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Effect of MnO/PP separator on morphologies of Li metal deposition

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Abstract: The application of lithium metal anodes is hindered by low Coulombic efficiency (CE), serious lithium dendrites and volume expansion. An MnO/Polypropylene (PP) composite separator was developed to regulate lithium metal deposition behaviors through *in situ* forming stable artificial solid electrolyte interface (SEI) passivating layers. The concentration of MnO in the cells can be maintained at a constant based on quite low solubility of MnO in the liquid electrolyte, and the dissolved MnO can be reduced to produce Li_2O and Mn metal nanoparticles, which can not only function as nucleating seeds of lithium metal deposits but also repair the broken SEI layer. Dendritic-free Li deposits can be obtained by simple separator coating. It can also improve the electrochemical performance of lithium metal batteries. And it is benefit for applications of Li metal anodes.

Key words: Li metal anode; MnO/PP composite separator; morphology; solid electrolyte interface (SEI)

0 Introduction

Li metal anodes are known as the "holy grail" anodes with extremely high theoretical capacity (3 860 $mAh \cdot g^{-1}$) and the lowest reduction potential (-3.04 V vs standard hydrogen electrode), which is of great significance for the development of Li secondary batteries with high energy density[1,2]. Li metal anodes can be assembled into secondary batteries not only with Li-containing compounds such as Li iron phosphate, Li manganate and ternary materials, but also with Li-free compounds such as sulfur (S) or air[3]. Li metal has good electrical conductivity, so it does not need additional conductive skeleton when used as an anode material[4]. In addition, different from Li^+ ion insertion/extraction mechanism of graphite anode, Li metal anode is plating/stripping on the electrode surface, that is, the Li^+ ions in electrolyte migrate or diffuse to the anode surface and combine with the electrons transferred to the electrode through the external circuit[5]. In the process of charge and discharge cycling of Li metal batteries (LMBs), the diffusion rate of Li^+ ions in electrolyte is slower than the migration rate of electrons in the outer circuit, resulting in uneven deposition of Li metal on the electrode surface, forming Li metal dendrites[6]. The higher the current density, the faster the electron migration rate. The Li metal deposition may follow the "top growth mechanism", and it is easy to form antler-like dendrites, which can penetrate the separator, causing the battery internal short circuit[7].

Due to its strong reactivity, Li metal can react with many substances, including O_2, H_2O or organic electrolytes[8]. Solid electrolyte interface (SEI) layer is formed on the surface of Li metal anode once it contacts with electrolyte. Therefore, SEI layer is actually the reaction products of Li metal and liquid electrolyte. The SEI layer conducts Li^+ ions but not electrons, so it can protect the deposited Li from reacting with electrolyte[9]. However, the SEI layer has a certain brittleness. When the deposited Li metal expands, SEI layer will crack, exposing fresh Li into electrolyte. Moreover, the crack sites of SEI have local high electronic conductivity, and a lot of Li^+ ions tend to accumulate here, leading to uneven deposition and the formation of new SEI layer[10]. Li dendrites with large specific surface area will consume more electrolytes and gradually form thick SEI layer, which will also increase the internal resistance of the battery. At the same time, Li metal dendrites have high tortuosity and are prone to fracture from current collector, falling into electrolyte and becoming "dead Li"[11]. The formation of thick SEI layer and "dead Li" will lead to the reduction of Coulombic efficiency (CE) and poor cycling performance[12,13]. If the Li metal deposition is controlled to be dendritic-free deposition, the electrolyte consumption can be slowed down, and it is also not easy to become "dead Li", which increases the reversibility of Li plating/stripping.

