

Passive Wireless Sensors Fabricated by Direct-Writing for Temperature and Health Monitoring of Energy Systems in Harsh-Environments

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Outline

- 1) Background
- 2) Vision of Technology
- 3) Statement of project objective
- 4) Description of team
- 5) Task descriptions (with approach and previous work)
- 6) Important project milestones



Background- Harsh Environment Sensing Needs

- Online monitoring of energy systems in extreme conditions is required for mining/drilling, transportation, aviation, energy, chemical synthesis, and manufacturing applications.
- Harsh-environments include:
 - High temperature (1000°C-2000°C)
 - High pressure (up to 1000 psi)
 - Various pO₂ levels
 - Corrosive conditions (molten inorganics or reactive gasses)
- Ability to monitor:
 - Temperature
 - Stress/strain within energy or reactor components
 - Failure events
 - Overall health



Processing Vision

Item A represents the organic carrier film.

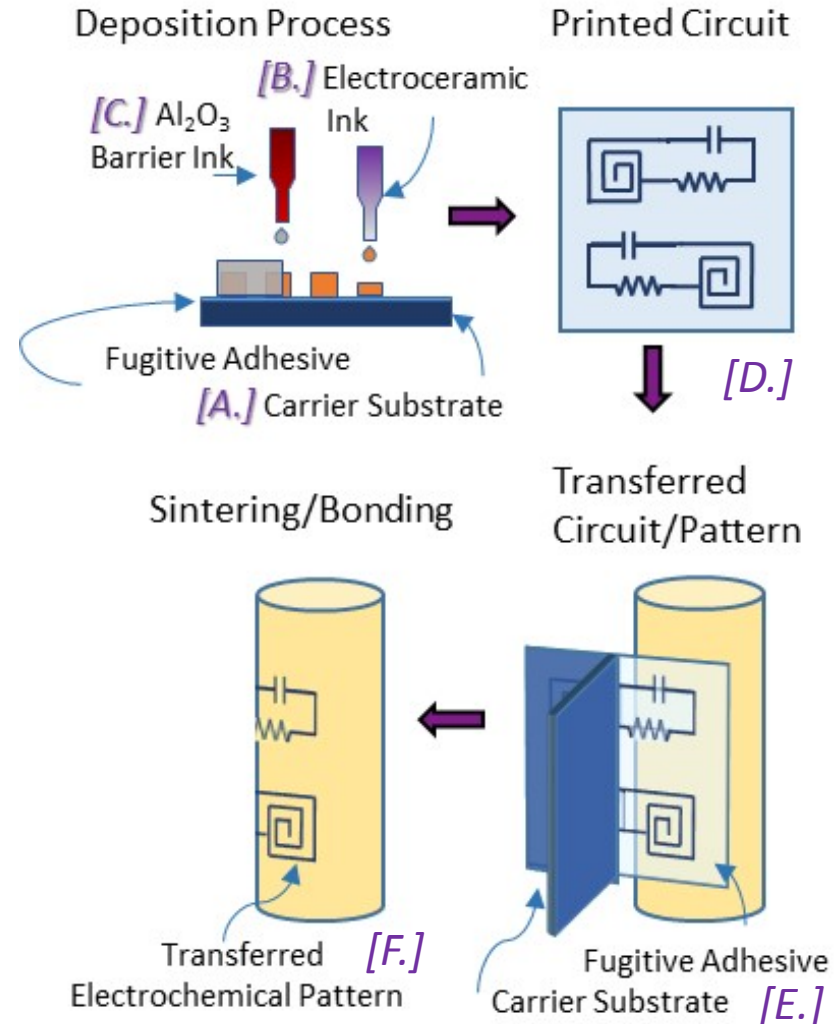
Item B represents the polymer-precursor ink (converts to an electroceramic after heat treatment).

Item C represents a possible barrier layer.

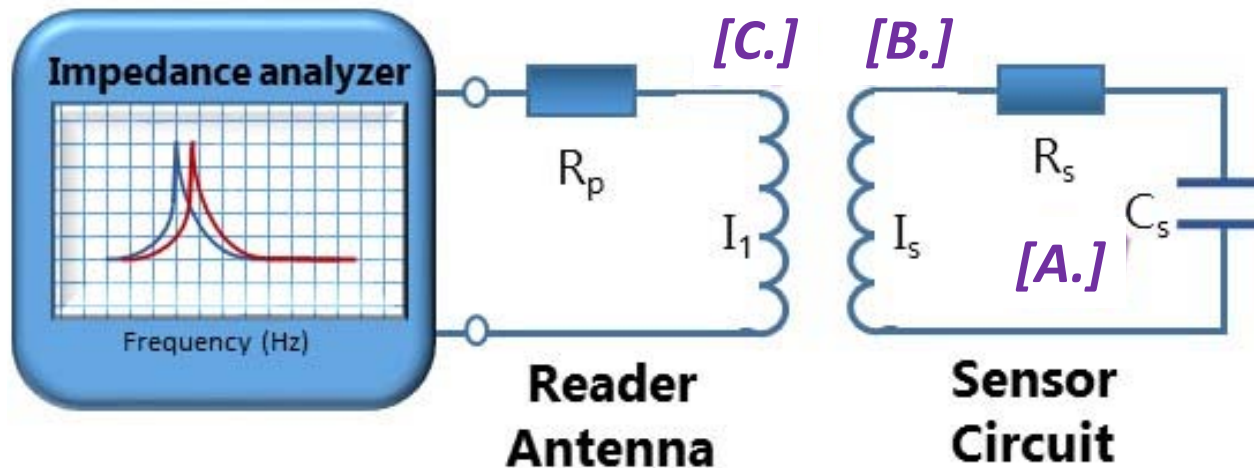
Item D represents printed sensor circuit on the transfer paper.

Item E shows the pattern being placed upon the energy-system component.

Item F represents the pyrolysis of the organic carrier and bonding.



Sensing Vision



Item A represents the LCR sensor and communication circuit.

Item B represents the inductor component (2D spiral) which act as a component for the sensor communication.

Item C represents the reader/powering antenna.

Alteration in the LCR components (due to temperature or strain changes) will result in a shift in measurable parameters (such as resonance frequency profile).



Program Objectives

- 1) Investigate phase formation, sintering/grain growth, and electrical properties of polymer-derived electroceramic composites between 500-1700 °C.
- 2) Define processes to direct-write through ink-jet and robo-casting the electroceramic composites onto oxide and polymer surfaces.
- 3) Develop methods to form monolithic “peel-and-stick” preforms that will efficiently transfer the sensor circuit to ceramic surfaces after thermal treatment.
- 4) Design of passive wireless LCR circuits and receiver (reader) antennas for communication and testing at temperature up to 1700°C.
- 5) Demonstrate the passive wireless sensor system developed for temperature and stress/strain measurements on a SOFC repeat unit and a singular gas turbine blade prototype as example applications.



R&D Team

Dr. Edward M. Sabolsky (WVU Mechanical and Aerospace Engineering) will act as PI of the program (both technical and administrative), and will be responsible for ceramics processing and sensor testing.

Dr. Kostas Sierros (WVU Mechanical and Aerospace Engineering) will lead development of micro-patterning and robo-casting of ceramic materials, and will be the co-developer of the printing inks and direct-writing tasks.

Dr. Daryl Reynolds (WVU Computer Engineering) will lead the electronics design, interfacing and circuitry, in addition to the development of the passive wireless communication and testing.

Dr. Andrew Nix (WVU Mechanical and Aerospace Engineering) 15 years of experience in turbine blade testing, and he will consult on the turbine blade demonstration testing.



***Task 2.0:
Fabrication and Characterization of
Polymer-Derived Electroceramic
Composites.
(Sabolsky)***



Task 2.0 Objectives:

- Investigate phase formation, sintering/grain growth, and electrical properties of polymer-derived electroceramic composites between 500-1700 °C.



