DOE/MC/29224 -- 5094
(DE96000626)

Processing of $LaCrO_3$ for Solid Oxide Fuel Cell Applications

Topical Report
April 1994 - April 1995

July 1995

Work Performed Under Contract No.: DE-FG21-93MC29224

For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By
University of Missouri-Rolla
Rolla, Missouri

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By
University of Missouri-Rolla
B-47 McNutt Hall, UMR
Rolla, Missouri 65401

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CONTRACT INFORMATION

Contract Number	DE-FG21-93MC29224
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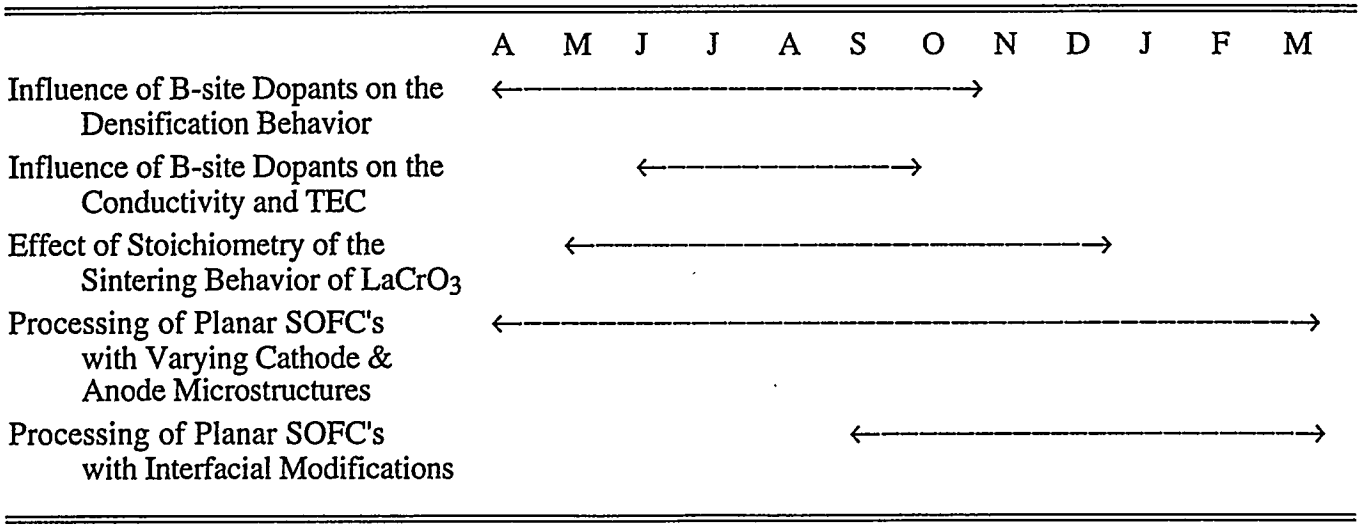
Contractor Project Manager	Wayne Huebner
Principal Investigators	Wayne Huebner Harlan U. Anderson
METC Project Manager	Bruce Harrington
Period of Performance	April 16, 1995 to April 15, 1996

Schedule and Milestones

FY95 Program Schedule



OBJECTIVES

The University of Missouri-Rolla is performing a 5 year research program with two primary objectives: 1) developing $LaCrO_3$-based interconnect powders which densify when in contact with anode and cathode materials for solid oxide fuel cells (SOFC), and 2) developing high performance cathodes, anodes and interfaces for use in planar SOFC's.

With regard to the processing and sintering of $LaCrO_3$, the specific objectives of this research program are to:

- Develop a non-liquid phase sintered $LaCrO_3$-based material sinterable in air.
- Improve and control the properties requisite of $LaCrO_3$ utilizing a B-site acceptor dopant
- Optimize and control the processing conditions associated with $LaCrO_3$.
- Incorporate materials developed in this program into planar cells and measure their performance.

With regard to developing high performance materials for use in planar SOFC's, the specific objectives of this research program over the last year have been to:

- Fabricate single cells with controlled microstructures (i.e. grain size and porosity of electrodes) for operation at 1000°C.
- Gain a better understanding of the mechanisms involved in improving cell performance via electrochemical and impedance techniques.
- Develop processing ↔ microstructure ↔ property relations of electrodes and their corresponding interfacial reactions.

BACKGROUND INFORMATION

One of the primary factors limiting the commercialization of SOFC's is the inability to fabricate $LaCrO_3$-based interconnects cheaply, and in contact with other SOFC components. The very properties which make $LaCrO_3$ a good interconnect material, also make it very difficult to sinter. Microstructural evolution ↔ property relationships of the $LaCrO_3$-based interconnect need to be understood. This was recently confirmed again at the SOFC IV conference held in Yokohama, Japan. At this meeting numerous research groups identified the cost of processing $LaCrO_3$ was prohibitive, as well as its tendency to change dimensions upon reduction. Indeed, the inability to co-sinter $LaCrO_3$-based compounds in air at T<1550°C with other SOFC components leads to unacceptable interfacial reactions, and resultant losses in efficiency. Many researchers have addressed this problem, yet progress has been hindered due to recognized difficulties in repeatable processing. Phase development, stoichiometry, particle size/distribution, agglomerate/ aggregate formation, volatility of components, and their dependence on dopant levels and sintering temperature, time and atmosphere are very difficult to systematically take into account.

The results of our work and other investigators[2-10] have lead us to the conclusion that the densification of $LaCrO_3$-based oxides is controlled by the ability for mass transport (sintering) to occur under conditions which minimize Cr oxide volatilization (Cr activity, a_{Cr}). We demonstrated that this could be accomplished by sintering at temperatures >1750°C and oxygen activities of about 10^{-10} atm[2] but clearly these sintering conditions are not compatible with the stability of other SOFC components. Another approach to reduce a_{Cr} is to lower the sintering temperature with liquid fluxes, which was initially pursued by Argonne National Labs[8]. However, the high mobility of the fluoride fluxes detrimentally affected the other cell components. More recent studies including our own have also shown that while transient liquid phase sintering may allow for lower temperature densification of the interconnect alone, the use of large amounts of A-site dopants (i.e. Ca,Sr) in amounts excess of stoichiometry create problems with second phase formation and limited mechanical stability in reducing atmospheres. Hence in some ways we are back to square one, and in this program we have re-focused our attention on densifying acceptor-doped $LaCrO_3$ without the addition of a liquid phase.

Materials research in the area of SOFC's is also driven by the recognition that processing and operating at lower temperatures would circumvent most of the reliability problems which are currently preventing these devices from achieving wide-scale commercialization. These considerations have directed interdisciplinary research thrusts in this field, namely:

- Alternate Materials
  - → higher conductivities at lower temperatures for all four SOFC components
  - → mixed-conducting cathodes
  - → greater phase stability at low pO_2's
- Processing
  - → novel synthesis techniques (for powders and thin films)
  - → controlled microstructure with desired properties
- Reliability Issues
  - → chemical, mechanical, and electrical stability under the temperature/time/atmosphere conditions of cofiring and operation

Hence studies on the optimization of components use of mixed conductors and thin film processing techniques[11-16], as well as studies on structure↔property relationships represent an enabling technology; what remains is to utilize this technology for the fabrication of planar SOFC's. Key to this next stage is the development of a porous anode substrate with the requisite microstructural, electrical and mechanical properties. While many researchers have investigated Ni:ZrO_2 cermet anodes[7], many issues related to microstructural evolution and stability, as well as the impact of Ni scale and connectivity on the electrical properties remain unresolved.

PROJECT DESCRIPTION

This report is divided into two primary sections:

#1 $LaCrO_3$ Sintering Studies

#2 SOFC Performance Studies

Results from these studies are presented in the following sections.