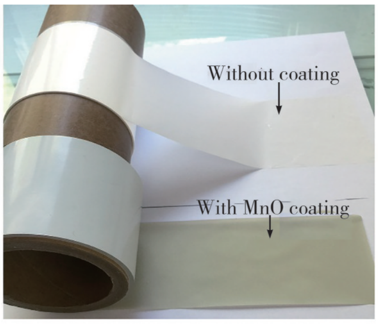
In view of these deficiencies, a series of improvement measures are put forward. The separator and electrolyte are in direct contacting with Li metal anode in LMBs. Therefore, the performance of Li metal anode can be regulated by modifying Li metal, improving electrolyte and separator modification. When 5% $LiNO_3$ was successfully dissolved in ester electrolyte by using dimethyl sulfoxide (DMSO), the CE was improved to 99.55%[14]. With the help of trace CuF_2 as solution accelerator, Huang Jiaqi successfully dissolved $LiNO_3$ in ester electrolyte, forming dense and stable SEI layer on the surface of Li metal anode, and the CE reached 99.5%[15]. Cui Yi evenly coated Cu_3N solution on the surface of Li metal anode to promote the formation of Li_3N protective layer, and the ionic conductivity reached 10^{-3} S·cm^{-1}—10^{-4} S·cm^{-1}, which could effectively promote the uniform migration of Li^+ ions[16]. A thickness of Al_2O_3 protective layer was constructed on the surface of Li metal anode based on atomic layer deposition technology, and the protective layer could effectively reduce the corrosion of electrolyte to Li metal, and gave Li||S cells good cycle performance[17]. It has been reported that piezoelectric ceramic transducer (PZT) can be *in situ* transferred to the surface of Li metal anode to form a passivation layer, which effectively inhibits the volume expansion of Li metal anodes[5].

An MnO/PP composite separator was developed to regulate the morphologies of deposited Li. MnO has a certain solubility in organic liquid electrolyte. MnO can be released from the coating layer into the electrolyte sustainedly, so as to continuously control the deposition behavior of Li metal. Microdissolved MnO can also repair the broken SEI layer and make the interface layer between Li metal and electrolyte become a mixed ion electron conductive layer, which can not only homogenize the electric field, but also promote the migration of Li^+ ions. In this paper, the deposition mechanism and the influence of the MnO/PP composite separator on the morphologies of Li metal deposition are studied, and the electrochemical performance using different separators for Li||Li cells are compared.

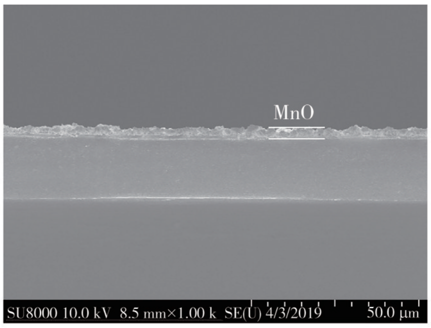
1 Results and discussion

1.1 Morphologies of Li deposits on Cu subtrate

Chemical redox reaction can occur between Li metal and MnO, and the products are Mn metal and Li_2O. MnO particles are first uniformly coated on the surface of porous PP separators with a thickness of around 3.5 μm, as shown in Fig.1. The transition metal oxide is slightly dissolved in organic liquid electrolyte, and the solubility of MnO in ether electrolyte is about 1.5 μmol detected by ICP-AES. MnO could be released continuously and slowly into the electrolyte until the coating layer is exhausted.