Task 2.0 Approach:

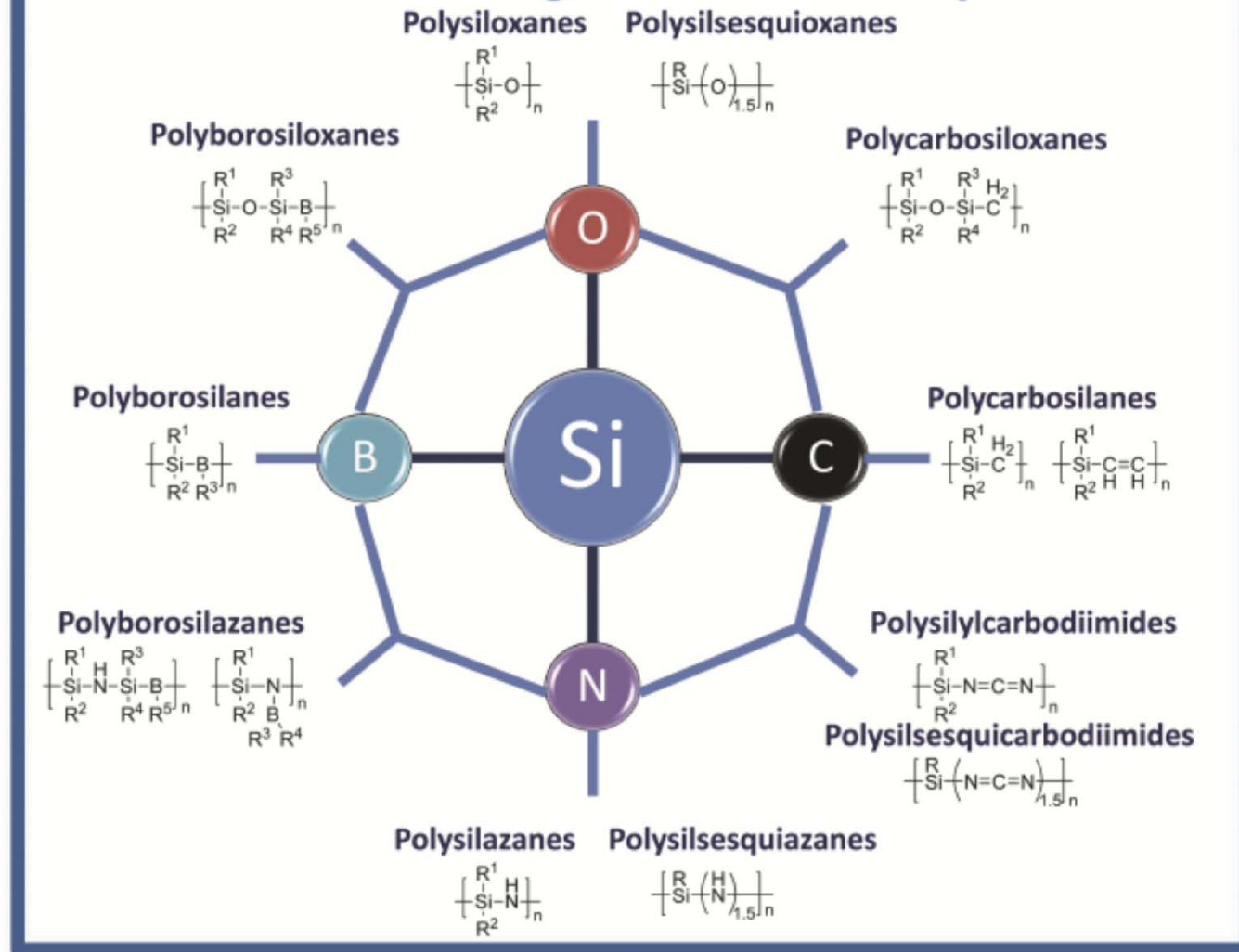
- **Subtask 2.1 Synthesis of Multifunctional Electroceramic Composites through Polymer-Derived Precursors. (Q1-Q3)-**
- **Subtask 2.2 Thermal Processing of Composite Compositions. (Q1-Q3)-**
- **Subtask 2.3 Composite Material Testing and Characterization. (Q1-Q4)-**

Full activity will not initiate until staffing completed in Jan.



Polymer-Derived Ceramics (PDCs):

Preceramic Organosilicon Polymers

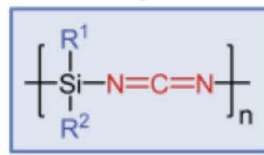
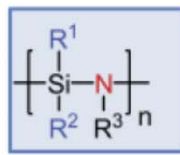
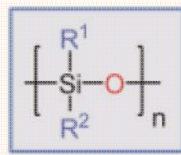
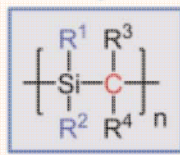
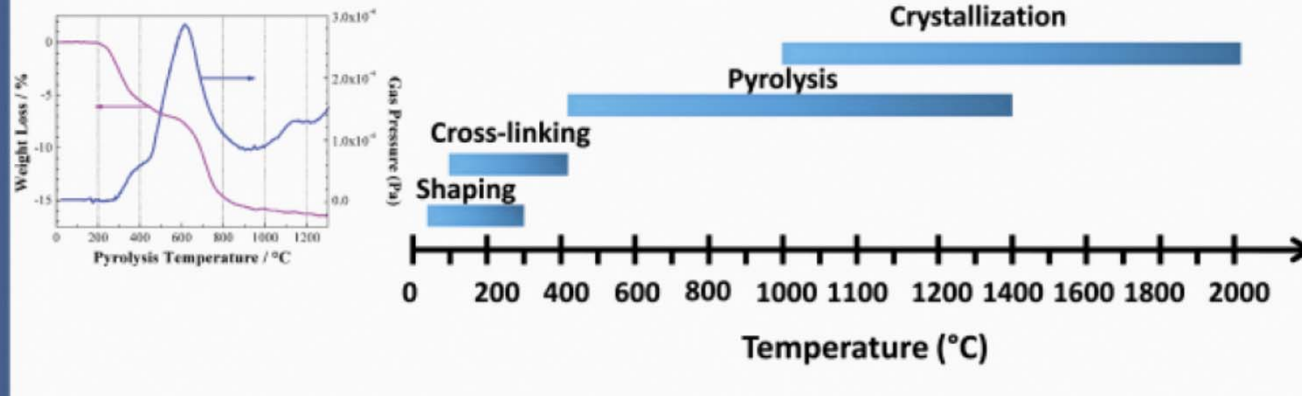


J. Am. Ceram. Soc., 93 [7] 1805–1837 (2010)



Polymer-Derived Ceramics (PDCs):

Polymer to Ceramic Transformation

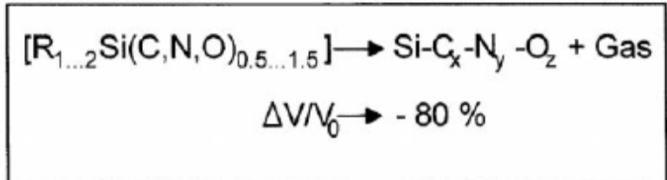
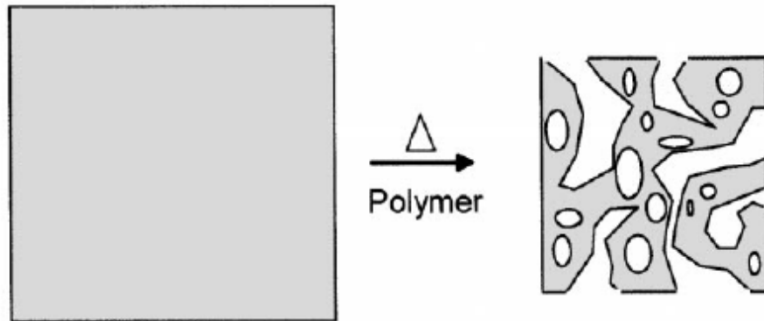


Poly(organocarbosilanes) $\xrightarrow{\Delta T}$ SiC
 Poly(organosiloxanes) $\xrightarrow{\Delta T}$ Si_xC_yO_z

