

Multi-Constituent Airborne Contaminants Capture with Low Cost Oxide Getters and Mitigation of Cathode Poisoning in Solid Oxide Fuel Cell

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Federal Agency and organization Element to which Report is Submitted

Department of Energy
National Energy Technology Laboratory

Work Performed under Federal Grant

DE-FE-0031647

Program Manager

Dr. Patcharin Burke
National Energy Technology Laboratory

Submission Date

12/01/2023

Report Submitted by

University of Connecticut, Storrs, CT 06269

Final Scientific/Technical Report

1. Programmatic Information

- **Federal Agency and organization Element to which Report is Submitted**

Department of Energy

National Energy Technology Laboratory

- **Federal Grant or Other Identifying Number Assigned by Agency**

DE-FE-0031647

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- **Submission Date:**12/01/2023

- **DUNS Number:** 614-20-9054

- **Recipient Organization (Name and Address):** University of Connecticut, Storrs, CT 06269

- **Project/Grant Period (Start Date, End Date):** 10/01/2018-08/16/2023

- **Reporting Period End Date:** 08/16/2023

- **Report Term or Frequency Number:** F

- **Signature of the Submitting Official**

Program Manager:

Dr. Patcharin Burke

National Energy Technology Laboratory

Acknowledgment

The technical report documents and describes the work performed under Award Number DE-FE-0031647 from the US Department of Energy, National Energy Technology Laboratory.

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2. Executive Summary

The technical effort and scientific findings, discussed in this report, documents operational barriers and associated long term performance stability challenges posed by the presence of trace airborne multi-constituent contaminants present in high-temperature electrochemical systems, including Solid Oxide Fuel Cells (SOFCs), Solid Oxide Electrolysis Cells (SOECs), Ion Transport Membranes, and Gas Separation systems. Above systems, offering promises for cleanliness and energy efficiency, face challenges with electrode poisoning stemming from the presence of trace contaminants including gaseous Cr/B/Si vapors in the presence of intrinsic contaminants SO₂/CO₂/H₂O gases. The study indicates that the long-term electrical performance degradation in SOFC systems can be traced to electrochemical, structural, and mechanical changes across cell, stack, and balance of plant components resulting from interactions with trace contaminants leading to increase in both ohmic and non-ohmic polarizations. The degradation primarily results from solid-state and gas-phase materials migration, electrode poisoning, and interactions at the cell and stack levels. Cathode degradation emerges as a significant factor impacting overall SOFC performance, especially related to the presence of intrinsic and extrinsic airborne impurities such as SO_x, CrO_x(OH)_y, SO_x, Si(OH)_x, and HBO_x. Although at trace levels, prolong systems operation at higher airflow (3-10X stoichiometric) allow the accumulation of contaminants within the cell components leading to electrical performance degradation through poisoning and electrode deactivation.

With focus on air electrode poisoning and electrochemical performance degradation in the presence of airborne trace contaminants, the research efforts were directed towards the identification, fabrication and test validation of cost-effective alkaline earth metal based getters to effectively capture the trace contaminants and prevent electrode poisoning. Thermochemical analysis and thermodynamic models have been developed to quantify gaseous contaminants (types and levels) under SOFC systems operating conditions. Contaminants interactions with select getter constituents leading to thermodynamically stable reaction products have been analyzed based on available literature. Select getter formulations have been synthesized using oxide and chemical precursors and getters have been fabricated using ceramic porous substrates. Fabricated getters have been tested in the laboratory to measure their efficacy for contaminant capture. Mitigation of the cathode poisoning in the presence of getter was demonstrated through electrochemical tests.

The research program has successfully identified the role of both intrinsic and extrinsic airborne trace contaminants on the electrochemical performance degradation and electrode poisoning under SOFC system operating conditions. Thermochemical models have been developed to account for the evaporation of the oxides in the presence of air constituents as well as interactions of the gaseous contaminants with the getter. The research effort has led to the successful development and validation of low cost getters for the efficient capture of trace contaminants and mitigation of electrode poisoning and performance degradation.

Based on the findings of the research, it is recommended that the developed getters be used in SOFC systems for long term mitigation of cathode electrode poisoning.

3. Project objectives

The objectives of this research program include development of a broader understanding of the origin, reaction processes and the nature of airborne contaminants (intrinsic and extrinsic); contaminant interactions with the air electrode materials and identification of mechanisms for the electrochemical poisoning and performance degradation at cell, stack and system levels; and development and validation of approaches for the mitigation of performance degradation during the SOFC systems operation in 600°C-800°C temperature range. Our research findings based on the experimental results indicate that the reactive oxides (Cr_2O_3 and SiO_2) formed at the metal-oxide interface during exposure to ambient air leads to continuous evaporation of the above oxides. The exposure conditions lead to the electrode poisoning and deactivation resulting in electrical performance degradation. The research program aims at identifying, fabricating, test and validating cost effective getter formulations and associated designs for the capture of airborne gaseous contaminant species. Efficacy of the getters will be validated in the lab and selected getter configurations will be subsequently integrated and tested in a prototype SOFC system test bed to validate the technology readiness level (TRL) 5. The main objectives of the project are directed towards the:

- Development of a comprehensive understanding of the origin, formation processes and the nature of gas phase airborne contaminants present in the air stream entering elevated temperature electrochemical systems
- Identification of trace airborne gas phase contaminants (intrinsic and extrinsic) and develop mechanistic understanding of interactions (chemical, electrochemical and structural) with conventional air electrode materials.
- Identification of cost effective getter materials and processing techniques to capture trace contaminants. Synthesize and validate getter performance and efficacy.
- Design and fabrication of getters for stack and BOP applications. Validate the above at stack/system level. Transfer technology to industrial partners.

The milestone log is shown in Table-1

Table 1. Milestone Log

Task/ Subtask	Milestone Title & Description	Planned Completion Date	Actual Completion Date	Verification
I	Establishment of program priorities with program manager	09/20	09/20	Technical reports/ Meetings
II	Identification, selection and optimization of getter materials.	12/19	08/23	Technical reports/ Meetings

III	Synthesis of HSA nano-porous coating and optimized getter architecture	11/19	08/23	Technical reports/ Meetings
IV	Getter validation for combined capture of Cr, S, Si and B impurities	11/19	08/23	Technical reports/ Meetings
V	Getter design optimization through computational flow analysis.	02/20	08/23	Technical reports/ Meetings
VI	Scale-up and long-term performance testing under system conditions	06/20	08/23	Technical reports/ Meetings
VII	Post-test characterization and mechanistic understanding of capturing impurities.	09/20	08/23	Technical reports/ Meetings
VIII	Documentation, reporting, and publication	09/20	08/23	Technical reports/ Meetings
IX	Intellectual property and technology transfer	09/20	08/23	Technical reports/ Meetings

4. Summary of accomplishments

Trace (ppm and lower) levels of airborne contaminants, present in ambient air under the SOFC systems operating conditions, influence the long term electrical performance stability by adversely affecting the electrode processes and increased polarization losses. Individual and combined effects of Cr and S gaseous species on the poisoning of LSM and LSCF electrodes have been studied. Chromium poisoning is mainly attributed to the electrochemical reduction of Cr vapors at catalytic active sites and surface reaction products formation while sulfur poisoning is primarily considered to be due to the strong chemical affinity of alkaline-earth dopants in the electrode with SO₂ resulting in the formation of thermodynamically stable reaction products. Experimental results indicate that the interactions of Cr/S gaseous species remain influenced by air electrode material type, operating temperature, Cr/S concentrations, and electrode polarization conditions.

The use of getters offers a scalable and cost-effective pathway for mitigating electrode contamination without the need to replace established cell and stack component materials. Considering the thermodynamics of related solid-gas reactions, the combination of alkaline-earth and transition-metal oxides Sr–Ni–O and Sr–Mn–O systems have been selected as absorbent materials for the multi constituent contaminants such as Cr, SO₂, Si, B, CO₂ etc. Their efficacy for capturing these gaseous species was validated by benchtop transpiration as well as electrochemical tests. Sr–Ni–O system was found to be structurally unstable accompanied by phase separation in

humid environments at high temperatures (≥ 950 °C). Sr–Mn–O system, on the other hand, showed excellent thermal stability at higher temperature and in humid environment. Post-test characterization of the getter showed the elongation of absorbent particles and the formation of SrSO_4 and SrCrO_4 indicating the high mutual affinity of alkaline-earth-metals and Cr/S species.

Major technical accomplishments and research findings include:

- Identification of intrinsic and extrinsic trace gaseous contaminants present in ambient air stream, entering the elevated temperature electrochemical systems
- Identification of reaction processes between gas phase acidic airborne impurities with basic air electrode constituents to form stable reaction products at the free surface and TPB.
- Development of thermodynamic models based on the Gibbs free energy minimization of reaction processes and validation of the co-capture of trace sulfur and chromium impurities in a wide temperature range.
- Development of getter formulations along with fabrication and experimental test validation for the capture of trace S (intrinsic) and Cr, Si, B (extrinsic) gaseous contaminants.
- Fabrication of supported getter structures using monolithic cordierites and foam substrates.
- Successful demonstration of getter for the capture of multi constituent contaminants individually and combined in gas phase.
- Development and validation of mechanisms responsible for the generation and capture of trace contaminants.
- Use of cost effective getters in the cell stack and BOP for the capture of gaseous contaminants present in fuel cells, electrolyzers and chemical reactor systems.

