



Short communication

A high energy density lithium/sulfur–oxygen hybrid battery

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ABSTRACT

In this paper we introduce a lithium/sulfur–oxygen (Li/S–O₂) hybrid cell that is able to operate either in an air or in an environment without air. In the cell, the cathode is a sulfur–carbon composite electrode containing appropriate amount of sulfur. In the air, the cathode first functions as an air electrode that catalyzes the reduction of oxygen into lithium peroxide (Li₂O₂). Upon the end of oxygen reduction, sulfur starts to discharge like a normal Li/S cell. In the absence of oxygen or air, sulfur alone serves as the active cathode material. That is, sulfur is first reduced to form a soluble polysulfide (Li₂S_x, $x \geq 4$) that subsequently discharges into Li₂S through a series of disproportionations and reductions. In general, the Li/S–O₂ hybrid cell presents two distinct discharge voltage plateaus, i.e., one at ~ 2.7 V attributing to the reduction of oxygen and the other one at ~ 2.3 V attributing to the reduction of sulfur. Since the final discharge products of oxygen and sulfur are insoluble in the organic electrolyte, it is shown that the overall specific capacity of Li/S–O₂ hybrid cell is determined by the carbon composite electrode, and that the specific capacity varies with the discharge current rate and electrode composition. In this work, we show that a composite electrode composed by weight of 70% M-30 activated carbon, 22% sulfur and 8% polytetrafluoroethylene (PTFE) has a specific capacity of 857 mAh g⁻¹ vs. M-30 activated carbon at 0.2 mA cm⁻² in comparison with 650 mAh g⁻¹ of the control electrode consisting of 92% M-30 and 8% PTFE. In addition, the self-discharge of the Li/S–O₂ hybrid cell is expected to be substantially lower when compared with the Li/S cell since oxygen can easily oxidize the soluble polysulfide into insoluble sulfur.

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1. Introduction

Sulfur and oxygen are among the highest theoretical capacities for the cathode of primary lithium batteries in comparison with all other cathode materials such as carbon monofluoride (CF_x, $x = 1$), SO₂, SOCl₂, and MnO₂. Based on complete reactions with metal lithium to form Li₂S and Li₂O, they have a theoretical specific capacity of 1675 mAh g⁻¹ vs. sulfur and 3351 mAh g⁻¹ vs. oxygen, respectively. Due to the high theoretical capacity and low cost of sulfur, the lithium/sulfur (Li/S) battery has been long studied as a rechargeable battery [1–11]. In the nature sulfur exists in more than 30 allotropes, of which ring-structural cyclooctasulfur (S₈) is the most stable form [12]. In discharge of a fresh Li/S battery, the ring-structural sulfur (S₈) is first broken an S–S covalent bond to form a chain-structural polysulfide anion (S_x²⁻, $x = 8$), which is further reduced into Li₂S through multistage reactions including a series of disproportionations and reductions [2,10]. Due to their high solubility in the organic electrolyte, polysulfide anions (S_x²⁻, $x \geq 4$) are easy to dissolve into the electrolyte, migrate to the surface of the lithium anode, and react with metal lithium. Therefore, the

cycling efficiency and cycle life of rechargeable Li/S batteries are rather poor. For the same reason, the Li/S battery suffers very high self-discharge. It has been reported that the self-discharge rate of a rechargeable Li/S battery can reach 8–15% per month [6]. Therefore, the Li/S battery is not suitable for a high energy density primary battery that is required to have long calendar life and service time.

On the other hand, the practical capacity of a Li/oxygen battery does not correspond to the theoretical capacities of oxygen and metal Li because of these reasons: (1) catalytic reduction of oxygen by carbon in the organic electrolyte takes place mainly through a two-electron process, only small amount of Li₂O₂ formed by the reduction of oxygen can be further reduced into Li₂O [13–16], and (2) the discharge products (Li₂O₂ and Li₂O) of oxygen are insoluble in non-aqueous organic electrolyte, they deposit on the surfaces of the catalytic sites of carbon and block oxygen from accessing to the reaction sites. Therefore, the real capacity of a Li/oxygen battery is determined by the carbon air electrode, or more specifically by the pore volume accessible for the deposition of discharge products. In addition, the operation of a Li/oxygen battery requires an oxygen or air environment, which is not the case in many applications.

