A thin inorganic composite separator for lithium-ion batteries

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A thin inorganic composite membrane composed of 94 wt\% Al\textsubscript{2}O\textsubscript{3} and 6 wt\% styrene-butadiene rubber (SBR) polymer binder is prepared via an aqueous solution casting process. 1 wt\% polyethylene glycol (PEG) is introduced into the casting suspension for the preparation of a 37 \mu m-thick inorganic composite separator. PEG plays a key role to enhance the stability of the casting suspension to separate the thin membrane from the substrate, and to increase the porosity of the membrane. The as-prepared Al\textsubscript{2}O\textsubscript{3}/SBR separator shows a superior thermal stability under 130 °C with no any shrinkage, higher electrolyte uptake/retention and ionic conductivity than the common polyethylene (PE) separator. In LiNi\textsubscript{1/3}Co\textsubscript{1/3}Mn\textsubscript{1/3}O\textsubscript{2}/graphite cells, the inorganic composite separator exhibits excellent cycling stability and good rate performance.

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1. Introduction

After successful applications in mobile electronic devices, lithium-ion batteries (LIBs) have been regarded as promising power sources for electric vehicles (EVs) and large-scale energy storage systems (ESSs) in recent years because of their advantages on energy density, power density and cycle lifetime [1–4]. In a typical LIB, an electrolyte-permeable porous separator is necessarily used to keep active electrodes (cathode and anode) apart but to permit the free flow of lithium ions through the liquid electrolyte filling in their open porous structure [5]. Even though the separator does not directly participate in the electrochemical reactions in a battery, it does significantly affect the cell performance of the battery as well as safety characteristics. Microporous membranes made from polyethylene (PE) and polypropylene (PP) matrices have widely used as separators for commercial lithium-ion batteries due to their good chemical stability and mechanical strength at room temperature. However, these organic separators usually have low melting points (135 °C for PE and 165 °C for PP) so that they could undergo obvious dimensional shrinkage even at the lower temperatures than their melting points, which may result in the direct contact of the anode and the cathode, i.e., short circuit. This internal short circuit may trigger thermal runaway of LIBs and even a fire or explosion. Additionally, poor wettability of the polyolefin separators to nonaqueous electrolytes limits their high-power applications for EVs and ESSs [6,7].

Inorganic ceramic materials have been investigated as fillers into the polymer matrix to obtain the composite polymer separator with an enhanced thermal stability and electrolyte wettability [8–11]. In our previous papers [12,13], we prepared purely inorganic Al\textsubscript{2}O\textsubscript{3} and SiO\textsubscript{2} porous membranes, which was used as separators in LiFePO\textsubscript{4}/graphite or LiMn\textsubscript{2}O\textsubscript{4}/Li cells and showed higher discharge capacity and rate capability, and better low-temperature performance than those using the commercial polyolefin separators due to their high porosity and wettability. In order to combine the advantages on the flexibility of composite polymer separators and the thermal stability of purely inorganic separators, inorganic composite separators containing inorganic ceramic powders as the main component are attractive [13–15]. The inorganic composite separators are composed of a high content of ceramic powders and a small amount of polymer binders or matrix. The former provides the separator with excellent thermal stability and electrolyte wettability, while the latter is designed for the flexibility of the separator. Recently, ceramic layers including SiO\textsubscript{2} [16], Al\textsubscript{2}O\textsubscript{3} [17,18], TiO\textsubscript{2} [19,20] were coated on commercial polyolefin separators in order to minimize the thermal shrinkage of polyolefin separators. This type of composite separators inherited the shutdown function of polyolefin separators, but also their high cost. Recently, Jung et al. [21] developed a ceramic separator composed of Li\textsubscript{1/2}La\textsubscript{1/4}Zr\textsubscript{1/4}O\textsubscript{2} and 10–20% polymer binder on graphite negative electrode. Zhang et al. [14] blended CaCO\textsubscript{3} powders with Teflon emulsion (61.5% solid content in water) and hot-rolled the mixture into a self-standing membrane. However,
that inorganic composite separator had the thickness of 175–190 μm, which was much thicker than commercial separators (25–40 μm). So far, it still keeps challenges to prepare a thin and self-standing inorganic composite separator. In order to achieve an inorganic composite separator with high porosity, the content of the polymer binder should be a low level, e.g. < 10%. Afterwards, the main difficulty is the poor mechanical strength of a thin composite separator with a small content of binder.

