 °C





Increase in Z' up to 150 °C

Z' drops beyond 150 °C

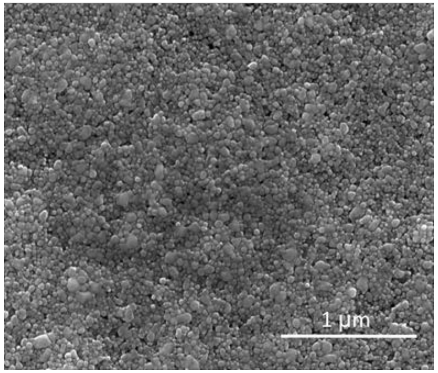


TCR for 24-150°: 0.000302/°C (at 20 Hz)

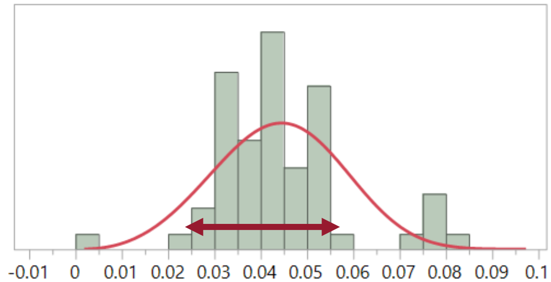
TCR for 150-300° C: -0.0059 /°C, at 20 Hz

Unstable impedance behavior

Microstructure Analysis of a Post Impedance Sample

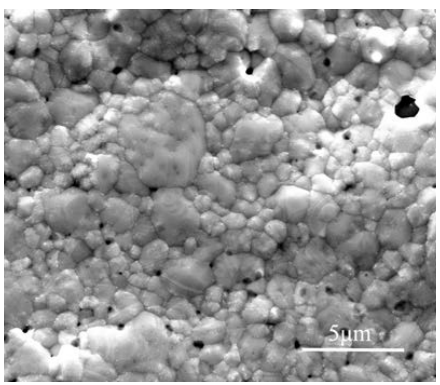


As-sintered

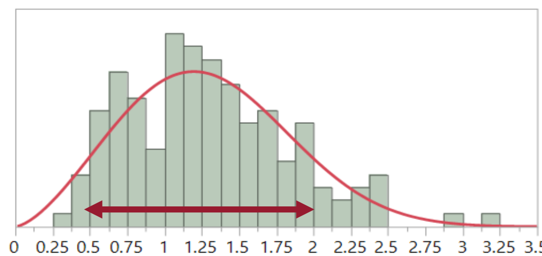


Grain Size Distribution (µm)

D_{avg} : 40 nm



After impedance test



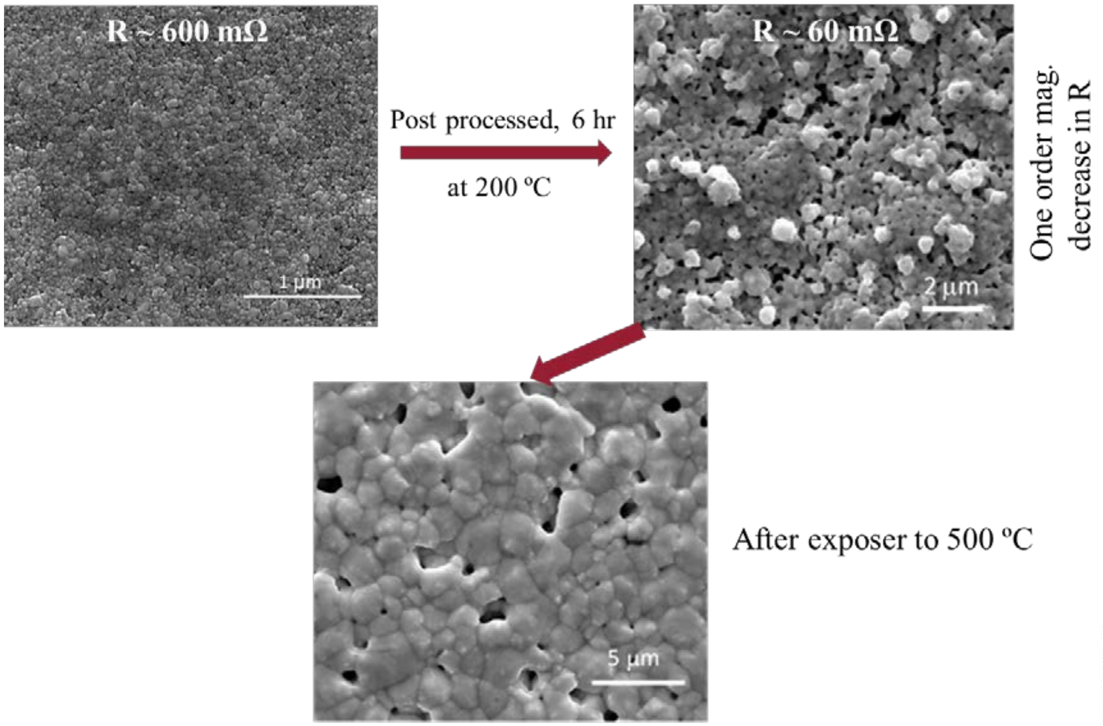
Grain Size Distribution (µm)

D_{avg} : 1 µm

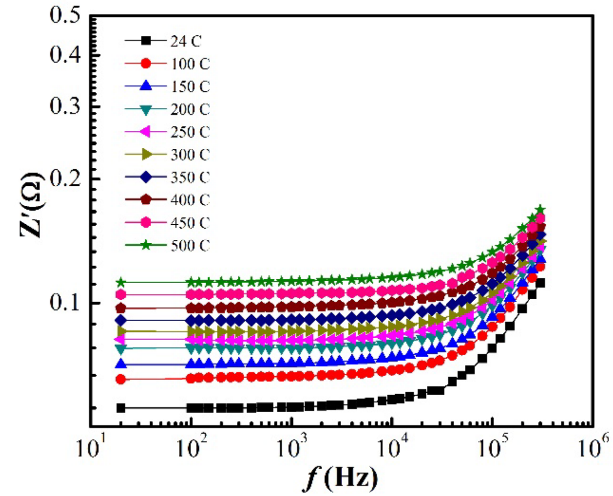
Increase in grain size

Additional sintering during impedance testing

Impedance Spectroscopy: Effect of long sintering time

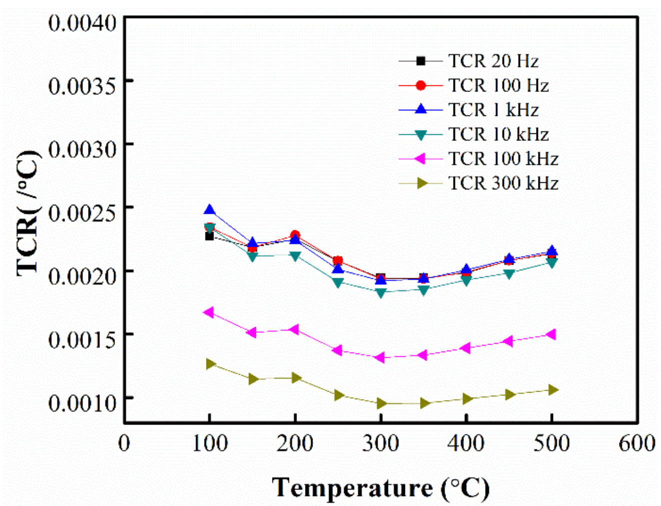


Sintered for 30 min at 200 ºC, post processed 6 hr at 200 ºC

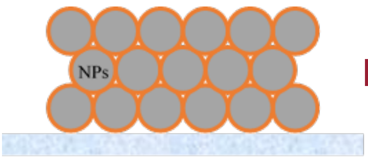


We observed instability in the impedance data. The instability was traced to grain growth, which increases conductivity

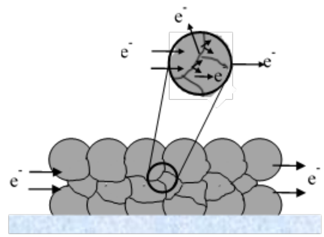
Issue solved by increasing the sintering time at the beginning of the sensor test to stabilize (increase) the grain size



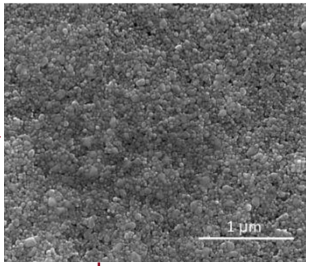
Impedance Spectroscopy: Effect of long sintering time

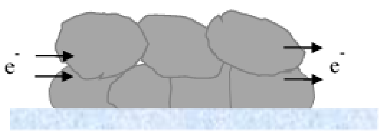


As Printed

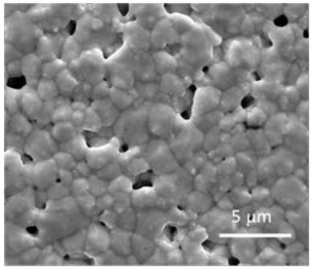


Sintered at 200 °C for 30 min (Higher grain boundary contribution to resistivity)





Heated to 500 °C (Grain growth and lower grain boundary contribution to resistivity)



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

GB width (1-10nm)

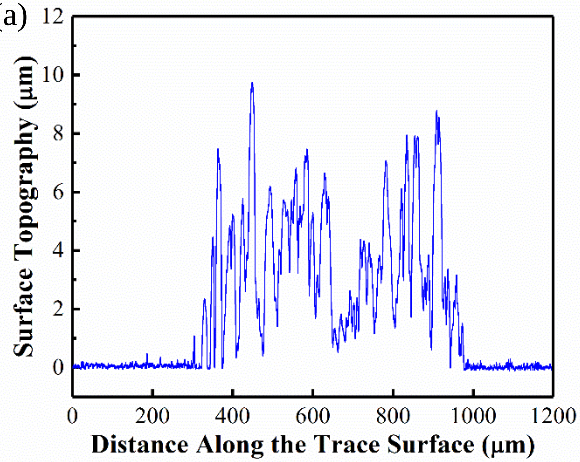
Grain size

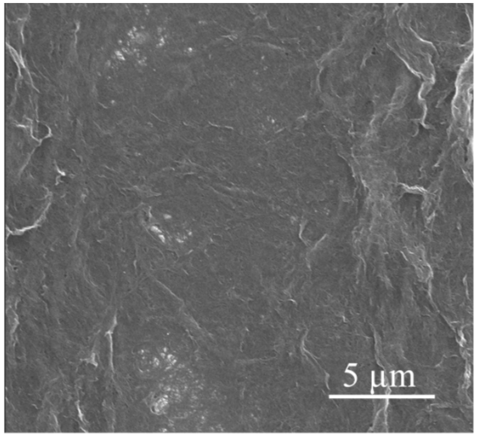
Bulk resistivity

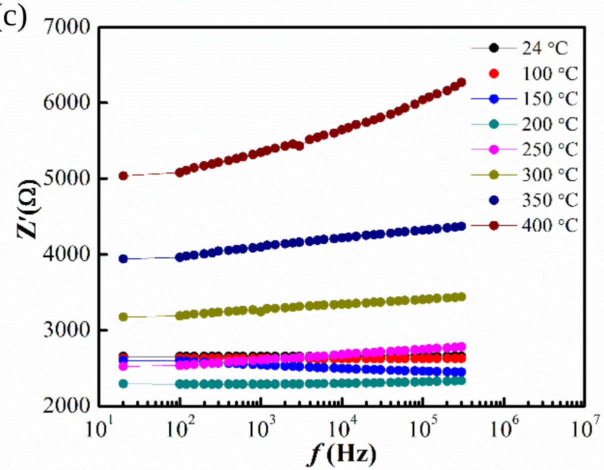
ρ_{gb} for silver $\sim 10^{-7}/10^{-8}\ \Omega$-cm

