Production of Surface Functionalized Powders For Solid Oxide Fuel Cell Cathodes

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Introduction:
Recent works have shown the benefits of using surface functionalized powders for fabrication of solid oxide fuel cell (SOFC) cathodes. With this approach, the best characteristics of two different materials can be fully optimized and exploited. The present state of the art for surface functionalization uses an aqueous process to infiltrate the cathode after sintering. Multiple liquid infiltration and annealing cycles are required, which are both time consuming and expensive to implement in commercial production.

This SBIR project is developing an alternative approach for production of surface functionalized cathode powders prior to sintering. The resulting surface functionalized powders represent a "drop-in" replacement for presently available cathode powders, and can be seamlessly integrated into industrial SOFC manufacturing.

In Phase I, Structured Materials Industries, Inc. (SMI) and FuelCell Energy, Inc. (FCE) demonstrated technical feasibility of this approach, as detailed in this poster presentation. In Phase II, the SBIR team will refine and optimize the process and the surface functionalized cathode materials. We will demonstrate improved efficiency and long term stability in actual fuel cell operation. We will also demonstrate the potential for scale-up to low-cost, high volume production. Ultimately, SMI will implement the technology by producing and selling surface functionalized cathode powders, as well as commercial processing equipment for their production.
The SMI surface functionalization process produces uniform coatings on powders. A wide range of coating compositions can be produced. A wide range of base powder materials can be processed. In this SBIR Phase I and Phase II project, we used a commercial \( \text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-\delta} \) (LSCF) powder with 0.7 \( \mu \text{m} \) average particle size, as shown in the image on the left. The image on the right shows a typical powder after surface functionalization by the SMI process.

During Phase I, SMI produced several samples of surface functionalized LSCF powder. The typical batch size was 20 to 25 grams in Phase I. This can easily be scaled to larger batch sizes in the future. The primary variable investigated in Phase I was the total amount of surface material applied, expressed in moles per kg of LSCF base powder.

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<th>Sample ID</th>
<th>Moles / kg</th>
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<td>11-06</td>
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Electrochemical characterization of the surface functionalized powders was performed at FuelCell Energy Inc. Screen printing slurries were prepared with each of the processed powders. These pastes were used to prepare LSCF electrodes, symmetrically screen printed onto scandia stabilized zirconia electrolyte disks (25 mm diameter and 150 µm thickness) with a gadolinium doped ceria barrier layer (2 µm thickness). A schematic diagram of the symmetrical test cell is shown in the Figure above. A similar symmetrical cell was also prepared using uncoated LSCF powder, as a baseline sample. The cathode coated discs were sintered at 1000 C for 3 hours. Silver paste was applied, and used as the current collector. Electrochemical Impedance Spectroscopy (ESI) was done to characterize the electrochemical performance of the symmetrical cells under open circuit potential. Four probe tests were performed at 800 C, 700 C and 600 C in air.

The Figure to the right shows example results of electrochemical impedance spectroscopy at 700 C, for all samples of surface functionalized cathode powders processed by SMI in Phase I. The total polarization resistance is the difference between the high-frequency and low frequency intercepts with the real axis. For the sake of simplicity, the Ohmic resistance contribution has been subtracted.
The above Figures show total polarization resistance results for all samples tested during Phase I; at 600 C and 800 C. In all tests, the surface functionalized powders outperform the uncoated LSFC powder, as indicated by lower polarization resistance. There appears to be an optimum surface loading at around 0.18 moles/kg.
The upper Figure shows total polarization resistance results for all samples tested during Phase I at 700 C. The lower Figure shows results of testing a symmetrical cell prepared with the optimum surface loading composition, under a constant current of 100 mA at 700 C. The surface functionalized powder outperforms the uncoated LSFC powder, as indicated by lower polarization resistance and lower degradation rate.
Summary and Conclusions for Phase I:

• The SMI surface functionalization process can produce a wide range of surface coatings on powders.

• The SMI surface functionalization process can be applied to a wide range of base powders.

• Phase I of this SBIR project produced samples of surface functionalized LSCF powders, with surface loading ranging from 0.041 to 0.392 moles/kg of LSCF base powder.

• The surface functionalized LSCF powders were fabricated into cathode structures, using standard screen-print and sintering processes.

• Evaluation of the surface functionalized cathodes was done at FuelCell Energy, Inc., by electrochemical impedance spectroscopy; at 600 C, 700 C and 800 C.

• In all cases, the surface functionalized powders outperformed uncoated LSCF, as measured by electrochemical impedance spectroscopy.

• There appears to be an optimum surface loading amount at around 0.18 moles/kg of LSCF base powder.

• Preliminary results of cathode testing as a function of time show the surface functionalized LSCF powder to result in lower polarization and lower degradation rate than uncoated LSCF powder.

• The SMI surface functionalization process can be scaled to large production volume.

• A preliminary cost analysis showed the SMI surface functionalization process to have minimal impact on overall SOFC production costs.

• Technical feasibility of using the SMI surface functionalization process to enhance the performance and economic benefits of SOFC systems was demonstrated in Phase I.

• The SMI surface functionalized powders represent a "drop-in" replacement for presently available cathode powders, and can be seamlessly integrated into industrial SOFC manufacturing.
Objectives for Phase II:

- Optimize the SMI process in terms of highest throughput and minimum cost for production of surface functionalized powders.
- Demonstrate the effects of composition for the surface functionalized material and identify the optimum composition and morphology for the resulting cathode powders.
- Investigate the use of final stabilization layers such as ZrO$_2$, deposited using the SMI surface functionalization process.
- Identify the mechanisms of SOFC performance enhancement for the surface functionalized cathode materials.
- Demonstrate the long term stability of the performance enhancement in SOFC operation.
- Demonstrate the potential for scale-up of the SMI surface functionalization process to high volume powder production.
- Demonstrate the pathway forward to implementation and commercialization in Phase III.

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