5. Project Details

The research effort focused on the thermodynamic and kinetic interaction studies between multi-constituent airborne gaseous impurities (predominantly acidic) and alkaline earth-transition metal oxides (basic) along with the analysis of reaction processes and identification of steps that can lead to continued surface reaction under SOFC systems operating conditions. High surface area (HSA) nano-rods and nano particles were synthesized and incorporated in the porous getter coating formed over ceramic substrates. Getter fabrication processes were subsequently scaled up for integration in prototype stack for electrical performance testing. The technical activities performed during the course of the research program are described in detail in the following sections.

5.1. Task 1.0 - Project Management and Planning

The research program was managed and directed in accordance with the proposed and agreed upon Project Management Plan (PMP) to meet all technical and budgetary objectives and requirements. UConn coordinated activities in order to effectively accomplish the project objectives and ensured that project plans, results, and decisions are appropriately documented and project reporting and briefing requirements were satisfied. Project risks were managed in accordance with the risk management methodology delineated in the Project Management Plan in order to identify, assess, monitor and mitigate technical uncertainties as well as schedule, budgetary and environmental risks associated with all aspects of the project. The results and status of the risk management process were presented during project reviews and in Progress Reports with emphasis placed on the medium- and high-risk items.

5.2. Task 2: Co-getter materials identification, selection and synthesis

The objective of the proposed task includes the identification of alkaline earth and transition metal oxides, oxide solid solutions, perovskites, and spinel compounds capable of forming thermodynamically stable reaction products (chromites, chromates, silicates, sulfates and borates) after interactions with gas phase impurities present in humidified air in the temperature range of 600-900°C and evaluation of their application as getter materials to capture the multi constituent contaminants that limit the performance of air electrode in long term operation.

The subtasks include:

5.2.1. Sub-task 2.1 Rational selection of candidate co-getter materials

The sub task focused on the identification and assessment of candidate metal oxides, solid solutions, perovskites and spinel structures that have shown ability co-capture air impurity species (Cr, S, Si, and B) from humidified air at high temperature on the cathode side. Existing literature and thermochemical databases (HSC Chemistry) were used for the identification of candidate formulations. Selected getter materials were synthesized and experimentally investigated and validated

5.2.2. Sub-task 2.2 DFT modeling to screen potential getter materials

Under this subtask, thermochemical databases were utilized to screen the getter materials and to determine the minimization of surface segregation and compound formation on the LSM and LSCF type cathode materials. The methodology considers structural and thermodynamic factors to evaluate surface energetics for oxide segregation and formation energy for the introduction of dopants in lanthanum manganite lattice. The developed methodology will also be used to account for simultaneous interactions in the presence of CO₂, H₂O, Cr, S, Si and B species in air over wide temperature range (500-1000°C). The simulation work [1] which depicts that honeycomb architecture permit steady, incompressible and laminar flow of the airstream for SOFC systems have been published in peer reviewed journal.

5.2.3. Sub-task 2.3 Co-Getter materials synthesis

This sub task focused on the development of synthesis techniques to obtain high surface area fibers and powder getter materials (mono-modal or multi-modal size distribution). Mainly two different synthesis routes including the conventional solid state and sol get method was used for the synthesis of high surface area powders. The citrate-nitrate route for the powder synthesis followed by dip coating to form a thin porous surface layer on cordierite channels and alumina foam and the nanofiber deposition process for the manganese oxide (MnO) fiber deposition on the select substrates.

5.2.4. Methods & approaches:

a) Materials Selection

Alkaline earth metals have been considered as the materials of choice for the capture of gaseous contaminants such as Cr, S, CO₂, Si, B etc. present in the oxidant and fuel gas streams in the trace amount because of their affinity to form thermodynamically favored stable compounds such as chromates, sulfates, carbonates, silicates and borates. For instance, SrO and BaO, segregated onto the surface from SOFC cathodes under the operating conditions, readily react with airborne Cr, S, Si, B and C containing volatile species [2–4]. It is recognized that Sr has high reactivity with SO₂ (high to low: Sr > Ca > Ba > Mg) [5], and CaO is also capable of scavenging Cr vapors as well as

SO₂ evolved during coal combustion[6–8]. Beryllium and Radium were excluded from the list because of their high toxicity and radioactivity. In the present work we have mainly focused on the evaluation of Strontium manganese oxide Sr-Mn-O based getter system due to its robustness in harsh environment and higher affinity to form the stable compounds with the air and fuel borne intrinsic and extrinsic contaminants and co capturing capabilities. Figure 1 shows the drop in the partial pressure of contaminants during reaction with different alkaline earth metal oxides over a temperature range (500°C-1000°C).

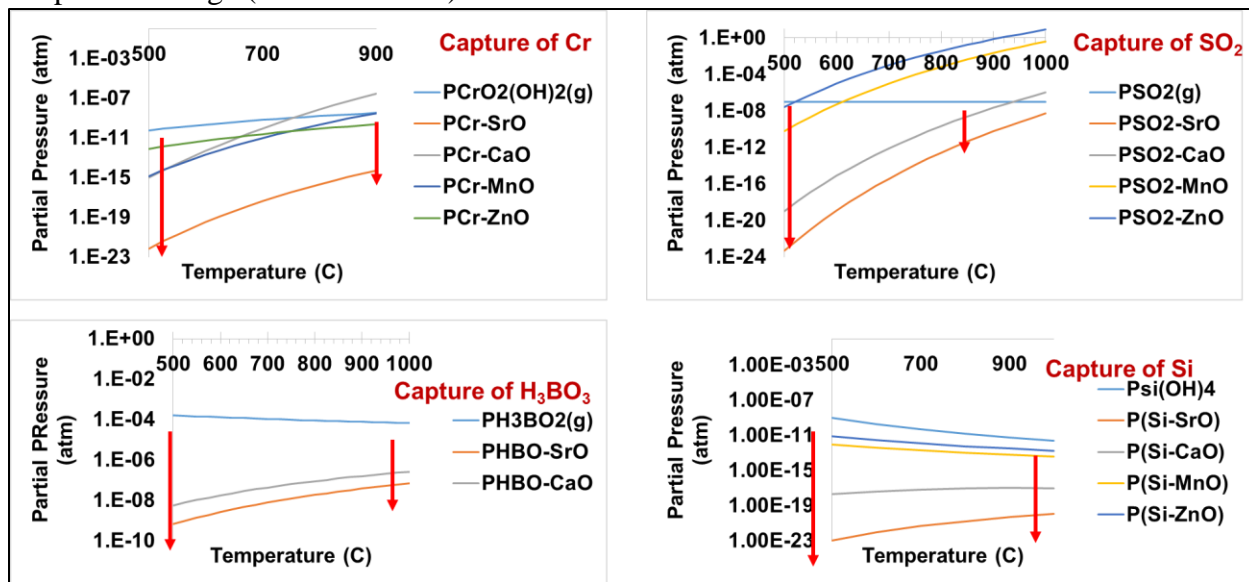


Figure1. Partial Pressure of Cr, S, B and Si gaseous species in presence of alkaline earth oxide materials

b) Materials Synthesis

Getter materials were synthesized based on alkaline-earth metals. The synthesis process has been described in detail in our published article [9] and sol-gel method[10]. In a typical co-precipitation route, chemical precursors such as nitrates, carbonate or oxides of Sr and Mn are dissolved in deionized water in a desired ratio and stirred thoroughly at 80°C and then Ammonium hydroxide (NH₄OH) is slowly added to the aqueous solution until pH becomes 8.5. The solution was subsequently rinsed with DI water to remove the remaining ammonium nitrate (NH₄NO₃) and then dried for 24 h in an oven. A sol-gel powder synthesis method was utilized to produce the high surface area getter powder. In a typical synthesis method, a nitrate of the alkaline-earth metal was mixed into DI water set to 80°C on a hot plate with other binding metal nitrate. The solution pH is made basic with the addition of ammonium hydroxide and left to gel overnight while stirring on the hot plate. The resulting gel is then calcined and sintered at 1300°C in ambient air to obtain a single-phase compound. The sintered powder was finally ground using a planetary ball mill with yttrium stabilized zirconia milling media. The structure and chemical composition was analyzed using an x-ray diffractometer (XRD) (D8 Advance, Bruker, with Cu α radiation). Peaks were indexed using National Institute of Standards and Technology (NIST) and International Center for Diffraction Data (ICDD) databases with ICDD PDF-2 XRD data mining software.

