AOI[3]: Smart Refractory Sensor Systems for Wireless Monitoring of Temperature, Health, and Degradation of Slagging Gasifiers

Team:

Dr. Debangsu Bhattacharyya a
Mr. Jeff Bogan b
Dr. David Graham c
Dr. Vinod Kulathumani c
Dr. Edward M. Sabolsky d

aDepartment of Chemical Engineering, WVU
bHarbisonWalker International Technology Center
cLane Department of Computer Science and Electrical Engineering, WVU
dDepartment of Mechanical and Aerospace Engineering, WVU
Background- Gasifier Sensing Needs:

- Online monitoring sensors of refractory used in coal gasifiers under extreme conditions including high temperature (>1300°C) and high pressure (up to 1000 psi) for >20,000 hr.
- Erosive and corrosive conditions (due to slag and high pressure, in addition to various pO₂ levels) causes degradation of refractory over time.
- Ability to monitor the integrity of the refractory materials during gasifier operation would contribute significantly to improving the overall operational performance and reliability of coal gasifiers.
  - Temperature
  - Stress/strain within refractory liner
  - Spallation events
  - Refractory liner health

- Monitoring interior thermochemical conditions allows for efficient control of the gasification process.
**Technology Vision:**

**Item A** represents the “smart refractory” material.

**Item B** is an interconnection (alignment) pin.

**Item C** is an interconnection brick, which will permit transfer of the signal to the exterior wall.

**Item D** is the sealed electrical access port to connect to the signal acquisition/processing units.

**Item E** is low-power electronics and wireless communication.
1) Investigate chemical/thermal stability, thermomechanical properties, and electrical properties of refractory ceramic composites at temperatures between 750-1450ºC.

2) Define processes to pattern and embed the conductive ceramic composites within refractory materials to incorporate temperature and strain/stress sensors into refractory bricks.

3) Develop methods to interface the electrical sensing outputs from the smart refractory with an embedded processor and to design a wireless sensor network to efficiently collect the data at a processing unit for further data analysis.

4) Develop algorithms for model-based estimation of temperature profile in the refractory, slag penetration depth, spallation thickness, and resultant health by using the data from the wireless sensor network.
Task Assignments:

- Task 2: Fabrication and Characterization of Oxide-Silicide Composites.
- Task 3 and Task 4: Sensor Patterning and Embedding and Static and Dynamic Sensor Testing.
- Task 5: Data Ex-Filtration Using a Wireless Sensor Network.
- Task 6: Model-Based Estimation of Refractory Degradation/Temperature.
- Task 7: Smart Brick System DEMO in Simulated Reactor (to be completed in summer).


Products:


*8 More publications currently being prepared, 2 patents, and 2 more oral presentations in the summer/fall 2017

Students worked/graduated:

- Rajalekshmi C. Pillai (Post-doc)
- Gunes A. Yakaboylu (PhD)
- Qiao Huang (PhD)
- Spencer Clites (MSc)
- Steven Andryzcik (MSc)
- Priyashraba Misra (MSc)
- Brian Armour (Undergraduate)
- James Meyer (Undergraduate)
- Aaron Teter (Undergraduate)
Task 2: 
Fabrication and Characterization of Oxide-Silicide Composites. 
(Sabolsky)
Task 2.0 Objectives:

- Investigate chemical/thermal stability, thermomechanical properties, and electrical properties of refractory silicide-oxide composites at temperatures between 750-1450°C.
**Chemical Stability (XRD):**

<table>
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<th></th>
<th>Al₂O₃</th>
<th>Y₂O₃</th>
<th>ZrO₂</th>
<th>Cr₂O₃</th>
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</tr>
<tr>
<td>TaSi₂</td>
<td>TaSi₂, Al₂O₃, Ta₅Si₃, Ta₃Si, SiO₂</td>
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<td>(Cr₀.₈₈Ti₀.₁₂)₂O₃, Cr₃Si, SiO₂</td>
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<tr>
<td>CrSi₂</td>
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<td>CrSi₂, ZrO₂</td>
<td>CrSi₂, Cr₂O₃, Cr₃Si, SiO₂</td>
</tr>
</tbody>
</table>

