Development and Characterization of High Performance and Robust Mixed Conducting Cathodes Supportive of Lower Cost SOFCs

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Zhien Liu                      Senior Investigator
Rich Goettler                  Senior Investigator
## Summary of Our Preliminary and Year 1 Research

### Results and Technical Approaches

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<tr>
<th>Identifying a series of nickelates with an active performance</th>
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<td>(+) High D and k in a wide temp region (+) Cr resistance; (-) Phase decomposition</td>
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<th>Identifying a series of nickelates with a stable and active performance (LGFCS proprietary work)</th>
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### Testing methodology

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<th>Reproducibility and reliability</th>
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## Comparison between Nickelates and Perovskite Cathodes

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<th>Nickelates</th>
<th>Advantage</th>
<th>Disadvantage</th>
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<tbody>
<tr>
<td></td>
<td>1. Superior D and k</td>
<td>Phase transition in ( A_{n-1}A_2B_nO_{3n+1} ), from ( n=1 ) to ( n&gt;1 ).</td>
</tr>
<tr>
<td></td>
<td>( 600^\circ C: D = 2 \times 10^{-8} \text{ cm}^2/\text{s}; k = 4.2 \times 10^{-7} \text{ cm/s} )</td>
<td></td>
</tr>
<tr>
<td></td>
<td>( 800^\circ C: D = 1.1 \times 10^{-7} \text{ cm}^2/\text{s}; k = 3 \times 10^{-6} \text{ cm/s} )</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2. Active over wide range of temperatures</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. Thermal expansion matching with electrolytes</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4. Absence of Sr and kinetic demixing</td>
<td></td>
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<th>LSCF</th>
<th>Advantage</th>
<th>Disadvantage</th>
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<tr>
<td></td>
<td>1. Extensive research has been carried out</td>
<td></td>
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<tr>
<td></td>
<td>2. Active electrode</td>
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<tr>
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<td>( 600^\circ C: D = 4 \times 10^{-10} \text{ cm}^2/\text{s}; k = 5 \times 10^{-8} \text{ cm/s} )</td>
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<td></td>
<td>3. Long-term operation has been demonstrated at multiple institutions.</td>
<td></td>
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<tr>
<td></td>
<td>1. Sr segregation</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2. Volatile Co densification</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. Demixing</td>
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(Boehm et al., SSI, 176, 2005)
Developing Cathode Materials with Low Polarization Resistance

Rationales
1. Rapid surface exchange and oxygen ion diffusion in \( \text{Pr}_2\text{NiO}_4 \) – based oxides
2. Activity of nickelates over a wide temperature region (550 – 900°C)
3. CTE Compatibility between nickelates and electrolytes (doped ceria or zirconia)

Current Status

Approaches
1. Compositions:
   - \((\text{Pr}_{0.50}\text{Nd}_{0.50})_2\text{NiO}_4 \) – PNNO5050
   - \((\text{Pr}_{0.25}\text{Nd}_{0.75})_2\text{NiO}_4 \) – PNNO2575
   - Substituted PNNO (LGFCS proprietary work)

2. Measurements
   - Half-cell measurements: ScSZ as the electrolyte measured at 750, 790, 830, and 870°C
   - Full-cell measurements: ScSZ electrolyte-supported and anode-supported cells
   - Durability (250 – 4000 hours) and reproducibility (3-5 cells per condition)
   - Current-potential sweep, impedance/differential relaxation time analysis, \( \text{pO}_2 \)
# Summary of the Preliminary and Year 1 Research

## Results and Technical Approaches

Identifying a series of nickelates with an active performance

(+ Higher D and k in a wide temp region (+) Cr resistance; (-) Phase decomposition

Identifying a series of nickelates with a stable and active performance

*(LGFCs proprietary work)*

### Thermally Stable Compounds:

- Doped PNNO5050, PNNO2575, and doped PNNO2575

### Electrochemically Stable Compounds:

- To be reported

## Testing methodology

### Reproducibility and reliability

- Multiple cells; statistic distribution of data

### Quantification of Phase Evolution in a Single Cell

- Standard and Au grid electrode

### Accelerated Testing Protocols

- Theoretical analysis and methods
Stabilize the Structure
What are the roles of substituting elements?

