**Quasi-Solid-State Li−O2 Batteries with Laser-Induced Graphene Cathode Catalysts**

Muqing Ren, Jibo Zhang, Chenhao Zhang, Michael G. Stanford, Yieu Chyan, Yan Yao,* and James M. Tour*  

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**ABSTRACT:** Li−O2 batteries are attracting considerable interest due to their outstanding theoretical capacities and energy densities. The liquid electrolyte and insulating nature of discharge products hinder further development. We present a practical strategy for durable Li−O2 batteries with high cyclable capacity up to 2 mAh/cm². A dual polymer gel electrolyte (DPGE) networking strategy is proposed for the quasi-solid electrolyte. Lowering the use of liquid components in the electrolyte would suppress the possible side reaction with superoxide intermediates. The DPGE is further combined with a highly effective Mn-based catalyst that is prepared by direct laser writing on polymers to produce the quasi-solid-state Li−O2 batteries. The DPGE sustains a highly reversible Li plating/stripping process without short-circuiting or an increase in interfacial resistance for over 2000 h. Furthermore, the Li−O2 battery demonstrated stable galvanostatic charge/discharge performance for over 200 cycles (2000 h) with a cutoff capacity of 0.4 mAh/cm². The battery delivers a high reversible capacity of 2.0 mAh/cm² at an elevated current density of 0.4 mA/cm², while the charging potential is maintained at a low value. Studies were performed during discharge and charge process to investigate the underlying mechanism. This work could contribute to a better practical design of Li−O2 batteries.

**KEYWORDS:** Li−O2 batteries, dual polymer gel electrolyte, laser-induced graphene, LIG, oxygen reduction reaction

**INTRODUCTION**

The increasing demand for energy resources encourages the development of high energy density storage systems. During recent years, Li−O2 batteries are attracting considerable interest due to their outstanding theoretical capacities and energy densities. Combining the Li metal anode with O2 as the cathode, a Li−O2 battery can, in principle, deliver a specific energy density of ~3500 Wh/kgLi2O2, which is several times higher than conventional Li-ion batteries (LIBs). Since the first demonstration of a rechargeable Li−O2 battery by Abraham and Jiang in 1996, an improved rechargeable Li−O2 battery was presented by Bruce and co-workers in 2006. The applicability of a delicately designed air cathode was then illustrated, and studies on nonaqueous Li−O2 batteries have ensued. The use of metallic Li and liquid electrolytes in Li−O2 batteries has introduced the same issues found in LIBs, such as electrolyte leakage, poor stability, flammability and volatility of the electrolyte, and Li dendrite growth. The sluggish oxygen electrochemistry at the O2 cathode contributes to additional problems, including the accumulation of discharge products and blockage of O2 diffusion. Moreover, the intermediate oxygen radical anion, superoxide (O2−), formed during the reduction process is active in decomposing the organic carbonate electrolyte and presents a challenge when using conventional electrolytes. The use of solid-state electrolytes over liquid electrolytes has been considered as an efficient method to overcome the safety issues caused by organic solvents. The well-studied inorganic solid electrolytes, including lithium superionic conductors such as garnets, exhibit stable potential window and high ionic conductivity for Li−O2 electrochemistry. However, poor interfacial properties raise the interface impedance and block ionic transport, which is essential for the cathode reactions, since Li2O2 has very low Li+ ionic and electronic conductivities. Although recently developed sulfide-based compounds have demonstrated high ionic conductivity and modulus as solid electrolytes, they exhibit high sensitivity to O2 and moisture. Alternatively, polymer gel electrolytes (PGEs) might provide a better electrolyte—electrode interface due to their viscoelasticity and low modulus. Moreover, PGEs were developed to improve the ionic conductivity and solvation of Li+ while maintaining a relatively high mechanical strength. Yet, when considering incorporation of PGEs with the O2 cathode, the high charging potential (due to the high
overpotential of O2 electrochemistry) would not only challenge the anodic stability of the electrolytes but also diminish the energy efficiency of the Li−O2 battery. The incorporation of a redox mediator, for example, LiI, InI3, and tetrathiafulvalene, into the electrolytes could facilitate the decomposition of Li2O2 and lower the charge potential, at the possible cost of migration of the redox mediator toward the anode and subsequent passivation of Li anode.30 Therefore, a careful consideration of the overall design and components of the Li−O2 battery is necessary when considering the use of PGEs.