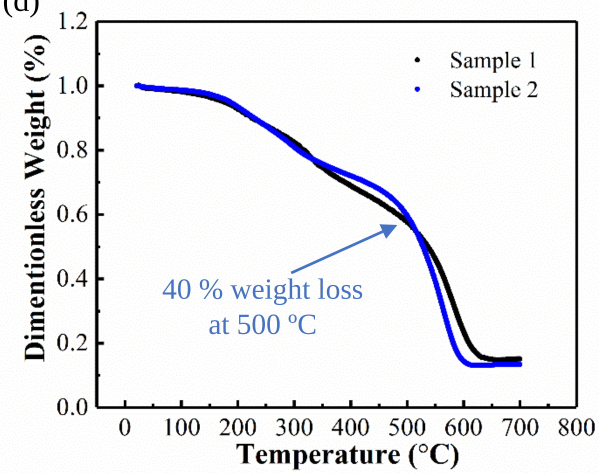
High Temperature Stability of CNTs

- 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

(a)


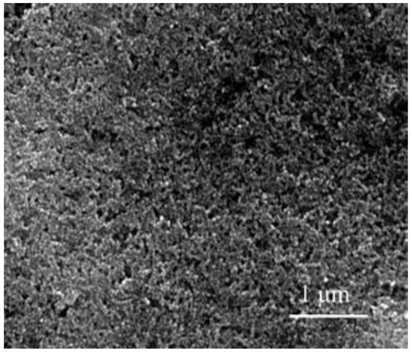
(b)


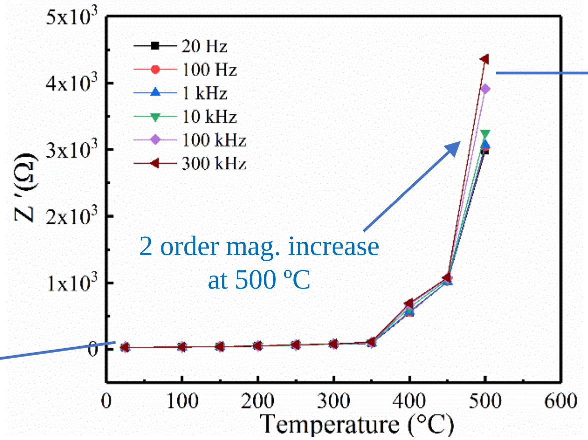
(c)


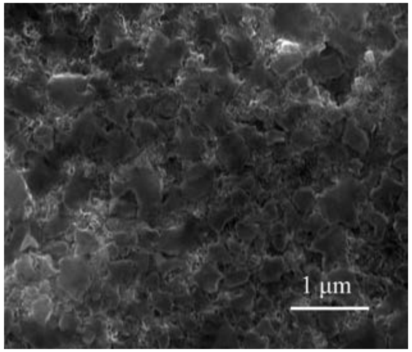
(d)


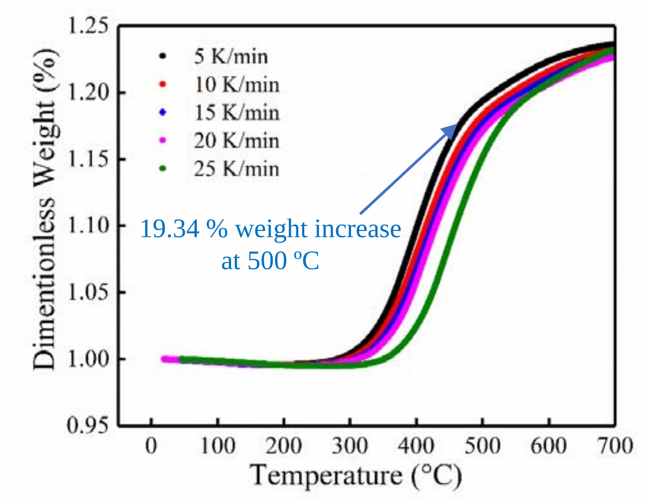
Based on this study we conclude that use of CNT films is not feasible at high temperatures (above 200-300 °C)

High Temperature Behavior of Ni NP Films







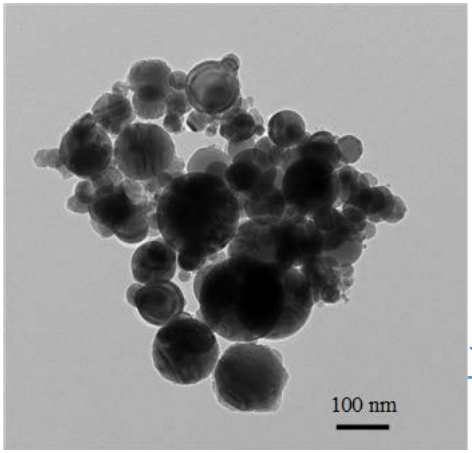


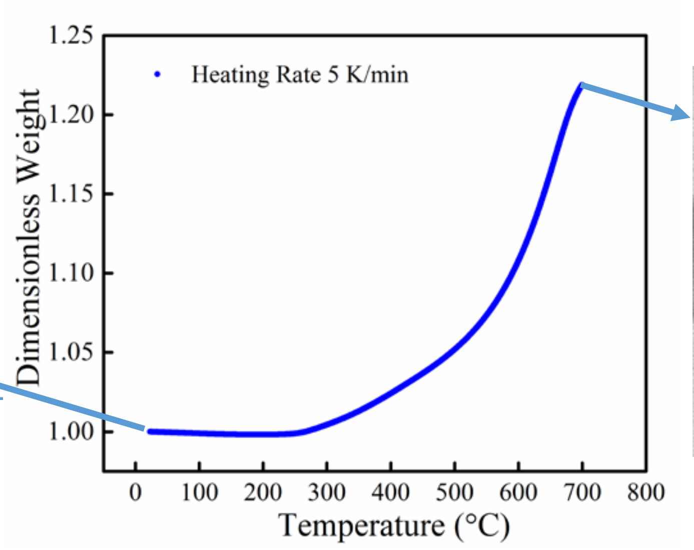
- ❑ Impedance increases by 2 order of mag. compared to RT beyond 350 ºC indicates an onset of oxidation
- ❑ TGA analysis shows high weight gain about 19.34 % at 500 ºC indicating heavy oxidation of Ni NPs

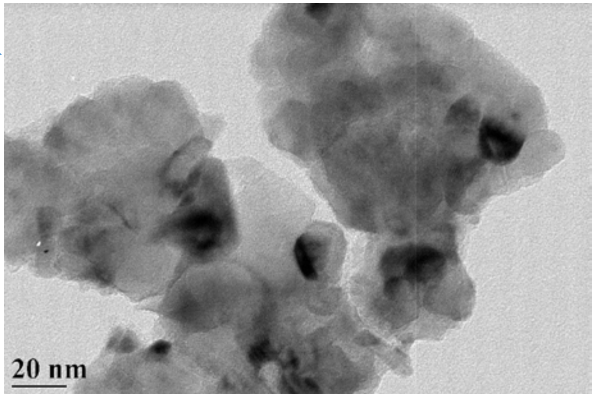
Use of Nickel NP films challenging beyond 300 °C due to oxidation

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







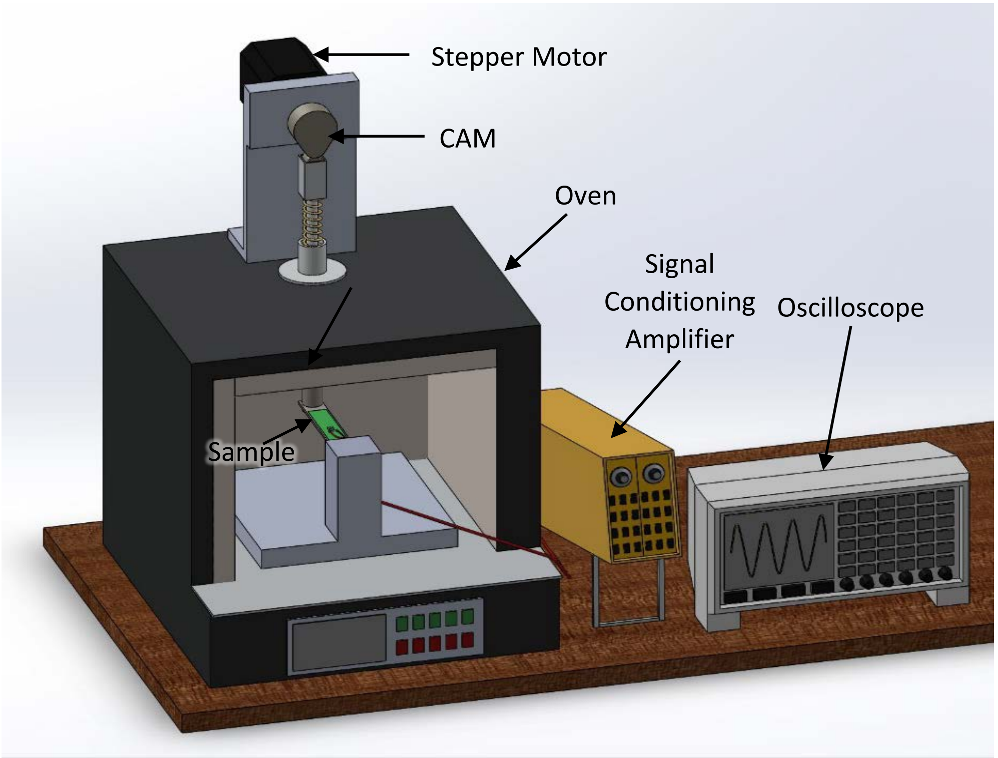
Materials Characterization

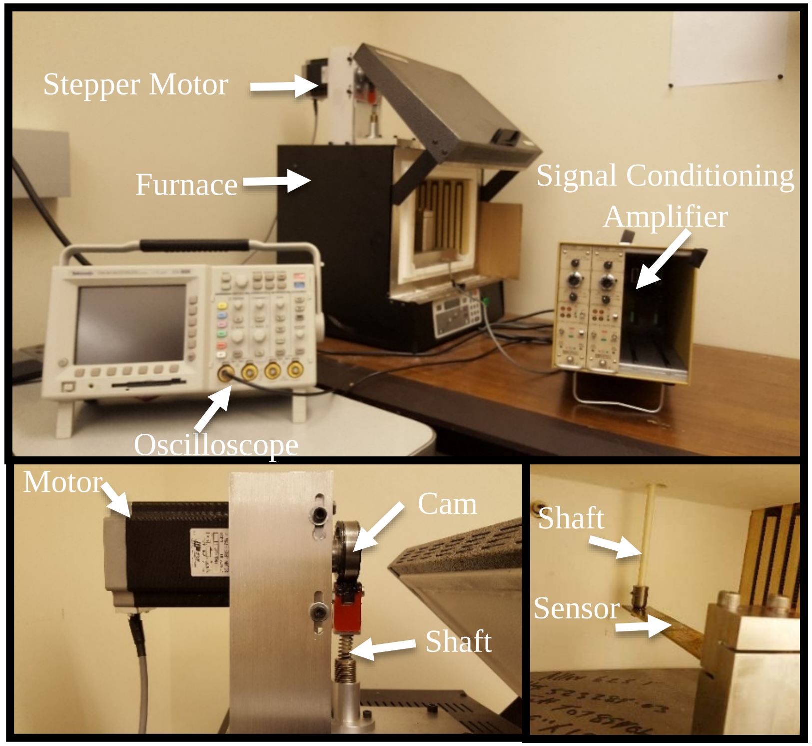
- ❑ Ag undergoes no/minimal oxidation up to 500 ºC
- ❑ Use of CNTs is challenging at above 200 ºC-300 ºC
- ❑ Ni is a suitable candidate for sensors application up to 350 ºC
- ❑ NiCr alloy can be useful in the temperature of RT-450 ºC