With the facts above in mind, in this work we attempt to develop a lithium/sulfur–air hybrid cell by using a low sulfur-content car-

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bon composite cathode as the air electrode. Such a cell is able to operate either in air or in the environment without air. Since oxygen has a higher electrode potential than polysulfide, it can easily oxidize the soluble polysulfide to insoluble sulfur. Therefore, the lithium/sulfur–air hybrid cell is expected to have substantially lower self-discharge than the Li/S cell while still retaining the nature of a Li/air and a Li/S cell. In this paper we will present and discuss the discharge characteristic of Li/S–O₂ hybrid cells. To avoid the effects of moisture and carbon dioxide in air, we sealed the cell in a small oxygen plastic bag, and called it a lithium/sulfur–oxygen (Li/S–O₂) cell.

2. Experimental

Sulfur (>99.5%, m.p. = 112.8 °C, Aldrich) and M-30 activated carbon having a specific surface area of 2500–3200 m² g⁻¹ and a mean diameter of 25–30 μm (Osaka Gas Chemicals Co. Ltd.) were used as the active materials of sulfur–carbon composite air electrode. Using polytetrafluoroethylene (PTFE) emulsion (Teflon®, solid content = 61.5%, DuPont Co.) as the binder, a free-standing sulfur carbon composite electrode with a composition of 70M-30/22S/8PTFE by weight was prepared as follows. A calculated amount of S and M-30 was mixed by ball-milling, the resulting mixture was wetted with small amount of alcohol, followed by mixing with PTFE emulsion to make a uniform paste. The paste was heated in a 120 °C oven for 1 h to evaporate solvent, and hot-rolled into a free-standing electrode sheet. Using the same procedure, an air electrode with a composition of 92M-30/8PTFE (wt.) was prepared and used as the control electrode. The purpose of selecting 120 °C as the processing and drying temperatures is to melt sulfur so that it can easily wet and adhere to the surfaces of the carbon. The resulting electrode sheets in a thickness range of 0.56–0.63 mm were punched into small disks with an area of 0.97 cm², dried at 120 °C for 2 h for future use. The porosity of the air electrode was calculated from the weight change before and after absorption of liquid propylene carbonate. To ensure full liquid absorption, a weighted carbon electrode was dipped into propylene carbonate and then a vacuum was applied until no bubbles came out of the carbon electrode. Before weighing, the extra liquid on the surface was removed by lightly wiping a filter paper on two sides of the electrode. A solution consisting of 0.2 m LiSO₃CF₃ (Aldrich) dissolved in a 1:3 (by weight) mixture of propylene carbonate (PC, electrolyte grade, Ferro) and 1,2-dimethoxyethane (DME, electrolyte grade, Ferro) was used as the electrolyte.

In a dry-room having a dew point of below –90 °C, a dry Li/S–O₂ hybrid cell was assembled by in sequence stacking a Li foil, a Celgard® 3500 membrane, an air electrode, a Ni mesh as the current collector, and an air window into a coin cell cap. To activate the cell, 200 μL of liquid electrolyte was added through the air window, followed by transferring the cell into a vacuum chamber and applying a vacuum to ensure complete wetting. Extra liquid electrolyte was removed by lightly wiping a filter paper on top of the Ni mesh. The electrolyte-activated cell was clamped on a cell holder and sealed in a dried air or an O₂ plastic bag to obtain a Li/S–O₂ cell. A detailed description for the assembly of such cells can be found in our previous paper [16].

Li/S–O₂ cell was galvanostatically discharged on a Maccor Series 4000 cycler with a 1.5 V cutoff voltage. Before discharge, the cell was rested for 2 h to reach equilibrium between the air electrode and O₂ in the plastic bag. Since the specific capacity of a Li/O₂ cell is determined by carbon [13–17], we here defined the specific capacity of the Li/S–O₂ hybrid cell in reference to the weight of M-30 activated carbon unless specified otherwise. All discharges were carried out at room temperature (about 22 °C).