We demonstrate a strategy toward practical Li−O2 batteries using a dual polymer gel electrolyte (DPGE) with an efficient MnO2/laser-induced graphene (LIG) cathode catalyst. In this design, the commercially available poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF−HFP) was used as the backbone of the DPGE to provide mechanical stability. Tris(methyloxytetraacrylate (TEMPT) was UV-polymerized in situ inside the PVDF−HFP network, providing a high ionic (Li+) conductivity due to abundant ethylene oxide moieties that efficiently solvate Li+ ions in the gel electrolyte. The DPGE demonstrated high stability through a >2000 h Li plating/stripping process without short-circuiting or increasing the interfacial resistance. The DPGE was further processed noble-metal-free catalysts contributes a new direction to the development of practical Li−O2 batteries.

RESULTS AND DISCUSSION

The synthesis of the DPGE was assisted by UV-activating polymerization of TEMPT with PVDF−HFP as the backbone, as shown in Figure 1a. Briefly, PVDF−HFP was dissolved in N-methyl-2-pyrrolidinone (NMP). This solution was mixed with lithium bis(trifluoromethanesulfonimidate/tetraethylene glycol dimethyl ether (LiTFSI/TEGDME). After addition of 2-hydroxy-2-methyl-1-phenyl-1-propanone (HMPP, photoinitiator) and TEMPT to the solution, it was cast on top of a glass substrate and cured with UV irradiation. The DPGE obtained through this method was semitransparent with a typical thickness of ∼287 µm (Figure 1b) by scanning electron microscopy (SEM). The X-ray photoelectron spectroscopy (XPS) results shown in Figure S1 demonstrate the distribution of Li+ in the polymer network. Figure 1c displays the cross-sectional morphology of DPGE that exhibits a dense structure, indicating the well-formed interpenetrating polymer network between PVDF−HFP and PEG. The broad and weak diffraction peak in Figure S2 suggests there is no significant aggregation/crystallization of PVDF−HFP or PEG in the DPGE. The slightly rough surface may be beneficial for interfacial contact with the O2 cathode, as it provides a highly active surface with the cathode catalysts to facilitate the Li+ transportation. The Li+ transference number (tLi+) and conductivity of the DPGE are summarized in Figure 1d. The DPGE exhibits a high tLi+ of 0.6 and a total conductivity of 3.25×10−4 S/cm at room temperature, which are comparable to the liquid electrolytes. The electrolyte’s electrochemical potential window is a critical factor to ensure Li−O2 battery...
cycling reversibility. Therefore, linear sweep voltammogram (LSV) testing was performed on a Li/DPGE/stainless steel cell to test DPGE’s anodic stability between 2.0 and 5.0 V, as shown in Figure S3. No obvious decomposition was observed before the voltage reaches 4.7 V, which demonstrates the chemical stability of DPGE in withstanding a high oxidation potential. A Li/DPGE/Li cell was fabricated to examine the compatibility of the DPGE with metallic Li. As shown in Figure S4, no short-circuit or buildup of interfacial resistance was observed after a 2000 h galvanostatic test.

Because of the sluggish O2 electrochemistry at the O2 cathode, an efficient bifunctional catalyst is desired to lower the overpotentials in both discharge and charge process. Instead of incorporating redox mediators (RMs, also known as soluble homogeneous catalysts in aprotic Li−O2 batteries) in the electrolytes, we proposed to use a porous heterogeneous catalyst at the cathode to avoid possible “crosstalk” between Li and RMs. MnO2 has shown high activity and durability in electrocatalysis and therefore was selected as the cathode catalyst for our study. The in situ synthesis of MnO2/LIG catalysts was done by a relasing method as previously reported (Scheme 1).35,36 PI is lased to make LIG and then exposed to an oxygen plasma to increase the hydrophilicity. An aqueous solution of the MnSO4 is applied, and then the LIG relased under the same conditions. The MnO2/LIG is then scraped from the PI surface to afford the active catalytic material. The morphology structure of MnO2/LIG catalyst was characterized by SEM (Figure 2a−c). The SEM images show that the typical porous structure of LIG was maintained after MnO2 was in situ grown by direct laser writing. Additionally, there is no obvious aggregation of MnO2 nanoparticles found on the LIG, indicating the homogeneous distribution of nanoparticles. The 3D porous structure will benefit the diffusion of O2 to the active catalyst, which also benefits contact between the electrode and DPGE. The LIG will contribute to a high discharge capacity since the capacity is determined by the porous cathode catalyst structure’s ability to accommodate the accumulation of discharged products.37,38

