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# Development and Validation of Chromium Getters for Solid Oxide Fuel Cell Power Systems

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Cover Letter

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March 12, 2019

Dr. Bing Wu
Review Editor
JoVE
1 Alewife Center Suite 200
Cambridge MA 02140

Subject: Submission of revised manuscript titled “Development and Validation of Chromium Getters for Solid Oxide Fuel Cell Power Systems” for publication in JoVE

Dear Dr. Wu,
Following your comments, we have revised our manuscript. It is our pleasure to re-submit the revised manuscript titled, “Development and Validation of Chromium Getters for Solid Oxide Fuel Cell Power Systems” for publication in the JoVE.

Two versions with and without marked changes are provided. The revised contents are marked in red in one attached manuscript.

The manuscript contains original research work and it has not been published earlier. Also, the manuscript is not under consideration for publication elsewhere. The manuscript is approved by all authors and the host authorities and there are no conflicts of interest. If I may be of any further assistance, please do not hesitate to call me at 860-381-0131 or email me at boxun.hu@uconn.edu.

Thank you!

Sincerely,

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**TITLE:**

# Development and Validation of Chromium Getters for Solid Oxide Fuel Cell Power Systems

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**KEYWORDS:**
chromium evaporation, high temperature electrochemical systems, solid oxide fuel cell stability, cathode degradation, chromium capture, electrochemistry.

**SUMMARY:**

Cathode poisoning from airborne contaminants in trace levels remains a major concern for long-term stability of high-temperature electrochemical systems. We provide a novel method to mitigate the cathode degradations using getters, which capture airborne contaminants at high temperature before entering electrochemically active stack area.

**ABSTRACT:**

Degradation of cathode in solid oxide fuel cells (SOFC) remains a major concern for the long-term performance stability and operational reliability. The presence of gas phase chromium species in air has demonstrated significant cathode performance degradation during long-term exposure due to unwanted compound formation at the cathode and electrolyte interface which retards the oxygen reduction reaction (ORR). We have demonstrated a novel method to mitigate the cathode degradation using chromium getters which capture the gas phase chromium species before it is ingested in the cathode chamber. Low-cost getter materials, synthesized from alkaline earth and transition metal oxides, are coated on the cordierite honeycomb substrate for application in the SOFC power systems. As-fabricated getters have been screened by chromium transpiration tests conducted for 500 h in humidified air atmosphere in presence of chromium vapor. Selected getters have been further validated utilizing electrochemical tests. Typically, electrochemical performance of SOFCs (lanthanum strontium manganite (LSM) ‖ yttria stabilized zirconia (YSZ) ‖ Pt) was measured at 850 °C in the presence and absence of Cr getter. For the 100 h cell tests containing getters, stable electrochemical performance was maintained, whereas the cell performance in the absence of Cr getters rapidly decreased in 10 h. Analyses of Nyquist plots indicated significant increase in

the polarization resistance within the first 10 h of the cell operation. Characterization results from posttest SOFCs and getters have demonstrated the high efficiency of chromium capture for the mitigation of cell degradation.

## INTRODUCTION:

Solid oxide fuel cell (SOFC) power system, a high temperature direct electrochemical energy conversion device, offers an environmentally friendly pathway to generate electricity from a wide variety of fossil and renewable fuels. SOFC technology finds its applications in centralized as well as distributed power generation areas[1]. This technology relies on electrochemical conversion of chemical energy stored in the fuels into electricity. Numerous advantages are offered by SOFCs in terms of high energy efficiency, high quality heat, ease of modularity, and no or negligible carbon footprints[2]. Several individual SOFC cells are connected in series or parallel fashion (namely SOFC stacks) to obtain desired output voltage. SOFC stacks consist of components such as dense electrolyte, porous electrodes, interconnection (IC) and seals[3,4]. Anode and cathode of adjacent cells are connected using IC, which not only serves as a separator to prevent any mixing of oxidant with fuel but also provides electrical connection between the adjacent anode and cathode[5].

Improvements over decades of research and development in materials engineering have led to reduction in operating temperature for SOFCs, enabling replacements of ceramics materials with inexpensive stainless-steel alloys for the fabrication of electrochemically active cell and stack components and balance-of-plant (BOP) sub-systems. Commercially available ferritic and austenitic stainless steels are utilized for the fabrication of system components due to their low cost, matched coefficient of thermal expansion (CTE) and resistance to oxidation and corrosion at high operating temperatures[6]. Formation of $Cr_2O_3$ type passivating oxide scale on the alloy surface acts as a barrier layer against inward diffusion of oxygen from air or outward diffusion of cations from bulk alloy[7].

In the presence of humidified air, $Cr_2O_3$ undergoes significant chemical transformation leading to hydrated chromium vapor species formation at SOFC operating temperatures. The gaseous chromium vapor is subsequently carried through the air stream into the cathode leading to surface and interface reactions with the cathode materials. Such cathode experiences both ohmic and non-ohmic increases in the polarization and electrical performance degradation. Details of the cathode degradation mechanisms have been illustrated elsewhere[8–10].

The state-of-the-art methods to reduce or eliminate the above cathode degradation processes commonly consist of modifications of the alloy chemistry, application of surface coating and the use of chromium tolerant cathodes[11,12]. Although these techniques have demonstrated reduction of the cathode degradation due to Cr vapor interactions (namely Cr poisoning) for short-term, long-term efficacy for performance stability remains a concern, mainly due to cracking and spallation within the coating and interdiffusion of cations.

We have demonstrated a novel method to mitigate the problem of chromium poisoning by capturing the incoming chromium vapor before it reacts with the cathode materials[13]. The getters have been synthesized from low-cost alkaline earth and transition metal oxides using conventional ceramic processing techniques. The cost advantage of this approach is use of non-noble and non-strategic materials as well as conventional processing methods to fabricate getters for the mitigation of cathode degradation arising from airborne contaminants. The placement of the getter can be tailored to capture chromium vapor arising from BOP components or it can also be tailored to be placed within the electrochemically active stack components[14,15]. Here, we present methods to validate the chromium getters using transpiration and electrochemical tests. Experimental setup and characterization results will also be demonstrated to show the getter effectiveness and the mechanisms of Cr capture on the getter under typical SOFC operating conditions.

## PROTOCOL:

### 1. Synthesis of chromium getter

1.1. Synthesize the precursor powder using alkaline earth and transition metal oxide salts via a conventional coprecipitation synthesis route as depicted in **Figure 1**[16].