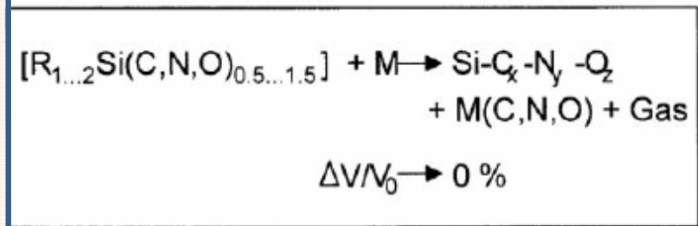
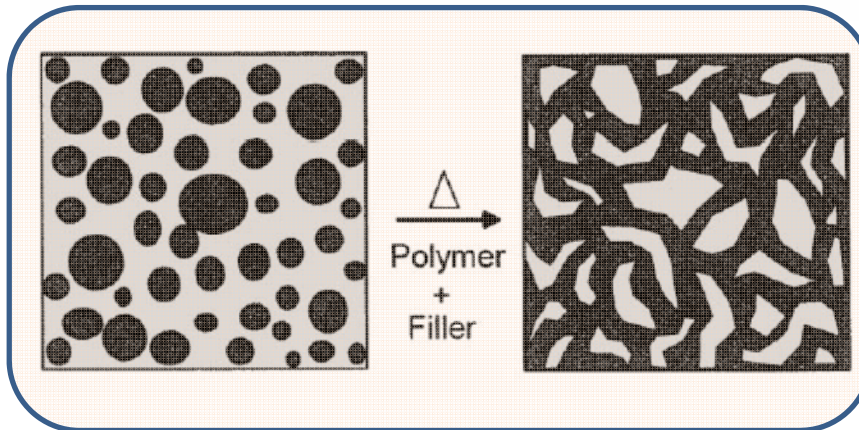
Poly(organosilazanes) $\xrightarrow{\Delta T}$ Si_xC_yN_z
 Poly(organosilylcarbodiimides) $\xrightarrow{\Delta T}$ Si_xC_yN_z



Polymer-Derived Ceramics and Effect of Fillers:



Cracks, porosity, and voids!



with R = H, CH₃, CH=CH₂, C₆H₅, etc.

Inert Filler= additional inorganic particles that **do not react** with polymer as it decomposes.

Active Filler= additional inorganic particles that **react** with polymer precursor.



Active Fillers for PDCs:

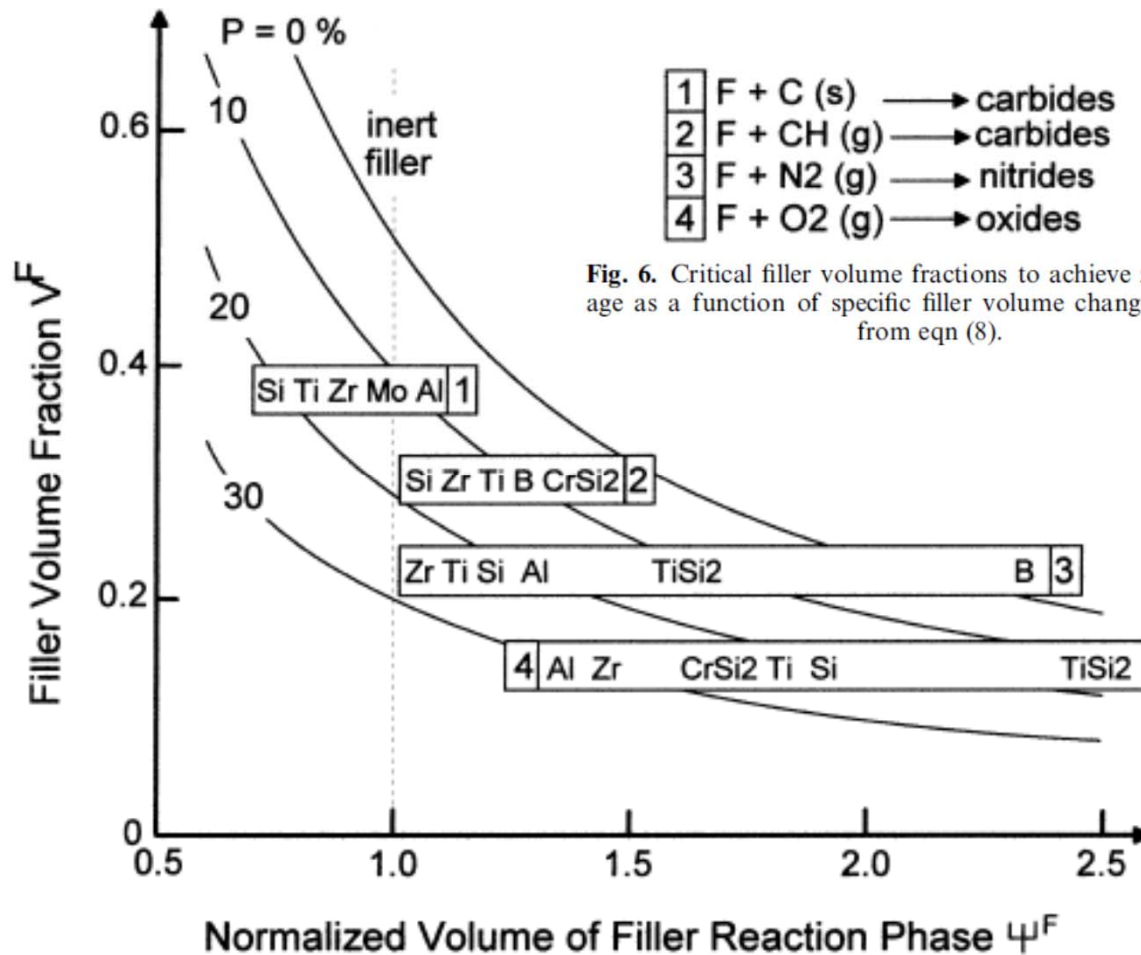


Fig. 6. Critical filler volume fractions to achieve zero shrinkage as a function of specific filler volume change calculated from eqn (8).

Note: Current sensor application does not required full densification!

- Reactive additions may reduce level of shrinkage which could maintain electrical percolation and bonding to substrate.
- Critical balance between transformation content, shrinkage, and printability.



Few Reasons for Oxide and Silicide Additions:

- 1) Highly conductive interconnects can be fabricated
(from metallic-like silicide compositions ($\sigma > 100 \text{ S/cm}$)).
- 2) Silicides are highly resistant to oxidation
(at temperatures up to 1800°C due to a passivation layer).
- 3) Silicides show high chemical stability
(at high-temperature (do not decompose) like many carbides and nitrides in oxygen).
- 4) Silicide/Oxide composites show even higher chemical and microstructure stability.
- 5) Heating elements, glow plugs and igniters composed of Silicide/Oxide composites have functioned in various harsh-environments for $>10,000$ s cycles
(such as those fabricated by Saint-Gobain, Kyocera, NGK...)



***Task 3.0:
Direct-Writing, Patterning, and
Transfer of the Sensor System.
(Sierros/Sabolsky)***



Task 3.0 Objectives:

- To define processes to direct-write through ink-jet and robo-casting the polymer-derived electroceramic composites onto oxide and polymer surfaces.
- To develop a method to transfer the pattern from an organic film to a ceramic surface and bond after thermal treatment.