*Prepared via mixed oxide route followed by sintering in argon atmosphere at 1400°-1600°C.*

- Metal silicides show high stability in Al₂O₃ and ZrO₂ matrix only with formation of different type of silicides (Mo₅Si₃, W₅Si₃, Nb₅Si₃, Cr₃Si, etc.) and SiO₂ (highlighted).
- They partially react with Y₂O₃ and Cr₂O₃ to form undesired secondary phases.
Composite Microstructures (SEM):

- 60-40 MoSi₂-Al₂O₃
- 60-40 MoSi₂-Y₂O₃
- 60-40 MoSi₂-ZrO₂
- 60-40 WSi₂-Al₂O₃
- 60-40 WSi₂-Y₂O₃
- 60-40 WSi₂-ZrO₂

Chemically etched in 1:1:1 HCl:HNO₃:H₂O

3/27/2017
Electrical/Thermal Properties:

4-probe electrical conductivity up to 900°-1000°C

CTE (100°-1000°C)

- 60-40 MoSi$_2$-$\text{Al}_2\text{O}_3$ (coarse) and WSi$_2$-$\text{Al}_2\text{O}_3$ (fine) exhibited higher electrical conductivity at 900°C.
- Density, particle distribution/size and secondary phase highly influenced the physical properties.
They showed high thermal stability at 1400°C up to 48h, only with formation of 5-3 silicide and silica phases.

The rates of grain growth were highly decreased by grain boundary pinning effect.
Task 2 Conclusions and Future Work:

- Metal silicides show high chemical/thermal stability in Al₂O₃ and ZrO₂ matrix (with occasional formation of sub-silicides).
- Electrical conductivity of composites characterized with various silicide content → consistent performance transferred to sensor design and fabrication task.

Future Work:

- Alternative compositions (Cr-silicides, etc.) and designs will be investigated to prevent the reaction between silicide/oxide composites and Cr₂O₃.
- Process parameters will be optimized by correlating the homogeneity (D index) with the physical properties of the composites (percolation, etc.) at high temperatures.
Task 3: Sensor Patterning and Embedding. (Sabolsky/HWI)

Task 4: Static and Dynamic Sensor Testing of Smart Refractory Specimens. (Sabolsky/HWI)

*US Provisional Patent Number 61/941,159
**Task 3 Objectives:**

- To develop methods for patterning technology the ceramic composites within the refractory matrix.

**Task 4 Objectives:**

- To test the electrical performance of the smart refractory brick (with embedded thermocouple or thermistor sensors).
- To investigate corrosion/erosion kinetics in static and dynamic tests on smaller prototype and full-size smart cups and bricks (at WVU and HWI).
- To implement and test methods for data collection on initial prototypes.
Smart Refractory Fabrication:

General Smart Refractory Processing Method

Examples of Sensor Preforms

- Thermocouple/Thermistor
- Resistive Circuit
- Temperature
- Spallation
- Strain Sensor (Rosette)
- Capacitive Sensor
- Stress/Strain

1st Part of a Refractory Brick

Ceramic Preform or Organic Film with Sensor Pattern

2nd Part of Refractory

Combined (or Stacked) Parts form Embedded Sensor Structure

Refractory with Embedded Sensor is Thermally-Processed (Sintered)
High-Temperature Thermocouple Performance:

The thermocouple with composition MoSi$_2$//TiSi$_2$ exhibited 34 mV at 1400 °C.

90 vol% silicide and 10 vol% oxide.

Various thermocouple compositions studied at 1500 °C.

