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Fabrication of antimony-doped tin oxide/carbon black composite with oxygen plasma treatment for lithium-air batteries

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ABSTRACT

In this paper, carbon black (CB) was treated by oxygen plasma as electrode active material and antimonydoped tin oxide based catalyst was prepared in the form of nanoparticles. The initial discharge of asprepared cathode for lithium-air battery was investigated. We found the surface of CB after oxygen plasma for 10 min at 200 W had successfully grafted functional groups, which were helpful to increase the binding sites between carbon material and tin oxide. More importantly, the results showed that functional CB exhibited excellent dispersing property. The synthesis of antimony-doped tin oxide (ATO) had a significant role in the growth of initial discharge plateaus of lithium-air battery. The discharge capacity of carbon black antimony-doped tin oxide (ATO/CB-10) was also superior to that of non-catalyst cathode.

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1. Introduction

Lithium-air batteries have received considerable attention for their various advantages with high theoretical specific energy and energy density since K.M. Abraham et al. reported a novel lithiumair battery [1,2]. For the past of two decades, the lithium-air batteries have ever faced many big challenges, such as the decomposition of electrolyte and the polarization of electrode. For catalysts, the chemical reactions are so sluggish that electrocatalysts as additives are used in the cathode to improve the electrochemical performance of lithium-air cells, such as Pt, Pd, MnOx and their hybrids [3–6]. Among them, ATO has high electrical conductivity and electrochemical stability. For cathode, carbon materials especially carbon black can be used as electrode active materials in lithium-air batteries due to their good conductivity and a large amount of mesopores in the carbon structure[7]. Reported in previous work, the surface area and microstructure of the carbon sources are strongly related to the electrical properties of lithiumair battery [8,9]. Besides, carbon sources can be ideal support to hold metal or metal oxides to form hybrid catalysts. However, the agglomerate carbon black blocks its employment and the carbonsupported catalyst is fabricated by complicated procedures, even introducing by-products.

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http://dx.doi.org/10.1016/j.matlet.2016.05.129 0167-577X/© 2016 Elsevier B.V. All rights reserved. In this work, the catalysis of ATO towards oxygen reduction reaction (ORR) was explored, which were easily prepared by solgel method. The plasma etching machine was used to deal with carbon black by low temperature plasma treatment due to its controllable processing, low cost, and environmental friendliness [10]. The moderate thin film as air cathode was made by spin coating method. The as-prepared cell including initial discharge capacity and plateaus shows a satisfying result. The findings proved the nanoparticles ATO with mesopores, an efficient catalytic activity for ORR, were successfully synthesized.

2. Experimental

2.1. Preparation of CB-supported ATO

The conductive carbon black VXC-72(diameter: 30 nm; resistivity: 0.067ohm-cm) was purchased from the Cabot Company in USA. They were put into the quartz tube of the oxygen plasma stripping machine, processing 2 min, 3 min, 5 min, 10 min at 200 W (CB-n). The illustration of oxygen plasma process is shown in Fig. 10.5.6 g SnCl₄ · 4H₂O and SbCl₃ (9:1 mol ratio), along with 0.5 g CB-10 dissolved in distilled water were sonicated for 30 min at room temperature, and then the solution were added ammonium hydroxide drop by drop until the pH up to 7. Hydrochloric acid was then added to the mixing solution until the pH up to 1–2 and mechanically stirred for 60 min. After 24 h standing, the deposition was washed several times by silver nitrate solution to



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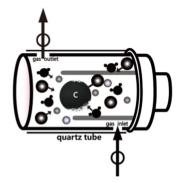


Fig. 1. Schematic diagram of air plasma etching for CB.

omit chloride ion. The resulting gel then were annealed at 500 °C under nitrogen atmosphere for 2 h.

2.2. ATO/CB-10 cathode and assembling lithium-air battery

N,N-dimethyl formamide (DMF, Sigma-Aldrich) as solvent, polyvinylidene fluoride (PVDF, FR904) (40 wt%) as solute material, CB-10(48 wt%) and ATO(12 wt%) as additives were mixed together and mechanically stirred until they are mixed homogeneously. The carbon slurry was coated onto a commercial carbon paper going through a spin coating method. Finally, the cathode was treated at 95 °C in vacuum for 12 h. Approximately 6.0 mg of carbon slurry was loaded on the carbon paper. Lithium-air cell was assembled with Li foil/electrolyte/carbon slurry in Ar-filled glove box filled with less than 5 ppm of moisture and O₂. The electrolyte contains 1 mol/L LiPF₆ and EC+ DEC+ DMC with a volume ratio of 1:1:1. The cell without catalysis was also prepared using the same method for comparison.

