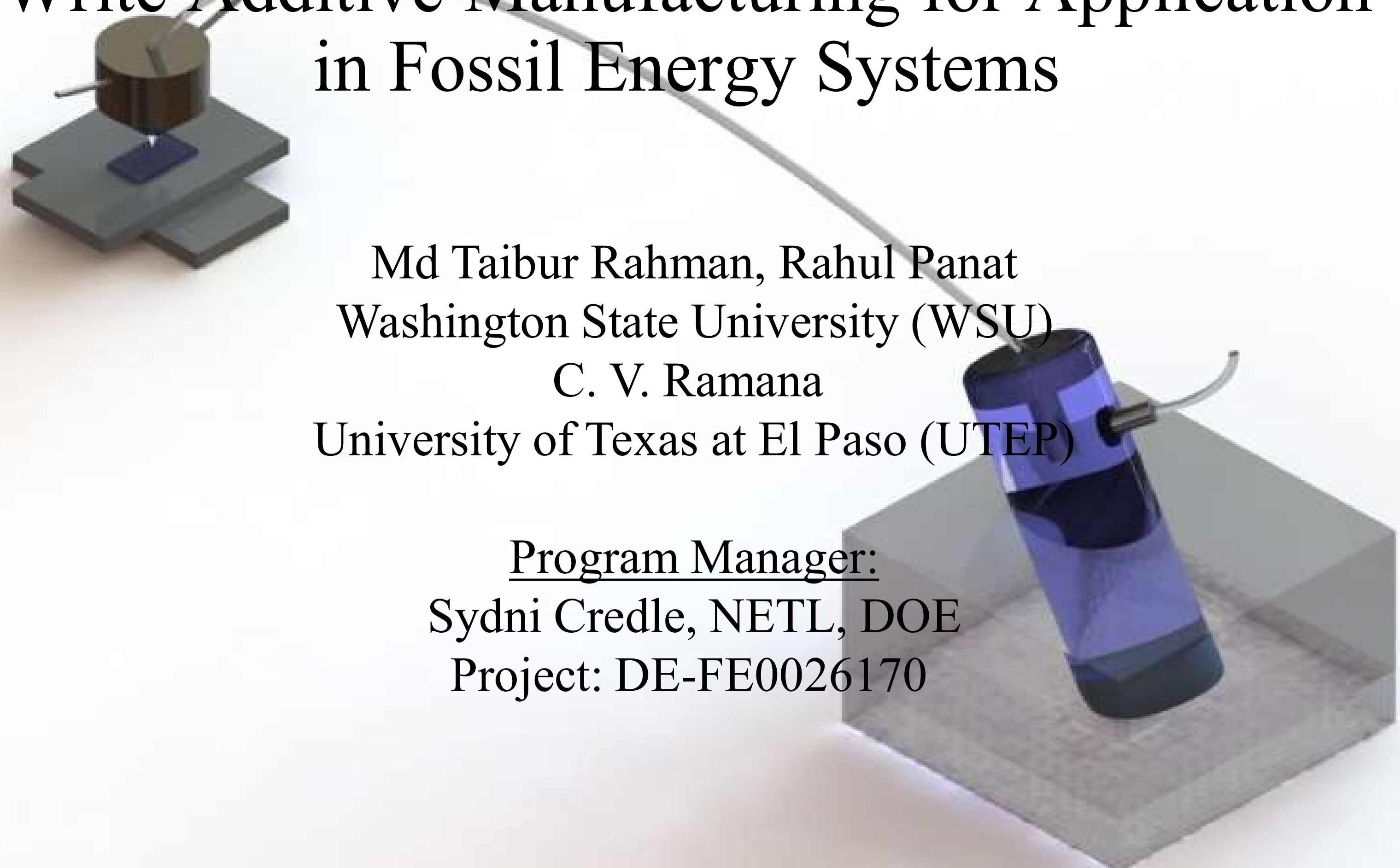
Low-Cost, Efficient and Durable High Temperature Wireless Sensors by Direct Write Additive Manufacturing for Application in Fossil Energy Systems



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Project: DE-FE0026170

Agenda

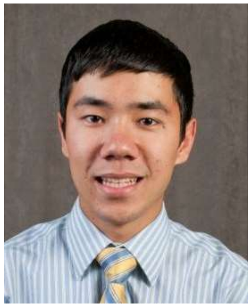
- Introduction and Background
  - Team
  - Project Goals and Objectives
  - Tasks and Timelines
- Tasks/ Research Accomplished
  - Manufacturing: Additive Printing Technique
  - Material Selection and Sensor Characterization
  - Single Sensor Design/ Printing and High Temperature Test Set-up
  - Sensor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

The Team

Washington State University

Rahul Panat
Project Lead PI

Md. Taibur
Rahman (PhD)



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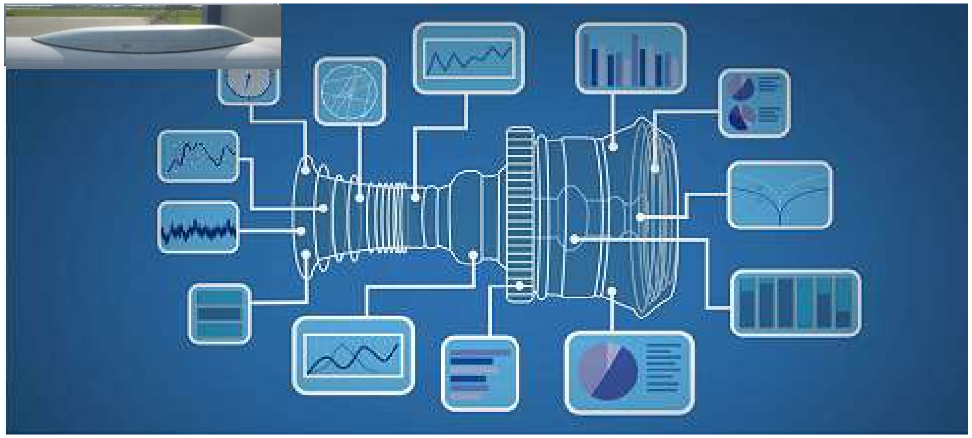
Project Co-PI

Juan Gomez (PhD)

Dr. P. Dubey

Background

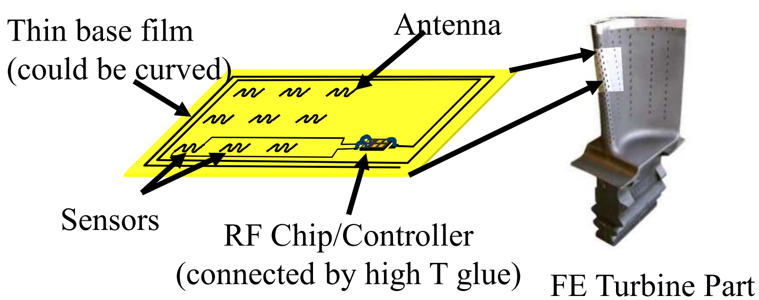


200 sensors across the turbine generate 300 data points per second

- 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
- Objectives:
  - Developing novel materials and manufacturing method for wireless strain sensors and pressure sensors that can operate at high temperatures (>350 ^{o}C up to 500 ^{o}C)
  - Integration of electronic circuitry on a curved 3-D surfaces such as those observed in gas turbine engines
  - Improvement of reliability issues for wireless sensors that arise from the demanding FE environments.



Schematic of a fully integrated high temperature wireless sensor system

Tasks and Timelines

	2015			2016												2017												2018								
	Q4			Q1			Q2			Q3			Q4			Q1			Q2			Q3			Q4			Q1			Q2			Q3		
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
Task 0.0: Feedback to DOE																																				
Task 1.0: Single Sensor Elements - Material System and Manufacturing Methods																																				
Task 2.0: Single Sensor Design and Testing																																				
Task 3.0: Reliability of Sensors at High Temperature																																				
Subtask 3.1: Work of Adhesion and Nanoindentation																																				
Subtask 3.2: Interfacial TEM observations																																				
Task 4.0: Wireless System Design and Fabrication																																				
Subtask 4.1. Sensor integration over a substrate																																				
Subtask 4.2.Design of a Wirelessly-Powered Integrated RF Transceiver																																				
Subtask 4.3: Integration over a Platform																																				

- Key Milestones (2016):
  - Develop/optimize Additive Manufacturing method
  - Sensor Material Characterizations (Impedance analysis, Oxidation study, Micro/Nano structure study)
  - Primary Material Selection
  - Reliability Test Setup

Task 1: Manufacturing Method and Material System

	2015			2016												2017												2018								
	Q4			Q1			Q2			Q3			Q4			Q1			Q2			Q3			Q4			Q1			Q2			Q3		
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
Task 0.0: Feedback to DOE																																				
Task 1.0: Single Sensor Elements - Material System and Manufacturing Methods																																				

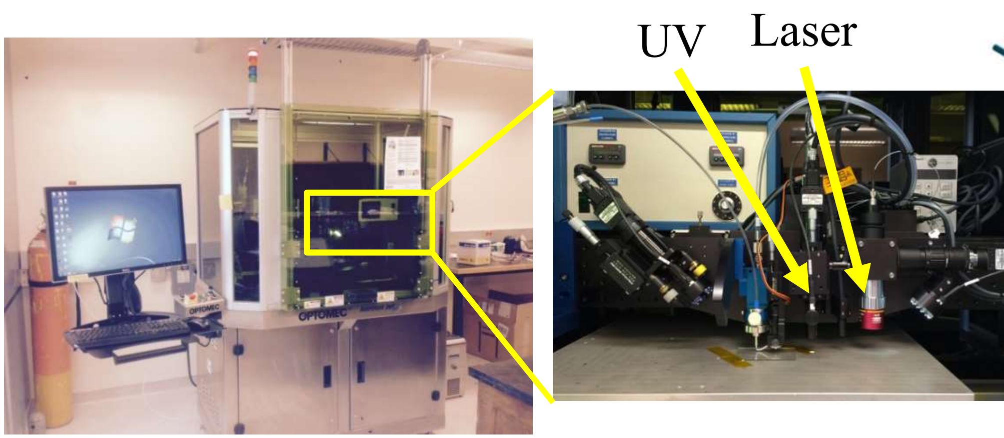
- ❑ Manufacturing Method: Aerosol Jet Additive Printing
- ❑ Material Selection:
  - Study of electrical characterization by impedance spectroscopy
  - Microstructuctural observation through SEM, TEM, XRD, AFM
  - Study of oxidation resistance by TEM/SAED, XRD, XPS, TGA

