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Molten Carbonates as an Effective Oxygen Reduction Catalyst for 550–650°C Solid Oxide Fuel Cells

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We report the first study that investigates the use of molten carbonates as an effective catalyst to promote electrochemical oxygen reduction reaction (ORR) at the cathode of intermediate temperature solid oxide fuel cells (IT-SOFCs). A series of binary Li-K carbonate compositions were incorporated into the porous backbones of a commercial cathode assembled in symmetrical impedance cells for electrochemical characterization. Within the temperature range of 550–650°C, we observed that the polarization and ohmic area-specific resistances of the original sample can be significantly reduced by the introduction of molten carbonates. A new ORR charge-transfer model involving two intermediate species CO52− and CO42− as the fast oxygen absorber and transporter, respectively, was presented as the mechanism for the facile ORR kinetics promoted by molten carbonates.

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Solid oxide fuel cell (SOFC) offers a great potential to produce electricity, heat and fuels with high efficiency and low emissions.1 To compete with conventional internal combustion engines (ICEs), the high-efficiency and low-emission SOFC needs to excel in performance and cost. A rational approach for SOFC to achieve that goal is to lower its operating temperature from current 800–1000°C to ≤650°C, so that the overall system cost as well as degradation rate can be reasonably reduced by the use of low-cost materials and increased reliability.2–5 A challenge to the pursuit of such an optimization of cathode microstructure to maximize the ORR-active porous backbones of a commercial cathode can substantially reduce the polarization resistance of the ORR kinetics at t ≤ 650°C.

A new comprehensive electrochemical charge-transfer model is subsequently proposed to understand the fundamentals of the enhanced ORR-kinetics. We elect a commercial MIEC comprised of LaxSr1−xGa0.83Mg0.17O3−δ and Gd0.2Ce0.8O1.9 (GDC) as the original cathode for the reason that the ORR-kinetics of this composite cathode is rate-limited by the dissociative adsorption of O2 at t ≤ 650°C.6–10 If the MC can indeed promote the rate of surface oxygen dissociative adsorption, the MC-modified LSCF + GDC cathode will show a lowered polarization resistance.

Experimental

Fabrication of symmetrical impedance cells.—The electrochemical performance of cathodes under investigation was evaluated with a symmetrical impedance cell configuration. The symmetrical impedance cell consisted of a dense LSGM cathode (La0.8Sr0.2Ga0.83Mg0.17O3−δ) electrolyte membrane and two identical thin films of porous LSCF+GDC cathode screen-printed on the two sides of the LSGM electrolyte. The LSGM electrolyte membrane made by tape-casting was approximately 15 μm in thickness after final sintering.

The ink used to screen-print the two identical layers of porous LSCF+GDC cathode on the LSGM was purchased from a commercial source (Sku: 232202, Fuel Cell Materials). The final tri-layer symmetrical cells were fired at 1100°C for 1 h to achieve a good adhesion of the cathode to the electrolyte. There were no concerns over chemical reactions between LSGM and LSCF at this temperature due to the excellent chemical compatibility between the two perovskite structured materials. The final cathodes were porous and had an effective surface area of 0.75 cm2 and thickness of 30 μm, respectively. For all electrochemical tests (≤650°C), silver paste (C8829, Heraeus) and silver mesh were used as the current collector.