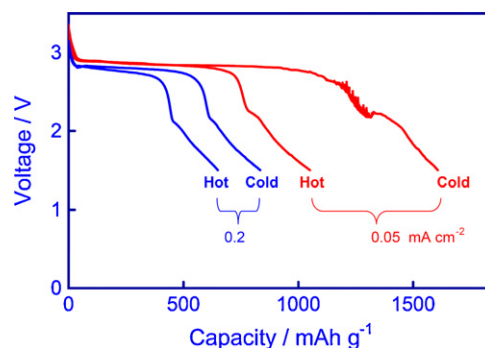


Fig. 1. Comparison of the discharge performance of carbon air electrodes prepared by “cold-process” and “hot-process”, respectively, in which both electrodes had a composition of 92M-30/8PTFE. The cold-process results in a porosity of 3.470 mL g⁻¹ vs. M-30 while the hot-process results in a porosity of 2.7789 mL g⁻¹ vs. M-30.

3. Results and discussion

3.1. Carbon air control electrode

In order to obtain good adhesion of the sulfur to the surface of carbon, a temperature (120 °C) that is slightly higher than the melting point (112.8 °C) of sulfur was selected to make the sulfur–carbon composite cathode. For a fair comparison, the same procedure was used to prepare the carbon air control electrode. To distinguish the air electrode described previously [16], we referred to the present electrode as “hot-processed” electrode and the previous one made at room temperature as “cold-processed” electrode. Fig. 1 compares discharge capacities of two carbon air electrodes that have the same composition but were made using different processes. Obviously, the “cold-processed” electrode has significantly higher capacity than the “hot-processed” counterpart. The similar result also was observed previously [14]. This phenomenon can be attributed to a difference in the porosity of two electrodes. It has been determined that the “cold-processed” electrode has an averaged porosity of 3.470 mL g⁻¹ M-30 while the “hot-processed” counterpart has only 2.7789 mL g⁻¹ M-30. The “hot-process” results in low porosity since at high temperature PTFE binder softens, resulting in dense bonding between carbon particles. In order to obtain the same conditions for comparison, all electrodes in the present work were made using hot process.

3.2. Li/S cell

According to the previous publications [3–11], the discharge of Li/S cells consists of two voltage plateaus: (1) the upper plateau from open-circuit voltage (OCV) to 2.1 V and (2) the lower plateau from 2.1 V until the discharge ends. The upper plateau corresponds to a reduction from elemental sulfur to soluble polysulfide (Li₂S_x, x ≥ 4), which can be written as



In organic electrolyte solution, the resulting polysulfide anions are chemically unstable, they will disproportionate into elemental sulfur and sub-polysulfide (Li₂S_{x-1}) having shorter S–S chain as follow:



Therefore, Eq. (1) is affected by the solubility and stability of polysulfide in the electrolyte, and its capacity changes with the kinetics of Eq. (2). The lower plateau relates to a series of reductions

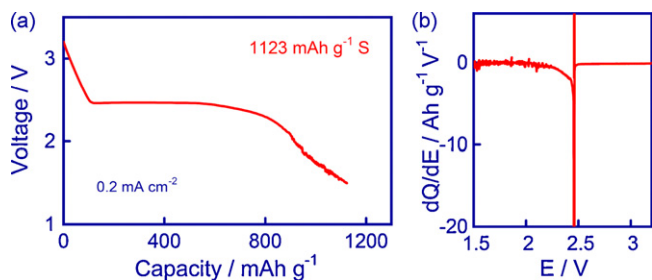
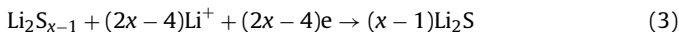


Fig. 2. Discharge voltage curve and differential capacity–voltage plot of a Li/S coin cell, which were recorded at 0.2 mA cm^{-2} .

of the sub-polysulfide, which can be representatively expressed as



The potential of Eq. (3) can be varied slightly with the length of the sub-polysulfide anion chain (i.e., the x value in $\text{Li}_2\text{S}_{x-1}$).