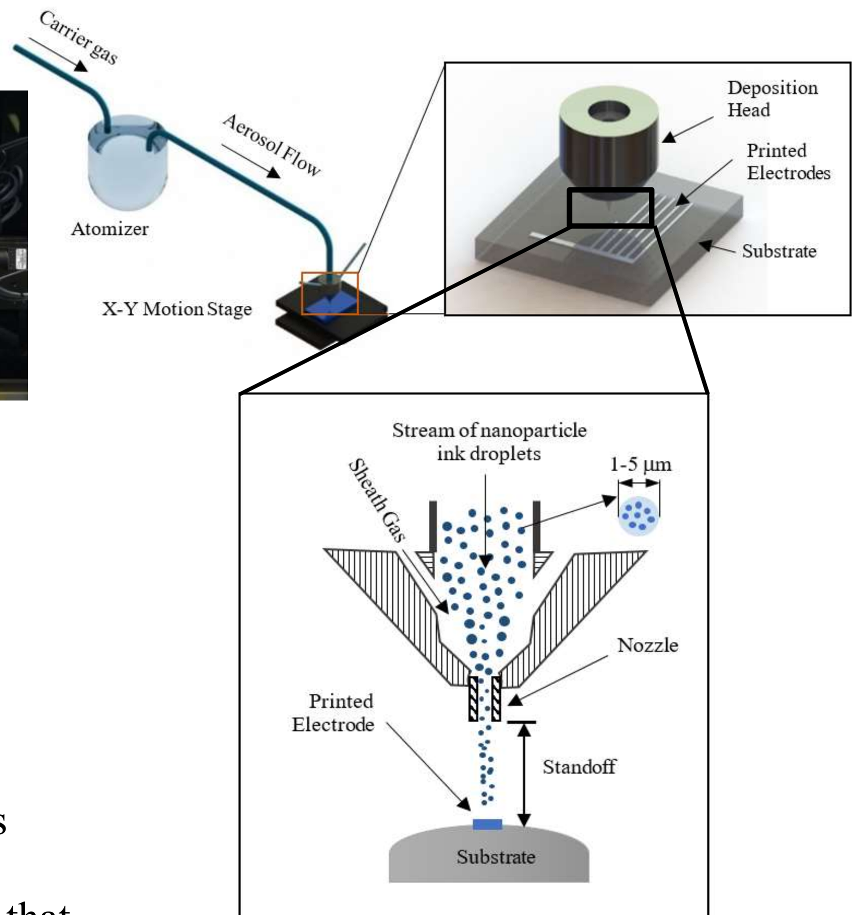
Additive Manufacturing/Printing for Sensors

Advantages:

- Environmentally sustainable manufacturing due to minimal waste
- High surface to volume ratio/porosity of sintered film can improve sensitivity of detection
- Flexibility of using different materials – any material in nanoparticle form can be printed
- Capability to rapidly produce custom sensors
- Possibility of arbitrary 3D substrates/surfaces

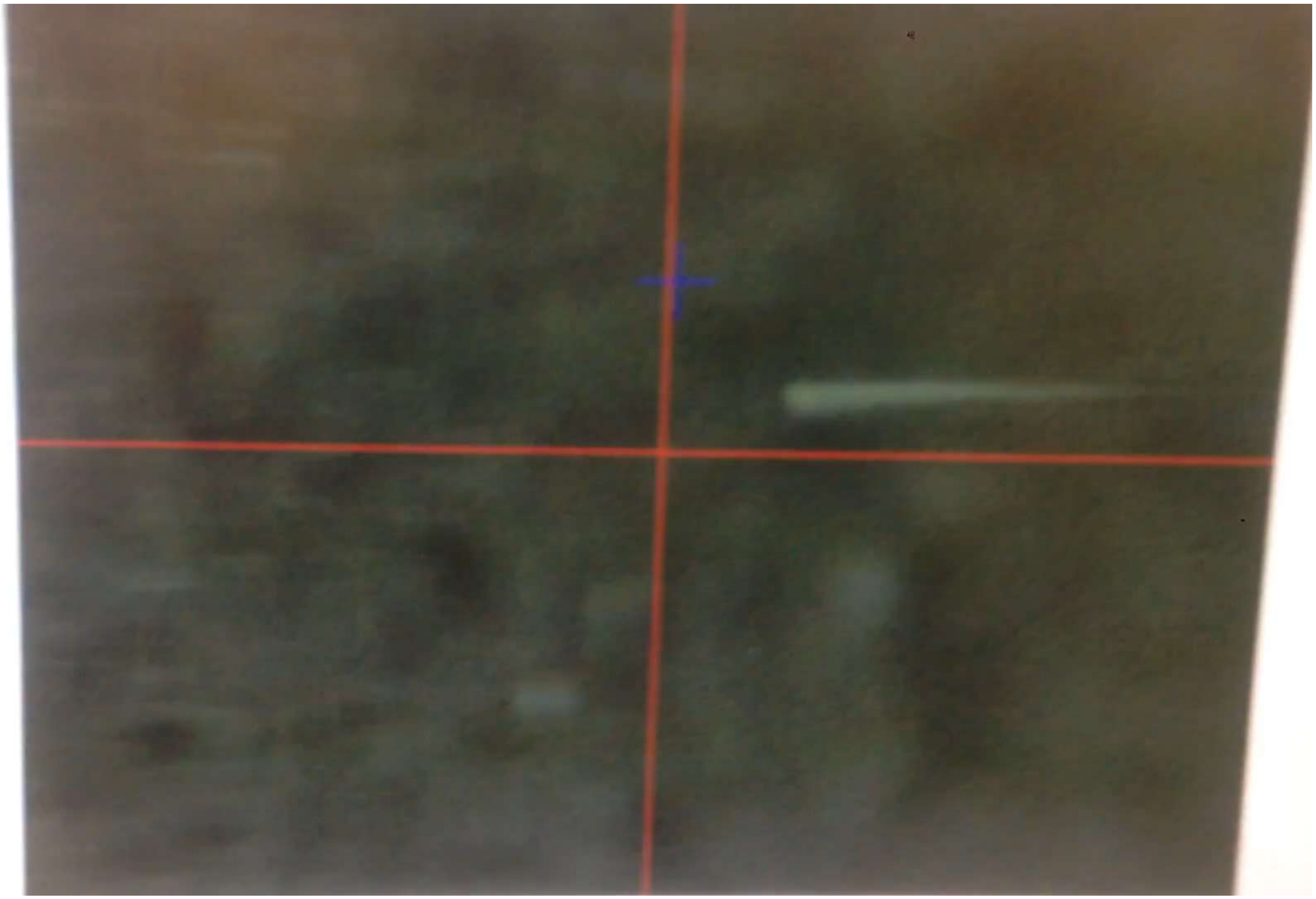
Approach: Aerosol-Jet Direct-write Printing





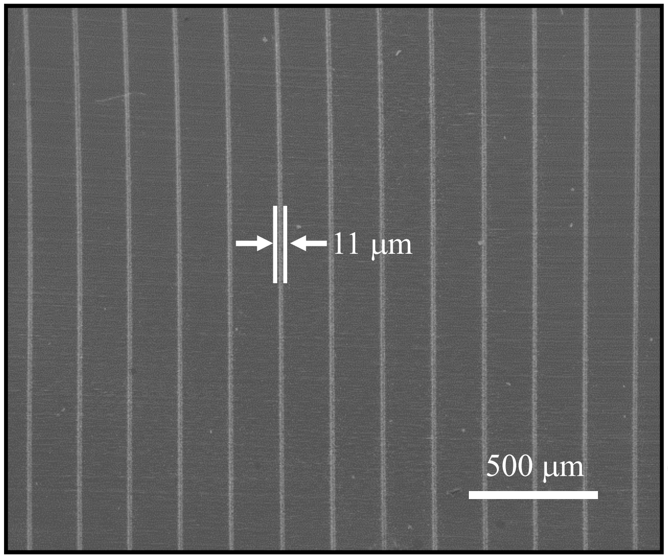
- ❑ Operates by creating aerosol mist of 1-5 µm droplets, each containing nanoparticles
- ❑ Clog resistant nozzles
- ❑ Advantages:
  - Minimal waste and manufacturing steps
  - Possibility of arbitrary 3D substrates/surfaces
  - Flexibility of using different materials
  - High surface to volume ratio of sintered film that can improve sensitivity for certain applications

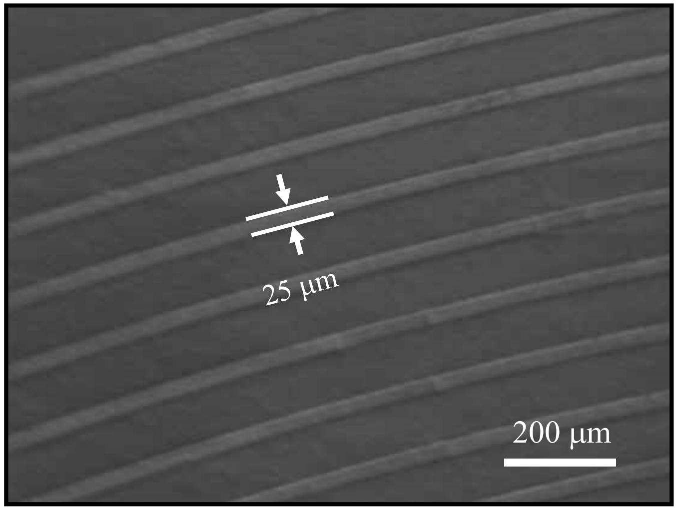
Aerosol-Jet Printing Video

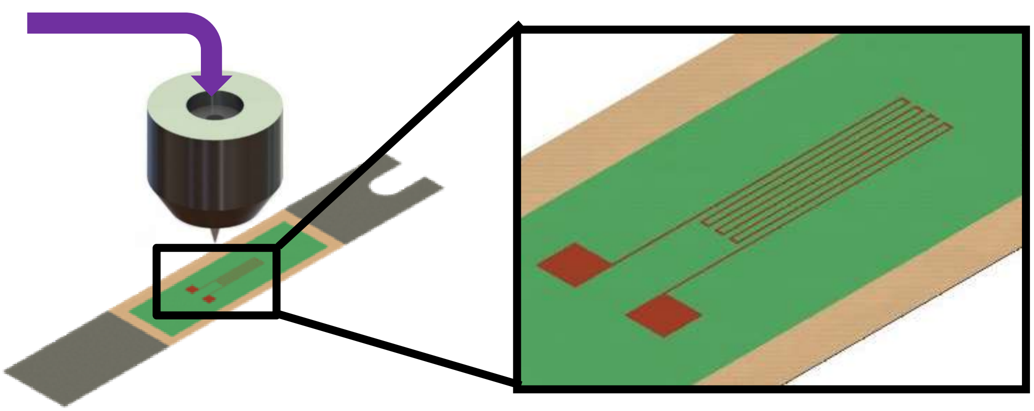


MTR2

High Resolution Printing





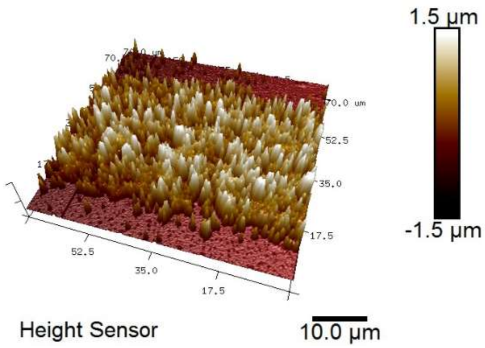


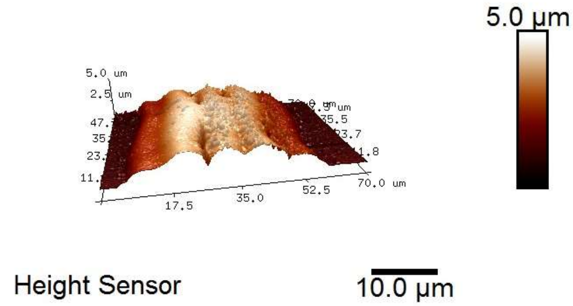
- High spatial resolution
- Feature size down to 10 μm
- High consistency in width