In this study, a thin and self-standing inorganic composite membrane composed of 94% Al2O3 particles and 6% styrene-butadiene rubber (SBR) binder was prepared by a solution casting method. In order to obtain a 37 μm-thick inorganic composite separator with the comparative thickness of commercial polyolefin separators, polyethylene glycol (PEG) was used as a multifunctional agent in the membrane-making process. Firstly, it was helpful to evenly mix Al2O3 particles with the SBR binder. Secondly, it acted as a pore-forming agent for increasing the porosity of the separator. Finally, it was the key to separate the thin membrane from a glass substrate. During the membrane-making process, deionized water was used as the only solvent/dispersant, which made the preparation process environmental friendliness. Besides no thermal shrinkage, the thin inorganic composite separator exhibited attractive cell performance compared to the commercial PE separator in LiNi1/3Co1/3Mn1/3O2|graphite cells. In the LiNi1/3Co1/3Mn1/3O2|graphite cells, the Al2O3/SBR separator exhibits excellent compatibility with both the electrodes, long cycling stability and superior rate capability.

2. Experimental

2.1. Preparation of the composite separators

The inorganic composite separators were prepared by a solution casting method. Neutral activated alumina (Al2O3) (300 mesh, Sinopharm Chemical Reagent Co., Ltd.) and Al2O3 ultrathin particles (100–300 nm, Xianzheng Co., Ltd.) were mixed firstly by the mass ratio of 1:3. Then the mixed Al2O3 particles and styrene-butadiene rubber (SBR, Jingbang Power Source Co., Ltd.) with polyethylene glycol (PEG, M.W. = 2000, Sinopharm Chemical Reagent Co., Ltd.) were mixed thoroughly in deionized water to form a viscous slurry at room temperature. The mass of PEG is 1 wt% based on the total mass of Al2O3 and SBR. Then the slurry was cast on a clean glass substrate and spread by blade to obtain a membrane with a controllable thickness. After drying at 55 °C for 6 h, the glass substrate with the membrane was immersed in deionized water for 1 h to remove PEG. At the same time, the wet membrane was spontaneously separated from the substrate. After drying at 55 °C for 24 h, the as-prepared membrane was cut into discs (Φ16 mm) for testing. For comparison, the no-PEG-assisted membranes with different ratios of Al2O3 particles and SBR binder were prepared in a similar process with the addition and removal of PEG.

2.2. Physical and electrochemical measurements of the separators

The morphologies of the composite separators were investigated by a field emission scanning electron microscopy (FESEM, Hitachi S-4800). The thermal shrinkage of separators was observed after they were placed in an oven and heated at 130 °C for 40 min. The thermal stability of the separators was evaluated by differential scanning calorimetry (DSC, TA DSC 2920) in the temperature range of 50–200 °C at a heating rate of 10 °C min⁻¹ and thermogravimetric (TG) analysis (TGA 6000, Seiko Instruments) in N2 atmosphere from 100 to 800 °C at a heating rate of 20 °C min⁻¹. The contact angles between separators and the electrolyte of 1 M LiPF6/ethylene carbonate (EC)+dimethyl carbonate (DMC) (1:1, w/w) were tested by the contact angle tester (DSA10-MK2, KRUSS Gmbh Germany). As described in our previous paper [12], the electrolyte uptake (U) and retention ratio (R) were calculated according to the following equations.

Electrolyte uptake \( U = \frac{W_f - W_0}{W_0} \times 100\% \)  

(1)

Electrolyte retention \( R = \frac{W_e - W_0}{W_i - W_0} \times 100\% \)  

(2)

where \( W_0 \) is the net weight of pristine separator, \( W_i \) and \( W_e \) are the initial and equilibrium weights of the wet separator after absorbing the electrolyte solution and standing at 50 °C for 30 min at least, respectively. For each testing point, three parallel measurements were carried out under the same conditions. For comparison, the electrolyte retention (R) of some separator was normalized based on the 1st point of each measurement. All the

![Fig. 1. Photographs of the membranes containing different contents of SBR. (a) 6%, (b) 20% and (c) 30%.