Fig. 2 shows a discharge curve and a differential capacity–voltage plot for a Li/S coin cell made with a cathode having a composition of 70M-30/22S/8PTFE (by weight). Since in the coin cell neither oxygen nor air is available, the results in Fig. 2 entirely reflect the electrochemical characteristics of a Li/S cell. As indicated in Fig. 2a, the upper voltage plateau is so short that it cannot be presented distinctly. This is likely due to the fast disproportionation (Eq. (2)) of the polysulfide anions in the presence of significant amount of M-30 activated carbon. In this case more sub-polysulfide with long S–S chain (i.e., high x value in $\text{Li}_2\text{S}_{x-1}$) can be left to the subsequent Eq. (3). As a result, the lower plateau presented by Eq. (3) gives higher voltages and it counts for the majority of the capacities of sulfur. As shown in Fig. 2, the Li/S cell has a specific capacity of $1123 \text{ mAh g}^{-1} \text{ S}$ (Fig. 2a) vs. the theoretical value of $1675 \text{ mAh g}^{-1} \text{ S}$, and it gives a 2.48 V of voltage plateau (see Fig. 2a and b), being at least 0.2 V higher than those reported elsewhere [6–11].

3.3. Li/O₂ cell

Fig. 3 shows a discharge curve and a differential capacity–voltage plot for a Li/O₂ control cell made with a carbon air electrode composed of 92M-30/8PTFE (by weight). As shown in Fig. 3a, the discharge curve consists of such three voltage regions as: the first region starting from OCV to 2.8 V , this region is very short and the voltage declines rapidly; the second region showing a long voltage plateau at $2.6\text{--}2.8 \text{ V}$, followed by a dramatic decrease in the voltage until 2.1 V ; the third region starting at 2.1 V and declining rapidly. As discussed previously [16], the first voltage region is due to the reduction of oxygen on fresh catalytic sites of carbon, which results in the formation of a monolayer coverage (i.e., a nucleation process) of Li_2O_2 . The second voltage region, which counts for the most of the capacities of a Li/O₂ cell, relates to a reduction of oxygen

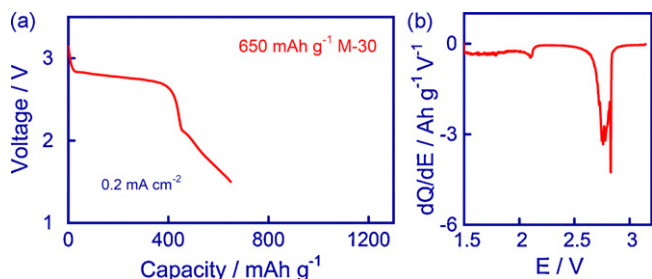


Fig. 3. Discharge voltage curve and differential capacity–voltage plot of a Li/O₂ cell, in which the air electrode had a composition of 92M-30/8PTFE and the discharge test was carried out at 0.2 mA cm^{-2} .

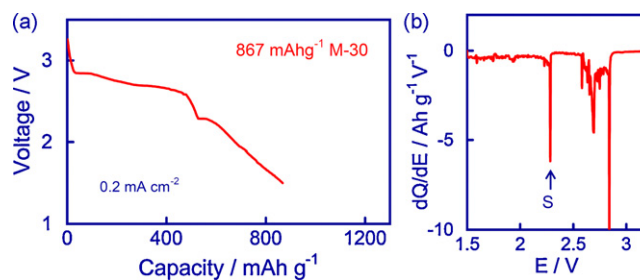


Fig. 4. Discharge voltage curve and differential capacity–voltage plot of a Li/S–O₂ hybrid cell, in which the air electrode had a composition of 70M-30/22S/8PTFE and the discharge test was carried out at 0.2 mA cm^{-2} .

and its resulting growth of the Li_2O_2 layer on the surface of carbon as described below:



The third voltage region relates to a continuous discharge of Li_2O_2 into Li_2O , which can be written as Eq. (5):



Rapid decline in the discharge voltage of the third region is because Li_2O_2 is neither ionically conductive nor electronically conductive. The third discharge region may vanish as the discharge current density rises since in this case the high polarization results in the region too short to be shown [16]. Fig. 3 indicates that the “hot-processed” carbon air electrode gives a specific capacity of 650 mAh g^{-1} at a discharge current density of 0.2 mA cm^{-2} , which is lower than that (834 mAh g^{-1}) obtained from the “cold-processed” electrode with the same composition and discharge conditions [16].