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

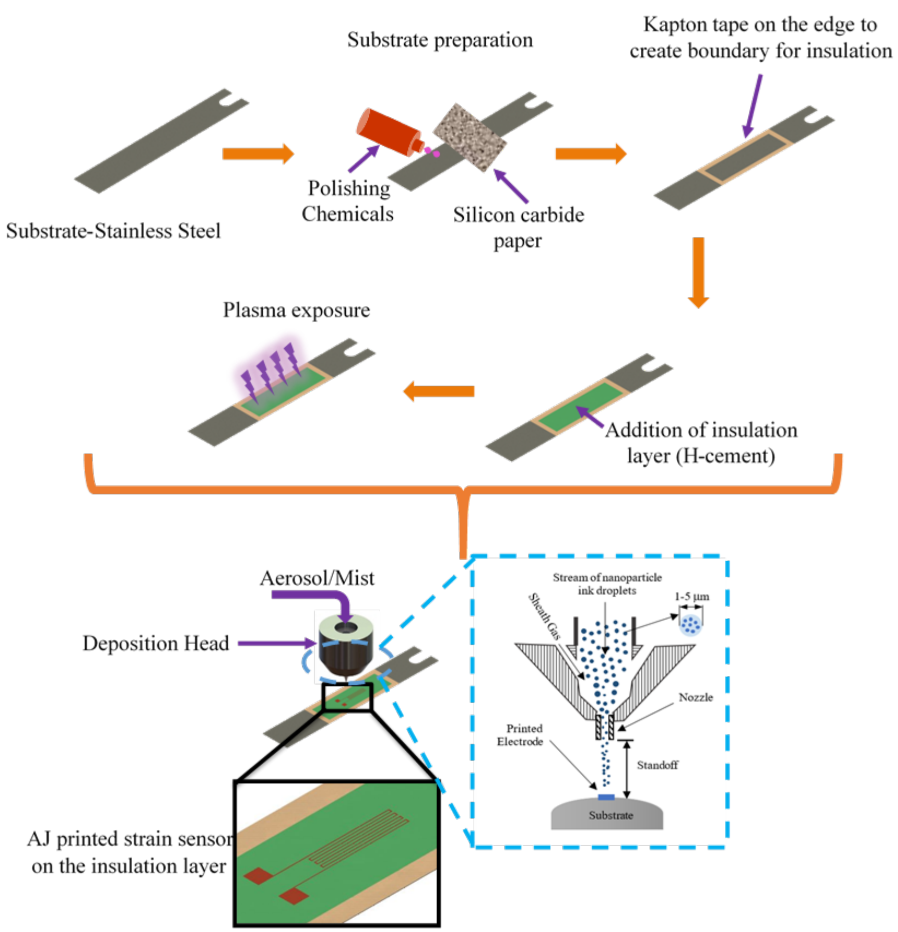


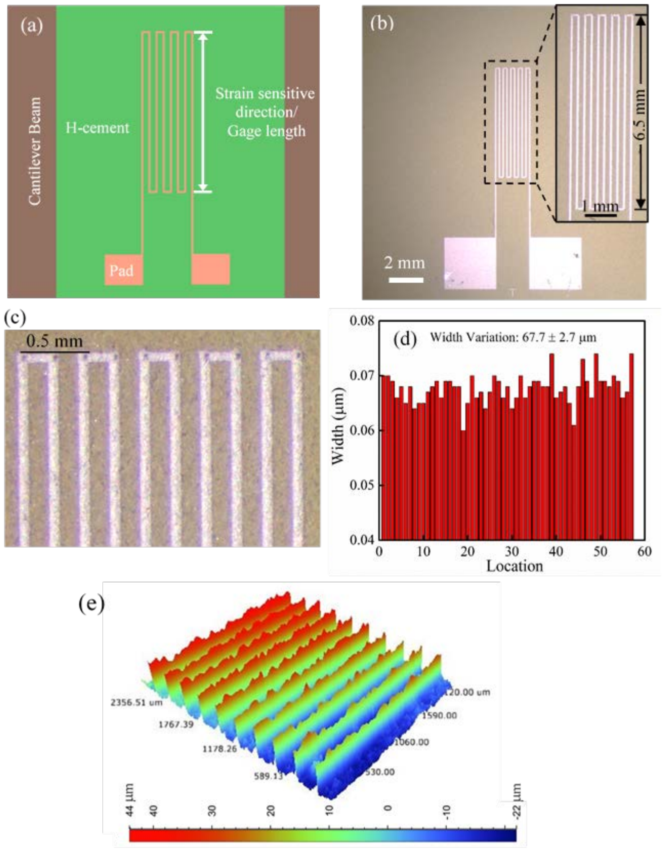


Actual Strain Sensor Test Set up

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

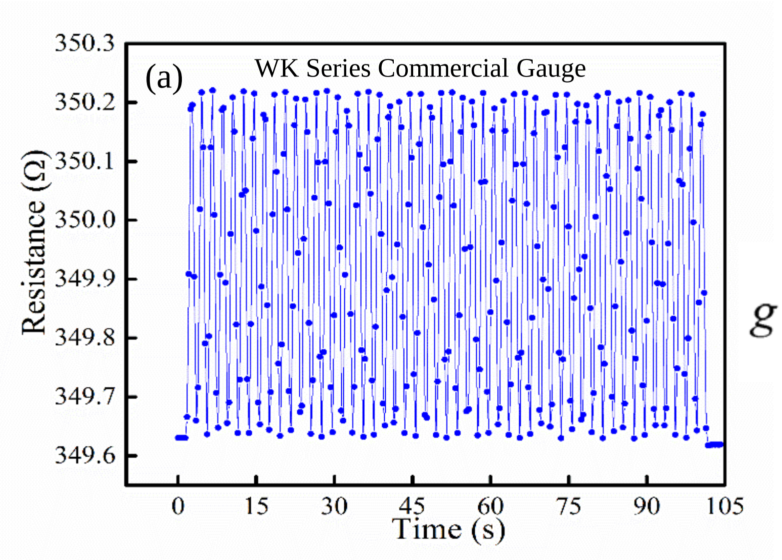
Sensor Fabrication



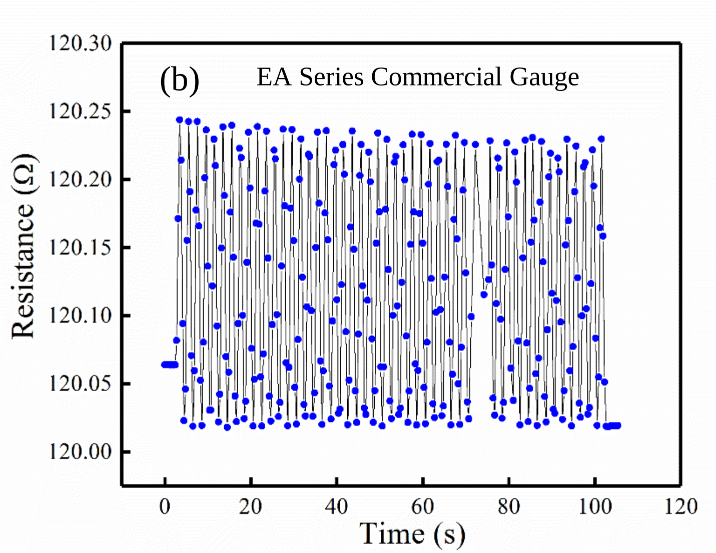


We have developed a robust protocol for sensor design and fabrication which give rise to repeated properties

Calibration of the High Temperature Sensing Test Set up



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



- 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: 350 Ω
- Given GF: 2.0 (from manufacturer)
- Measured strain in the beam: 862 με using the following equation:

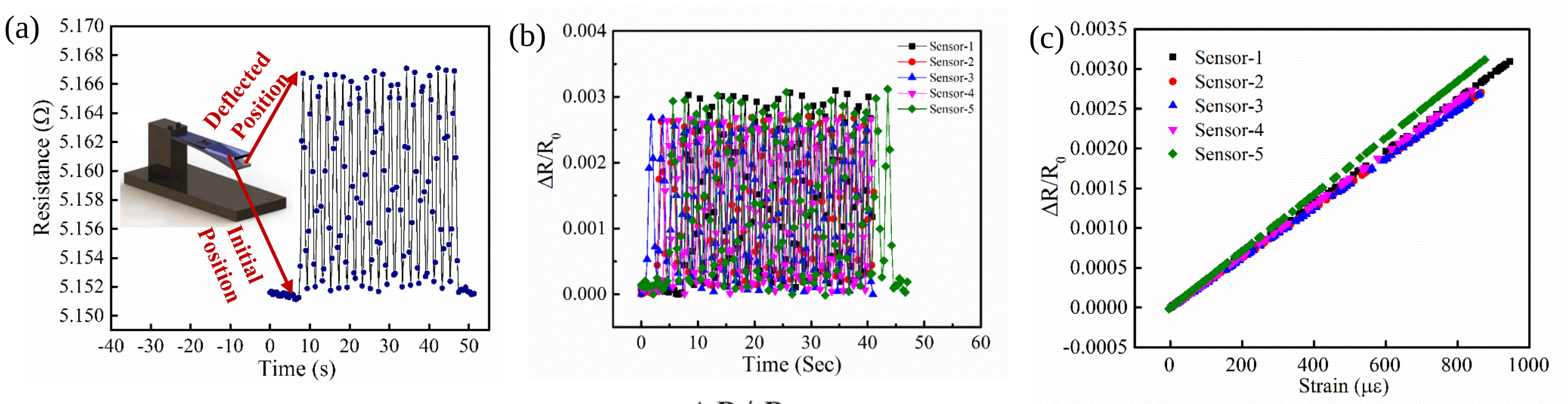
- 4-wire probing was used to measure the R change using $7_{1/2}$-digit precision multimeter
- Sensor was installed in a SS substrate
- Resistance of the Sensor: 120 Ω
- Given GF: 2.0 (from manufacturer)
- Measured strain in the beam: 878 με using the following equation:

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

Video

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

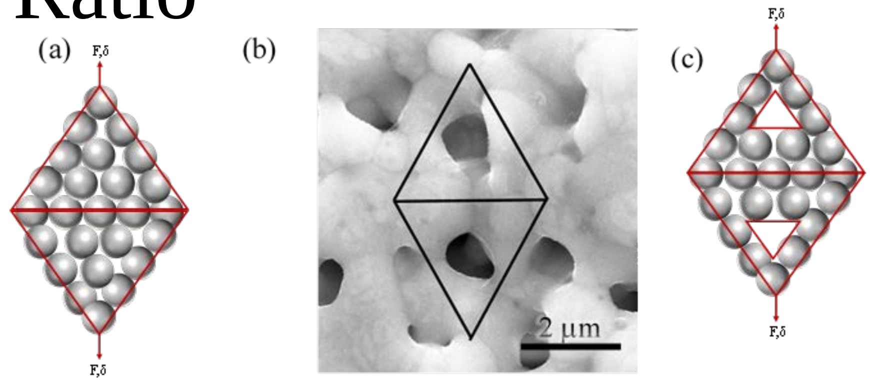
- GF: 3.15 ± 0.086,
- Commercial sensor has a GF of 2.0