Synthesis of Li-K carbonates modified and LSCF-infiltrated cathodes.—A series of binary Li-K carbonate compositions were prepared by melting a mixture of lithium carbonate (99%, Alfa Aesar) and potassium carbonate (99%, Alfa Aesar) at 650°C for 2 hours. The resultant melts were then broken into micron-size powders through ball milling, which were subsequently dispersed into ethanol under ultrasonic stirring to yield a suspension. A controlled amount of the suspension was then uniformly spread over the cathode surface using a pipette, followed by drying at 100°C. A final heat-treatment was carried out at 650°C for 2 hours to allow carbonates to melt and incorporate into pores of the cathode backbones. The compositions of the binary Li-K carbonate melts varied from Li/K = 10/90 to 16/84.
After each infiltration, the samples were fired at 500°C for 5 hours, and nitrates with a concentration of 0.5 mol·L⁻¹ were prepared by mixing reagent grade La(NO₃)₃·6H₂O (Alfa Aesar), Sr(NO₃)₂·6H₂O (Alfa Aesar), Co(NO₃)₂·6H₂O (Alfa Aesar) and Fe(NO₃)₃·6H₂O (Alfa Aesar) in a desirable stoichiometry to achieve the final nominal composition of La₀.₆Sr₀.₄Co₀.₂Fe₀.₈O₃₋ₓ (δ) or other compositions at Li/K ratios of 70/30, 30/70 and 10/90. Similar to Figure 1c, all the MC compositions led to reduction in RP and RO in compared to the pristine sample. The highest RP-reduction in Figure 2b is observed at 3.6 mg of MC mass-loading at 550, 600, and 650°C.

The mass loading effect of MC on area-specific resistance (ASR) of the cathode is shown in Figure 1. For this study, the MC composition was fixed at the eutectic composition of Li/K = 62/38 (mol%). At the first glance of EIS spectra at 600°C in Figure 1a, the total ASR of the pristine cathode is largely dominated by the high polarization resistance (ASR). The frequency was swept from 10⁷ to 0.01 Hz with 20 mV of stimulus AC amplitude. In order to avoid the interference from silver paste, the highest temperature of all the tests was limited to 650°C. The long-term stability of both original and MC-added cathodes was simultaneously evaluated at 600°C for a total of 1,500 hours with a side-by-side cell arrangement.

All of the MC compositions led to reduction in RP and RO in compared to the pristine sample. The highest RP-reduction in Figure 2b is observed at 3.6 mg of MC mass-loading at 550, 600, and 650°C. Figure 2a, extracted RP in Figure 2b and RO in Figure 2c, respectively. For this study, the MC loading is fixed at 3.6 mg·cm⁻². All of the MC compositions led to reduction in RP and RO in compared to the pristine sample. The highest RP-reduction in Figure 2b is observed on the eutectic composition Li/K = 62/38 (mol%), followed by compositions of Li/K = 30/70, 70/30 and 10/90. Similar to Figure 1c, RO does not show appreciable variations among compositions of Li/K = 70/30, 30/70 and 62/38, but indeed increase with the composition of Li/K = 10/90.

Figure 1. (a) EIS spectra measured from pristine and MC-modified LSCF+GDC cathodes at 600°C; (b) normalized RP and (c) RO as a function of MC mass-loading at 550, 600, and 650°C.

Electrochemical and microstructural characterizations.— The spectra of Electrochemical Impedance Spectroscopy (EIS) was collected from the fabricated symmetrical impedance cells using an electrochemical workstation (IM6, Zahner) under open-circuit conditions, and further analyzed with the Thale equivalent circuit software (Zahner) to extract electrode polarization resistance. The area specific resistance (ASR) was converted from the obtained raw polarization resistances by normalizing it to the actual cathode area (0.75 cm²). The frequency was swept from 10⁷ to 0.01 Hz with 20 mV of stimulus AC amplitude. In order to avoid the interference from silver paste, the highest temperature of all the tests was limited to 650°C. The long-term stability of both original and MC-added cathodes was simultaneously evaluated at 600°C for a total of 1,500 hours with a side-by-side cell arrangement.