3.4. Li/S–O₂ hybrid cell

Fig. 4 shows a discharge curve and a differential capacity–voltage plot for a Li/S–O₂ hybrid cell made with a sulfur–carbon composite electrode in a composition of 70M-30/22S/8PTFE (by weight). Fig. 4a shows that the Li/S–O₂ cell gives a specific capacity of 867 mAh g^{-1} , being about 200 mAh g^{-1} higher than the control Li/O₂ cell (see Fig. 3a). There is no doubt that these additional capacities are contributed by sulfur. As indicated in Fig. 4b, there are two major reductions in the hybrid cell as presented by the differential capacity peaks at voltages from OCV to 2.6 V and at 2.3 V , respectively. The reduction of oxygen in the hybrid cell, presented by the differential capacity peaks between OCV and 2.6 V in Fig. 4b, is more complicated than that in the Li/O₂ cell (see Fig. 3b). This is due to the fact that in the sulfur–carbon composite electrode some catalytic sites on the surface of carbon are covered fully or partially by sulfur during the hot-process preparation. The difference in the coverage of sulfur results in different conductivity and catalytic activity of carbon, which causes more scattered distribution of the differential capacities against the cell voltage as indicated in Fig. 4b.

Fig. 5 shows the effect of oxygen partial pressure on the specific capacity of the Li/S–O₂ cells, in which cell-1 was sealed in an oxygen plastic bag and cell-2 was sealed in an air plastic bag. The dot line shows the additional capacities of cell-2 after the cell was discharged to 1.5 V and then the cell was open and filled with pure oxygen to replace air, followed by discharging at the same current density. As indicated in Fig. 5, these two cells have significantly different discharge behaviors. For the reduction of oxygen, the discharge voltage and specific capacity of cell-2 are much lower than those of cell-1, showing a significant impact of the oxygen partial pressure. This result is in good agreement with our previous observation [17]. For the reduction of sulfur, however, cell-2 presents higher discharge voltage and larger specific capacity than cell-1

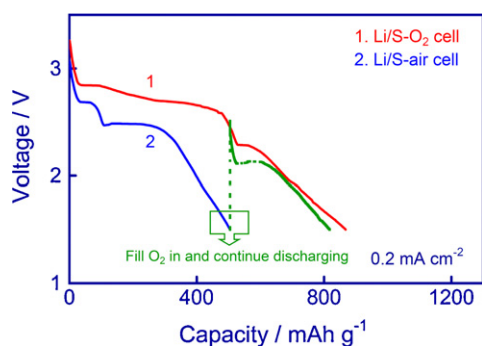


Fig. 5. Comparison of the discharge characteristic of Li/S-O₂ cells in an oxygen and an air environment, respectively, in which the discharge test was carried out at 0.2 mA cm⁻². Note that upon the end of discharge, cell-2 was opened and replaced air with pure oxygen, followed by discharging at the same current density.

although its overall capacity (505 mAh g⁻¹) is still lower than cell-1 (868 mAh g⁻¹). This is because at the end of oxygen discharge, most surfaces of carbon in cell-1 are covered by non-conductive Li₂O₂, resulting in a significant decrease in the conductivity of the composite electrode. Therefore, the subsequent discharge of sulfur in cell-1 suffers high polarization and low utilization. After the discharge ended, cell-2 was opened, filled with pure O₂, and discharged using the same discharge current. This process gives an additional capacity of 312 mAh g⁻¹, as shown by the dot line in Fig. 5. Thus, cell-2 eventually gives the similar capacity as cell-1, suggesting that the overall specific capacity of a Li/S-O₂ cell be limited by the carbon composite electrode, or more specifically by the pore volume accessible for the deposition of insoluble discharge products such as Li₂O₂, Li₂O, Li₂S₂, and Li₂S.