$LaCrO_3$ SINTERING STUDIES

Experimental Approach:

Powders of the composition $LaCr_{1-x}M_xO_3$ were prepared by a modified liquid mix process, in which ethylene glycol and citric acid are added to a stoichiometric mixture of carbonates and nitrates of the constituent ions in a 1:1:1 ratio. Starting materials were thermogravimetrically standardized to ± 0.1mg and x-rayed to ensure correct oxide composition. BET measurements indicate resulting powders have a specific surface area of ~5.0 m^2/g. Sintering studies were conducted with 1/2" pellets uniaxially pressed to 3ksi and isostatically pressed to 40ksi. Green densities were taken geometrically, while final densities were measured by the Archimedes technique with kerosene as the medium. Sintering studies were performed on an Orton 1600D dilatometer with 1/4" pellets uniaxially pressed to 0.2ksi and isostatically pressed to 40ksi. Crystallinity and phase purity were determined by x-ray on a Rigaku Miniflex system. Conductivity was determined with a 2-point d.c. apparatus with platinum electrodes over the temperature range of 25-1300°C.

RESULTS

A) Sintering

Recent studies in our laboratory have shown that compositions of $LaCr_{1-x}M_xO_3$ with $x<0.10$ exhibit enhanced sintering in air at temperature up to 1600°C. X-ray and TGA results (Figures 1 and 2) show that the material remains phase pure perovskite throughout the temperature range and that no apparent liquid is formed in the material. Figure 3 shows densification studies performed at 1600°C/6hrs as a function of dopant concentration. Despite problems with the processing resulting in green densities of 48-51% theoretical density, samples were still found to sinter to 90% of theoretical density. The relative densities level off with about 7% dopant additions. Figure 4 shows a polished and thermally etched

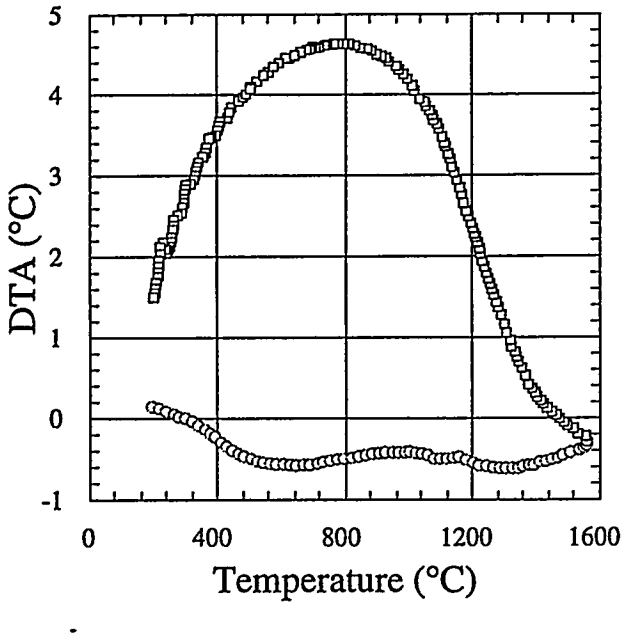


Figure 1: DTA Results on $LaCr_{0.9}M_{0.1}O_3$

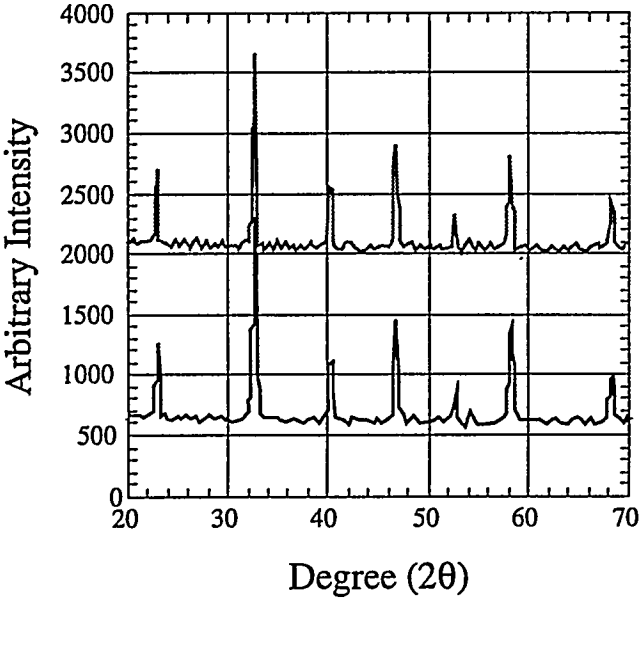


Figure 2: XRD Results on $LaCr_{0.9}M_{0.1}O_3$

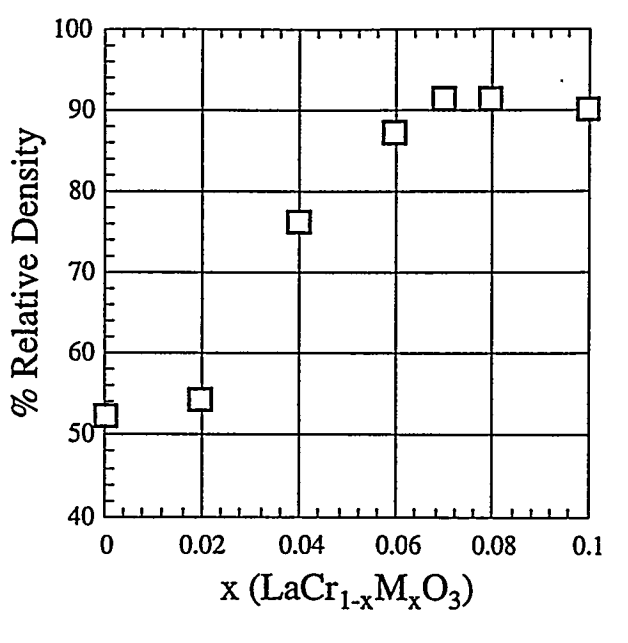


Figure 3: % Relative Density vs. Dopant Concentration for $LaCr_{1-x}M_xO_3$ (1600°C - 6h)

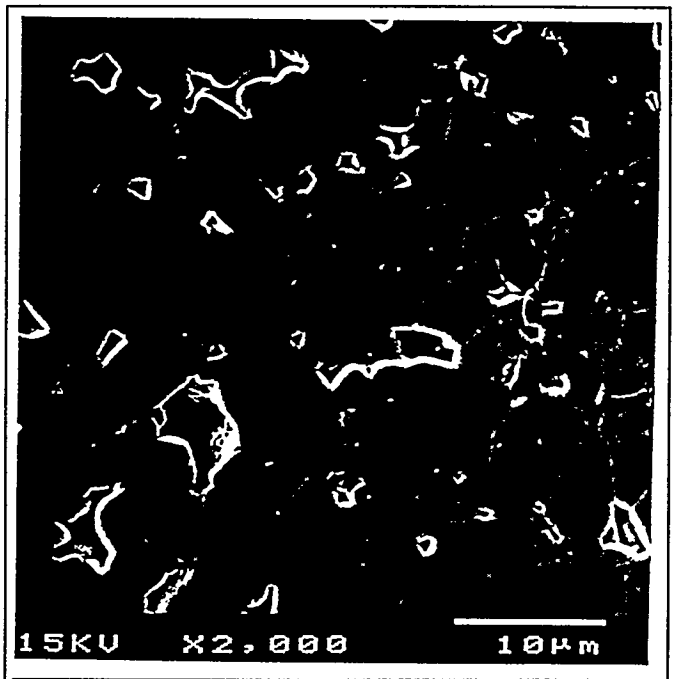


Figure 4: Polished and Thermally Etched Specimen of $LaCr_{.90}M_{.10}O_3$ (1600°C - 6h)

specimen of $LaCr_{.90}M_{.10}O_3$ corresponding to a relative density of ~90%. Tests on the effects of stoichiometry were performed for lanthanum contents of 0.98, 0.99, and 1.00. Figure 5 shows that when the lanthanum content is reduced 1% to a nominal composition of $La_{.99}Cr_{.90}M_{.10}O_3$ the