The microstructures of cathodes in all the symmetrical impedance cells, either pretested or post-tested, were examined with a field-emission scanning electron microscopy (FESEM, Zeiss Ultra) equipped with an energy dispersive X-ray spectroscopy (EDS) analyzer.
LSCF-infiltrated counterparts. Although the two modified cathodes exhibit considerable reduction in $R_p$ over the pristine one, the degree of $R_p$- and $R_O$-reduction achieved by MC is far superior to that by infiltrated LSCF. Figure 3b summarizes all the $R_p$ and $R_O$ values obtained from three cathodes within 550–650°C. It is clear that the MC is an excellent catalyst for ORR, exhibiting the lowest $R_p$ and $R_O$

It should be noted that preparation of MC-modified cathode is a rather easy process. It can be performed by single-step dispersion of a small fraction of MC ethanol suspension into the cathode backbones, followed by a one-time heat-treatment at 650°C. In contrast, the solution-infiltrated LSCF cathode requires a repetitive and tedious process to prepare, making it production unfriendly.

**Long-term stability.**—Long-term stability of a cathode is an important requirement for SOFCs to be a commercial product. To demonstrate the suitability of MC-modified cathodes for practical long-term uses, the $R_p$ and $R_O$ of the pristine and MC-modified cathodes were constantly monitored at 600°C over 1,500 hours shown in Figure 4. The behavior of the pristine sample can be characterized by an initial decrease in $R_p$ and $R_O$ for the first 300-hour, followed by a gradual increase. Such an early-stage reduction in $R_p$ and $R_O$ is attributed to the well-known “break-in” phenomenon involving interfacial reconfiguration. Since there is no obvious microstructural change found (see later Figure 5), the increases in $R_p$ and $R_O$ of the pristine LSCF cathode are likely attributed to the Sr segregation on the surface of cathode as suggested in the literature. In contrast, the MC-modified cathode shows not only much lowered $R_p$ and $R_O$ but
also no “break-in” effect. The absence of the “break-in” effect for the MC-modified cathode implies that the cathode may have been pre-activated by the presence of MC phase during the sample preparation. The similar rate of $R_p$-increase observed for both samples during the entire testing period seems to suggest a similar degradation mechanism, i.e., Sr-segregation. Furthermore, lower and smoother variation of $R_D$ of the MC-modified sample than the pristine one during the test also suggests that the effective conductivity of the cathode has been enhanced by MC.

**Microstructural observations.**—To further facilitate the understanding of MC-promoted ORR kinetics, the microstructural features of pristine and MC-modified cathodes before and after 1,500-hours testing were characterized with SEM/EDS. The cross-sectional views of microstructures near the cathode/electrolyte interface of the two samples are shown in Figure 5. The pristine cathode in Figure 5a and 5b shows a typical porous microstructure with connected LSCF and GDC networks. After the 1,500-hours test at 600°C, no obvious difference in microstructure can be discerned. However, for the MC-modified cathode, a clearly distinguishable microstructure is observed in Figure 5c and 5d. For the pre-test sample, randomly distributed needle-like species are seen over the internal surfaces of cathode backbones. Local EDX analysis indicated that these slender needles belong to the Li/K carbonates. The EDX analysis on the post-tested sample (1,500-hours), however, revealed no trace of carbonates on the surfaces of cathode grains. The surface of the MC-modified sample seems uneven in Figure 5d, a sign of MC dissolution into LSCF. Decomposition of carbonates into oxide and CO$_2$ should not be the cause for the MC disappearance as it has been previously reported in the literature that 0.03% CO$_2$ in air is sufficient to prevent the decomposition. While the exact chemical change occurred within the cathode during testing is unclear and worth further investigating, it is interesting to note that alkaline-earth and rare-earth elements such as those used in LSCF and GDC have been reported as the enhancers for oxygen solubility in MC.

**Single cell performance.**—Figure 6 compares the power characteristic of the pristine and MC-modified single SOFCs. Both cells exhibited reasonable OCV, indicating a good sealing. The performance of the pristine cell was very poor, in part due to the use of a thicker LSGM membrane, but mainly due to the poor cathode performance as was also confirmed by the EIS results of half-cell impedance.

