“New Cathode Materials for Intermediate Temperature Solid Oxide Fuel Cells”
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Allan J Jacobson
Center for Materials Chemistry
University of Houston
Houston, Texas 77204-5003

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Abstract

Operation of SOFCs at intermediate temperatures (500 – 800 °C) requires new combinations of electrolyte and electrode materials that will provide both rapid ion transport across the electrolyte and electrode - electrolyte interfaces and efficient electrocatalysis of the oxygen reduction and fuel oxidation reactions. This project concentrates on materials and issues associated with cathode performance that are known to become limiting factors as the operating temperature is reduced.

The specific objectives of the proposed research are to develop cathode materials that meet the electrode performance targets of 1.0 W/cm² at 0.7 V in combination with YSZ at 700 °C and with GDC, LSGM or bismuth oxide based electrolytes at 600 °C. The performance targets imply an area specific resistance of ~0.5 Ωcm² for the total cell. The research strategy is to investigate both established classes of materials and new candidates as cathodes, to determine fundamental performance parameters such as bulk diffusion, surface reactivity and interfacial transfer, and to couple these parameters to performance in single cell tests.

The initial choices for study are perovskite oxides based on Sr substituted LaFeO₃, where significant data in single cell tests exists at PNNL for cathodes on both YSZ and CSO/YSZ, and Ln₂NiO₄ compositions. A key component of the research strategy is to evaluate for each cathode material composition, the key performance parameters, including ionic and electronic conductivity, surface exchange rates, stability with respect to the specific electrolyte choice, and thermal expansion coefficients. Results on electrical conductivity relaxation measurements on additional compositions in the La₂NiO_{4+x} and Pr₂NiO_{4+x} series are presented in this report. Synthesis and characterization of the P1 compositions La_{0.8}Sr_{0.2}FeO_{3-x} and La_{0.7}Sr_{0.3}FeO_{3-x} is described.

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1. List of Graphical Materials

- Figure 1. Thermogravimetric analysis data for $\text{La}_x\text{Pr}_{2-x}\text{NiO}_{4+\delta}$ ($x = 1.9(a), 1.2(b)$ and $1.0(c)$).
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2. Introduction

The objectives of the project are to discover new oxide cathode materials that meet a performance target of 1.0 W/cm^2 at 0.7 V in combination with YSZ at 700°C and with CGO, LSGM electrolytes at 600°C . An ancillary objective of the project is to increase fundamental understanding of the intrinsic transport properties of mixed electronic ionic conducting oxides and oxide-oxide interfaces that can be used to accelerate further progress in the development of cost effective high performance solid oxide fuel cells. In Phase I, we are measuring the surface exchange rates, diffusion coefficients and interfacial transport for an initial set of perovskite related oxide materials. In Phase II we will synthesize and characterize new cathode materials and measure their kinetic parameters. The thermal and chemical compatibility with different electrolytes will be determined. Based on the results, a subset of the best materials will be selected for single cell tests. The phase III objectives are to evaluate the performance of the best materials identified in Phase I and II. The optimum electrode composition and microstructure will be determined and the longer term performance characteristics evaluated.

3. Executive Summary

The project began on October 1, 2003 and this is the fourth quarterly report. In the fourth period, we have prepared and characterized additional single phase samples of $\text{La}_x\text{Pr}_{2-x}\text{NiO}_{4+x}$ ($x = 0.5, 1.5$) for comparison with the end members $\text{La}_2\text{NiO}_{4+x}$ and $\text{Pr}_2\text{NiO}_{4+x}$.¹⁻⁹ We have measured the dc conductivities as a function of $p\text{O}_2$ in several oxygen partial pressures and confirmed the discontinuity that we reported last quarter. Thermogravimetric analysis measurements were completed and the oxygen ion diffusion coefficients and the surface exchange coefficients extracted for comparison with previous measurements. We have also synthesized two examples of P1 compositions and started to characterize the surface chemistry. We have continued to investigate synthesis conditions for $\text{LaFeO}_3 - \text{SrFeO}_{2.5}$ thin films. Two layer thin films of LaFeO_3 and $\text{SrFeO}_{2.5}$ have been prepared by PLD in amorphous form and their inter-diffusion investigated at different temperatures studied. The surface roughness problems have been eliminated but further work is necessary to find the optimum conditions (temperature and film thickness) to prepare single phase crystalline compositions.

