# *AOI [1] Advanced Manufacturing of Ceramic Anchors with Embedded Sensors for Process and Health Monitoring of Coal Boilers*

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2022 Crosscutting Research and Advanced Energy Systems Project Review Meeting
SENSORS & CONTROLS PROGRAM
May 4, 2022 (1:00 pm)

Contract DE-FE0031825

# *Initial Acknowledgments:*

**Acknowledgment:** "This material is based upon work supported by the Department of Energy Award Number DE-FE0031825"



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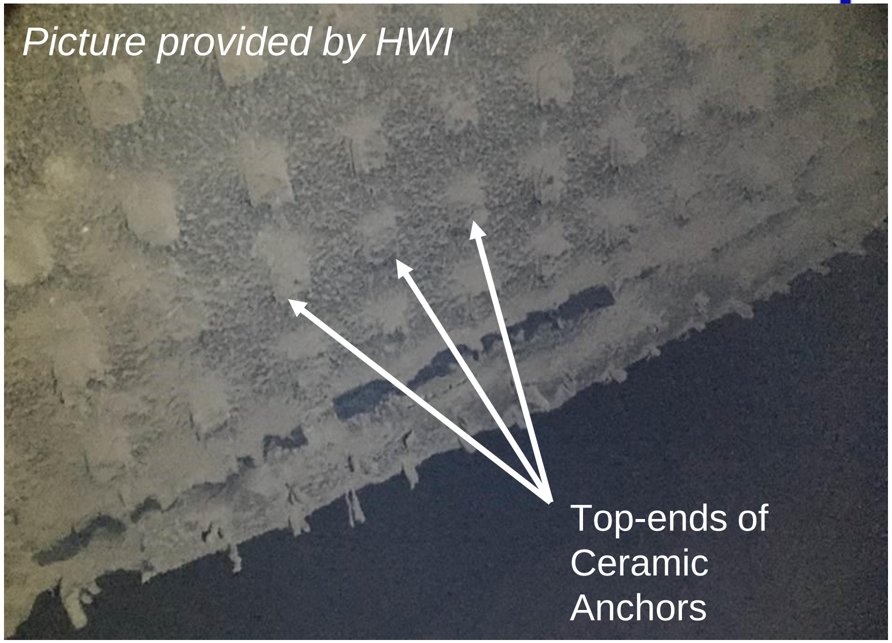
# Background:

**Monolithic Refractory Furnace Wall after Shut-Down**




- Large volume of ceramic refractory goes into the various boilers
  - Boiler floors and walls stand out as locations that contribute the most to refractory maintenance cost.
  - Less critical are ash hoppers and refractory failure there

- Understanding <u>thickness loss and degree of thermal shock</u> would be very important in these areas for precise operational planning and refractory replacement.

- Monitoring of <u>crack propagation and stress/strain development</u> during dry-out of the monolithic liner; this process would also be important for the application of these sensor systems.

# *Program Objectives:*

The specific project objectives are as follows:

1) Define high-temperature, stable conductive ceramics to be embedded within refractory anchors; **(Task 2)**

2) Develop and implement the 3D printing technology to pattern and control the microstructure of the ceramic anchor and embedded sensor circuits; **(Task 3)**

3) Develop low power analog electronics and wireless communication hardware to efficiently collect the sensor signal at each processing unit and transmit data to a central hub for data analysis; **(Task 5)**

4) Demonstrate the smart ceramic anchor system for temperature and liner fracture within a boiler furnace simulated environment *(which includes better interconnection strategy).* **(Task 4 & 6)**

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# Publications/Presentations:

**Publications**

1) *Coaxial Ceramic Direct Ink Writing on Heterogenous and Rough Surfaces: Investigation of Core-Shell Interactions*. *ACS Appl. Mater. Interfaces.* 2022
2) *The effect of multivalent elemental doping concentration on crystalline structure and electrical conductivity of lanthanum chromite perovskites obtained by Pechini Sol Gel method*. *ACS Appl. Mater. Interfaces.* 2022
3) *Theoretical and experimental analysis of dopant concentration and atmosphere nature effects on electrical properties and defect chemistry of doped lanthanum chromite pervoskites*. *Journal of Alloys and Compounds.* 2022.

**Presentations**

1) J.A. Mena, K. Sabolsky, A.A. Abrahamian, D.T. Cipollone, K. Sierros, E. M. Sabolsky, V. Mendoza-Estrada[2,], " High Temperature Thick Film Sensors Based on Doped Lanthanum Chromite Refractory Semiconductor Materials," Oral presentation ICMSN 2022 (2022 The 6th International Conference on Materials Sciences and Nanomaterials), July 10-142022.
2) D. Cipollone, J.A. Mena, K. Sierros, and E.M Sabolsky, "Out of the Lab: 3D Printing on Non-Ideal Surfaces." Oral presentation at the Materials Science & Technology 2021 (MS&T'21) Virtual Meeting, October 17-20, 2021.
3) J. A. Mena, K. Sabolsky, E.M Sabolsky, K. Sierros and K. S. Varadharajan, "Electrical, Structural and Thermomechanical Properties of Doped-$LaCrO_3$ Ceramics for High Temperature Electronics and Sensing Applications," Oral presentation at the Materials Science & Technology 2021 (MS&T'21) Virtual Meeting, October 17-20, 2021.
4) Z. Yang, J. A. Mena, J. Conte, B. Jordan, K. Sabolsky, K. A. Sierros, and E. M. Sabolsky, "Characterization and High Temperature Electrical Properties of Brazed Joints of $La_{0.8}Sr_{0.2}CrO_3$ with Nickel and Nickel Alloys. " Oral presentation at the Materials Science & Technology 2021 (MS&T'21) Virtual Meeting, October 17-20, 2021.
5) J.A. Mena, K.V. Sivaneri, G.A. Yakaboylu, E.M. Sabolsky, K. Sabolsky, and K. Sierros, "Evaluation of Doped-$LaCrO_3$ Ceramics for High Temperature Sensor Applications", Oral presentation at the Materials Science & Technology 2020 (MS&T'20) Virtual Meeting, November 2-6, 2020.

# *SUMMARY of TECHNICAL TASKS and MILESTONES*

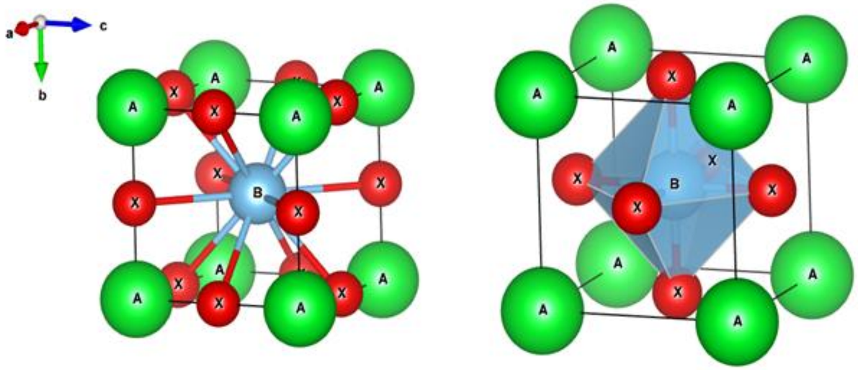
# *Task 2.0 – Fabrication and Characterization of Conductive Ceramic Composites (Sabolsky)*

# *Task 2.0 – Fabrication and Characterization of the Conductive Ceramic Composites*

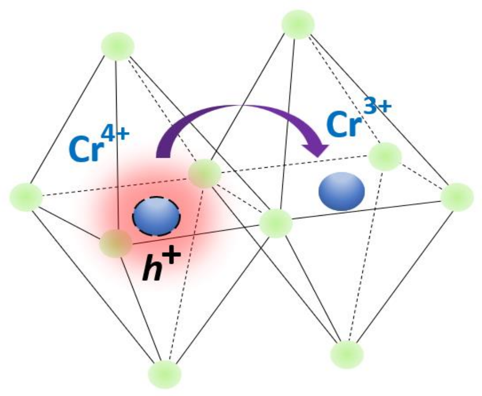
- ***Subtask 2.1- Synthesis and Thermal Processing of Perovskites and Composites***
  - The conductive oxide composites will be synthesized in-house.
  - Thermal processing in an atmosphere (e.g. air, argon) will be completed at high temperatures (up to 1500°C ).
- ***Subtask 2.2- Materials Characterization and Testing***
  - Characterize initial powder and monoliths using SEM, XRD, XPS and TGA.
  - Characterize stability in various environment, non-isothermal, isothermal and cyclic oxidation experiments will be conducted using a high temperature thermogravimetric analyzer (TGA) in an oxidizing gas flow (e.g. ambient air) at various temperatures
  - Electrical conductivity of the materials will be measured in a broad temperature range up to 1500°C to understand their electrical performance.

# *Initial High-Temperature Conductor for Sensors: Doped Lanthanum Chromites :*

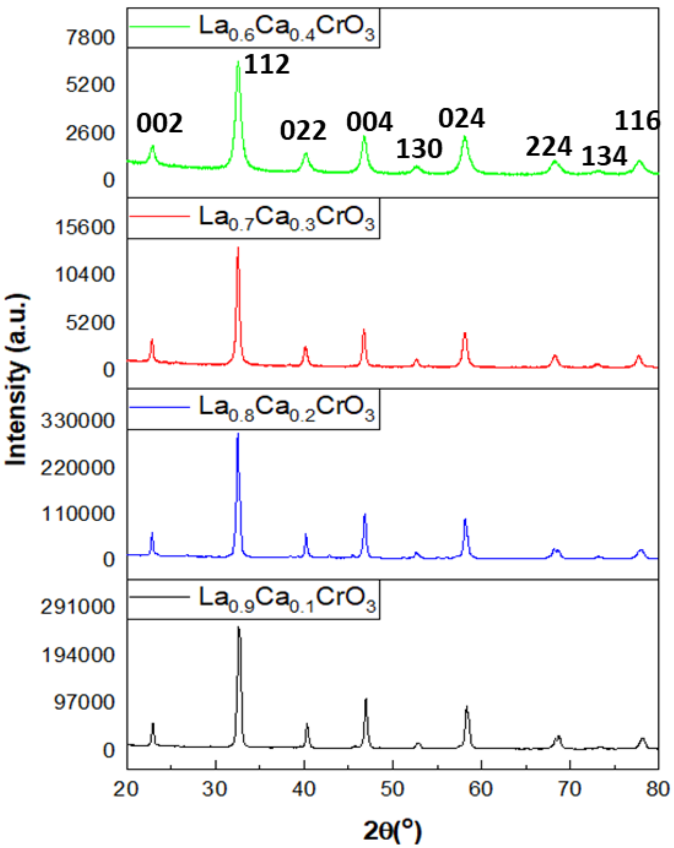
- High melting point (~2500 °C).
- Chemical stability under oxidative and reducing atmospheres.
- Pure $LaCrO_3$ shows semiconducting behavior with no ionic conduction.
- σ = 10 – 200 S·cm-1 (RT – 1500°C).
- Compatibility (thermal expansion coefficients matching) near refractory materials (~10×10$^{-6}$ C$^{-1}$).