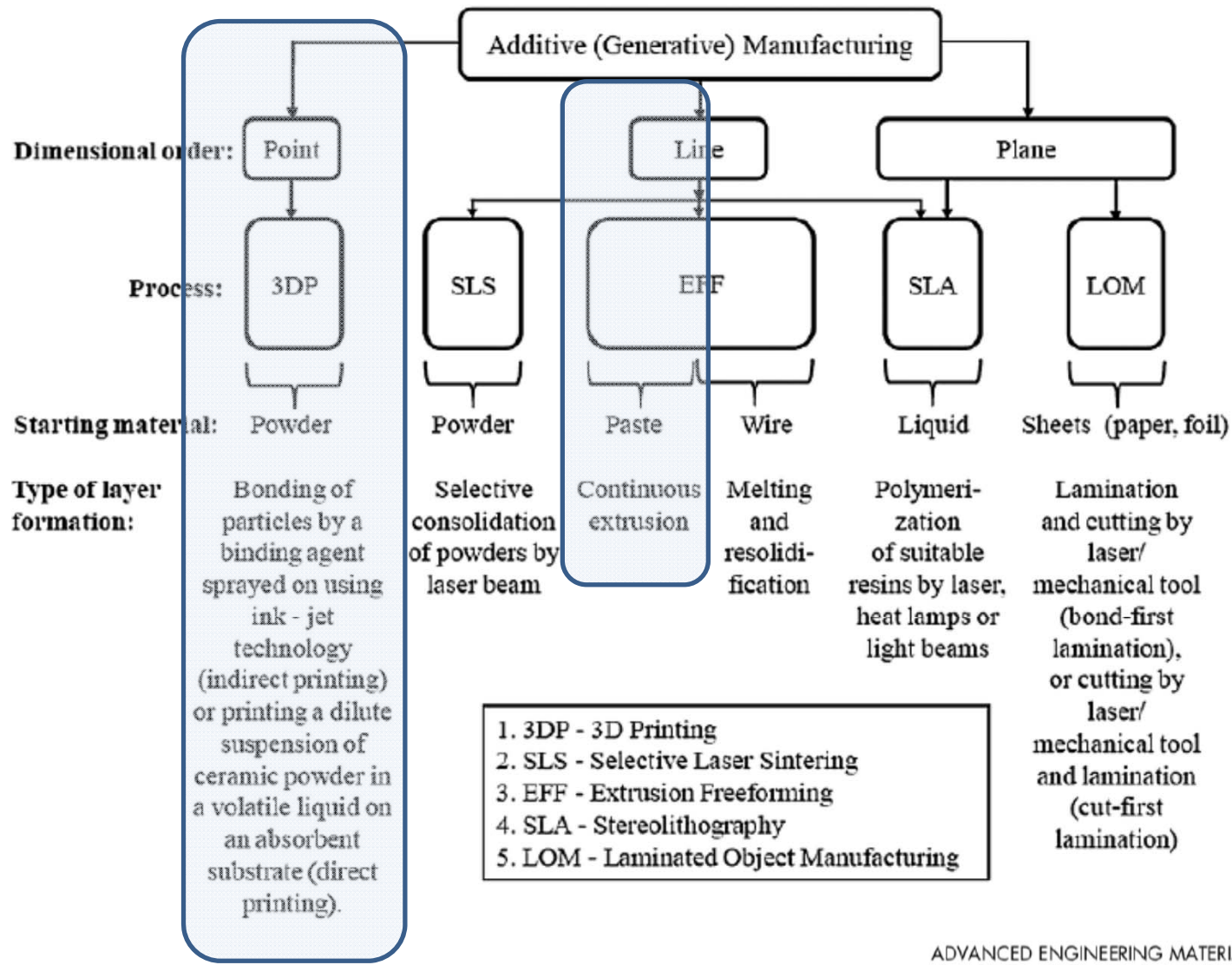


Task 3.0 Approach:

- **Subtask 3.1 Direct-Writing Ink Development. (Q2-Q4)-**
- **Subtask 3.2 Direct-Writing/Patterning and Drying Characterization. (Q2-Q6)**
- **Subtask 3.3 Thermal Processing Development and Structure Tailoring. (Q2-Q5)-**
- **Subtask 3.4 Baseline Sensor Testing and Design Optimization. (Q3-Q8)-**
- **Subtask 3.5 “Peel-and-Stick” Development. (Q3-Q8)-**



Additive Manufacturing of Ceramics:



Shaping of Polymer-Derived Ceramics:

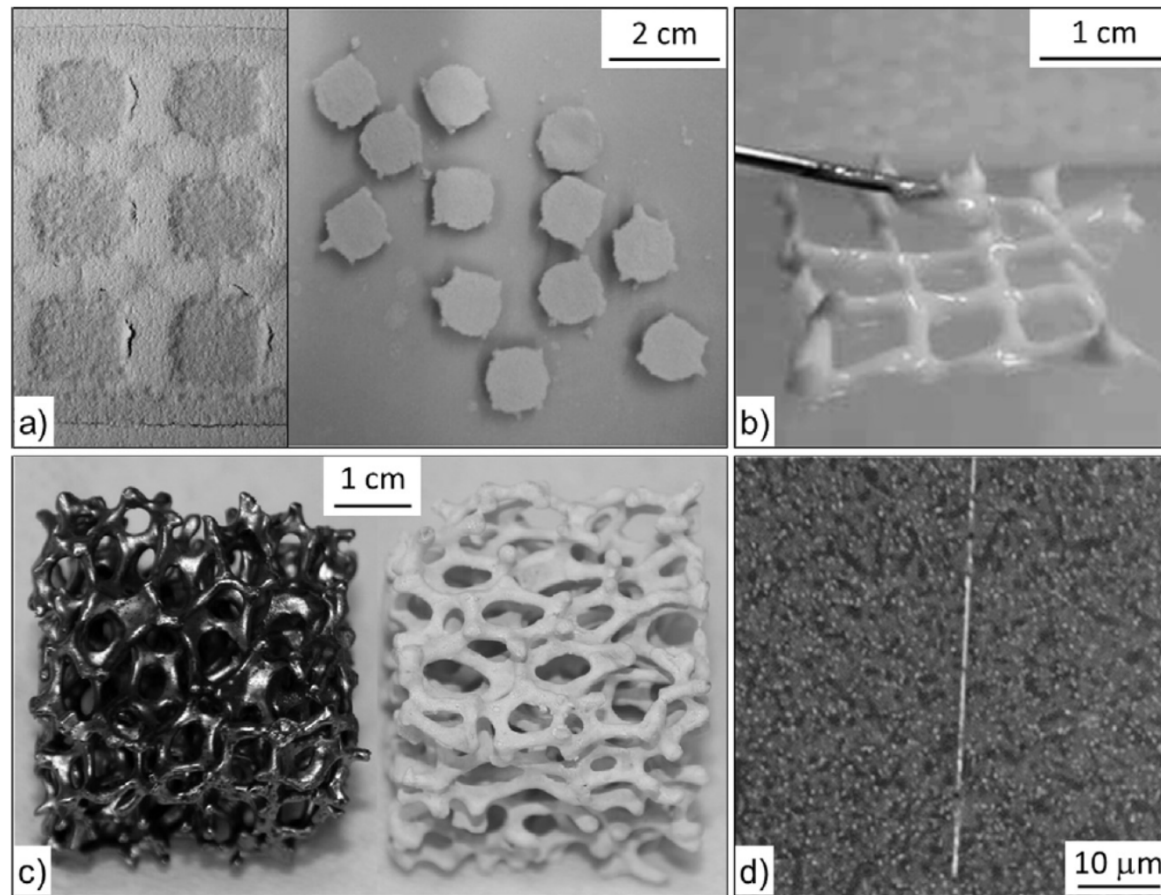


Fig. 7. Images of various components produced by applying different shaping techniques to preceramic polymer plus nano-sized filler mixtures. (a) Small features produced by indirect 3D printing of organic binder droplets onto a powder bed of silicone plus calcium carbonate filler (left: printed parts embedded in the powder bed; right: parts extracted from the powder bed. Before firing. Image courtesy of C. Gomes and J. Guenster, BAM, Germany); (b) paste extrusion through a syringe of a silicone plus calcium carbonate filler (before firing); (c) 2 cm × 2 cm × 2 cm SiC foam block before (left) and after (right) coating by dipping into a silicone plus zirconia suspension (after firing at 1250 °C in air; foams samples courtesy of A. Ortona, SUPSI, Switzerland); (d) Sialon blocks joined by a layer of silicone resin plus γ alumina (bright vertical line) deposited by spraying (after firing at 1550 °C in N_2 ; image courtesy of J. Hellmann, PSU, USA).