Rationale
1. It is unknown why the performance of Pr$_2$NiO$_4$ is stable, but the phase transition from Pr$_2$NiO$_4$ to Pr$_6$O$_{11}$ and higher ordered phases (e.g. Pr$_3$Ni$_2$O$_7$ and Pr$_4$Ni$_3$O$_{10}$) was observed.
2. Why are some compositions more stable than the others?
3. Charge/valance ratio and strain effects on the structural stability at A-site are contributing factors.

Current Status
Migration of oxygen ion to interstitial site creates vacancy available for adsorption of oxygen (Boehm et al., SSI, 176, 2005)

Approaches
1. Electrochemical measurements of doped PNNO5050 and PNNO2575
2. Phase stability in doped (Pr$_{0.25}$Nd$_{0.75}$)$_2$NiO$_4$ – PNNO2575 calcined at various temperatures and measured at 790 and 870°C for a long duration.
3. A-site deficient compounds has shown improved activity, but its durability is unknown. The chemical potential of A-site element will influence the formation of Pr$_6$O$_{11}$ or Nd$_2$O$_3$, thus controlling the phase stability.

Ionic Radii (3+ and CO#: 8)
La: 1.16 Å; Nd: 1.11 Å
Pr: 1.13 Å; Pr (4+): 0.96 Å
Sm: 1.08 Å; Sm (2+): 1.27 Å
## Results and Technical Approaches

| Identifying a series of nickelates with an active performance  
(+): High D and k in a wide temp region (+): Cr resistance; (-): Phase decomposition  
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**Quantification of Phase Evolution in a Single Cell**
- Standard vs. Au grid electrode

**Accelerated Testing Protocols**
- Theoretical analysis and methods
Phase Purity and Stability of PNNO
Phase Purity And Stability Challenges in R-P Nickelates

Pr$_2$NiO$_{4+\delta}$ (PNO)

PNO at 1150 °C 5h

Pr$_{12}$O$_{22}$, Monoclinic, PDF #97-008-2107

Pr$_2$NiO$_4$, Orthorhombic, PDF #97-008-1577

NiO PDF #97-004-3740

Preliminary work under LGFCS Phase II subcontract
Phase Purity And Stability Challenges in R-P Nickelates

$\text{Pr}_2\text{NiO}_{4+\delta}$ (PNO)

$\text{Pr}_0.5\text{Nd}_0.5\text{NiO}_{4+\delta}$ (PNNO)

Preliminary work under LGFCS Phase II subcontract
Phase Purity And Stability Challenges in R-P Nickelates

Pr$_2$NiO$_{4+\delta}$ (PNO)

$+$ Pr$_6$O$_{11}$

* PrNiO$_3$

PNO at 1150 °C 5h
annealed at 790 °C for 50h

Intensity (a.u.)

NiO PDF#97-004-3740

Pr$_{12}$O$_{22}$, Monoclinic, PDF#97-008-2107

Pr$_2$NiO$_4$, Orthorhombic, PDF#97-008-1577

2θ (degree)

20 25 30 35 40 45 50 55 60

PNNO at 1150 °C 2h
annealed at 790 °C for 150h in air

Intensity (a.u.)

NiO PDF#97-004-3740

Pr$_{12}$O$_{22}$, Monoclinic, PDF#97-008-2107

Pr$_2$NiO$_4$, Orthorhombic, PDF#97-008-1577

2θ (degree)

20 25 30 35 40 45 50 55 60

Preliminary work under LGFCS Phase II subcontract
Phase Purity And Stability Challenges in R-P Nickelates

$\text{Pr}_2\text{NiO}_{4+\delta} (\text{PNO})$

$\text{Pr}_0.5\text{Nd}_{0.5}\text{NiO}_{4+\delta} (\text{PNNO})$

$\text{Pr}_6\text{O}_{11}$

$\text{PrNiO}_3$

$\text{PNO at 1150 } ^\circ\text{C 5h}$

annealed at $790 ^\circ\text{C}$ for 50h

$\text{PNO at 1150 } ^\circ\text{C 5h}$

$\text{PNO at 1150 } ^\circ\text{C 5h}$

annealed at $790 ^\circ\text{C}$ for 50h

$\text{Pr}_{12}\text{O}_{22}$, Monoclinic, PDF#97-008-2107

$\text{PrNiO}_4$, Orthorhombic, PDF #97-008-1577

$\text{NiO PDF#97-004-3740}$

Intensity (a.u.)