*A atom cubic unit cell* *B atom cubic unit cell*



# Doped Lanthanum Chromites XRD Characterization:



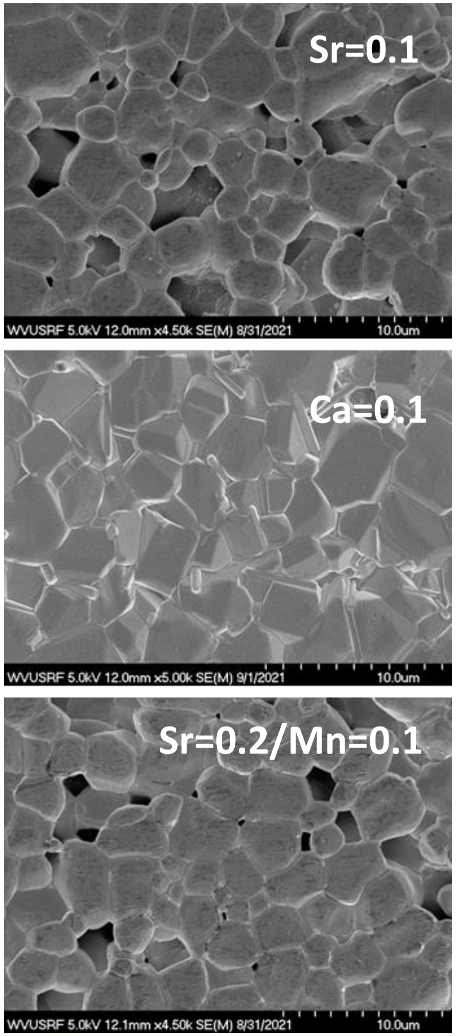

**X-ray diffractograms for the samples of the $La_{1-x}Ca_xCrO_3$ series**

**Lattice parameters, unit cell volume and XRD theoretical density for doped lanthanum chromites perovskites**

| Composition | Lattice parameters (Å) a | c | b | Volume ($Å^3$) | $\rho XRD_{Theoretical}$ ($g/cm^3$) |
|---|---|---|---|---|---|
| $La_{0.9}Sr_{0.1}CrO_3$ | 5.5124 | 7.7926 | 5.5668 | 239.1299 | 6.0658 |
| $La_{0.8}Sr_{0.2}CrO_3$ | 5.4988 | 7.7853 | 5.5425 | 237.2747 | 6.2568 |
| $La_{0.7}Sr_{0.3}CrO_3$ | 5.4769 | 7.7580 | 5.5233 | 234.6839 | 6.4710 |
| $La_{0.6}Sr_{0.4}CrO_3$ | 5.4524 | 7.7407 | 5.5122 | 232.6441 | 6.6721 |
| $La_{0.9}Ca_{0.1}CrO_3$ | 5.4180 | 7.7332 | 5.5039 | 230.6050 | 6.5962 |
| $La_{0.8}Ca_{0.2}CrO_3$ | 5.4092 | 7.7264 | 5.4982 | 229.7898 | 6.3340 |
| $La_{0.7}Ca_{0.3}CrO_3$ | 5.3994 | 7.7058 | 5.4877 | 228.3264 | 6.0871 |
| $La_{0.6}Ca_{0.4}CrO_3$ | 5.3897 | 7.6853 | 5.4622 | 226.2520 | 5.8528 |
| $La_{0.8}Sr_{0.20}Cr_{0.90}Mn_{0.10}O_3$ | 5.4734 | 7.7765 | 5.5648 | 236.8595 | 6.4197 |
| $La_{0.8}Sr_{0.20}Cr_{0.80}Mn_{0.20}O_3$ | 5.4705 | 7.7702 | 5.5587 | 236.2829 | 6.4437 |
| $La_{0.8}Sr_{0.20}Cr_{0.70}Mn_{0.30}O_3$ | 5.4598 | 7.7498 | 5.5398 | 234.4020 | 6.5037 |
| $La_{0.8}Sr_{0.20}Cr_{0.60}Mn_{0.40}O_3$ | 5.4146 | 7.7065 | 5.4981 | 229.4225 | 6.6534 |

- Decrease in lattice parameters were observed when dopant cations is introduced in the lattice.
- To achieve neutrality chromium oxidation states, change from $Cr^{+3}$ to $Cr^{+4}$, reduction in the chromium ionic size occurs.

# Doped Lanthanum Chromites Microstructural Characterization:



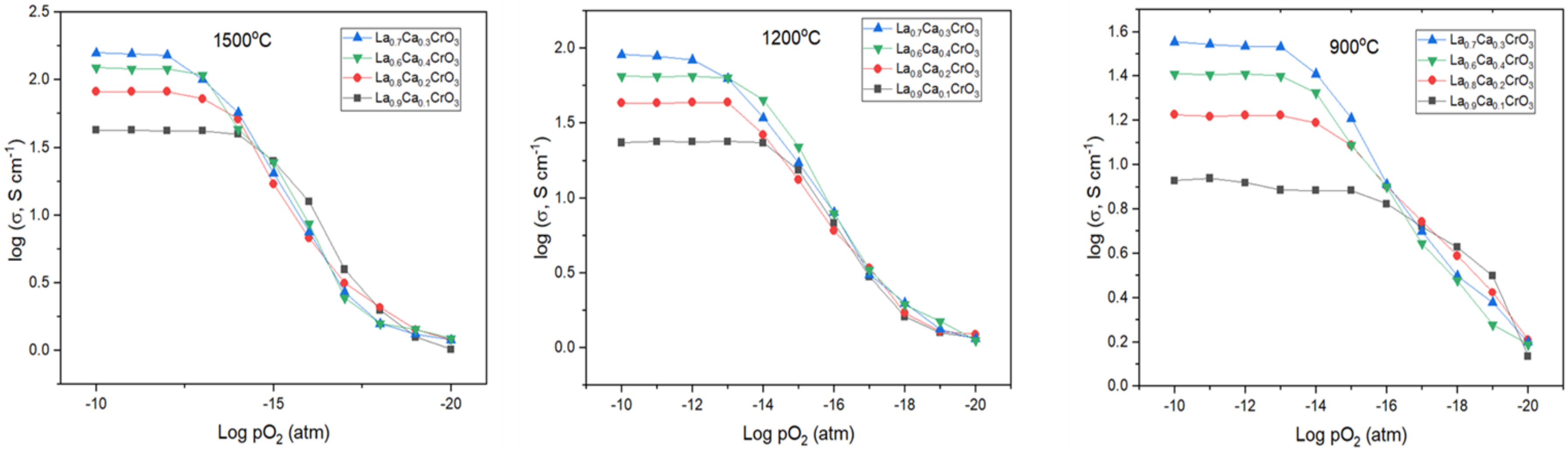

**Average grain size and bulk density distribution for $La_{1-x}Sr_xCrO_3$, $La_{1-x}Ca_xCrO_3$, $La_{1-x}Sr_xCr_{1-y}Mn_yO_3$ series**

| Composition | Average grain length (µm) | Relative Percentage Bulk Density (%) |
|---|---|---|
| $La_{0.9}Sr_{0.1}CrO_3$ | 3.6480 | 94 |
| $La_{0.8}Sr_{0.2}CrO_3$ | 3.4898 | 95 |
| $La_{0.7}Sr_{0.3}CrO_3$ | 3.6290 | 95 |
| $La_{0.6}Sr_{0.4}CrO_3$ | 3.2455 | 94 |
| $La_{0.9}Ca_{0.1}CrO_3$ | 4.1367 | 96 |
| $La_{0.8}Ca_{0.2}CrO_3$ | 3.7727 | 97 |
| $La_{0.7}Ca_{0.3}CrO_3$ | 3.6883 | 97 |
| $La_{0.6}Ca_{0.4}CrO_3$ | 3.6548 | 98 |
| $La_{0.8}Sr_{0.20}Cr_{0.90}Mn_{0.10}O_3$ | 3.3257 | 95 |
| $La_{0.8}Sr_{0.20}Cr_{0.80}Mn_{0.20}O_3$ | 3.5142 | 96 |
| $La_{0.8}Sr_{0.20}Cr_{0.70}Mn_{0.30}O_3$ | 3.4771 | 95 |
| $La_{0.8}Sr_{0.20}Cr_{0.60}Mn_{0.40}O_3$ | 3.3782 | 97 |

- Pechini Sol Gel prepared calcium, strontium, manganese doped lanthanum chromite powders exhibit better sinterability and densification under oxidizing conditions.
- The samples of Ca doped lanthanum chromite powder have more dense microstructures.

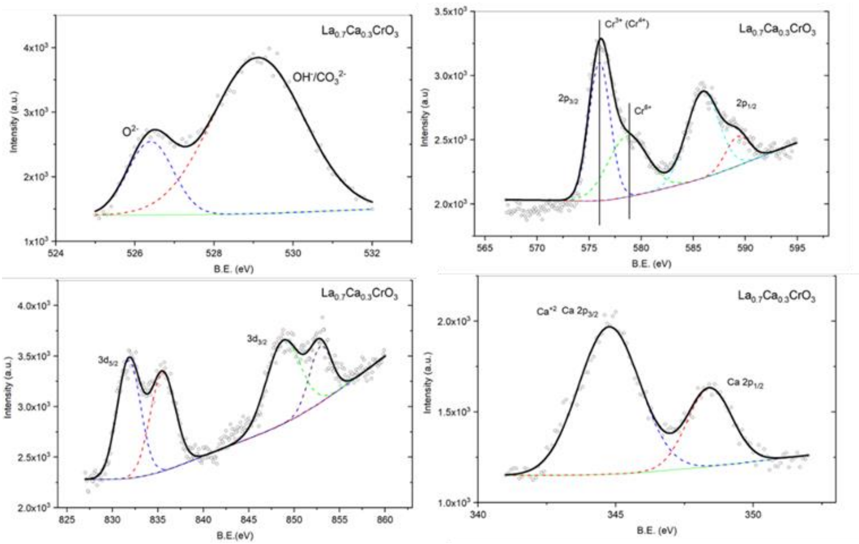
# *Doped Lanthanum Chromites Electrical Characterization:*

**Electrical conductivity dependence of oxygen partial pressures at different temperatures for calcium doped lanthanum chromites**




- When the oxygen partial pressure goes below a critical value, the oxygen vacancies are generated at expense of electron holes and conductivity decrease for all compositions.
- At lower temperatures (600°C - 900°C) not significant change in conductivity occurs during the equilibrium time used (90 minutes).
- At higher temperatures (1200°C and 1500°C) the conductivity drops exponentially at lower oxygen partial pressures. Increasing the strontium concentration, the conductivity drop significantly.