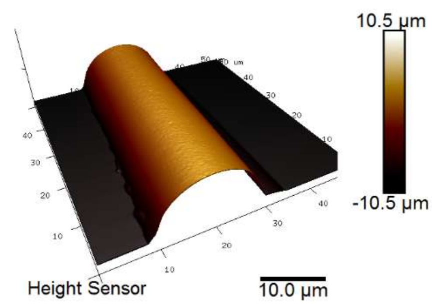
Slide 11

MTR2 add a sesnor design image
MD TAIBUR RAHMAN, 3/8/2017

Good Control Over Printed Lines



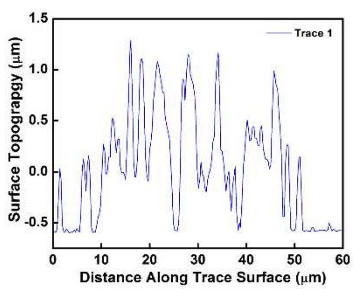


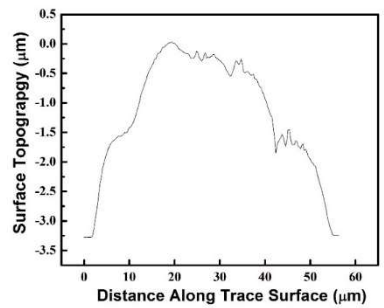


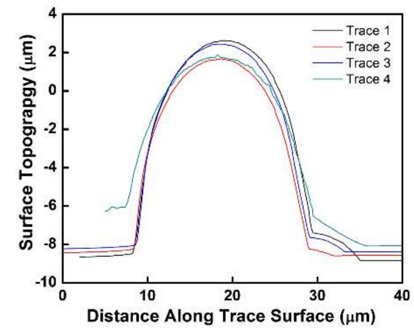
Single layer

Three layers

Multiple layers



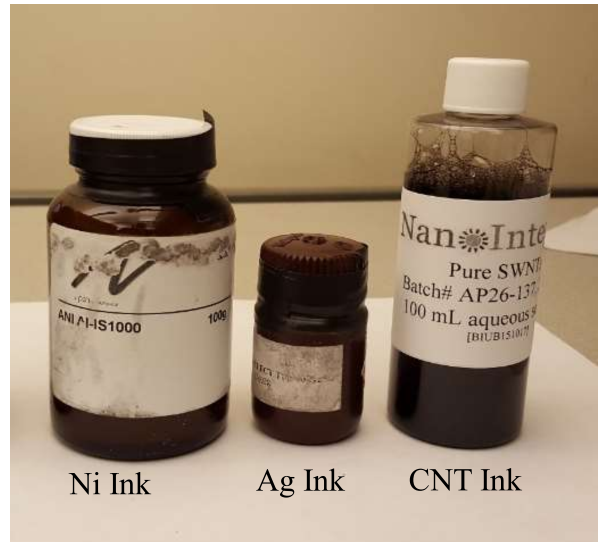




Good control over printed lines/films, roughness of the film can be minimized by multiple printed layers

Material Systems

- Silver (Ag) Nanoparticles
  - Viscosity: 1cP
  - Particle Size: 20-30 nm
- Dispersed Carbon Nanotubes (CNTs)
  - Viscosity: 1cP
  - Diameter : 100 nm
- Nickel (Ni) Nanoparticles
  - Viscosity: 16-25cP
  - Particle Size: 20-100 nm
- Nichrome (NiCr) Alloy Nanoparticles
  - Viscosity: 1-5cP
  - Particle Size: 100 nm



Slide 13

MTR3 put a pic of vial with ink
MD TAIBUR RAHMAN, 3/9/2017

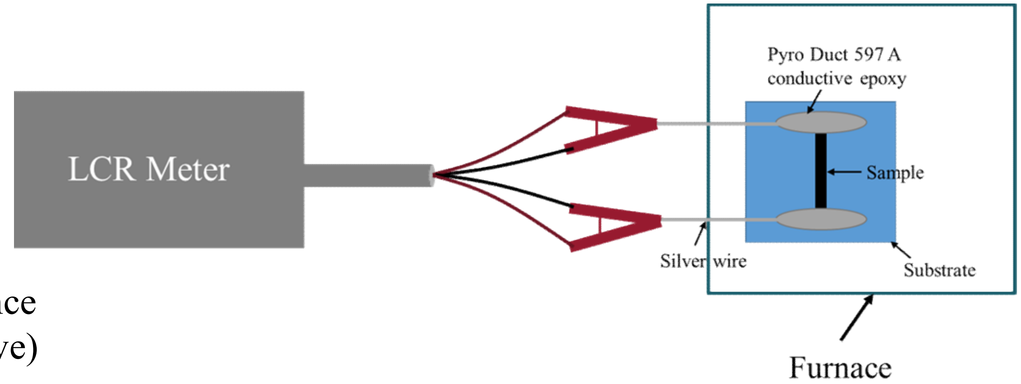
Electrical Characterization on Sensor Segment

Impedance Spectroscopy

$$Z = R + jX$$

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

Obstacle to current flow in AC circuit



DC circuit does not have reactance

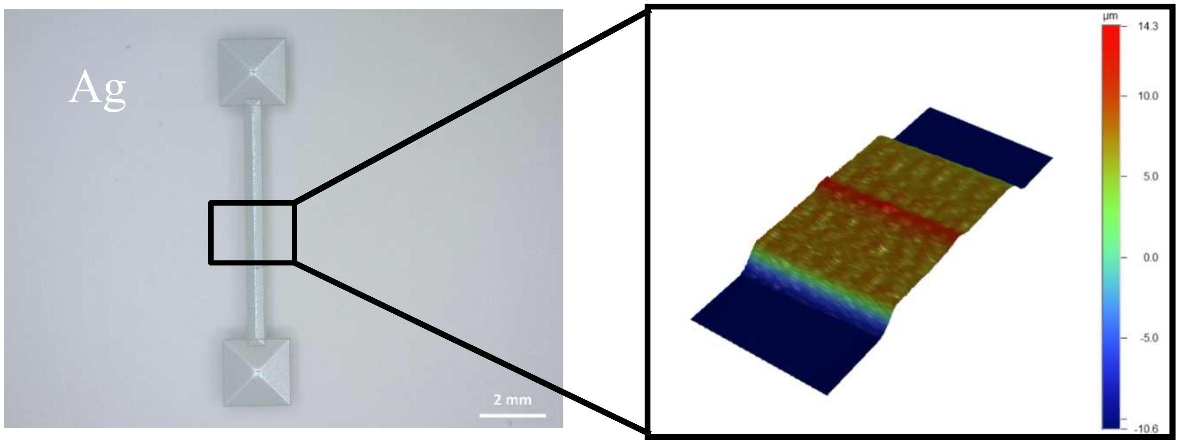
High T sensors will have an AC coupled system

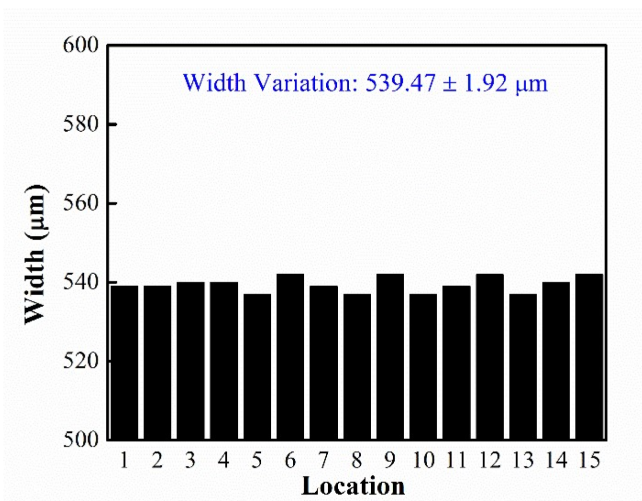
- Z change with T
- Z change with f

Impedance characterization carried out in-situ for temperatures up to 500 °C

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

Printed Sensor Segment

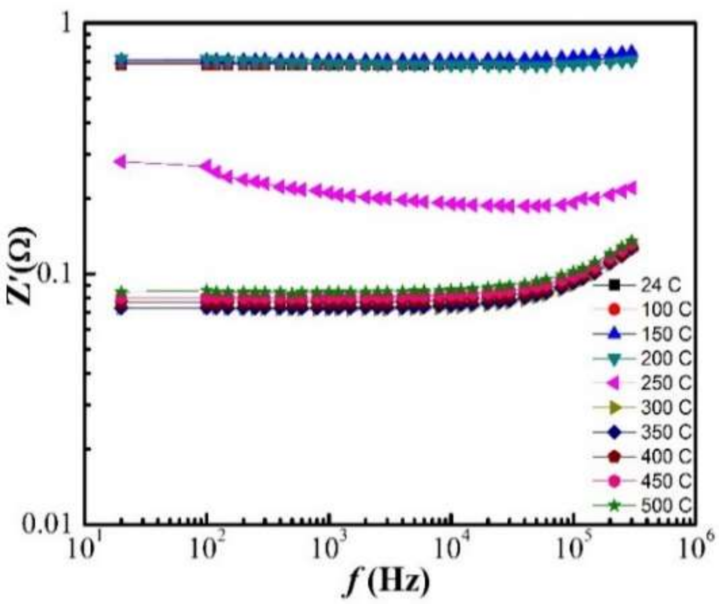




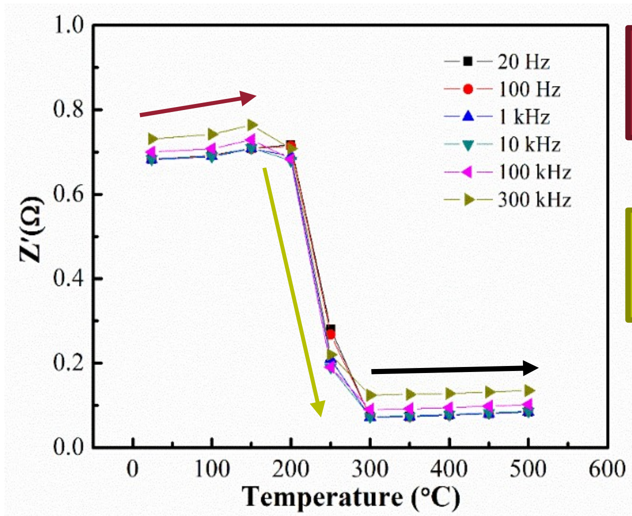
- Metal electrode that can response to a stimulus such as strain to provide require output for sensor action
- Highly repeatable printing of sensor segments
- Low surface roughness achieved with printing parameters (e.g. multiple passes, nozzle size etc.)