(a) Optic images of PP and MnO/PP separators



(b) Cross-sectional SEM image of MnO coated PP separator

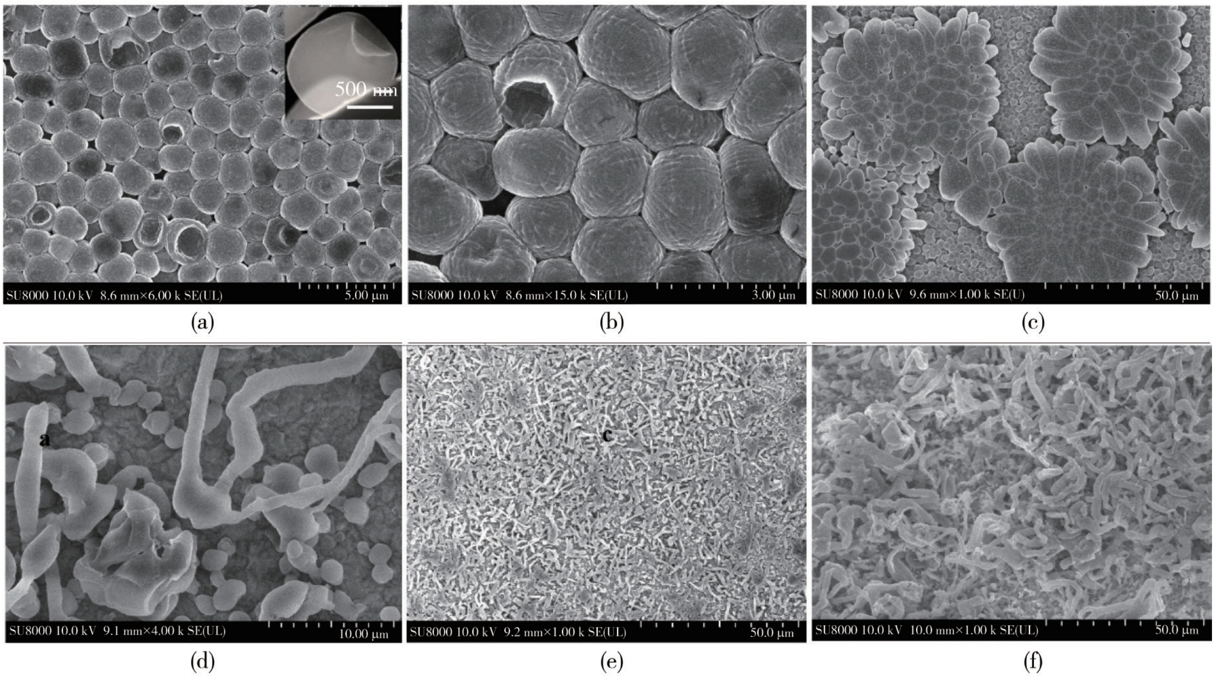
Fig. 1 Images of MnO/PP separator

Therefore, the equation for what happens inside the LMBs is

$$x\mathrm{Li}+y\mathrm{MnO}\rightarrow x\mathrm{Li_2O}+x\mathrm{Mn}+(y-x)\mathrm{MnO}.$$

Li||Cu cells were assemblied with MnO/PP or PP separators to study the MnO effect on Li metal deposition morphologies. When plating 0.2 mA·h·cm^{-2}, it is observed that a uniform and compact spherical layer of Li metal is deposited on the Cu, with a small dimple on its surface (Fig.2(a)—(b)). Ether electrolytes containing $LiNO_3$ are used. Since the reactions between ether solvents and Li metal are rather mild, $LiNO_3$ contributes to the formation of stable SEI layer, and the initial depositions are generally spherical particles[18]. The spherical Li deposition is further observed by scanning transmission electron microscopy (STEM), and the outermost layer is SEI layer (Insert of Fig. 2(a)). With the increase of the area capacity, an interesting two-layer deposition phenomenon is observed on the Cu, that is, the bottom layer is distributed with small balls with a diameter of about 2.0 μm, and the top layer is distributed with large pieces of sheet-like Li (about 58.5 μm in diameter and 6.5 μm in thickness) (Fig. 2(c)). The reason why there are two kinds of morphologies of Li deposition is that the deposition substrate changes from Cu to Li, that is, dense spherical deposition becomes the substrate of subsequent sheet-like deposition. Different substrates have different wettability and surface energy, which affects the morphologies of Li deposition. Therefore, Cu foil are often used to observe the morphologies of Li metal deposition because it can directly reflect the change process of Li deposition from the perspective of heterogeneous nucleation.