- 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,



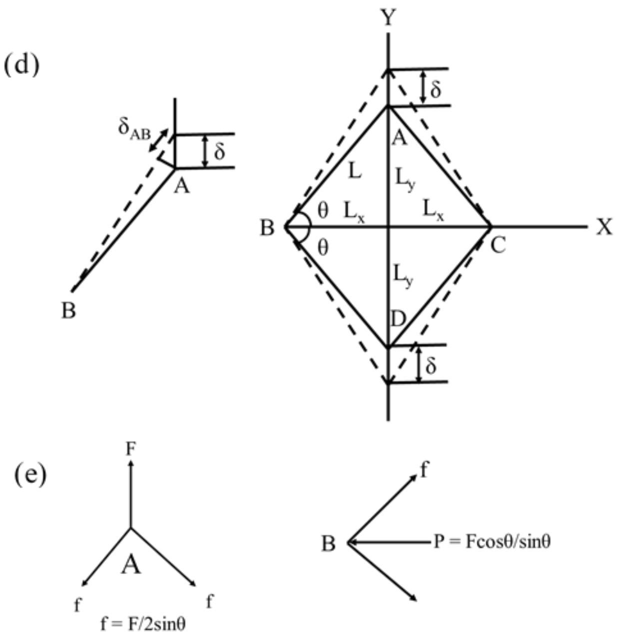
$$\delta_{AB} = \delta \sin\theta$$

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

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

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

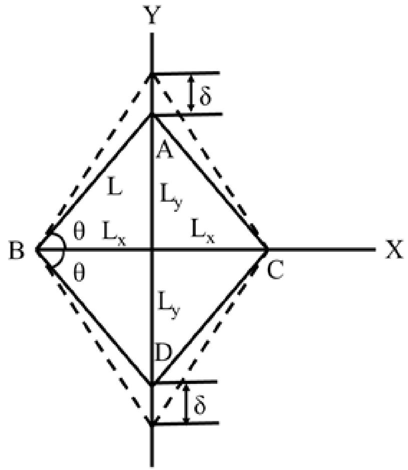
$$\delta_{BC} = \frac{P\left(2 \times L_x\right)}{AE} = \left(\frac{2F}{AE}\right) \times \left(\frac{L_x^2}{L_y}\right)$$

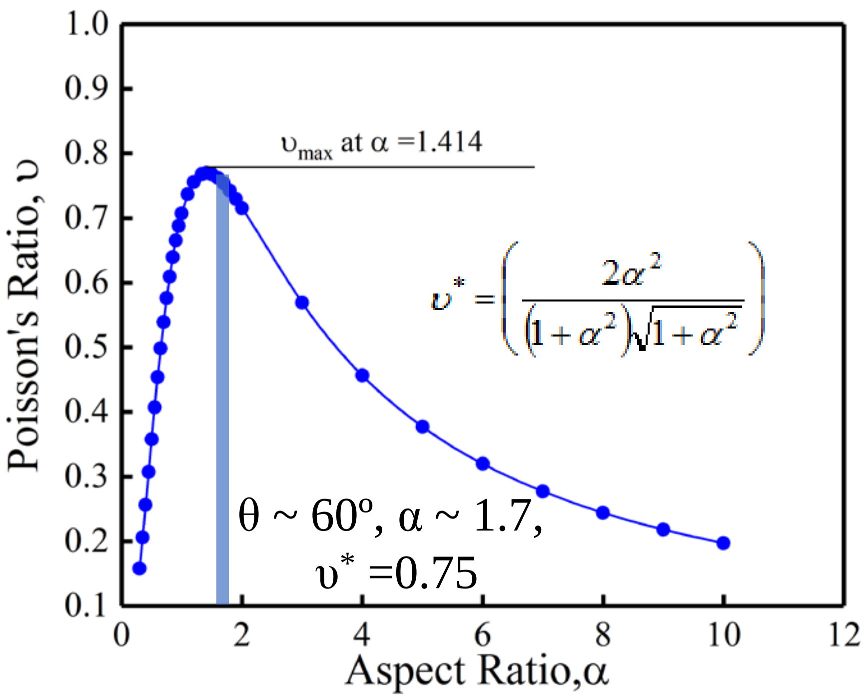


Analytical Model for Effective Poisson Ratio

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

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

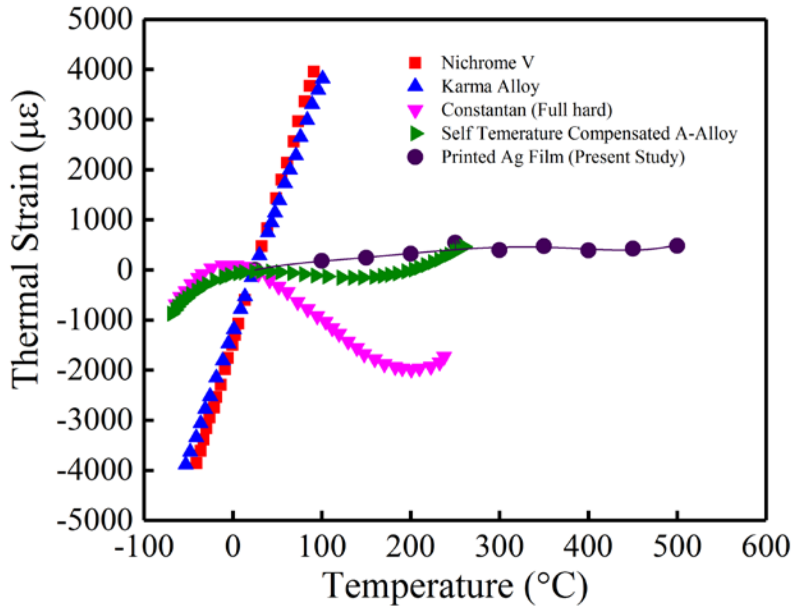




Porosity can result in higher Poisson ratio for 3-D printed films

Modelling was performed by Prof. H. Zbib, WSU

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:

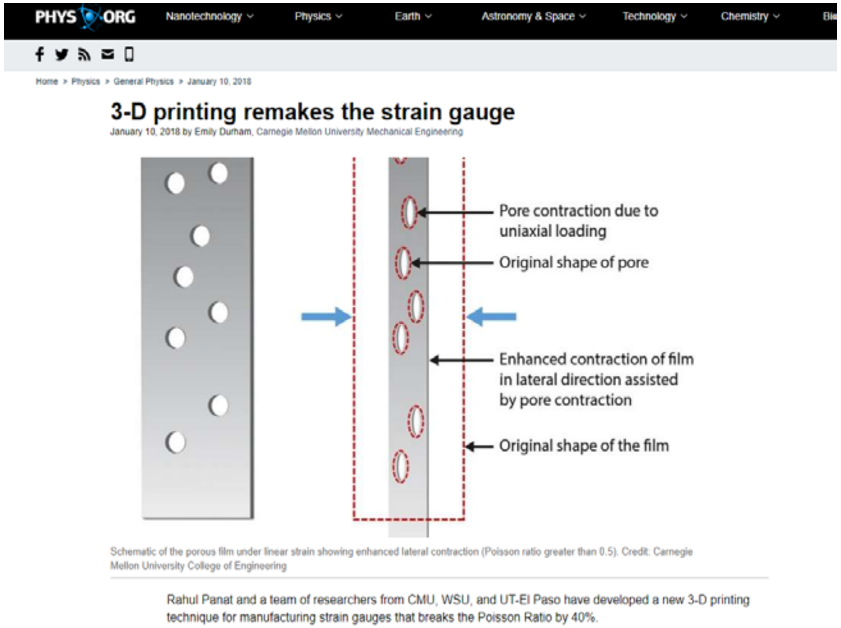
CMU News: 3-D printing remakes the strain gauge

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



PHYS ORG Nanotechnology Physics Earth Astronomy & Space Technology Chemistry

Home > Physics > General Physics > January 10, 2018

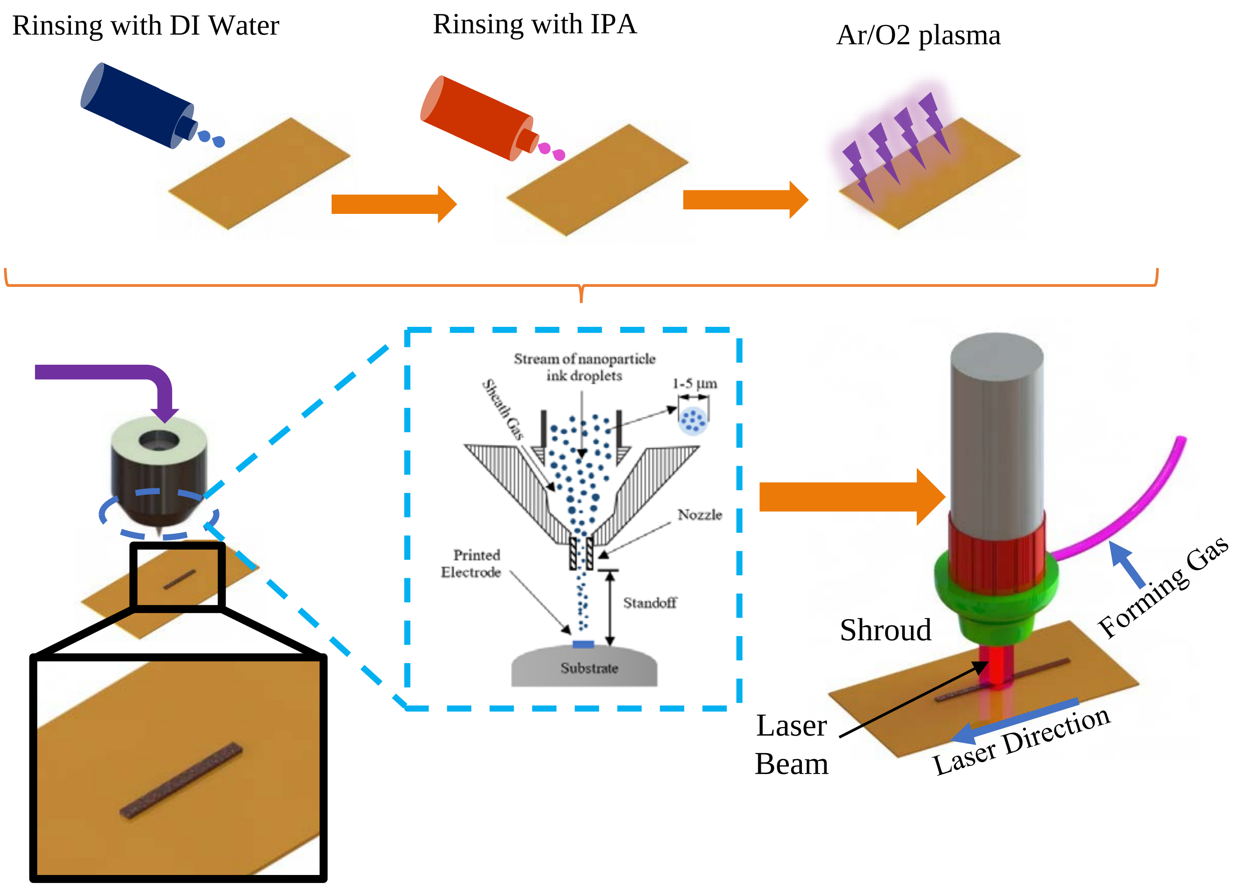
3-D printing remakes the strain gauge

January 10, 2018 by Emily Durham, Carnegie Mellon University Mechanical Engineering