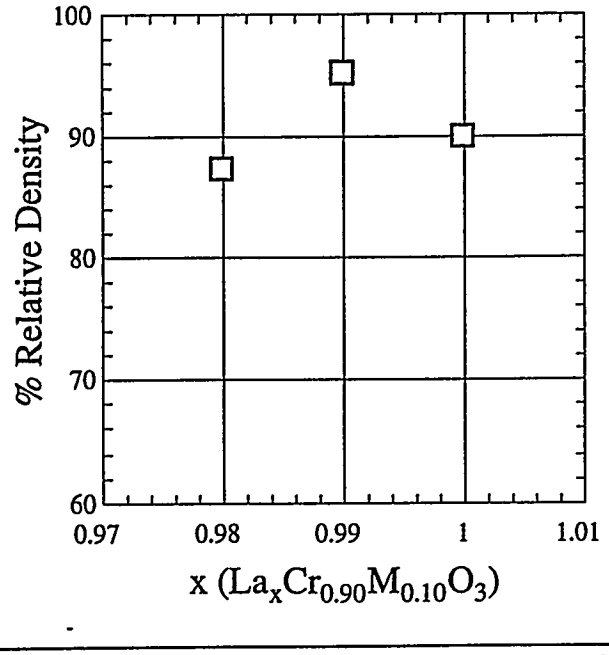


Figure 5: % Relative Density vs. Lanthanum Content for $La_xCr_{0.9}M_{0.1}O_3$ (1600°C - 6h)

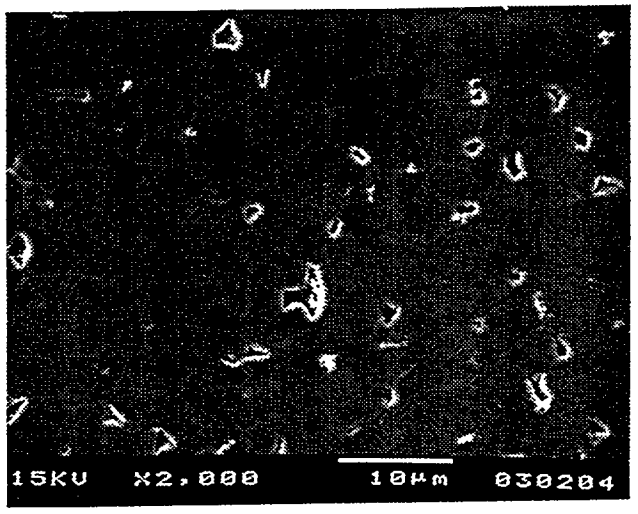


Figure 6: Polished and Thermally Etched Specimen of $La_{.99}Cr_{.90}M_{.10}O_3$ (1600°C - 6h)

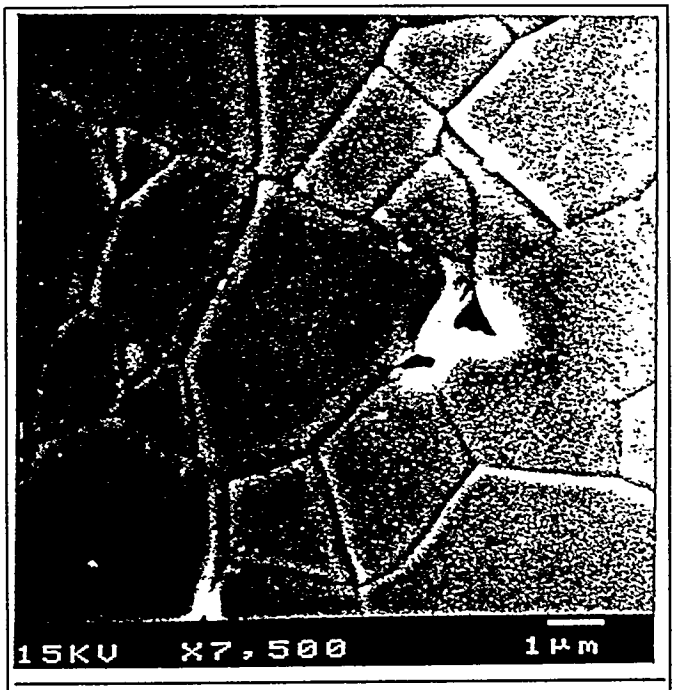


Figure 7: Polished and Thermally Etched Specimen of $La_{.99}Cr_{.90}M_{.10}O_3$ (1600°C - 6h)

relative density is increased to 95%. Figures 6 and 7 show a polished and thermally etched $La_{.99}Cr_{.90}M_{.10}O_3$ specimen . The percent linear shrinkage of $La_{.99}Cr_{.90}M_{.10}O_3$ is shown is figure 8, with the sample exhibiting ~28% shrinkage to a final relative density of ~90% after 6 hours at 1600°C. Sintering starts at ~1100°C reaching a shrinkage of 17% before the hold at 1600°C. Further results have shown that 4 hrs is sufficient to reach the maximum shrinkage. Improvements in processing related issues should allow for even higher sintered densities to be achieved, possibly allowing the lowering of the sintering temperature. Current work is focusing on theses issues.

B) Property Control

Preliminary studies have shown that $La_{.99}Cr_{.90}M_{.10}O_3$ has a conductivity of ~8S/cm at 1000°C, sufficient for fuel cell applications (Figure 9). As seen in figure 10 the plot of $Log(\sigma^*T)$ vs. 1000/T is linear, as is expected under a small-polaron model. Current work is progressing on the evaluation of other properties such as: mechanical and electrical integrity under ambient and hydrated reducing conditions, match of thermal expansion with other cell components, and long term chemical stability with other materials in the fuel cell.

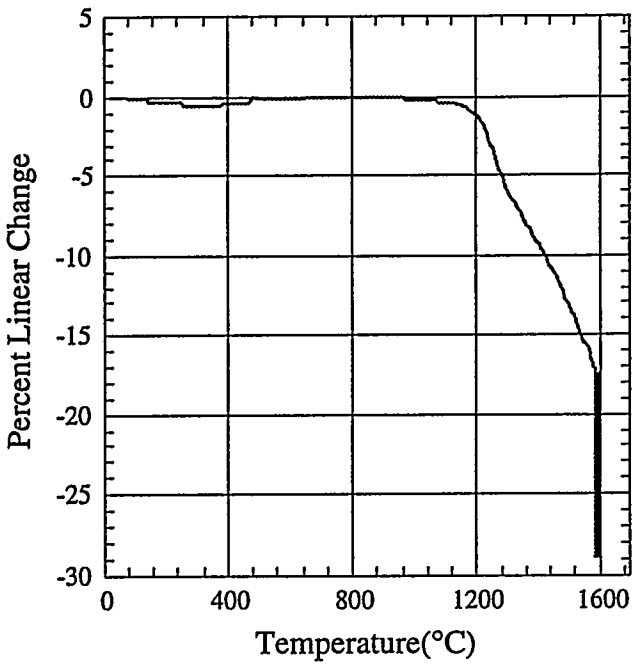


Figure 8: Percent Linear Shrinkage of $La_{.99}Cr_{.90}M_{.10}O_3$

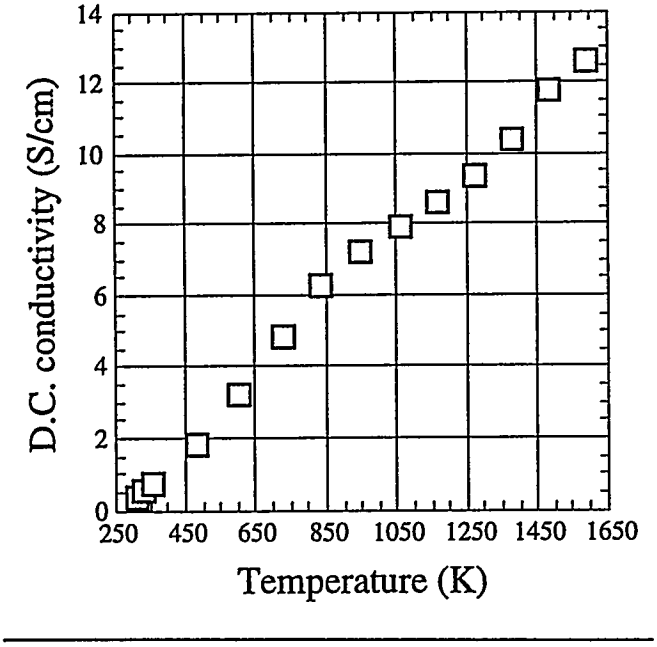


Figure 9: DC Conductivity of $La_{.99}Cr_{.90}M_{.10}O_3$ sintered at 1600°C - 6h

Major Achievement: The development of a non-liquid phase sintered $LaCrO_3$-based interconnect system that utilizes a B-site dopant.