Generally, crystal growth includes nucleation and growth, and the same rule can be applied to Li metal deposition. The produced mixed nanoparticles of Mn metal and Li_2O can be used as the crystal nucleus of deposited Li, which belongs to heterogeneous nucleation, because the nucleation overpotential based on mixed nanoparticles is lower than that on Cu (Fig.3). When crystals grow in ideal conditions, one row grows first, then adjacent rows. After a layer is overgrown, a neighboring layer is grown, layer by layer outward parallel migration. However, the actual crystal growth cannot achieve such an ideal situation. It is often the case that one layer is not fully covered, another layer begins to grow. The initial spherical deposition of Li is arranged on the Cu foil in a single layer and then grows up layer by layer, as shown in Fig.2(a)—(c). After layer by layer arrangement of Li, in fact, a uniform layer of Li substrate has been formed. For subsequent deposition, it is to continue to deposit on Li metal, rather than on the original Cu foil, that is, the substrate has changed from a poor wetted Cu substrate to a good wetted Li substrate.



(a) (b) (c)

(d) (e) (f)

Fig. 2 Top view SEM image of deposited Li on Cu foil after the first Li deposition in an Li||Cu cell with MnO/PP separator at 0.5 $mA\cdot cm^{-2}$ for (a–b) 0.2 $mA\cdot h\cdot cm^{-2}$ with different magnification and (c) 1.0 $mA\cdot h\cdot cm^{-2}$. Illustration is HRTEM of lithium deposition. Surface SEM images of deposited Li on Cu foil after the first deposition in Li||Cu cell with PP separator at 0.5 $mA\cdot cm^{-2}$ for (d) 0.2 $mA\cdot h\cdot cm^{-2}$ (e) 0.5 $mA\cdot h\cdot cm^{-2}$ and (f) 1.0 $mAh\cdot cm^{-2}$. Electrolyte of 1.0 $mol\cdot L^{-1}$ LiTFSI in DOL/DME (1∶1, v/v, 1.0% $LiNO_3$) is employed here

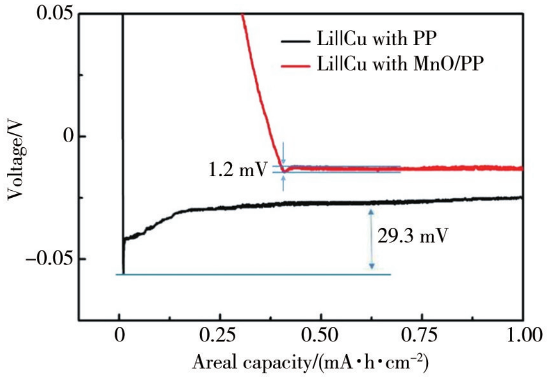


Fig. 3 Overpotential of Li||Cu cells at 0.01 $mA\cdot h\cdot cm^{-2}$ with different separators.

For Li|PP|Cu cells, the initial deposition can also be seen with some balls. However, as the deposition time increases, dendritic Li deposits begin to occur (Fig.2(d)). With the increase of Li deposition, the unstable SEI layer will rupture, and the electron density at the crack sites will increase, attracting a large number of Li^+ ions and forming Li dendrites. With the increase of area capacity, the Li dendrite formation becomes more and more serious. By comparing Fig.2(a)—(c) and Fig.2(d)—(f), it can be found that the morphologies of Li deposition obtained based on the two types of separator are quite different.

1.2 Mechanism of MnO composite separator

In order to study the deposition mechanism of Li metal more directly, Cu substrate is selected in this experiment. Cu is a lithophobic substrate with poor wettability, so Li metal cannot be spread out on Cu, and the contact angle will be greater than 90°. Li metal can be spread out unless some modification is made on Cu, such as the formation of an alloying layer to be a lidenophile substrate[19]. Mn cannot form alloy with Li in the battery system, so the substrate is still Li-phobic Cu[20]. After the initial deposition of the bottom layer of Li metal on Cu foil, the surface of deposited Li metal will be covered with a layer of SEI. The SEI is characterized by conducting ions instead of electrons, which can prevent the deposited Li from continuing to react with MnO in electrolyte, so the MnO is suspended outside the SEI layer at this time. The process of Li deposition is accompanied by volume expansion, while the toughness of the SEI layer is not particularly strong. When the Li deposition continues to a stage, the SEI layer will fracture. After fracture, Li metal and electrolyte are no longer isolated, but in direct contact, and local electronic conductivity increases. As the reduction potential of Mn^{2+} is higher than that of Li^+, Mn^{2+} is preferentially reduced to form Mn metal and Li_2O at the crack sites, thus repairing the broken SEI layer (Fig.4(a)). Mn and Li_2O can also be used as nucleating agents to promote uniform deposition of Li metal. It is recently demonstrated that Li_2O containing SEI layer shows better mechanical strength and stability during cycling of Li-metal cells, compared with LiF or Li_2CO_3 that has long been considered playing an important role in stable SEI[21].