4. Experimental

4.1 Characterization of K1 Compositions $\text{La}_x\text{Pr}_{2-x}\text{NiO}_{4+x}$ ($x = 1.9, 1.2, 1.0$).

The syntheses and compositional characterization of additional K1 compositions in the series $\text{Ln}_2\text{NiO}_{4+x}$, $\text{Ln} = \text{La, Pr}$, have been prepared using the synthesis method described in the previous report. The phase purity and lattice parameters were determined by powder X-ray diffraction. Thermogravimetric analyses were performed to determine the equilibrium oxygen nonstoichiometry. Measurements of the relative values of δ in $\text{La}_x\text{Pr}_{2-x}\text{NiO}_{4+\delta}$ ($x=1.9, 1.2$ and 1.0) obtained from thermogravimetric analysis were placed on an absolute basis by determining the starting composition using iodometric titration. Figure 1 shows

the results for the three compositions as a function of temperature and pO_2 in the range 0.5% to 5%.

Figure 1. Thermogravimetric analysis data for $La_xPr_{2-x}NiO_{4+\delta}$ ($x=1.9$ (a), 1.2 (b) and 1.0 (c)).

4.2 DC conductivity Measurements of $La_xPr_{2-x}NiO_{4+\delta}$ ($x = 0.5, 1.5$)

Two additional compositions were prepared with $x = 0.5$ and 1.5 to verify the abrupt change of conductivity between $x = 1 \sim 1.2$. The new samples give results consistent with the previous data. The origin of this discontinuity is not yet known but may reflect a miscibility gap.

Figure 2. Comparison of the DC conductivities for $\text{La}_x\text{Pr}_{2-x}\text{NiO}_{4+\delta}$ at $1000/T = 2.0$.

4.3 Electrical Conductivity Relaxation Measurements

The results from the TGA measurements were used to convert the values of D_{chem} and k_{chem} reported previously using the relations $D_{\text{O}} = D_{\text{chem}} / \Gamma$ and $k_{\text{ex}} = k_{\text{chem}} / \Gamma$ where Γ is the thermodynamic factor. The results are shown in Figure 3.

Figure 3. Values of D_{O} (left) and k_{ex} (right) at 3 % pO_2 except 0.5 % for $\text{La}_{1.9}\text{Pr}_{0.1}\text{NiO}_{4+\delta}$ determined by electrical conductivity relaxation

The diffusion coefficients vary by about a factor of 2 with $\text{La}_{1.9}\text{Pr}_{0.1}\text{NiO}_{4+\delta}$ being the highest and $\text{La}_2\text{NiO}_{4+\delta}$ the lowest. The activation energies are similar within the experimental errors. In contrast, much more variation is found in the surface exchange velocities. Two compositions $\text{La}_{1.9}\text{Pr}_{0.1}\text{NiO}_{4+\delta}$ and $\text{La}_{1.0}\text{Pr}_{1.0}\text{NiO}_{4+\delta}$ were found to have much higher k values at low temperature and lower activation energies. The data may be

compared with literature results for pure La_2NiO_4 determined by isotope exchange and depth profiling as shown in Figure 4. The diffusion results obtained from the two techniques agree within a factor of 2 with the ECR results systematically higher. The values of the surface exchange coefficient vary by more than an order of magnitude with the ECR results falling between the two IEDP studies. This comparison and the differences in k values for the Pr substituted samples indicate that further work is needed to investigate the surface structure and composition of these phases.

Figure 4. Comparison of the D and k values for La_2NiO_4 measured by ECR and IEDP.