# Doped Lanthanum Chromites Optical and Defect Chemistry Characterization:



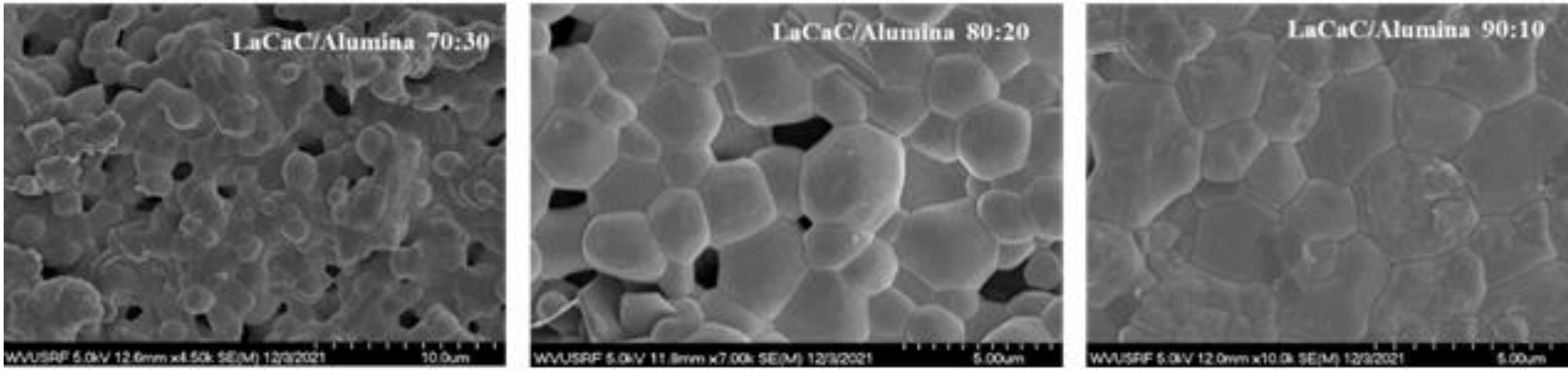

***High resolution XPS spectra of $Ca^{+2}$, $La^{+3}$, $O^{-2}$, $Cr^{+3}$, $Cr^{+4}$ and $Cr^{+6}$ in pure calcium doped lanthanum chromite.***

**Optical band gap for $La_{1-x}Sr_xCrO_3$; $Ca_{1-x}Sr_xCrO_3$; $La_{0.8}Sr_{0.2}Cr_{1-y}Mn_yO_3$ compositions**

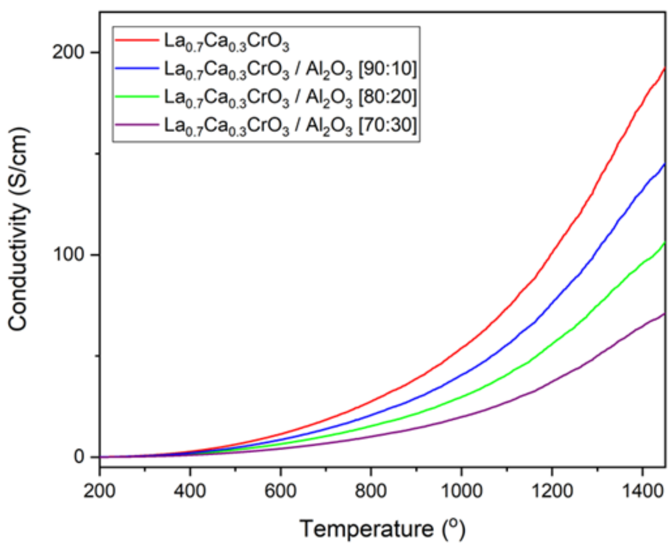
| Composition | Band Gap (eV) |
|---|---|
| $La_{0.9}Sr_{0.1}CrO_3$ | 2.03 |
| $La_{0.8}Sr_{0.2}CrO_3$ | 1.86 |
| $La_{0.7}Sr_{0.3}CrO_3$ | 1.63 |
| $La_{0.6}Sr_{0.4}CrO_3$ | 1.55 |
| $La_{0.9}Ca_{0.1}CrO_3$ | 2.06 |
| $La_{0.8}Ca_{0.2}CrO_3$ | 1.97 |
| $La_{0.7}Ca_{0.3}CrO_3$ | 1.59 |
| $La_{0.6}Ca_{0.4}CrO_3$ | 1.42 |
| $La_{0.8}Sr_{0.2}Cr_{0.9}Mn_{0.1}O_3$ | 2.03 |
| $La_{0.8}Sr_{0.2}Cr_{0.8}Mn_{0.2}O_3$ | 1.82 |
| $La_{0.8}Sr_{0.2}Cr_{0.7}Mn_{0.3}O_3$ | 1.62 |
| $La_{0.8}Sr_{0.2}Cr_{0.6}Mn_{0.4}O_3$ | 1.72 |

- The peak in 576 eV correspond to chromium 2p state associated to the presence of $Cr^{+3}$ and $Cr^{+4}$ as expected.
- The formation of chromium higher oxidation states due calcium doping evidence the electron/ polaron hopping as electrical conductivity mechanism.
- Band gap values decrease at function of doping level for calcium and strontium content.
- The introduction of strontium and calcium increment the positive holes concentration forming shifts in the conduction bands.

# *Doped Lanthanum Chromites/ Alumina Composites Characterization :*

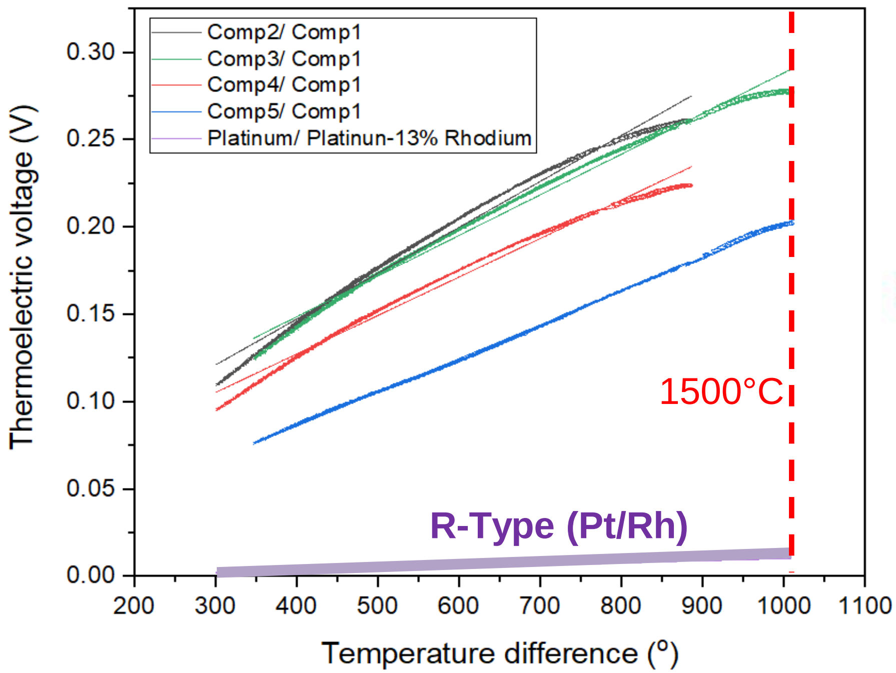


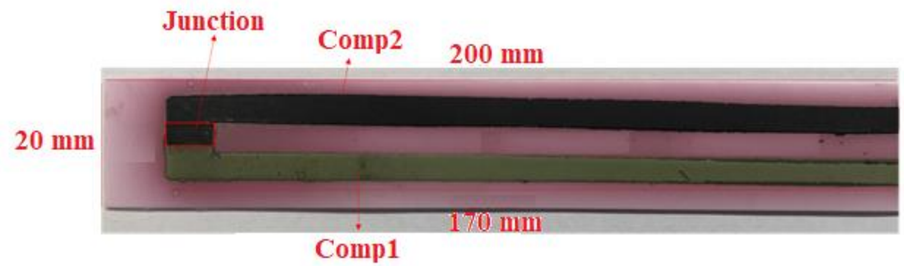

***ıcture of LCC/ Alumina composites pellets***




- Decreasing conductivity effect can be explained by low sinterabilty of composites with the alumina increment.
- Alumina aggregates affect the LCC grain grow and pore formation increasing the resistance inside the composite.

# *Ceramic-Based High-Temp Thermocouples (Provisional Patent Application Submitted):*




**U-shape ceramic printed thermocouple prototype**

- ➢ The thermoelectric voltage is direct proportionally correlated with temperature difference.
- ➢ The thermoelectric testing was completed up to 1500°C; the conductivity and microstructural stability was proven for these materials at higher temperatures.
- ➢ Thermocouples were tested in a range between 30 to 1500°C , showing an excellent reproducibility indicating that chromium evaporation has little effects on the device sensitivity.

# Task 2 Summary:

**Completed Work**

- Doped chromites perovskites characterized by XRD, SEM, XPS, UV-Vis and Archimedean density.
- Electrical conductivity testing of all prepared doped lanthanum chromite compositions at temperatures up 1500°C under air, reducing and different oxygen partial pressures atmospheres.
- Optimal compositions passed to Task 3 for embedding in smart refractory.
- New all-ceramic refractory thermocouples invented and implemented into alternative refractory brick (US Patent applied).

**Future Work**

- Test embedded thermocouples versus cycling and long-term operation at high temperature.
- Investigate alternative n-type ceramic compositions for further development of thermocouples and strain sensor materials.

# *Task 3.0 – Direct-Writing of Refractory and Sensor System (Sierros)*

# Task 3.0 – Direct-Writing (2D/3D Patterning) of Refractory and Sensor System

- ***Subtask 3.1- Direct-Writing Ink Development***
  - Ink formulations will be fabricated within a permissible surface tension and viscosity range for direct-writing.
  - Characterization of drying behavior of printed lines at room/moderate temperature. Ink formulations will be characterized using thermogravimetric analysis to understand organics decomposition.

- ***Subtask 3.2 Direct-Writing/Patterning and Drying Characterization***
  - Direct writing deposition, drying, and thermal post-processing will be defined, which includes methods to control wetting and drying characteristics of the deposited composite solutions (for both refractory and sensor circuit formulations).
  - Ink formulations, direct writing parameters (printing nozzle shape and size, writing speed, extrusion pressure), drying procedures, and post-processing temperatures will be included as variables, and printing fidelity will be characterized by optical, SEM and AFM microscopy.