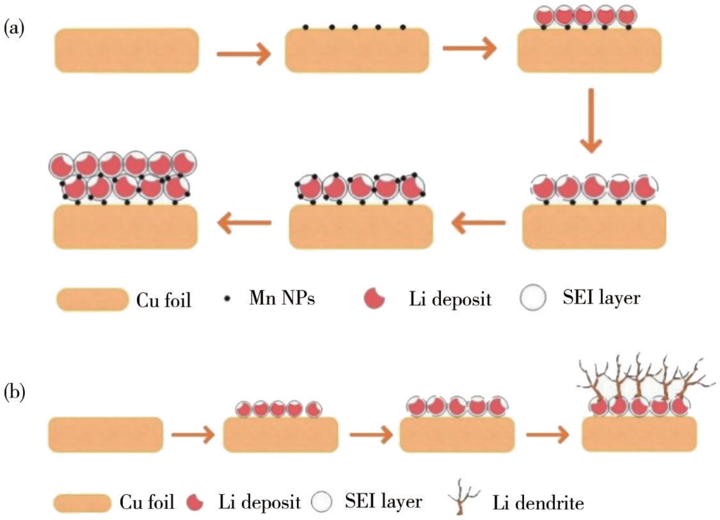


Fig. 4 Mechanism of MnO composite separator. (a) Schematic diagram of dendrite-free Li depositions in Li||Cu cell with MnO/PP separator in ether electrolyte. (b) Schematic diagram of dendrite growth in Li||Cu cell with PP separator.

For the Li||Cu cells using PP separator, initial lithium deposits are also spherical. However, there are differences in diameter between Li deposites by using different separators. Compared with PP separator, the cell with MnO/PP composite separator forms larger initial spherical lithium deposition. The deposition size is related to the overpotential[19]. The smaller the overpotential, the larger the size of the deposition.

With the increase of deposition time, the size of deposition increases, which leads to the fracture of SEI layer. There is a sudden increase of electron density at the crack. It will induce further reduction of Li^+ ions, resulting in dendritic Li metal deposition (Fig.4(b)). Dendrites growth becomes more serious when the deposition area capacity increases. Dendritic lithium metal deposition has a large specific surface area, consuming more electrolytes to form thick SEI layers and leading incresed impedance. At the same time, Li dendrites have high tortuosity and are easy to fracture and be “dead lithium”[22]. The subsequent deposition with MnO/PP composite separator is sheet-like rather than spherical due to the change of the deposition substrates from Cu to Li. The initial spherical deposition of the bottom layer becomes the substrate for subsequent Li deposition, that is, the wettability and surface energy of the interface change, so the Li in the upper layer is spread out in sheet-like shape. Sheet-like Li deposition has a small specific surface area, consuming less electrolyte. The interface layer between Li deposition and liquid electrolyte contains Mn metal and Li_2O, and it is a kind of mixed ion electron conductive layer, which can reduce the impedance of the battery and regulate the formation of dendrite-free Li deposition morphology owing to uniform electric field and Li^+ flux.