4.4 Synthesis of Perovskite Compositions P1, $\text{La}_{1-x}\text{Sr}_x\text{FeO}_3$, $x = 0.2, 0.3$

The glycine-nitrate and solid-state methods were used to synthesize $\text{La}_{0.7}\text{Sr}_{0.3}\text{FeO}_3$ and $\text{La}_{0.8}\text{Sr}_{0.2}\text{FeO}_3$ (LSFO). The LSFO compounds were then characterized with powder X-ray diffraction (XRD), microprobe, and density measurements. Pressed pellets of the four compounds were sintered for subsequent gas-solid interface reactions.

La_2O_3 (Aldrich 99.9%), SrCO_3 (Aldrich 99.9%), and Fe_2O_3 (Aldrich 99.9%) were used in the solid-state synthesis method. Iron metal (Aldrich (99.99%)) is used in place of Fe_2O_3 in the glycine-nitrate synthesis method. La_2O_3 was dried at 1000 °C, Fe, Fe_2O_3 , and SrCO_3 are dried at 125 °C. All reagents are dried for 16 h. Stoichiometric quantities are then used for synthesis.

After ignition in the glycine-nitrate process and grinding the compositions were heated at 1000 °C. The samples were then ground and reheated at 1200, 1250, and 1300 °C. Phase pure and homogeneous samples were obtained except $\text{La}_{0.8}\text{Sr}_{0.2}\text{FeO}_3$, prepared via the glycine-nitrate method. A representative X-ray powder pattern and backscattered electron image is shown in Figure 5. The stoichiometries determined by microprobe analysis and the densities measured by the Archimedes method are given in Tables 1 and 2. The analytical data $\text{La}_{0.8}\text{Sr}_{0.2}\text{FeO}_3$, prepared via the glycine-nitrate method show significant variations in the La and Sr compositions at different points on the sample indicating poor homogeneity. This sample will not be considered further.

Table 1 Microprobe analysis results for $\text{La}_{1-x}\text{Sr}_x\text{FeO}_3$, $x = 0.2$ or 0.3

$\text{La}_{0.8}\text{Sr}_{0.2}\text{FeO}_3$ glycine-nitrate							
	I	II	III	IV	V	average	range
La	0.89	0.81	0.84	0.88	0.80	0.85	0.09
Sr	0.11	0.19	0.16	0.12	0.20	0.15	0.09
Fe	1.12	1.05	1.02	1.12	1.08	1.08	0.10
$\text{La}_{0.8}\text{Sr}_{0.2}\text{FeO}_3$ solid-state							
La	0.79	0.80	0.80	0.79	0.79	0.79	0.02
Sr	0.21	0.20	0.20	0.21	0.21	0.21	0.02
Fe	1.06	1.08	1.09	1.07	1.08	1.08	0.02
$\text{La}_{0.7}\text{Sr}_{0.3}\text{FeO}_3$ glycine-nitrate							
La	0.71	0.71	0.70	0.71	0.70	0.71	0.01
Sr	0.29	0.29	0.30	0.29	0.30	0.29	0.01
Fe	1.09	1.09	1.06	1.10	1.07	1.08	0.03
$\text{La}_{0.7}\text{Sr}_{0.3}\text{FeO}_3$ solid-state							
La	0.71	0.69	0.69	0.69	0.70	0.70	0.02
Sr	0.29	0.31	0.31	0.31	0.30	0.30	0.02
Fe	1.10	1.06	1.07	1.07	1.08	1.08	0.04

Figure 5 XRD powder pattern and backscattered electron image of $\text{La}_{0.8}\text{Sr}_{0.2}\text{FeO}_3$, prepared via solid-state synthesis.

Table 2 Densities of $\text{La}_{1-x}\text{Sr}_x\text{FeO}_3$, $x = 0.2, 0.3$ samples

	$\text{La}_{0.8}\text{Sr}_{0.2}\text{FeO}_3$		$\text{La}_{0.7}\text{Sr}_{0.3}\text{FeO}_3$	
	glycine-nitrate	solid-state	glycine-nitrate	solid-state
d g/cm ³	5.80	6.11	5.96	5.93
% Theoretical	88.3	93.1	92.5	91.6