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Photographs of the membranes containing different contents of SBR. (a) 6%, (b) 20% and (c) 30%.
measurements were carried out in an Ar-filled glove box (MBraun).

2.3. Electrochemical performance measurements

The ionic conductivities of the separators immersed with the electrolyte solution of 1 M LiPF$_6$/EC+DMC (1:1, w/w) were measured by an electrochemical impedance spectroscopy (EIS) method using a CHI660e electrochemical work station (Shanghai Chenhua). The impedance measurements were performed on the electrolyte-immersed separators sandwiched between two stainless steel electrodes over a frequency range of 100 kHz to 1 Hz with AC amplitude of 10 mV. The ionic conductivity ($\sigma$) was calculated by the following equation:

$$\sigma = \frac{d}{R_b \times A}$$

where $d$ and $A$ are the thickness and the effective area of the separator, respectively, and $R_b$ is the bulk impedance of the wet separator obtained at the high frequency intercept of the Nyquist plot on the real axis.
Cell performance was evaluated in CR2032-type coin cells with a LiNi$_{1/3}$Co$_{1/3}$Mn$_{1/3}$O$_2$ positive electrode and a graphite negative electrode. To make a positive electrode laminate, 80 wt% LiNi$_{1/3}$Co$_{1/3}$Mn$_{1/3}$O$_2$ (Umicore, Belgium), 6 wt% conductive carbon additive (KS-6, TIMCAL), 6 wt% conductive carbon additive (Super-P, TIMCAL) and 8 wt% polyvinylidene fluoride (PVDF, aladdin) were dispersed in 1-methyl-2-pyrrolidinone (NMP, Sinopharm Chemical Reagent Co., Ltd.) to form a slurry, which was then spread onto an aluminum foil current collector. To make a negative electrode laminate, 90 wt% commercial graphite (FT-1), 5 wt% conductive carbon additive (SFG, TIMCAL), 2 wt% carboxymethyl cellulose (CMC, aladdin) and 3 wt% SBR were dispersed in deionized water, which was then spread onto a copper foil current collector. After vacuum drying at 80 °C for 10 h, both the positive and negative laminates were punched into discs (Φ14 mm) for assembling the cells. The mass loadings of the positive and negative electrodes were controlled at ~4.0 mg cm$^{-2}$ and ~1.9 mg cm$^{-2}$, respectively. The specific capacity of the LiNi$_{1/3}$Co$_{1/3}$Mn$_{1/3}$O$_2$|graphite full-cell was calculated on the mass of the LiNi$_{1/3}$Co$_{1/3}$Mn$_{1/3}$O$_2$ cathode. The electrolyte used was 1 M LiPF$_6$/EC+DMC (1:1, w/w). The LiNi$_{1/3}$Co$_{1/3}$Mn$_{1/3}$O$_2$|graphite cells were performed between 2.5 and 4.3 V on an Arbin BT2000 battery cycler. All the cells were initially cycled twice at 0.1 C (1 C = 150 mA g$^{-1}$) for activation. The cycling performance was measured at 0.5 C and rate capabilities was obtained at different current densities ranging from 0.5 to 8 C for charge and discharge.

3. Results and discussion

Zhang et al. [14] prepared the inorganic composite separator containing 92% CaCO$_3$ and 8% PTFE by a facile solution casting method, but its big thickness of 175–190 μm was not welcome in views of volumetric energy density and cell resistance. However, there are many challenges for the thin inorganic composite separator. Firstly, a big challenge is originated from the preparation of thin inorganic composite membranes by solution casting, e.g., the comparable thickness of commercial polyolefin separators, <40 μm. As shown in Fig. 1, it is quite difficult to prepare an unbroken membrane using a low content of polymer binder (6% SBR in Fig. 1a).