The effects of different separators on the SEI layer morphologies were futher studied based on SEM and HRTEM characterization after the initial plating/stripping in the Li||Cu cells. It can be observed that the Cu foil is covered with an uniform layer of thin film in Li||Cu cell with MnO/PP separator after the first cycle (Fig.5(a)). The thin film could be attributed to the reduction of MnO and electrolyte before and during the Li-deposition, which functions as a composite SEI layer protecting the deposited Li metal. There are depressions similar to large pores on the film, which is caused by the collapse of SEI layer with the same shape after the spherical Li deposition is stripped off. The flat part covered on the surface of Cu foil are products of functional coating and electrolytes reduced by electrochemistry. The diffraction rings of Mn metal particles are detected in HRTEM image of SEI layer by using MnO/PP composite separator (Fig.5(b)), which further indicates that Mn metal particles are not oxidized to $\mathrm{Mn^{2+}}$ during Li metal stripping. However, after plating/stripping, a lot of dendritic Li deposition can be seen on the Cu substrate in Li||Cu cell containing PP separator (Fig.5(c)), which is actually "dead Li", i.e., lithium deposits that have not been effectively stripped. Dendritic Li deposition has a high tortuosity, and is prone to fracture and fall into liquid electrolyte, thus losing connection with the Cu foil. Meanwhile, SEI layer continues to be generated on the surface of "dead Li". Since SEI is not conductive, "dead Li" loses activity and cannot be stripped again. Dendritic free Li deposits can be got by using MnO/PP composite separator in Li||Cu cells, and the plating/stripping reversibility is good, so only a layer of thin film can be observed on the Cu foil surface, and there is almost no traces of Li deposits.

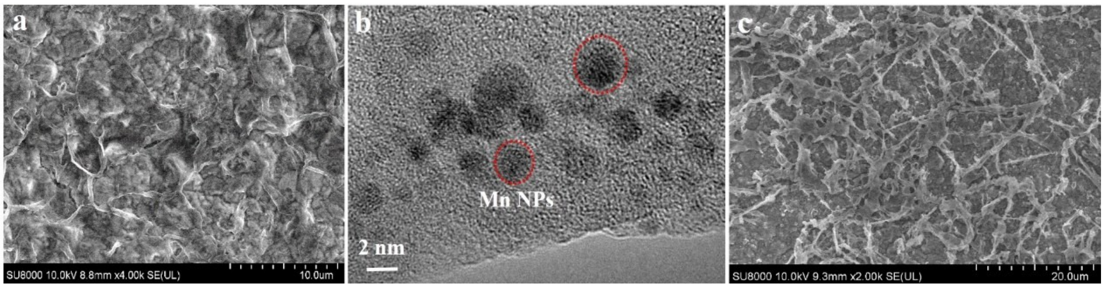
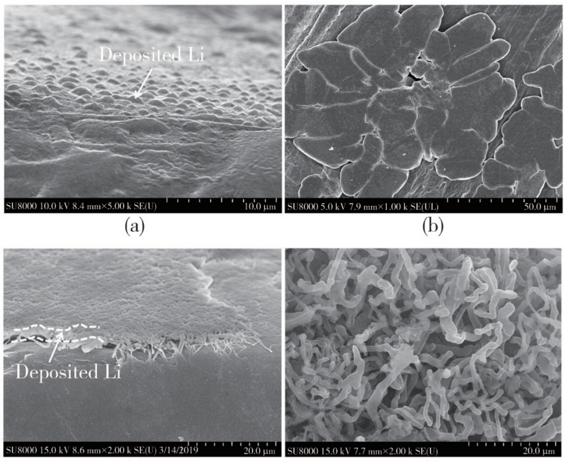


Fig. 5 Top-view SEM image of Cu foil after the first plating/stripping cycle at $0.5\ \mathrm{mA\cdot cm^{-2}}$ for $1.0\ \mathrm{mAh\cdot cm^{-2}}$ with (a) MnO/PP and (c) PP separators. (b) HRTEM image of SEI layer after one plating/stripping cycle in Li||Cu cell with MnO/PP separator at $0.5\ \mathrm{mA\cdot cm^{-2}}$ for $1.0\ \mathrm{mA\cdot h\cdot cm^{-2}}$. Electrolyte of $1.0\ \mathrm{mol\cdot L^{-1}}$ LiTFSI in DOL/DME (1∶1, v/v, 1.0% $\mathrm{LiNO_3}$) is employed here