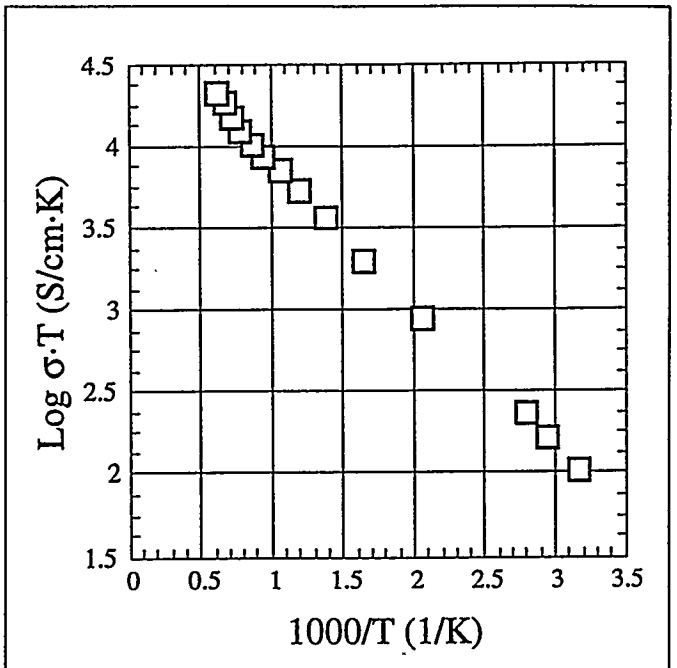


Figure 10: Arrhenius Plot of the DC Conductivity of $La_{.99}Cr_{.90}M_{.10}O_3$ sintered at 1600°C - 6h

SOFC PERFORMANCE STUDIES

In this portion of the research we are focusing on the microstructure ↔ property relations in solid oxide fuel cells (SOFC's) to better understand the mechanisms involved in cell performance. The aim is to fabricate SOFC's with controlled microstructures utilizing $La_{1-x}Sr_xMnO_3$ (LSM), yttria stabilized zirconia (YSZ), and Ni-YSZ composites as the cathode, electrolyte, and anode, respectively. The specific objectives of this research project are to:

→ Fabricate SOFC components with controlled microstructures (i.e. grain size and porosity of the cathode/anode).

→ Gain a better understanding of the mechanisms involved in improving cell performance via electrochemical and impedance techniques, and correlate these results to the microstructure.

This aspect of the project is divided into three phases:

I. Construct a system for measuring planar SOFC's

II. Processing and forming of trilayer structures of anode/electrolyte/cathode

III. Electrical characterization of single cells by electrochemical and impedance techniques.

Phase I was completed during Year 2, whereas Phases II and III are presently ongoing.

Experimental Approach:

Sample Preparation

Single cells were fabricated by conventional thick film processing techniques as described in Figure 11. Commercial YSZ powder (Zirconia Sales of America) was mixed with appropriate binders, solvents, plasticizers, and surfactants to prepare a castable slip. The slip was ball milled with ZrO_2 media then cast at a thickness of 10 mils using the doctor blade technique. Circular samples were punched out of the tape and a thin Pt reference probe was screen printed onto the YSZ tape. The samples were laminated and fired flat at 1450°C for 2 hours. Fired thicknesses of the Pt reference electrode and the YSZ electrolyte are 5 and 200μm, respectively (Figures 12 and 13).

Anode powders of NiO, $Ni_{1-x}Mg_xO$, YSZ (Zirconia Sales of America) and Sm doped CeO_2 were used as starting materials. NiO, $Ni_{1-x}Mg_xO$, and Sm doped CeO_2 were prepared by the Pechini liquid mix and glycine -nitrate methods to give powders of varying particle size and surface area. Powder crystallinity, phase and surface area were characterized by X-ray diffraction and BET techniques as a function of calcination temperature. Anode powders were mixed with appropriate binders, solvents, and surfactants and three roll milled to prepare high viscosity pastes with correct rheological behavior for screen printing. Anode pastes were screen printed onto dense YSZ electrolytes and fired at various temperatures (1200 - 1500°C). A typical anode microstructure, NiO - YSZ, fired in air is shown in Figure 14. The anode thickness can be varied depending on the mesh size, emulsion thickness, and wire diameter of the screen.