Impedance Spectroscopic Characterization of Ag

❑ Sintered for 30 min at 200 °C

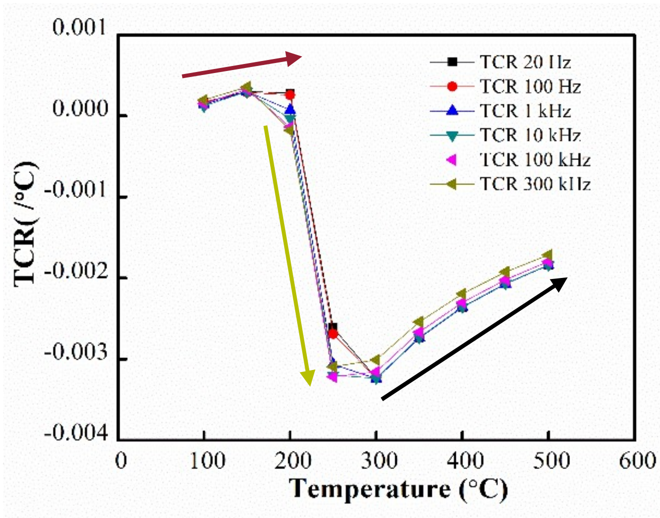


Problem:
Unstable impedance behavior



Increase in Z' up to 150 °C

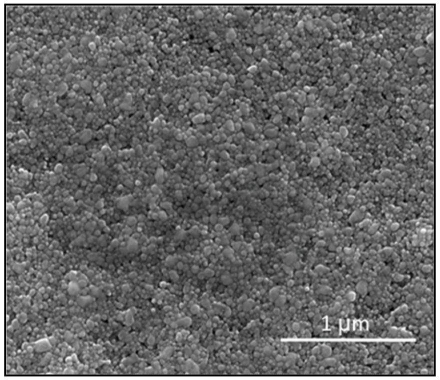
Z' drops beyond 150 °C



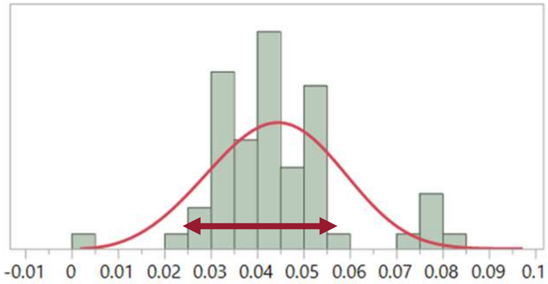
TCR for 24-150°: 0.000302/°C (at 20 Hz)

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

Microstructure Analysis of a Post Impedance Sample



As-sintered

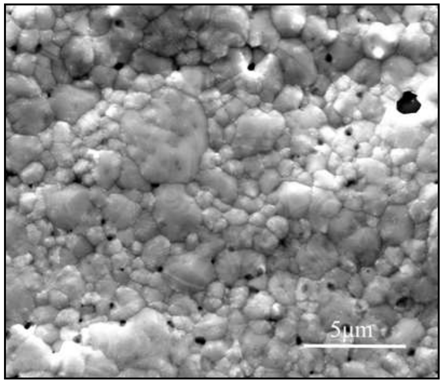


Grain Size Distribution (µm)

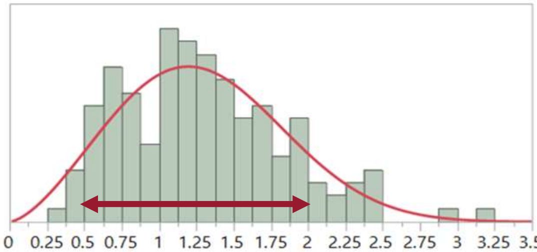
D_{avg} : 40 nm

Increase in grain size

Additional sintering during impedance testing



After impedance test

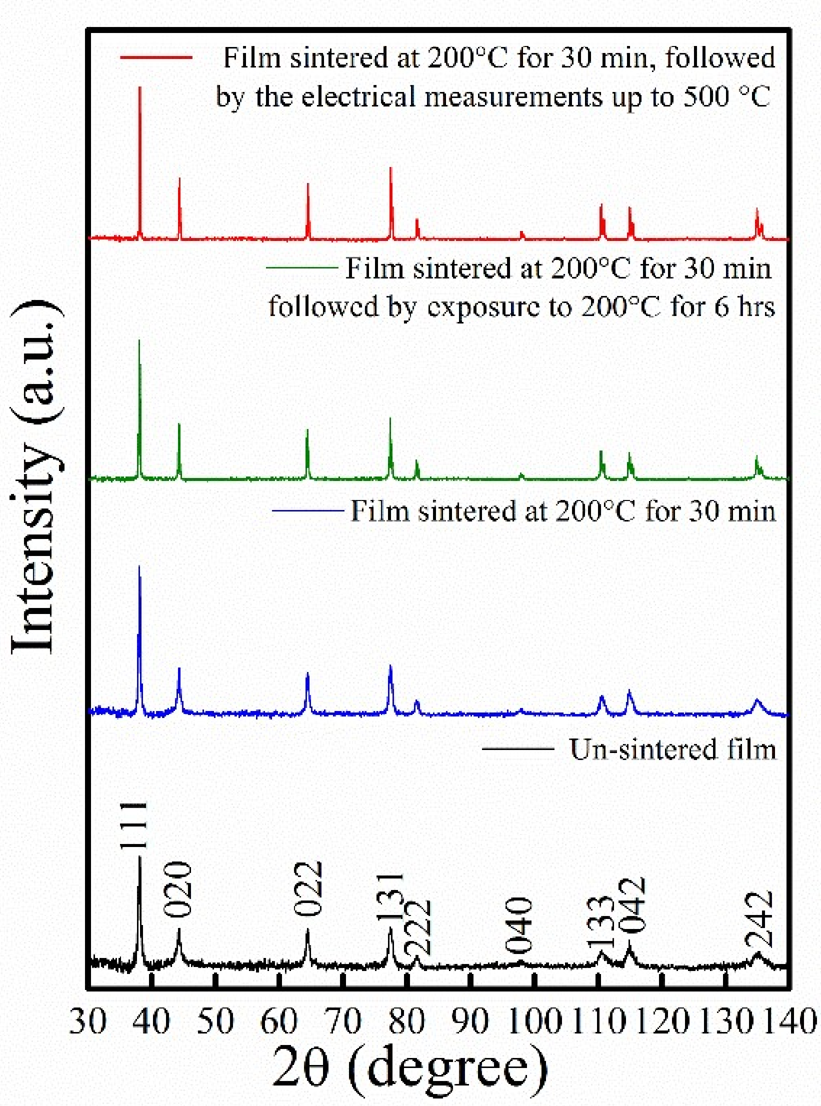


Grain Size Distribution (µm)

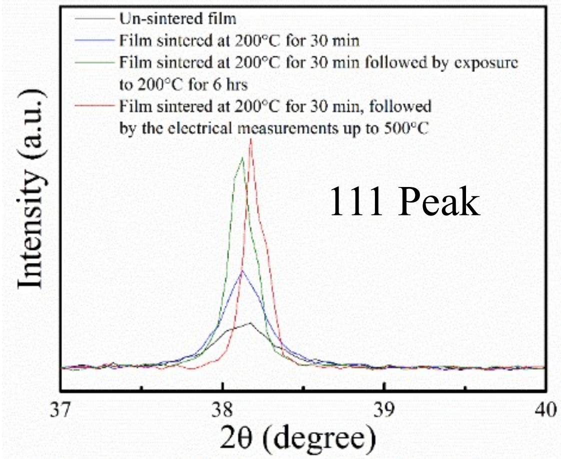
D_{avg} : 1 µm

Is the grain growth causing a drop in Z' ?

X-ray Diffraction Analysis

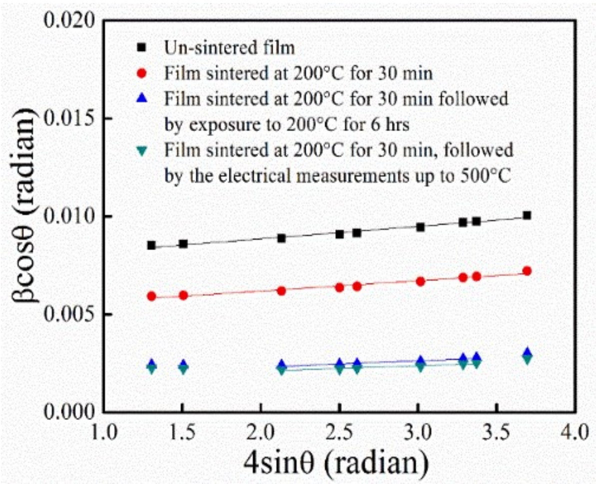


- All peaks correspond to fcc Ag
- No formation of secondary or oxide phases



- Sintered samples the peak width decreased
- Peak shift: indicates crystallite size increase

Analysis of Crystallite Size and Microstrain



Williamson-Hull Plot

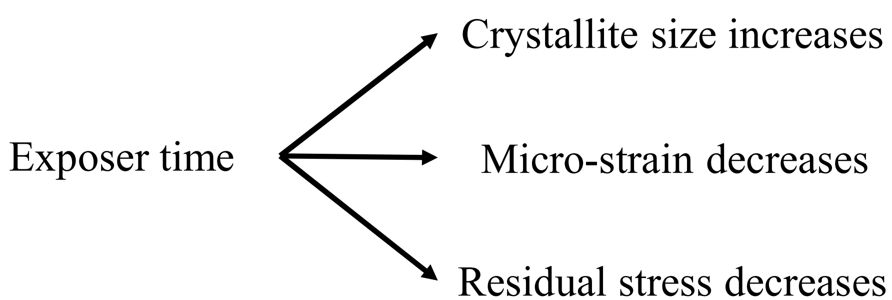
Sample	Lattice Constant, a (Å)	Unit Cell Volume ($Å^3$)	Crystallite Size, D (W-H) Plot (nm)	Crystallite Size, D (from Scherer's Eqn.) (nm)	Micro Strain (W-H) Plot (%)	Dislocation Density (δ) (m^{-2})
1	4.08755	68.2952	19.30	16.27	0.000636	6.15E14
2	4.08755	68.2952	28.174	23.38	0.000527	4.28E14
3	4.08835	68.3352	114.028	57.13	0.000455	1.75E14
4	4.07991	67.9130	127.0319	61.18	0.000273	1.63E14