A high content of polymer binder, exceeding 20% (Fig. 1b), even 30% SBR is necessary to get an unbroken membrane, but the porosity of the membrane will dramatically decrease with
increasing the polymer composition (Fig. 1c). The porosity is tightly related to the mass ratio of Al$_2$O$_3$ and SBR. The higher mass ratio of Al$_2$O$_3$ and SBR (the lower content of SBR), the higher porosity of the received membrane. In order to prepare a flexible, thin and highly porous membrane, two issues need be solved: (1) a low content of polymer binder is used for high porosity of the composite membrane, but the binder can strongly anchor the tiny inorganic powers to build up a flexible membrane. One of the key issues is the good dispersibility of the binder in the inorganic powers suspension; (2) it is a big challenge to separate a thin membrane from a substrate after the solution casting followed by drying. It seems to be impossible to peel off a dry membrane with less than 40 $\mu$m-thickness and a low content of polymer binder strongly adhered to the glass substrate.

In order to weaken the connection of the membrane and the substrate, we introduce 1 wt% PEG into the suspension for solution casting. After immersing the dry membrane adhered to the substrate into water, PEG will be dissolved into water and the membrane be separated spontaneously from the substrate. As shown in Fig. 2a, the membrane prepared without PEG can be hardly separated from the glass substrate and broke into pieces since the content of polymer binder is low and the membrane is too thin. The membrane prepared with PEG can be easily peeled off and the as-prepared membrane is flexible (Fig. 2b). The top surface morphology of the membrane with PEG was characterized by SEM as shown in Fig. 2c. It is clearly shown that Al$_2$O$_3$ particles are homogeneously distributed and connected by SBR binders. The clear and homogeneous pores with the size of several hundred nanometers are formed after removing distilled water and PEG. The pore size of the inorganic composite membrane will be mainly related to the size of Al$_2$O$_3$ particles. In our study, we use the mixture of neutral activated alumina (Al$_2$O$_3$) (300 mesh) and Al$_2$O$_3$ ultrathin particles (100–300 nm) by the mass ratio of 1:3. The big Al$_2$O$_3$ particles are beneficial to the mechanical strength of the membrane, and the ultrathin particles provide the abundant microporous structure. The porosity of the Al$_2$O$_3$/SBR separator is also estimated to be around 68% based on the electrolyte uptake, the electrolyte density and the geometrical volume of the separator, which is much higher than that of the commercial polymer separators (~40%) [13]. From the cross-section SEM image (Fig. 2d), the thickness of the Al$_2$O$_3$/SBR membrane is about 37 $\mu$m. It is also possible to obtain much thinner membrane by increasing the content of SBR or using a nonwoven matrix. The bottom surface (Fig. 2e) of the Al$_2$O$_3$/SBR membrane is also highly porous, quite similar as its top surface, suggesting that the membrane prepared with PEG is symmetrical. However, the bottom surface (Fig. 2f) of the membrane prepared without PEG is dense, which clearly suggests the high content of the polymer binder in the side close to the glass substrate. The inhomogeneous dispersing of the polymer binder directly results to the difficulties for the preparation of the membrane and the poor mechanical properties of the membrane as well as the limitation for the ionic transportation between-in. In addition, it is also found that 6% SBR is the optimal content for the preparation of inorganic composite separators with 1% PEG. As shown in Fig. 3, when the SBR content is lower than 6% (4% or 5%) it is difficult to form a thin and flexible membrane. That is, Al$_2$O$_3$ ultrathin particles (100–300 nm) with high surface area require a content of SBR binder no less than 6%. Therefore, the inorganic composite separator containing 6% SBR and with the assistance of 1% PEG is indicated as the Al$_2$O$_3$/SBR separator below.
Fig. 4 shows schematic illustration of the membrane preparation process with and without PEG. Without PEG, the SBR in the wet membrane cast on the glass substrate is more or less apt to precipitate to the bottom of the membrane during the drying process. As a result, the dense bottom part of the composite membrane was tightly adhered to the glass substrate so that the membrane cannot be separated from the substrate. Since the content of SBR is low, the Al₂O₃ particles on the top of the membrane lead to the poor mechanical strength and the membrane was very brittle. PEG has three roles during the membrane preparation process. Firstly, it acts as a dispersing agent, which enhances the stability of the casting suspension during the dry process. PEG helps the polymer binder uniformly sticking the Al₂O₃ particles like a surfactant, which is quite important to obtain the relatively symmetrical porous membrane. Secondly, PEG as a soluble polymer can help weaken the connection between the inorganic composite membrane and the glass substrate in water so that the thin membrane could be separated spontaneously from the substrate. Last but not the least, PEG is a good pore-forming agent after its removal from the membrane in water.