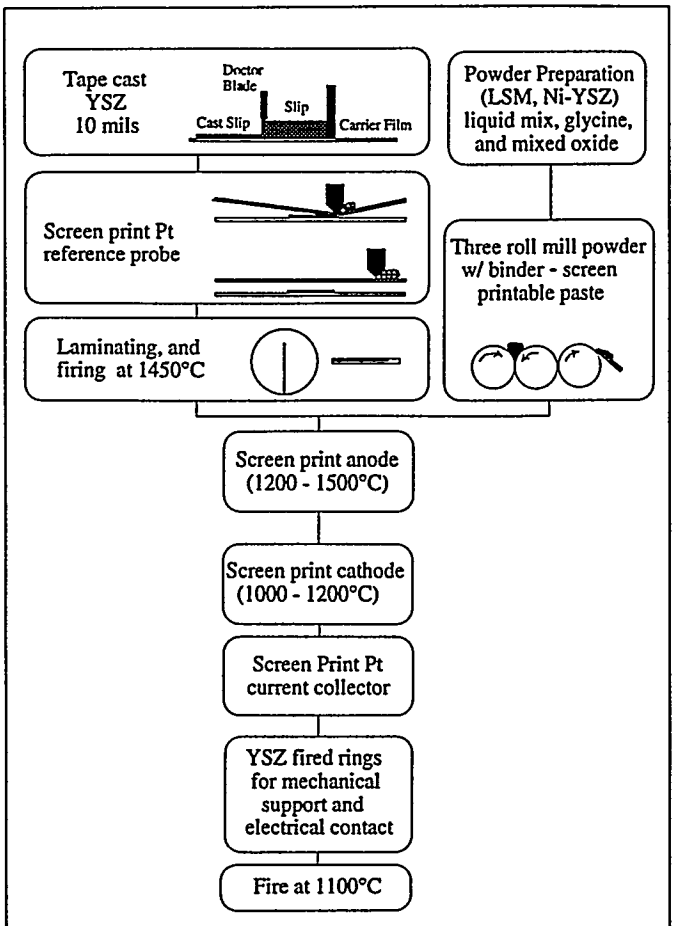


Figure 11. Experimental Overview

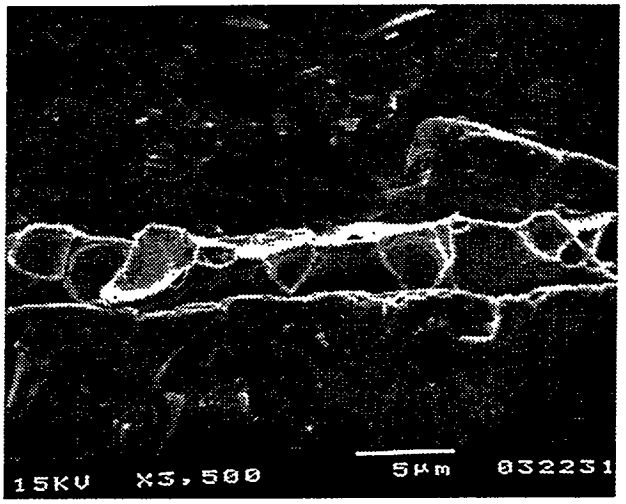


Figure 12: Pt Reference Electrode within the YSZ Electrolyte

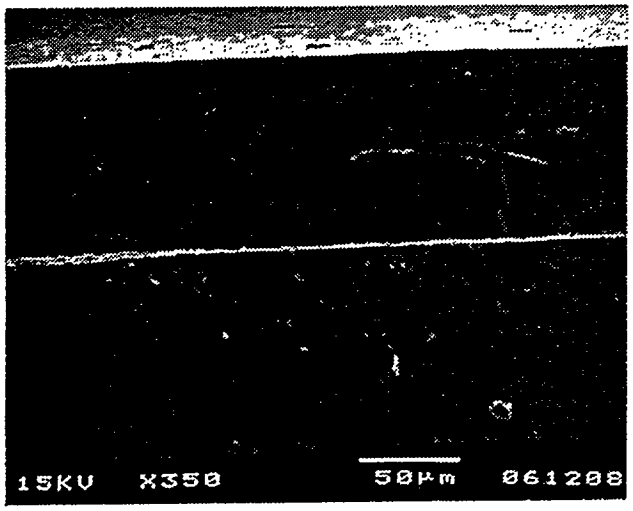


Figure 13: Pt Reference Electrode within the YSZ Electrolyte

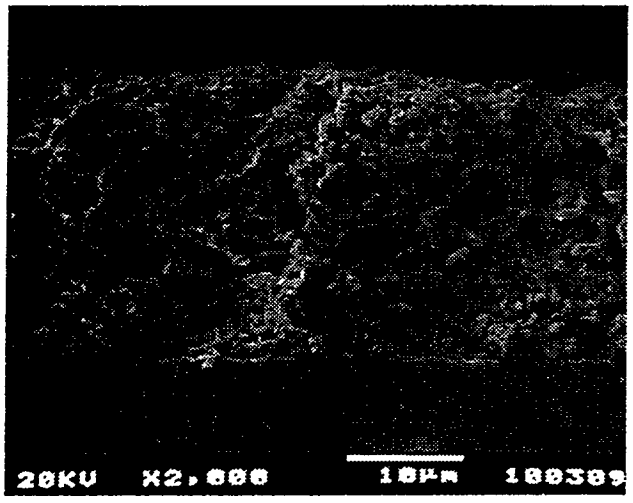


Figure 14: Typical Screen-printed Ni:YSZ Anode

Different cathode powders of composition $La_{0.79}Sr_{0.2}MnO_3$ (LSM-20) were made by various techniques (Pechini liquid mix, glycine - nitrate, and mixed oxide) to give powders of varying particle size and surface area. Powder crystallinity, phase and surface area were characterized by X-ray diffraction and BET techniques as a function of calcination temperature. Cathode powders were prepared into pastes by a similar method as described above, screen printed onto the YSZ electrolyte and fired at various temperatures (1000 - 1200°C). Various cathode microstructures have been developed for the same firing temperature (1100°C/1 hour) with different processing techniques (Figures 15 - 18).

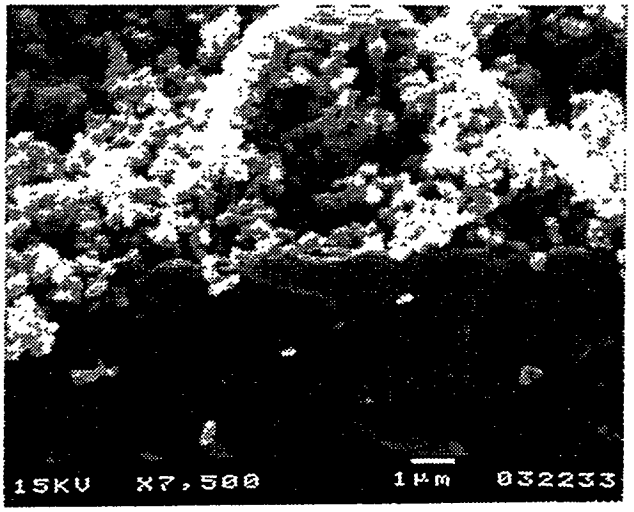


Figure 15: Screen Printed LSM Cathode → Glycine Nitrate Precursor Calcined at 800°C - 1h; fired on at 1100°C - 1h.

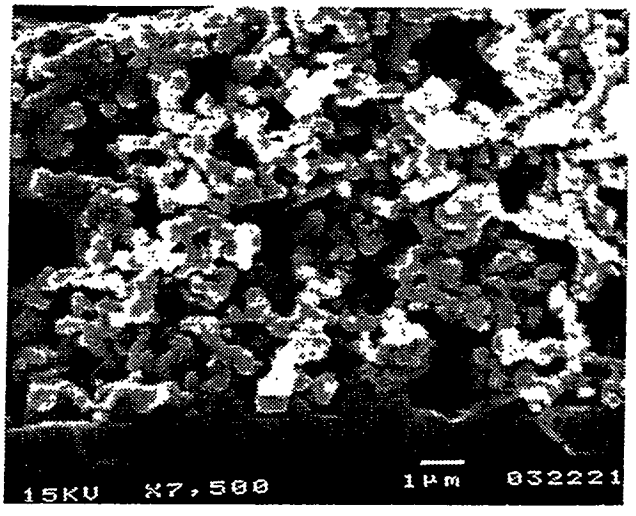


Figure 16: Screen Printed LSM Cathode → Pechini Precursor Calcined at 900°C - 1h; fired on at 1100°C - 1h.

A Pt current collector was printed onto the LSM-20 to mimic a Pt mesh screen. A typical microstructure of the Pt current collector is given in Figure 19.

Dense YSZ rings with a thickness of ~ 400µm were cemented on both sides of the electrolyte for mechanical support and electrical contact.

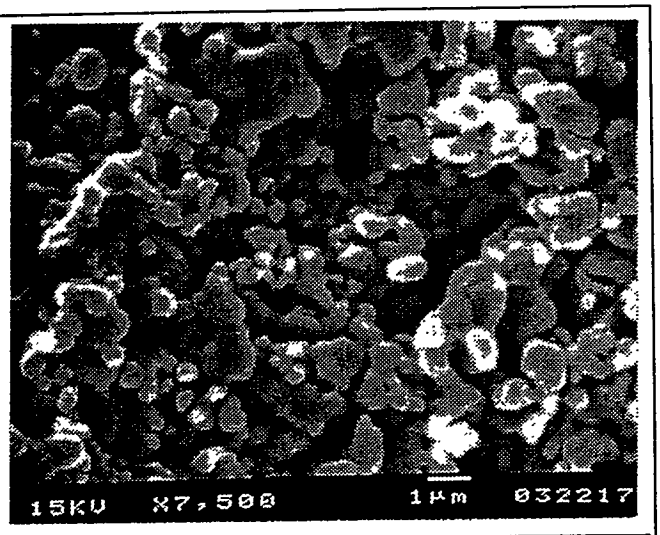


Figure 17: Screen Printed LSM Cathode → Pechini Precursor Calcined at 1100°C - 1h; fired on at 1100°C - 1h.

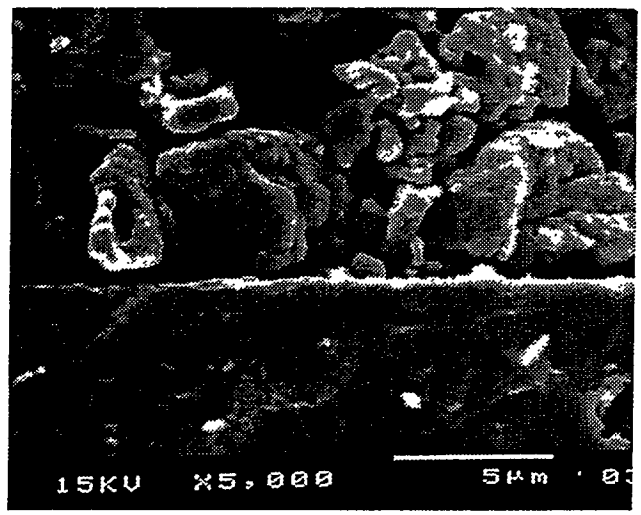


Figure 18: Screen Printed LSM Cathode → Mixed Oxides Calcined at 1100°C - 1h; fired on at 1100°C - 1h.

