Designing Internal Surfaces of Porous Electrodes in Solid Oxide Electrolysis Cells for Highly Efficient and Durable Hydrogen Production

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DOE Award – FE-0032112
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**HIGHLIGHTS**

- Exceptional Stability of Nano ALD Layer in SOEC Operated @ 850°C.

- ALD coated cell: H₂ Production @ >2.5A/cm²; 1.2V, superior stability for the first 1000 h; H₂ Production @ >~2.5A/cm² exceed DOE 2025 electrolysis goal of 1.5A/cm².

- Conformal ALD layer is **Cr tolerant** mixed conductor, further increasing the SOEC durability.
OVERVIEW

Project Background & Challenges

Solid Oxide Electrolysis Cells (SOEC) and Degradation

- Fuel electrode: Ni migration
- Oxygen electrode: LSCF/SDC decomposition, Sr surface segregation, Cr contamination
- Oxygen electrode: LSM/YSZ delamination, Cr contamination

Project Scope & Activities

- Project Objective
- Technical Approach
- Project Structure & Success Criteria

Current Results

On-Demand Design of ALD Layer Structure & Chemistry for Inherently Functional Commercial Cells

- Commercial cells with LSCF/SDC: ALD Coating LSCF/SDC Induced High Current Density
- Commercial cells with LSM/YSZ: Designing Internal Surface of LSM/YSZ by ALD Coating
  - ALD Layer of Conformal YSZ Nanoionics: Exceptional Stability in SOEC Operated @ 850°C
  - ALD Layer of Mixed Conductor:
    ✓ SOFC: Increase of cell peak power density up to 250%
    ✓ SOEC: High H₂ production rate @ >2.5A/cm² 850°C, superior stability >1000h
    ✓ Cr tolerant ALD layer is tunable from discrete particles to conformal layer
- Direct Impact of High Hydrogen Production Rate on SOEC Commercialization.
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Solid Oxide Cells, SOEC Electrolyzing water to produce hydrogen

Solid Oxide Fuel Cells-SOFC:
- Oxidizing a fuel to produce electricity.
- High efficiency, fuel flexibility, low emissions.

Solid Oxide Electrolysis Cells- SOEC:
- Electrolyzing water to produce hydrogen and oxygen gases.
- Produce pure green hydrogen.

SOEC systems are practically adapting the well-developed SOFC systems to shorten the development of SOEC devices.

Operation of the SOEC stacks at higher current densities over 0.75A/cm² and a low degradation rate could enable cost-competitive production of synthetic hydrocarbon and hydrogen.

Increasing hydrogen productivity >0.75A/cm² being pursued in Europe

Cell-level. (A) Current-voltage curves for cells fabricated in 2006 and 2020 with data from at 750°C, measured in H₂O/H₂ = 1 or CO₂/CO = 1. (B) Durability test of H₂O electrolysis at 1 A/cm² on a cell fabricated in 2005 measured at 850°C and a cell fabricated in 2015 measured at 800°C. All cells were supported by a Ni-YSZ electrode and had an active area of 16 cm².

- Extensive (> 9000 hours) long term SOEC testing has been performed in Europe.
- Significant degradation during initial operation (first ~ 500 hours), regardless of temperature and cells.
- Degradation is severe especially at the higher production rate with the current density > 1A/cm²

- Hauch A. Recent advances in solid oxide cell technology for electrolysis. Science. 2020;370(6513)
- Electricity consumption < 40 kWh/kg
- Production loss rate < 1.9%/1000h
- Availability >95%
- Reversible FC efficiency 54%
- Current density 1.25A/cm²
- Steam conversion rate > 85%


Chen M, Tong X, Ovitar S. Lessons Learned from Operating a Solid Oxide Electrolysis Cell at 1.25 A/cm² for One Year. Ecs Transactions. 2021;103(1):475.
SOEC usually presents more severe degradation than SOFC.

SOEC Degradation of is strictly dependent on the electrolysis operating conditions.

SOEC degradation is rooted in electrolyte, both fuel and oxygen electrodes.

SOEC degradation varies based on the chemistry of the electrodes.

- Ni/YSZ fuel electrode is the primary source of degradation for most long-term SOEC tests.

Significant Ni migration upon current load, operation for 9000 hours, 800C.