1.3 Morphologies of Li deposits on Li subtrate

The morphologies of initial Li deposition on Li metal substrate with different separators are further investigated, as shown in Fig.6. When the area capacity of $0.1\ \mathrm{mA\cdot h\cdot cm^{-2}}$ is deposited, hemispherical bulges can be observed on the Li metal substrate with contact angles less than 90°, while contact angles of initial spherical deposits on Cu foil are greater than 90°. When $0.5\ \mathrm{mA\cdot h\cdot cm^{-2}}$ is deposited, a single-layer large-size sheet-like structure is obtained, which is also different from the double-layer structure presented on Cu. The difference of Li deposition between the two substrates is mainly due to the different wettability of substrates to Li and the different surface energy. When the bottom layer of Cu foil is covered with a layer of dense spherical Li, the subsequent deposition is also sheet-like structure. Therefore, researchers often control the deposition of Li metal by changing the surface energy of Cu substrate through various measures, such as forming a liophilic alloy layer.

The MnO/PP composite separator proposed in this study does not form an alloying layer with Li metal on Cu, so it does not completely change the wettability of Cu base and still has a certain over-potential, so the initial Li deposition formed is still spherical rather than hemispherical. For Li|PP|Li cells, the surface of the lithium metal presents disorganized dendrite lithium.



(a) (b)

(c) (d)

Fig. 6 Cross-sectional and top view SEM images of Li foil in a Li||Li cell with a MnO/PP separator at $0.5\ \mathrm{mA\cdot cm^{-2}}$ with different areal capacities of (a) $0.1\ \mathrm{mA\cdot h\cdot cm^{-2}}$ and (b) $0.5\ \mathrm{mA\cdot h\cdot cm^{-2}}$. Cross-sectional and top view SEM images of Li foil in a Li||Li cell with a PP separator at $0.5\ \mathrm{mA\cdot cm^{-2}}$ with different areal capacities of (c) $0.1\ \mathrm{mA\cdot h\cdot cm^{-2}}$ and (d) $0.5\ \mathrm{mA\cdot h\cdot cm^{-2}}$. $1.0\ \mathrm{mol\cdot L^{-1}}$ LiTFSI in DOL/DME (1∶1, v/v, 1.0% $\mathrm{LiNO_3}$) is used as electrolyte

1.4 Electrochemical performance of different separator

Li||Li symmetrical cells with PP and MnO/PP/MnO separators are assembled to monitor interface stability during long-term plating/stripping (see Fig.7).

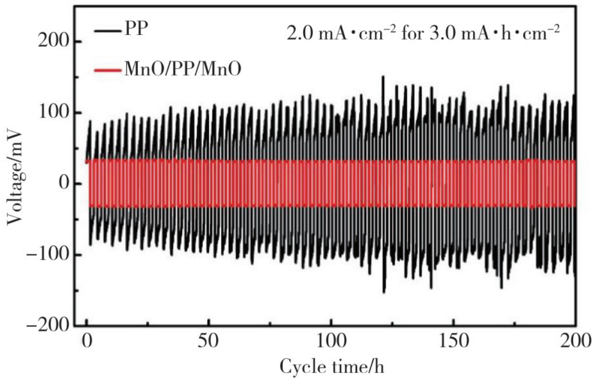
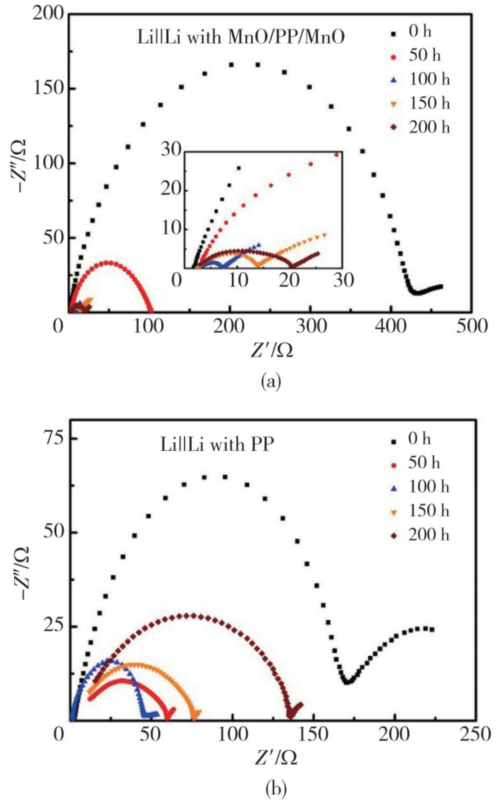