5.3. Task 3: Synthesis of high surface area (HSA) nano rods, powder and tailored porosity coatings

In this task, particular emphasis was given to the synthesis techniques for obtaining HSA nano and meso-scale powder, nanofibers and nano-rods for the identification and preparation of getter coating. Getter materials were evaluated and selected processes of interest were further optimized and tested in the laboratory. The as-synthesized high surface area nano and meso scale powder and nano-fibers were characterized for their chemistry, and morphology. The as prepared HSA materials were subsequently used for the development of getter coatings applied over ceramic backbone such as honeycomb monolith and alumina foams.

5.3.1. Sub-task 3.1 High surface area (HSA) getter powder synthesis

High surface area powder were synthesized utilizing conventional precursor based techniques, supercritical and gas phase precipitation. Getter coating techniques ranging from conventional dip coating to methods involving aerogel, hydrothermal deposition and additive manufacturing were assessed based on specific coating materials chemistry. The role of processing parameters such as chemical precursor's type and concentration, pre and post treatment conditions, particle size, coating thickness, and thermal treatment in controlling the microstructure, surface coverage uniformity and adherence were addressed.

5.3.2. Sub-task 3.2 Characterization of synthesized getter materials

Surface and bulk characterization techniques were used to analyze as-synthesized getter materials. Structural studies were performed using X-ray powder diffraction for crystalline materials. Functional group analyses such as Sr-O, Ni-O, and Sr-O-Ni (and other dopant) vibrational modes were analyzed using Fourier transform infrared spectroscopy and Raman spectroscopy. Electrochemical methods like cyclic voltammetry and impedance spectroscopy were performed to study electrode stability and conductivity.

5.3.3. Sub-task 3.3 Identification of materials properties and development of coating technique

Powder classification by size were used to develop suspensions with targeted pore size distributions, with and without polymer bead pore formers. Reactivity between various getter phases were assessed in a rapid and efficient manner by utilizing in-situ X-ray diffraction (under controlled humidity and temperature) to screen reactivity. Sintering cycles for the composite getters were determined wherein pellet densification is tracked during the sintering cycle via an automated optical imaging system, for both monoliths and on-cathode layers, where the cathode can be co-sintered with the getter.

5.3.4. Experimental methods & approaches:

c) Fabrication of Getter

High purity Al_2O_3 foam (>97% Al_2O_3) with high surface area (Figure 2a) were selected as getter support to achieve a relative clean baseline (mitigate the contribution of Si, Cr, and B contribution from the support) for the getter validation tests. The Sr and Mn oxide compound (SMO) was used as getter materials for coating on cordierite. This selection of materials is based on the thermodynamic calculations conducted and reported in earlier reporting period which indicated significant reduction in the Si vapor pressure under SOFC operating conditions. The SMO getter was fabricated by dip-coating of the cordierite in an aqueous precursor slurry comprised of SrMnO_3 , MnCO_3 , $\text{Sr}(\text{NO}_3)_2$ and poly-(vinylpyrrolidone), followed by heat treatment at 1000 °C (Figure 2b).

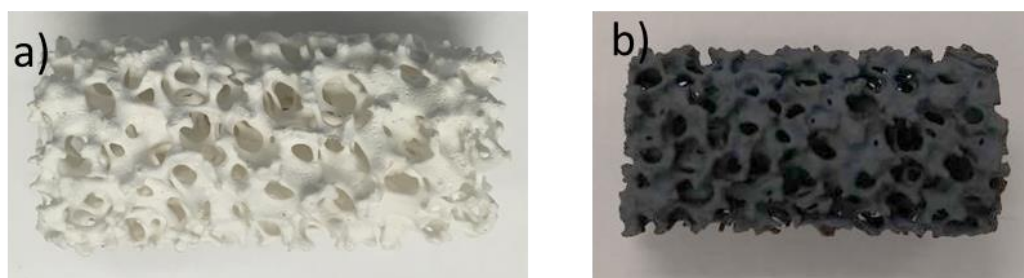


Figure 2. Fabricated getter after heat treatment at 1000 °C.

In collaboration with Alfred University, a getter fabrication technique including coating process was developed and experimentally evaluated using the synthesized SMO coating powder for getter fabrication. The coating process includes dispersing the powder in water in a shaker mill (~35 volume percent solids), followed by addition of Darvan C to deflocculant the particles. Subsequently, the getter substrates were fabricated through dip coating by the slurry and then subjected to a compressed air stream to remove excess slurry that was captures via capillary forces. The alumina filter substrates were coated twice; the average weight gain on the first coat was 0.75 grams with a standard deviation of 0.1 grams. After the first coat, fabricated getter containing coating materials were heated at 1000°C for five hours, followed by re-coating to obtain ~1 gram of the select getter material on each getter support element. These samples were heated again at 1000 C for 5 hours, followed by heating to 1050°C and 1150°C for 5 hours each. Coatings were found quite stable (no flaking/ chipping) against the impingement of high pressure compressed air. Getters using Cordierite substrates were also fabricated (400/in square cordierite). The cordierite getters were sintered at 1150°C for 5 hours and contained 0.71 grams of getter material. That sample was made with a single coating.

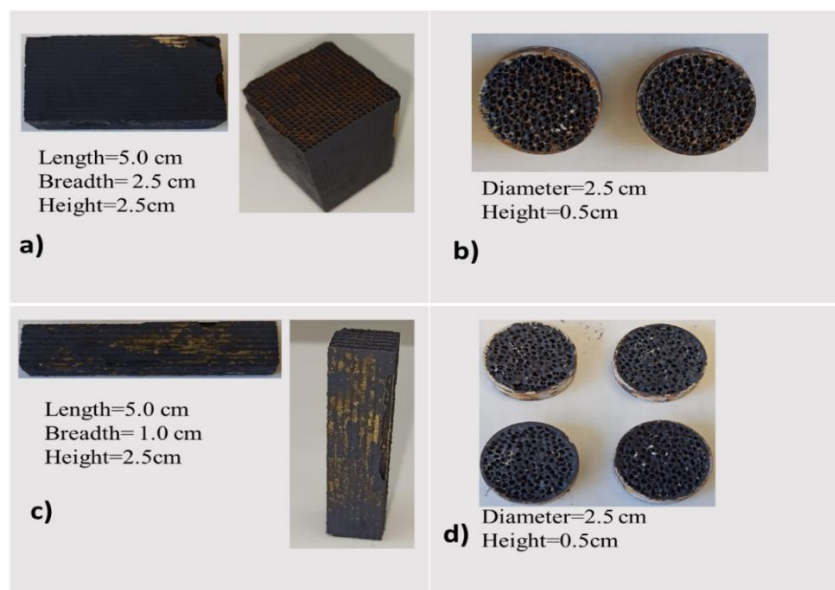


Figure 3: Cordierite and Alumina substrates coated with getter coatings developed in collaboration with Alfred University.

5.4. Task 4: Co-getter validation for combined capture of Cr, S, Si and B

The ability of the developed advanced getters to capture multiples of airborne gaseous impurities were experimentally validated under simulated cell and stack operating conditions. Two components of this task included *in-situ* evaluation of the getter using electrochemical technique followed by an in-depth characterization of the cathode and getter to understand the capture profile of each Cr, S, Si and B elemental distribution.

5.4.1. Sub-task 4.1 Transpiration test of select getters

The effectiveness of the getters and coating layer formulations were experimentally evaluated under simulated cell and stack operating conditions (temperature and flow rates). A parametric study was performed to map the presence of Cr, S, Si and B along the X-Y (cell plane) and Z (component thickness) to examine the efficiency of the getters.

5.4.2. Sub-task 4.2 Electrochemical validation of getters

Ability to capture gas phase impurities and electrochemical performance stability of the cathode against airborne impurities were examined using the existing symmetrical and half-cell test setup for up to 500 hrs. in 600-900°C temperature range and ambient humid air. Impurities in the air stream were introduced through the use of respective oxide or compound sources in the inlet air stream. Standard cell tests were also conducted to examine the cell performance stability. The cell test performance was compared with the base line test conditions using air containing air impurities. Performance stability and electrode polarization changes with time were analyzed.

5.4.3. Sub-task 4.3 Posttest getter parametric study

This subtask focused on conducting parametric study on the presence and deposition profile of impurities in the getter, contact layer and the cathode. Type of impurities and their concentration were mapped along the X-Y (cell plane) and Z (component thickness) direction of the cathode electrode and the getter cross-section. The parameters of interest for investigation included the role of cathode atmosphere chemistry (levels and type of airborne impurities including humidity levels), cell operating temperature (600-900°C), exposure time (100-2000 hrs.), and electrical load. External sources of impurities in presence of water vapor were used to simulate the ‘real world’ cathode atmosphere and to obtain detectable changes in polarization resistances through accelerated testing. Post-test analytical study were performed to determine the improvement in the thermo-chemical stability of the cathodes.

