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Bismuth Oxide: A New Lithium-Ion Battery Anode

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Abstract

Bismuth oxide directly grown on nickel foam (p-Bi₂O₃/Ni) was prepared by a facile polymer-assisted solution approach and was used directly as a lithium-ion battery anode for the first time. The Bi₂O₃ particles were covered with thin carbon layers, forming network-like sheets on the surface of the Ni foam. The binder-free p-Bi₂O₃/Ni shows superior electrochemical properties with a capacity of 668 mAh/g at a current density of 800 mA/g, which is much higher than that of commercial Bi₂O₃ powder (c-Bi₂O₃) and Bi₂O₃ powder prepared by the polymer-assisted solution method (p-Bi₂O₃). The good performance of p-Bi₂O₃/Ni can be attributed to higher volumetric utilization efficiency, better connection of active materials to the current collector, and shorter lithium ion diffusion path.

Introduction

In the past decades, lithium-ion batteries (LIBs) have been considered as the most effective and practical technology for power supply of small electronic devices due to its flexible design and long lifespan. However, with the rapid development of electronics and increasing trend of renewable energy, improved electrode materials for LIBs are needed to meet the increasing demand for higher energy density, larger gravimetric/volumetric capacity, and better cycle performance.^{1,2} To address this concern, a great deal of efforts have been devoted to the fabrication of various materials for lithium-ion battery electrodes. Metal oxides have been intensively studied as one of the most promising candidates for LIBs, because of their high theoretical capacities and low cost.³⁻⁷

In addition, alloying anode materials, which mainly include Group IVA and Group VA elements, have been investigated as potential anode materials. For example, SnO₂, Sn, and Sb together with their composites with carbon, were widely studied.⁸⁻¹⁵ Based on the diagonal relationship between Bi and Sn, Bi is believed to be able to work as an anode material for LIBs as well. Despite the relatively low gravimetric capacity of bismuth (386 mAh/g), which is comparable to that of the commercial carbon anode (372 mAh/g), it has a quite high volumetric capacity of about 3765 mAh/cm³.^{13,14} This establishes a good

potential for bismuth based compounds and composites to work as the anode materials for LIBs. Recently, bismuth sulfide and bismuth telluride were studied as the anode materials.^{16–20} It was also demonstrated that the electrochemical behaviour of conventional transition metal oxides can be improved by doping them with Bi_2O_3 ^{21,22,23} or by synthesizing Bi involved binary metal oxides.^{24,25}

Bi_2O_3 is an important metal-oxide semiconductor with a band gap of 2.8 eV.²⁶ Many efforts have been made to synthesize various nanostructures of Bi_2O_3 .^{27–30} However, to the best of our knowledge, direct application of Bi_2O_3 as anode materials for LIBs has not been yet reported. Herein, we report a facile polymer-assisted solution method to directly grow Bi_2O_3 on Ni foam (p- $\text{Bi}_2\text{O}_3/\text{Ni}$) for the use as a binder-free LIB anode. The p- $\text{Bi}_2\text{O}_3/\text{Ni}$ shows superior electrochemical properties in comparison to the polymer-assisted solution prepared Bi_2O_3 powder (p- Bi_2O_3) and a commercial Bi_2O_3 powder (c- Bi_2O_3). P- $\text{Bi}_2\text{O}_3/\text{Ni}$ kept a capacity of 782 mAh/g after 40 cycles at a current density of 100 mA/g and still delivered a capacity of 668 mAh/g at a current density of 800 mA/g.

Experimental

Sample Preparation

The precursor solution was prepared by dissolving 1 g of bismuth nitrate hydrate ($\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$) into a polymer solution, which was prepared by dissolving 2 g of polyethylenimine (PEI, 50 wt% in water, branched polymer, average $M_n \sim 60,000$ by GPC, average $M_w \sim 750,000$ by LS, Aldrich) and 1 g of ethylenediaminetetraacetic acid (EDTA, anhydrous, 99 %, Aldrich) in 7 g of de-ionized (DI) water. To prepare p- $\text{Bi}_2\text{O}_3/\text{Ni}$, the Ni foam (MTI) was immersed into the precursor solution in an alumina boat. The crucible with immersed Ni foam was transferred into a box furnace and heated at 450 °C for 3 h in air. Finally, the Ni foam covered with yellowish Bi_2O_3 was sonicated in DI water for 2 min to remove the unbound Bi_2O_3 powder followed by drying in a vacuum oven at 70 °C for 12 h. The Bi_2O_3 powder was prepared by heating up the same precursor solution directly in a crucible using the same temperature program (p- Bi_2O_3). The commercial Bi_2O_3 powder (Aldrich, 99.999 %, c- Bi_2O_3) was also used for comparison. Negligible amount of NiO on the surface of Ni foam in p- $\text{Bi}_2\text{O}_3/\text{Ni}$ sample was confirmed by additional control experiments as explained in the supporting information.

