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

Team:

Dr. David Graham[a]

Dr. Edward M. Sabolsky [b]

Dr. Kostas Sierros [b]

Ms. Margaret Raughley[c]

[a]Lane Department of Computer Science and Electrical Engineering, WVU

[b]Department of Mechanical and Aerospace Engineering, WVU

[c]HWI International, Advanced Technology and Research Center

2021 Crosscutting Research and Advanced Energy Systems Project Review Meeting

SENSORS & CONTROLS PROGRAM

May 19, 2021 (9:00 am)

Contract DE-FE0031825

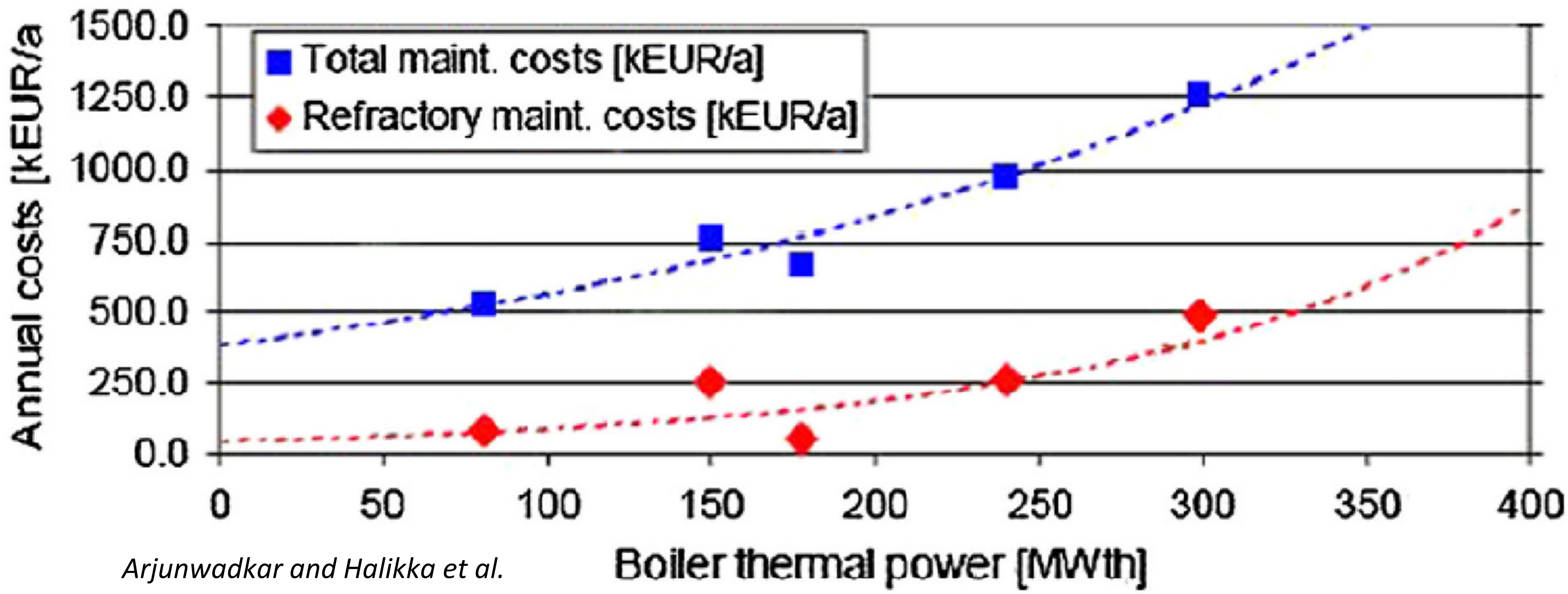
Initial Acknowledgments:

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

HWI

Background:

- Many different coal boiler technologies, but generally they have similar harsh environment refractory requirements.
- Refractory must resist high temperatures (~850-1500°C), erosion (by particles up to 6 mm OD and velocity of 30 m/s), and corrosion by alkali/alkaline-earth slag.
- Refractory maintenance is on average 25% of the annual total cost of coal boilers, and can reach as high as 40% of the total.

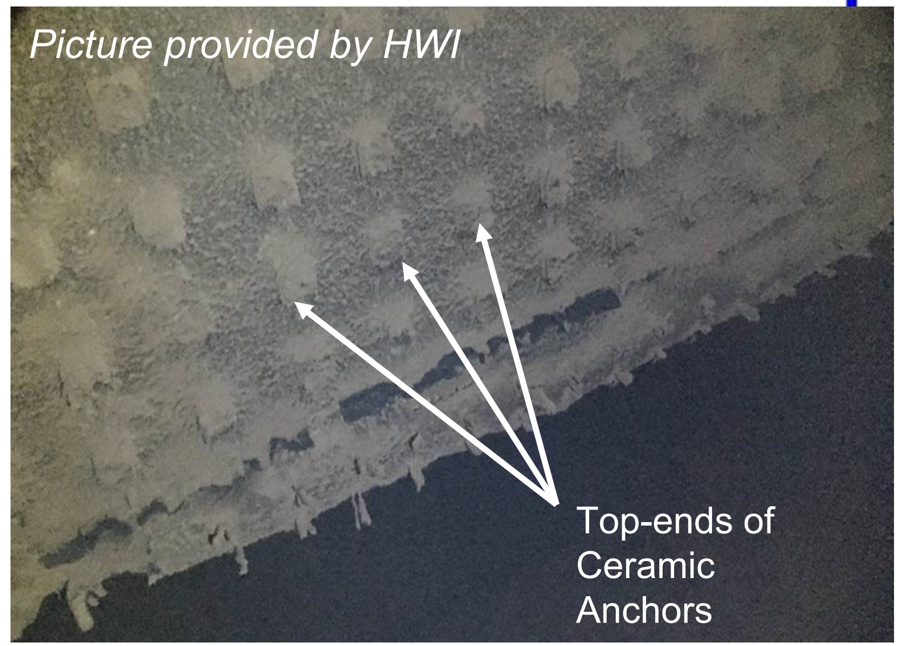


Arjunwadkar and Halikka et al.

Background:

- 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

Monolithic Refractory Furnace Wall after Shut-Down



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

- Monitoring of crack propagation and stress/strain development 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 with anchor clip).* **(Task 4 & 6)**

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

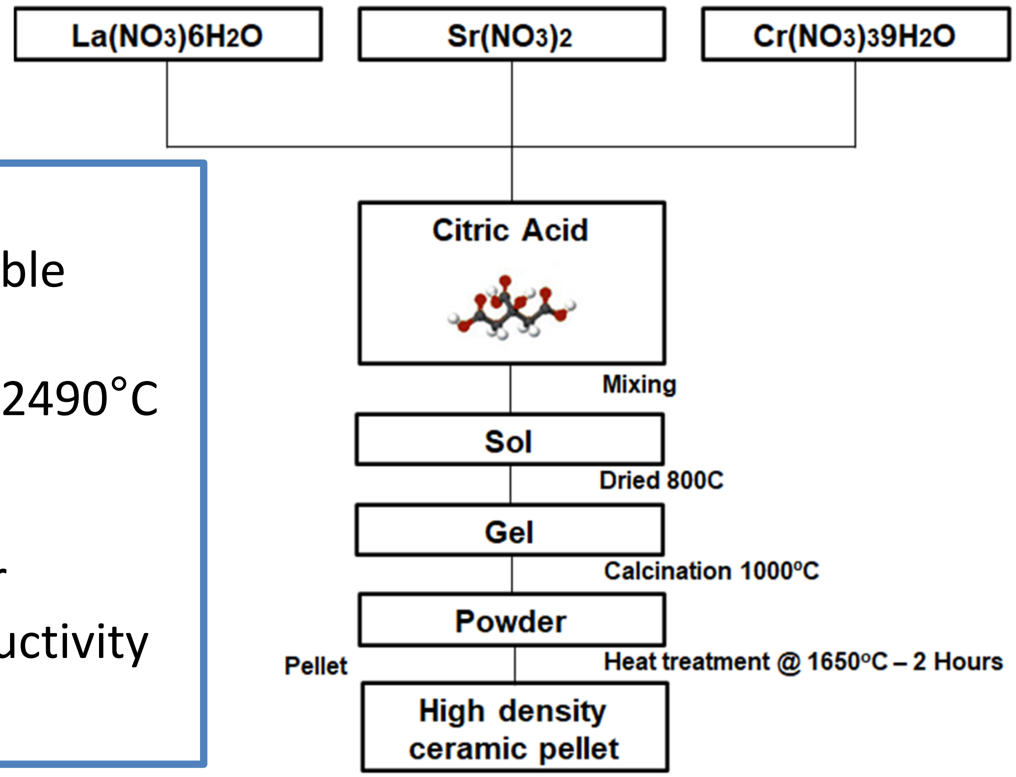
- ***Subtask 2.1- Synthesis and Thermal Processing of Perovskites and Composites***
  - The conductive oxide composites will be synthesized in-house.
  - Thermal processing in an atmosphere (e.g. air, argon) will be completed at high temperatures (up to 1500°C).