Robo-casting of Electroceramic Patterns:

- Robocasting of numerous ink formulations including;
 - ZnO sols
 - Nanoparticle-based Ag
 - TiO₂ aqueous solutions
 - Graphene
 - Nanoparticle C

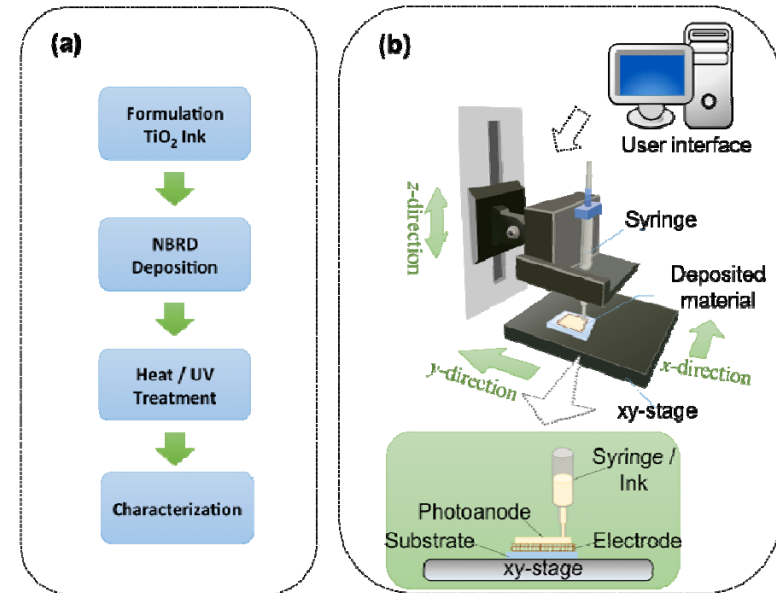


Figure 1: (a) Proposed approach; (b) Nozzle-based robotic deposition (NBRD) system and ink printing.

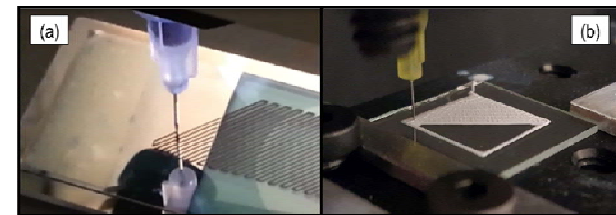
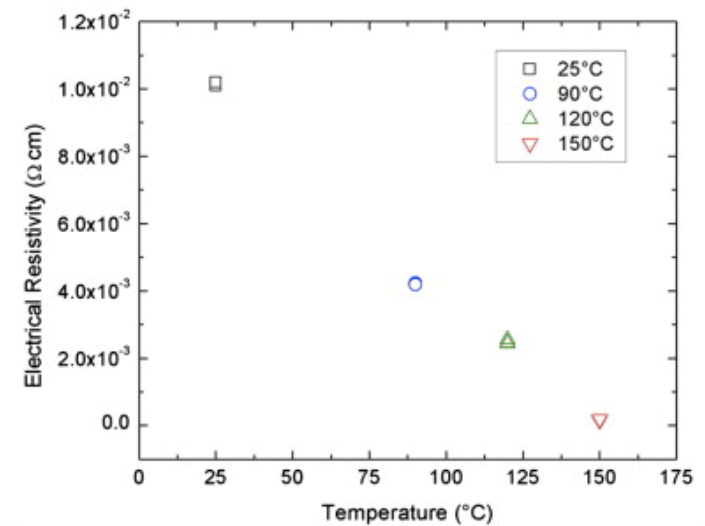
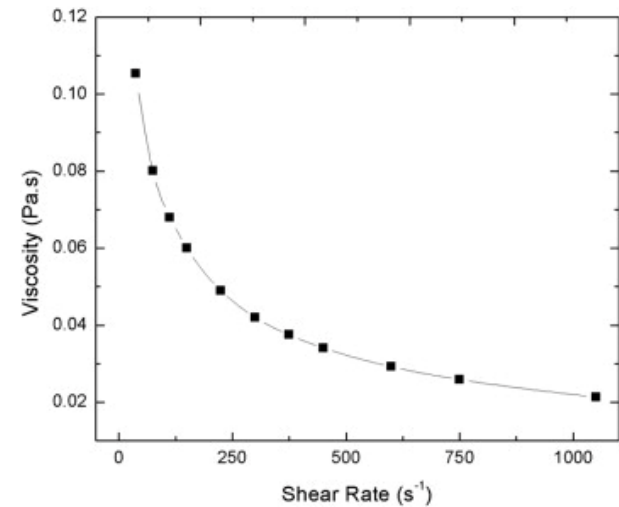
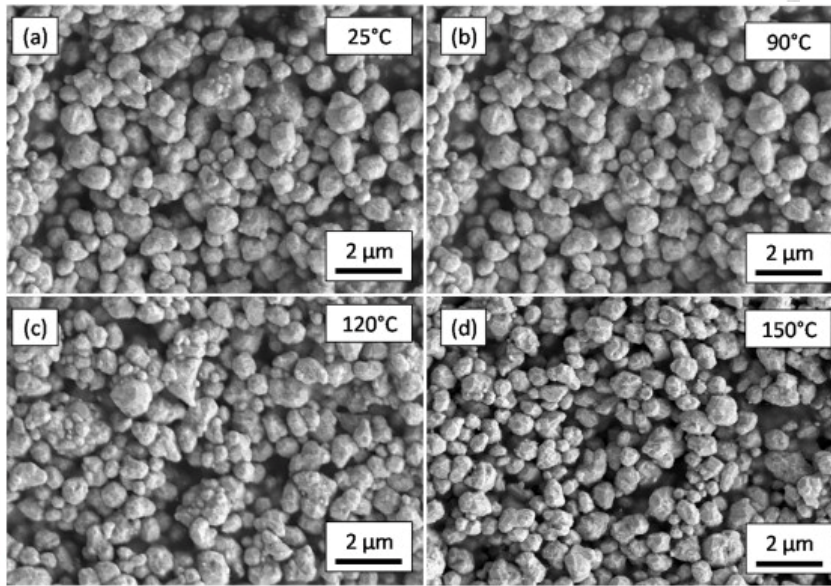
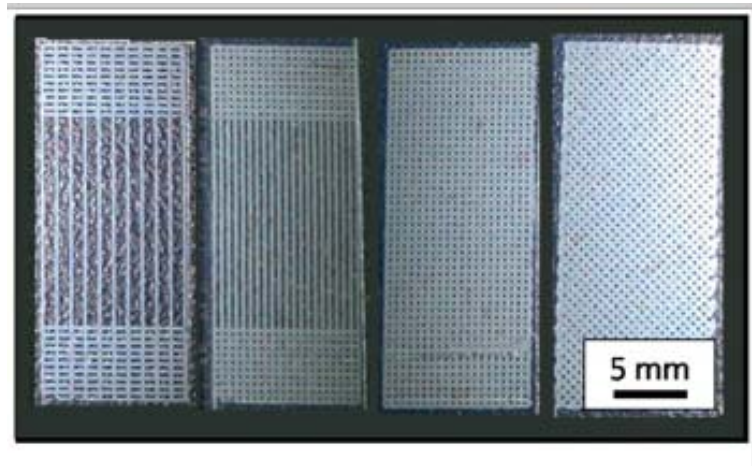


Figure 2: Examples of direct writing at WVU. (a) Ag pattern for flexible electrodes ; (b) TiO₂-TAHL aqueous film.