1) Un-sintered sample, 2) Sample was sintered at 200 °C for 30 min, 3) Sample was sintered at 200 °C for 30 min + post processed at 200 °C for 6hr, 4) Sample was sintered at 200 °C for 30 min + exposed to 500 °C for impedance measurement. Lattice constant, unit cell volume and crystallite Size (from Scherer's Eqn.) was calculated based on most intense peak (111)

$$\beta \cos\theta = \frac{0.9\lambda}{D} + 4\varepsilon \sin\theta$$

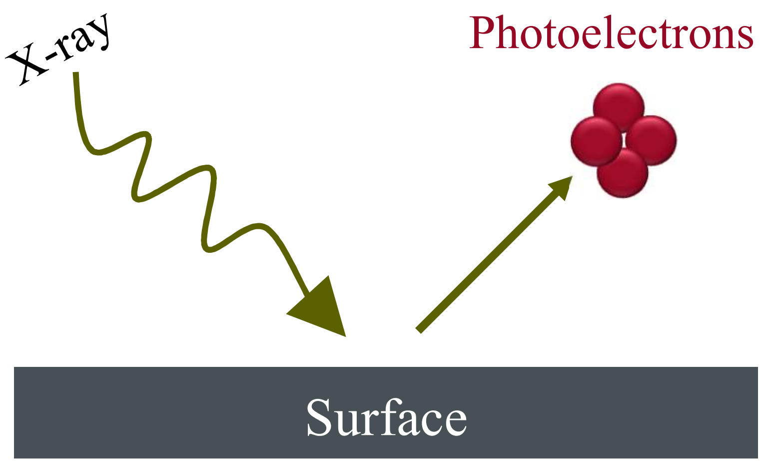
Crystallite size → D; Micro-strain → ε

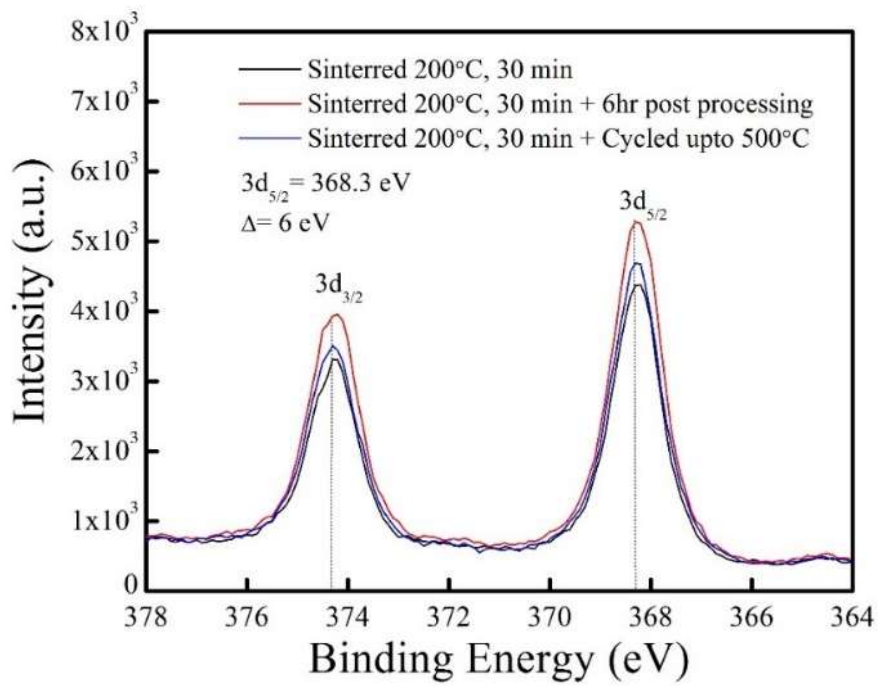
Crystallite size ↓ Dislocation density $\delta = \frac{15\beta cos\theta}{4aD}$



Crystallite size increase confirmed by XRD analysis

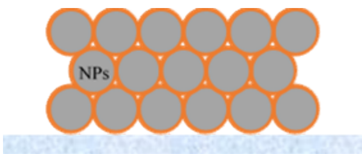
X-ray Photoelectron Spectroscopy (XPS) Analysis



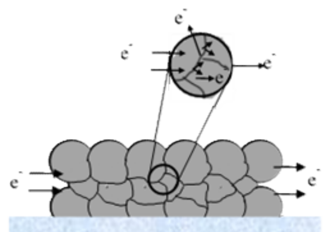


- No peak shift or change in peak shape due to heating
- Ag did not oxidize when exposed to 500 °C

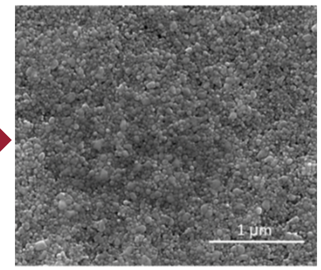
Hypothesis



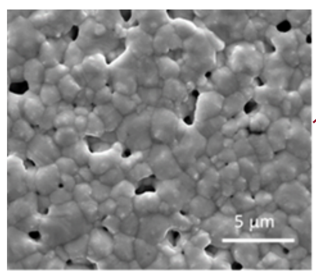
As Printed



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

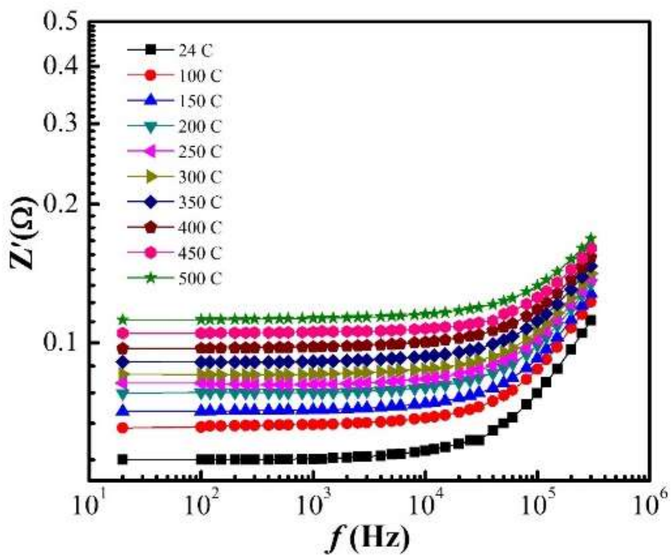
Bulk resistivity

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

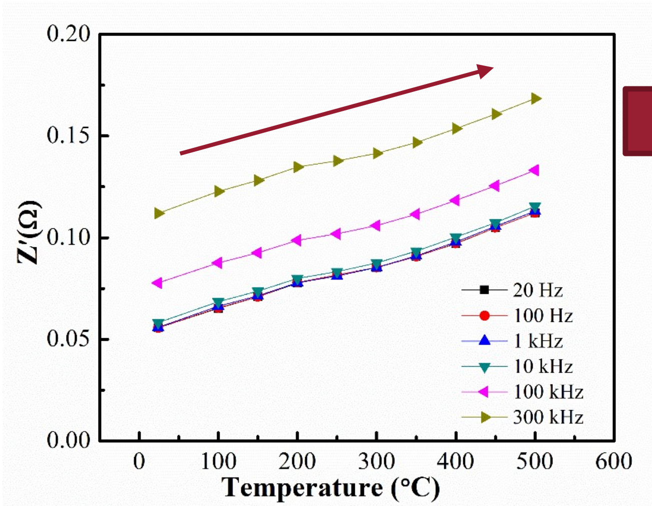
Solution: Increase grain size prior to high temperature sensor usage

Stable Electrical Behavior for Printed Sensors

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

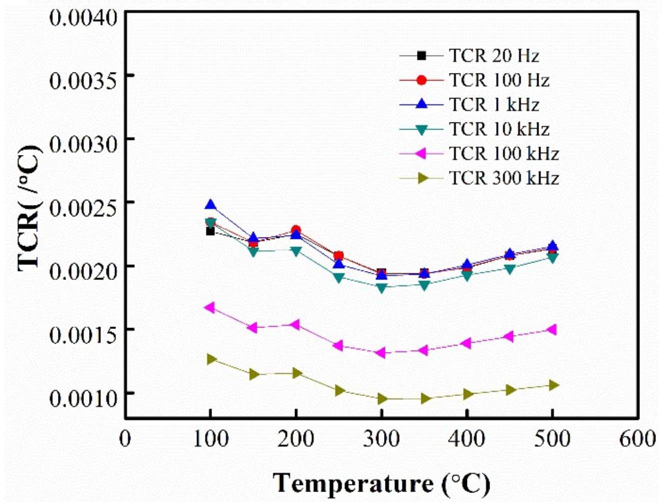


Solution:
Tailored micro-structure for stable electrical behavior of sensors



Increases with T

Positive TCR

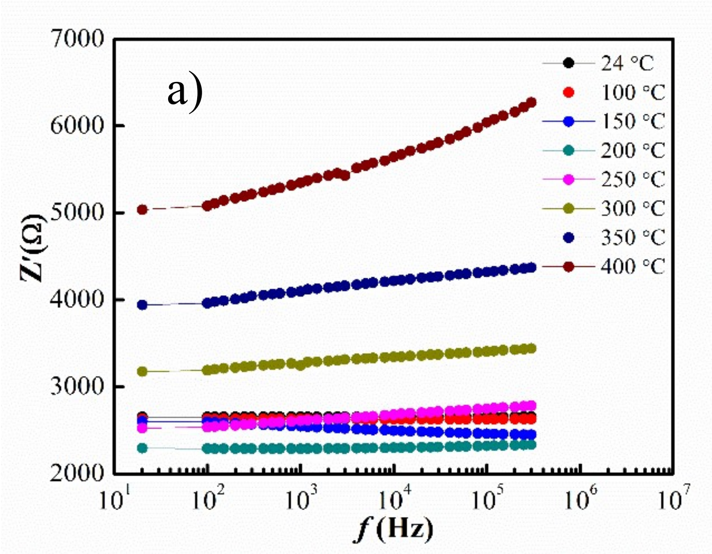


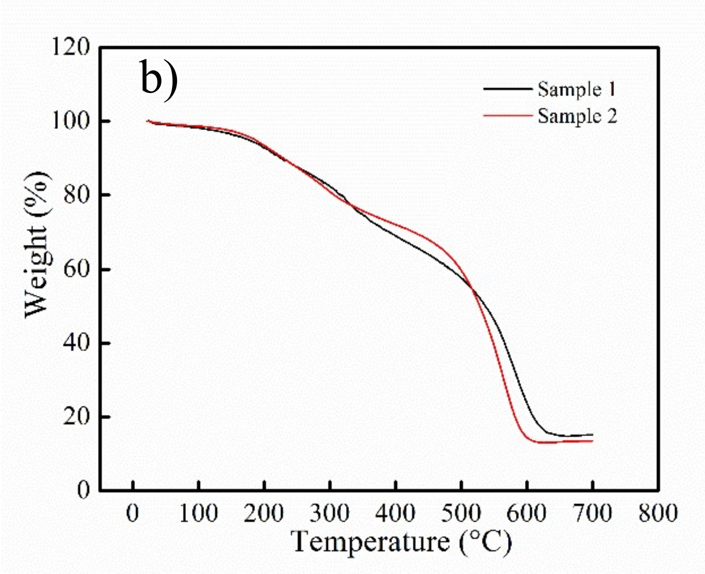
Key Conclusions

- ❑ Electrical resistivity can be tailored by controlling the microstructure of the printed film
- ❑ Ag undergoes minimal oxidation up to 500 ºC
- ❑ Silver is a potential material candidate for room and high temperature sensor application