An advantage of Li/S-O₂ cell over the traditional Li/S cell could be its substantially low self-discharge rate. A well-known reason for the high self-discharge of Li/S cell is the soluble polysulfide anions that migrate to the surface of the Li anode and react with metal Li. In Li/S-O₂ hybrid cell, however, this unwanted phenomenon can be minimized since oxygen has higher electrode potential than polysulfide and it can easily oxidize the soluble polysulfide back to insoluble sulfur. Our experiment appears to show that Li₂O₂ cannot oxidize polysulfide although it has higher electrode potential than polysulfide. This is likely related to the insolubility of Li₂O₂ in the organic electrolyte, which significantly reduces the reactivity of the solid Li₂O₂. On the other hand, the presence of oxygen causes negative effect when the content of sulfur in the composite electrode is high. In an effort to increase the content of sulfur, we found that the composite electrodes with more than 60% (wt.) sulfur gave very low specific capacities (results are not shown). To clarify the reasons, we opened the cell after the discharge test ended, and found that the air window of the electrode was completely covered by a dense and yellow-color sulfur layer, which certainly blocks the access of oxygen to the reaction sites of carbon. This could be one of the most important reasons resulting in the low specific capacity for high sulfur-containing electrodes. Another reason is that when the content of sulfur is high, the molten sulfur could cover and clog the catalytic sites of carbon as a result of the hot-process in the preparation of the composite electrode. From the view of this point, therefore, the “hot-process” may be not suitable for the preparation of sulfur-carbon composite electrode of Li/S-O₂ cells.

Fig. 6 compares discharge performance of a Li/O₂ control cell and a Li/S-O₂ hybrid cell at different current densities. At low current density (0.05 mA cm⁻², Fig. 6a), the control cell outperforms Li/S-O₂ hybrid cell in both specific capacity and discharge voltage. This is because the low current density allows gaseous oxygen to have sufficient time for dissolution into the electrolyte and diffusion to the other side of the air electrode [16,17], which favors

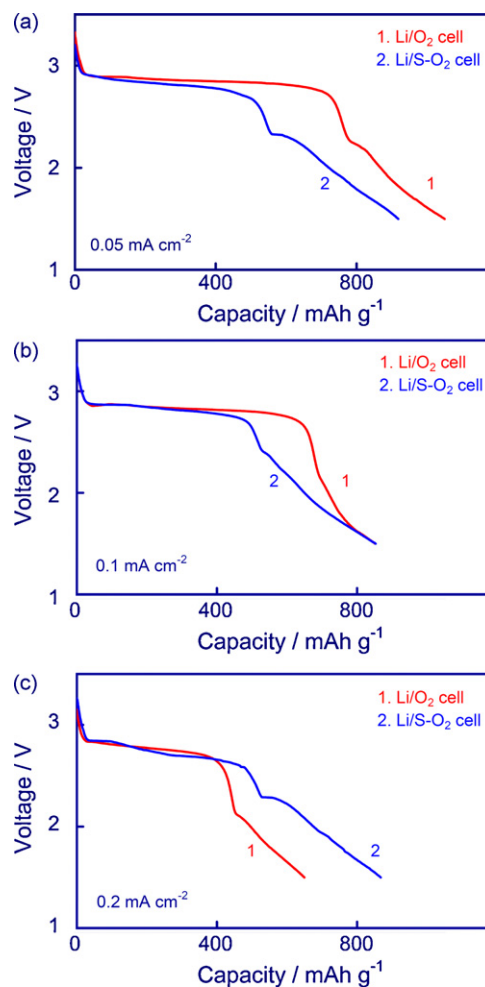


Fig. 6. Comparison of the discharge performance for a Li/O₂ cell and a Li/S-O₂ cell at different current densities. (a) 0.05 mA cm⁻², (b) 0.1 mA cm⁻², and (c) 0.2 mA cm⁻².