Schematic of the porous film under linear strain showing enhanced lateral contraction (Poisson ratio greater than 0.5). Credit: Carnegie Mellon University College of Engineering

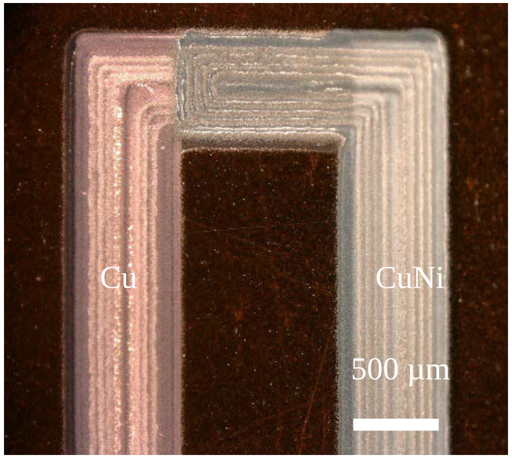
Rahul Panat and a team of researchers from CMU, WSU, and UT-El Paso have developed a new 3-D printing technique for manufacturing strain gauges that breaks the Poisson Ratio by 40%.

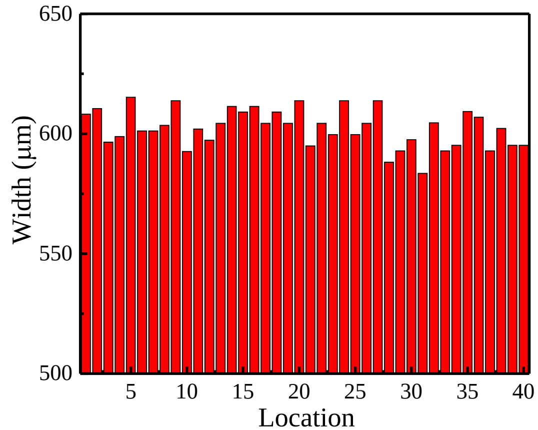
Single Sensors: Printing Temp Sensors

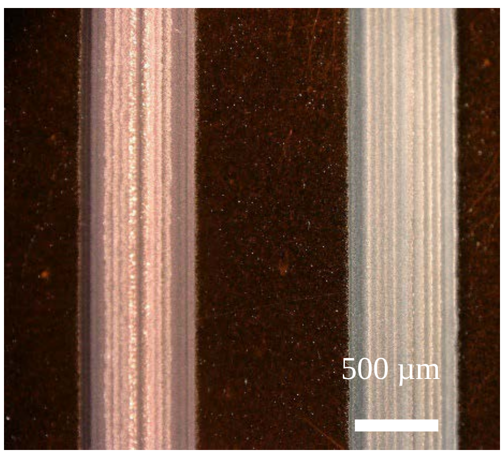


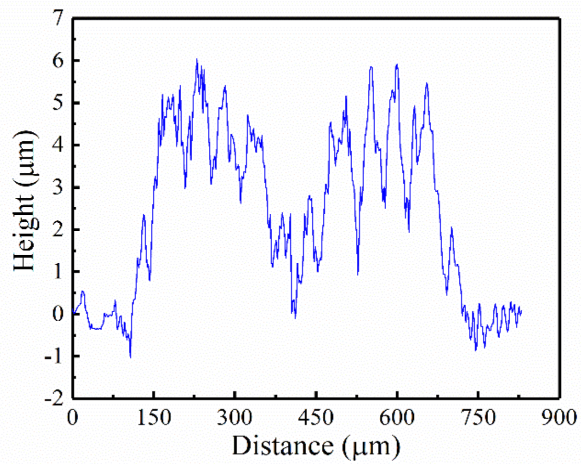
- Printing followed by laser sintering at 100mW and 400mW
- Does not affect the underlying polyimide substrate

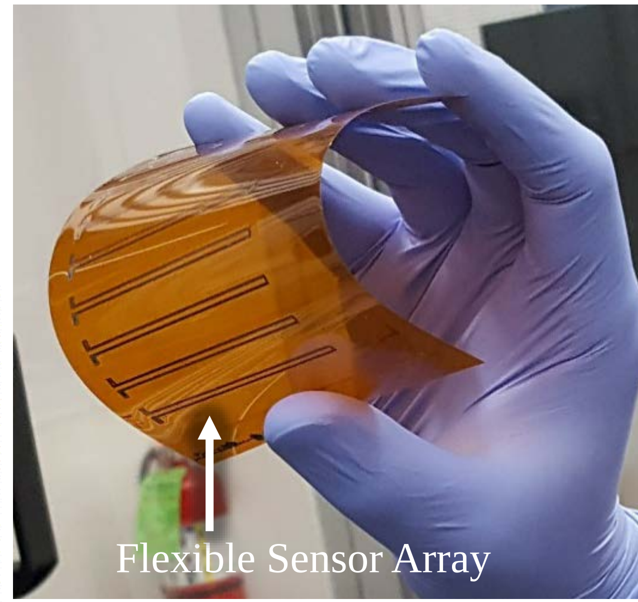
Fabricated Sensor





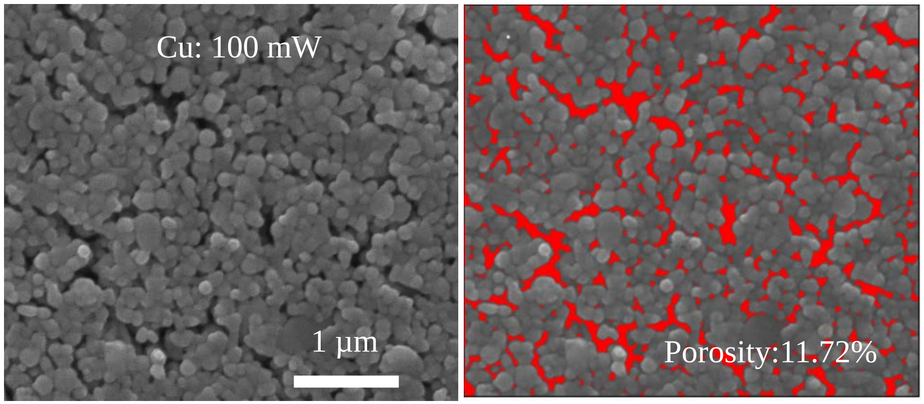


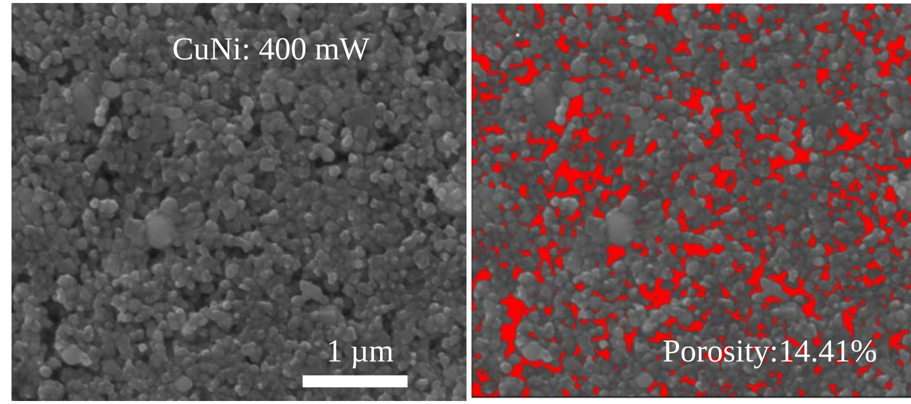


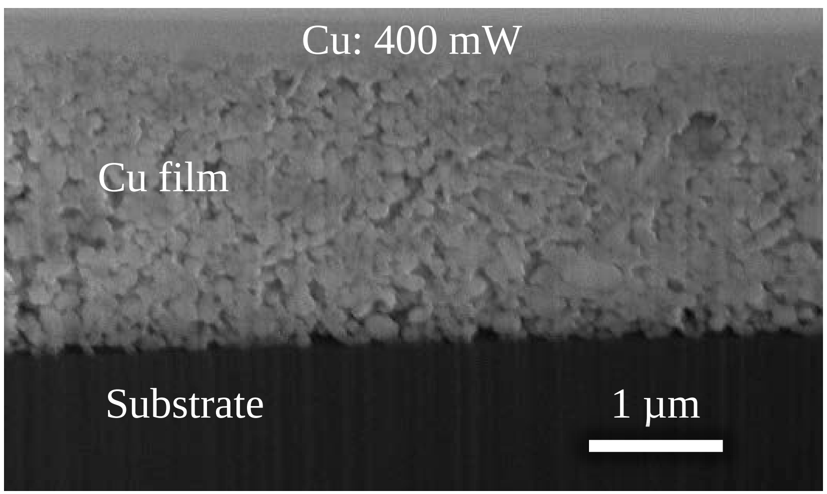


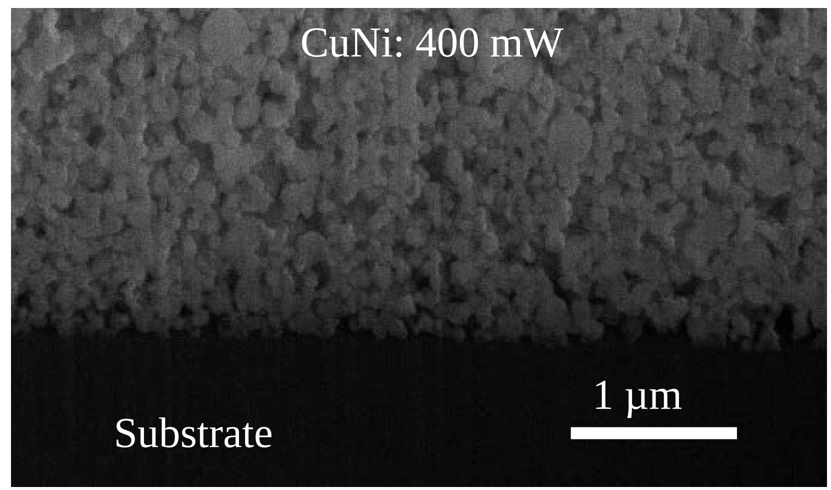
- Flexible geometry realized by direct printing