![Figure 4](image-url)  
*Figure 4.* Long term stability of $R_p$ and $R_D$ of pristine and MC-modified LSCF+GDC cathodes evaluated at 600°C; (a) $R_p$, (b) $R_D$.  

![Figure 5](image-url)  
*Figure 5.* Microstructural changes of the cathodes recorded before and after 1,500-h test at 600°C. Pristine cathode: (a) before and (b) after 1500-h test; MC-modified cathode: (c) before and (d) after 1500-h test.
analysis shown in the previous section. A factor of three performance improvement by MC-incorporation in cathode microstructure is seen from Figure 6, although the overall performance was not as high as expected.

**Discussion**

*New ORR charge-transfer model involving MC phase.*—The advantages of MC as an effective ORR-catalyst to reduce $R_p$ and $R_o$ of the pristine LSCF+GDC cathode are clearly demonstrated. To understand how MC involves in and promotes the ORR kinetics, a new charge-transfer model is herein proposed. The model is centered on the hypothesis of two oxygen-rich peroxycarbonate-ions $\text{CO}_5^{2-}$ and $\text{CO}_4^{2-}$ species as the fast oxygen absorber and transporter, respectively. Figure 7a illustrates schematically the concept of the chemisorption of $\text{CO}_2$ on the surface of MC and subsequent reaction with $\text{CO}_5^{2-}$ to form $\text{CO}_5^{2-}$ and $\text{CO}_4^{2-}$ as the intermediate species, respectively.

The theoretical support to the proposed ORR charge-transfer model is provided by DFT calculations. First, the surface chemisorption of $\text{O}_2$ molecules on MC is a thermodynamically favorable process as indicated by the negative Gibbs free energy at the standard state ($\Delta G^\circ = -101.6 \text{kJ/mole}$) for the reaction of $\text{CO}_3^{2-} + \text{O}_2 = \text{CO}_5^{2-}$. Partial charge-transfer occurred from MC to oxygen during the formation of $\text{CO}_5^{2-}$ lowers the energy of stabilization. Second, the equilibrium constant $K = 1.1$ at 873K for the subsequent reaction of $\text{CO}_5^{2-}$ with $\text{CO}_3^{2-}$, i.e., $\text{CO}_5^{2-} + \text{CO}_3^{2-} = 2\text{CO}_4^{2-}$, suggests a good chemical stability of $\text{CO}_4^{2-}$ in the bulk of MC. Third, Figure 7b of molecular orbitals and energy eigenvalues calculated by DFT shows that $\text{CO}_5^{2-}$ has the lowest reduction potential of oxygen compared to $\text{CO}_5^{2-}$ and $\text{O}_2^{2-}$, suggesting $\text{CO}_5^{2-}$ as a likely active species to be involved in the charge-transfer step of the ORR. From these preliminary DFT calculations, it is reasonable to assert that $\text{CO}_5^{2-}$ and $\text{CO}_4^{2-}$ other than $\text{O}_2^{2-}$ or $\text{O}_3^{2-}$ play an active role in the MC-involved ORR kinetics.

The experimental support to the proposed charge-transfer model is the spectroscopic observation of $\text{CO}_4^{2-}$ and the measured high oxygen solubility in MC. The strong affinity of $\text{O}_2$ to MC in the form of $\text{CO}_5^{2-}$ can lead to a very high sticking coefficient of oxygen molecules onto the surface of MC. On the contrary, classical
dissociative chemisorption of gas molecules on ionic solid surfaces typically has a very low sticking coefficient, thus severely limiting the rate of chemisorption.