(-137) (135) (-331) (-133) (131)

50% Nd-substitution does not stabilize R-P Structure

Preliminary work under LGFCS Phase II subcontract
Phase Purity And Stabilizing The PNO Composition B at 1150 °C 2h annealed at 790 °C for 150h in air
Composition A at 1150 °C 2h annealed at 790 °C for 150h in air
Composition A at 1150 °C 2h
Composition B at 1150 °C 2h

\[
\text{Pr}_6\text{O}_{11}, \text{ Orthorhombic, PDF#97-008-1577}
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\text{NiO PDF#97-004-3740}
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\[
\text{Pr}_{12}\text{O}_{22}, \text{ Monoclinic, PDF#97-008-2107}
\]

\[
\text{Pr}_3\text{NiO}_4, \text{ Orthorhombic, PDF #97-008-1577}
\]

PrStabilizing The R-P Structure
Phase Purity And Stabilizing The PNO

Composition B at 1150 °C 2h annealed at 870 °C for 150h in air
Composition A at 1150 °C 2h annealed at 870 °C for 150h in air
Composition B at 1150 °C 2h annealed at 790 °C for 150h in air
Composition A at 1150 °C 2h annealed at 790 °C for 150h in air
Composition B at 1150 °C 2h
Composition A at 1150 °C 2h

Pr$_{12}$O$_{22}$, Monoclinic, PDF#97-008-2107
Pr$_6$O$_{11}$, Orthorhombic, PDF #97-008-1577

LGFCS proprietary work
Preliminary work under LGFCS Phase II subcontract
Phase Purity And Stabilizing The PNNO

Composition B at 1150 °C 2h annealed at 870 °C for 150h in air
Composition A at 1150 °C 2h annealed at 790 °C for 150h in air
Composition B at 1150 °C 2h annealed at 790 °C for 150h in air
Composition A at 1150 °C 2h annealed at 790 °C for 150h in air

Pr$_6$O$_{11}$, Higher order phase
Pr$_{12}$O$_{22}$, Monoclinic, PDF#97-008-2107
Pr$_2$NiO$_4$, Orthorhombic, PDF #97-008-1577
Pr$_{12}$O$_{22}$, Monoclinic, PDF#97-008-2107
Pr$_2$NiO$_4$, Orthorhombic, PDF #97-008-1577

Intensity (a.u.)

2θ (degree)

Composition A at 1150 °C 2h
Composition B at 1150 °C 2h
Composition A at 1150 °C 2h
Composition B at 1150 °C 2h
Composition A at 1150 °C 2h
Composition B at 1150 °C 2h

2θ (degree)
Phase Purity And Stabilizing The PNNO

Composition B at 1150 °C 2h annealed at 870 °C for 150h in air
Composition A at 1150 °C 2h annealed at 790 °C for 150h in air
Composition B at 1150 °C 2h annealed at 790 °C for 150h in air
Composition A at 1150 °C 2h
Composition B at 1150 °C 2h
Composition A at 1150 °C 2h

Pr$_6$O$_{11}$, Orthorhombic, PDF #97-008-1577
Pr$_{12}$O$_{22}$, Monoclinic, PDF #97-008-2107
Pr$_2$NiO$_4$, Orthorhombic, PDF #97-008-1577
NiO PDF #97-004-3740

Phase Purity And Stabilizing The PNO

Composition B at 1150 °C 2h annealed at 870 °C for 150h in air
Composition A at 1150 °C 2h annealed at 790 °C for 150h in air
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Composition A at 1150 °C 2h
Composition B at 1150 °C 2h
Composition A at 1150 °C 2h

Pr$_6$O$_{11}$, Orthorhombic, PDF #97-008-1577
Pr$_{12}$O$_{22}$, Monoclinic, PDF #97-008-2107
Pr$_2$NiO$_4$, Orthorhombic, PDF #97-008-1577
NiO PDF #97-004-3740

higher order phase

distorted orthorhombic or tetragonal phase

Composition B at 1150 °C 2h annealed at 790 °C for 500h in air
Composition A at 1150 °C 2h
Composition B at 1150 °C 2h
Composition A at 1150 °C 2h