# Task 3.0 – Direct-Writing (2D/3D Patterning) of Refractory and Sensor System

- ***Subtask 3.3- Thermal Processing Development and Structure Tailoring***

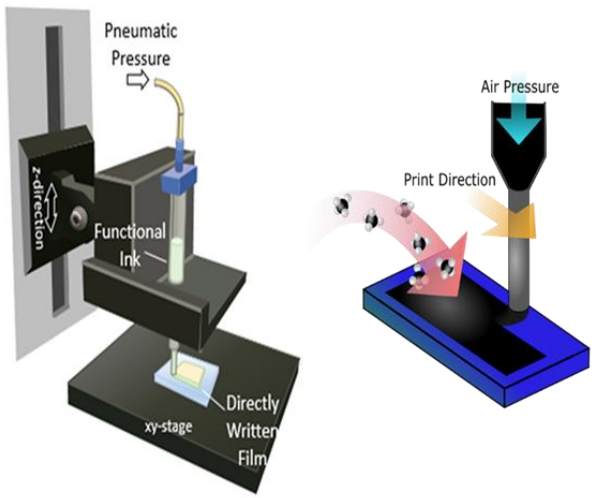
  - Post-processing thermal treatment as defined by thermal schedules in Task 2.0.
  - Printed samples will be analyzed by optical microscopy and SEM/EDS as a function of thermal processing (in the bulk and film printed states). The propagation of defects and sintering mismatch, as well as shrinkage, will be studied and analyzed for each layer.

- ***Subtask 3.4-Baseline Sensor Testing and Design Optimization***

  - Three (3) initial sensor configurations will be designed using CAD. Electroceramic patterns will be directly written from CAD onto ceramic preforms and directly onto refractory anchor substrates, respectively.
  - Feature sizes will be measured by surface profilometry and optical microscopy. After post-processing, electrical performance characterization will be conducted at high-temperatures, ranging from 500-1500°C, in varying atmospheres. Baseline electrical performance will be assessed, and optimized designs will be completed and compared in Task 5.

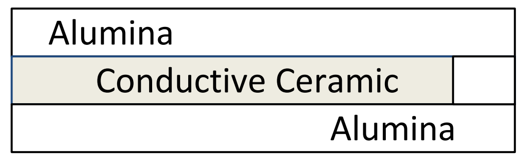
# *Direct Ink Writing:*

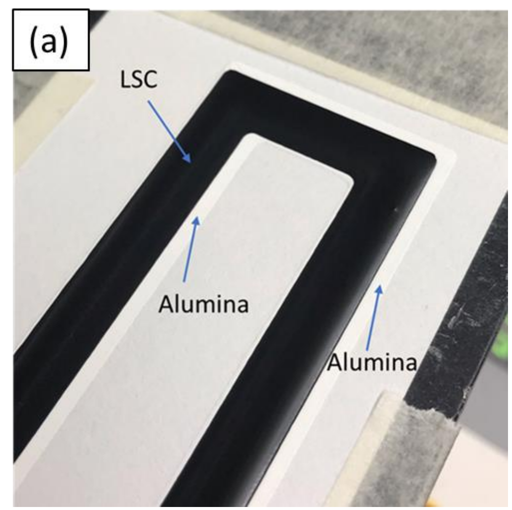
- Traditionally layer-by-layer
- Adequate for most applications
- Time to dry increases manufacturing time
- Surface protrusions may induce discontinuities



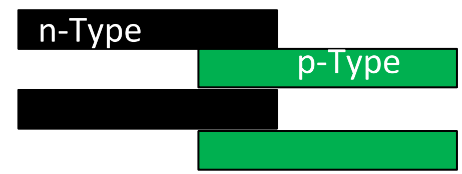

- Extrusion Based
- Ambient Deposition
- Lax Material Constraints

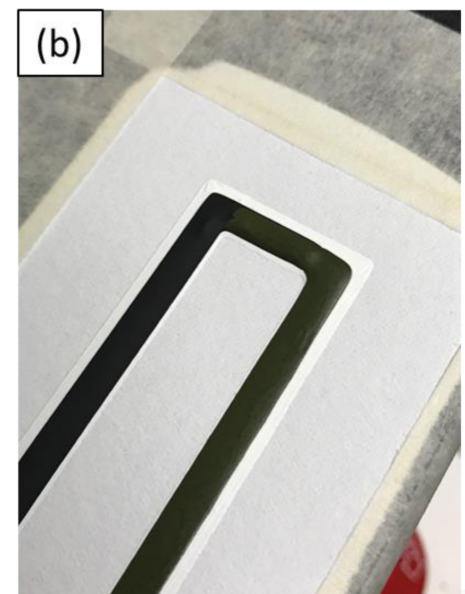
**Thermistor**




**Thermocouple**

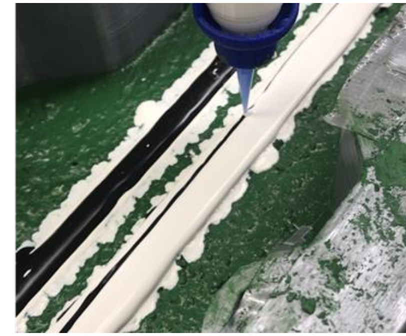


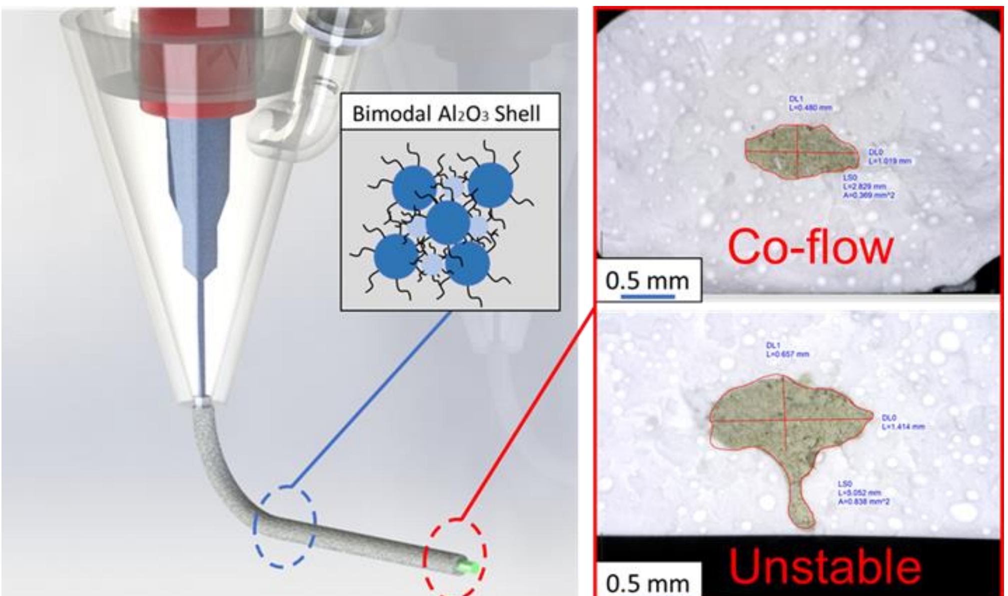



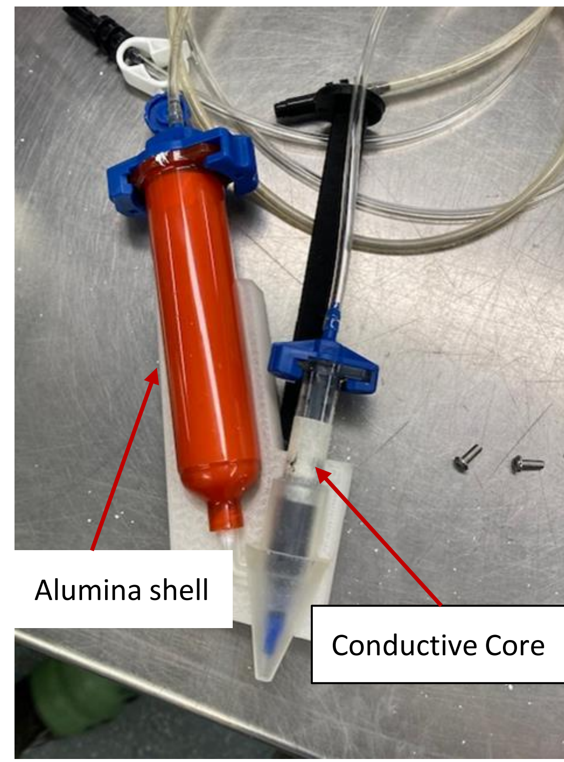
# Coaxial DIW and Platform Preparation:

*Can we simplify the manufacturing process through materials engineering?*

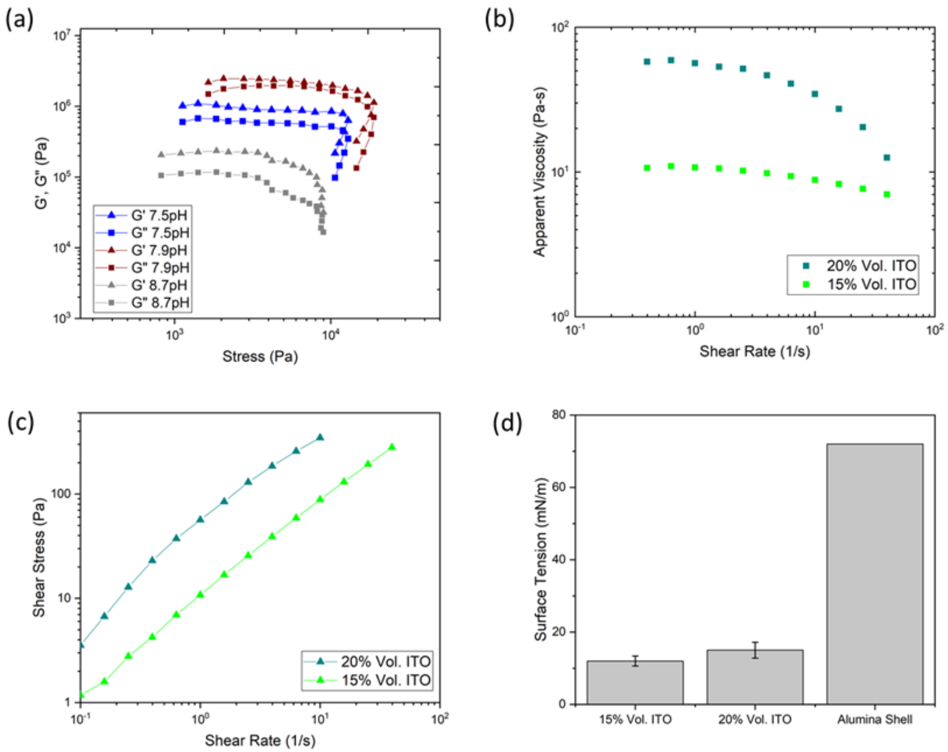
- ➢ Co-extrusion of core-shell ink systems
- ➢ Hydrophilic alumina shell
- ➢ Hydrophobic conductive core
  - ○ Facilitate core fidelity
- ➢ Microfluidic co-flow inspired analyses