1.1.1. Prepare stock solutions using 50.33 g of strontium nitrate $Sr(NO_3)_2$ and 43.97 g of nickel nitrate hexahydrate $Ni(NO_3)_2.6H_2O$ in order to prepare 2.4 M solutions in 100 mL of de-ionized water.

1.1.2. Use 9 mL of 2.4 M $Sr(NO_3)_2$ and add 7 mL of 2.4 M solution of $Ni(NO_3)_2.6H_2O$, followed by the addition of 30 mL of 5 M $NH_4OH$ to increase the pH to 8.5.

1.1.3. Stir the mixed solution and heat it up to 80 °C for precipitation. Then, dry the solution in a dry oven and ensure that all the water evaporates until a blue waxy compound is observed. Rinsed the powder in deionized (DI) water to ensure that residual ammonium nitrate is removed by filtration. Finally, dry the powder at 120 °C for 2 h.

NOTE: This will result into precursor powder for strontium nickel oxide (SNO) getter.

1.2. Dissolve the powder in water to prepare a slurry.

1.3. Immerse the cordierite substrate in the slurry for dip-coating, followed by drying in air at ~120 °C for at least 2 h with a ramp rate of 5 °C.

1.4. Calcine the substrate in air at the temperature of 650 °C for 12 h with a ramp rate of 5 °C to produce SNO getter.

### 2. Screening of chromium getter using Cr transpiration test

2.1. Set up the experiment following the illustration in **Figure 2a** for the validation of Cr getters.

2.1.1. Place 2 g of sintered chromium oxide pellet (1200 °C, 2 h) as a chromium source in a quartz tube.

NOTE: The quartz tube is specifically designed with a diffuser inside (shown in **Figure 2**) to prevent any back diffusion of chromium vapor during operation. The dimensions of the fabricated getter cartridge match the inside diameter of the quartz tube. Getter cartridge is placed between the chromium source and the outlet elbow (shown in **Figure 2**).

2.1.2. Flow the compressed air at a flow rate of 300 sccm through a mass flow controller (MFC). Bubble the air at room temperature water to ensure that the humidity is 3% $H_2O$. This humidified air passes through the chromia pellets, evaporates chromium vapor and flows through the getter.

NOTE: The chiller and condenser are placed at the outlet of the transpiration setup to enable condensation of chromium-containing vapor which deposits at the outlet elbow (at the low temperature zone).

2.1.3. Place additional wash bottles before venting the gas at the outlet to ensure that the evaporated chromium is captured.

2.1.4. With the setup, purge the tube with air for at least 1 h to ensure that there is no leaks or contaminants.

2.1.5. Start the furnace to heat up to the desired temperature (for example, 850 °C in this case) and hold it there for 500 h.

2.1.6. Monitor the color change of the outlet elbow and record for any discoloration due to deposited chromium compounds.

2.1.7. Lower the furnace temperature back to room temperature (RT) after the completion of the test. Turn off the air flow until the furnace temperature reaches RT.

2.1.8. Remove the getter sample for post-test analyses and characterization.

### 2.2. Quantitative analysis of chromium species by inductively coupled plasma mass spectroscopy (ICP-MS)

NOTE: ICP-MS sample preparation of post Cr transpiration test[17].

2.2.1. Wash the deposited chromium from glass elbow, condenser, wash bottles and quartz tubes using 20% nitric acid ($HNO_3$) to extract the chromium after conducting transpiration test for 500 h.

2.2.2. Extract the deposited chromium by dissolving it in 20% $HNO_3$ for 12 h.

2.2.3. Further remove any undissolved chromium species from the glass wall by dissolution in alkaline potassium permanganate solution upon heating at 80 °C.

NOTE: Convert any partial unreacted $Cr^{3+}$ species to $Cr^{6+}$ species in this step.

2.2.4. Analyze the DI water and nitric acid blank sample by ICP-MS.

2.2.5. Divide each sample into three parts for ICP-MS analysis and report the average value.

## 3. Electrochemical validation of chromium getter using SOFC cells with and without getter

### 3.1. Cell fabrication and in-operando electrochemical testing of Cr getters[18,19]

3.1.1. Fabricate SOFCs by screen printing LSM paste on the surface of YSZ electrolyte (**Figure 3a**).

3.1.2. Sinter the applied LSM ink at 1200 °C for 2 h, heated with a ramp rate of 3 °C/min.

3.1.3. Use a Pt electrode as the anode. Attach a Pt on the YSZ disc (anode side) as a reference electrode, and attach Pt gauze and Pt wires to YSZ electrolyte disc using Pt ink and then cure the SOFC at 850 °C for 2 h at a ramp rate of 3 °C/min.

3.1.4. Conduct three distinct experiments using three identical SOFCs (namely Cell a, b, and c) to validate the efficacy of getters and demonstrate chromium poisoning without a getter.

NOTE: Make sure to use the identical test conditions to simulate nominal SOFC operating conditions of 850 °C and maintain the anode air (dry) for all the tests at 150 sccm.

3.1.5. Assembly the Cell-a in the tube reactor in the absence of chromium source using paste for sealing. Heat the furnace with a ramp rate of 5 °C/min up to a designed temperature (for example: 850 °C in this study). Then, flow the 3% $H_2O$/air (for example 300-500 sccm) to the LSM cathode.

3.1.6. Measure the electrochemical performance of the SOFC using a multi-channel potentiostat[9].

3.1.7. Record the cell current every minute with a bias of 0.5 V applied between the cathode and the reference electrode.

3.1.8. Conduct electrochemical impedance spectroscopy (EIS) analyses between cathode and reference electrode using three electrode mode in the frequency range of 0.5 Hz to 200 KHz with 10 mV sinus amplitude at an interval of 1 h. After a 100 h test, cool down the furnace to room temperature and take the Cell-a for characterization.

3.1.9. Place 2 g of chromium oxide ($Cr_2O_3$) pellets (source of chromium vapor) in a porous container at the constant heating zone of the alumina tube. Assembly the Cell-b in the tube reactor using paste for sealing. Heat the furnace with a ramp rate of 5 °C/min up to 850 °C. Then, flow the humidified air (for example 300-500 sccm) through the chromia pellets and ensure a constant generation of the chromium vapor species[9].

3.1.10. Repeat Steps 3.1.6 – 3.1.8. After a 100 h test, cool down the furnace to room temperature and take the Cell-b for characterization.