Pr$_6$O$_{11}$, Orthorhombic, PDF #97-008-1577
Pr$_{12}$O$_{22}$, Monoclinic, PDF #97-008-2107
Pr$_2$NiO$_4$, Orthorhombic, PDF #97-008-1577
NiO PDF #97-004-3740

Phase Purity And Stabilizing The R-P Structure

Composition A at 1150 °C 2h annealed at 870 °C for 150h in air
Composition B at 1150 °C 2h
Composition A at 1150 °C 2h
Composition B at 1150 °C 2h
Composition A at 1150 °C 2h
Composition B at 1150 °C 2h

Pr$_6$O$_{11}$, Orthorhombic, PDF #97-008-1577
Pr$_{12}$O$_{22}$, Monoclinic, PDF #97-008-2107
Pr$_2$NiO$_4$, Orthorhombic, PDF #97-008-1577
NiO PDF #97-004-3740

LGFCs proprietary work
Phase Purity And Stabilizing The PNO

Composition B at 1150 °C 2h annealed at 790 °C for 150h in air
Composition A at 1150 °C 2h annealed at 790 °C for 150h in air
Composition B at 1150 °C 2h annealed at 790 °C for 150h in air
Composition A at 1150 °C 2h annealed at 790 °C for 150h in air
Composition B at 1150 °C 2h annealed at 870 °C for 150h in air
Composition A at 1150 °C 2h annealed at 870 °C for 150h in air
Composition B at 1150 °C 2h annealed at 870 °C for 150h in air
Composition A at 1150 °C 2h annealed at 870 °C for 150h in air

Composition A at 1150 °C 2h annealed at 790 °C for 500h in air
Composition B at 1150 °C 2h annealed at 790 °C for 500h in air
Composition B at 1150 °C 2h annealed at 790 °C for 500h in air
Composition A at 1150 °C 2h annealed at 790 °C for 500h in air
Composition B at 1150 °C 2h annealed at 870 °C for 500h in air
Composition A at 1150 °C 2h annealed at 870 °C for 500h in air
Composition B at 1150 °C 2h annealed at 870 °C for 500h in air
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Phase Purity And Stabilizing The PNNO

Composition B at 1150 °C 2h annealed at 870 °C for 500h in air
Composition A at 1150 °C 2h annealed at 870 °C for 500h in air
Composition B at 1150 °C 2h annealed at 790 °C for 500h in air
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Composition B at 1150 °C 2h annealed at 870 °C for 150h in air
Composition A at 1150 °C 2h annealed at 870 °C for 150h in air
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Composition B at 1150 °C 2h annealed at 790 °C for 150h in air
Composition A at 1150 °C 2h annealed at 790 °C for 150h in air
Composition B at 1150 °C 2h annealed at 790 °C for 150h in air
Composition A at 1150 °C 2h annealed at 790 °C for 150h in air

Phase Stabilized with Combination of A and B site Substitution and Doping
Fully Stabilizing the R-P Structure via A-site Doping

Composition C at 1150 °C 2h

Pr$_2$NiO$_4$, Orthorhombic, PDF #97-008-1577

Pr$_{12}$O$_{22}$, Monoclinic, PDF#97-008-2107

Pr$_2$O$_2$, Monoclinic, PDF #97-006-2107

NiO PDF#97-004-3740

Pr$_2$NiO$_{4+x}$, Orthorhombic, PDF #97-006-1577

PNO at 1150 °C 5h annealed at 790 °C for 50h

PNO at 1150 °C 2h annealed at 870C for 150h in air

PNO at 1150 °C 2h annealed at 870C for 150h in air

Intensity (a.u.)

2θ (degree)
Fully Stabilizing the R-P Structure via A-site Doping

Pr$_2$NiO$_4$, Orthorhombic, PDF #97-008-1577

NiO PDF#97-004-3740

Composition C at 1150 °C 2h annealed at 790 °C for 150h in air

Pr$_{12}$O$_{22}$, Monoclinic, PDF#97-008-2107

Pr$_{12}$O$_{22}$, Monoclinic, PDF#97-008-2107

Pr$_{2}$NiO$_4$, Orthorhombic, PDF #97-008-1577

Composition C at 1150 °C 2h

PNNO2575
Fully Stabilizing the R-P Structure via A-site Doping
Substituted PNNO2575
Phase Stable: 790 and 870 °C, 3% H₂O, dopant%, and T_{calcining}

Comp 1 at 870 °C for 150 h in 3% humidified air

Comp 2 at 870 °C for 150 h in 3% humidified air

Comp 2: doped PNNO2575 after calcining at T2

Comp 1: doped PNNO2575 after calcining T1

Pr$_2$NiO$_4$, Orthorhombic, PDF #97-008-1577

Pr$_{12}$O$_{22}$, Monoclinic, PDF #97-008-2107

Nd$_2$O$_3$, PDF #00-006-0408

NiO, PDF #97-004-3740

Intensity (a.u.)