The thermocouple with composition MoSi$_2$//TiSi$_2$ exhibited 34 mV at 1400 °C.
Monoliths of sensors were fabricated via tape casting, laminated and sintered at 1500 °C. These laminates were embedded in the Cr$_2$O$_3$ brick while slip casting and co-sintered at 1500º C in Argon atmosphere.
The temperature sensor (thermistor) with composition (60-40) vol% MoSi$_2$-Al$_2$O$_3$ embedded within the Cr$_2$O$_3$ refractory exhibited stable behavior for more than **250 hours at 1350 °C** in argon atmosphere.
Embedded Thermocouple: Smart Chromia Brick

- Sensor preform embedded HWI high-chromia formulation.
- Sensor embedded below opening to insert slag composition for corrosion testing.

Thin Embedded TC Sensor

Interfaced Smart Brick

Long TC embedded Chromia Refractory Brick
Current Issue: Brick Interconnection

- Loss of electrical connection to bricks during testing.
- Metal lead delamination due to wetting limitations and Pt-Si alloy formation.
- Phase oxide formation in locations that are not embedded causing drift in response.

Efforts are focusing on better understanding the issue and developing the proper ceramic and/or metal connections.
Alternative Sensor Materials
(Heading towards Demonstration Task)

Risk Mitigation:
• Two alternative oxide compositions initiated in August 2016.
• Fabrication and testing completed, and new bricks for summer Task 7 DEMO being manufactured at HWI.

Approach:
High conductivity electrode material, \( \text{La}_2\text{NiO}_4 \) (LNO) and \( \text{Sr(Ti,Nb)}\text{O}_3 \) (STNb) was evaluated as alternative sensor materials, stable in air and slightly reducing atmospheres.
• Stable interconnect junction and does not react with Pt leads.
• Synthesized at WVU by solid state processes.
• Screen printed on and within zirconia, and embedded within high-chromia bricks.
Alternative Sensor Material: $\text{La}_2\text{NiO}_4$

- $\text{La}_2\text{NiO}_4$ (LNO) - High p-type semi-conducting material.
- Initial LNO thermistor sensors showed an increase in resistance during 5 hr hold.
- Increased sintering temperatures destabilized microstructure and caused variable response.

![Graphs showing resistance and temperature changes for LNO sensor tested to 1000°C and 1350°C](image)
**Alternative Sensor Material: La$_2$NiO$_4$**

- Coarser (more aggregated sensor material, >10 µm average particle size) currently being tested to show stable high-temperature response.
- The thermistor tested to 1400°C showed nice stability.

YSZ imbedded thermistor imaged over light box
Alternative Sensor Material: $\text{Sr(Ti}_{0.8}\text{Nb}_{0.2})\text{O}_3$

- $\text{Sr(Ti}_{0.8}\text{Nb}_{0.2})\text{O}_3$ (STNb) - High n-type semi-conducting material.
- The thermistor was fabricated using zirconia substrate.
- Thermistors were sintered at 1450°C and tested to 1400°C.

Screen printed sensor material on green zirconia
Task 3 and 4 Conclusions and Future Work:

- All-ceramic thermocouple and thermistor preforms (and smart bricks) were fabricated and successfully tested, but issues with interconnection.
- Two new alternative oxide-based systems also being investigated in parallel to show full brick and system demonstration.

Future Work:

- Optimize method to interconnect to embedded sensors (in order to stabilize sensor signal and sensor long-term response).
- Complete fabrication and testing of all-oxide based sensor preforms in smart brick architecture.
- Scale-up all sensor preforms and smart refractory brick for FULL-TECHNOLOGY DEMO IN TASK 7.
Task 5: Data Ex-Filtration Using a Wireless Sensor Network. (Graham/Kulathumani)
**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.
Electronics interfacing – Approach:

**Aim**: To reliably collect data from the sensors embedded within the smart bricks and interface them to wireless sensor nodes for communication

**Approach**:

(i) Iterative approach to sensor interface circuitry in parallel with the sensor development
   a) Initial sensor interface circuitry using off-the-shelf circuitry
   b) Move to integrated circuits for lower-power and more compact solutions

(ii) Investigate energy harvesting using thermoelectric devices to help power the sensor motes and interface circuitry
Custom Integrated Circuit:

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

Resistance-Based Sensor

\[ V_{out} = V_{ref2} + \frac{R_2}{R_1} \delta (V_{ref1} - V_{ref2}) \]