M. T. Rahman et al., J. Appl. Phys. Vol. 120, Issue 7, 2016

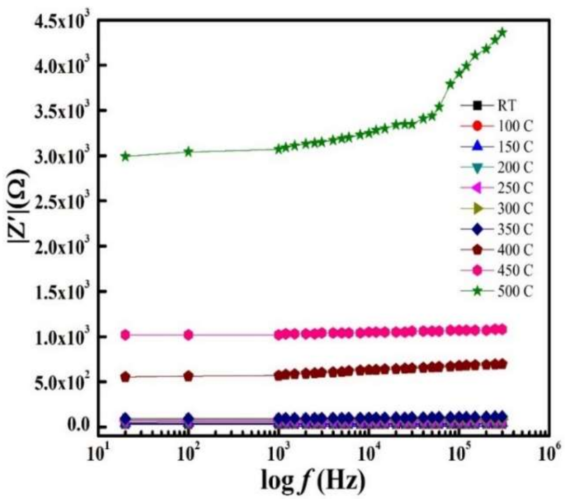
CNTs as a Potential Sensor Material

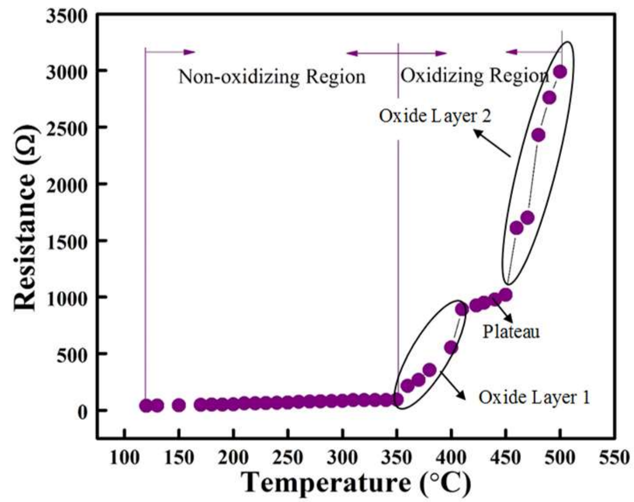


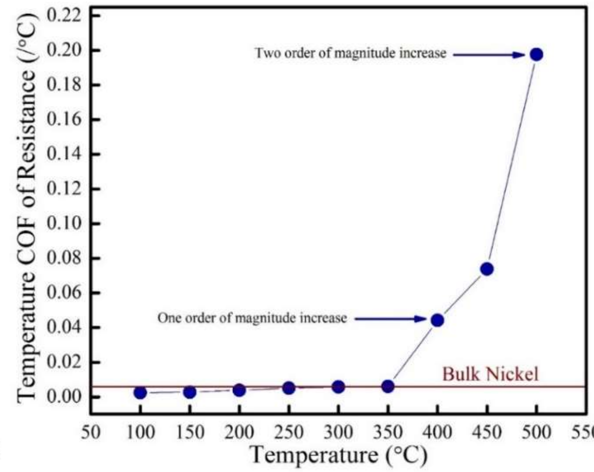


- ❑ Impedance analysis TGA was performed to understand the weight loss percentage of the CNTs.
- ❑ Use of CNTs challenging due to mass loss and high impedance

Ni Nanoparticle Films as a Potential Sensor Material







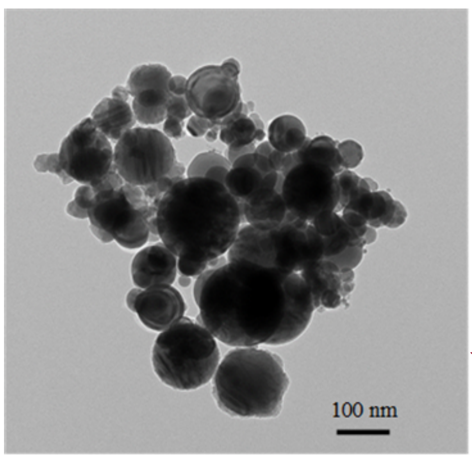
- TCR of Ni increases significantly beyond 350 °C indicating an onset of oxidation
- Ni shows a two stage oxidation behavior, with accelerated oxidation beyond 450 °C

Use of Nickel NP films challenging beyond 350 °C due to oxidation

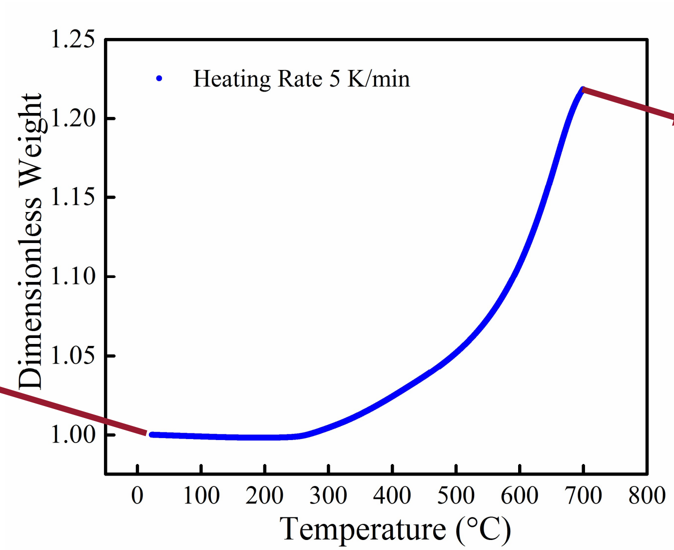
Publication under preparation

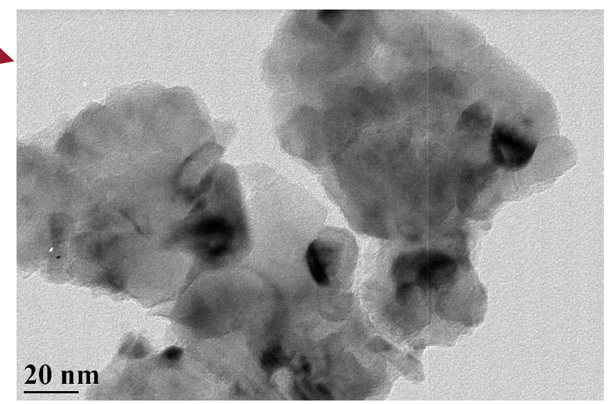
NiCr Nanoparticles as a Potential Sensor Material

- Bulk NiCr (>10 wt% Cr) is a highly oxidation resistant alloy and has been used as resistive heating element for over a century
  - Oxide film is several microns thick and predominantly stable Cr_2O_3
- NiCr NPs as potential materials for additive printing of high T sensors
- Thermogavemetric analysis was performed on the NPs at different heating rates up to 700 ºC
- TEM/SAED, XRD analysis performed