Alternative Ni-free ceramic oxide-based hydrogen electrodes generally have low electrical conductivity which can cause a significant increase in the resistance of the cell. Accordingly, the well-developed Ni/YSZ currently is still the best-performing fuel electrode for SOEC.
SOECs present more severe degradation than SOFCs, Oxygen electrode

**LSM/YSZ Oxygen electrode**, severe delamination at the electrolyte and electrode interface

- Delamination, due to lack of ionic conductivity in LSM, could happen ~48 hours after SOEC operation.
- Cr contamination also cause the degradation.

LSCF/SDC oxygen electrode, (1). LSCF decomposition, loss of the electrocatalytic activity, Sr surface segregation. (2). Cr vapor contamination from the interconnect, and their reaction with Sr.

SOEC degradation is taking place on both the fuel and oxygen electrodes.
- Ni/YSZ, LSM/YSZ, LSCF/SDC degradation due to different nanostructure/chemistry reasons.

This project:
- Improving of SOEC performance through modifying internal surface of porous electrodes.
- Atomic layer deposition of porous Ni/YSZ, LSM/YSZ, LSCF/SDC (separately) of commercial SOCs.
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**Objective** of this research is to achieve highly efficient and durable hydrogen production.

**Approach:** Decrease electrode resistance, increase hydrogen production rate, increase durability, by ALD coating.

- The commercial cells adapted for this study consist of the Ni/YSZ fuel electrode and La$_x$Sr$_{1-x}$Mn$_y$O$_3$-(LSM)/YSZ and La$_x$Sr$_{1-x}$Co$_y$Fe$_{1-y}$O$_3$ (LSCF)/Sm$_2$O$_3$ doped CeO$_2$ (SDC) oxygen electrodes.
- Highly active and robust nano-scale coating layers will be developed using the ALD and implanted to the internal surface of the porous electrode of as-fabricated commercial cells.
- For both the fuel and oxygen electrodes, innovative conformal surface consisting of nanocrystalline ionic conducting materials will be incorporated into the ALD coating layer to provide structure protection for increased durability towards both the intrinsic and extrinsic degradation.
**SCOPE OF WORK & common approaches for tasks.**

Commercial Cells: We will work on state-of-the-art commercial cells provided by different manufacturers that are being developed and matured worldwide during the past two decades.

- **ALD processing:** ALD coating using a commercial ALD system: Computer-controlled automatic switching of the precursor with a one-step processing. The ALD processing procedure will be developed to control the chemistry and the thickness of the ALD layer.

- **SOEC hydrogen production evaluation:** Lab-scale cell testing of the commercial button and planar cells: The cell testing will be performed on baseline and surface-modified cells. The cells will be tested using the state-of-the-art test stations at the PI’s lab. Testing conditions such as operation temperature, operation current density, operation voltage and durability, will be adjusted at the industrially relevant operating conditions.

- **Nanostructure evaluation:** ALD layer nanostructure evaluation of as-deposited cells and ALD layer nanostructure evaluation of cells after electrochemical testing.

**Success Criteria.**

The proposed work will accomplish the validation of concepts at a laboratory scale by direct comparison of the electrolysis performance of the as-received commercial baseline cells and the commercial cells subjected to subsequent to ALD coating developed through this project.

All lab-scale cell testing will be performed at the industry-relevant operation conditions as detailed below:

- Cells for testing will be state-of-the-art commercial cells (SOFCs), with excellent performance repeatability.

- Long-term stability tests will be carried out over 500 hours of continuous operations. The desired operation current density is over 0.75A/cm².

**Very challenging to achieve for the commercial cells.**

For instance, baseline cells with LSM/YSZ experience delamination under high current even after ~50 hours operation.
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On-Demand Design of Nanostructure & Function of ALD Layer

- Single layered nanoionics
- Discrete core-shelled catalysts
- Multilayered nano-composite

- Multilayered nano-composite
- Porous composite having pores filled with nano catalyst

- ALD layer could be single layered, either conformal continuous layer or discrete particles.
- ALD layer could be heterogenous composite, layered or porous filled with nano catalysts.
ALD Coating of Elements We Work On

- ALD layer nanostructure and chemistry could be designed on demand, based on the electrode backbone structure & chemistry.
- ALD coating as-made inherently functional fuel cells, (1). increase cell power density to 300%, (2). mitigate intrinsic degradation caused by cation segregation, (3). mitigate the Cr contamination.