5.4.4. Experimental methods & approaches:

a) Transpiration Tests

Transpiration technique was utilized in our laboratory to generate, transport and react gas phase trace contaminants under SOFC exposure conditions (Figure 4). The test technique consisted of the use of contaminant source (select porous oxide pellets) to generate gaseous contaminants during exposure to elevated temperature gas atmospheres, and transport of generated gaseous contaminants to reaction sites including electrode and getter samples. Porous pellets containing oxides of Cr, Si, and B were fabricated and used to generate specific contaminants. Reaction processes involving interactions of contaminants and getters along with the effectiveness of developed getters were evaluated. Getter capacity for the capture of contaminants was examined using coated foam and monolith supports. In a typical experiment, 200 sccm of dry air was flown into a water bubbler set at 25°C to raise the humidity of the air to 3% H₂O before reacting with the contaminant source and produce the Cr/Si/B gaseous volatile species. The gaseous contaminants subsequently were allowed to react with the LSM or LSCF electrode. The tests were conducted for 100h.

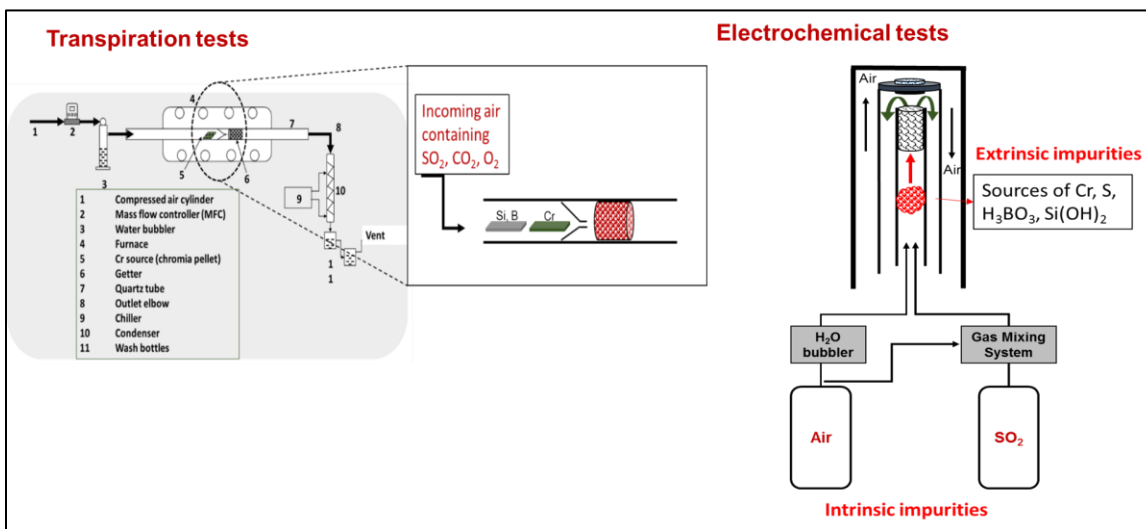


Figure 4. Schematics of bench top transpiration test with airborne intrinsic and extrinsic contaminant such as Cr, Si, B and S and electrochemical test setup for the validation of electrode poisoning under the contaminant such as Cr, Si, B and S volatile species and getter efficacy.

b) Electrochemical Validation Test

Electrode performance decay, in terms of increase in the polarization losses due to the deactivation and poisoning of electrochemical activity, was studied using well established electrochemical techniques in half and full cell configurations. The electrochemical characteristics of LSM and LSCF air electrodes were assessed in the presence of gaseous contaminants through the use of the electrochemical impedance spectroscopy technique, which involved subjecting the electrodes to 10 mV electrical perturbation under OCV conditions. The experiments were conducted over a broad frequency range of 200 KHz to 10 mHz in a specially designed test reactor as shown in Figure 4. The reactor consists a quartz tube with a 2-inch outside diameter for the introduction of select gas mixtures, an additional quartz tube with a 1-inch diameter to introduce humid air or air containing trace level of contaminant gas such as SO_2 or CO_2 . For the introduction of extrinsic contaminants containing species of Cr, Si and B, high surface area chromia, silica and borosilicate powders were used as source (dispersed on the quartz wool) placed before the LSM/LSCF electrode in the gas passage connected to the electrode. The experimental setup provided an ideal configuration and environment for assessing the performance of the electrodes under conditions that mimic SOFC operating conditions.

5.4.4. Results & Discussion:

a) Transpiration Tests

Experimental results were analyzed in terms of changes in the electrochemical performance of the electrodes, surface and interface morphology, structure and chemistry of the bulk electrode materials exposed to the gaseous atmospheres (with and without the presence of contaminants). Figure 5 shows the surface morphology of the as fabricated LSCF powder that was evaluated for Si contamination after being exposed to high humidity (90% H_2O -Air) at 800°C for 100 hours. The surface morphology exhibited changes as shown by SEM micrographs, and the EDS analysis confirmed the presence of the Si on the LSCF surface. Pre-test samples exhibited a multi-modal

particle distribution that is clean and devoid of deposits or exsolution. It is, however, noticed that the surface of the posttest electrode has changed as well there is evidence of chemical changes and formation of new compounds. It is hypothesized that the interaction of LSCF powder with volatile silica species result in the formation of glassy phase deposits on the surface as well as compositional changes under high humidity conditions.

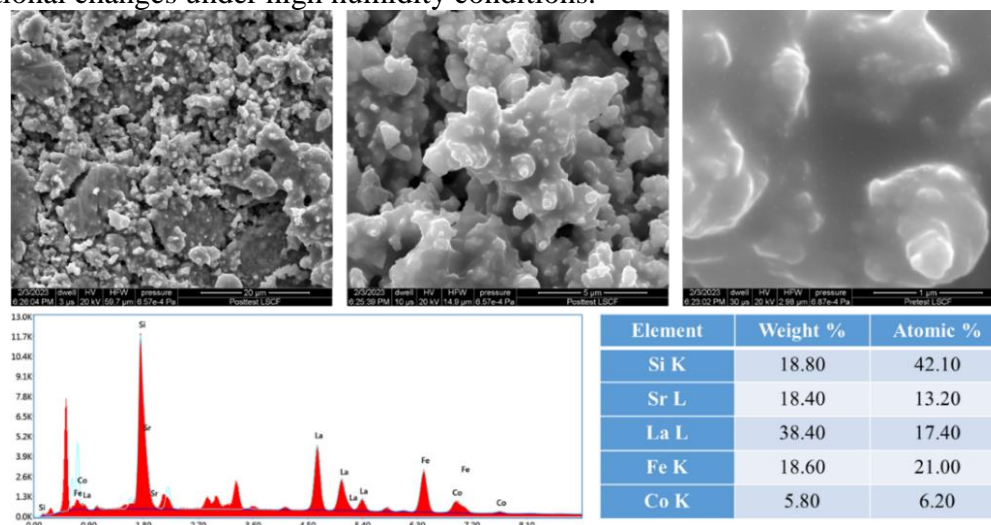


Figure5: Surface morphology and surface chemistry of posttest LSCF pellets tested in Si gaseous environment in high humidity atmosphere.

b) Transpiration Tests with SMO Getter

The role of getter in capturing the gas phase contaminants was examined using transpiration technique. Figure 6. Shows the surface morphology of the SMO getter after the transpiration test in humidified air for 300h at 700°C using borosilicate glass beads as the source of B and Si species. Si deposition was observed at the getter surface. The deposition is associated with the formation of gaseous species and its subsequent deposition/ interaction with the getter.

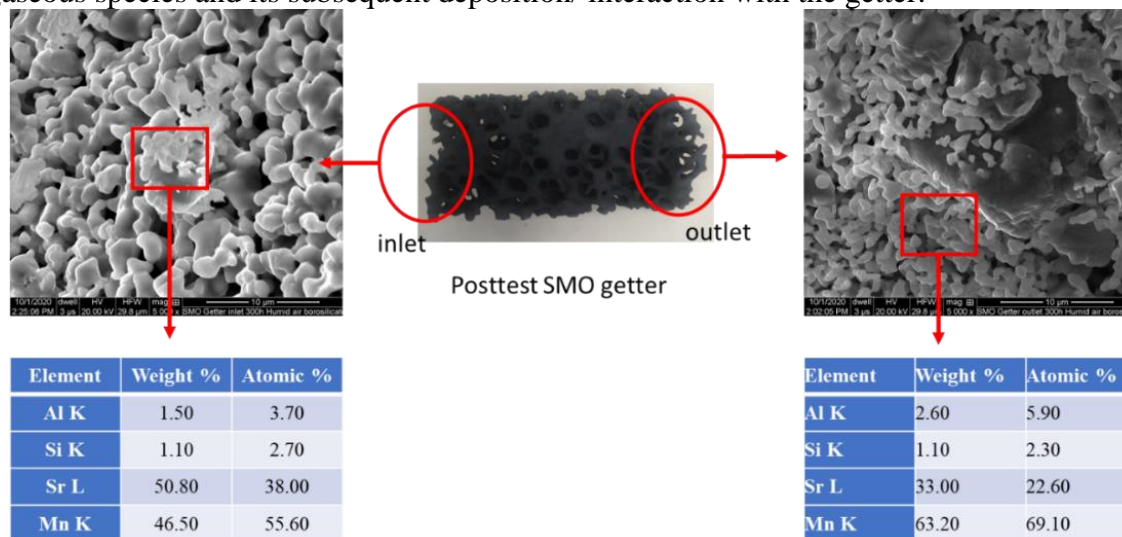


Figure 6. Surface morphology and EDS analysis of posttest SMO getter at inlet and outlet after test in humidified air for 300h at 700C

ICP-OES analysis was carried out on the Na_2CO_3 containing aqueous solution to evaluate the SMO getter performance. The result of ICP-OES (inductively coupled plasma optical emission spectroscopy) is tabulated in Table 1.