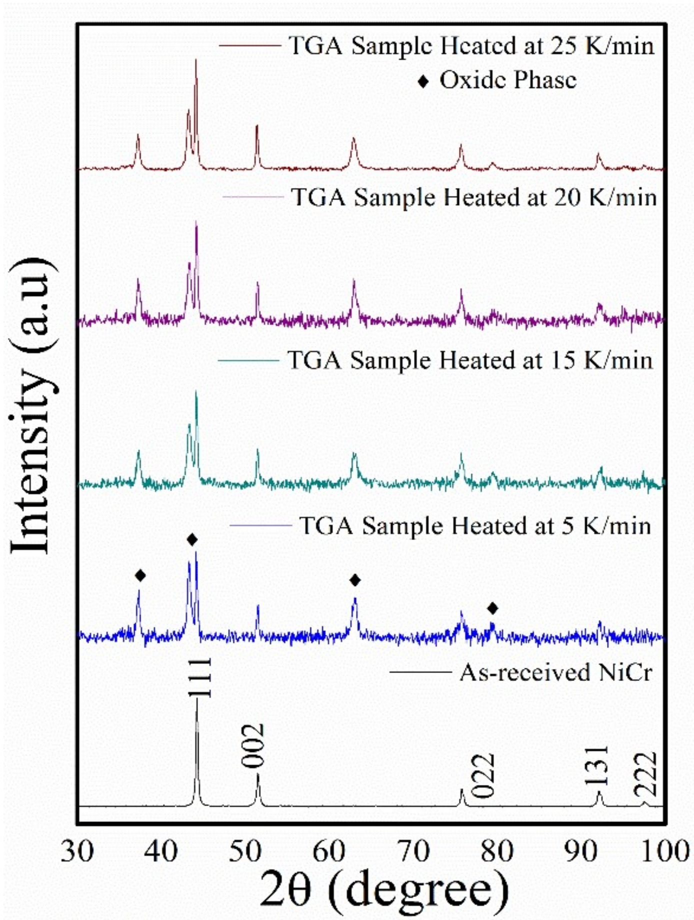
TEM image

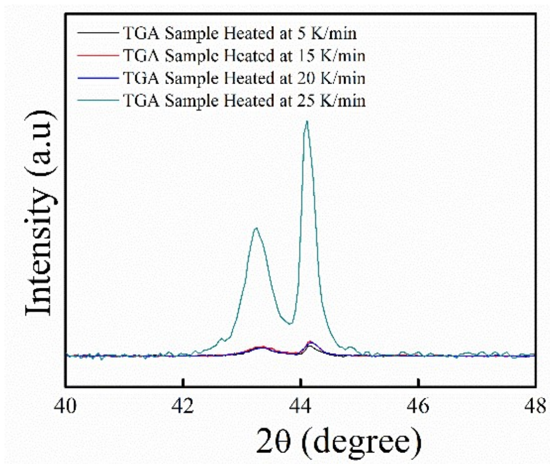


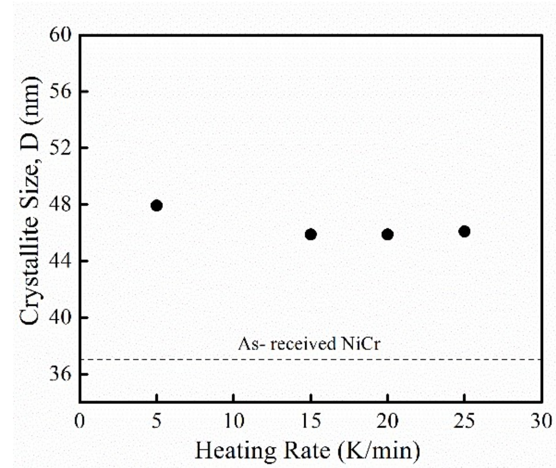


TEM image

X-ray Diffraction (XRD) results for the NiCr NPs at Different Conditions

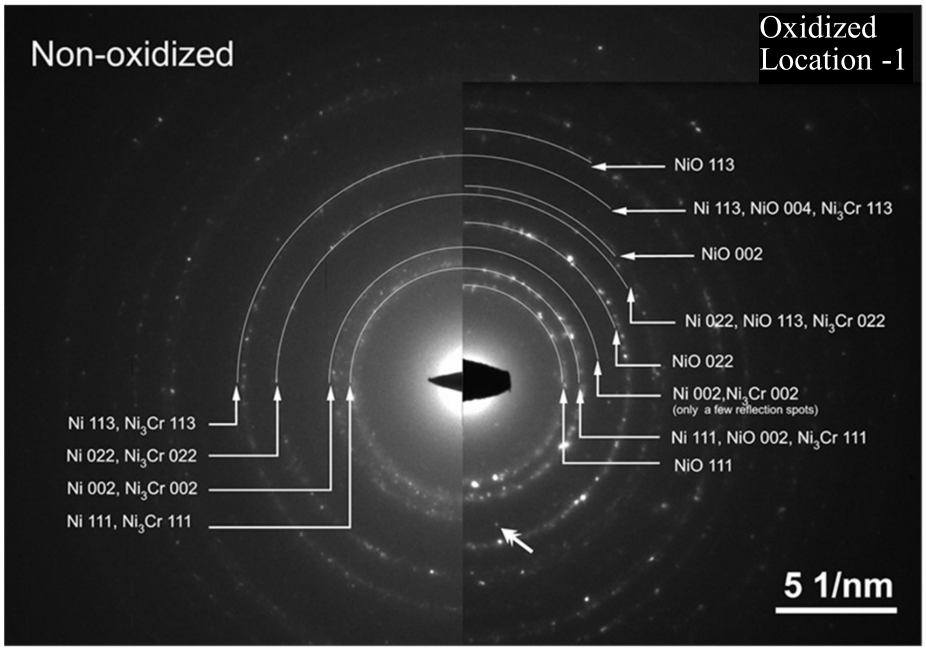


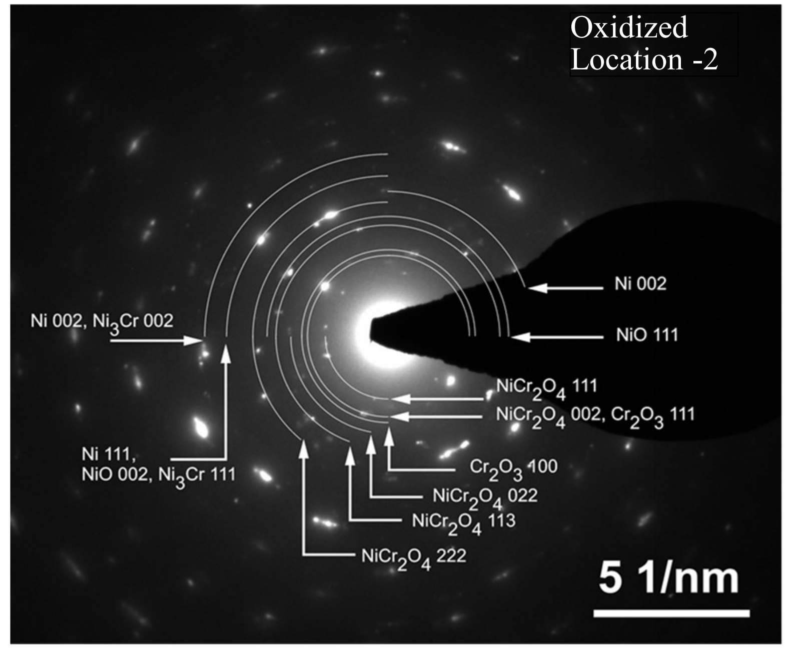




- ❑ The multiple peaks observed indicate that the as-received NiCr NPs had a polycrystalline structure and that no oxide phases were detected.
- ❑ For the oxidized samples, the Ni oxide phases appeared for all the heating rates, along with a distortion of the most intense (111) peak
- ❑ The crystallite size increased for the heat treated samples by about 24%

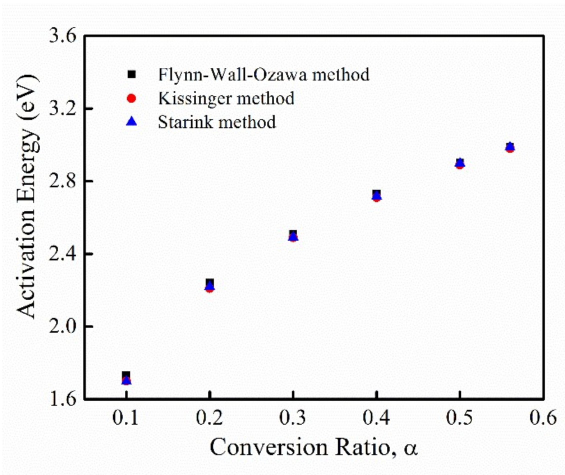
TEM Selective Area Electron Diffraction of NiCr

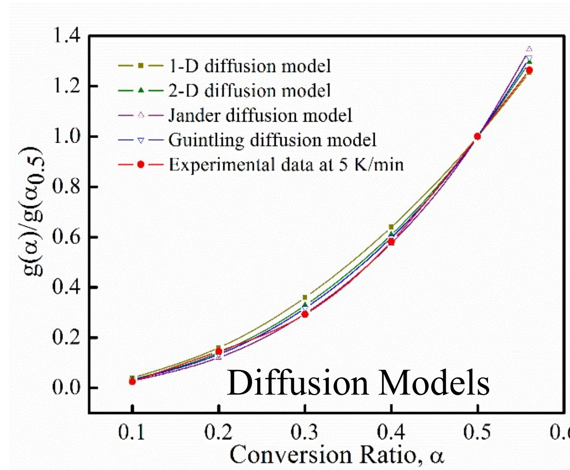




- ❑ Rings represents diffraction different atomic plane.
- ❑ Non-oxidized sample shows strong reflections close to Ni and Ni_3Cr
- ❑ Oxidized sampleshows existence of NiO, Cr_2O_3 and $NiCr_2O_4$

NiCr Oxidation Kinetics by Continuum Model





- ❑ We calculated the activation energy for oxidation
- ❑ We compared the results with diffusion models
- ❑ Experimental data shows good fit with 3-D Jander model shown below:

$$g\ (\alpha) = \left[1 - (1-\alpha)^{1/3}\right]^2$$

NiCr Nanoparticles can act as a back up material for FE sensors

M. T. Rahman et al., J. Phys. Chem. C, 2017, 121 (7), pp 4018–4028

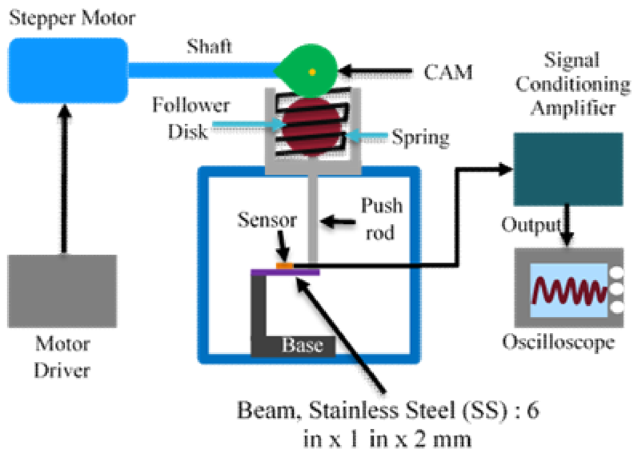
Materials Conclusions

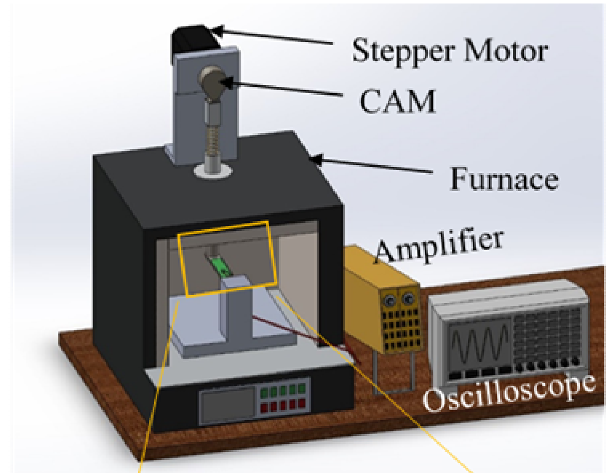
- ❑ Lead material: Ag nanoparticles
- ❑ Back up material: Ni-Cr nanoparticles

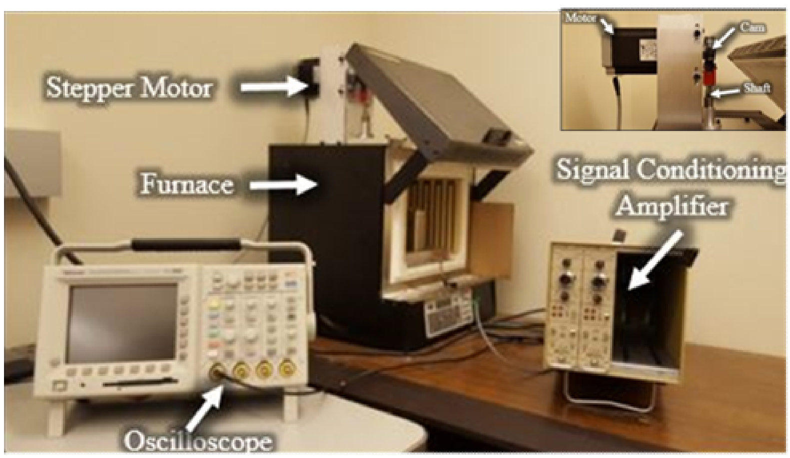
Task 2: Single Sensor Design and Testing

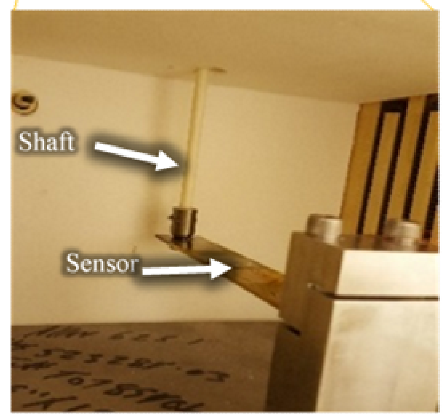
	2015			2016												2017												2018								
	Q4			Q1			Q2			Q3			Q4			Q1			Q2			Q3			Q4			Q1			Q2			Q3		
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
Task 0.0: Feedback to DOE																																				
Task 2.0: Single Sensor Design and Testing																																				