Sample Characterization

The system built for measuring cell performance is shown in Figure 20. The apparatus is a two tube design capable of operating at temperatures up to 1100°C. The oxygen activity of the oxidant and fuel is controlled by mass flow controllers and the pO_2 is monitored using a ZrO_2 sensor. Partial pressures of O_2 are controlled by O_2 - N_2 mixtures or air on the cathode side and H_2-H_2O, and H_2-N_2-CO_2 mixtures on the anode side. Values obtained from the ZrO_2 sensor are in good

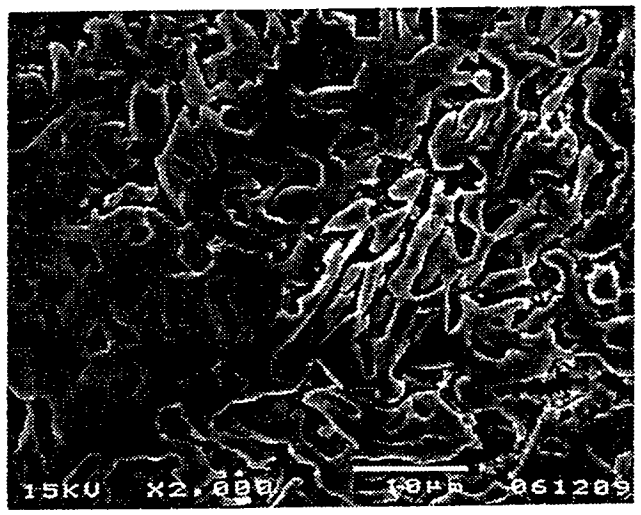


Figure 19: Screen Printed Pt Current Collector.

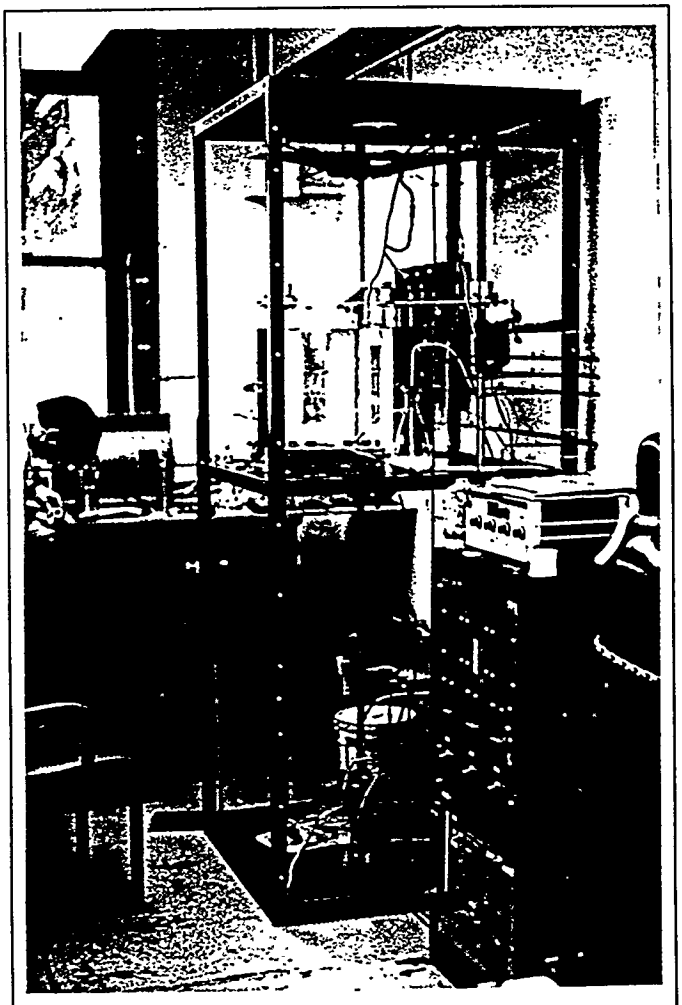


Figure 20: Cell Measurement System

agreement with values derived from thermodynamic data.

Electrochemical measurements are carried out using a three electrode configuration which allows for separation of electrode effects during operation. Pt lead wires are used on the cathode side for electrical connection to both the cathode and reference electrodes while Ni wire is used on the anode side. Electrical characterization, I - V, current density, and interfacial characterization, is performed using a Keithley 197 Microvolt DMM, a Hewlett Packard 4192A Impedance Analyzer, and a computer controlled Solartron Electrochemical Interface (1286) and Impedance/Gain-Phase Analyzer (1260).

Results and Discussion

Electrochemical data obtained at present has been focused on the overpotential associated with the anode, namely, coarsening of the Ni particles. All cell have an effective electrolyte thickness of 200μm with a Pt reference electrode buried in the center of the electrolyte. The same cathode ($La_{0.79}Sr_{0.2}MnO_3$) has been used for all experiments and was fired on the electrolyte at 1100°C for 1 hour. The only variables to present are the anode composition, Ni content, firing temperature, and gas composition.

Experiments were initially performed on YSZ-NiO anodes containing 43 volume % Ni after reduction with various firing temperatures (1250, 1300, 1400, and 1500°C). Cells were measured with air as the oxidant and a H_2-N_2-CO_2 mixture as the fuel. The theoretical open circuit voltage is 1.00 V. Plots of cell voltage, anode overpotential, and cathode overpotential versus current density measured under load are given in Figure 21. Anode and cathode overpotentials are the voltages measured from the electrodes (cathode or anode) to the Pt reference electrode and do not include any IR losses associated with the anode, cathode, and electrolyte. The IR drop across the electrolyte and anode at the given current density does not have any significant contribution to the total anode overpotential although the IR loss across the cathode may contribute to the total cathode

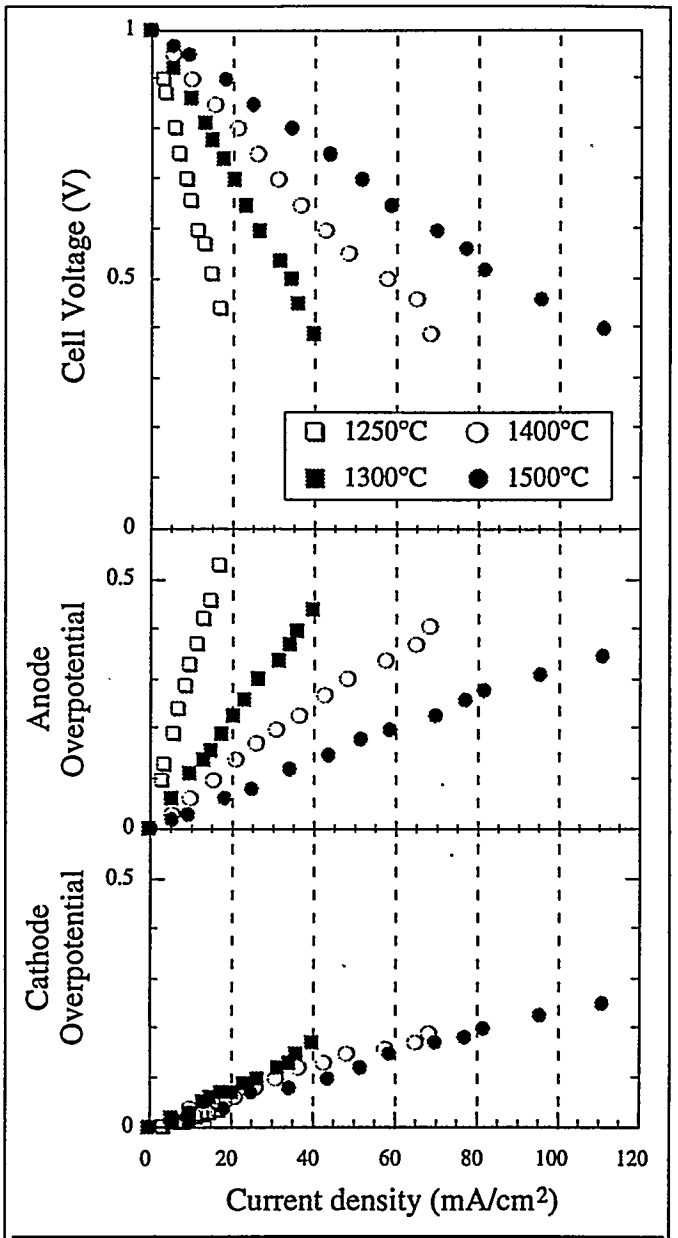


Figure 21: I-V Results on Cells with Anodes Sintered at Different Temperatures

overpotential. Sheet resistance measurements need to be performed on the cathode material to successively separate the cathode overpotential into a IR drop and a nonohmic loss.