- ***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.

Compositional Research:

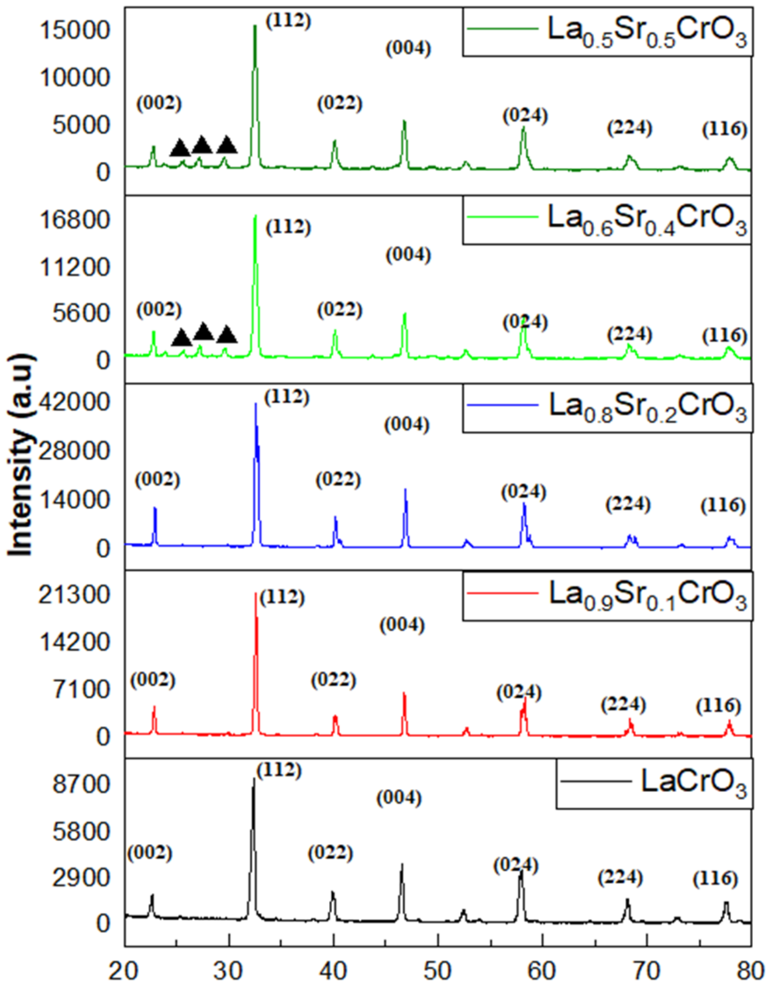
LaCrO$_3$

- High-temperature stable perovskite ceramic
- Melting temperature 2490°C
- CTE ~9.5×10^{-6} /K^{-1}
  (near Al_2O_3 and Cr_2O_3)
- P-type semiconductor
- Dope to control conductivity behavior

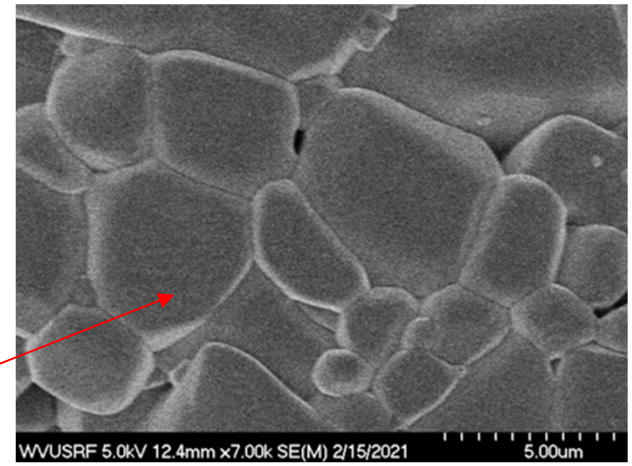


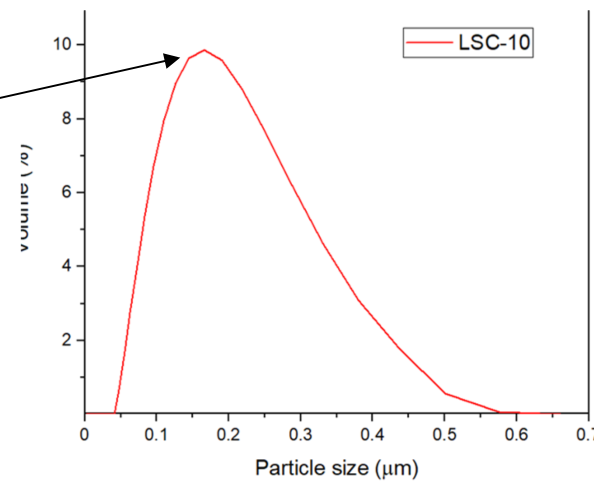
- ➢ Doped A- and B-site lanthanum chromites (LC) compositions were prepared (such as A-site= Sr, Ca and B-site= transition metals)
- ➢ All compositions were prepared by Pechini modified sol–gel method.

Strontium-doped LC characterization:



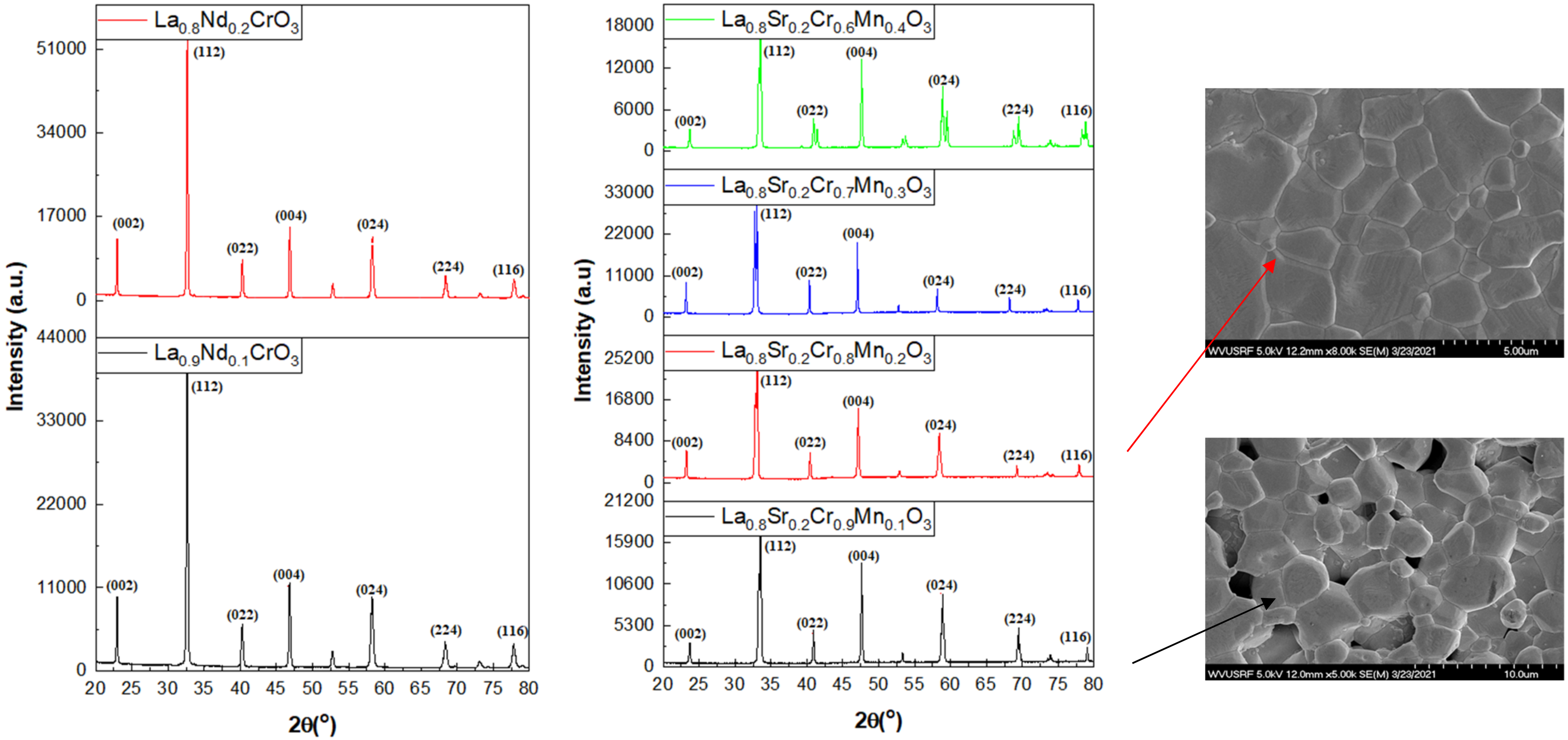
▲ $SrCrO_4$





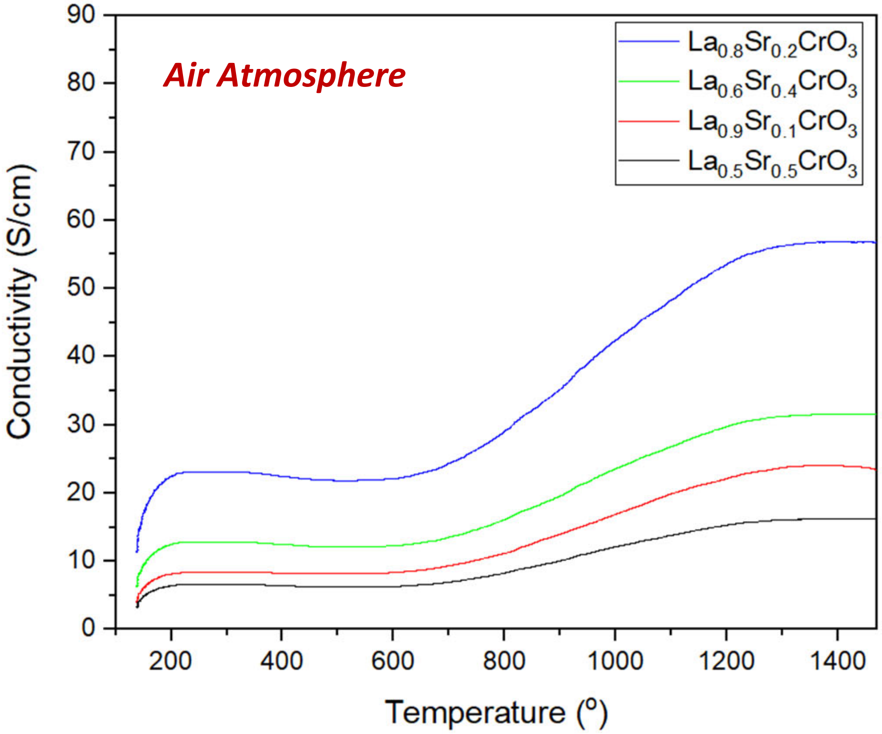
➢ High theoretical density was achieve for all compositions (94-96%).

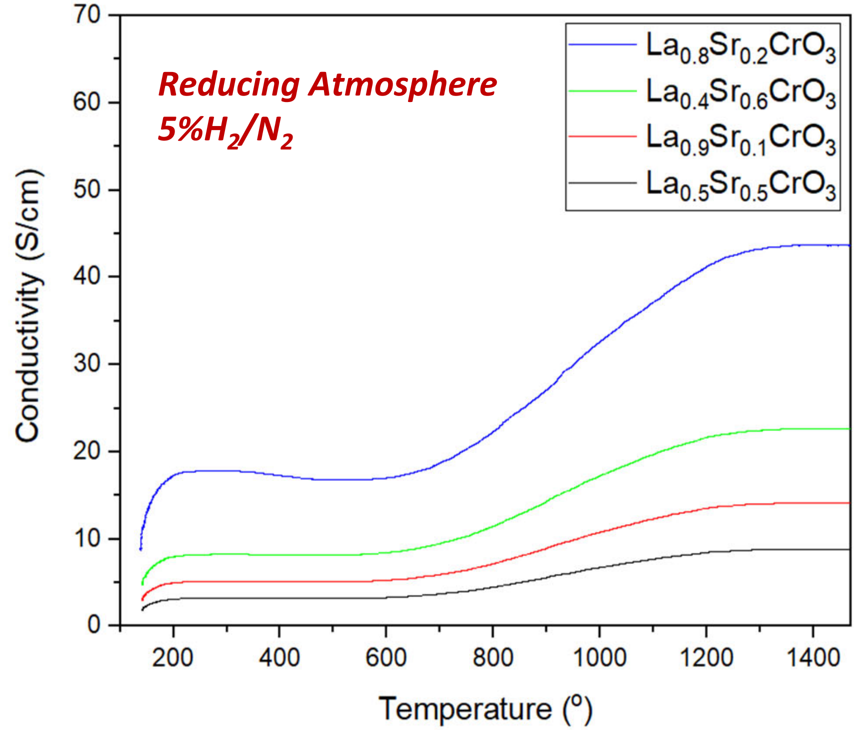
Nd and Mn doped LC characterization:



- ➢ Single phase powders materials were obtained by Pechini method at various doping levels.
- ➢ Particle size and chemistry was controlled to achieve high densities for all compositions (93-96%).

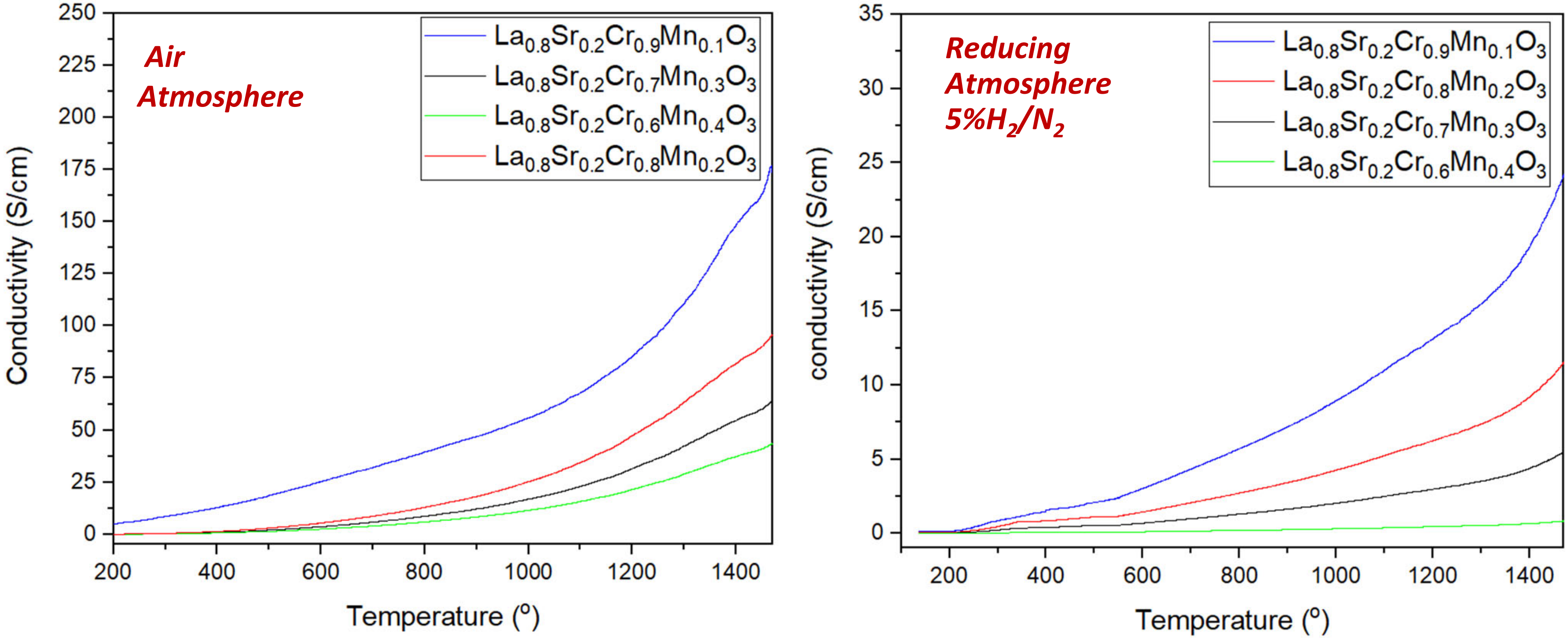
LSC Electrical Conductivity Testing:





- Under high pO_2, there is high p-type conduction (~60 S/cm at high temperature).
- Constant trends in specific temperature regimes (not good for thermal sensors).
- Lower lower pO_2, compensation by oxygen formation lowers electrical conductivity.
- At higher strontium doping levels, conductivity decrease by secondary phase formation.