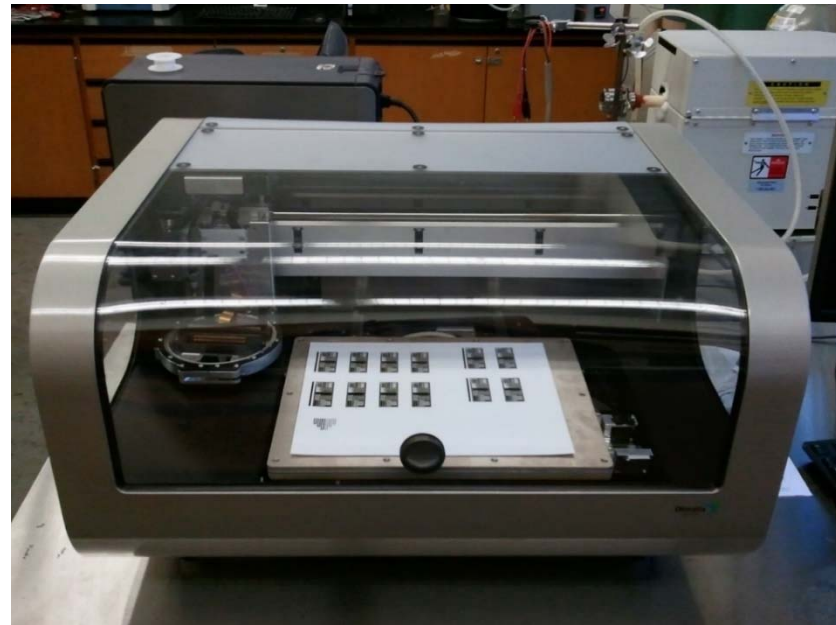


Example: Robo-casting of large-area conductive Ag patterns for flexible electrodes:



Sensors and Circuits by Ink Jet Writing:

- Dimatix DMP-2981 uses disposable piezo inkjet cartridge.
- Replaceable small capacity (1.5ml) cartridges.
- Cartridge consists of 16 independently controllable nozzles which allow for 10 pl drop size.
- Deposits nano-suspensions, organic fluids or metal salt solutions.
- <20 cP viscosity is targeted for printing with ink jet.



Potential Issue ⇒ **Achieving proper kinematic rheology criteria with PDC precursors.**

$$\text{Weber}(W) = \frac{\text{Internal Forces}}{\text{Surface Tension}} = \frac{\rho L v^2}{\sigma} < 35$$

$$Z = \frac{\sqrt{\text{Tension} \times \text{Internal}}}{\text{Viscous Forces}} = \frac{\sqrt{\rho \sigma L}}{\mu} < 25$$

ρ = density
 v = drop velocity
 σ = surface tension
 L = nozzle diameter
 μ = ink viscosity



Transfer of Patterns to Energy Component:

- Process has been demonstrated for sensor components using Ag, Ni, and oxide inks.
- **Potential issues:**
 - Re-dispersion of aqueous inks with water-release mechanism.
 - Surface roughness and porosity effects on bonding (during release and final sensor bonding).
 - Effects of thermolysis on microstructure and sensor electrical properties (and requirements for in-service firing).



***Task 4.0:
Passive Wireless Communication
Circuit Design and Testing.
(Reynolds)***



Task 4.0 Objectives:

- To design and model a passive wireless LCR circuit and receiver (reader) antennas for communication.
- To fabricate and test the sensor design and circuit at room temperature and up to 1700°C.



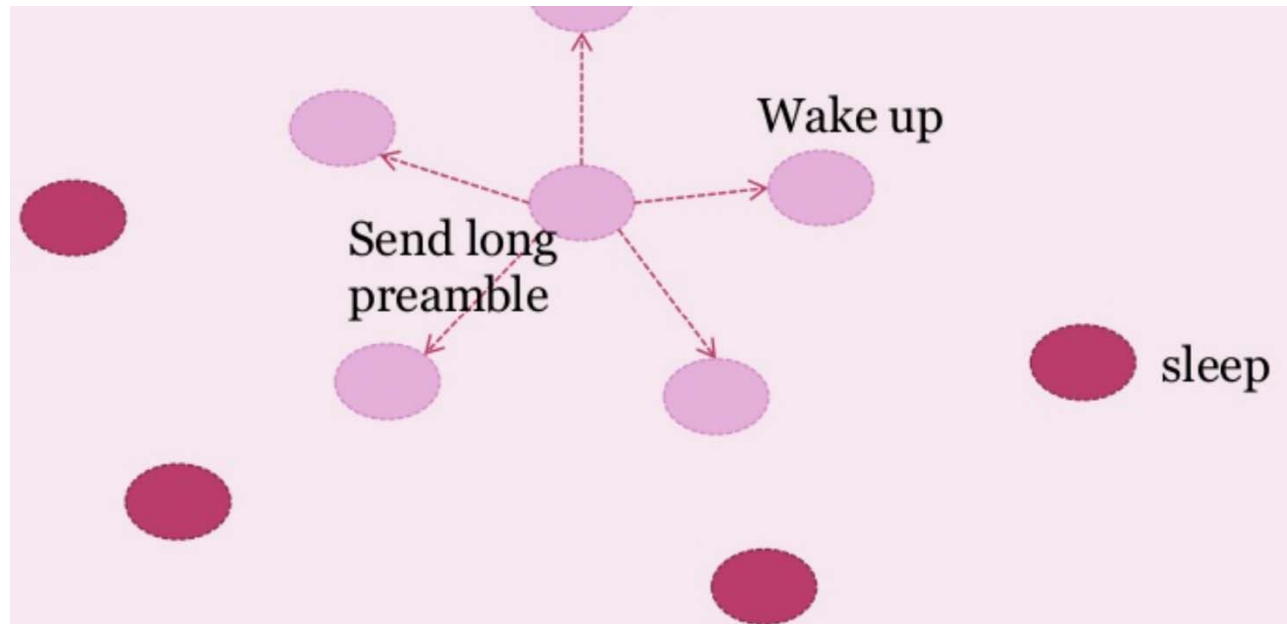
Task 4.0 Approach:

- **Subtask 4.1: Passive Wireless Communication Circuit Design and Testing. (Q1-4)-**
- **Subtask 4.2: Circuit Fabrication and Testing at Lower Temperatures. (Q3-9)-**



Reynolds Group Previous Work I/III

Wake-up signaling for wireless sensor networks:

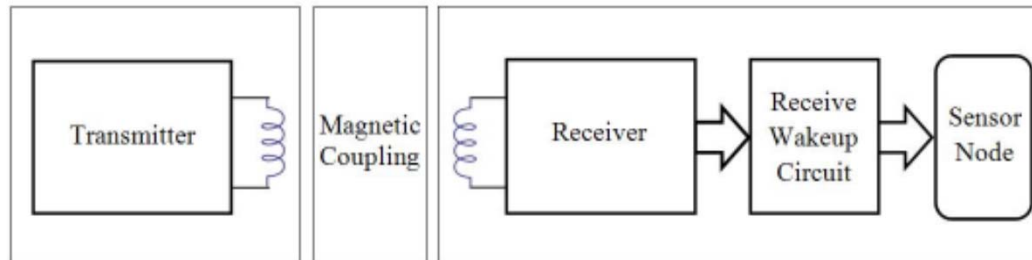


Conventional approach: periodic polling of the communication channel;
consumes lots of energy