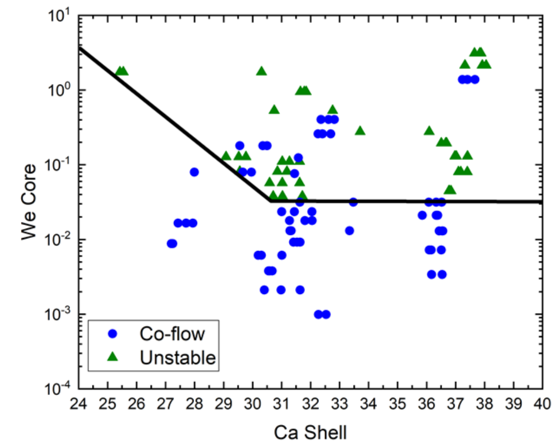


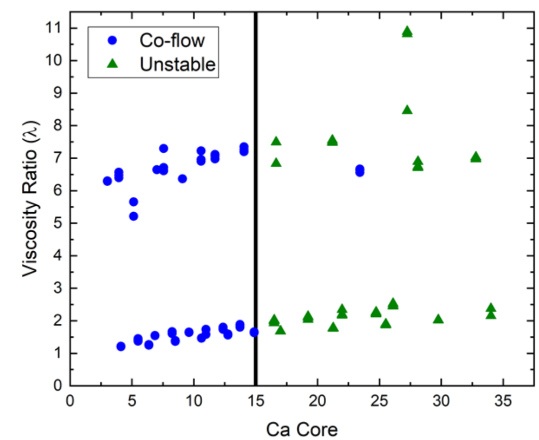


# *Ink Engineering and Flow Analysis:*

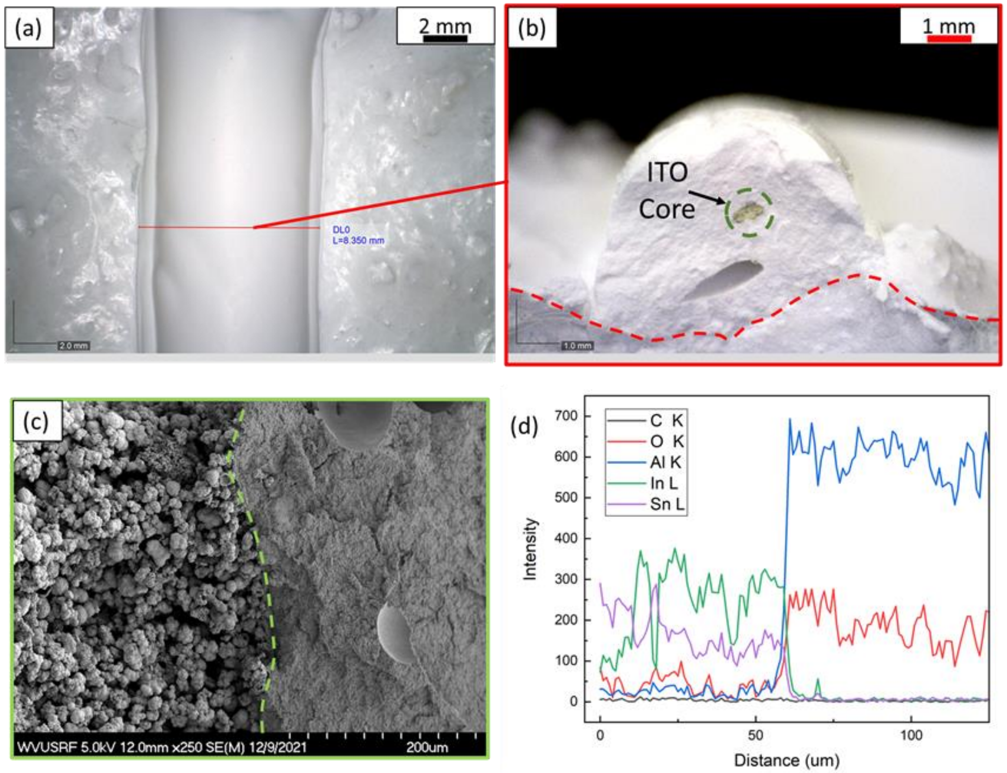


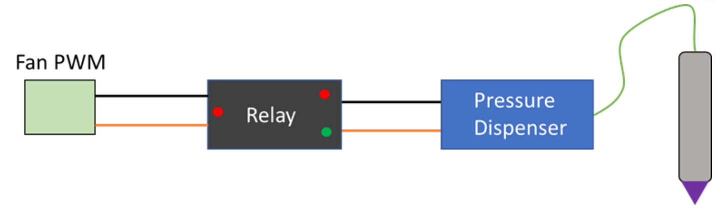




- ➢ NaOH addition enables tailoring ink stiffness one order of magnitude
- ➢ Flow stability characterized through mappings of We, Ca, Flow rate ratios, and viscosity ratios

# Smart Refractory and Coaxial Printing:





- ➢ Alumina barrier maintains core continuity across rough substrate
- ➢ Hydrophobic-hydrophilic ink suite maintains interface
  - Sharp transition shown in EDX scan

# *Future Work:*

- Expand direct ink written sensor suite
  - Thermistors, thermocouples, and *strain sensing elements*
  - Study microstructure-performance relationships

- Further develop coaxial ceramic DIW technology
  - Study of viscous co-flow and stable flow regimes
  - Potential to harness deformations and instabilities

# Summary and Completed Milestones:

| Subtask | Milestone | Planned Completion | Actual Completion |
|---|---|---|---|
| 3.1 | Ink Formulation | 08/31/2020 | 08/27/2020 |
| 3.2 | Ink Characterization | 08/31/2020 | 08/31/2020 |
| 3.3 | Printing of Preforms (2 designs) | 12/31/2020 | 11/04/2020<br>03/15/2022 |
| 3.4 | 3D Printing of Sensor During Refractory Fab. | 10/31/2021 | 03/25/2021<br>03/25/2022 |
| 6.1 | Delivery and Setup of 3D Printer at HWI | 06/30/2021 | 03/25/2021<br>03/25/2022 |

# *Task 4.0 – Development of Embedded Interconnection Design and Smart Anchor Testing (Sabolsky)*

# Task 4.0 – Development of Embedded Interconnection Design and Smart Anchor Testing

- ***Subtask 4.1- Anchor Clamp Interconnection Development***
  - Ceramic/metal brazes will be evaluated to connect the electroceramic embedded sensor to an insulated metal wire/spring that will pass through the anchor clamp.
  - The electrical (and chemical) stability of the connection will be evaluated as a function of time/temperature to insure a stable interconnection to the electronics.

- ***Subtask 4.2- Corrosion Testing of Smart Anchor Prototypes***
  - Prototype ceramic anchors will be fabricated and tested within static slag conditions at 900-1350ºC extending over 24-500 hours.
  - Interconnect clamps designed and fabricated in Subtask 4.1 will be electrically connected to the prototype anchors.
  - The anchors will undergo static slag exposure within the refractory testing system already available to test sensored refractory brick.

## *Subtask 4.3- Quality Control Testing of Prototype Smart Anchor Samples*

- The Recipient will measure the modulus of rupture (MOR) and cold crushing strength (CCS) (after drying and firing) using ASTM C-133.
- Other important characteristics that will be measured will be followed in order qualify the anchors for potential service.
  - Bulk density (by ASTM C-134)
  - Linear change (by ASTM C-113)
  - Abrasive change (by ASTM C-704).
- Post-mortem microstructure will be evaluated by SEM for the tested prototypes.

**Smart Anchors with Slag Insertion Ports Available**

# Sensor Interconnection by Ag-based Cement:

- Embedded sensors require strong ceramic/metal ohmic joints to interconnect the sensor to the electronics (through an insulated wire).
- Ag-based high temperature conductive and ceramic-metal cements have been identified for mechanical ceramic/metal joints.

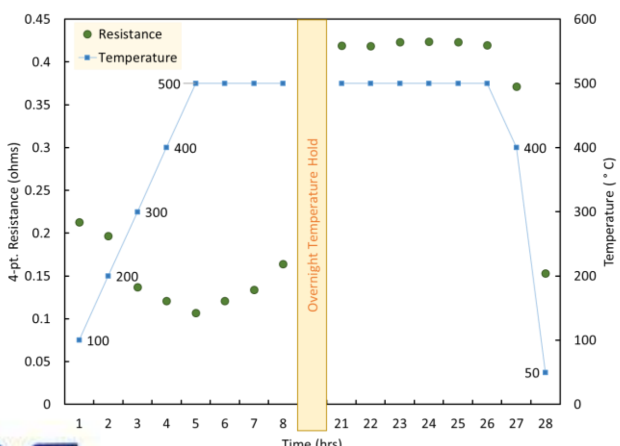
**Pyro-duct 597-A (Aremco)**

- Silver-filled, electrically and thermally conductive adhesive up to 927°C
- Simple application: air dry 1-4 hrs; cure at 100°C for at least 1 hr
- *Connects leads to sensor material*

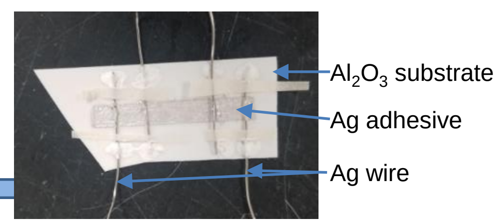
**Ceramabond 668 (Aremco)**

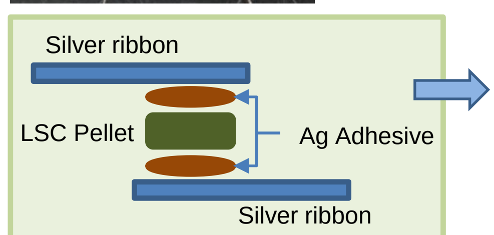
- High temperature adhesive up to 1371°C
- Bonds and seals ceramics to ceramics and ceramics to metals
- Connects and secures wires to the brick