The thermal shrinkage of separators is closely related to the safety of lithium-ion batteries, since a severe shrinkage caused by the heat generation during cycling especially at high current rates would result in the short circuit of batteries [22,23]. The thermal shrinkages of separators were observed by measuring the dimensional change (area-based) after they were subjected to heat treatment at 130 °C for 40 min (Fig. 5a and b). Since PE has a melting point of about 135 °C and the corresponding microporous separator is prepared through stretching processes, the PE separator easily lose dimensional stability above 100 °C [24,25]. As shown in Fig. 5b, the PE separator has a big shrinkage of 84%. However, the Al₂O₃/SBR separator has almost no shrinkage. The good thermal stability of the Al₂O₃/SBR separator can be attributed to the thermally stable Al₂O₃ particles and high heat resistance SBR as binder. It must be pointed out that compared with other binders such as PVDF and polymethyl methacrylate (PMMA), the elastomeric SBR possesses higher flexibility, stronger binding force and higher heat resistance [17,26].

Fig. 5c and d shows the DSC and TG curves of the PE and the Al₂O₃/SBR separators, respectively. In Fig. 5c, the PE separator shows an endothermic peak at 147.5 °C in the DSC curve, corresponding to the melting point of PE. Meanwhile, the Al₂O₃/SBR membrane shows no endothermic peak at the same temperature range, which indicates that the Al₂O₃/SBR separator is thermally stable between 50 and 200 °C. From Fig. 5d, it can be seen that up to 600 °C the PE separator experiences a weight loss of over 90%, but the inorganic composite separator only has a small weight loss of about 5% due to its low content of polymer binder. The low weight loss of the inorganic composite separator demonstrates its qualification to separate the cathode and anode at high temperatures.

The wettability of separators was evaluated by electrolyte contact angle test, and the results are shown in Fig. 6a and b. The Al₂O₃/SBR separator was quickly wetted by electrolyte with contact angle of 0°, while the PE separator was hardly wetted by electrolyte (with contact angle of 44.8°) because the nonpolar PE separator has intrinsically poor wettability with the commercial nonaqueous electrolytes [13,27]. The superior electrolyte
wettability of the Al2O3/SBR separator is attributed to the hydrophilic nature of the oxide particles and the capillary force of the pores in the Al2O3/SBR separator. Moreover, the electrolyte uptake of the Al2O3/SBR separator (0.8159 g cm⁻³) was larger than that of the PE separator (0.6768 g cm⁻³), due to the good electrolyte wettability of inorganic particles and high porosity of the Al2O3/SBR separator. The time dependence of the normalized electrolyte retention of the separators at 50 °C is shown in Fig. 6c. After 3 h the electrolyte stored in the Al2O3/SBR separator is 40%, but only 23% in the PE separator. The result of electrolyte uptake is well accordance with the electrolyte contact angle testing. The Al2O3/SBR separator presents superior electrolyte wettability than the PE separator, owing to the nano-porous structure and high hydrophilicity of Al2O3 particles toward nonaqueous electrolytes. It is known that the wettability of separator greatly affects cell performance [28]. Considering the excellent electrolyte wettability, the Al2O3/SBR separator is expected to provide higher ion conductivity. Fig. 6d shows the Nyquist curves of the liquid electrolyte-soaked separators. The straight line inclined towards the Z axis represents the electrode/electrolyte double layer capacitance behavior. Thus, the bulk resistance Rb of the separators can be acquired from the high-frequency intercept of the Nyquist plot on the Z axis. It is seen that the bulk resistance of the Al2O3/SBR separator (3.45 Ω) is slight lower than the PE separator (3.86 Ω). According to the Eq. (3) in Section 2, the ionic conductivity of the Al2O3/SBR separator is calculated to be 0.93 mS cm⁻¹, which surpasses that of the PE separator (0.40 mS cm⁻¹). The higher ionic conductivity of the Al2O3/SBR separator is due to its better electrolyte wettability and higher porosity than the PE separator [29]. These results suggest that the ionic conductivity of the Al2O3/SBR separator can well meet the conductivity requirement of lithium-ion battery applications.