High Temperature Sensor Set Up



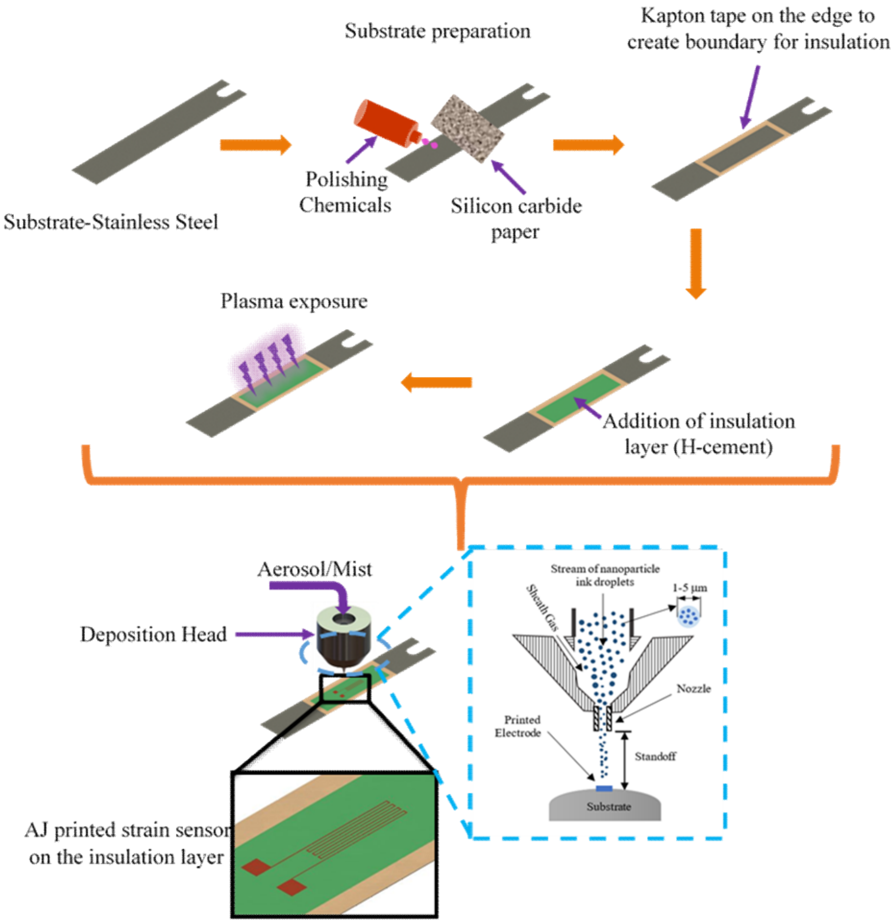


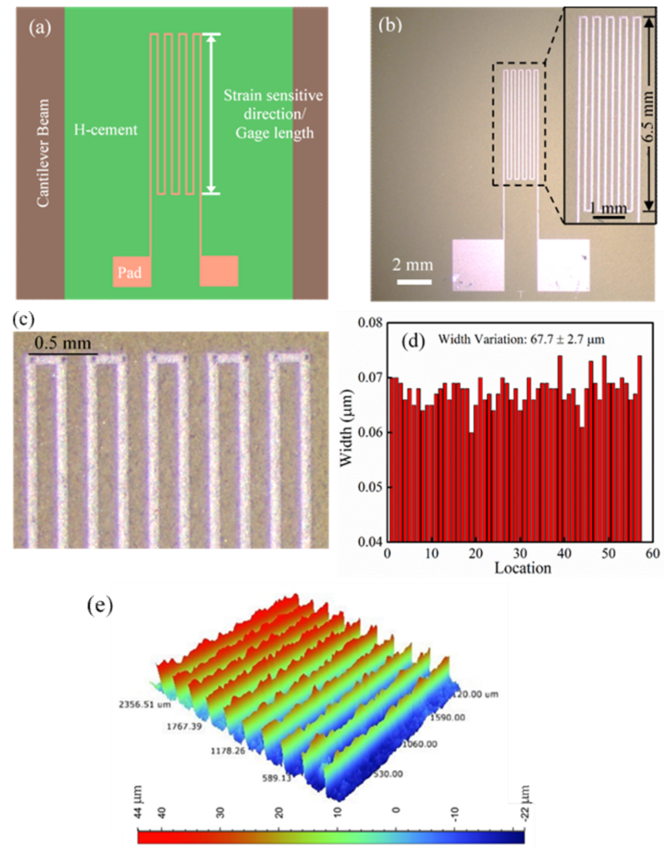




- Able to provide 2000 micro strain on the beam
- Deflection frequency: up to 10 Hz

Sensor Fabrication





Fabrication/manufacturing protocols have been developed with initial sensor fabrication and calibration completed

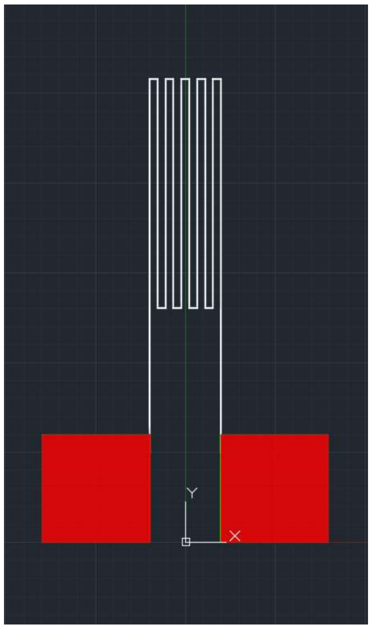
Slide 33

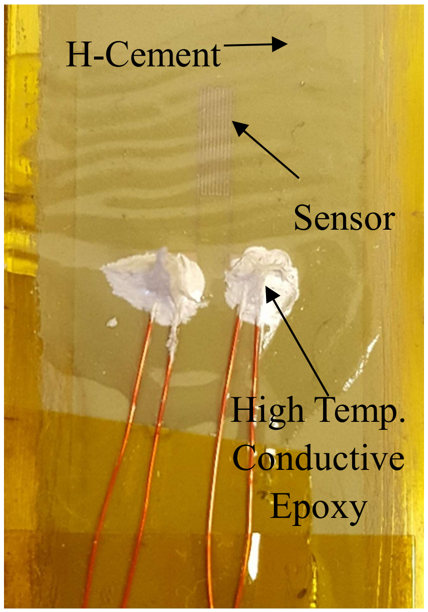
MTR4 insert video

MD TAIBUR RAHMAN, 3/9/2017

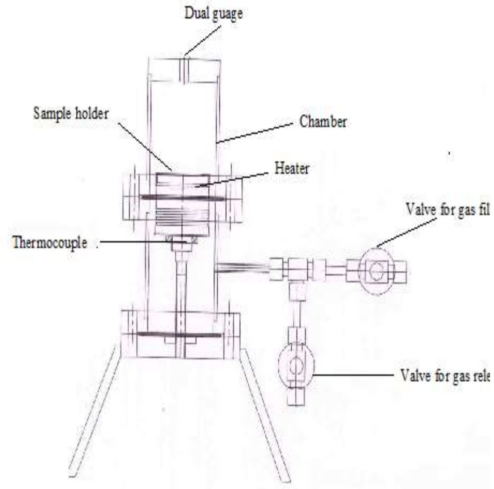
High Temperature Testing Protocols

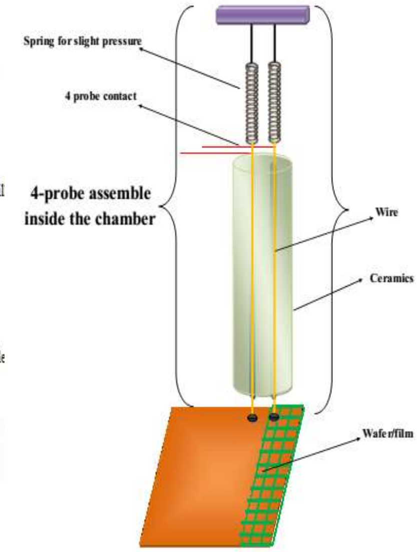
Strain Sensor testing





Pressure Sensor testing





High temperature protocols have been developed for the sensors

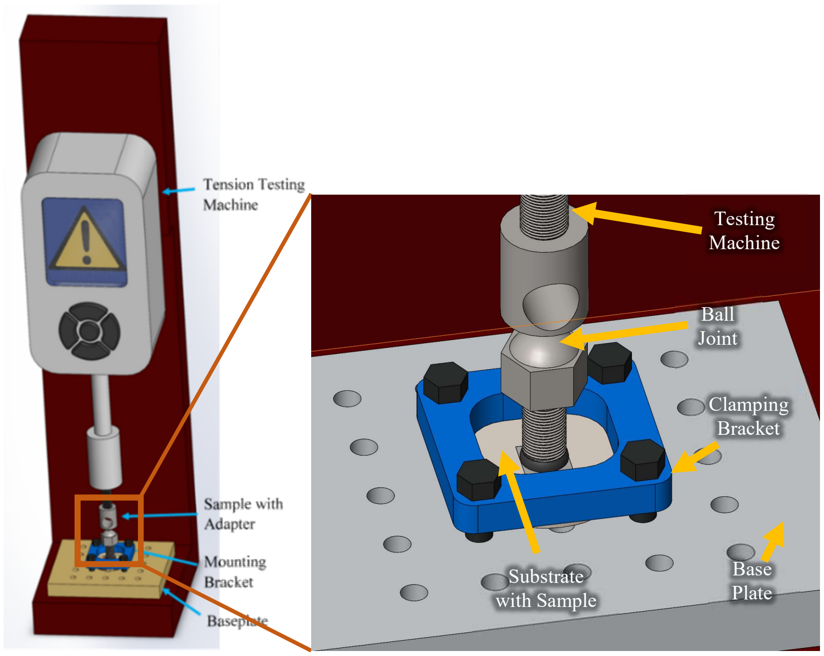
Slide 35

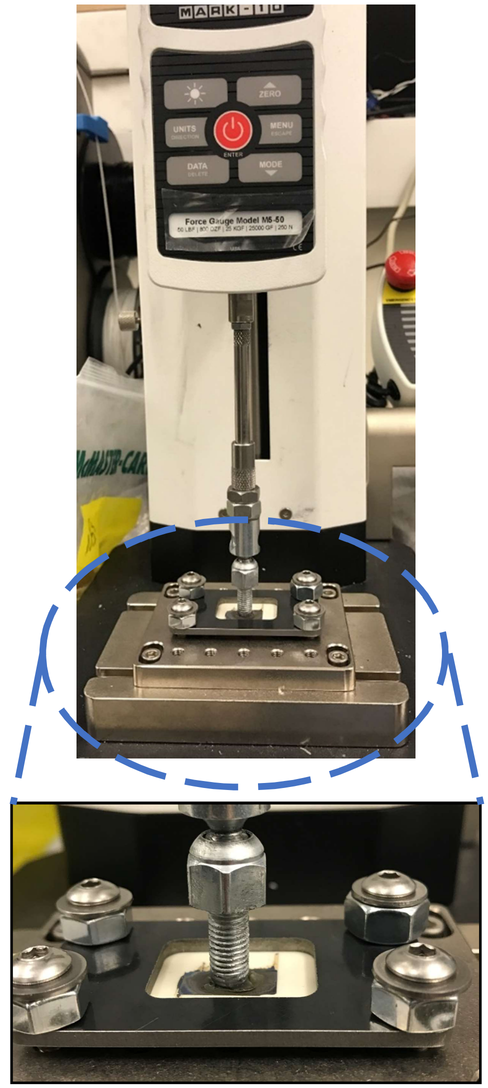
MTR4 insert video
MD TAIBUR RAHMAN, 3/9/2017

Task 3. Reliability Study of the Sensor

	2015			2016												2017												2018									
	Q4			Q1			Q2			Q3			Q4			Q1			Q2			Q3			Q4			Q1			Q2			Q3		
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
Task 0.0: Feedback to DOE																																				
Task 3.0: Reliability of Sensors at High Temperature																																				

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
- After sintering, ball joint is attached to the sample by Cyanoacrylate glue (superglue), allowing for sample to be attached to tension testing machine

Summary of Research

Deliverables of year 1-

- Manufacturing Process Selection ✓
- Material characterization and selection (lead and backup) ✓
- High temperature testing set up ✓
- Reliability Study
  - Work of adhesion test set up ✓

Year-1: Student Training and Research Outcomes

Student Training

1. 2 students pursuing PhD (1 minority student-first generation college graduate)
2. 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, P. Dubey, C. V. Ramana, and Rahul Panat, “3D Printed High Performance Sensors for High Temperature Applications”, *to be submitted to ACS Sensors*.
4. M. T. Rahman, Juan J. Gomez Chavez, P. Dubey, C. V. Ramana, and Rahul Panat, “High temperature stability of 3D printed Ni films”, *in preparation for submission to Journal of Applied Physics*.

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)

2017 Deliverables

- Printing and testing of reliable high temperature sensors
- Design and fabrication of workable antenna at high temperatures
- Wireless system design

	2015			2016												2017												2018								
	Q4			Q1			Q2			Q3			Q4			Q1			Q2			Q3			Q4			Q1			Q2			Q3		
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
Task 0.0: Feedback to DOE																																				
Task 4.0: Wireless System Design and Fabrication																																				
Subtask 4.1. Sensor integration over a substrate																																				
Subtask 4.2.Design of a Wirelessly-Powered Integrated RF Transceiver																																				
Subtask 4.3: Integration over a Platform																																				

Acknowledgement

- DOE/NETL through grant # DE-FE0026170
- Sydni Credle
- Franceschi imaging center, WSU
- Mr. Bob Lentz, WSU for experimental test set-up