From Figure 21 it can be seen that the cell performance increases as the firing temperature increases. The increase in performance with firing temperature is related to the decrease in the potential drop across the anode. The higher firing

temperature may create a more rigid network of YSZ-YSZ contacts thus reducing the tendency of the Ni to sinter. Figure 22 is an SEM micrograph of an anode fired on at 1400°C, reduced and operated under load for 8 hours. The YSZ particle size is approximately 1 μm whereas the Ni particles have coarsened to approximately 5 μm. Before reduction the YSZ and NiO particles were both roughly 1 μm (Figure 14). The cathode overpotentials are essentially the same for a given current density for all samples. Figure 23 shows the cathode microstructure after load for 8 hours; no changes in microstructure are evident.

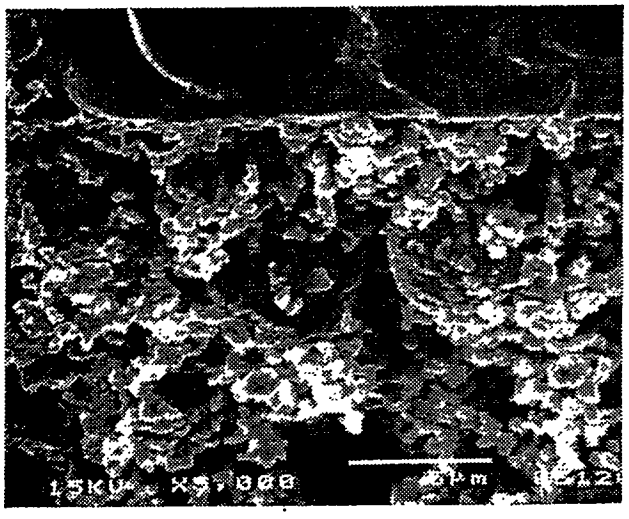


Figure 22: Anode Fired on at 1400°C; Operated as a Cell for 8 h at 1000°C.

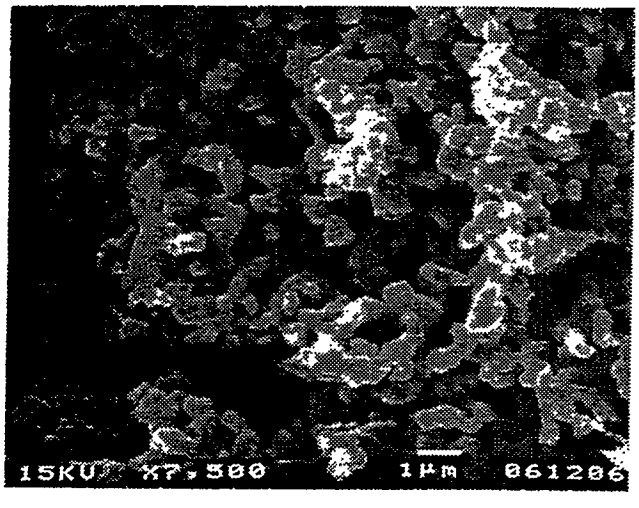


Figure 23: LSM Cathode after Operation as a Cell for 8 h at 1000°C.

The effect of Ni content was investigated with a constant anode and cathode sintering temperature, 1400 and 1100°C, respectively. The same experimental parameters were used as above (i.e. gas composition, electrolyte thickness, and placement of Pt reference electrode). Figure 24 shows the improvement of the cell with a higher loading of NiO, 54 versus 43 volume % Ni after reduction. The anode overpotential is smaller for a higher Ni loading for a given current density

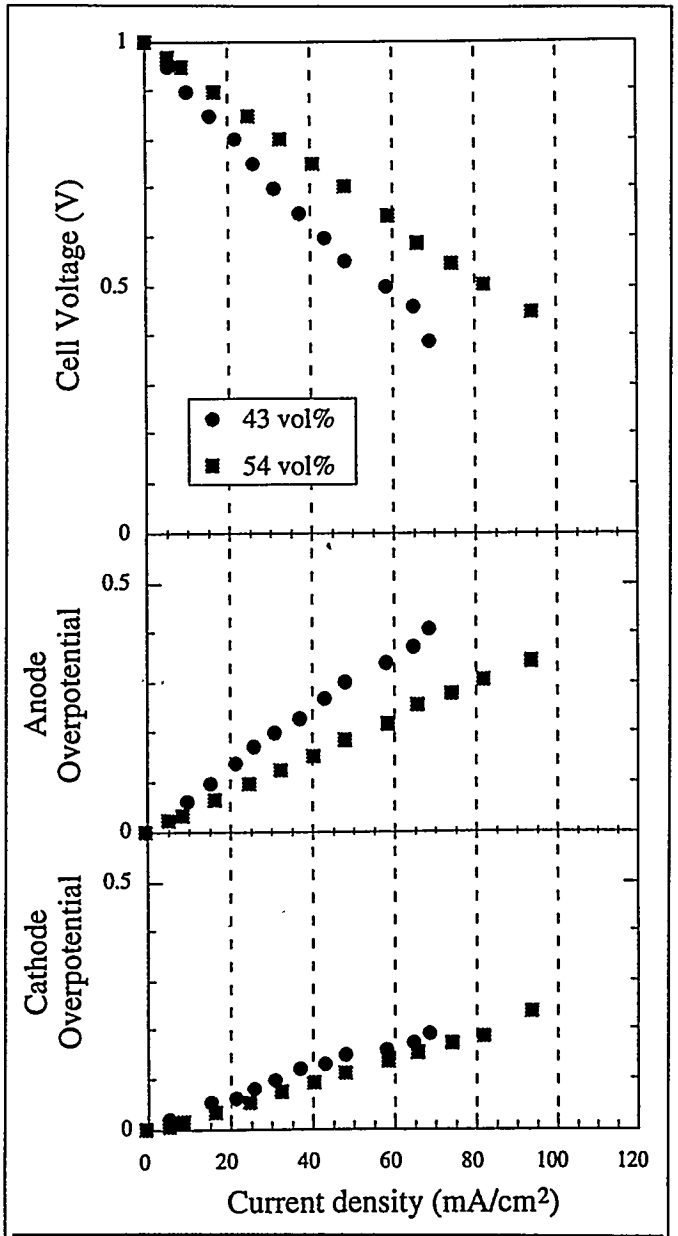


Figure 24: I-V Results on Cells with Anodes Containing Different Amounts of Ni

whereas the cathode overpotentials are essentially the same. A reason for the improvement with a higher Ni loading may be that a higher percentage of contact points (three phase boundaries) are present which increase the electrochemical oxidation reaction rate.

The anode composition was also investigated by replacing YSZ with Sm doped CeO_2, a mixed conductor at low oxygen partial pressures. Figure 25 shows the results of samples containing 54 volume % Ni after reduction with YSZ versus Sm doped CeO_2. Experimental conditions are the same as described above. There is no significant difference between samples but the Sm doped CeO_2 sample does show a slight improvement and a lower overpotential. A possible explanation for the similar results may be that the current density is not high enough to see a significant difference between the two samples. Future investigations will be focus on improving cell performance (i.e. microstructure, anode thickness, etc.) such that the anode composition (YSZ vs. CeO_2) can be better understood.