uniform and dense deposition of the reduction products (Li₂O₂ and Li₂O) throughout the air electrode and as a result makes the Li/O₂ cell higher capacity. On the contrary, the uniform and dense deposition of Li₂O₂ and Li₂O suppresses the subsequent discharge of sulfur as the non-conductive Li₂O₂ and Li₂O layer adds high electric resistance (polarization) to the discharge of sulfur. At 0.1 mA cm⁻² (Fig. 6b), less reduction products of oxygen are deposited, and hence the Li/S-O₂ cell reaches the same specific capacity as Li/O₂ control cell although its voltages near the end of discharge, which are contributed by Li₂O₂ and sulfur, are relatively lower than Li/O₂ cell. At 0.2 mA cm⁻² (Fig. 6c), the specific capacity contributed by the reduction of oxygen is reduced, resulting in less deposition of the reduction products. In this case, the carbon still retains high electronic conductivity and enables sulfur to discharge effectively. Therefore, the discharge capacity of Li/S-O₂ hybrid at 0.2 mA cm⁻² is significantly higher than the Li/O₂ cell (see Fig. 6c). The merit of Li/S-O₂ hybrid cells in the rate capability can be better displayed by Fig. 7. It is shown that there are few changes in the specific capacity and discharge voltage when the discharge current is increased from 0.05 mA cm⁻² to 0.1 mA cm⁻². The cell still retains unchanged in the specific capacity except for an about 0.1 V decrease in the discharge voltage when the discharge current is further increased from 0.1 mA cm⁻² to 0.2 mA cm⁻². These merits can be attributed to the faster kinetics of sulfur reduction than oxygen reduction. At even higher discharge rate (not done in the present work), the reduction of oxygen may not contribute to the specific capacity of the Li/S-O₂ hybrid cell due to its significantly increased polarization.

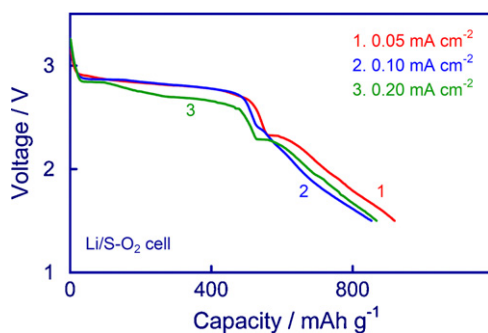


Fig. 7. Discharge rate performance of Li/S–O₂ cells at (1) 0.05 mA cm⁻², (2) 0.1 mA cm⁻², and (3) 0.2 mA cm⁻², respectively.

In this case the discharge performance of a Li/S–O₂ hybrid cell is expected to be determined entirely by sulfur, and the cell performs like a pure Li/S battery.

From the results above, we find that the benefit of a Li/S–O₂ hybrid cell can be maximized either by optimizing the composition of sulfur–carbon composite electrode or by adjusting discharge current density. The Li/S–O₂ hybrid cell becomes advantageous over the Li/O₂ cell only when the discharge current density reaches or exceeds a certain value. This value varies with the design of air composite electrodes, such as the loading of cathode active materials, the thickness and porosity of the electrode.

4. Conclusions

Based on the results of this work, the following conclusions can be made: (1) The concept of a Li/S–O₂ hybrid cell is practically

feasible, the Li/S–O₂ cell is able to operate either in an air or in an environment without air; (2) The overall specific capacity of the Li/S–O₂ cell is limited by the maximum deposition of the insoluble discharge products (i.e., Li₂O₂, Li₂O, Li₂S₂, and Li₂S) of oxygen and sulfur; (3) There is a trade-off between the specific capacities of oxygen and sulfur, the overall specific capacity of the Li/S–O₂ cell can be maximized by optimizing the composition of the sulfur–carbon composite electrode; and (4) The advantage of a Li/S–O₂ cell over the Li/O₂ cell can be presented only when the discharge current density reaches or exceeds a certain value.

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