Bridge Circuit Temperature vs Actual Temperature

Within ±2% accuracy
Circuits for Thermocouple-Based Sensors:

- Compensates for measurement error at thermocouple cold junction
- Adds offset to thermocouple input to allow for the correct temperature measurement
- Greatly improves accuracy over a large range of temperature values
Circuits for Energy Harvesting:

- Leverages COTS-based DC/DC converter circuit (LTC3108)
- Start-up Sequence shows the output of Thermoelectric Generator, LDO Regulator, Storage Buffer, and $V_{OUT}$
- The Mote Experiment was done using a TelosB. The results shown are 10 minutes into the test. Once a minute, the TelosB turned on and was powered by the energy harvesting system for a 5 second radio transmission.

$V_{OUT}$ – System Output
Storage Buffer – Energy Storage Output
LDO Regulator – Internal Regulator Output
TEG – Thermoelectric Generator Output
Reconfigurable Circuits:

- Maintain flexibility—in-the-field updates to sensor-interfacing circuits
- A variety of sensor interface circuits can be constructed from a single chip
- Internal temperature compensation using floating-gate transistors
Wireless sensor network overview:

- Collect refractory sensor data over wireless medium
- (data exfiltration)
- Enable remote configuration of parameters
  - (over-the-air programming)
Previous work

- Assembled full signal chain
  - Smart bricks with embedded sensors were interfaced with a wireless mote yielding a complete signal chain comprising
    - the smart brick,
    - resistance measurement / amplifier circuit,
    - and wireless data transmission.
- Verified wireless signal chain performance on smart brick prototype
- Developed visualization tools and sensor parameter control interface
- Tested network performance at scale using network simulator
Current work: Model based strategies

- **Goal: Talk less, convey more information**
  - Critical for scaling system to large network sizes
  - Use information-centric models to compress data being transmitted

- **Linear estimation based data reduction**
  - Sender computes dynamic linear estimator based on sampled data
  - Transmits linear estimator
    - Only if estimation error exceeds a preset threshold
  - Receiver uses most recent linear model to estimate sensor data
  - Only models communicated
    - Not data
**Algorithm for sensor**

**Algorithm 1** Algorithm for sensor model $m_s$

1: **Initialize:**
   Transmit the values $(v_0, v_1)$ for time $(t_0, t_1)$
2: **for** Each time $t$ **do**
3:   Solve for $m$, $c$ in $e_t = m \times t + c$ using $(v_{t-1}, v_{t-2})$ and $(t - 1, t - 2)$
4:   Estimate sensor data value $e_t$ at current time $t$
5:   Calculate difference percentage $\delta_t \leftarrow |e_t - v_t|/v_t \times 100$
6:   **if** $\delta_t > th$ **then**
7:     Transmit $v_t$
8:   **Send control message**
9:   **end if**
10: **end for**

Model updated here
Algorithm for receiver / controller

Algorithm 1 Algorithm for controller model $m_c$

1: Initialize:
   Receive the values $(v_0, v_1)$ for time $(t_0, t_1)$
   Solve for $m, c$ in $e_t = m \cdot t + c$ using $(v_0, v_1)$ and $(t_0, t_1)$
2: for Each time $t$ do
3:    Estimate sensor data value $e_t$ at current time $t$
4:    if control message is heard then
5:       return $v_t$
6:    end if
7:    return $e_t$
8: end for

Model updated here
Evaluation setup

- Sensor Network Simulated with sensor model and controller model

- Data from Mica2Dot sensors with weather boards deployed at Intel Berkeley Research Lab
  - Mimics slow changing, low sampling rate phenomena like the smart brick monitoring
  - The data was sampled once every 31 secs and collected between February 28th and April 5th, 2004

- The model is validated for three different threshold percentage values i.e. $th = 0.1, 0.5, 1$ for all 54 sensors

- System also evaluated under simulated packet drops
Results (1)