Microstructure



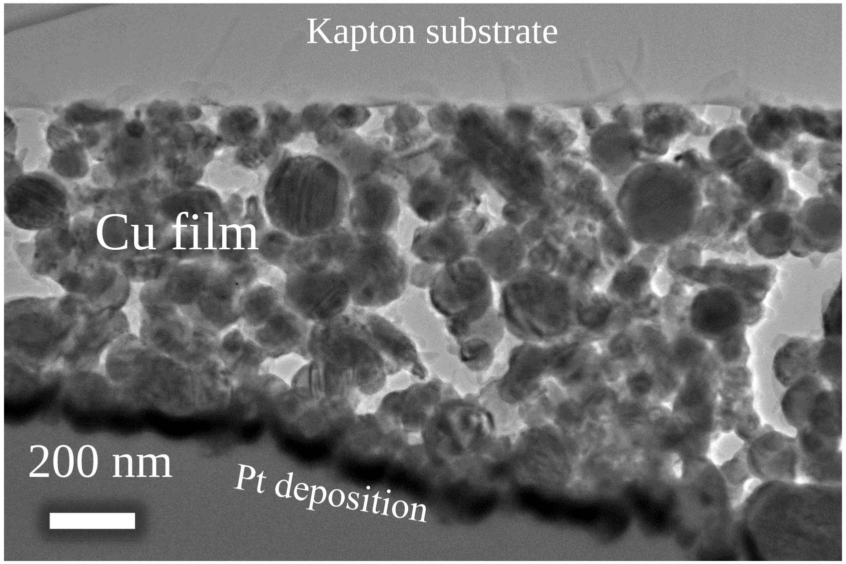


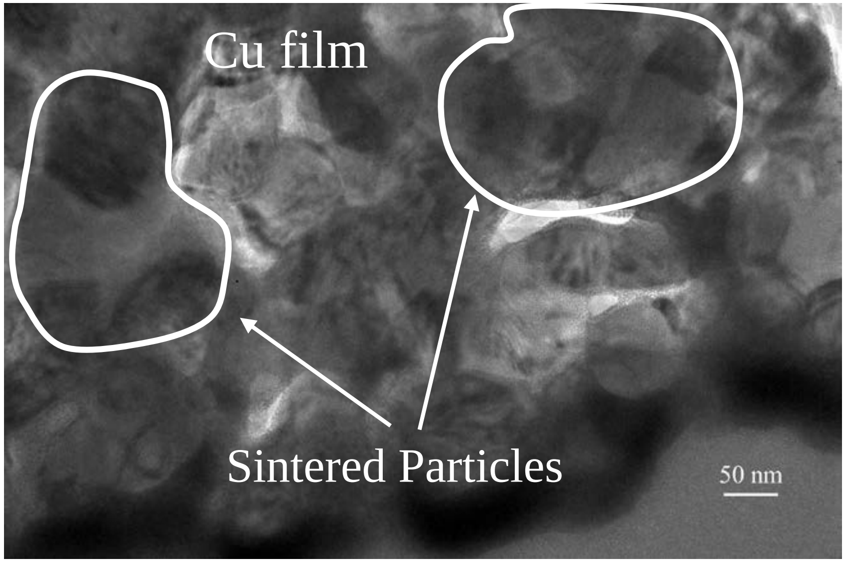


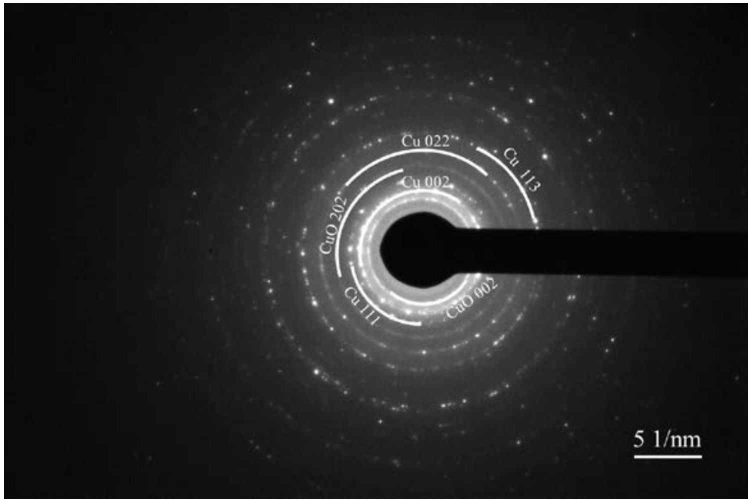


- Fused particles observed in SEM and FIB sections with porosity up to 20%

TEM and SAED Studies

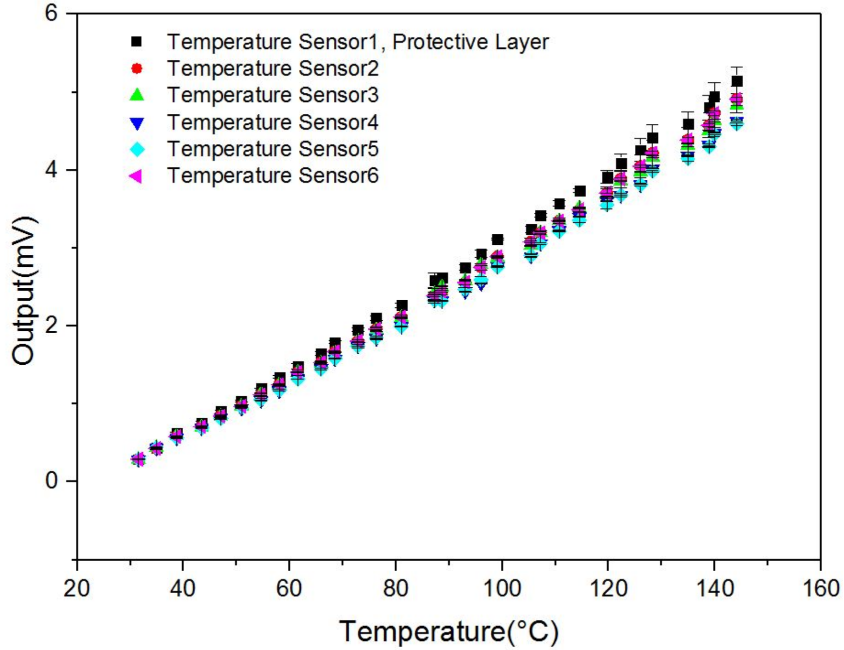


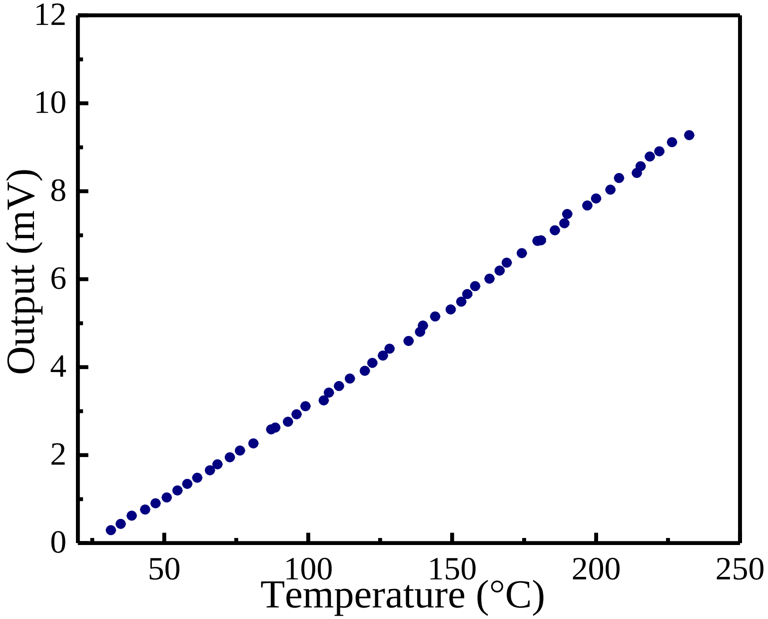




- TEM observations show sintered particles
- SAED pattern shows presence of oxide

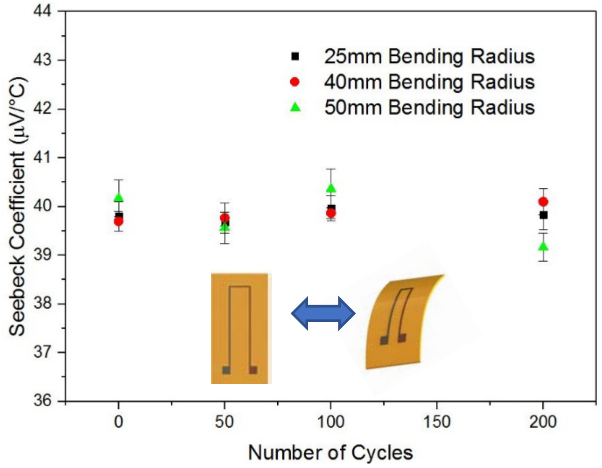
Sensor Characteristics

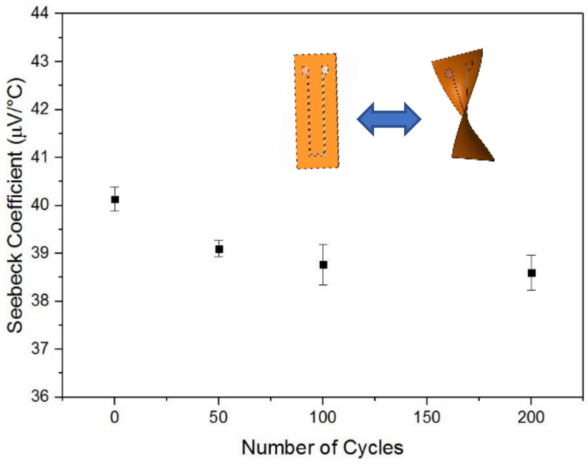


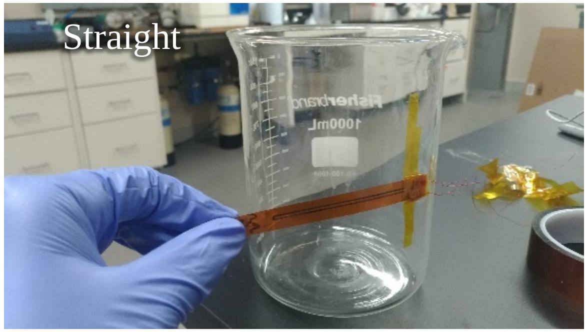


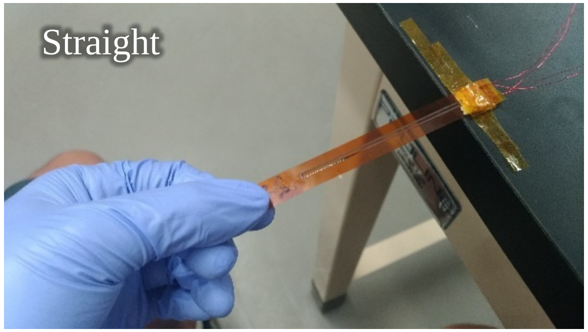
- Highly linear response up to 260 °C

Flexibility Tests









- Established high resilience (strain tolerance) for sensors made by advanced manufacturing

Single Sensors: Ceramic Sensors

- This single sensor investigation was motivated by creating sensing technologies that stable at high temperatures
- **Perovskite oxide** $BaFe_{1-x}Ta_xO_{3-\delta}$ ($x = 0.3$) ceramics were explored for oxygen sensing using conventional solid-state reaction route
  - Other sensing materials such as metal oxides show limited use at high temperatures due to change in conductivity with temperature
- Tailoring the grain size, porosity and morphology with different sintering/annealing temperatures to give different sensing properties
- Phase purity and structural analysis and explore the effect of grain morphology and grain size on sensing response