3.1.11. Place 2 g of chromium oxide ($Cr_2O_3$) pellets (source of chromium vapor) in a porous container at the constant heating zone of the alumina tube. Place a chromium getter above the chromium source. Assembly the Cell-c on the top of the tube reactor using paste for sealing. Heat the furnace with a ramp rate of 5 °C/min up to a designed temperature (for example: 850 °C in this study). Then, flow the 3% $H_2O$/air (for example 300-500 sccm) to the LSM cathode.

3.1.12. Repeat Steps 3.1.6 – 3.1.8. After a 100 h test, cool down the furnace to room temperature and take the Cell-c for characterization.

## 3.2. Posttest getter morphological and chemical characterization

NOTE: Posttest characterization is conducted using scanning electron microscopy (SEM) coupled with energy dispersive spectroscopy and scanning transmission electron microscopy (STEM) coupled with EDS analyses. Focused electron and ion-beam technologies (FIB) have been utilized for the preparation of nanoscale samples.

3.2.1. Analyze the microstructures of the cell component by fracturing after the electrochemical test. Use SEM instrument for morphological analysis. Ensure that both the morphologies and chemical compositions of LSM cathode surface and LSM/YSZ interface are analyzed[13, 14]

3.2.1.1. Prior to conducting SEM analysis, prepare the samples by sputter coating of gold (Au) films to make sure the sample surface is conductive (avoiding charge on the sample surface). The coating chamber is under a vacuum (< 50 mm Torr). The applied current is at 40 mA and the coating time is 1 min.

3.2.1.2. Conduct quantitative elemental distribution using energy dispersive X-ray spectroscopy (EDS) technique. Set the distance between the specimen and the lower pole piece in SEM system at 10 mm. Apply a voltage of 20 KV for the SEM and EDS analysis.

3.2.2. Conduct the chemical, structural and morphological analyses of the chromium getter using the SEM-EDS technique to obtain the chromium capture profile across the getter channels.

3.2.2.1. Prepare the posttest getter sample by dissecting the getter sample into half using a knife.

3.2.2.2. Repeat Step 3.2.1.1 to coat conductive gold films on the getter surface.

3.2.2.3. Repeat Step 3.2.1.2. Ensure that detailed EDS analyses are conducted from the inlet of the getter towards the outlet along the central channel as shown in **Figure 2b**. Use weight (wt.) % of total chromium measured against the channel length to plot the chromium profile.

3.2.3. Conduct in-depth chemical, structural and morphological analyses of the chromium getter using the FIB-STEM-EDS technique[17,20].

3.2.3.1. Repeat Step 3.2.1.1 to coat conductive gold films on the getter surface.

3.2.3.2. Load the sample in the FIB-STEM instrument, select the region of interest (ROI) for sample extraction, deposit four layers of Pt to mark and protect the sample (a typical area of 30 µm length × 15 µm width).

3.2.3.3. Mill the channels around the above ROI using the FIB beam until a "bridge-like" strip is left. Then, make wedges at both sides of the strip to make sure the depth is enough for the analysis (a typical depth is 10-20 µm).

3.2.3.4. Mount the micromanipulator needle and cut the FIB sample by milling using an ion beam with 15 nA current. Then lift the FIB sample from the bulk getter sample to the FIB-STEM grid holder, which is perpendicular to the electron beam. After the specimen touch the grid at right position, deposit Pt using an ion beam current of 0.5 nA to connect the specimen to the grid.

3.2.3.5. Make the specimen thinner using a FIB current of about 20 pA at 2 kV to obtain a 50-60 nm sample thickness. Perform a final clean-up of the specimen using argon-milling at an extra low current (0.5 pA at 1 kV).

3.2.3.6. Conduct STEM-EDS mapping of the above getter specimen. Operate the scanning transmission electron microscope at 200 kV. A High Angle Annular Dark Field (HAADF) image of the selected area on the getter specimen was obtained and elemental maps of relevant elements (such as Cr and Sr) were taken.

## REPRESENTATIVE RESULTS:

A Cr transpiration experiment is a screening test for the selection of Cr getters. Cr transpiration setup was utilized to validate the performance of chromium getter under the SOFC operating conditions. Experiments were conducted in the presence of a chromium getter operated at 850 °C in humidified (3% $H_2O$) air for 500 h. Visual observations during Cr transpiration tests indicated significant discoloration of the outlet elbow during 500 h in the absence of getter. However, placing a getter next to the chromium source demonstrated no discoloration of the outlet elbow. This indicated that the getter can effectively capture all the incoming chromium vapor species under nominal SOFC operating conditions. To validate the chromium capture and understand the gettering mechanism, the posttest getter was dissected and observed under the SEM. Detailed EDS analyses were also conducted to measure the elemental distribution of chromium (wt.%) along the central channel of the getter (shown in **Figure 2b**). EDS elemental distribution of chromium shows that majority of Cr (wt.%) is captured within first 4000 µm of distance from the getter inlet. SEM-EDS data further shows that the middle and the outlet of the getter contains no or negligible chromium during transpiration test.

Proven Cr getters have been utilized for electrochemical validation tests. The electrochemical performance comparisons of the three LSM‖YSZ‖Pt SOFCs under different experimental test conditions are shown in **Figure 3b.** The LSM cathodes of the three SOFCs were exposed to humidified air (air-3% $H_2O$) to control the Cr vapor exposure to the cathodes. The cathode from **cell a** was exposed to 3% $H_2O$ air with no getter and no Cr vapor for 100 h. The results show stable I-t curve with a presence of cathodic activation period (0-20 h). The **cell b,** exposed to 3% $H_2O$ air with Cr vapor and no getter, shows a rapid drop in the current within the first few hours of the test. This indicated chromium poisoning of the cell. For the **cell c** in presence of a chromium getter and 3% $H_2O$/air at the cathode side with chromium vapor, the electrochemical performance of the **cell c** demonstrated a significant improvement, which was very close to that of the **cell a** (not shown in figures).