**Initial burn-in required for stability**

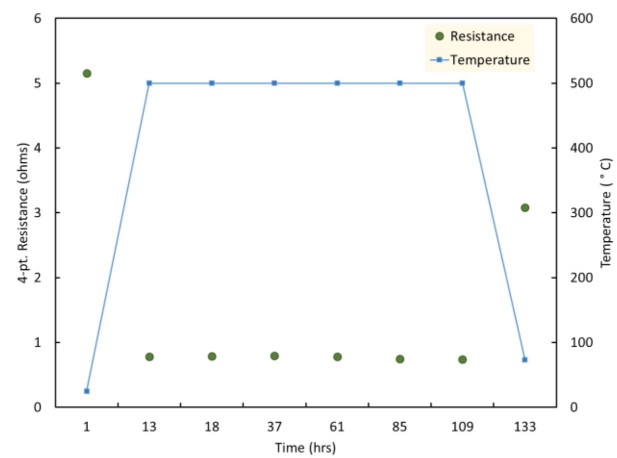



4-pt Resistance Samples



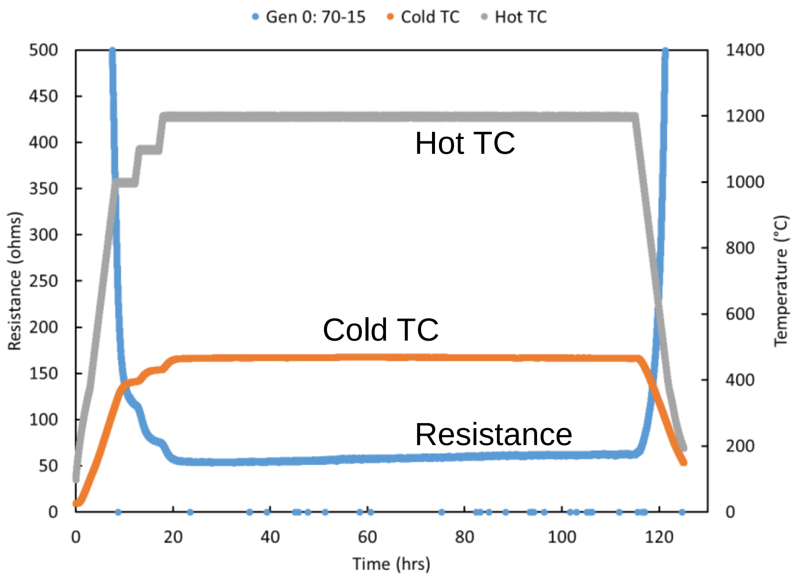



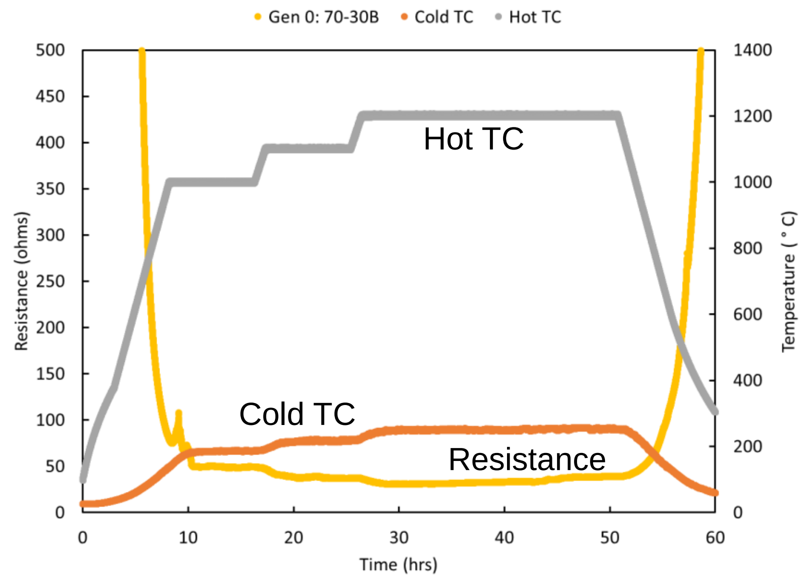

**Long term stability with LSC**



# Gen0: Braze vs. Ag-based cement

Full size anchor w/ hole and Gen0: CompA




**Ag-based braze:**
- annealed under 5% $H_2$-95%$N_2$ gas at

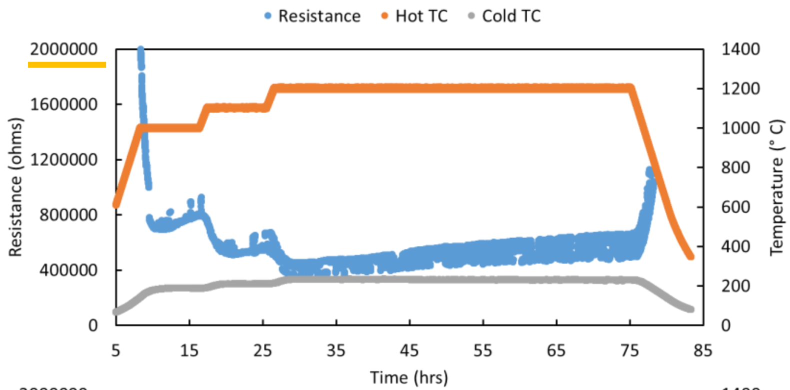
**Ag-based cement:**
- cured in air at 100°C for 12 hrs

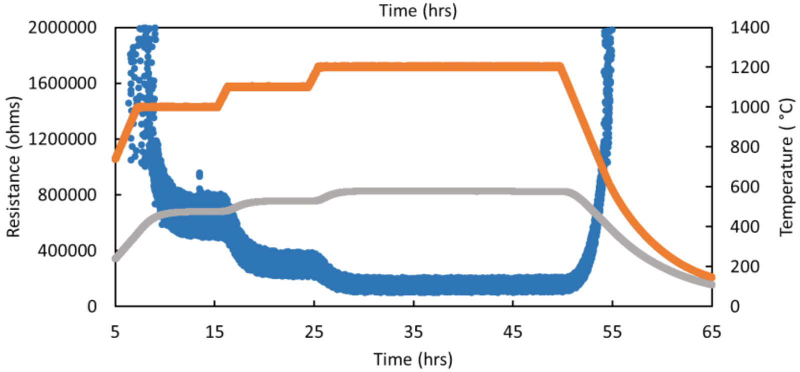
- Ag-based cement provides results comparable to brazed leads
- Ag-based cement allows for faster and easier application and preparation of bricks for testing

61-53-04-02H (w/ hole)
61-34 LSC Ink (Gen0)

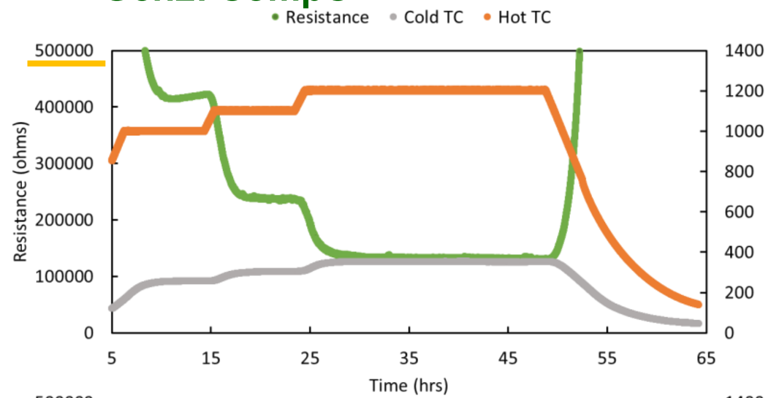
# Gen1 and Gen2 Sensor Testing:

## Gen1: CompB:$Al_2O_3$

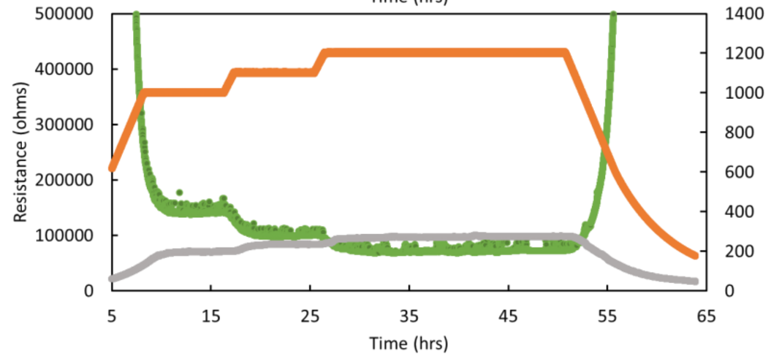


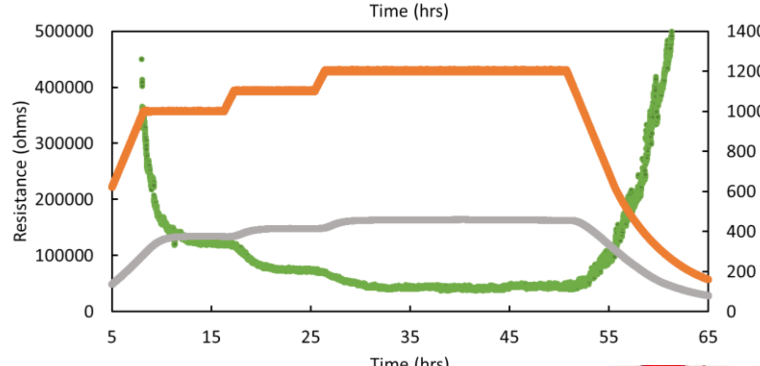

## Gen2: CompC




2 Layers




3 Layers




4 Layers

Sensor Material Thickness

- Testing protocol:
  - 1000°C/8h, 1100°C/8h, 1200°C/24-48h with 2°C/min heating and cooling rates
- Gen 1 sensors:
  - High resistance, unstable and broad data range
  - Does stabilize with thicker sensor layers
- Gen 2 sensors:
  - Resistance under 100 kΩ
  - Improved stability over 24 hr hold

# Quality Control Testing of Protypes (Go-NoGo):

- **Demonstrate smart anchor prototype which achieves >75% quality control standards for high-alumina ceramic anchor composition (Modulus of Rupture, Cold Crushing Strength)**

| Test Type | Criteria | Tested Results |
|---|---|---|
| Density after drying, UNITS: pcf | | 184-188 |
| Using ASTM C-133 MOR after drying, UNITS: psi | >1125 | 2980-3200 |
| Using ASTM C-133 CCS after drying, UNITS: psi | >6000 | 11650-16270 |
| | | |
| Density after 1500F (firing), UNITS: pcf | | 178-181 |
| Using ASTM C-133 MOR after 1500F firing, UNITS: psi | >375 | 2080-2430 |
| Using ASTM C-133 CCS after 1500F firing, UNITS: psi | >5250 | 10650-15010 |

- Samples are quite robust with embedded sensors and achieved >200% of the minimum criteria set for smart anchors at that point.

# *Task 5 Summary:*

**Completed Work**

- Evaluated castable Ag/refractory composite potting compositions to bond interconnect wires to embedded sensor design.
- Results show conductive composites show higher stability and strength over brazed leads (and easier thermal processing methodology).
- Quality control testing (MOR and CCS testing) showed that the smart anchors are comparable to current anchor products.

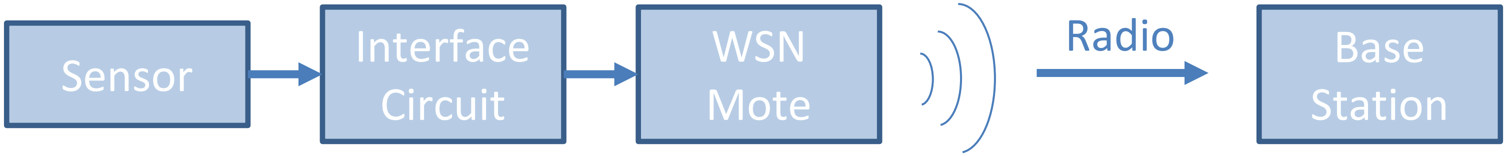
**Future Work**

- Complete evaluation of Gen3 sensors with lower temperature cold end connection.
- Complete in-house corrosion testing.
- Initiate testing of thermocouple sensors.
- Develop engineering method to secure connection for deployment in a commercial application (discussed further in Task 6).