Transmission electron microscope (TEM) images demonstrate that MnO2 nanoparticles were homogeneously distributed on the graphene, as shown in Figure 2d−f. The nanoparticles exhibit a uniform size distribution of ~10 nm. The high-resolution TEM (HRTEM) images reveal that the lattice spacing of MnO2 is ~0.24, 0.30, and 0.49 nm, respectively. These values correspond to the interplane spacing of the (211), (310), and (200) planes of α-MnO2 (PDF44-0141), which is in accordance with the X-ray diffraction (XRD) data (Figure 3a). It further confirms the formation of MnO2 nanoparticles embedded on graphene structures. The multilayer graphene structure arranged around the metal oxides is consistent with the Raman data (Figure 3b). The results confirm that the nanoparticles and LIG were formed during the direct laser writing. Figure 3a shows the crystalline structure of the catalysts. The characteristic peak of LIG is observed at 25.6°, corresponding to the multilayer graphene structure. The crystal planes of (200), (310), (211), (301), and (510) are well-defined from the XRD pattern and match the lattice spacing of α-MnO2. The intensities of these peaks are relatively weak, which may be caused by the small size of the crystallized nanoparticles and rapid coverage of LIG on the surface of MnO2.

The Raman-active mode located at 643 cm−1 in Figure 3b is attributed to the symmetric stretching vibration (Mn−O) of the MnO6 octahedron. This result is consistent with the XRD evidence that MnO2 is the primary phase of manganese species in the catalyst. The clearly identified D, G, and 2D peaks suggest the existence of defective or bent multilayer graphene as the characteristic sign of LIG.

The elemental composition and chemical valence state were analyzed by X-ray photoelectron spectroscopy (XPS). A XPS survey spectrum of the MnO2/LIG catalyst illustrates characteristic Mn, O, and C species (Figure 3c). Figure 3d presents the high-resolution XPS spectrum in the Mn 2p
region for the MnO₂/LIG catalyst. It has spin–orbit split peaks of Mn 2p₃/2 and 2p₁/2 at 642.1 and 653.8 eV, respectively. The spin-energy separation is 11.7 eV, which suggests the existence of Mn⁴⁺ species. The O 1s spectra show a broad peak with
of the MnO2 surfaces to O2, which is a key procedure of O2 window (0.25-0.4 V). The Koutecky-Levich (K-L) equation ( Experimental Section), and the results are plotted in Figure 4b. In the potential window (0.25–0.55 V vs RHE), the average n for MnO2/LIG is ~4.0, suggesting a predominantly direct four-electron transfer process. These results demonstrate the strong affinity of the MnO2 surfaces to O2, which is a key procedure of O2 reduction in aprotic electrolyte. Figure 4c shows the OER performance of MnO2/LIG. The onset potential is ~1.5 V, corresponding to an overpotential of 270 mV. The Tafel slope is ~148 mV/dec ( Figure 4d), and the potential to reach 10 mA/cm2 is 1.85 V. The effect of precursor concentration on the morphology and the activity of MnO2/LIG catalyst was investigated ( Figures S7 and S8). It is concluded that a moderate loading of nanoparticles on LIG could ensure a uniform distribution of the active sites to avoid aggregation and maximize their ability to deliver a high mass activity. The control LIG exhibited much lower performance than MnO2/LIG in ORR and OER, suggesting a small contribution from the LIG structure on the intrinsic catalytic activity ( Figure S9).

The MnO2/LIG exhibits comparable performance to the Mn-based catalysts synthesized by conventional methods ( Tables S1 and S2), demonstrating the effectiveness and reliability of the LIG method for the facile preparation of catalysts. The nano- and mesoporous structure from LIG lead to higher surface area and better conductivity whereas the uniform distribution of nanosized MnO2 particles provides abundant surface active sites toward the O2 electrochemical process in a Li–O2 battery. Based on MnO2/LIG activity toward both OER and ORR performance, it was further applied in a quasi-solid-state Li–O2 battery to enhance the cycle reversibility and to study the mechanism.