2.3. Characterization

The resistivity of samples were measured by semiconductor resistivity of the powder tester(ST-2722). The surface area of CB samples were investigated by Brunauer-Emmett-Teller (BET, ASAP2000M). The morphology of samples were examined by field emission scanning electron microscopy (FESEM, HITACHIS-4800). The chemical elements of carbon black samples were studied by X-ray photoelectron spectroscopy (XPS, K-Sepna), X-ray diffraction (XRD,D/max-2500, Cu Ka radiation at 30 kV) and FTIR spectroscopy. The discharge capacity was measured with electrochemical work station (ZENNIUM).

Table 1.

Comparison of surface area and pore volume of three samples.

Carbon sample	Surface area (m ² /g)	Pore volume (cc/g)	Pore radius (nm)
Pristine CB	254.151	$\begin{array}{c} 3.834 \times 10^{-1} \\ 4.210 \times 10^{-1} \\ 3.671 \times 10^{-1} \end{array}$	1.745
CB-10	243.906		2.034
Milled CB	97.078		1.527

3. Results and discussion

3.1. BET

The resistivity comparison between parent CB and functional CB are shown in Fig. 2(a). Interestingly, the resistivity of functional CB-10 (\sim 0.016 Ω -cm) is much lower than the parent material. It needs to be noted these oxygen functional groups can increase the conductivity of CB due to lower the agglomeration and are helpful to improve the polarization of cathode [11,12]. Because the initial discharge capacity are highly related to the pore sizes of CB, the pristine CB, treated CB-10 and milled carbon black for 30 min in the agate mortar were analyzed by BET. In Fig. 2(b), they have the same mesoporous structures with broad pore size distribution. The more detail data are listed in Table 1. There is sharply decrease in total surface area from 254.151 m^2/g to 97.078 m^2/g for milled CB, while the structure of CB after plasma are rarely changed about either the surface area or pore radius. The small changes between pristine CB and functional CB may be the air plasma etching effect both physical and chemical etching. It is indicated oxygen plasma would not affect the initial discharge capacity by means of changing pore radius [13]. The functional groups on the surface of CB-10 with low-temperature oxygen plasma treatment were by XPS. It is observed that the peaks near 284.6 eV and 532.8 eV are attributed to C 1s and O 1s, respectively in Fig. 2(c). From the XPS spectrum of CB-10, the O 1s spectrum is relatively stronger than that of pure CB, suggesting an effective grafting of oxygen element on the carbon black surface.

3.2. SEM

The microstructure and morphology of CB-10 and the ATO/CB-10 cathode are shown in Fig. 3. CB-10 exhibited a uniform distribution like grapes around 30 nm in diameter [14]. Since plasma process causes a large amount of oxygen functional groups combined with carbon black, the interaction between each other is weak considering Van der Waals or the electrostatic repulsion generally strong enough. In addition, from the image of SEM at lower magnification, a number of cracks are moderate to ensure the cathode has good physical stability and prevents to be clogged by solid discharge products. Fig. 3(c) showed these mesopores are clear and the nanoparticles in smaller size is attributed to ATO, 15–

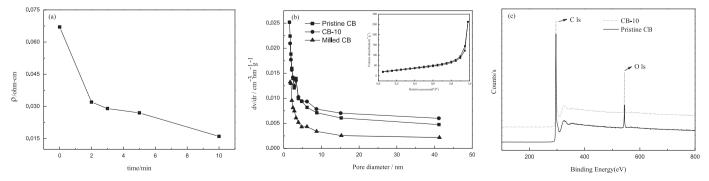


Fig. 2. (a) Effects of treatment time on the resistivity of samples; (b) Pore size distributions of pristine CB, CB-10 and milled CB. Inset: the nitrogen adsorption/desorption isotherm of CB-10; (c) XPS spectra of pristine CB and CB-10.

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