Table 1. B and Si concentrations absorbed in Na_2CO_3 , determined by inductively coupled plasma (ICP)

Sample Details	Boron concentration	Silicon concentration
A (Na_2CO_3 aqueous solution): without SMO getter	10680 $\mu\text{g/L}$	21570 $\mu\text{g/L}$
B (Na_2CO_3 aqueous solution) : With SMO getter	6500 $\mu\text{g/L}$	8027 $\mu\text{g/L}$
(Concentration in A / Concentration in B) X 100	60.9 %	37.2 %

From ICP data it is evident that both boron and silicon concentrations are significantly decreased in the exit gas due to the presence of SMO getter, indicating the ability of SMO getter to capture both boron and silicon contaminants.

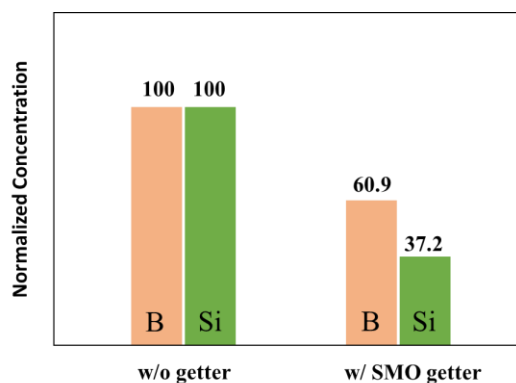


Fig 7. Comparison of the amount of boron and silicon concentration in the presence and in the absence of SMO getter

c) Electrochemical tests

Electrode and getter performance were examined after the completion of electrochemical tests. Electrical characteristics of LSM and LSCF based electrodes were analyzed. Figure 8a shows the electrode performance degradation of electrodes under electrochemical cell configurations LSM|YSZ|Pt and LSCF|GDC|Pt and operated in the presence of humid air containing SO_2 and humid Air-Cr vapors. It is observed that the current density of the LSM-based cell rapidly decreased for the first ~10 h, while that of the LSCF-based cell reduced gradually. Nyquist plots (Figure 8b&c) show that the polarization resistance increases in both cells whereas ohmic resistance changes significantly in only LSCF-based cell.

Cross-sectional analysis of LSM and LSCF containing cells were performed before and after the exposure to Cr and SO_2 containing air under experimental conditions (Figure 9). The granular

morphology of LSM with a smooth surface did not change after the exposure to Cr and S contaminants and elemental distributions of Cr and S are very similar to those in the LSM poisoned by Cr only and S only contaminants[1–3,11,12]. The morphology of LSCF before and after the exposure to gaseous Cr and S contaminants under polarization conditions, however, changes significantly from the initial smooth surface to an angulated surface over the exposed areas indicating morphology change due to contaminant coverage and interaction incorporation.

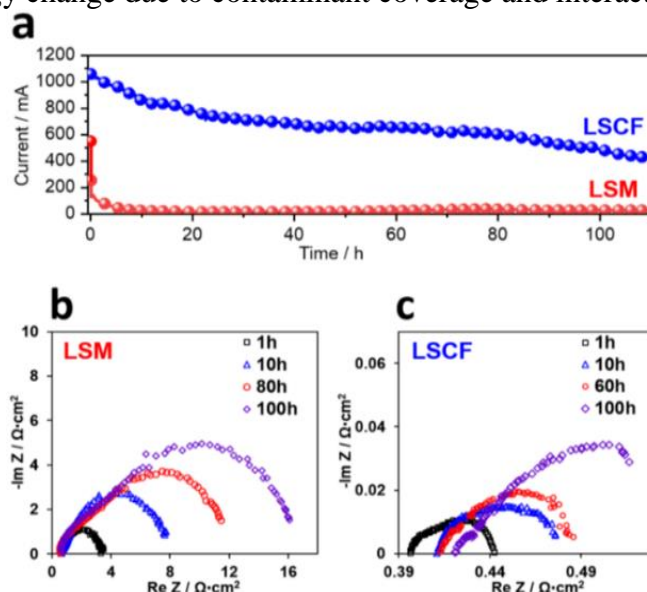


Figure 8: (a) Current density and (b and c) Impedance spectra of LSM|YSZ|Pt and LSCF|GDC|Pt of cells operated for 110 h at 750 °C under the exposure of air containing 500 ppb SO₂ and Cr vapor (~1 ppb CrO₂(OH)₂ (g)).

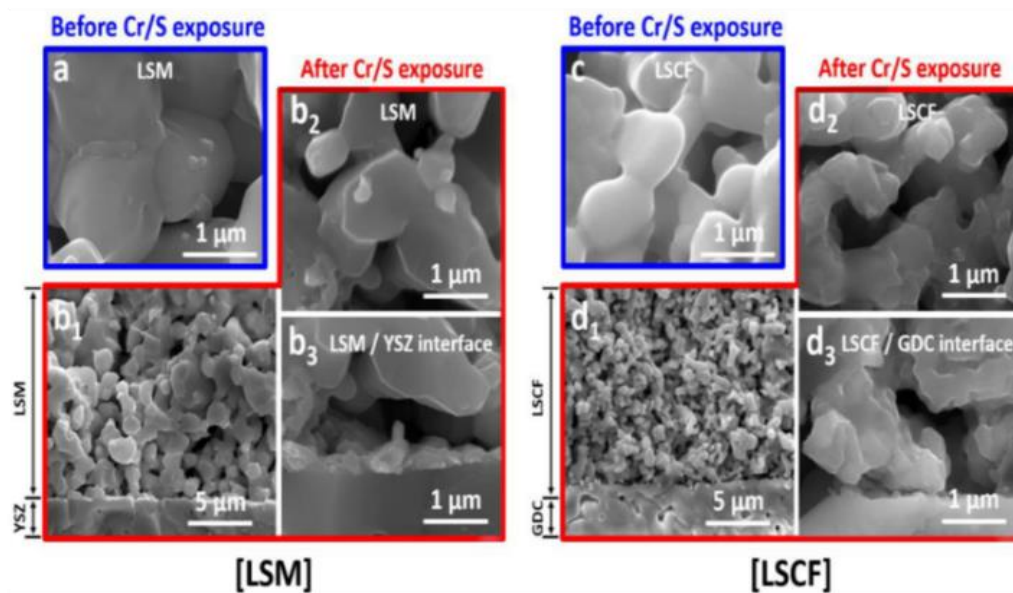
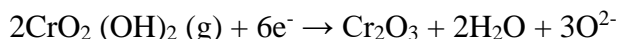


Figure 9: fracture surface morphology of as fabricated LSM and LSCF electrodes (a and c) and exposed to SO₂ and Cr vapors for 110 h at 750 °C during the electrochemical test (b and d).

In LSCF electrode, it is postulated that the Cr_2O_3 is deposited at the LSCF/GDC interface and SO_2 on the LSCF particle surface (combined Cr-S poisoning). From the thermodynamics of the $\text{SrO}-\text{CrO}_2(\text{OH})_2-\text{SO}_2$ system (Figure 4), it is proposed that the reactions between surface-segregated SrO and Cr-S contaminants form SrSO_4 under the experimental conditions. At high temperatures ($> 500^\circ\text{C}$), Sr-segregation occurs at the surface and the SrO particles react with Cr-S contaminants [14]. Continuous absorption and reaction lead to the formation of SrSO_4 on the LSCF particle surface. In the case of Cr vapors flowing over LSCF, since the surface Sr is covered by SO_2 , the SrO's ability to capture Cr species is diminished. Thus, the Cr vapors freely reach the LSCF/GDC interface resulting in the deposition of Cr oxides by electrochemical reduction reaction.

The absorption of airborne Cr and S contaminants on the SMO getter was examined using electrochemical cell (LSM|YSZ|Pt) operated in the presence of humidified air containing 4 ppm SO_2 and ~ 1 ppb $\text{CrO}_2(\text{OH})_2$ (interaction of Cr_2O_3 with H_2O) and the presence of SMO getter. The electrochemical performance was compared with those recorded under the flow of either the SO_2 or $\text{CrO}_2(\text{OH})_2$ gases in the absence of the getter. Figure 10 summarizes the electrical behavior of cells. The current density remained stable (Curve a, 0-110 hrs.) under the flow of humidified air ($\sim 3\%$ H_2O). Immediately after the injection of 4 ppm SO_2 gas at 110 hrs., the current density decreased (Curve a, 110–150 h). Similarly, under the presence of Cr vapor, the current density decreased (Curve b) due to electrode poisoning and preferential its deposition at the triple phase boundary (TPB) via the following electrochemical reaction.