LSC Electrical Conductivity Testing:



- Electrical conductivity increases (to even low temperature) with Mn additions (>175 S/cm at high-temperature).
- No linear portions within conductivity trend with temperature (better for sensors and interconnects).
- P-type conduction continues to be reduced with lower pO_2.

Task 2 Future work:

- Synthesis and characterization of novel n-type semiconductors.
- Electrical conductivity measurements under oxidative, reducing (and inert atmosphere) for n-type and p-type doped-perovskite compositions.
- Electrical conductivity at different oxygen partial pressures for all conductive ceramic compositions.
- Thermoelectric and thermomechanical measurements for n-type and p-type doped conductive composite compositions.

Summary and Completed Milestones:

- Task 2 – *on schedule*
  - Doped lanthanum chromites perovskites prepared by Pechini sol-gel methods.
  - Doped chromites perovskites characterized by XRD, SEM, DLS and Archimedean density.
  - Electrical conductivity testing of all prepared doped lanthanum chromite compositions at temperatures up 1500°C under air and reducing atmospheres.

Subtask	Milestone	Planned Completion	Actual Completion
2.1	Downselect conductive composition for sensor printing	08/31/2020	08/27/2020
2.2	Materials Characterization and Testing	08/31/2020	08/31/2020

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.

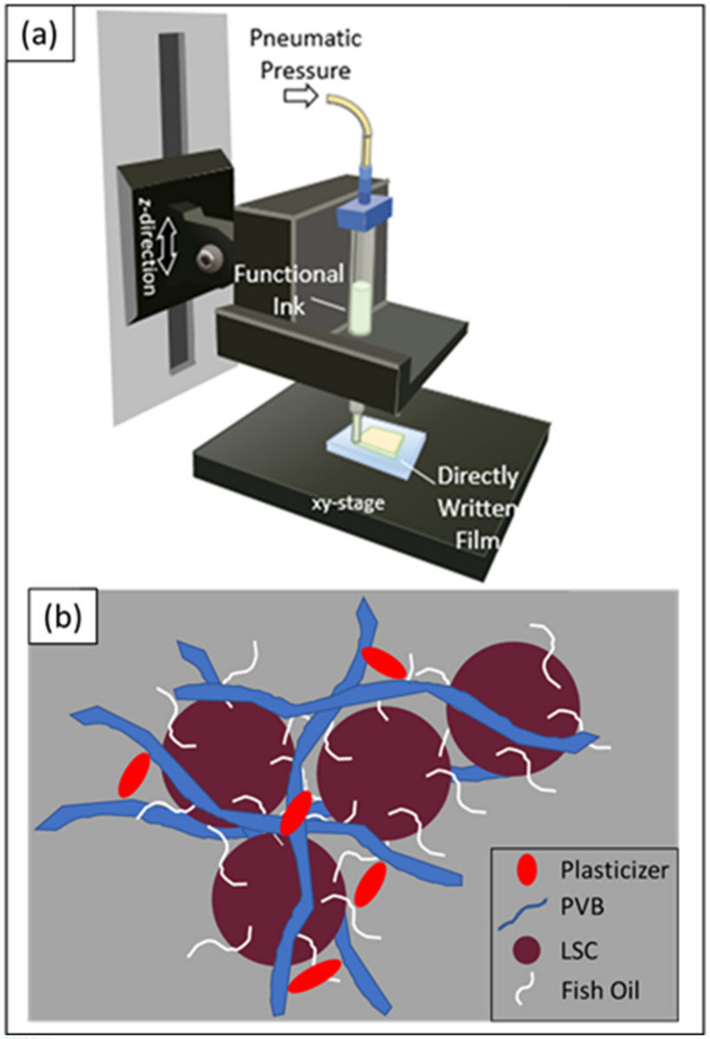
- ***Subtask 3.2 Direct-Writing/Patterning and Drying Characterization***
  - Direct writing deposition, drying, and thermal post-processing will be defined, which includes methods to control wetting and drying characteristics of the deposited composite solutions (for both refractory and sensor circuit 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 Process:



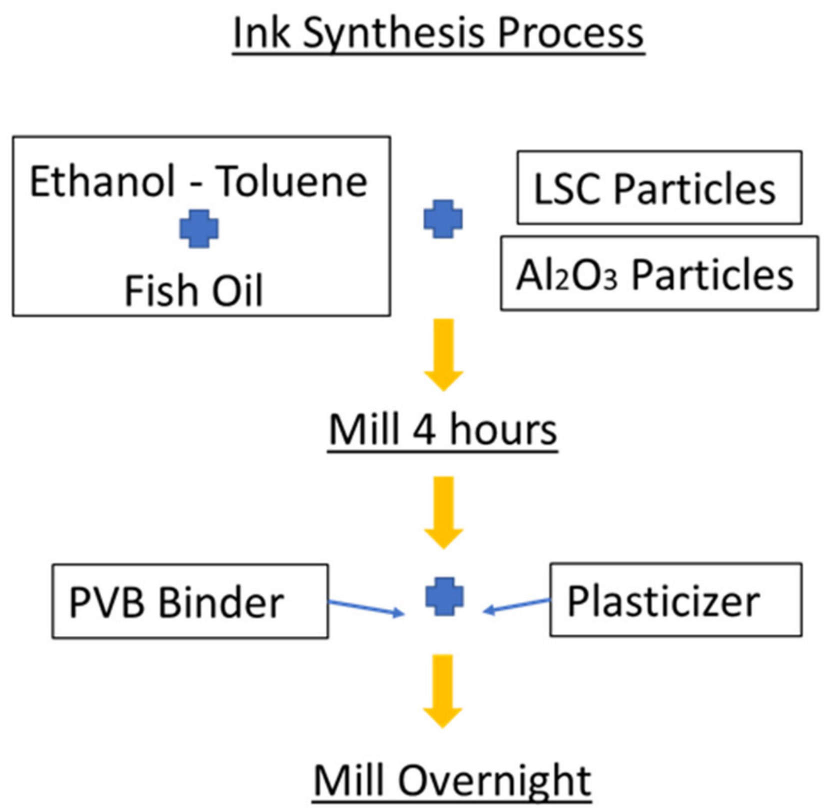
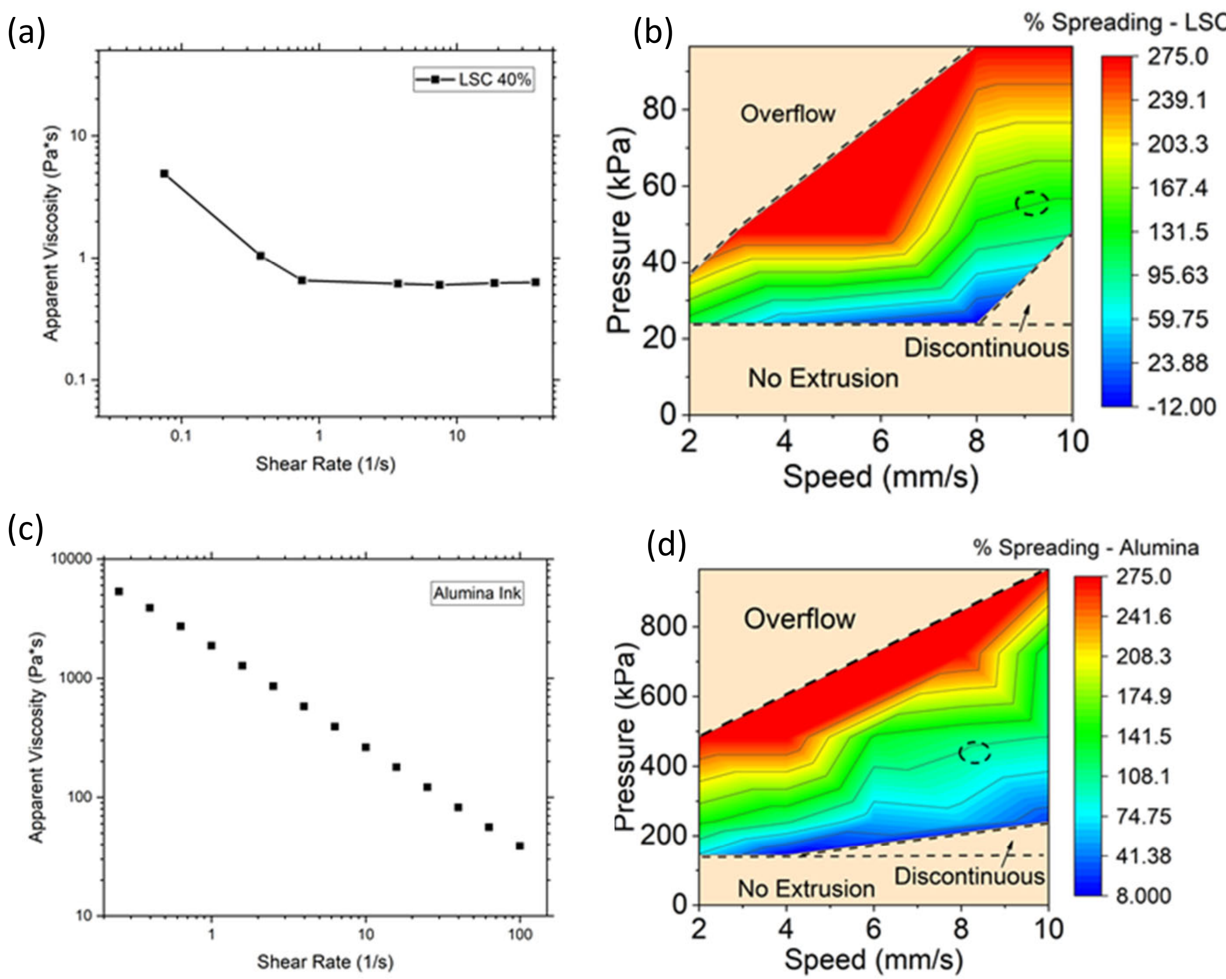
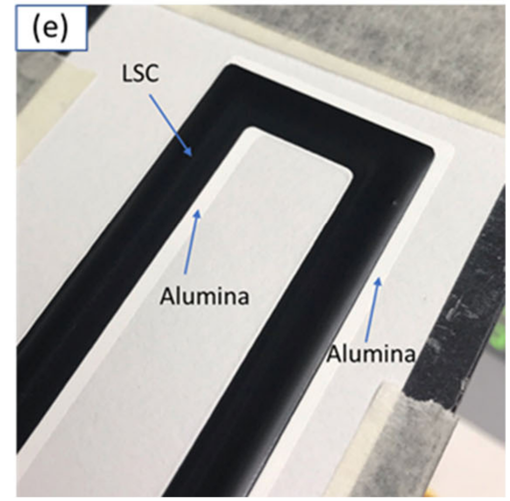


Fig 1: (left) (a) Direct Ink Writing (DIW) process schematic and (b) LSC ink schematic. Fish Oil acts as a dispersant, and PVB the matrix material. Ethanol – Toluene cosolvent system (gray) prevents nozzle clogging, while enabling high resolution features.

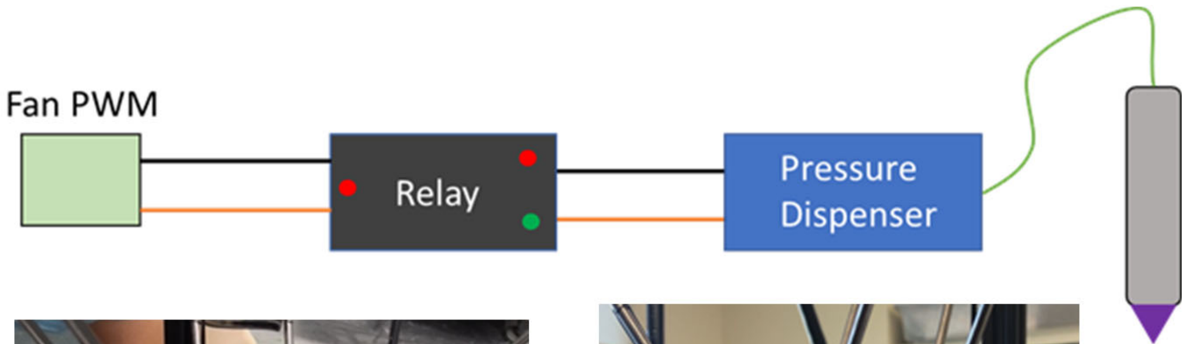
Ink Characterization:

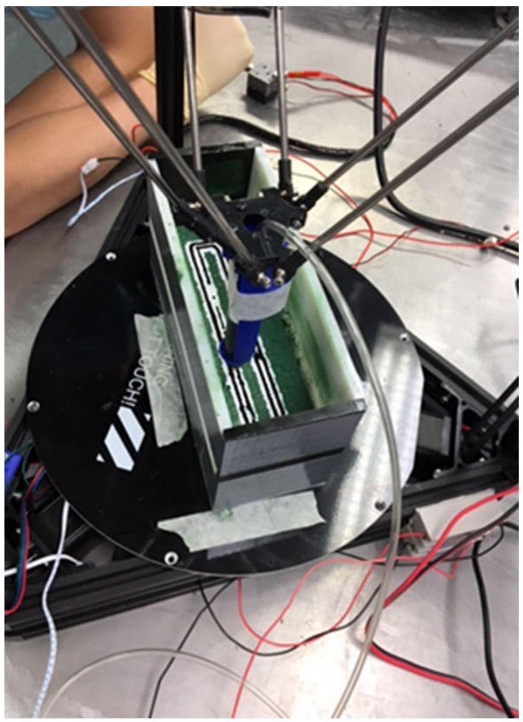


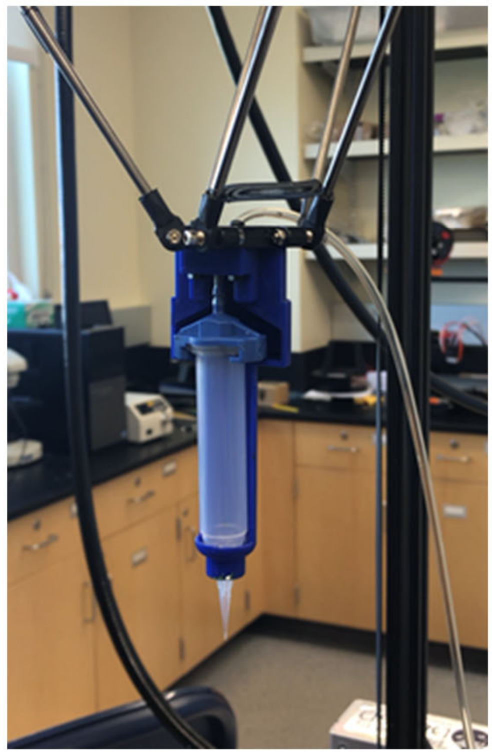


- ➢ Inks display shear thinning behavior
- ➢ Circled regions in b) and d) indicate regions used to print sensors with a 250 μm nozzle

Printing Platform Preparation:

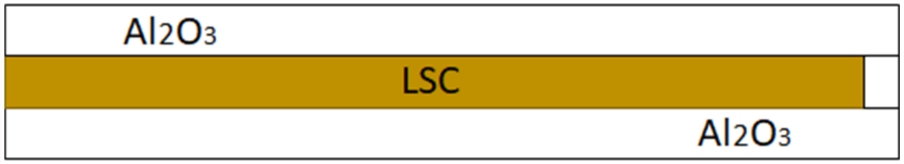






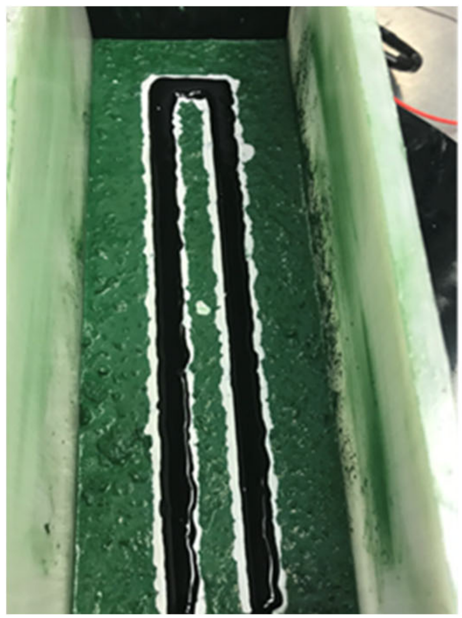
- ➢ Delta printer hardware and software modified for syringe printing
  - G-code triggers PWM control pins
- ➢ XYZ travel controlled by carriage system
  - Reduced toolpath profile

Smart Refractory Printing:



Thermistor Architecture

- ➢ Control of ink-substrate interaction critical to manage spreading
- ➢ Perform complete refractory-ink parametrizations

Mold ⇨ Printing first layers ⇨ Encasing thermistor ⇨ Finished cast

Future Work:

- Refractory substrate parametrization
  - Adjust toolpath and printing parameters to achieve uniform first layer

- Surface mapping and real-time nozzle adjustment for rough terrain
  - Maintain constant ink flow rate and nozzle height
  - Integrate laser adjusted feedback
  - Surface scan and adjust toolpath

- Adjustment of sensor geometry for electrical team
  - Use revised LSC conductivity and shrinkage

Summary and Completed Milestones:

- Task 3 – *on schedule*
  - LSC ink formulated and characterized
  - Preform fabrication time reduced considerably by use of 3D printing
  - 3D printing system delivered and used to print thermistors in refractory

- Sensor fabrication for quality control testing complete

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	12/31/2020	11/04/2020
3.4	3D Printing of Sensor During Refractory Fab.	10/31/2021	03/25/2021
6.1	Delivery and Setup of 3D Printer at HWI	06/30/2021	03/25/2021

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.

Sensor Interconnection by HT Brazing:

- Embedded sensors require strong ceramic/metal ohmic joints to interconnect the sensor to the electronics (through an insulated wire).
- Brazes have been developed for *mechanical* ceramic/metal joints, but have not been evaluated extensively for high-temperature *electrical* connection applications.

- Current work investigating Ni- and Ag-based brazes to act as ceramic/metal ohmic contacts.

Ni-based brazes:

a) Ni (~3.5% Si, ~1.9% B), AWS BNi-3 and BNi-4

b) Brazing temperature ≈1150-1250 °C

c) Wire Ni, Ni Alloy600, and Ag

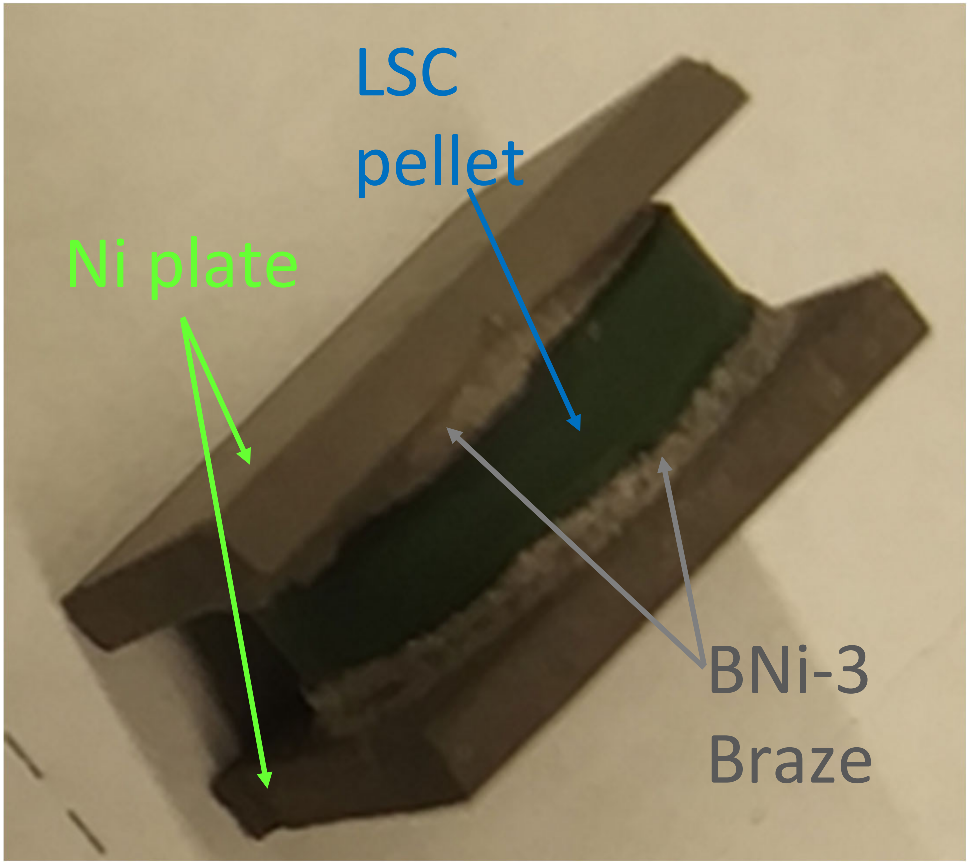
Ag-based brazes:

a) Ag (~27-30% C), AWS BAg-8

b) Brazing temperature ≈770-890 °C

c) Wire Ni, Ni Alloy600, and Ag

Ni and Ag Brazed Interconnects:



- 1160 °C for 20 min in Ar gas flow.
- Symmetrical sample fabricated to measure interfacial resistance by 4-point method.
- Ag-braze showing good adhesion and low resistance currently (carried to Task 6).

- Matrix of brazing experiments are being completed with Ni- and Ag-brazes with various thickness, brazing temperature/time, pressure, and atmosphere.
- Interfacial resistance, strength, and thermal stability are being evaluated for sensor interconnect application.

Future Work:

- Complete evaluation of processing variables to study ceramic/metal symmetrical samples of Ni, alloy 600, and Ag metals with LSC.

- Define contact resistance and change over time (stability) and effect on sensor signal.

- Develop engineering method to braze connection onto smart refractory anchors (discussed further in Task 6).

Summary and Completed Milestones:

- Task 4 – *behind schedule*
  - Ni-based and Ag-based brazes were used to connect to Ag and Ni wire to LSC ceramics.
  - 4-point resistance tests at elevated temperature were initiated for this tests, but not completed at this time.
  - Ag-based brazing filler metal paste, BAg-8, was used to braze the sensor of the smart anchor with silver wire (and sensor was tested to 1200°C, discussed further in Task 6).