The migration of $\text{CO}_2^{2-}$ from the $\text{O}_2$/MC surface of chemisorption to the MC/LSCF interface of $\text{CO}_2^{2-}$ reduction can be achieved through a cooperative “cogwheel” (or “paddle-wheel”) mechanism shown in Figure 8, involving the breaking and reforming $\text{O}-\text{CO}_3^{2-}$ bond. Similar mechanism has also been previously proposed for the superionic conduction phenomenon in fast alkali-metal ionic conductors with rotationally disordered complex anions and Ga-based oxides with tetrahedral moieties. The weakly bonded O in $\text{CO}_2^{2-}$ ($\text{O}-\text{CO}_3^{2-}$) ions can swiftly migrate toward the MC/LSCF interface, whereas $\text{CO}_3^{2-}$ ions return to the MC bulk to replenish the preceding reactions. Again, the electrons transfer is a much faster process at liquid/solid interfaces than at gas/solid interfaces, resulting in facile ORR kinetics. Figure 9 illustrates schematically the five basic steps of the MC-involved ORR, along with the conventional gas/solid charge-transfer pathway, and Table I further summarizes the elementary ORR steps stated above involving $\text{CO}_2^{2-}$ and $\text{CO}_4^{2-}$ as the intermediate species.

Application of the new ORR charge-transfer model to Figure 2, the observed lowest $R_p$ at the eutectic composition can then be understood as a result of the maximum amount of melt available for catalyzing the ORR. Other compositions are two-phase mixtures according to the phase diagram, containing a portion of solid phase that is inactive to the ORR. On the other hand, finely dispersed MC particles within

Table I. A new charge-transfer model for MC-promoted ORR.

<table>
<thead>
<tr>
<th>Step</th>
<th>Reaction</th>
</tr>
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<tbody>
<tr>
<td>Step-❶</td>
<td>$\text{CO}_2^{2-} (\text{MC}) + \text{O}_2(\text{air}) = \text{CO}_2^{4-} (\text{MC})$</td>
</tr>
<tr>
<td>Step-❷</td>
<td>$\text{CO}_2^{4-} (\text{MC}) + \text{CO}_2^{2-} (\text{MC}) = 2\text{CO}_2^{3-} (\text{MC})$</td>
</tr>
<tr>
<td>Step-❸</td>
<td>$\text{CO}_2^{3-} (\text{O}_2/\text{MC}) = \text{CO}_2^{3-} (\text{MC}/\text{LSCF})$</td>
</tr>
<tr>
<td>Step-❹</td>
<td>$\text{CO}_2^{3-} (\text{MC}) + 2e^- (\text{LSCF}) = \text{CO}_2^{2-} (\text{MC}/\text{LSCF})$</td>
</tr>
<tr>
<td>Step-❺</td>
<td>$\text{O}_2(\text{LSCF}) = \text{O}_2^{2-} (\text{LSCF})$</td>
</tr>
</tbody>
</table>

Overall ORR at LSCF cathode:
$\text{O}_2(\text{air}) + 4e^- (\text{LSCF}) = 2\text{O}_2^{2-} (\text{LSCF})$
the porous backbones of the cathode are of high preference in terms of maximizing the reactive area and shortening the distance for CO$_2^-$ to migrate. Overloading the MC in the cathode’s porous backbones may, therefore, reduce the reactive area and lengthen the migrating distance of CO$_2^-$, thus causing impedance to charge-transfer. This is why an optimal MC mass-loading is observed in Figure 1.

$R_0$ reduction by MC.—The $R_0$ measured from the symmetrical impedance cells is the sum contributions from electrolyte, cathode, interfacial contacts, current-collector and measuring leads. Since there are nearly no difference in electrolyte, current-collector and electrical contact among cells tested, the observed reduction in $R_0$ by MC must be originated from the cathode itself. However, the relatively low conductivity of MC phase (e.g., 1.1 S·cm$^{-1}$ at 600°C) in comparison to that of LSCF cathode is not sufficient to justify MC as the sole source for the reduced $R_0$. Formation of more conductive phase and/or to that of LSCF cathode is not sufficient to justify MC as the sole source for the reduced $R_0$. Formation of more conductive phase and/or to that of LSCF cathode is not sufficient to justify MC as the sole source for the reduced $R_0$. 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