The electrode performance degradation due to the presence of SO_2 contaminants is considered to be associated with the chemisorption of SO_2 on the LSM surface. In the presence of SMO getter, the current density remained stable for 230 h (Curve c) despite the injection of the same levels of Cr vapor and SO_2 gas (i.e., ~ 1 ppb $\text{CrO}_2(\text{OH})_2$ for the initial 100 h, and then both ~ 1 ppb $\text{CrO}_2(\text{OH})_2$ and 4 ppm SO_2 for another 120 h). Cross-section of the LSM tested in the presence of the SMO getter under the flow of Cr and S gases shows a clean surface morphology whereas the sulfur-contaminated LSM shows a modified surface with nano protrusions of SO_2 precipitates.

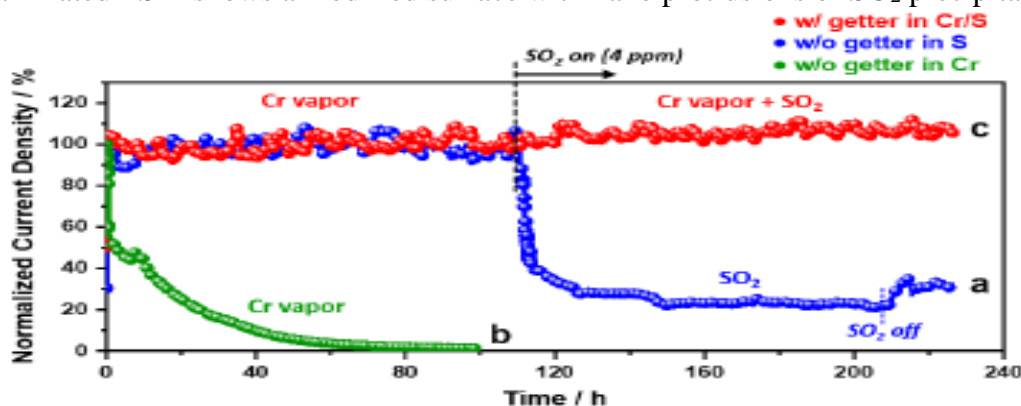


Figure 10. Current density of the LSM|YSZ|Pt cells over time under the flow of (a) 4 ppm SO_2 gas (injected after 110 h); (b) Cr vapor (~ 1 ppb $\text{CrO}_2(\text{OH})_2$) in the absence of SMO getter; and (c) under the presence of both Cr vapor and 4 ppm SO_2 gas (injected after 110 h) in the presence of SMO getter.

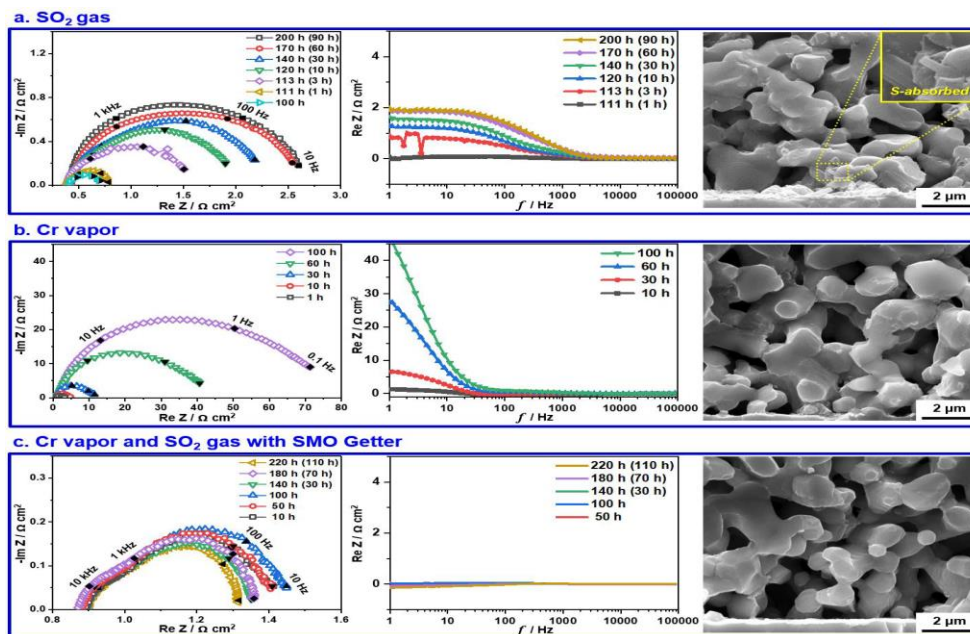


Figure 11. Impedance spectra (Nyquist and Bode plots) of the LSM|YSZ|Pt cell and cross-sectional SEM images at the LSM/YSZ interface exposed to (a) SO₂ gas, (b) Cr vapor, and (c) both Cr vapor and SO₂ gas in the presence of the SMO getter, respectively.

Electrochemical characteristics of the LSCF electrode in the presence and absence of gas phase contaminants are presented in Figure 11. Nyquist and the polarization behavior of the electrode in the presence of high humidity (90% H₂O-Air) at a temperature of 700°C for a duration of 100 hours are presented. The Nyquist plot shows a continuous increase in polarization when LSCF half cells were subjected to the high humidity air containing Si gaseous species indicating that the introduction of Si gas as a contaminant leads to irreversible degradation the cell. It is noted that the ohmic polarization experienced little/ no change, while the non-ohmic polarization exhibited a steep incline, implying impact of Si gaseous species on electrochemical reaction steps.

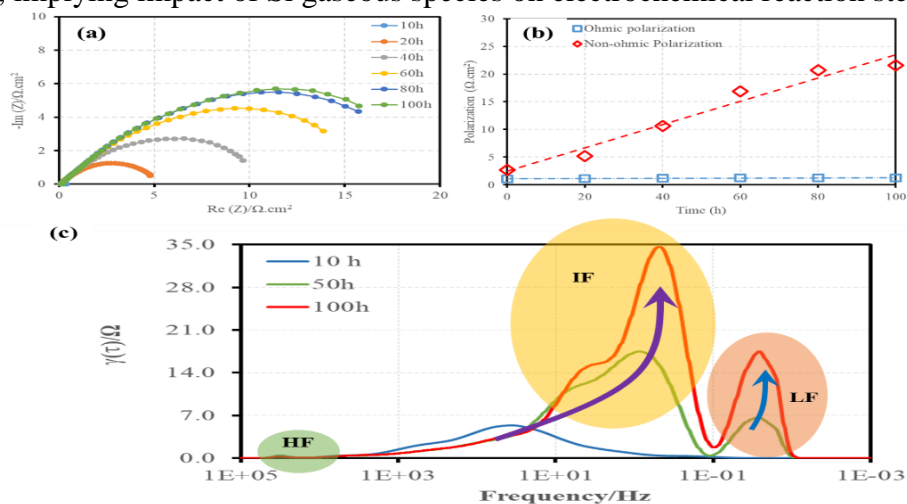


Figure 12: Nyquist plot and polarization changes for LSCF electrode in high humid air (90% H₂O-Air) containing Si gaseous species (700 °C, 100 hours).

DRT (Distribution of Relaxation Time) analysis was used to assess the electrode behavior and analysis of the rate-determining steps and mechanisms. The DRT analysis shows four noticeable peaks, each corresponding to distinct rate-limiting steps or processes taking place at the electrode. The peak at lower frequencies, are associated with the adsorption/desorption of gas indicating that the interaction between Si and the electrode surface obstructs the gas's dissociative adsorption/desorption. Within the intermediate frequency range of 1 Hz to 10^3 Hz, two distinct processes occur. The first process is attributed to surface exchange reactions occurring at the solid-gas interface, while the second process is associated with ionic transport within the bulk electrode material. Exposure of the electrode to a high humidity environment containing Si gas leads to a shift towards lower frequencies indicative of a reduction in catalytically active sites and a retardation in reaction rates. It is hypothesized that the electro catalytic performance of the LSCF electrode is adversely impacted by the formation of a resistive layer induced by Si contamination. It is suggested that the presence of Si contamination impedes the electro catalytic performance of the LSCF electrode, as evidenced by the DRT analysis.

The surface morphology of the posttest LSCF electrode is shown in Figure 12(a-c) after a 100h electrochemical test. The electrode morphology exhibits noticeable changes in surface appearance. The EDS analysis of the electrode, from marked region, also shows the change in the chemistry. Figure 12(c) shows the surface coverage of LSCF electrode. The EDS analysis reveals enrichment Sr and presence of silica at the surface suggesting the formation of the strontium silicate layer corroborating the XRD results.