Subtask	Milestone	Planned Completion	Actual Completion
4.1	Demonstrate initial braze for sensor interconnection	03/31/2021	04/08/2021 using Ag-based braze, (AWS BAg-8)

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

Task 5 Objectives:

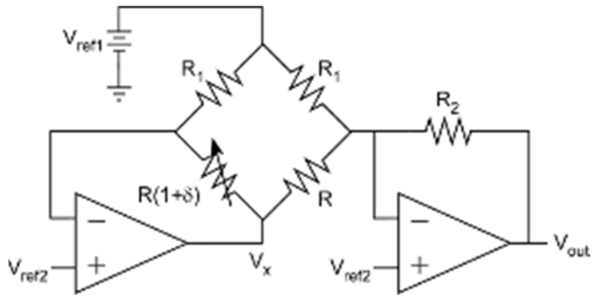
- To develop methods to interface the electrical sensing outputs from the smart refractory with an embedded processor
- To design a wireless sensor network to efficiently collect the data at a processing unit for further data analysis

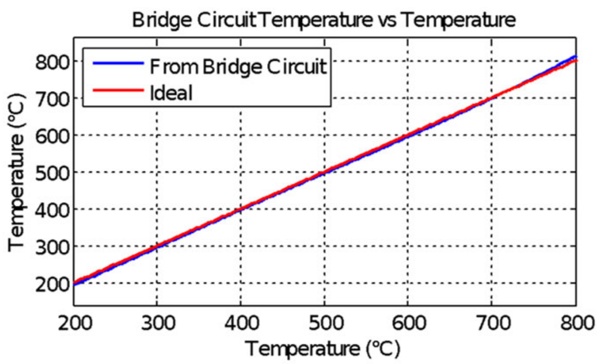
Task 5.0 – Electronics and Wireless Communication Interfacing with Smart Ceramic Anchors

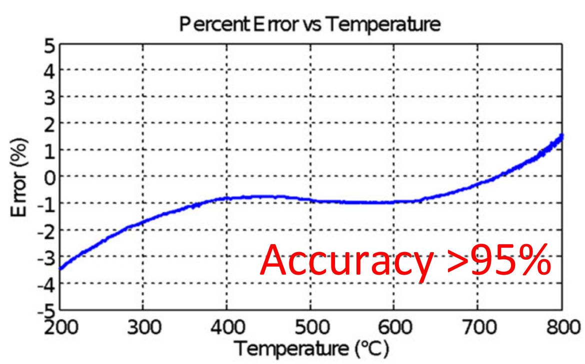
- ***Subtask 5.1- Interfacing Electrical Outputs to Motes***
  - Initial interface circuitry will be designed using discrete off-the-shelf components to obtain a working system and to allow for full system integration.
  - These interface circuits will be used for monitoring the sensors within the ceramic 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

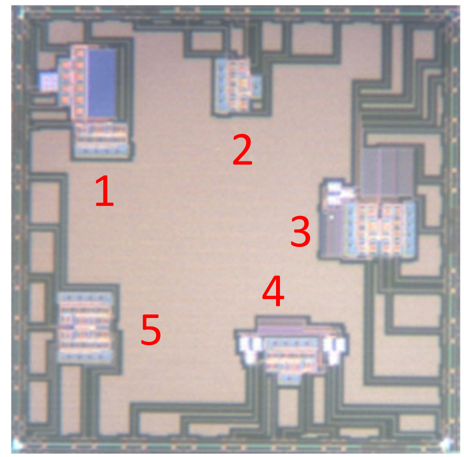
Discrete Sensor-Interfacing Circuits





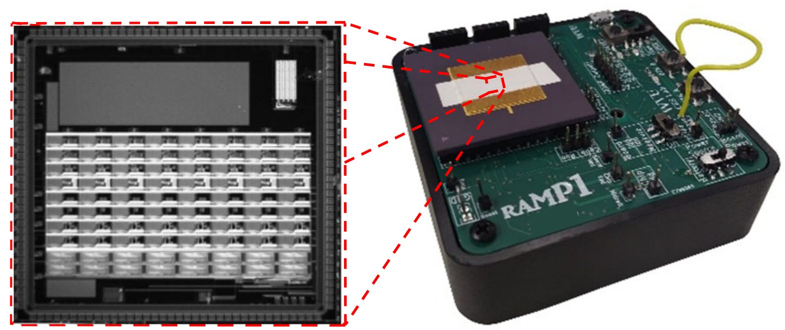


Custom Integrated Circuits



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

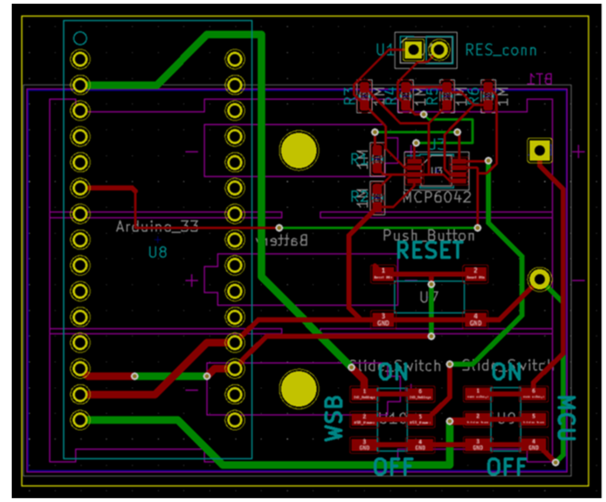
Reconfigurable Integrated Circuits

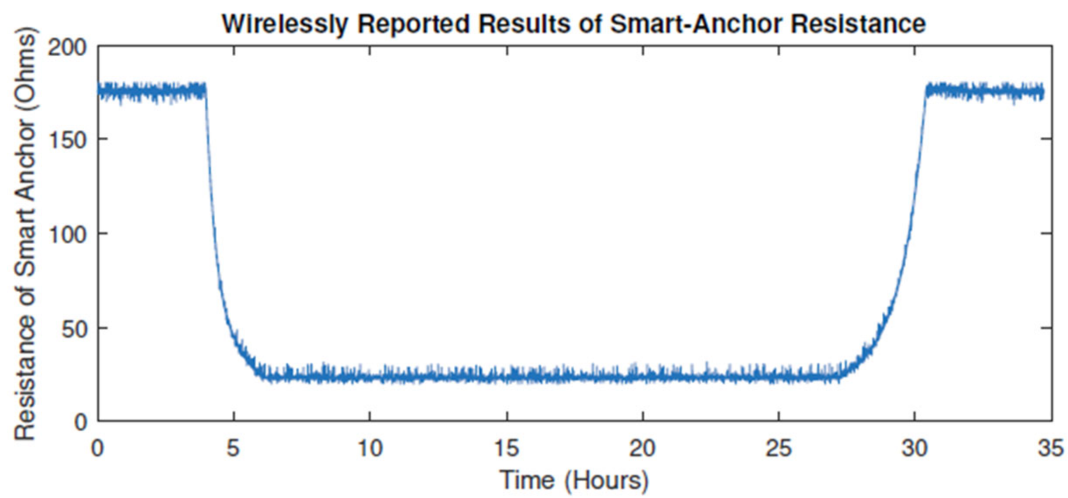


Flexibility post-deployment
Ultra-low power implementations

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
- Printed circuit board (PCB) designed

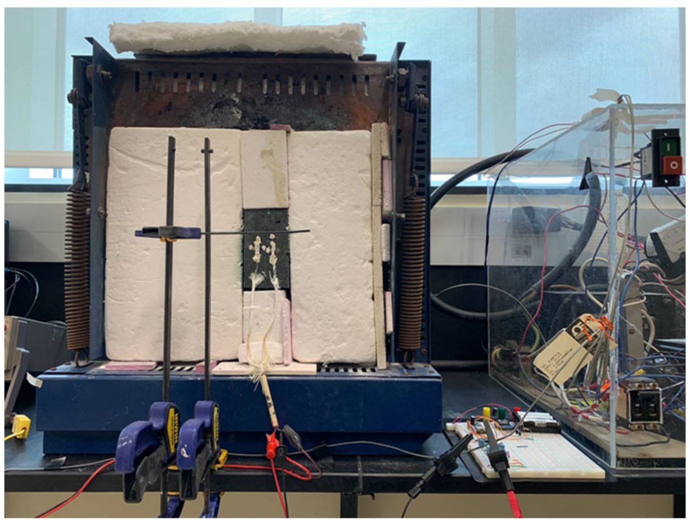




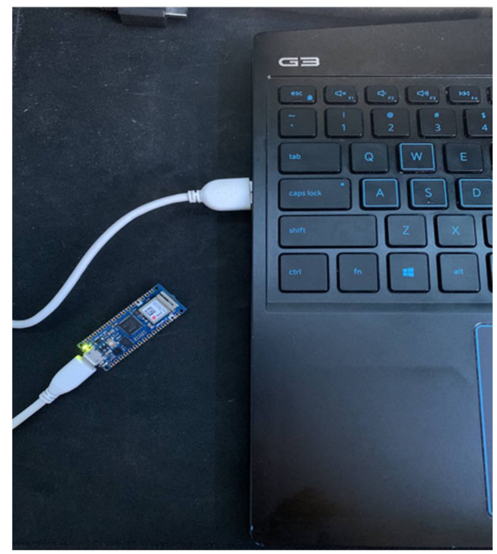
Task 5.0 System and Software Improvements

- Matlab -> Python
  - Library for reading serial interface on Nano 33 IoT
  - Library for automated email/text alarm system
  - Live update plot for visual monitoring

- Arduino Nano 33 IoT
  - Built-in ADC used to convert voltage to digital data
  - BLE transmits digitized data for Base Station to interpret



BLE



BLE

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

Task 5 Summary:

Task 5 Summary – *on schedule*

- Built prototype sensor interfacing circuit/platform
- Implemented better microcontroller/radio platform
- Developed prototype Python software infrastructure for data collection and analysis
- Demonstrated
- 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.)
- Implement auto ranging functionality on the sensor circuitry for accommodating a larger range of resistance/temperatures
- Improve the software infrastructure
- 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.