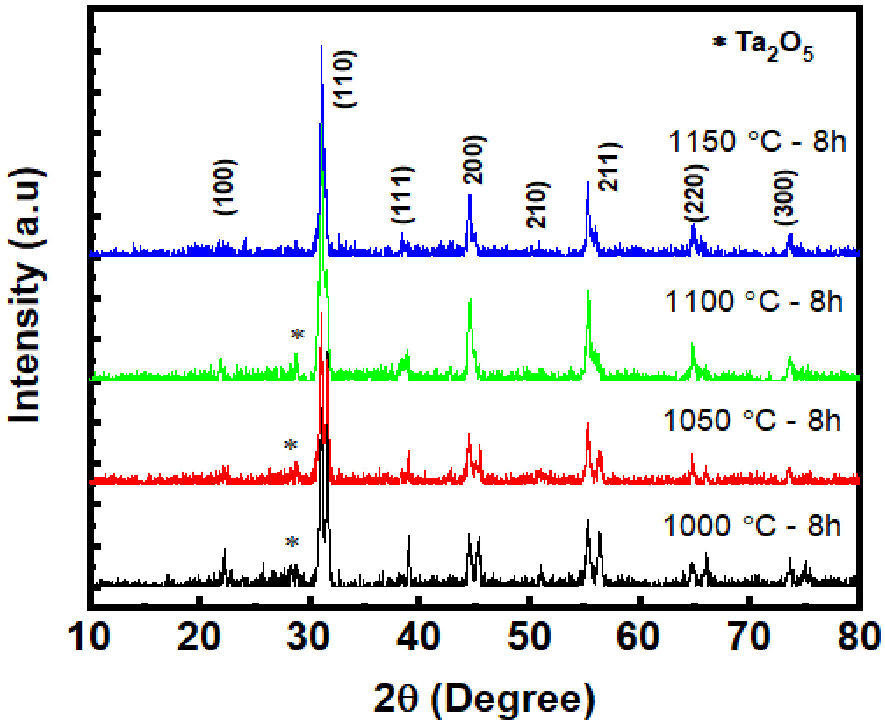
Ceramic Sensors: Materials and Methods

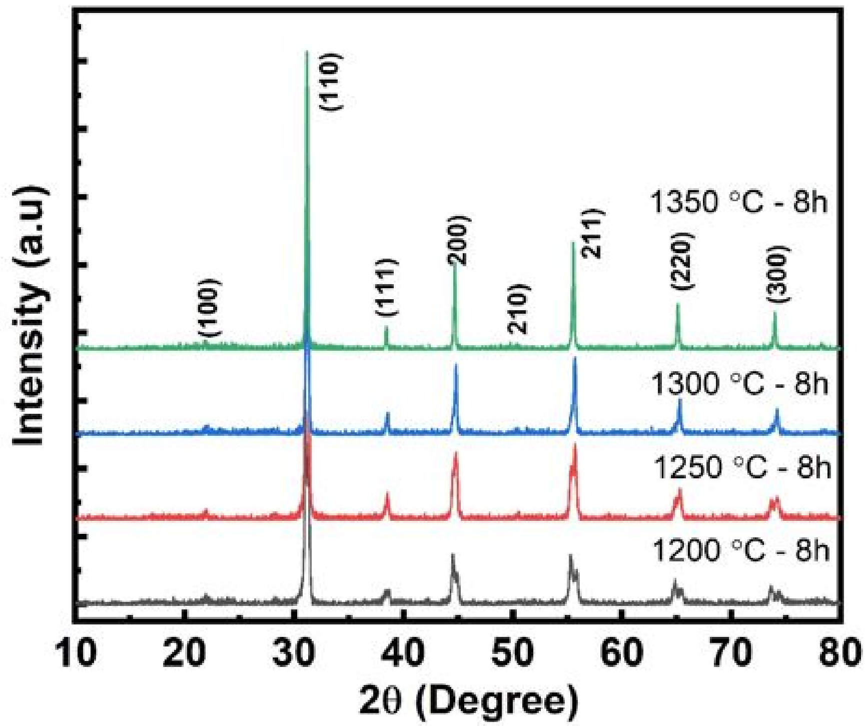
Synthesis of Bulk Ceramics – Solid State reaction

- Precursors - $BaCO_3$, Fe_2O_3 and Ta_2O_5
- Weighing the suitable precursors in stoichiometric proportion
- Calcination – Decomposition and reaction
- Sintering following by palletization
- $BaCO_3 + 1/2(1\text{-}x)\ Fe_2O_3 + 1/2\ (x)Ta_2O_5$

$BaFe_{1\text{-}x}Ta_xO_{3\text{-}\delta}$

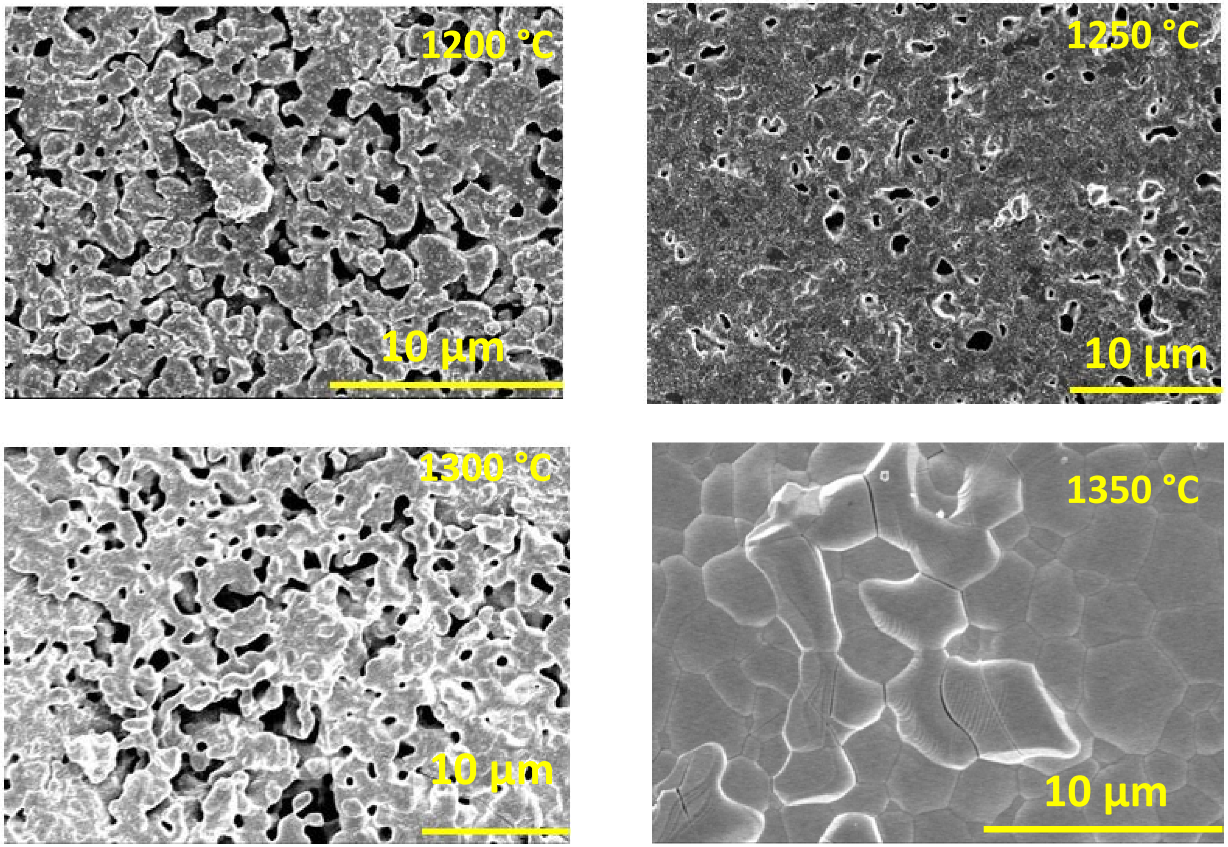
X-ray Diffraction Results





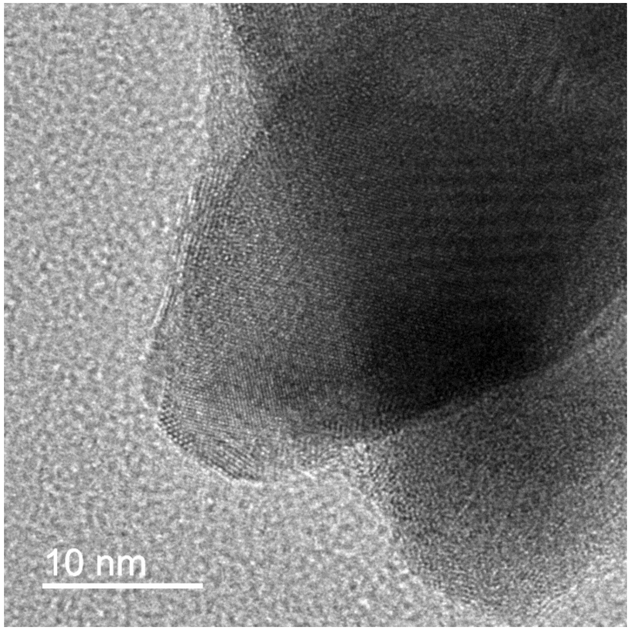
- X-ray diffraction of $BaFe_{0.7}Ta_{0.3}O_{3-\delta}$ calcined and sintered at different temperatures
- Phase purity - 1150 °C

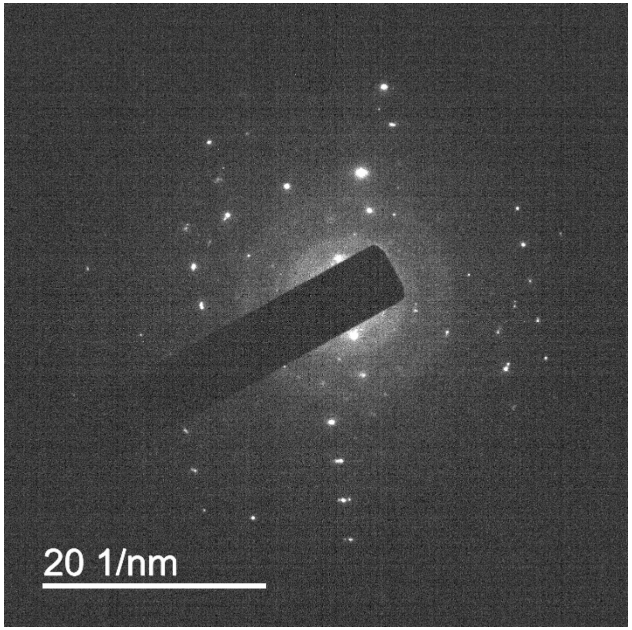
Morphology of $BaFe_{0.7}Ta_{0.3}O_{3-\delta}$ sintered at different temperatures



- Dense packing at 1350 °C

TEM Studies (ongoing)





- TEM and electron diffraction studies currently ongoing to understand the structure of the sensors

Ceramic Sensors

- $BaFe_{1-x}Ta_xO_{3-\delta}$ (x = 0.3) compounds were synthesized through solid state reaction route

- X-ray diffraction of sintered compounds reveals that compounds are phase pure without any secondary phases