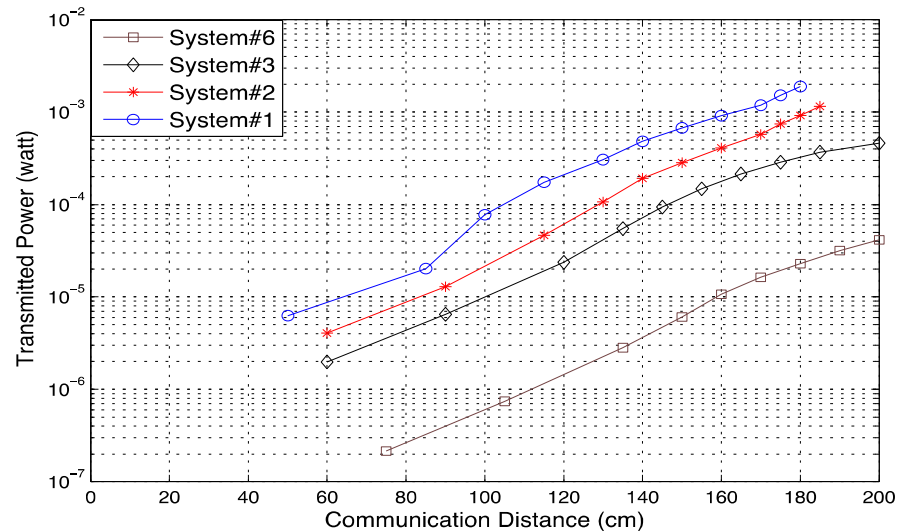
Reynolds Group Previous Work II/III

Our approach: ultra-low power magnetic coupling for wakeup



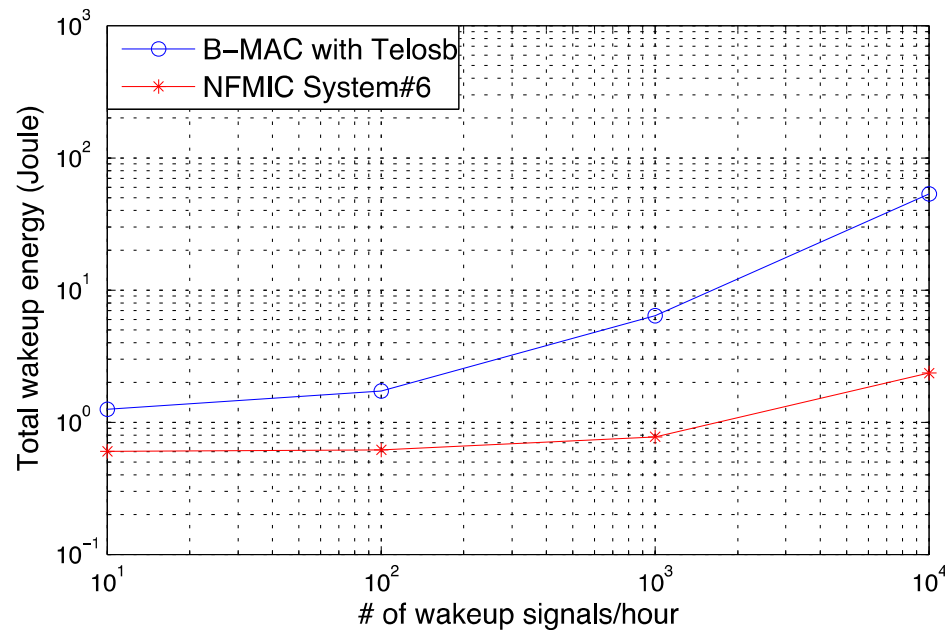
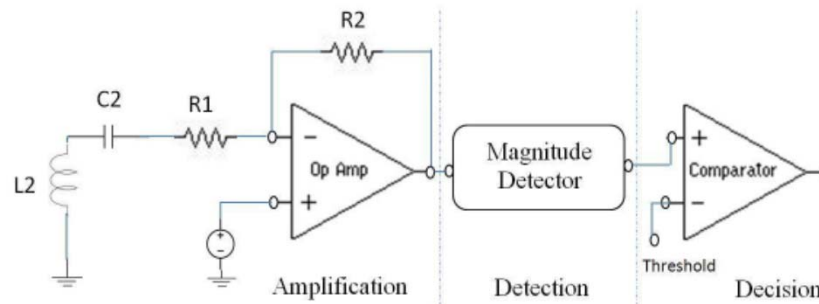
- Considered coil gauge, resistance, turns, diameter, etc.

Coil Type	TX Coil	RX Coil	Resonance Frequency
System#1	TC_1	TC_1	60 khz
System#2	TC_3	TC_1	60 khz
System#3	TC_4	TC_1	60 khz
System#4	TC_4	TC_2	60 khz
System#5	TC_5	TC_1	100 khz
System#6	TC_5	TC_2	60 khz



Reynolds Group Previous Work III/III

With low-complexity, low-power circuitry, we achieved **order of magnitude improvements in energy efficiency:**

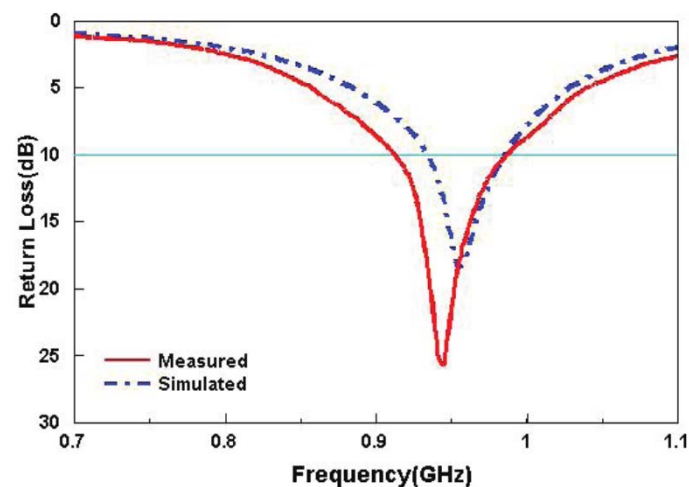
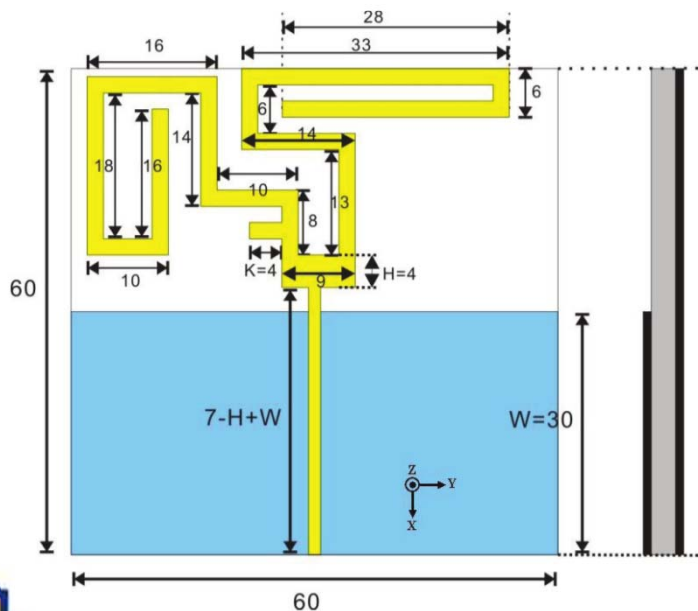


Task 4 Proposed Work

Optimize both sides of the wireless link

- **Sensor side**

- **Frequency considerations: which frequency range(s) penetrate barriers and provide good reader range.**
 - **HF (13.56 MHz):** phone chips, short range, well standardized
 - **UHF (902-928 MHz):** good range, small antennas, standards exist
- **Optimizing antenna configurations:**

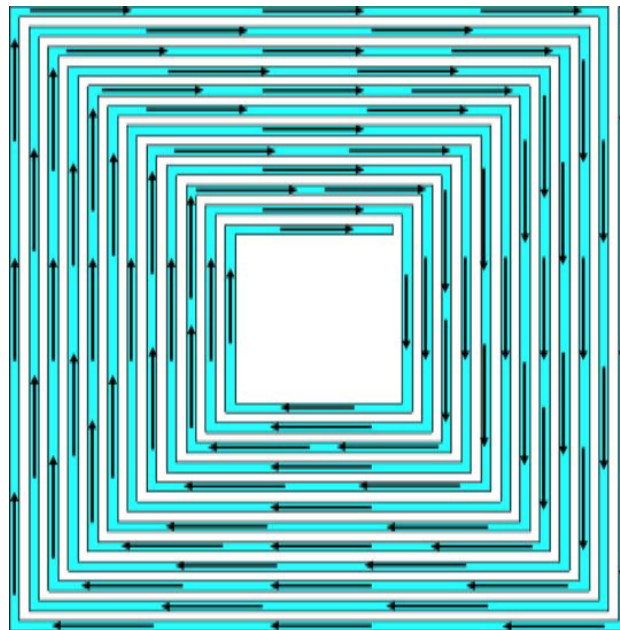


(Chen, et al, IEEE Antennas and Propagation Magazine, 2013.)