Effect of chromium vapor on cathode degradation, also known as chromium poisoning, has been studied as shown in **Figure 4**. A representative Nyquist plot is provided for **cell b** (**Figure 4b**), which was exposed to chromium vapor but without the getter. The electrochemical impedance of the cathode was measured independent of changes that may occur at the anode electrode using a three-electrode mode. When the cathode of the **cell b** was exposed in chromium vapor, the semi-circles of the Nyquist spectra of the cathode stretched with time, indicating an increase of polarization resistance with increase in exposure time. During the 100-h tests, the polarization resistance of the cathode in **cell b** exhibited a rapid increase during first 20 h, followed by slower change during the next 40 h, and significantly no change after 60 h. The non-polarization resistance (Rnp) of cathode showed only negligible changes. Above experiments indicate that chromium vapor mainly resulted in a rapid change of Rp which led to the cathode degradation. The increase in the polarization resistance of the cathode occurs mainly due to retardation of oxygen reduction reaction (ORR) at the electrode/electrolyte interface. To demonstrate this, morphological characterization of the fractured SOFC was conducted and compared with the morphology at the cathode surface. **Figure 4 (c1 and c2)** shows the SEM micrographs of the LSM surfaces and LSM/YSZ interfaces from **cell b**, respectively. The SEM-EDS analysis shows ~2.5 wt.% chromium on the cathode surface while 11.2 wt.% of chromium was observed at the electrode/electrolyte interface layer (**Table 1**). Significant deposition of chromium vapor takes place at the cathode-electrolyte interface, which inhibits the ORR and degrades the cathode performance with time.

After electrochemical tests, the chromium getter was prepared for microstructural analysis. The morphology of the deposited chromium on fiber supported getters was examined by SEM-EDS (**Figure 5a**). At localized areas near the air inlet, large particles rich in Cr (44.8 atom%) and Sr (54.3 atom%) were formed on the getter fibers. The getter fiber support from the mid-section and outlet remains free of chromium (not shown here).

To further investigate the reaction processes for the capture of chromium, a posttest getter fiber was milled using FIB technique (**Figure 5b**). **Figure 5c** shows the HAADF image of the cross-section of the posttest getter fiber by STEM. From the elemental mapping, a surface layer containing Sr and Cr has been observed as shown in **Figure 5d,e**, indicating a stable reaction product ($SrCrO_4$) formation on the surface of the alumina fiber. Near the surface of the $SrNiO_x$ coated alumina fiber, the outward diffusion of Sr from the $SrNiO_x$ coated getter material.

## FIGURE AND TABLE LEGENDS:

**Figure 1. Procedures for getter synthesis and fabrication.** Schematic illustration of getter power synthesis and coating method using conventional ceramic processing route.

**Figure 2. Illustration of Cr transpiration test setup and testing results.** (**a**) Experimental setup to conduct transpiration test under nominal SOFC conditions. (**b**) Distribution of chromium (wt.%) profile along the getter length from inlet to outlet. This figure has been modified from reference [14] with a permission.

**Figure 3. Electrochemical validation of getter and testing results.** (**a**) Schematic of LSM‖YSZ‖Pt SOFC, (**b**) I-t plots in presence and absence of getter, and (**c**) I-t plots in various cathode air flow rates.

**Figure 4. Effect of chromium poisoning on LSM cathode surface and LSM/YSZ interface.** (**a**) Illustration of chromium evaporation processing in presence of $O_2$ and $H_2O$. (**b**) Nyquist plot of the SOFC exposed to Cr vapor in 3% $H_2O$ air. (**c**) 1. Surface morphology of the LSM cathode exposed to Cr vapor, and 2. deposition of $Cr_2O_3$ along the interface of LSM and YSZ indicating electrode poisoning.

**Figure 5. Characterization results of posttest getter by EDS and FIB-TEM.** (**a**) Morphology of deposited Cr vapor on the surface of getter fiber long with respective elemental distribution. (**b**) Cross-section of a getter sample with deposited Cr using focused ion beam (FIB) technique. (**c**) High-angle annular dark-field imaging (HAADF) of the getter sample prepared by the FIB technique (**d**, **e**) Elemental maps of the FIB sample showing presence of Cr and Sr on the getter surface.

## DISCUSSION:

The experimental results clearly demonstrate the effectiveness of chromium getters during long-term chromium transpiration tests and electrochemical tests. Presence of getters successfully mitigates the contamination of the electrode which otherwise would lead to rapid increase in polarization resistance and electrochemical performance degradation.

The formation of gas phase chromium species from chromia is favored and enhanced with an increase of water vapor concentration (humidity level)[16]. The water content in the cathodic air is maintained at 3% representing room temperature humidification and saturation. A high temperature cell exposure condition of 850 °C has been selected to demonstrate the effectiveness of the prepared chromium getters in this study.

For the rational design of Cr getters, the first step is to identify various chromium species present in the humid air environment. Thermodynamic calculations indicated significantly different equilibrium partial pressures of chromium vapor species in dry and humidified air. $CrO_3$ was found as predominant gaseous species at elevated temperatures in dry air whereas hydrated oxides such as $CrO_2(OH)$ and $CrO_2(OH)_2$ were identified as prevalent species in humid air at elevated temperatures[15]. Amongst all the chromium vapor species, the partial pressure of $CrO_2(OH)_2$ remained relatively high throughout the entire temperature range (**Figure 4a**). It is noted that lowering of the temperature did not significantly lower the chromium vapor pressure. Presence of alkaline oxide (SrO for example) containing getter, however, has indicated significant reduction in the equilibrium chromium vapor pressure due to the formation of thermodynamically stable compound ($SrCrO_4$)[14]. In this study, our observations indicate that the cordierite supported SNO getter reacts with chromium vapors to form crystalline $SrCrO_4$ and that also lowers the partial pressure of Cr vapors considering the reaction (Eq. 1):

$$SrO_{(s)} + CrO_2(OH)_{2\,(g)} \rightarrow SrCrO_{4\,(s)} + H_2O_{(g)} \qquad (1)$$

During the long-term capture test using the transpiration method, discoloration of the outlet elbow was not observed, indicating absence of gaseous chromium vapor in the exiting air stream and hence the formation of yellowish deposit at lower temperatures on the exposed outlet elbow area. Most Cr vapor was captured within 5 mm of inlet getter (**Figure 2b**). In contrast, the outlet elbow shows significant discoloration after 500 h chromium transpiration test due to deposition of chromium species in the absence of getter. The discoloration on the outlet quartz tube is a visual indication of the presence of Cr vapor species in the gas phase. More precisely, the Cr capture efficiency has been evaluated by the ICP-MS analysis method. After conducting transpiration test for 100-500 h, the deposited chromium from glass elbow, condenser, wash bottles and quartz tubes were washed to extract the chromium by a fixed volume of 20% $HNO_3$ acid (for example, 1 L). The total moles of Cr species released per hour, measured by ICP-MS in different transpiration experiments, are compared for getter optimization. In our experiments, sintered $Cr_2O_3$ pellets were utilized as a stable chromium source of chromium vapor to minimize the carryover of fine $Cr_2O_3$ particles.