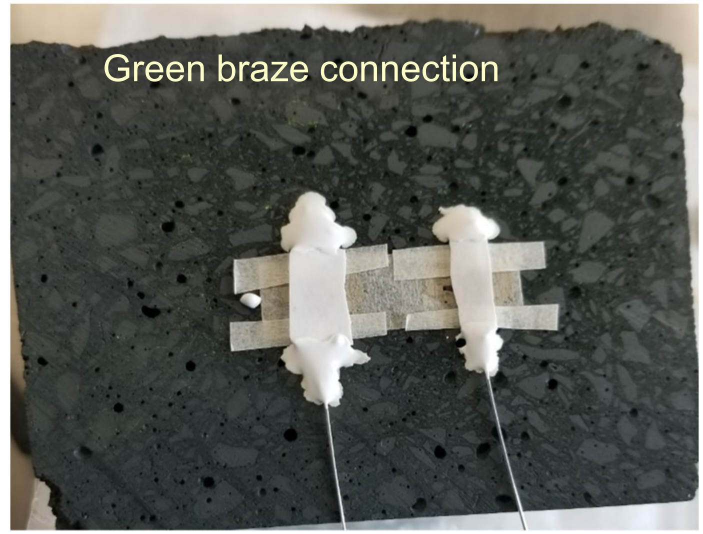
Task 6.0 Ceramic Anchor Manufacturing and Concept Demonstration.

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

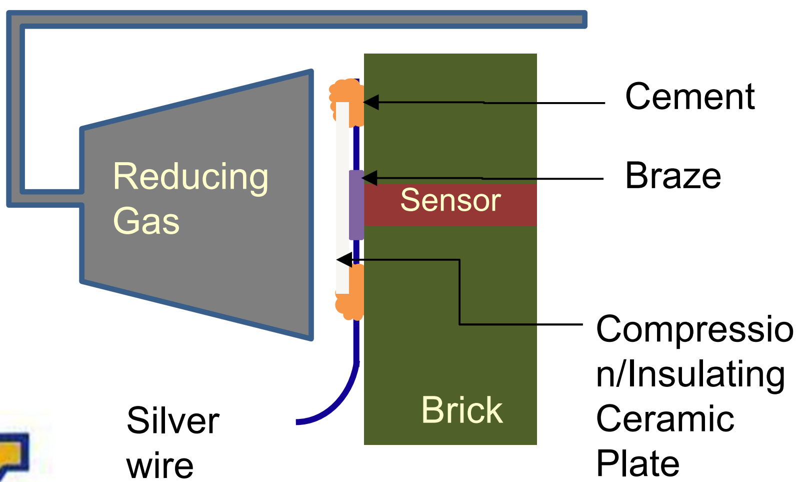
- A minimum of three (3) smart anchors will be installed within the liner of a coal boiler, ash hopper, or glass furnace floor.
- The demonstration will include at least three wireless sensor nodes utilizing the optimal electronic interface, power configuration, and wireless network defined in Task 5.0.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

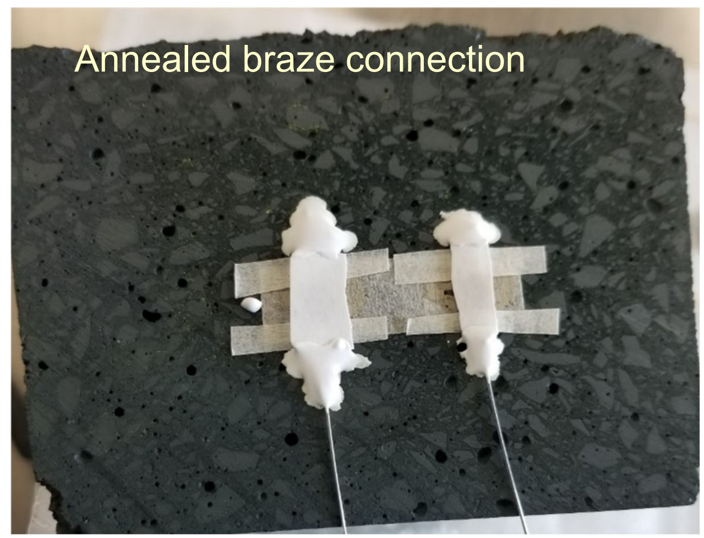
Anchor with Brazed Sensor Connection:

- Anchors were cast with printed sensors and cavity for slag testing
- Silver-Copper braze was used to attach sensor to silver leads



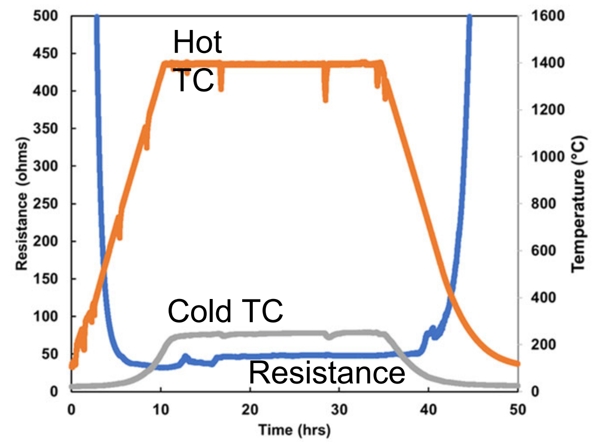
Braze Annealing under Localized Reducing Gas





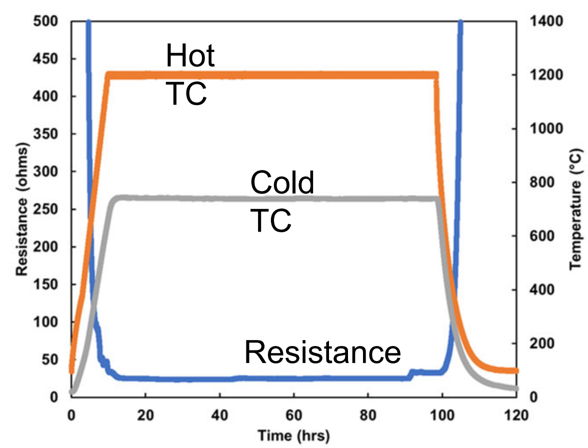
Anchor with Brazed Sensor Connection:

Test with LSC ink connection



R = 48 ohm at 25 hrs

Test with brazed connection



R = 24 ohm at 25 hrs

Future Work:

- Full-scale production and testing of smart anchors with all components and installed at HWI Research Center for testing.
  - Optimized sensor material system (Gen2 composition)
  - Brazed interconnection
  - Nears-sensor electronics with wireless communication

- Longer-term in application testing will be completed with entire system to better understand degradation and data acquisition issues.

Summary and Completed Milestones:

- Task 6 – *On schedule*
  - Fabricated smart anchors by 3D printing at HWI, initiated wiring of smart bricks and initial testing trials.
  - Also, began producing anchors for quality control testing for Go/No-Go set for June 30th, 2021.

Subtask	Milestone	Planned Completion	Actual Completion
4.1, 6.1	Initiate anchor property characterization to pass product standards	05/31/2021	Initiating next week.

Next Important Milestone:

Subtask	Milestone	Planned Completion	Actual Completion
4.3 Go/ No Go: B	Demonstrate smart anchor prototype which achieves >75% quality control standards (Modulus of Rupture, Cold Crushing Strength)	06/30/2021	