The cell performance of the inorganic composite separator is evaluated in the LiNi1/3Co1/3Mn1/3O2/graphite cells by comparing with the PE separator. Fig. 7a shows the initial charge–discharge voltage profiles of the LiNi1/3Co1/3Mn1/3O2/graphite cells using the PE separator and the Al2O3/SBR separator. The first discharge capacity of the cell using the PE separator at 0.1 C is 161 mAh g⁻¹, which is slightly higher than the cell using the Al2O3/SBR separator (156 mAh g⁻¹). However, the cell using the Al2O3/SBR separator exhibits the high Coulombic efficiency of 88%, compared that using the PE separator of only 81%. The improvement on Coulombic efficiency is because of the reduced side reactions during the activation process of the battery, since Al2O3 can capture the trace amounts of moisture and acidic impurity in the electrolyte [30]. Fig. 7b shows the cycling performance of the cells with the PE separator and the Al2O3/SBR separator. At 0.5 C, the cycling performance of both cells is very stable. But the discharge capacity of the cell using the Al2O3/SBR separator remains 90% of its initial capacity after 100 cycles, while the cell using the PE separator remains 87%. It can be attributed to the good wettability of Al2O3 particles which offered expedite channels for Li⁺ ions to pass through. The excellent cycling stability of the cell using the Al2O3/SBR separator strongly supports the good compatibility between this inorganic composite separator with both the cathode and the anode.

Rate performance of the LiNi1/3Co1/3Mn1/3O2/graphite cells with the PE separator and the Al2O3/SBR separator were also evaluated at current rates of 0.5 C, 1 C, 2 C, 4 C and 8 C (Fig. 7c and d). At 0.5 C, the discharge capacities of the cells using the polymer
separator and the Al₂O₃/SBR separator are 160 and 156 mAh g⁻¹, respectively. At 1 C, the cell using the Al₂O₃/SBR separator delivers the comparative capacity to that using the PE separator. However, the cell with the Al₂O₃/SBR separator shows better rate capability than that with the PE separator at high current rates (2 C, 4 C and 8 C). When the current rate increases to 8 C, the discharge capacities of the cells using the Al₂O₃/SBR separator and the PE separator are 128 and 105 mAh g⁻¹, respectively. The reason for the improvement on rate capability can be also attributed to the higher uptake and the better wettability of the Al₂O₃/SBR separator toward the electrolyte as well as its high porosity. These results on the cell performance suggest that this inorganic composite separator could be a promising choice in power batteries for EVs.

4. Conclusion

A 37 μm-thick inorganic composite membrane, consisting of 94% Al₂O₃ and 6% SBR, has been prepared via a facile solution casting with 1% PEG. Since a very small amount of binder is needed and the only solvent is deionized water during the membrane-making process, the Al₂O₃/SBR membrane is attractive in views of environmental friendliness and cost. The effects of PEG during making the thin membrane are from three ways: to enhance the stability of the casting suspension as a dispersing agent, to separate the thin membrane from the substrate as a soluble polymer, and to increase the porosity of the membrane as a pore-forming agent. As the separator of LIBs, the Al₂O₃/SBR membrane shows superior thermal stability, enhanced wettability toward nonaqueous electrolyte, higher porosity and ionic conductivity compared with the commonly used PE separator. In the LiN11/2CO13Mn11/2O2-graphite, the Al₂O₃/SBR separator exhibits excellent compatibility with both the electrodes, long cycling stability and superior rate capability. Nevertheless, the mechanical strength of the inorganic composite membrane is not enough, still further work needs to be done for this inorganic composite separator with high strength. In conclusion, this work provides a facile PEG-assisting way to prepare thin inorganic composite separators, which would be attractive for high performance lithium ion batteries with high safety characteristics.

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References