During baseline electrochemical experiment performed in the presence of chromium without a getter, gaseous chromium species flow through the porous LSM cathode are further reduced to form a $Cr_2O_3$ layer (**Figure 4a**) at the gas/LSM cathode/YSZ triple phase boundaries and cathode/electrolyte interface under a bias as shown in Eq.2.

$$2CrO_2(OH)_2\,(g) + 6e^- = Cr_2O_3\,(s) + 3O^{2-}\,(ion) + 2H_2O\,(g) \qquad (2)$$

Stoichiometric LSM remains largely unreacted under the entire SOFC operation range of chromium vapor species[9]. Our observations indicate that smaller amounts of $Cr_2O_3$ deposits at the LSM cathode surface (**Figure 4C1**) whereas majority of the $Cr_2O_3$ deposits at the triple phase boundaries (TPB) blocking the active sites for further oxygen reduction, increase in the polarization resistance (**Figure 4b**) and poor electrochemical performance of the cell[16].

The three-electrode electrochemical cell design and test setup, utilized in our previous studies of LSM cathode stability in $CO_2$ and humidified air[18,19], has proven to be a powerful test vehicle and configuration for electrochemical impedance measurements. A reference electrode is added at the anode side near the periphery of the YSZ electrolyte using platinum paste and wire. This Pt electrode acts as a reference for measuring and controlling the working electrode potential, without current flow (ideal case). The stable Pt electrode remains free of Cr deposition on the anode site.

During the electrochemical experiments in the presence of chromium with a getter, sintered and pure $Cr_2O_3$ pellets are utilized as the stable Cr source. Due to the utilization of pure $Cr_2O_3$ pellets in our getter validation tests, the resulting concentration of Cr vapor species is expected to be much higher than those found in conventional fuel cell stacks and systems, in which protected coatings are added to reduce Cr evaporation. Our electrochemical experiments, hence, can be considered as accelerated tests. Pure LSM cathode are utilized as the cathode, which is very sensitive to chromium poisoning to validate the cathode poisoning and getter mechanisms. With an increase of air flow rates from 50 sccm to 500 sccm, which is similar to the real application conditions, the LSM‖YSZ‖Pt SOFCs maintain stable electrochemical performance as shown in **Figure 3C**, indicating the Cr getter still effectively capture Cr vapors at relatively high flow rates. In our ongoing and future work, high surface area getters and computational fluid dynamics (CFD) method are being developed for more active and longer lasting getters.

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## DISCLOSURES:

Authors do not have anything to disclose.