The most recognized provider of ALD equipment for R&D & Industry.
Overview

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    - Fuel electrode: Ni migration
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SOEC: ALD Coating LSCF/SDC of Commercial Cell Resulted in Hydrogen Production Rate > 1A/cm²

Experimental Conditions:
- Commercial Cell with LSCF/SDC oxygen electrode and Ni/YSZ fuel electrode.
- Ni/YSZ electrode is not ALD coated.
- ALD Coating on the LSCF/SDC electrode only.

- ALD coating on LSCF/SDC resulted in high current density of >1.1A/cm²; @ 750°C; 2.5A/cm²; @ 850°C.
- ALD coating of LSM/YSZ of commercial cells resulted in better SOEC performance than that of ALD-coated LSCF/SDC cells.
- ALD coating of LSM/YSZ of commercial cell, mitigate the electrode delamination, achieved >2.5A/cm² after > 1000 hours SOEC.
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Designing Internal Surfaces of Porous LSM/YSZ Electrode

- ALD layer thickness ~ 10 nm
- ALD YSZ ionic conductor
- ALD layer thickness ~ 15 nm
- ALD Cr Tolerant Mixed Conducting Oxide
- ALD layer thickness ~ 70 nm

- Internal surface of porous electrode of as fabricated commercial SOC offers the design space for further tuning the chemistry & structure of the electrode.

- ALD layer creating new sites for facilitating the electrochemical reactions for increasing SOEC hydrogen production rate, and cell durability.

- ALD layer protective coating for increasing cell tolerance of contaminants such as Cr.
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SOFC: ALD Coating YSZ Nanoionics (~ 10 nm thick)

<table>
<thead>
<tr>
<th>ALD coated cell</th>
<th>Baseline cell</th>
<th>Enhancement</th>
</tr>
</thead>
<tbody>
<tr>
<td>Operation</td>
<td>Peak W/cm²</td>
<td></td>
</tr>
<tr>
<td>0 hr 750°C</td>
<td>0.734</td>
<td></td>
</tr>
<tr>
<td>24 hr 750°C</td>
<td>1.264</td>
<td></td>
</tr>
<tr>
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<td>1.264</td>
<td></td>
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Experimental Conditions:
- Commercial Cell with LSM/YSZ and Ni/YSZ.
- Ni/YSZ electrode is not ALD coated.
- ALD Coating on the LSM/YSZ electrode only.

ALD YSZ nanoionics on LSM/YSZ of commercial SOFC
- ALD coating achieves a 163 % peak power increase at 750°C
- ALD coated reduced both the ohmic and polarization resistance of the entire cell.
Baseline SOEC with LSM/YSZ fast degradation after 26 hours at 850°C

SOEC at 850°C, ALD coating of LSM/YSZ resulted in:

- Reduced the cell resistance. Lower the SOEC operation voltage. **High SOEC current of >2A/cm²**
- Mitigate the cell delamination, increase the cell durability.
- ALD nano-YSZ, Exceptional stability. After 850 °C for 150 hours, ALD nano-YSZ layer remains to be conformal ~ 10 nm in thickness without any agglomeration.
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      ◦ SOEC: High $H_2$ production rate @ $>2.5A/cm^2$ 850°C, superior stability $>1000h$
      ◦ Cr tolerant ALD layer is tunable from discrete particles to conformal layer
  ◦ Direct Impact of High Hydrogen Production Rate on SOEC Commercialization.
Designing Internal Surfaces of Porous LSM/YSZ Electrode

- **Baseline**
  - ALD layer thickness ~ 10 nm
  - ALD YSZ ionic conductor

- **ALD Coated**
  - ALD layer thickness ~ 15 nm
  - ALD Cr Tolerant Mixed Conducting Oxide

- **Internal surface of porous electrode of as fabricated commercial SOC offers the design space for further tuning the chemistry & structure of the electrode.**

- **ALD layer creating new sites for facilitating the electrochemical reactions increasing increase SOEC hydrogen production rate, and cell durability.**

- **ALD layer protective coating for increasing cell tolerance of contaminants such as Cr.**
ALD layer: Cr-Tolerant Mixed Conductor Oxide

SOFC: Coating LSM/YSZ, Increase Cell Peak Power Density Up To >250%

![Graphs showing voltage and power density vs current density at different temperatures (700°C, 750°C, 800°C, 850°C).]