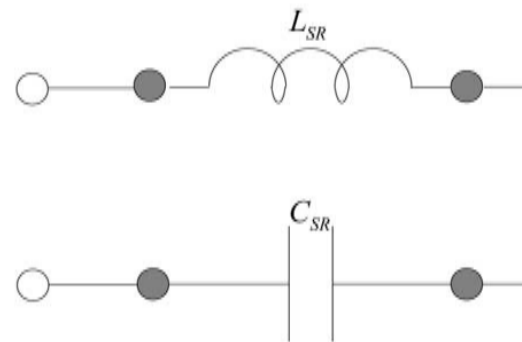


Task 4 Proposed Work

- Ring Resonators: closed or open rings in a dielectric ceramic matrix
- Behaves like an LC resonant circuit
- Can we achieve resonance in UHF band?
- What kind of range will be achievable?



a)



b)

(Bilotti et al, *IEEE Trans. Antennas and Propagation*, 2007)



Task 4 Proposed Work

- Reader side

- Goals

- Good read range
 - Ease of use
 - Cloud connected: automatic data upload; automatic event messaging
 - Reasonable cost

- Option 1: Modify off-the-shelf UHF readers

- Run Windows Embedded
 - Highly capable
 - Expensive: \$2,500+

- Option 2: UHF RFID Phone dongle

- Inexpensive: \$200
 - 1m read range



Task 4 Proposed Work

- Option 3: Construct our own reader:
 - High-Gain Antenna
 - Single Board Computer
 - Display
 - Housing
 - Cost: < \$1000



***Task 5.0:
Implementation of Passive Wireless
Sensors in Harsh-Environments.
(Sabolsky/Nix/Reynolds)
(Nexceris/GE)***



Task 5.0 Objectives:

- To investigate the *passive wireless sensor system developed (and method of transferring sensor system) for temperature and stress/strain measurements on:*
 - *SOFC repeat unit (with Nexceris LLC)*
 - *Singular gas turbine blade prototype as example applications (with GE Global Research)*



Task 5.0 Approach:

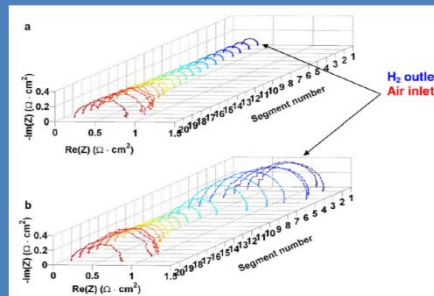
- **Subtask 5.1 Performance Evaluation of Passive Wireless Sensor System at High Temperature (Q4-Q11)-**
- **Subtask 5.2 Wireless Concept Demonstration for SOFCs (Q10-Q12)-**
- **Subtask 5.3 Wireless Concept Demonstration for Turbine Blades (Q10-Q12)-**



Solid-Oxide Fuel Cell Demonstration:

Instrumented PEM Fuel Cells

- Sensors used to understand temperature and fuel/oxidant composition.
- Used in polymer-based fuel cells.



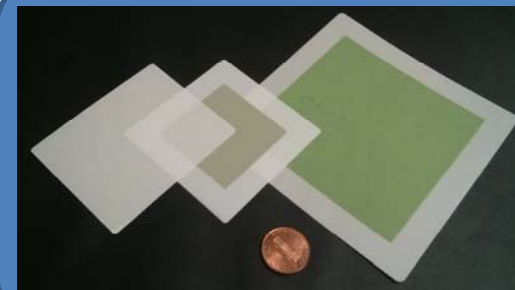
Instrumented PEM Fuel cell (Low-Temp fuel cell)



J. Power Sources.,196 [22] 9451-9458 (2011).

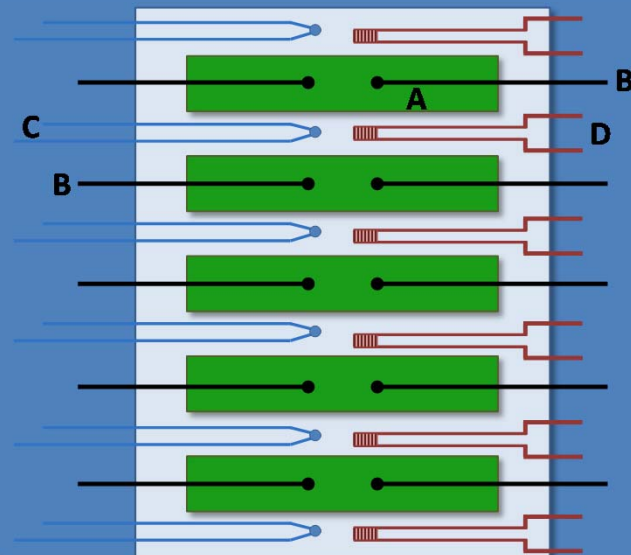
- WVU and Nexceris will fabricate SOFCs to allow for sensor deposition within the anode.
- Temperature will be measured during SOFC loading at 750°C.
- **Potential issue:** communicating with sensors through insulated, heated test chamber, and interference with SOFC output.

Instrumented Solid-Oxide Fuel Cells



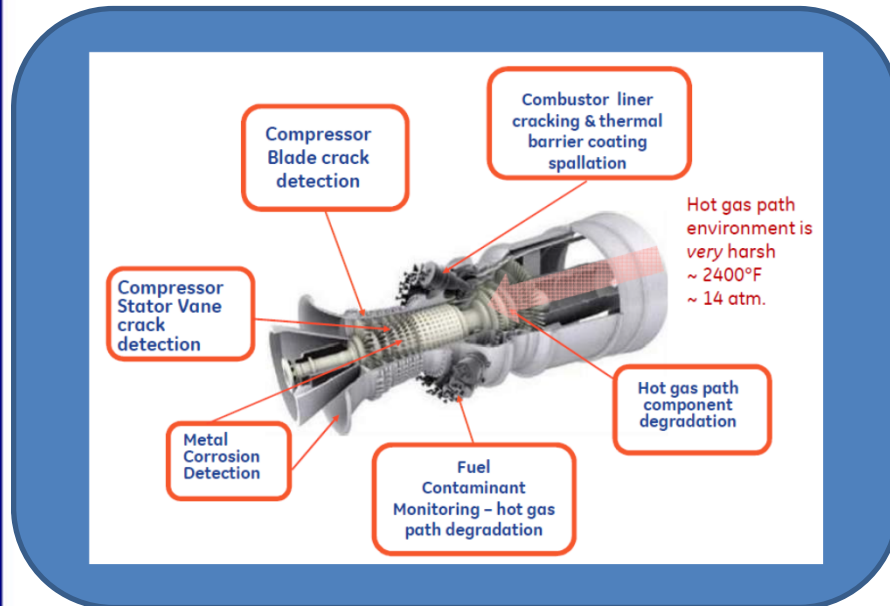
SOFCs fabricated at WVU

TCs and Strain Sensors and Voltage Probes



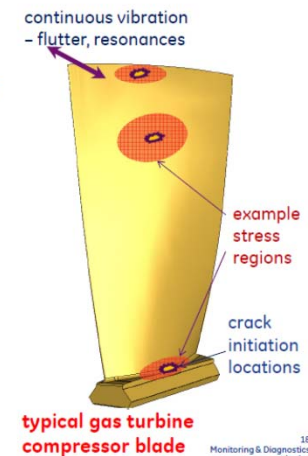
Turbine-Blade Demonstration:

GE Monitoring & Diagnostics



Typical failure drivers & mechanisms for turbine blades

- High cycle fatigue (HCF)
 - Normal corrosion can initiate tiny pits in metal
 - Continuous flexing of blades during operation can grow cracks from pits (high cycle fatigue)
 - When a crack gets large enough, the centrifugal force can pull blade apart (liberation)
- Foreign Object Damage (FOD):
 - Debris gets sucked in and damages blades
- Rubs
- A liberation could cause significant secondary damage -> millions of dollars



- WVU will place an array of temperature (potentially a strain) sensors onto turbine-blade simulant (with TBC) supplied by GE Global.
- Sensors will be monitored on blade at $>1200^{\circ}\text{C}$ (blade will be static, but methods to measure dynamic effects will be considered).
- Targeted Goal:** peel-&-stick on curved structure and sensor functionality at HT.
- Potential issue:** communicating with sensors through insulated, heated test chamber.



Acknowledgments:

- This funding by the U.S. Department of Energy (DOE) under contract DE-FE0001245.
- Funding by the U.S. Department of Energy (DOE) under contract DE-FE0012383.
- We also would like to thank to HarbisonWalker International for their support.
- We acknowledge use of the WVU Shared Research Facilities.
- We also would like the acknowledge Dr. Wei Ding, and Dr. Marcela Redigolo for their assistance.