# *Task 5.0 – Electronics and Wireless Communication Interfacing with Smart Ceramic Anchors (Graham)*

# *Task 5 Objectives:*

- To develop methods to interface the electrical sensing outputs from the smart refractory with an embedded processor
- To design a wireless sensor network to efficiently collect the data at a processing unit for further data analysis

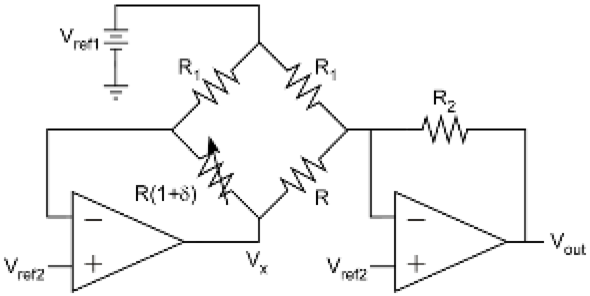


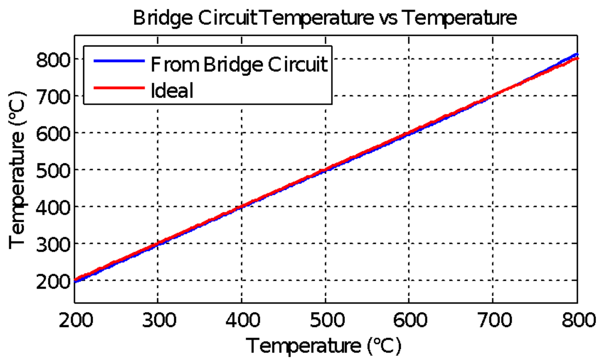
# Task 5.0 – Electronics and Wireless Communication Interfacing with Smart Ceramic Anchors

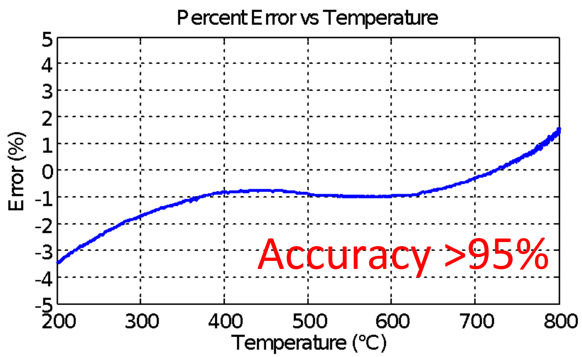
- ***Subtask 5.1- Interfacing Electrical Outputs to Motes***
  - Initial interface circuitry will be designed using discrete off-the-shelf components to obtain a working system and to allow for full system integration.
  - These interface circuits will be used for monitoring the sensors within the ceramic anchors and providing the appropriate signals to the wireless embedded processor.
- ***Subtask 5.2- Wireless Data Transport and Stand-alone Performance Evaluation with Simulated Electrical Signals***
  - The aim of this subtask will be to reliably transport the anchor sensing data from the individual motes to a data processing center over a wireless communication medium, and to allow convenient wireless configuration of sensor parameters from a remote processing center.
  - Initial sensor network prototype will be developed to evaluate in a stand-alone mode using simulated sensor data. The information delivery performance will be evaluated for the typical ceramic anchor application-parameters including data sampling requirements, sensor density, and number of sensors.
- ***Subtask 5.3- Investigating Reconfigurable Integrated Circuits Interfaces***
  - Reconfigurable IC solutions are a natural choice for this application because of the iterative nature of testing and the capability to refine performance after final deployment.
  - Further implication of this design choice is the ability to reconfigure the sensing circuitry wirelessly when interfaced with the motes, whether the circuitry is in-the-field or in a lab setting.
  - The reconfigurable ICs will interface the motes and anchor sensors in the same manner as the off-the-shelf sensing circuits without any needed modifications to the mote.

# Task 5.0 Sensor Interfacing Circuitry

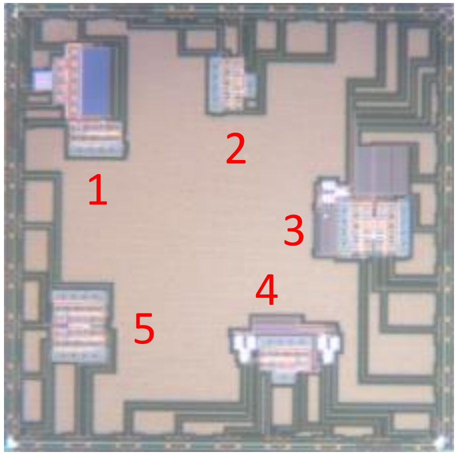
## Discrete Sensor-Interfacing Circuits



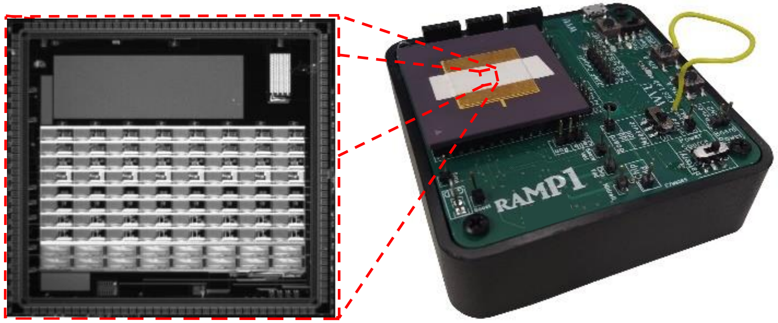




## Custom Integrated Circuits




1. Cold-Junction Compensator
2. Thermocouple Amplifier
3. Capacitive Sensor
4. Thermocouple Amplifier V2
5. Wheat-Stone Bridge

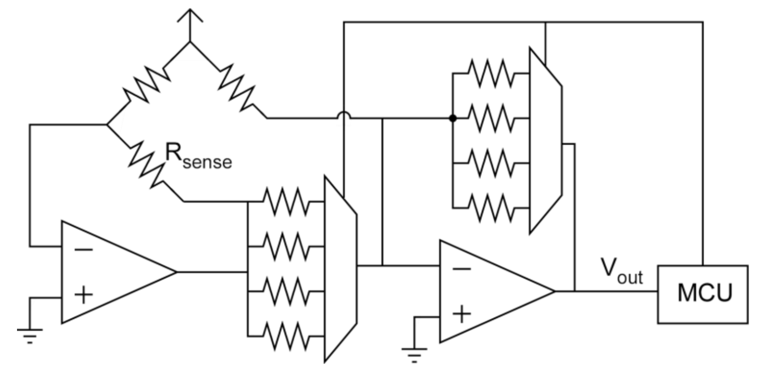
## Reconfigurable Integrated Circuits

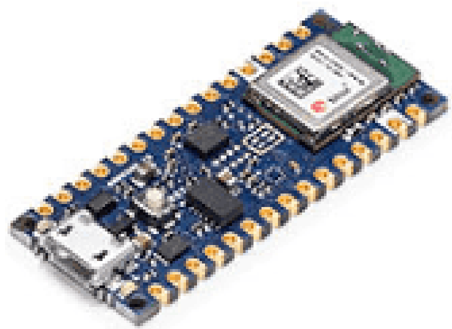


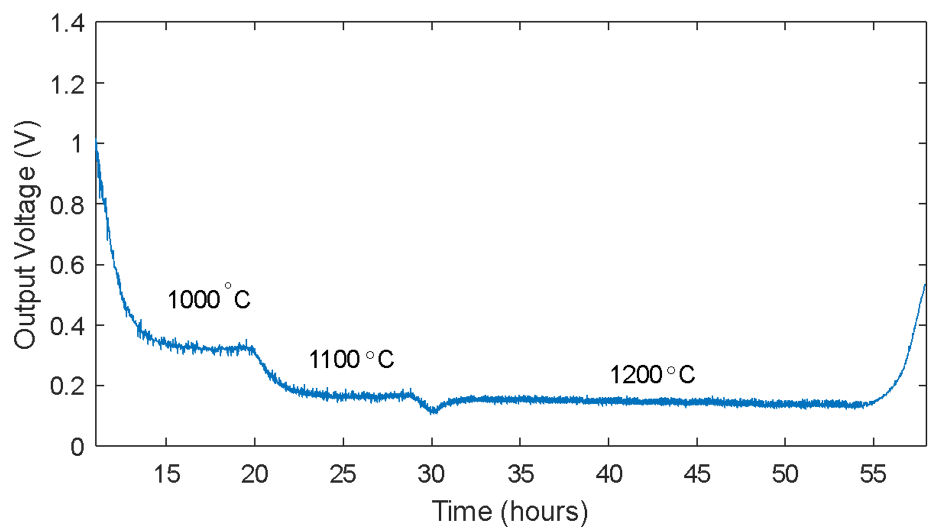

Flexibility post-deployment
Ultra-low power implementations

# Task 5.0 Prototype and Test Run Results

- Arduino Nano 33 IoT
  - Includes low energy Bluetooth (BLE)
  - Analog-to-Digital (ADC) converter for digitizing input data from WSB
- Test runs resulted with accurate received readings (resistance) from the wireless node
- Auto-ranging functionality to improve measurement resolution

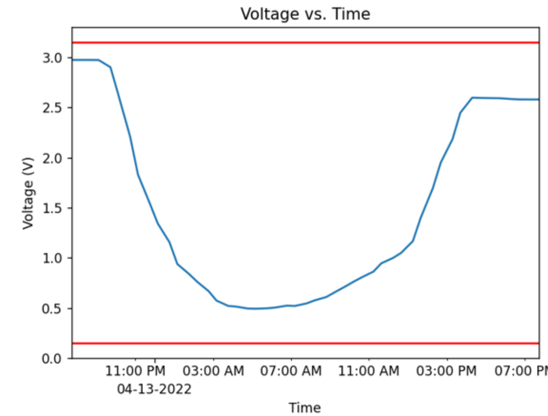


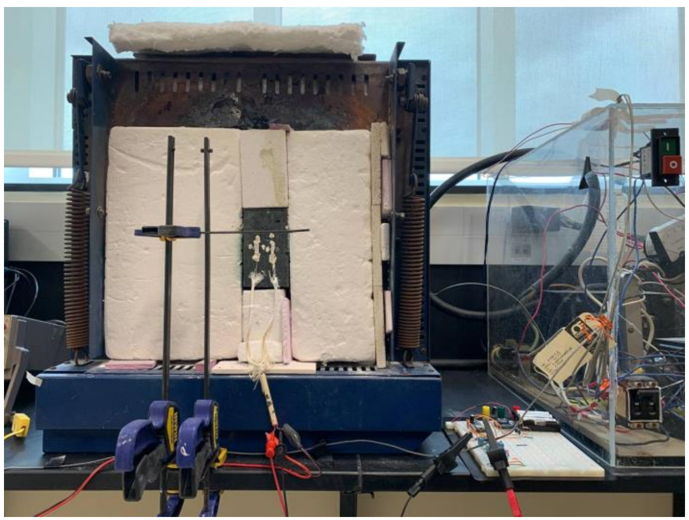





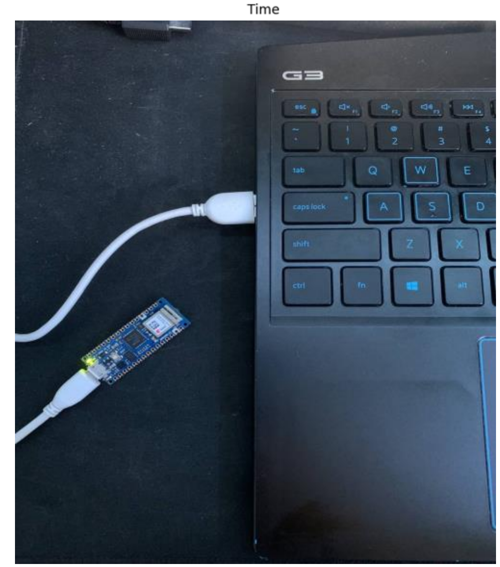
# Task 5.0 System and Software Improvements