20 25 30 35 40 45 50 55 60

2θ (degree)
Quantification of Cathode Durability
Quantification of phase evolution is necessary

1. to investigate the kinetics for phase decomposition (in LSCF) and phase transition (in nickelates).
2. to predict the durability of the cathodes.

Quantification is very challenging because of

1. the overlapping of XRD reflections and possible interactions between the cathode and its current collector (e.g. LSC, LNF).
2. the cathode thickness (30 μm), which might be too much for XRD analysis to study the cathode evolution occurring at the cathode/electrolyte interfaces.
3. the slow kinetics of phase evolution (could take 1000s-hours), which necessitate the development of accelerated test protocols.
Requirements and Cell Designs for Quantifications

Requirements for Current Collector
1. Chemically and mechanically compatible with the cathode
2. Chemically stable during operation
3. Electrically conducting
4. Its XRD reflections don’t overlap with major cathodes
5. Don’t interfere (deteriorate or enhance) cathode performance
6. Stable and reproducible XRD reflections before and after measurements to provide a baseline.

Cell Designs by using Au Current Collector
1. Design a series of Au grids with various open areas and grid width, which are used as current collector and baseline reference
2. Questions to be addressed: (1) which is the optimized thickness of Au grid (2) Au intensity and peak area vs. operation conditions.

From oxide current collector

       to gold collector
Validation of Stable and Reproducible Au Intensity

(LSCF and Au were printed with the same grid, so Au covered all LSCF)
Validation of Stable and Reproducible Au Intensity

YSZ/GDC/LSCF/Au (LSCF and Au were printed with the same grid, so Au covered all LSCF)
Validation of Stable and Reproducible Au Intensity

YSZ/GDC/LSCF/Au (LSCF and Au were printed with the same grid, so Au covered all LSCF)
Validation of Stable and Reproducible Au Intensity

YSZ/GDC/LSCF/Au (LSCF and Au were printed with the same grid, so Au covered all LSCF)
Electrochemical Measurements (Accelerated Test)
Accelerated Test: LSCF

(a) & (b) Potential vs. Current Density for LSCF-I and LSCF-III at 750 °C with humidified Air/H₂.

(c) Power Density vs. Current Density for LSCF-I and LSCF-III at 750 °C with humidified Air/H₂.

(d) & (e) Nyquist plots for LSCF-I and LSCF-III at different hours (0th, 50th, 100th, 150th, 200th).

(f) Nyquist plots for LSCF-III at different hours (0th, 50th, 100th, 150th, 200th).
Performance of PNNO5050 and Substituted PNNO5050

Cell performances are reproducible. Au was used as the current collector to quantify the kinetics of phase evolution.

The ohmic loss is small at all temperatures even though the electrolyte is ~ 20 μm. The cathodic resistance for gas diffusion seems large, which can be reduced by using LSC or LNF. But the reason why it was not observed in LSCF is unknown.

$R_{\text{ohm}}$ is 0.12 Ω cm$^2$ at 750 °C decreasing to 0.06 Ω cm$^2$ at 850 °C.
1. Is Au peak area consistent among the cells?
2. Does Au peak area change after accelerated test, if so by how much and why?
3. Can the crystal phase changes in the cathode be quantified in a reliable way?

- Cathode phase evolution can be calculated with respect to Au reference peak
- Average Au peak area before and after the measurements can be used directly to calculate the percentage of cathode phases.

Full cells with Au grid after the measurements.
## Results and Technical Approaches

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### Electrochemically Stable Compounds:
- To be reported

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**Reproducibility and reliability**
- Multiple cells; statistic distribution of data

**Quantification of Phase Evolution in a Single Cell**
- Standard and Au grid electrode

**Accelerated Testing Protocols**
- Theoretical analysis and methods