To explore the potential application of the bifunctional catalysts MnO2/LIG and solid electrolyte DPGE in Li–O2 batteries, the cells were tested through galvanostatic discharge and charge processes. The typical Li–O2 coin cell configuration consists of a Li foil as the anode, a piece of DPGE as the separator, and MnO2/LIG as the cathode catalyst, and dry O2 is maintained during the test as the cathode. Figure 5a shows the representative first discharge and charge voltage profiles for the MnO2/LIG electrode at various current densities with time limited to 5 h. The first cyclic voltage gap is 0.92 V when the current density is 0.08 mA/cm2; this voltage gap increased to 1.34 V at a current density of 0.4 mA/cm2. The MnO2/LIG exhibits comparable performance to the Mn-based catalysts synthesized by conventional methods (Tables S1 and S2), demonstrating the effectiveness and reliability of the LIG method for the facile preparation of catalysts. The nano- and mesoporous structure from LIG lead to higher surface area and better conductivity whereas the uniform distribution of nanosized MnO2 particles provides abundant surface active sites toward the O2 electrochemical process in a Li–O2 battery. Based on MnO2/LIG activity toward both OER and ORR performance, it was further applied in a quasi-solid-state Li–O2 battery to enhance the cycle reversibility and to study the mechanism.

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initial step of discharging, and then the sites with slightly lower activity would be accessed. Therefore, the former ones were preferentially decomposed at lower overpotential in the charge process. If there is heterogeneous precipitation of the discharge products during the discharge process (e.g., the accumulation of discharge products on the sites that are already covered), the charge process would exhibit a high overpotential even at the beginning due to the low electronic/Li$^+$ conductivity of the products, namely Li$_2$O$_2$, LiOH, or both.

Figure 5b shows the rate performance of MnO$_2$/LIG electrode for quasi-solid-state Li–O$_2$ battery (0.08–0.4 mA/cm$^2$) with a step current density of 0.08 mA/cm$^2$. At each representative current density, the battery was tested for five cycles. It is observed that the charged voltage potential gradually increased while the discharged voltage potential gradually dropped with the increased current densities. This could be attributed to the accumulation of insulating discharge product that results in higher overpotential and decomposition and the increasing difficulty for O$_2$ to diffuse through the discharged products to react with Li ions. The battery was discharged and charged for $\sim$200 cycles at the current density of 0.08 mA/cm$^2$. The MnO$_2$/LIG electrode for the Li–O$_2$ battery with a conventional Celgard separator and liquid electrolyte (1 M LiTFSI/TEGDME) was also tested at the current density of 0.08 mA/cm$^2$ for comparison (Figure S10). Although the battery shows a lower discharge–charge overpotential at the first cycle, its charging potential increased quickly with cycling. The charging potential reached over 4.5 V at the 80th cycle. The quasi-solid-state Li–O$_2$ battery with DPGE can be recharged for more times than with the conventional liquid electrolyte. The $\alpha$-MnO$_2$ is intrinsically active for O$_2$ electrochemistry in aprotic Li–O$_2$ batteries.\cite{39,40,41}

The improved cycling durability with DPGE further highlights the importance of a stable electrolyte system for a practical Li–O$_2$ battery. Figures 5c,d show the battery’s cyclic performance under a large current density of 0.4 mA/cm$^2$. The cells can be cycled $\sim$50 times with the cutoff capacity of 2 mAh/cm$^2$ without significant increase of the overpotentials (Table S3).

The morphology and crystal structure change of the electrode were investigated through SEM, XRD, and XPS characterizations, which help to further understand the reaction process inside the quasi-solid-state Li–O$_2$ battery. Small crystal particles were formed when the MnO$_2$/LIG electrode was discharged for 125 h (10 mAh/cm$^2$, Figure 6a), and the particles were decomposed after being charged back for 125 h (Figure 6b). Figure 6c demonstrates the crystalline structure of the MnO$_2$/LIG electrode after discharge (125 h) and charge (125 h). The characteristic peak of multilayer graphene (LIG, 25.6°) was maintained during the discharge and charge. The discharge products are identified as LiOH and Li$_2$O$_2$. Li$_2$O$_2$ is produced from the successive reduction of O$_2$ to O$_{2^-}$ and O$_{2^{2-}}$. LiOH is formed through several possible pathways, e.g., reaction with a trace amount of H$_2$O on the surface of the DPGE that might come from the air during the DPGE synthesis, moisture in the O$_2$, and reaction of superoxide with the PVDF binder. From a previous study, it was found that Mn species facilitate the decomposition of H$_2$O$_2$ and move the reaction forward to result in the formation of LiOH.\cite{42,43} Despite the dual discharge products, the catalyst could facilitate the decomposition process and enhance the cycling stability. During the charge process, all of the discharged Li$_2$O$_2$ and LiOH were decomposed. Figure 6d illustrates the Li 1s XPS survey corresponding to the electrode discharged (125 h) and then charged (125 h) states. The Li 1s XPS has a main peak at $\sim$54.4 eV, which is ascribed to Li$_2$O$_2$ and LiOH. Li$_2$O$_2$ and LiOH have the same Li 1s binding energy, and it is difficult to distinguish those two from XPS characterization.