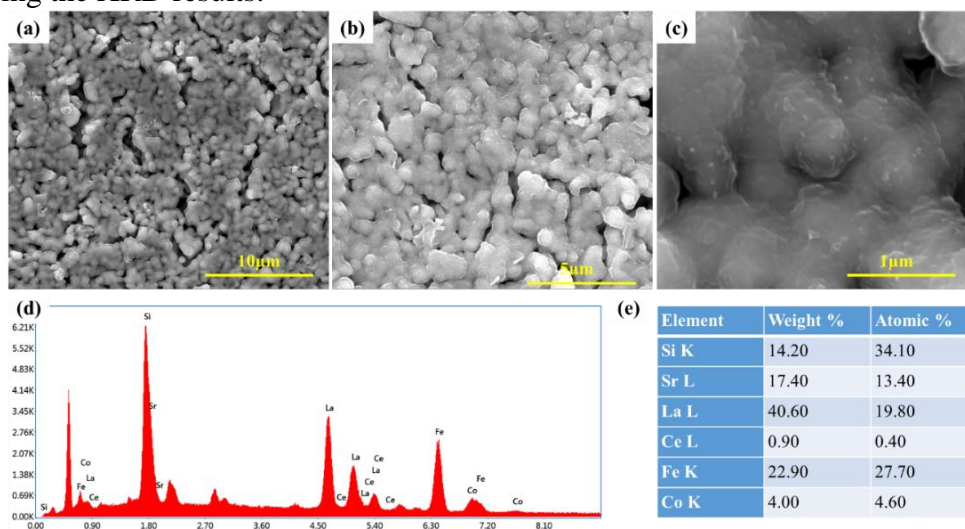


Figure 13: Surface morphology of LSCF electrode after the electrochemical test in high humid air (90% H₂O-Air) containing Si gaseous species (700 °C for 100h.)

To further examine the nature of the interaction of Si with the LSCF electrode and surface compound formation, the free electrode surface (gas-electrode interface) was further analyzed by TEM. Elemental distribution in the surface layer was obtained. TEM sample sections were prepared using by focused ion beam (Helios PFIB dual beam) technique. Elemental distribution and relative concentrations (mapping) was performed with STEM as shown in Figure 13. The

elemental mapping shows association of Sr and Si in the surface layer indicating the formation of strontium silicate surface layer. XRD analysis supports the observation. Analysis of the elemental distribution in the surface layer also indicates segregation of Sr on the grain boundary and localized enrichment of Co.

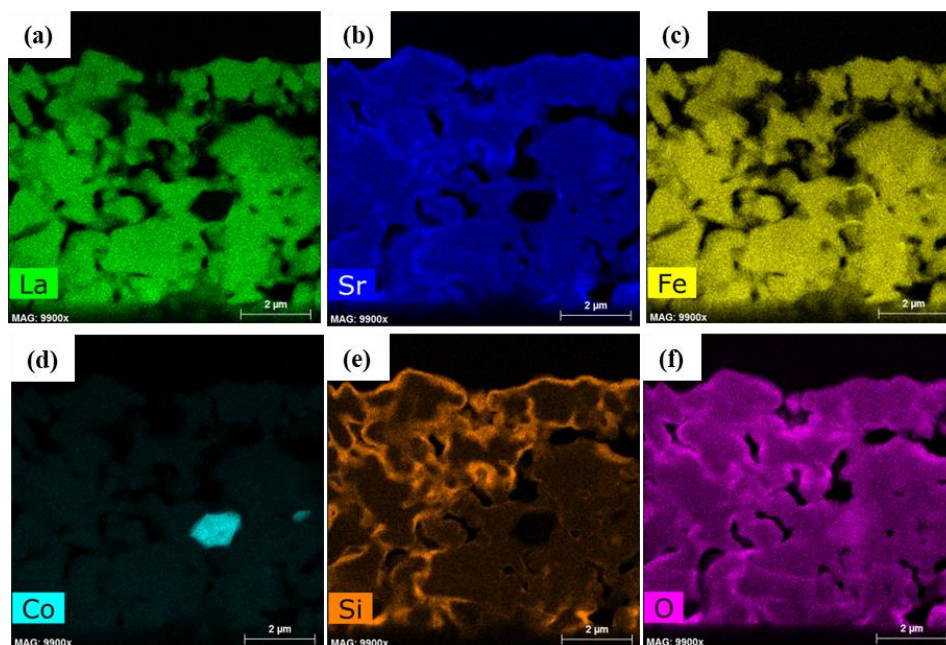


Figure 14: Elemental mapping of posttest LSCF electrode.

5.5. Task 5: Getter design optimization using computational flow analysis

The proposed task focused on the development materials chemistry along with getter design for achieving a uniform getter interaction with contaminants. The design considerations included minimization/ elimination of gas channeling, short circuit diffusion and mixing. The getter formulations have been considered to capture gas-phase sulfur, chromium, silicon, boron, and other trace-level contaminant gaseous species simultaneously. Thermodynamic principles and computational materials modeling research utilized to design oxides and related compounds capable of capturing these species from moist air, especially those interacting with chromia-forming alloys and silicon-containing sealants in SOFC stacks. Additionally, the study aimed to verify the stability of reaction products formed under diverse SOFC operating conditions, accounting for varying temperatures and contaminant levels.

5.5.1. Sub-task 5.1 Getter optimization using computational modeling

The subtask included the development of computational fluid dynamics CFD model to understand flow interaction pattern with the getter and fabrication of getter with developed optimized design for the validation through experimentation. A number of getter support designs will be analyzed through CFD analysis to visualize flow dynamics. A parametric study of the getter support and coating structures illustrate the effects of the key operating parameters such as geometric surface area, tortuosity and porosity, thickness of coating materials, pore size and its distribution across the coating thickness.

5.5.2. Sub-task 5.2 Development of optimal coating and getter design

Pertinent thermochemical data were obtained using HSC Chemistry software to create reaction models. This software allows the study of various gas-solid reactions at different temperatures and pressures. The data helps in comparing how stable different materials are when used in the specific conditions of solid oxide fuel cells (SOFCs).

An optimum getter support and micro and macro structural coating specifications were proposed to allow for extended gas-getter contact at low pressure drop requirement under systems operating conditions. The getters have been fabricated and tested to validate the model.

5.6. Task 6: Scale-up and long-term performance testing under system conditions

This task comprised of scaling up of the material synthesis and getter fabrication processes for the fabrication of large scale getters (support and coating) followed by integration and performance testing in prototypical SOFC stack and system test bed. After successful demonstration of the co-capturing of airborne impurities during bench top tests, processes for the fabrication of getter with optimized design were used for scaling up.

5.6.1. Sub-task 6.1 Materials scale-up to meet large systems requirements

This task addressed the identification and development of scalable manufacturing processes for the fabrication of select getter designs. Getter materials developed in the discovery stage of research are mostly produced in gram quantities. Initial synthesis processes were scaled to produce materials at hundreds of grams per batch after selected getter materials has been validated and proven to be effective.

5.6.2. Sub-task 6.2 Identification and development of quality control procedures

This sub-task addressed the identification and development of analytical methods and quality control procedures for the materials synthesis and getter fabrication processes. Getter materials were produced at batch level and examined for the chemical consistency in terms of elemental composition and phases, particle size and morphology. Existing and commonly practiced laboratory techniques were used to assure consistency of the material.

5.6.3. Sub-task 6.3 Independent validation of getters by SOFC industrial partners

This subtask involved getter materials electrochemical, physical and chemical evaluation by industrial partners. A batch of 200-250g getter powder, prepared and characterized, have been provided to the SOFC industry for their test validation under DOE guidance. Fabricated getters were provided to industrial collaborators for the cell and stack level evaluation. Details of the materials chemistry, reaction mechanisms, test procedure and materials limitations and other relevant information were transferred to industry for scale up and application in SOFC. UConn is able to provide any assistance to the SOFC industry partners with technology transfer, testing, characterizations, and on-site technical support as needed.

5.7. Task 7: Post-test characterization and mechanistic understanding of capturing impurities

In this task, posttest samples (obtained after completion of bench top tests, half-cell and full cell tests and long term endurance tests) were analyzed using a variety of analytical techniques to obtain and understand structural, chemical and morphological changes occurring at the cathode surface, cathode-gas and cathode-electrolyte interfaces and the bulk. The obtained information were used as a feedback to examine the poisoning mechanism of cathode under the presence of multi-constituent airborne impurities.

5.8. Task 8: Documentation, reporting, and publication

Technical progress, accomplishments, and experimental results have been documented and reported as required. Quarterly progress reports, technical and topical reports and technical

publications have been prepared and provided to the program manager. Findings and results of the program have been presented at technical meetings and published in peer reviewed journal.

5.9. Task 9: Intellectual property and technology transfer

The novelty of the technology has been documented through patent disclosures, while technical innovations have been shared with our industrial partners.