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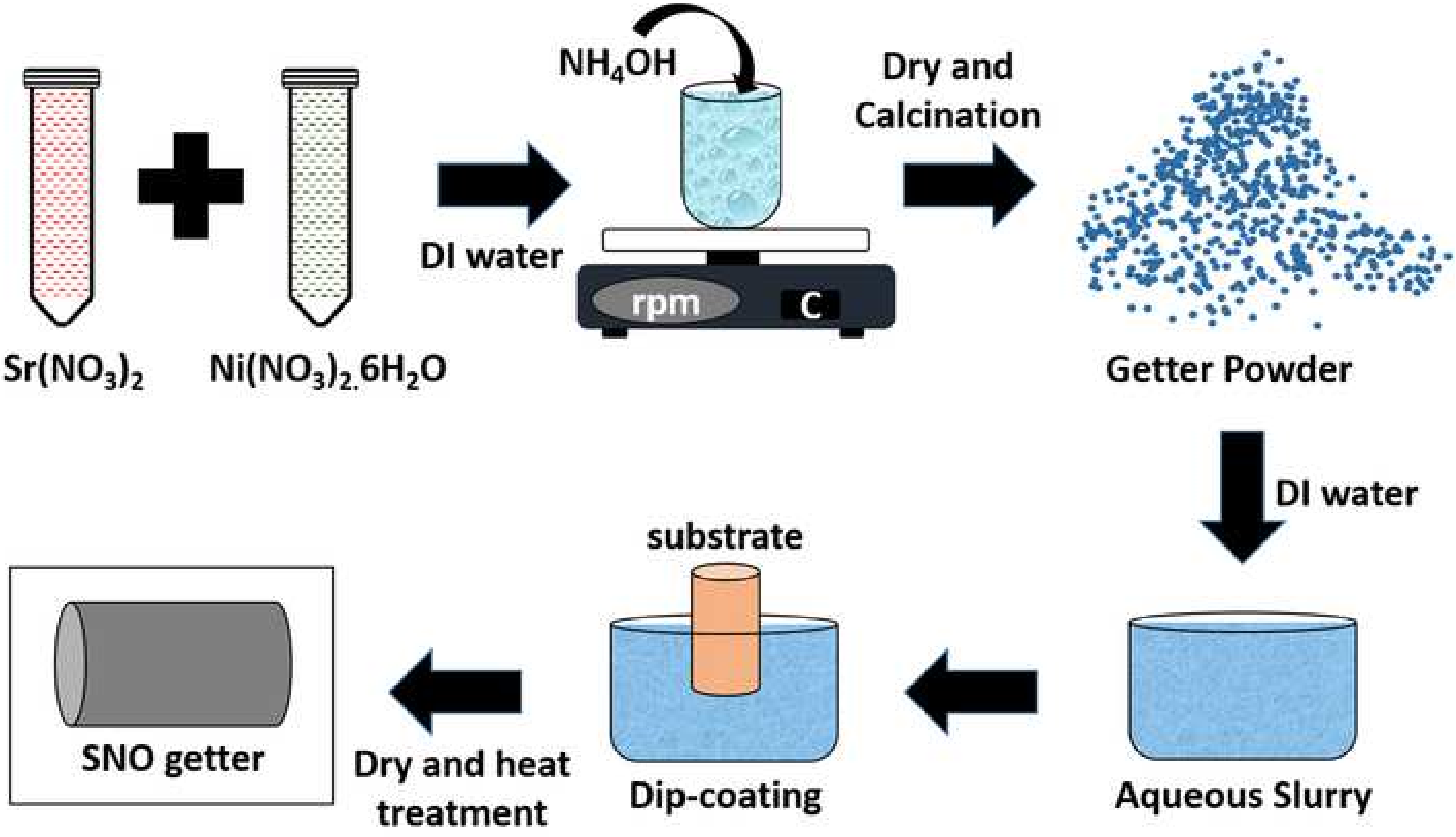
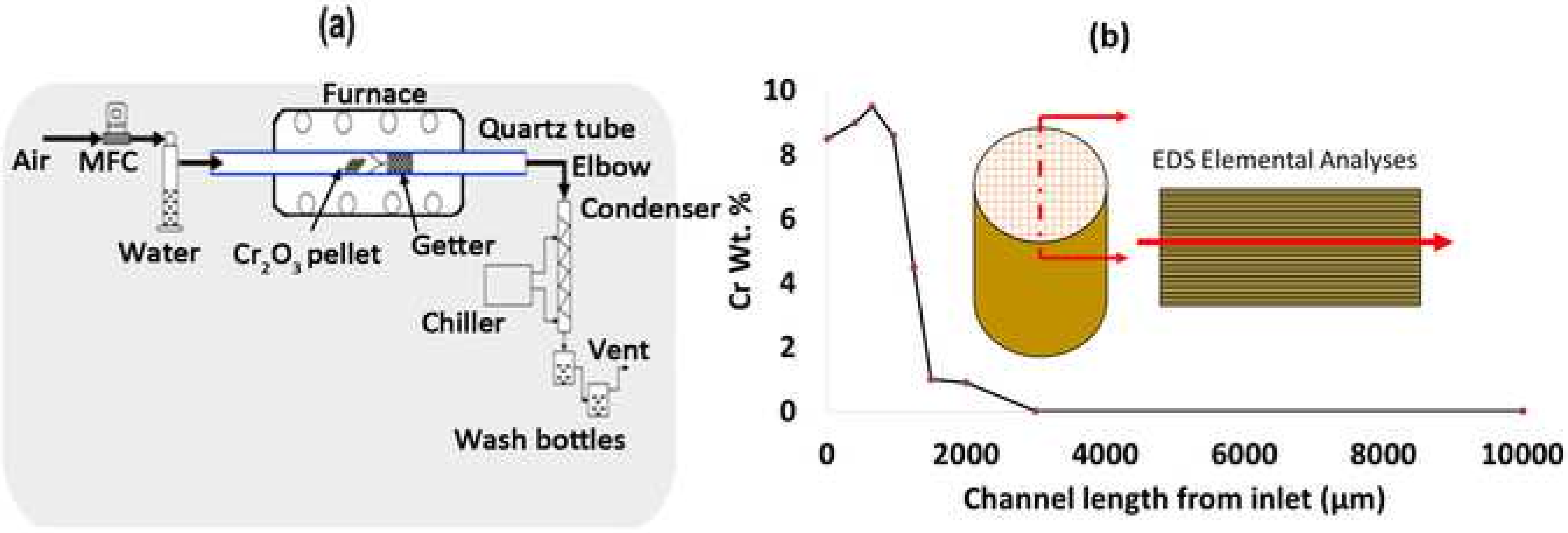