In our previous study of Li–O$_2$ batteries with glyme-based electrolytes,\cite{44} we found that the surface active sites are of great importance in modulating the size and distribution of discharge products. Because of the low donor number of glymes and high O$_2$ solubility in electrolyte (e.g., 0.6 mM in 1 M LiTFSI/G4), the nucleation process of LiO$_2$ highly depends on the accessible sites on the cathode/electrolyte interface. Similarly, in the quasi-solid-state Li–O$_2$ battery, where the O$_2$ electrochemical reactions take place at the electrolyte/electrode surface, a high density of accessible sites is a necessity for a high reversible capacity. Otherwise, the discharge products would fill the sites and lead to either high overpotentials for Li$^+$/e$^-$ conduction and/or blockage of O$_2$. Another key factor is the compatible interfacial contact between solid electrolyte and cathode. The intrinsic viscoelasticity and low modulus of the polymer-based quasi-solid electrolyte would alleviate the interfacial tension and possible loss of conductive contact due to volume change through cycling. Therefore, we suggest the use of porous cathode catalysts with the solid electrolytes for practical Li–O$_2$ batteries. The utilization of quasi-solid electrolyte has minimized the utilization of liquid electrolyte, the latter of which is unstable against superoxide as well as high anodic potentials. The incorporation of efficient cathode catalyst further lowers the overpotentials and enables a long cycle life of the batteries. However, although the $t_{Li^+}$ of the DPGE is higher than the liquid electrolytes, the overall Li$^+$ conductivity is still lower than that of the liquid electrolytes, which could lead to high overpotentials under high current densities. Hence, future optimization should be focused on improving the $t_{Li^+}$ and Li$^+$ conductivity as well as the activity of the cathode catalyst.
CONCLUSION
In this study, we demonstrated a quasi-solid-state Li−O2 battery with DPGE as the separator and solid-phase synthesized bifunctional catalyst MnO2/LIG as cathode catalyst. Benefiting from the intrinsic high activity of LIG and stability of the DPGE, the Li−O2 battery demonstrated enhanced performance in cycling life. The quasi-solid-state battery cycled ~200 times with a cutoff capacity 0.4 mAh/cm2 and cycled ~50 times with a high cutoff capacity 2.0 mAh/cm2 at a high current density of 0.4 mAh/cm2. The results here suggest that further developments in carbon−metal oxide composite cathodes are warranted in the quest to fabricate Li−O2 batteries with markedly higher energy density than most current commercial systems.

ASSOCIATED CONTENT

Supporting Information
The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsaem.9b02182.

Experimental details, additional SEM/TEM images, Raman spectra, XPS data, and electrochemical test results (PDF)

AUTHOR INFORMATION

Corresponding Authors
Yan Yao — Department of Electrical and Computer Engineering and Texas Center for Superconductivity, University of Houston, Houston, Texas 77204, United States; orcid.org/0000-0002-8785-5030; Email: yyao4@uh.edu

James M. Tour — Department of Chemistry, Smalley-Curl Institute and NanoCarbon Center, and Department of Materials Science and NanoEngineering, Rice University, Houston, Texas 77005, United States; orcid.org/0000-0002-8479-9328; Email: tour@rice.edu

Other Authors
Muqing Ren — Department of Chemistry, Rice University, Houston, Texas 77005, United States; orcid.org/0000-0003-1685-3764

Jibo Zhang — Department of Electrical and Computer Engineering and Texas Center for Superconductivity, University of Houston, Houston, Texas 77204, United States; orcid.org/0000-0002-9966-8195

Chenhao Zhang — Department of Chemistry, Rice University, Houston, Texas 77005, United States

Michael G. Stanford — Department of Chemistry, Rice University, Houston, Texas 77005, United States; orcid.org/0000-0001-9663-1138

Yieu Chyan — Department of Chemistry, Rice University, Houston, Texas 77005, United States

Complete contact information is available at: https://pubs.acs.org/10.1021/acsaem.9b02182

Author Contributions
M.R. and J.Z. contributed equally to this work.

Notes
The authors declare the following competing financial interest(s): Rice University owns intellectual property (IP) on the LIG process. The IP is being licensed to a company in which none of the authors are officers or directors, but J.M.T. is a stockholder. All potential conflicts are disclosed to and managed by the Rice University Office of Sponsored Projects and Research Compliance.

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