6. Summary and conclusions

Alkaline earth metal-based oxides serve as low cost active getter constituents for the capture of trace gaseous contaminants present in air streams entering high-temperature electrochemical systems. Getter constituents have been synthesized by conventional and co-precipitation routes including sol-gel method. Synthesized powder have been used for the dip coating of alumina-based porous foam and cordierite honeycomb monoliths. Transpiration tests, performed under SOFC operating conditions, have demonstrated the formation and transport of gaseous species of extrinsic contaminants such as Cr, Si and B in addition to the presence of airborne intrinsic contaminants such as SO₂, CO₂, and moisture. Transpiration tests performed with getter confirms the getters ability to capture intrinsic and extrinsic airborne contaminants. Thermodynamic models support the getter material's ability to reduce contaminant partial pressure due to the formation of stable reaction products. Electrochemical measurements with the LSM and LSCF air electrodes under individual and simultaneous exposure to airborne contaminants help in understanding the electrode poisoning mechanisms. Electrochemical tests conducted in the laboratory also confirm the efficacy of the getters in mitigation of poisoning due to the capture the trace contaminants. Alkaline earth metal-based getters offer a cost effective pathway for the mitigation of electrode poisoning and capture of contaminants in SOFC and other electrochemical systems.

Products

a. Publications, conference papers, and presentations

i) Journal publications:

1. Hong, J, Anisur, M. R., Heo, S J, Dubey, P K, and Singh, P. Sulfur Poisoning and Performance Recovery of SOFC Air Electrodes. *Frontiers in Energy Research*, 9, 2021.
2. Hong, Junsung, Su Jeong Heo, and Prabhakar Singh. "Water mediated growth of oriented single crystalline SrCO₃ nanorod arrays on strontium compounds." *Scientific Reports* 11, no. 1 (2021): 3368.
3. Hong, Junsung, Su Jeong Heo, and Prabhakar Singh. "Combined Cr and S Poisoning Behaviors of La_{1-x}Sr_xMnO_{3±δ} and La_{1-x}Sr_xCo_{1-y}Fe_yO_{3-δ} Cathodes in Solid Oxide Fuel Cells." *Applied Surface Science* (2020): 147253.
4. J. Hong, A.N. Aphale S.J. Heo, B. Hu, M. Reisert, S. Belko, and P. Singh, "Strontium Manganese Oxide Getter for Capturing Airborne Cr and S Contaminants in High-Temperature Electrochemical Systems" *ACS applied materials & interfaces*, 11(38), (2019) 34878-34888.
5. Hong, Junsung, Su Jeong Heo, Ashish N. Aphale, Boxun Hu, and Prabhakar Singh. "H₂O absorption assisted Sr-segregation in strontium nickel oxide based chromium getter and encapsulation with SrCO₃." *Journal of the Electrochemical Society* 166, no. 2 (2019): F59.
6. Aphale, Ashish, Junsung Hong, Boxun Hu, and Prabhakar Singh. "Development and validation of chromium getters for solid oxide fuel cell power systems." *JoVE (Journal of Visualized Experiments)* 147 (2019): e59623.

7. S.J. Heo, J. Hong, A. Aphale, B. Hu, and P. Singh, "Chromium Poisoning of $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3\pm\delta}$ Cathodes and Electrochemical Validation of Chromium Getters in Intermediate Temperature-Solid Oxide Fuel Cells" *Journal of The Electrochemical Society*, 166(13), (2019)
8. Aphale, Ashish, Boxun Hu, and Prabhakar Singh. "Low-Cost Getters for Gaseous Chromium Removal in High-Temperature Electrochemical Systems." *Jom* 71 (2019): 124-130.
9. Reisert, Michael, Ashish Aphale, and Prabhakar Singh. "Solid oxide electrochemical systems: Material degradation processes and novel mitigation approaches." *Materials* 11, no. 11 (2018): 2169.

ii) Books or other non-periodical, one-time publications: N/A

ii) Other publications, conference papers and presentations:

1. Belko, Seraphim, Pawan Kumar Dubey, Kevin X. Lee, Junsung Hong, Michael Reisert, Boxun Hu, and Prabhakar Singh. "Multi-Contaminant Getter for Trace Airborne Contaminant Capture in Elevated Temperature Electrochemical Systems." *ECS Transactions* 111, no. 6 (2023): 2385.
2. Belko, Seraphim, Pawan Kumar Dubey, Kevin X. Lee, Junsung Hong, Michael Reisert, Boxun Hu, and Prabhakar Singh. "Multi-Contaminant Getter for Trace Airborne Contaminant Capture in Elevated Temperature Electrochemical Systems." 18th International Symposium on Solid Oxide Fuel Cells (SOFC-XVIII) (243rd ECS Meeting) May 28-June 2, 2023 Boston, MA.
3. Pawan Kumar Dubey, Kevin Lee, Michael Reisert, Seraphim Belko and Prabhakar Singh "Multi-Constituent Airborne Contaminants Capture with Low Cost Oxide Getters and Mitigation of Cathode Poisoning in Solid Oxide Fuel Cell" 23rd Annual Solid Oxide Fuel Cell (SOFC) Project Review Meeting, Pittsburgh October 25-27, 2022.
4. Junsung Hong, Su Jeong Heo, Ashish N. Aphale, Boxun Hu, Prabhakar Singh, Cathode Poisoning and Mitigation in the Presence of Combined Cr and S Contaminants in SOFC , Presenting at the 2019 TMS Annual Meeting & Exhibition, TX, USA.
5. Aphale, Ashish, Michael Reisert, Junsung Hong, Su Jeong Heo, Boxun Hu, and Prabhakar Singh. "Capture of Trace Airborne Contaminants: Application to Electrochemical Systems." 49th International Conference on Environmental Systems, 2019.
6. Hu, Boxun, Su Jeong Heo, Junkai He, Yanliu Dang, Ashish Aphale, Aman Uddin, Junsung Hong et al. "High Surface Area Getter Materials for Chromium and Sulfur Capture in SOFC Systems."

b. Website(s) or other Internet site(s): None

c. Technologies or techniques: None

d. Inventions, patent applications, and/or licenses: None

e. Other products: None

Training and Professional Development

One post-doctoral fellow has been trained to perform the modelling work. The graduate students are being trained with experimental methods and analytical tools. The post-doctoral fellow and the students are being educated with the fuel cell knowledge.

Dissemination of Results

The targets of Milestone I, have been met with the management plan. Initial procedure for getter synthesis and materials identification has been achieved. The work plan, experimental

designs and the results are documented in laboratory notebook as well as the laboratory computers in the University of Connecticut.

Participants and Other Collaborating Organization

Prof. Prabhakar Singh

Project role: Principal investigator

Contribution to project: Provide guidance and oversee the project

Prof. Steven L. Suib

Project role: Co-investigator

Contribution to project: Leading the high surface area materials synthesis.

Dr. Boxun Hu

Project role: Co-investigator

Contribution to Project: Leading experimental research through planning, training graduate students, analyzing the results, and interfacing with the team.

Professor Scott Mixture

Alfred University, Co-investigator

Professor Avinash Dongare

Project role: Co-investigator

Contribution to project: Leading large scale assembly of getters using cordierite support

Professor Avinash Dongare

Project role: Co-investigator

Contribution to project: Leading DFT and energetic study of cathode and getter materials

Dr. Ashish Aphale

Project role: Post-doctoral fellow

Contribution to Project: Conducting the experiments, analyzing the data

Dr. Mohammad Anisur Rahman

Project role: Post-doctoral fellow

Contribution to Project: Conducting the experiments, analyzing the data.

Dr. Pawan Kumar Dubey

Project role: Post-doctoral fellow

Contribution to Project: Conducting the experiments, analyzing the data.

Dr. Kevin Lee

Project role: Post-doctoral fellow

Contribution to Project: Conducting the experiments, analyzing the data.

Mr. Junsung Hong

Project role: Graduate Student

Contribution to Project: Conducting experiments and analysis of results

Mr. Seraphim Belko

Project role: Graduate Assistant

Contribution to Project: Preparing the materials and assistance in conducting experiments

Impact

The topic of long term cathode degradation due to the ingestion of airborne gaseous impurities and development of cost effective materials and methodologies for the mitigation of the above

remains in the forefront of technical challenges limiting the long term stable operation of cells and stacks.

The proposed approach offers design flexibility and potential for operation under a wide temperature range (600-800°C) to capture multiple impurities under systems operating conditions.

Key advantages include:

- Use of non-noble and non-strategic materials
- Low cost conventional alkaline earth and transition metal oxide for getter formulation
- Conventional ceramic processing techniques and use of ceramic backbone
- Design flexibility for integration in BOP and within the cell stack
- System and stack design independent

Changes/Problems

There is no change on the goals of the project as well as experimental plans so far.

Special Reporting Requirements

Not Applicable

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- [1] Hong J, Aphale AN, Heo SJ, Hu B, Reisert M, Belko S, et al. Strontium Manganese Oxide Getter for Capturing Airborne Cr and S Contaminants in High-Temperature Electrochemical Systems. *ACS Appl Mater Interfaces* 2019;11:34878–88.
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