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<tr>
<td>Operation</td>
<td>Peak W/cm²</td>
<td></td>
</tr>
<tr>
<td>0 hr 700°C</td>
<td>0.99</td>
<td></td>
</tr>
<tr>
<td>0 hr 750°C</td>
<td>1.65</td>
<td></td>
</tr>
<tr>
<td>0 hr 800°C</td>
<td>1.90</td>
<td></td>
</tr>
<tr>
<td>0 hr 850°C</td>
<td>2.14</td>
<td></td>
</tr>
<tr>
<td>7 hr 700°C</td>
<td>0.40</td>
<td>248%</td>
</tr>
<tr>
<td>0 hr 750°C</td>
<td>0.63</td>
<td>264%</td>
</tr>
<tr>
<td>0 hr 800°C</td>
<td>0.97</td>
<td>196%</td>
</tr>
<tr>
<td>1 hr 850°C</td>
<td>1.23</td>
<td>174%</td>
</tr>
</tbody>
</table>

With same power output requirement, ALD-coated cell could operate at much lower temperature (100°C lower):

- ALD coating impact is more significant at lower temperature: 248% at 700°C, remains 174% at 850°C.
- ALD coated cell at 700°C outperforms baseline at 800°C.
- ALD coated cell operated at 750°C outperforms baseline at 850°C.
Both ohmic resistance and polarization resistance decreased at temperature of 700-850°C.

- $R_s$: 14%, 28%, 29%, and 30% reduction on the $R_s$ values for the ALD coated cell for the temperature range of 700-850 °C, with a $R_s$ value of 0.0351 Ωcm$^2$ at 850°C compared to 0.0499 Ωcm$^2$ for the LSM baseline.
- $R_p$: 65%, 61%, 42%, and 32% reduction on the $R_p$ values for the ALD coated cell for the temperature range of 700-850 °C, with a $R_p$ value of 0.1637 Ωcm$^2$ at 850°C compared to 0.2390 Ωcm$^2$ for the LSM baseline. While more notorious reduction of 0.2686 Ωcm$^2$ at 70°C compared to 0.7711 Ωcm$^2$ for the LSM baseline.
SOEC: ALD coating induced higher current, lower voltage

Fuel Cell Mode
Electrolysis Mode; @\~3A/cm²

Ni/YSZ-YSZ-LSM/SSZ cell, commercial button cell, **direct switch from SOFC to SOEC**.

**1.4V after 26 hr @2.2A/cm²**
**1.212V after 159 hr @2.9A/cm²**
**1.211V after 493 hr @2.8A/cm²**
**1.202V after 1030 hr @2.65A/cm²**

<table>
<thead>
<tr>
<th>Operation</th>
<th>Rs (Ω cm²)</th>
<th>Rt (Ω cm²)</th>
<th>Rp (Ω cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>500 h, Coated</td>
<td>0.0247</td>
<td>0.0901</td>
<td>0.0654</td>
</tr>
<tr>
<td>1050 h, Coated</td>
<td>0.0354</td>
<td>0.1087</td>
<td>0.0733</td>
</tr>
<tr>
<td>0 h Baseline</td>
<td>0.0359</td>
<td>0.1332</td>
<td>0.0972</td>
</tr>
</tbody>
</table>

- ALD coated cell process significant lower resistance than that of the baseline after 1000 hours operation.
- ALD coated cell increase in resistance, especially Rs, probably due to degradation from electrolyte and Ni/YSZ.
- ALD coating of LSM/YSZ alone mitigate the electrode delamination, resulted high current and superior stability for the first 1000 hours SOEC operation.

Baseline delaminated after 26 hours operation, ALD coated cell appears stable for the first 1000 hours. ALD coated cell: a high current density of >2.5 A/cm², with low voltage of 1.2V after 1000 hours operation.
SOEC: Record high SOEC performance achieved by ALD coating

- High hydrogen production rate over 2.5 A/cm², Highest reported water electrolysis performance to date for SOEC with LSM or LSCF-based electrodes.
- Low electricity consumption, operational voltage of ~ 1.2 V at 850°C.
- Superior stability for the first 1000 hours SOEC with no sign of delamination.