- Python User Interface
  - Library for reading serial interface on Nano 33 IoT
  - Handles auto-ranging & BLE communication
  - Provides graphical assessment of measurements



BLE



BLE

Sensor → Interface Circuit → Nano 33 Iot → Nano 33 Iot → Laptop Base Station

# Task 5 Summary:

**Completed Work**

- Built prototype sensor interfacing circuit/platform
- Implemented better microcontroller/radio platform
- Developed Python software infrastructure for data collection and analysis
- Implemented auto ranging functionality on the sensor circuitry for accommodating a larger range of resistance/temperatures
- Milestone 8 Completed – Demonstrate operational prototype of sensor interfacing electronics

**Future Work**

- Improve the power savings (implement a real-time clock for duty cycling, etc.)
- Expand to thermocouples and other sensors
- Implement a custom solution on our reconfigurable sensor-interfacing and analysis platform

# Task 6.0 Ceramic Anchor Manufacturing and Concept Demonstration (Sabolsky)

# Task 6.0 Ceramic Anchor Manufacturing and Concept Demonstration.

- ***Subtask 6.1- Scale-up of Smart Ceramic Anchor Production***
  - Fabricate the sensor preforms formed by 2D/3D printing, and begin to design and setup a printing system.
  - Implement trials of ceramic anchor manufacturing.  Anchors will undergo quality control analysis, where basic statistics on loss/failure rates may be estimated for Task 7.0.
  - Anchor clamping and wiring trials will be completed to mimic actual monolithic (castable) refractory installation within the primary furnace of a boiler.

- ***Subtask 6.2- Quarterly Ceramic Anchor Testing Trials***
  - Quarterly testing of anchor and interconnection performance (in a non-critical application),  the anchors will be inserted within the available kilns.
  - At least three (3) smart prototype anchors will be evaluated each quarter (starting at the onset of this task within the program) using the optimal smart anchor, interconnect clamp and electronics/wireless motes for the current quarter.

# Task 6.0 Ceramic Anchor Manufacturing and Concept Demonstration.

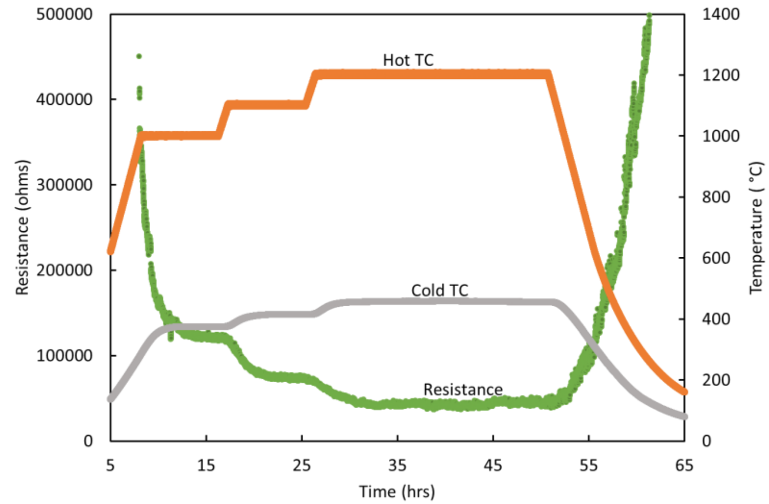
## *Subtask 6.3- End-user In-service Smart Ceramic Anchor Demonstration (DEMO)*

- A minimum of three (3) smart anchors will be installed within the liner of a coal boiler, ash hopper, or glass furnace floor.
- The demonstration will include at least three wireless sensor nodes utilizing the optimal electronic interface, power configuration, and wireless network defined in Task 5.0.
- The end-user demonstration customer(s) will be **<u>identified by quarter eleven.</u>**

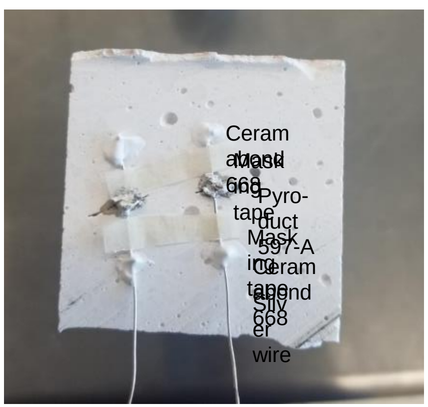
# *Smart Anchor with a Silver Sensor Connection:*

- Half-sized bricks were cast with printed sensors and cavity to facilitate testing
- Silver adhesive was used to attach sensor to silver leads
- Refractory mortar was applied to protect connections during testing in a commercial setting

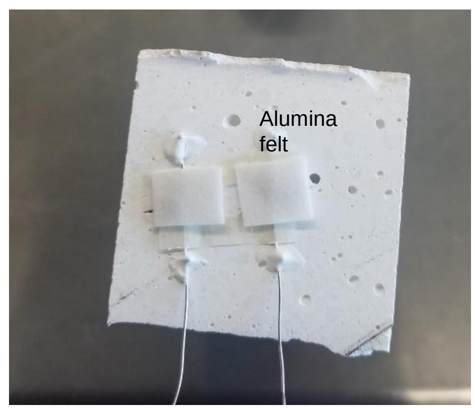
Test with Silver Adhesive Connection



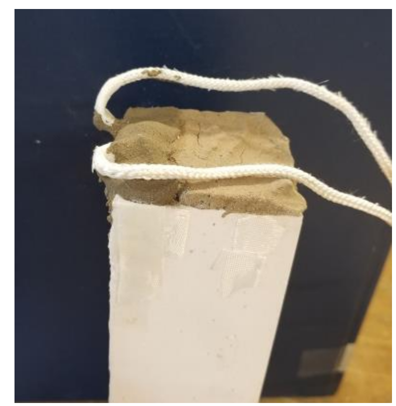

Silver wire leads connection with protective layers




Connection arrangement




Initial protection

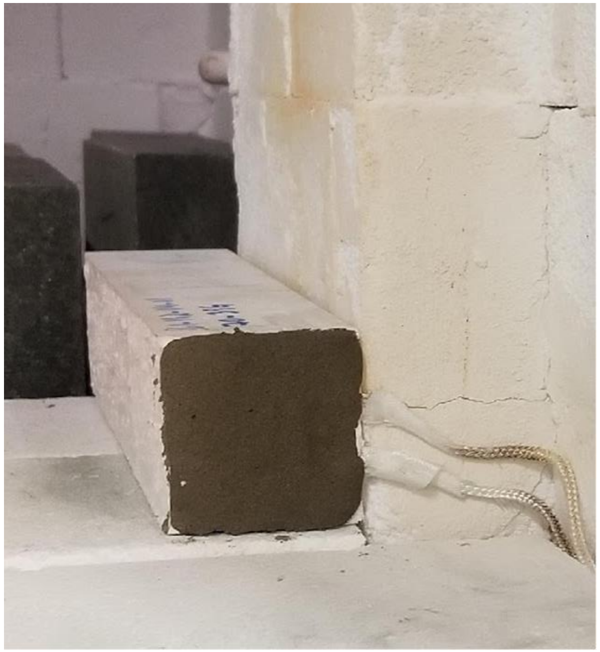


Refractory protection

# Gen 2 Sensor Brick Deployed at HWI:

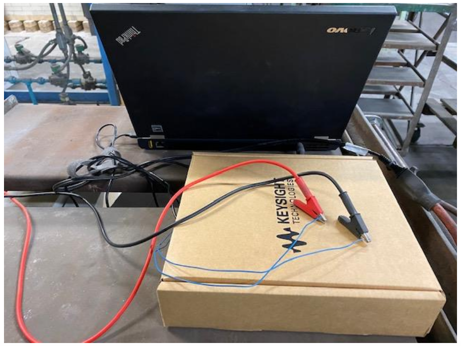
High Temperature Furnace at HWI

Gen2 Sensor Brick in door installation

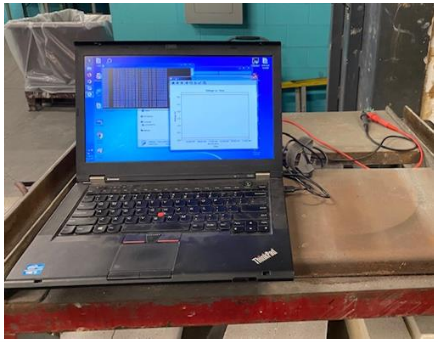


Connection to electronics

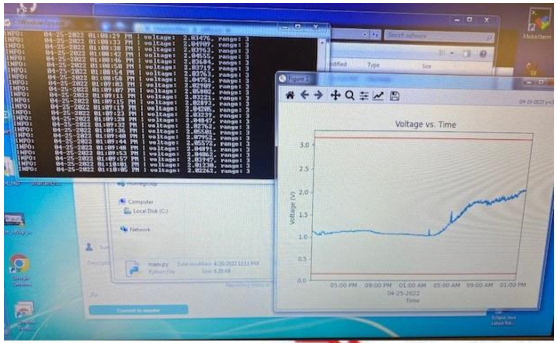

Connection to electronics




Connection to laptop




On-screen data monitoring



# *Task 6 Summary:*

**Completed Work**

➢ Full-scale production and testing of smart anchors with all components and installed at HWI Research Center for testing.

- Optimized sensor material system (Gen0, Gen2, and Gen3 sets)
- Low-power sensor electronics with wireless communication installed and testing

**Future Work**

➢ Continue longer-term testing of greater number of sensored anchors for statistics and better understanding of degradation mechanisms.

➢ Discussion with customers on issues for practical installation.

➢ Installation within continuous kiln production facilities within HWI Ohio and/or Kentucky (>1400°C operations for months). Next step for customer assurance of technology.

# *Acknowledgments:*


- We would like to thank **U.S. Department of Energy (DOE)** for sanctioning this project **DE-FE0031825**.

- **Robie Lewis and Maria Reidpath**, project managers at U. S. Department of Energy, is greatly appreciated for his insight and valuable guidance.

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- Kindly acknowledge faculty and staff of West Virginia University for their support.

## *Thank you!!!*

# Questions

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