4.5 XPS Analysis and Reaction of $\text{La}_{0.7}\text{Sr}_{0.3}\text{FeO}_3$

The sintered pellet of $\text{La}_{0.7}\text{Sr}_{0.3}\text{FeO}_3$, synthesized by the solid-state method, was analyzed by X-ray photoelectron spectroscopy (XPS) to characterize the surface composition. XPS analysis was performed at both 70° and 15° take-off angles. The 15° angle is used to probe the near surface region while the 70° angle data give information at greater depth. Combined, the data at the two angles reveal which binding energies correspond to lattice oxygen or surface oxides. The results are shown in Figure 6. The higher binding energies from 530 to 533 eV correspond to surface oxides and the lower binding energy of 528.5 eV corresponds to lattice oxygen. The very large peak height at the higher binding energies indicates that the surface is highly contaminated with carbonates.

Figure 6. Photoelectron spectra of the as received $\text{La}_{0.7}\text{Sr}_{0.3}\text{FeO}_3$ sample prepared via the solid-state method

Figure 7. Photoelectron spectra of the cleaned $\text{La}_{0.7}\text{Sr}_{0.3}\text{FeO}_3$ pellet prepared via the solid-state method.

The sample was then transferred from the XPS chamber to an attached reaction chamber where it was heated in 100% O₂ at 350 °C for 1.5 h to remove surface contaminants. The sample was cooled to room temperature and transferred back to the XPS chamber for subsequent analysis. XPS data were measured at both angles and confirm that the surface carbonates have been removed (see Figure 6). Further analysis of the surface oxides at the higher binding energies that remain after the heat treatment will be the subject of future work.

5. Combinatorial Approach to Measurement of Transport Parameters.

Perovskite and perovskite - related oxides are noteworthy for their tolerance of a very wide range of compositions. This is an advantage in that variations in composition can be used to simultaneously optimize several important properties such as surface exchange rate, diffusion coefficient, thermal expansion, and substrate reactivity. The disadvantage is that the matrix of possible compositions is very large and serial evaluation of properties is time consuming. Consequently, we have decided to adopt a combinatorial approach whereby many compositions can be screened for one or more properties using a single specimen. As a first step, we plan to investigate compositions in the LaFeO₃ – SrFeO_{2.5} system which have been shown recently to be of interest as cathode materials for intermediate temperature operation. Eventually, we plan to expand the scope to include four different oxide components. We will use the two oxide component system to develop the technique and the processing conditions needed to prepare samples and the evaluation techniques. Samples will be prepared in the form of thin films by pulsed laser deposition (PLD) on single crystal yttria stabilized zirconia substrates.

To date, we have made a number of bilayer films as discussed in previous reports and studied the effects on annealing on inter-diffusion. The surface roughness problems have been eliminated but further work is necessary to find the optimum conditions (temperature and film thickness) to prepare single phase crystalline compositions. More details will be given in the next report.

6. Conclusions

Single phase samples of La_xPr_{2-x}NiO_{4+d} (x = 1.9, 1.5, 1.2, 1.0 and 0.5) for comparison with the end members La₂NiO_{4+x} and Pr₂NiO_{4+x} have been prepared and characterized. Dense samples were prepared for conductivity and conductivity relaxation measurements. Conductivity measurements at different pO₂ values were measured for the samples and show significant variations with x. Conductivity relaxation measurements were made with different pressure switches. The oxygen ion diffusion coefficients and the surface exchange coefficients were extracted using the measured thermodynamic factors and compared with previous IEDP measurements. We have completed the synthesis of the first P1 compositions.

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8 List of Acronyms and Abbreviations

CGO	Cerium Gadolinium Oxide
ECR	Electrical Conductivity Relaxation
EPMA	Electron Probe MicroAnalysis
GDC	Gadolinia Doped Ceria (see CGO)
LSGM	Lanthanum Strontium Magnesium Gallate
PLD	Pulsed Laser Deposition
TGA	Thermogravimetric Analysis

YSZ Yttria Stabilized Zirconia
IEDP Isotope Exchange and Depth Profiling

9. Milestones and Scope of Work

The project is divided into three phases that will overlap as shown in the timelines below

Months	1-3	4-6	7-9	10-12
Year1	Phase I			
Year 2	Phase II			
	Phase II			
Year 3	Phase III			
	Phase II			
	Phase III			

Phase I: The Phase I objectives are to complete the characterization of a set of perovskite materials for which performance data already exists at PNNL. We will then measure the relevant kinetic parameters. The comparison the real performance data with the fundamental kinetic parameters will be used to guide materials selection in Phase II.