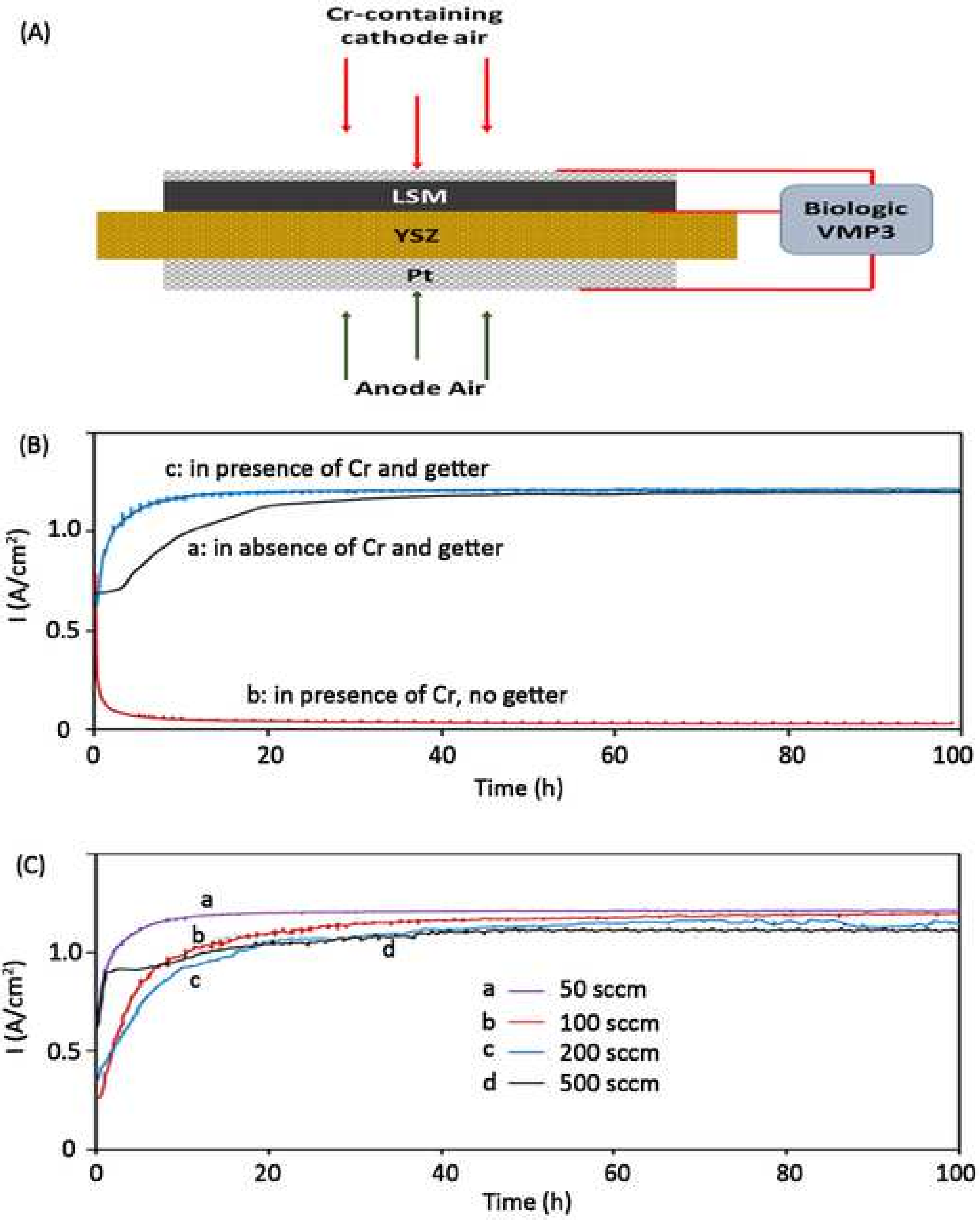
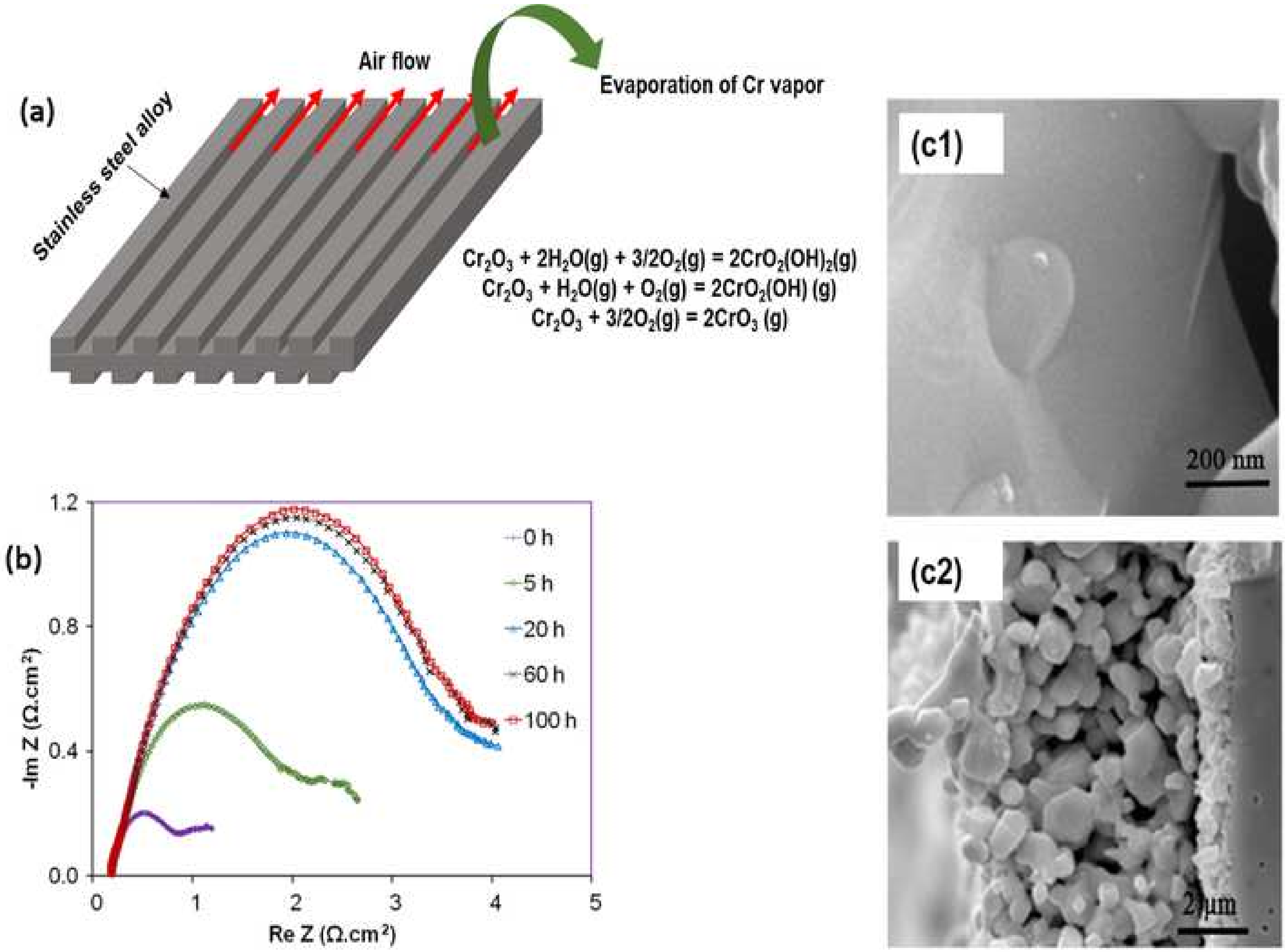
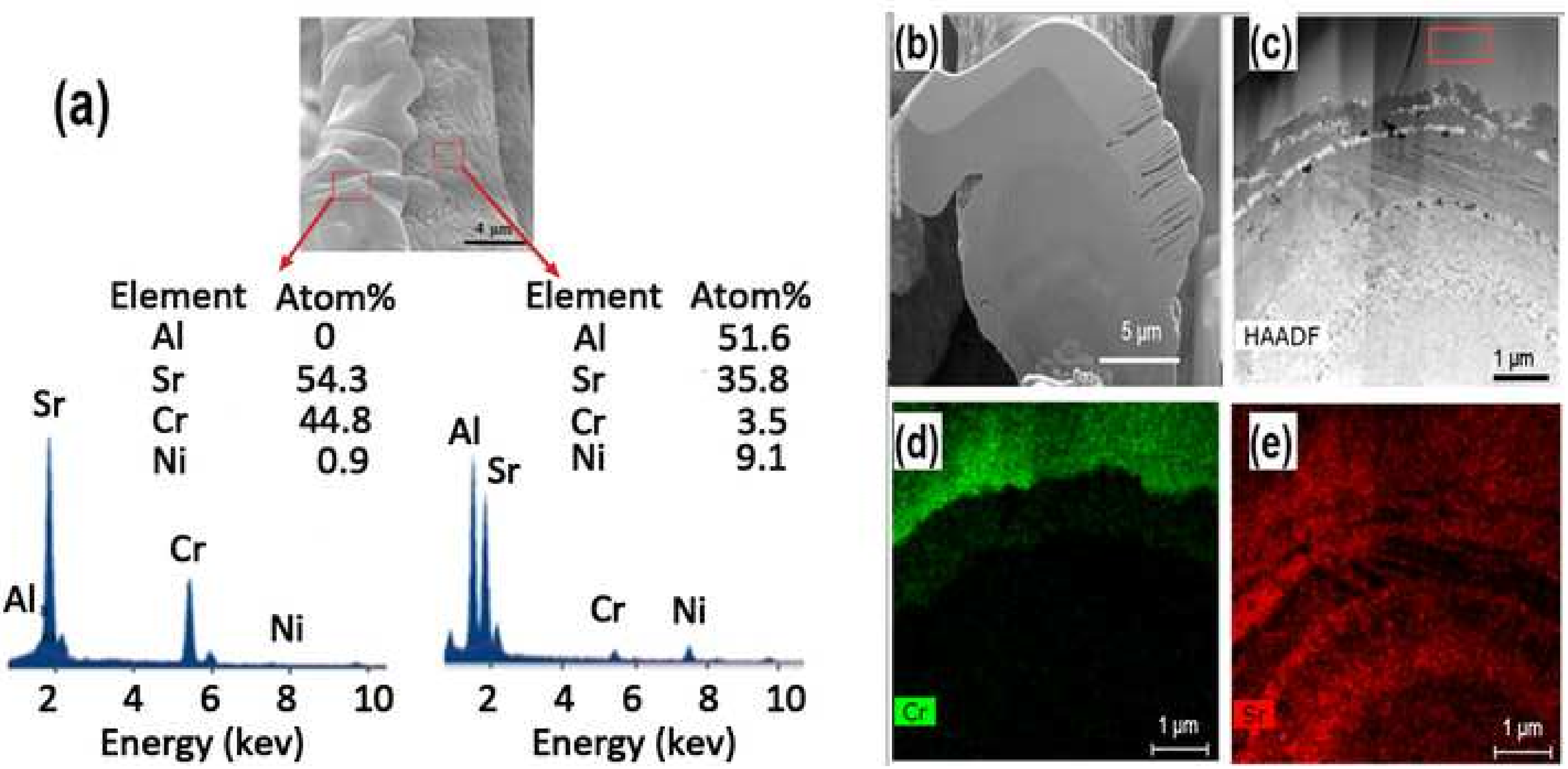