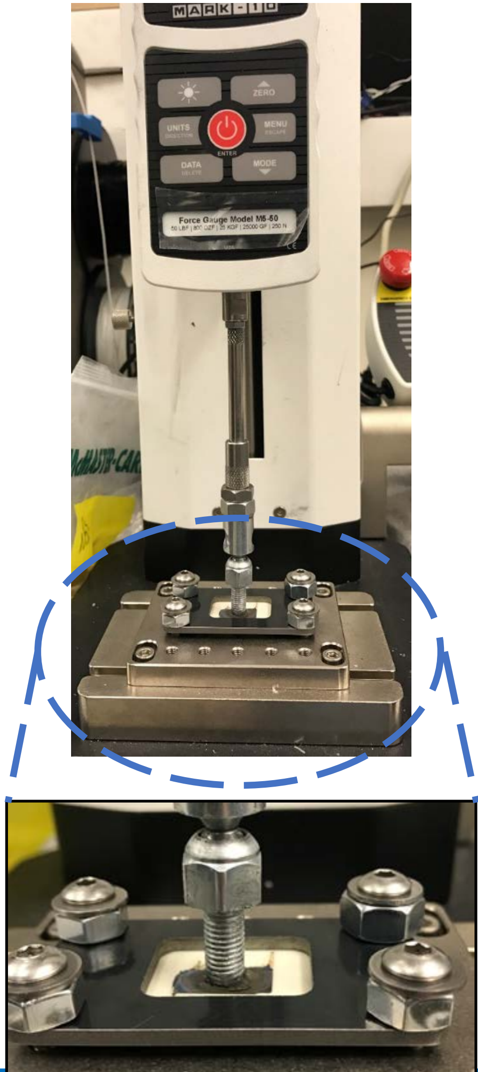
- X-ray diffraction of sintered compounds reveal a evident structural transformation with increasing sintering temperature

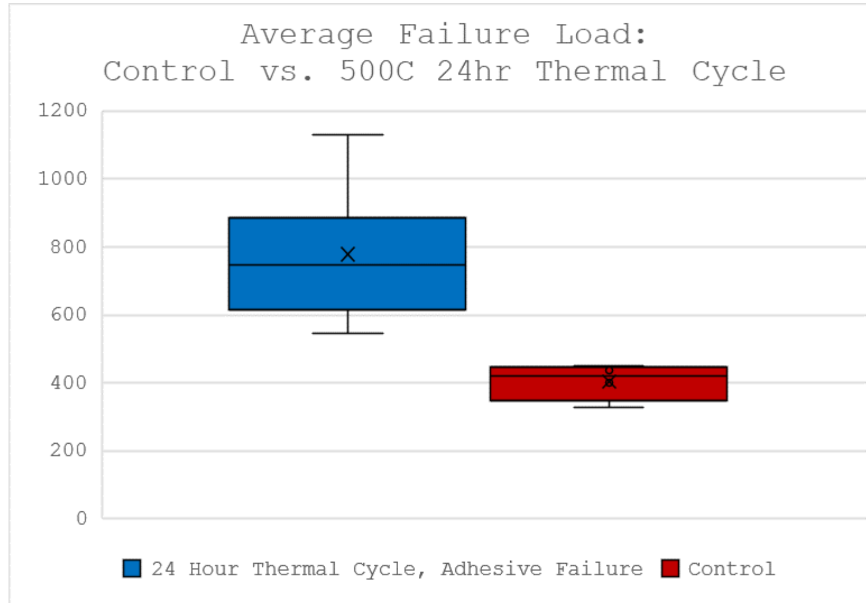
- $BaFe_{0.7}Ta_{0.3}O_{3-\delta}$ exhibits dense morphology at 1350 °C and porous morphology 1200 °C, 1250 °C and 1300 °C

- Next steps will be to explore this material for applications such as high temperature oxygen sensing

Task 3. Reliability Study of the Sensor

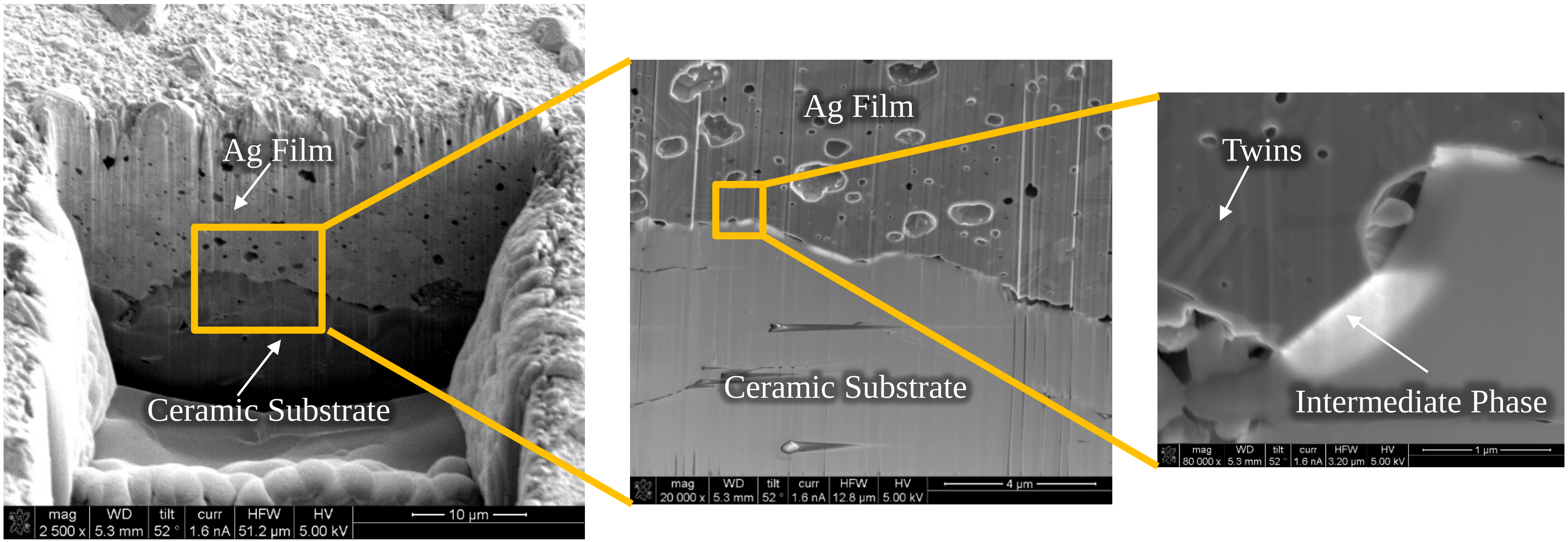
Work of Adhesion Test Setup





- 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



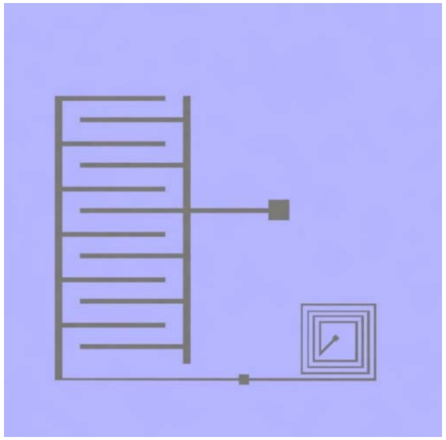
- Focused ion beam was used to observe cross section of the isothermal samples
- Existence of intermediate phase was observed

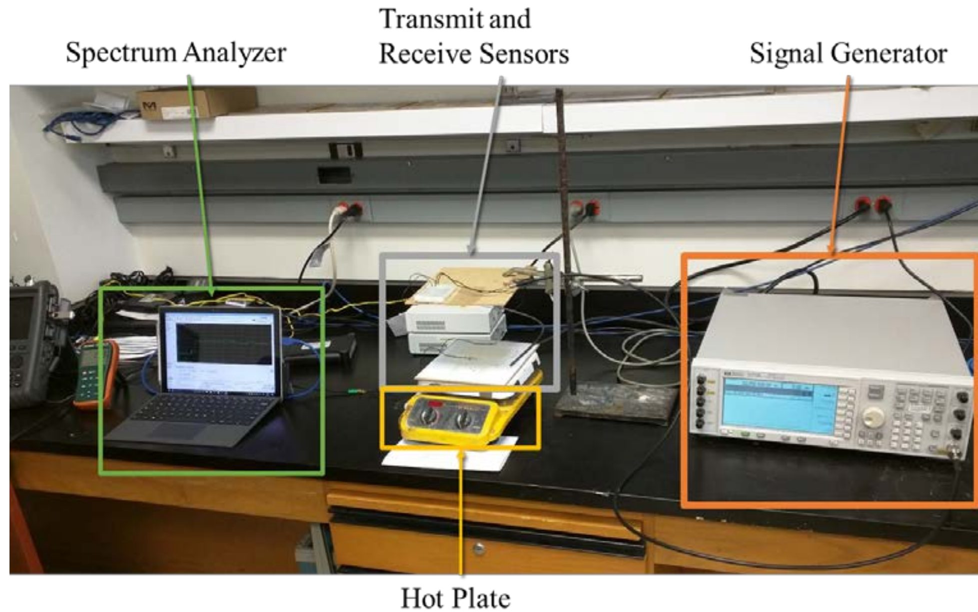
Summary of Research

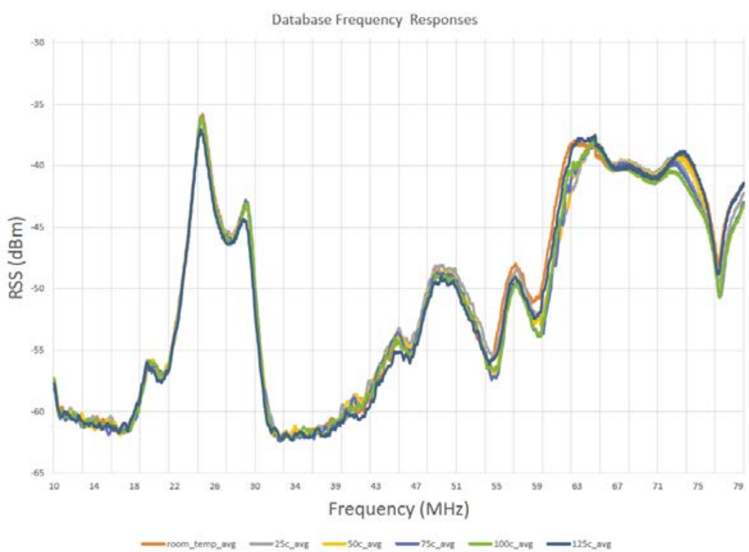
Deliverables:

- 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

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…….

Year-1-3: Student Training and Research Outcomes

Student Training

1. 1 PhD student graduated and joined Intel Corporation, 1 PhD student (minority) will graduate soon
2. 2 students pursuing MS, 1 MS student graduated in March 2018 and joined Janicki Industries
3. 3 Undergraduate researchers (1 minority through Louis Stokes Alliance for Minority Participation), 2 Postdocs

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)**
4. M. T. Rahman, C. H. Cheng, B. Karagoz, M. Renn, M. Schrandt, A. Gellman, and R. Panat, "High Performance Flexible Temperature Sensors via Nanoparticle Printing", in press, ***ACS Applied Nano Materials*** (2019).

Additional papers 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)

Year-1-3: Student Training and Research Outcomes

Conference Presentations:

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)
5. R. Panat, Md. T. Rahman, M. Schrandt, M. Renn, H. Zbib, C. Cheng, C. Ramana, “3D Printed High Performance Sensors”, at the annual TMS conference 2019
6. R. Panat, Md. T. Rahman,, C. V. Ramana, “3D Printed Metal Films”, Invited talk at the annual TMS conference 2019.

Acknowledgement

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

2019 Deliverables

- Completion of the ceramic sensors and testing their performance at high temperatures
- Fabrication of workable antenna at high temperatures

Thank You