Phase II: The Phase II objectives of the project are to synthesize and characterize new cathode materials and to measure their kinetic parameters. The thermal and chemical compatibility with different electrolytes will be determined. Based on these results, a subset of the best materials will be selected for single cell tests. Some additional compositions will be synthesized if indicated by the single cell test data.

Phase III: The phase three objectives are to evaluate the performance of the best materials identified in Phase I and II. The optimum electrode composition and microstructure will be determined and longer term performance characteristics evaluated.

Updated Milestones for Year 1

The updated milestones reflect two program adjustments since the original submission. The first is that we have started work on the K1 compositions ahead of the P1 compositions. The latter have been moved into the second half of the first year. The second is that in order to accelerate the thin film materials selection work we have decided to adopt a combinatorial approach which will permit the simultaneous synthesis of many compositions.

PHASE I

- Task 1.0 Four modified perovskite oxide compositions (**P1**) selected by UH and PNNL will be synthesized and characterized by X-ray diffraction (XRD) and electron microprobe analysis (EMPA) (Months 6-9) **completed**
- Task 2.0 The chemical compatibility with YSZ, CGO and LSGM electrolytes will be determined (Months 6-9) **in progress**
- Task 3.0 The temperature dependence of the dc conductivity and stoichiometry will be determined in air for the **P1** compositions. (Months 6-9) **in progress**
- Task 4.0 The diffusion coefficients and surface exchange rates will be measured by electrical conductivity relaxation (Months 6-12) **in progress**

- Task 5.1 Thin films of perovskite compositions will be synthesized by PLD using a combinatorial approach. (Months 3-9) **in progress**
- Task 5.2 Electrode-electrolyte interfaces will be characterized for thin films of cathode materials by AC impedance spectroscopy (Months 9-12)
- Task 6.0 Electrode-electrolyte interfaces will be characterized for thin films of cathode materials by IEDP (9-12)

PHASE II

- Task 1.0 Four modified perovskite oxide compositions (**P2**) will be synthesized and characterized by XRD and EMPA (Months 9-12)
- Task 1.1 Two A_2BO_4 compositions (**K1**) will be synthesized and characterized by XRD and EMPA. (Months 1-4) **completed**
- Task 1.2 Three additional A_2BO_4 compositions (**K2**) will be synthesized and characterized by XRD and EMPA. (Months 6-9) **completed**
- Task 2.0 The chemical compatibility with YSZ, CGO and LSGM electrolytes will be determined for **P2** (Month 12)
- Task 2.1 The chemical compatibility with YSZ, CGO and LSGM electrolytes will be determined for **K1** (Month 7) **in progress**
- Task 2.2 The chemical compatibility with YSZ, CGO and LSGM electrolytes will be determined for **K2** (Month 9)
- Task 3.0 The dc conductivity and stoichiometry will be determined in air for the **K1** compositions that have chemical compatibility. (Months 4-6) **completed**
- Task 4.0 The diffusion coefficients and surface exchange rates will be measured by electrical conductivity relaxation for the **K1** materials evaluated in Task 3 (Months 6-12). **completed**
- Task 4.1 K1 samples will be characterized in symmetric cells by AC impedance spectroscopy (Months 6-9) to confirm the measured values of k_{ex} . **in progress**
- Task 5.0 Electrode-electrolyte interfaces will be characterized for thin films of cathode materials selected from Task 4.0 by AC impedance spectroscopy (Months 10-33).
- Task 6.0 Electrode-electrolyte interfaces will be characterized for thin films of cathode materials selected from Task 4.0 by IEDP (Months 10-33)
- Task 7.0 Results from Tasks 4.0, 5.0, and 6.0 will be used as they become available to select cathode electrolyte combinations for single cell tests (Months 10-33).