- Percentage of packets saved as function of allowed error
- Even with an error of 0.1%, *about 40% packets saved*
Results (2)

- Actual observed error vs preset error threshold
- Actual observed errors are *actually much lower*
Results (3)

- Impact of data loss
  - Note that **model data more significant than raw data**

- Notice that errors are about 2-5% when crucial model data is lost
  - *But sending less data is actually likely to reduce data loss due to reduced interference*
**Task 5 Conclusions and Future Work:**

- Analog interface section
  - Developed an integrated circuit for sensor interfacing
  - Demonstrated the potential of using energy harvesting
  - Exploring using reconfigurable analog systems for providing long-term flexibility

- Wireless sensor network
  - Model based data reduction techniques are being explored
  - This can help reduce data rate without compromising with information required for analyzing system characteristics
  - Linear model based estimator yielded 40% savings with <1% error rate
  - Plan to continue exploring other model based ideas for data reduction e.g. Change based strategy
Task 6.0: Model-Based Estimation of Temperature Profile and Extent of Refractory Degradation. (Bhattacharyya)
Task 6 Objectives:

- To develop algorithms for model-based estimation of temperature profile in the refractory, slag penetration depth, spallation thickness, and resultant health by using the data from the wireless sensor network
Motivation & Approach:

Motivation:
- Typical correlation based approaches are inadequate
- Stiff temperature gradient along the sensor length
- Change in thermal and electrical properties over time due to slag penetration

Model-based approach:
Variable of interests:
- Temperature
- Extent of slag penetration
Properties (thermal, mechanical, electrical) Models

- For slag, sensor, and refractory materials
- For slag-infiltrated refractory

Process Models:
- Thermal model:
- Slag penetration model:
  - Capillary pressure
  - Simplified Poiseuille’s law
Effect of Slag Penetration on Wall Temperature

Steady state temperature profiles:

✓ Property models for slag penetrated refractory
✓ Slag penetration model

![Graph showing temperature changes with slag penetration](image)
Sensor Models:

Five different types of sensors:

- Interdigital capacitor (IDC)
- Strain gauge
- Resistive circuit
- Thermistor
- Thermocouple
Interdigital Capacitor (IDC) Sensor Model:

Sensitivity to slag penetration
- Composite property model
- Slag can only fill the pores

Sensitivity to the hot face temperature

**Estimation:**

**Methods:**
- Traditional Kalman Filter (TKF)
- Extended Kalman Filter (EKF)
- Unscented Kalman Filter (UKF)

**Difficulties:**
- Differential Algebraic Equations System
- Out-of-sequence measurements due to the wireless sensor network
- Multi-rate estimation
  - slag penetration: slow process
  - temperature: relatively fast process


**Wireless Sensor Network:**

Out-of-sequence measurements (OOSM):

- A random time delay due to communication delay
- Goal: Update the current states by using the measurements that arrive late
Estimations of the Extent of Slag Penetration Using IDCs:

- Constant hot face temperature
- Complex effect of slag penetration on:
  1. Temperature profile
  2. Dielectric constant
Estimations of Extent of Slag Penetration Using Thermistors:
Estimation of Multi-Rate KF:

- Both thermistors and IDCs are used
- Both temperature and slag penetration length are estimated
Estimations of Temperature with OOSM:

- The OOSM algorithm can successfully make use of the measurements that are received out-of-sequence.

\[ MSE = \frac{1}{N} \left( \sum_{i=1}^{N} (\hat{x}_i - x_i)^2 \right) \]

- Effect of a lossy measurement network evaluated-estimation accuracy decreases as the data loss rate increases.
Estimations of Slag Penetration with OOSM:

- 6 IDCs are embedded on the centerline (# of sensors is decided by the sensitivity study)
- EKF
- One-lag delay measurements
Task 6 Conclusions and Future Work:

- An algorithmic framework that can use the measurements from the smart refractory bricks through a wireless network to estimate the temperature profile and slag penetration depth in a gasifier has been developed.

- Further validation and testing of developed models and algorithms using experimental data are in progress.
Acknowledgements:

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