$Sr(NO_3)_2$
$Ni(NO_3)_2.6H_2O$
DI water
$NH_4OH$
rpm
C
Dry and Calcination
Getter Powder
DI water
Aqueous Slurry
substrate
Dip-coating
Dry and heat treatment
SNO getter


(a)
Furnace
Quartz tube
Air
MFC
Elbow
Condenser
Water
$Cr_2O_3$ pellet
Getter
Chiller
Vent
Wash bottles
(b)
10
8
6
4
2
0
Cr Wt. %
EDS Elemental Analyses
0
2000
4000
6000
8000
10000
Channel length from inlet (μm)


(A)
Cr-containing cathode air
LSM
YSZ
Pt
Biologic VMP3
Anode Air
(B)
c: in presence of Cr and getter
a: in absence of Cr and getter
b: in presence of Cr, no getter
I (A/cm²)
0
0.5
1.0
0
20
40
60
80
100
Time (h)
(C)
a
b
c
d
a 50 sccm
b 100 sccm
c 200 sccm
d 500 sccm
I (A/cm²)
0
0.5
1.0
0
20
40
60
80
100
Time (h)


(a)
Air flow
Evaporation of Cr vapor
Stainless steel alloy
$Cr_2O_3 + 2H_2O(g) + 3/2O_2(g) = 2CrO_2(OH)_2(g)$
$Cr_2O_3 + H_2O(g) + O_2(g) = 2CrO_2(OH)(g)$
$Cr_2O_3 + 3/2O_2(g) = 2CrO_3(g)$
(b)
-Im Z (Ω.cm²)
Re Z (Ω.cm²)
1.2
0.8
0.4
0.0
0
1
2
3
4
5
0 h
5 h
20 h
60 h
100 h
(c1)
200 nm
(c2)
2 µm



| Element | Atom% |
|---|---|
| Al | 0 |
| Sr | 54.3 |
| Cr | 44.8 |
| Ni | 0.9 |

| Element | Atom% |
|---|---|
| Al | 51.6 |
| Sr | 35.8 |
| Cr | 3.5 |
| Ni | 9.1 |

Table 1. SEM-EDS results of the posttest cell b cathode surface and interface

| | Cathode cell b |
|---|---|
| Cr atom% at surface | 2.5 ± 0.5 |
| Cr atom% at LSM/YSZ interface | 11.2 ± 2 |

| Item Number | Name of Material/ Equipment | Company | Catalog Number | Comments/Description |
|---|---|---|---|---|
| 1 | **$Sr(NO_3)_2$** | Sigma-Aldrich | 243426 | Getter precursor material |
| 2 | **$Ni(NO_3)_2$-$6H_2O$** | Alfa Aesar | A15540 | Getter precursor material |
| 3 | **$NH_4OH$** | Alfa Aesar | L13168 | Getter precursor material |
| 4 | **Pt ink** | ESL ElectroScience | 5051 | Current collector paste |
| 5 | **Pt wire** | Alfa Aesar | 10288 | Current collector wire |
| 6 | **Pt gause** | Alfa Aesar | 40935 | Current collector |
| 7 | **$Cr_2O_3$ powder** | Alfa Aesar | 12286 | Chromium source |
| 8 | **Nitric acid ($HNO_3$)** | Sigma-Aldrich | 438073 | Chromium extraction |
| 9 | **Potassium permanganate ($KMnO_4$)** | Alfa Aesar | A12170 | Chromium extraction |
| 10 | **LSM paste** | Fuelcellmaterials | 18007 | Cathode |
| 11 | **YSZ electrolyte** | Fuelcellmaterials | 211102 | Electrolyte |
| 12 | **Alumina fiber board** | Zircar | GJ0014 | Getter substrate |
| 13 | **Ceramabond paste** | AREMCO | 552-VFG | For cell sealing |
| 14 | **ICP-MS (7700s)** | Agilent | NA | For Cr analysis |
| 15 | **Potentiostat (VMP3)** | Biologic | NA | For EIS/I-t measurement |
| 16 | **FIB (Helios Nanolab 460F1)** | FEI | NA | For Nano-sample preparation |
| 17 | **TEM (Talos F200X S/TEM)** | FEI | NA | For composition analysis |

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Ashish Aphale, Junsung Hong, Boxun Hu*and Prabhakar Singh

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