**Electrolyzer Stack Goals by 2025**

<table>
<thead>
<tr>
<th></th>
<th>LTE PEM</th>
<th>HTE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Capital Cost</td>
<td>$100/kW</td>
<td>$100/kW</td>
</tr>
<tr>
<td>Elect. Efficiency (LHV)</td>
<td>70% at 3 A/cm²</td>
<td>98% at 1.5 A/cm²</td>
</tr>
<tr>
<td>Lifetime</td>
<td>80,000 hr</td>
<td>60,000 hr</td>
</tr>
</tbody>
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H₂ production from Present ALD coated cells at 2.5 A/cm² @ 1.2V, exceed DOE 2025 program goal of 1.5A/cm²

In terms of Hydrogen production, DOE 2025 program goal for HTE stack is 1.5A/cm²

ALD layer: Cr-Tolerant Mixed Conductor Oxide
SOEC: High H₂ Production Rate, Exceptional Stability

- Stable SOEC operation for the first 1000 hours.
- Exceptional Stability of nanostructure of ALD layer:
- ALD layer nano-grains, remained to be ~ 20 nm after 1000 hours SOEC @ 850C.
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Baseline

ALD Coated

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ALD YSZ ionic conductor

ALD layer thickness ~ 15 nm

ALD layer thickness ~ 70 nm
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Impact of High Hydrogen Production Rate on Commercialization.

- Commercial and state-of-the-art cells from different manufactures is \( \approx 1 \text{ A/cm}^2 \) at \( \approx 1.3 \text{V} \)
- ALD coated commercial cells (with LSM/YSZ electrode), achieved:
  - \( \approx 2.5\text{A/cm}^2 \) & \( \approx 1.2\text{V} \) is 2 times the Hydrogen Production Rate
  - \( \approx 2.5\text{A/cm}^2 \) & \( \approx 1.2\text{V} \) could achieve an estimated **two-fold reduction** in SOEC stack and Balance of Plant (BOP) cost and footprint.

**Brief technoeconomic analysis**

For a state-of-the-art commercial SOFC system and a stack (or portion of a stack) able to generate **1.5 kW** of electricity in **SOFC mode**, when operated reversely in the electrolysis mode:

- If the electrolysis current density is **2.5A/cm²**, the 1.5 kW stack is estimated to generate \( \approx 15 \text{ kg of green hydrogen per day} \) (or \( \approx 5,200 \text{ kg per year} \)).
- If the hydrogen market price is \( \approx \$5/\text{kg} \), this **1.5 kW SOFC stack** is estimated to generate revenue on the order of \( \approx \$25,000/\text{yr} \) when operated in electrolysis mode to produce green hydrogen.
ALD coating porous electrode increase hydrogen production rate and cell durability:

- Double or even triple the hydrogen production rate (Kg/h) from that of the baseline cell by decreasing cell resistance & increasing current density,
- Increase SOEC durability and significantly decrease cell degradation.

Scale-up ready:
The Beneq P1500 for large scale industry processing

- Can accommodate parts up to 1300 × 2400 mm (1.3 m x 2.4 m) in size,
- Enables the deposition of high-quality, functional optical coatings on wide area mirrors or lenses; Possible substrates include: Generation display glass; Photovoltaic glass sheets; Astronomical mirrors, Semiconductor chamber lids, liners & showerheads; Printed circuit boards
- The commercial ALD systems are ready for processing multiple SOC stacks simultaneously.
SUMMARY

- ALD coating of LSCF/SDC electrode of commercial cell result in SOEC of high current of 1.1A-2.5A @ 750°C.
- As-fabricated commercial SOFC with LSM/YSZ air electrode experienced degradation within first 50 hours.
- ALD coating of the LSM/YSZ oxygen electrode mitigate the cell degradation, lower the SOEC operation voltage and increase the hydrogen production rate.

  - **ALD Coating of YSZ ionic conductor**
    - Significantly increase SOFC peak power density to 160%, reduction both Rs and Rp.
    - Increase of SOEC current density for higher hydrogen production rate.
    - Increase SOEC durability. Extraordinary structure stability ALD nano-YSZ after SOEC @850°C.

  - **ALD coating mixed conducting metal oxide, discrete nano-grains of 15 nm**
    - ALD coating significantly increase the SOFC peak power density by up to 250%.
    - ALD coated SOEC is with high current density of >2.5A/cm2, low voltage of 1.2V.
    - ALD coated SOEC is with superior stability, for the first 1000 hours.

  - **ALD coating mixed conducting metal oxide, conformal coating, ~ 70 nm thick.**
    - ALD coating significantly increase the SOFC peak power density by up to 260%.
    - Conformal ALD coating layer is Cr-resistant, could further increase cell durability.

ALD coating of as-fabricated commercial SOFC for their SOEC application:

- Achieved the record high performance of >2.5A/cm², stable SOEC for the first 1000 hours.
- **ALD coated cell is with high hydrogen production rate 2.5A/cm², exceed the DOE 2025 target of 1.5A/cm².**
- ALD coating of commercial cells: Two granted patents, Four pending patents.
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