Anodes composed of 10% MgO doped into NiO to form a solid solution $Ni_{0.9}Mg_{0.1}O$ was investigated as a function of volume % Ni after reduction. Experimental conditions were similar to those given above except H_2 bubbling through H_2O at room temperature was used as fuel. YSZ was used as the other component in the cermet. Figure 26 shows the effect of Ni content for samples with 10% MgO, MgO additions, and gas composition on cell performance. There is a small improvement with increased Ni content for samples containing 10% MgO. The higher Ni loading may increase the number of reaction sites and thus performance as stated earlier. From the data in Figure 26, the improvement in cell performance can be attributed to MgO additions or gas composition, although, the exact parameter(s) controlling performance is not known. Experiments aim at understanding this need to be performed (i.e. YSZ-Ni cermet without MgO utilizing H_2/H_2O as the fuel). Figure 27 is an SEM micrograph of a Ni-MgO-YSZ/YSZ interface reduced at 1000°C for 8 hours. From the micrograph it can be seen that the MgO is successful in reducing the coarsening effect of the

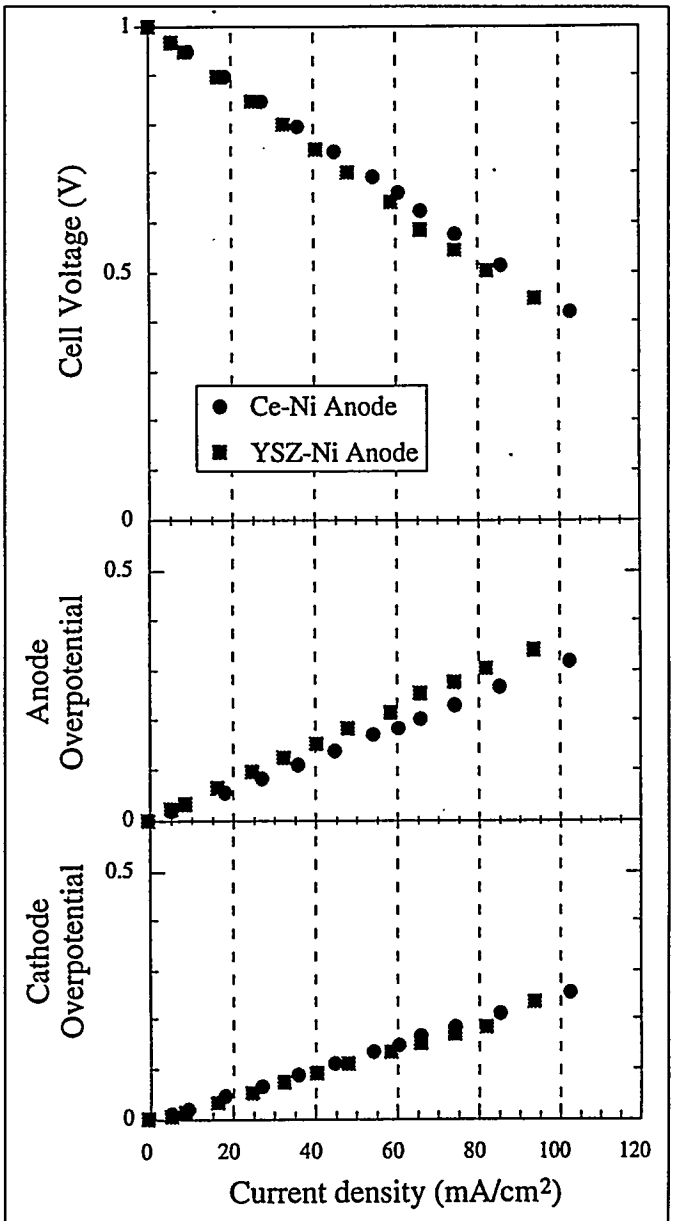


Figure 25: I-V Results on Cells with Anodes Containing Ni: CeO_2 and Ni:YSZ.

Ni in comparison to a Ni-YSZ cermet with no MgO additions (Figure 22).

Major Achievements During the Past Year

- Developed processing skills necessary to fabricate single cells.

- Incorporated a Pt reference electrode into the electrolyte for separation of electrode effects.
- Developed processing ↔ microstructure ↔ property relations for a number of anodes.
- Developed experimental techniques for measuring cell performance.

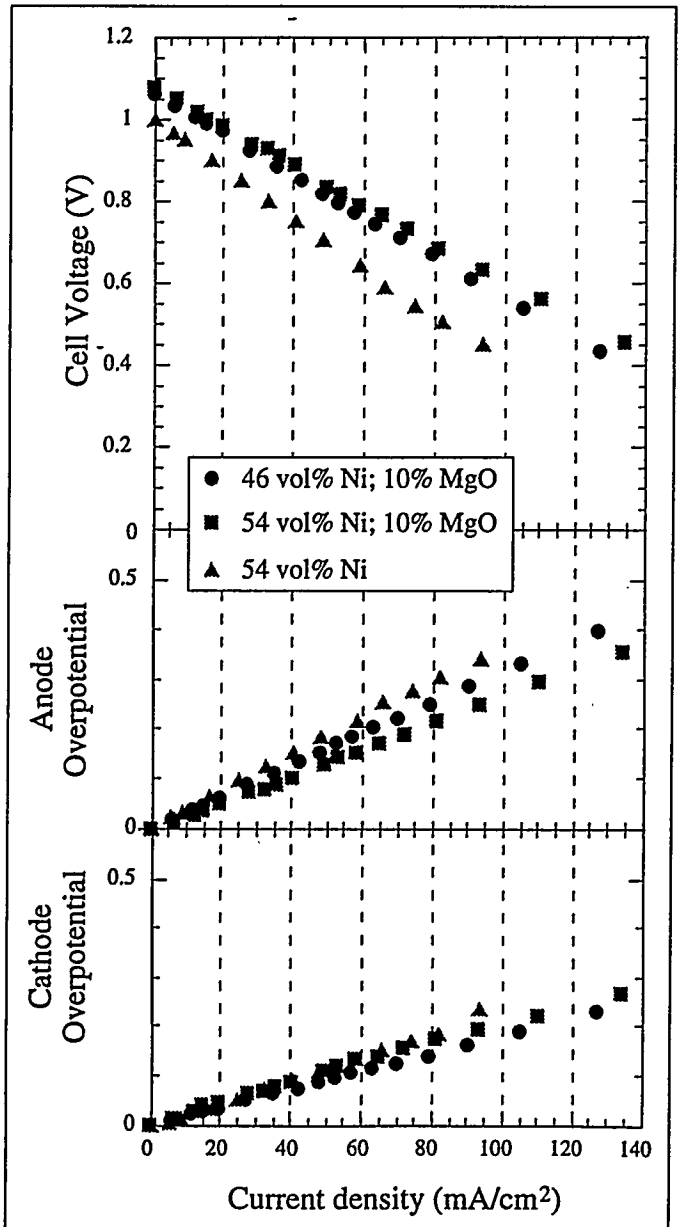


Figure 26: I-V Results on Cells with Anodes with and without MgO Additions to Stabilize the Ni Size

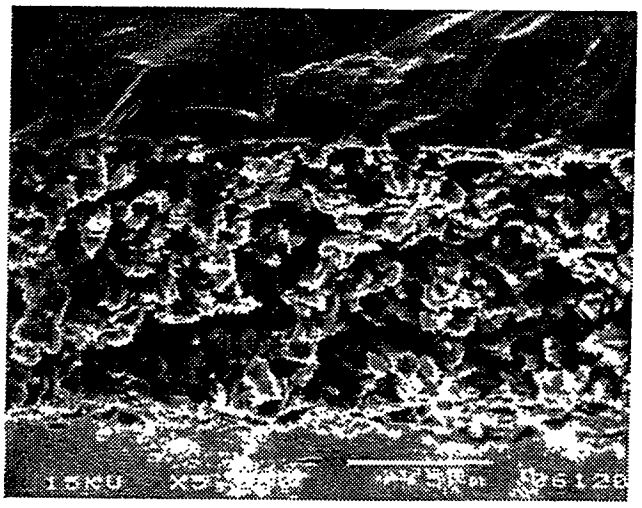


Figure 27: Anode Structure Stabilized with MgO Additions

FUTURE WORK

With respect to the major thrusts of this program:

$LaCrO_3$ Sintering Studies

- Continue development of a non-liquid phase sintered $LaCrO_3$-based material sinterable in air.
- Optimize and control the processing conditions associated with $LaCrO_3$→ minimize the particle size and increase the green density.
- Incorporate materials developed in this program into planar cells and measure their performance.

SOFC Performance Studies

- Fabricate single cells with controlled microstructures of the cathode; incorporate interfacial modifications to improve the catalytic activity.

- Gain a better understanding of the mechanisms involved in improving cell performance via electrochemical and impedance techniques.

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