Fig. 7 Cycle curves of Li||Li symmetric cells separated by PP and MnO/PP/MnO at the current density of 2.0 $mA \cdot cm^{-2}$ with the areal capacity of 3.0 $mA \cdot h \cdot cm^{-2}$ in 1.0 $mol \cdot L^{-1}$ LiTFSI DOL/DME (1∶1, v/v, 1.0% $LiNO_3$)



(a)

(b)

Fig. 8 Nyquist plots of Li||Li cells with MnO/PP/MnO (a) and PP (b) separators with 1.0 $mol \cdot L^{-1}$ LiTFSI in DOL/DME (1∶1, v/v, 1.0% $LiNO_3$) as electrolyte at 2.0 $mA \cdot cm^{-2}$ with an areal capacity of 3.0 $mA \cdot h \cdot cm^{-2}$ after different time, respectively

It can be seen that the voltage profiles gradually stabilize at around 30.0 mV for 200 h in a current density of 2.0 $mA \cdot cm^{-2}$, indicating the constant impedance and stable interface of cell during the repeated plating/stripping. In a striking contrast, the symmetric cell with uncoated PP separator shows a continuous voltage increase, which can be attributed to the endless consumption of electrolyte and lithium metal until the depletion of liquid electrolyte. The resistance of the Li||Li cell is monitored with the extension of cycling time, as shown in Fig. 8. Although the Li|MnO/PP/MnO|Li cell gives a larger initial resistance than Li|PP|Li cell owing to the additional layers of MnO, the Li|MnO/PP/MnO|Li cell shows a much more stable and lower resistance from the 100th h to the 200th h, indicating more stable interface between Li metal anode and electrolyte with composite separator.

2 Conclusions

An MnO/PP composite separator with slow releasing function is designed. The functional coating can regulate the behavior of Li metal deposition and repaire the broken SEI layer. MnO could be released continuously and slowly until the coating is exhausted, because it is slightly soluble in organic liquid electrolyte. MnO can be chemically or electrochemically reduced by Li metal to be Mn metal and Li_2O nanoparticles. The products can optimize the wettability and surface energy of substrate. Dendrite-free Li metal deposition with smaller specific surface area and lower tortuosity can be obtained based on composite separator no matter on Cu subtrate or Li subtrate. The sustainably existed trace of MnO ensures the artificial SEI layer can be re-healed once damaged by the volume expansion of lithium metal. However, the deposits obtained by using ordinary PP separator are lithium dendrites. Dendritic-free Li deposits have been proved to help improve the electrochemical performance of LMBs. The composite separator can be mass-produced in atmospheric environment, which can promote the practical application of LMBs to some extent.

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Declaration of conflicting interests

The authors have no conflict of interests related to this publication.

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MnO/PP隔膜对锂金属沉积形貌的影响

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摘 要: 低的库伦效率、严重的枝晶生长和体积膨胀限制了锂金属负极的应用。本文提出了一种MnO/聚丙烯(PP)复合隔膜,可以通过原位形成稳定的固态电解质界面(Solid electrolyte interface, SEI)层调控锂金属的沉积行为。由于MnO微溶于有机液态电解质,其可在电池中保持恒定浓度,同时,溶出的MnO可以被还原成Li_2O和Mn。还原产物不仅可作为锂金属沉积的成核剂,还可以修复破裂的SEI层。通过简单的隔膜涂覆可以获得无枝晶的锂沉积,还可以提升锂金属电池的电化学性能,有助于促进锂金属负极的应用。

关键词: 锂金属负极; MnO/PP复合隔膜; 形貌; 固态电解质界面

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