Characterization

The crystal structure characteristics of the samples were studied by X-ray diffraction (XRD) using a Rigaku Miniflex II X-ray powder diffractometer with $\text{CuK}\alpha$ ($\lambda = 0.15406$ nm) radiation. The morphology and microstructure were characterized by scanning electron microscopy (SEM, S-3400NII) and transmission electron microscopy (TEM, JEOL-2010). The elemental content was evaluated by energy dispersive x-ray spectroscopy (EDS) on S-3400NII.

Electrochemical properties were measured using CR-2032 coin cells. A piece of metallic lithium foil was used as the counter electrode. The p- $\text{Bi}_2\text{O}_3/\text{Ni}$ was directly used as the working electrode. For the p- Bi_2O_3 and c- Bi_2O_3 powder, the working electrode was prepared by casting slurry onto the Ni foam and drying it in a vacuum oven at 70 °C for 12 h. The slurry contained a mixture of either p- Bi_2O_3 or c- Bi_2O_3 powder as the active material, carbon black, and polyvinylidene fluoride in a weight ratio of 70:20:10 dissolved in N-methyl-2-pyrrolidone. The mass loading of active material on each anode was about 2 mg. For p- $\text{Bi}_2\text{O}_3/\text{Ni}$, we used directly the weight difference of the Ni foam measured before and after the whole process. The solution of 1 M LiPF_6 in a mixture of ethylene carbonate (EC)/dimethyl carbonate (DMC) (1:1 by volume) was used as the electrolyte in all cases. Coin cells were assembled in an argon-filled dry glovebox. Galvanostatic charge/discharge

cycling performance was evaluated on an LAND Battery Tester CT2001A at room temperature in a potential range of 0.05–2.50 V (vs. Li⁺/Li) at different current densities. The measured values were normalized per gram of active material. Cyclic voltammetry (CV) was carried out using a Princeton Applied Research Versa STAT4 electrochemical workstation. The electrochemical impedance measurement was performed on fresh coin cell samples at zero bias and with 5mV AC amplitude by CHI-680A (CH Instruments, Inc) workstation.

Results and discussion

Bi₂O₃ was successfully grown on Ni foam by our previously reported polymer-assisted solution approach.³¹ Scheme 1 illustrates the Bi₂O₃ synthesis. First, Bi³⁺ ions were bonded with EDTA/PEI polymer, forming a homogenous precursor solution. The polymer served as an adhesion agent between Bi³⁺ ions and the Ni foam in the second step of immersing Ni foam in the solution. After the heat treatment in air, Bi³⁺ ions were converted into Bi₂O₃, and the residual carbon left from the polymer acted as a binder between Bi₂O₃ particles and the current collector Ni foam.

Fig. 1 shows the XRD patterns of p-Bi₂O₃/Ni, p-Bi₂O₃ and c-Bi₂O₃ powder. The noticeable sharp peaks indicate that all samples have good crystallinity. The relatively broad peaks of p-Bi₂O₃ compared to those of c-Bi₂O₃ suggest that the p-Bi₂O₃ prepared by the polymer-assisted solution method has a smaller particle size. From the Scherrer's equation, the particle sizes can be estimated ca. 18 and 31 nm for the p-Bi₂O₃ and c-Bi₂O₃ powder, respectively. However, as shown in Fig.1, the XRD patterns, for p-Bi₂O₃ can be interpreted as consisting of two phases: monoclinic Bi₂O₃ (JCPDS No. 41-1449) and tetragonal Bi₂O_{2.33} (JCPDS No. 27-0051), while commercial powder has only monoclinic Bi₂O₃. Note that p-Bi₂O₃/Ni has exactly the same XRD pattern as that of the p-Bi₂O₃, plus additional three peaks from the Ni foam substrate.

Fig. 2(a) shows a SEM image of the typical morphology of p-Bi₂O₃/Ni over a large area, indicating that the surface of Ni foam is well covered by a sheet of Bi₂O₃. While the insert images in Fig. 2(a) and (b) shows a smooth surface of blank Ni foam. The magnified images in Figs. 2(b,c) suggest a network-like sheet morphology of the product and a good connection between Bi₂O₃ and the Ni foam. TEM image in Fig. 2(d) of the p-Bi₂O₃ powder shows the particles of 15–20 nm in size (Fig. S2 for the histogram), consistent with the XRD estimate, which are embedded in a matrix of thin carbon layers. The carbon wrapped structure helps connecting the Bi₂O₃ particles with each other. The carbon content was determined as 10.8 wt% by EDS (see Fig. S3 and Table S2). The high resolution TEM image in Fig. 2(e) shows that the lattice spacing ca. 0.408 nm, corresponding to spacing between the (020) planes of monoclinic Bi₂O₃. For comparison, the TEM image of c-Bi₂O₃ powder, given in Fig. 2(f) and Fig. S4, shows disordered fragments and aggregated larger particles.

The electrochemical properties of p-Bi₂O₃/Ni were investigated by galvanostatic charging and discharging in a voltage range of 0.05–2.5 V (vs. Li⁺/Li). For comparison, p-Bi₂O₃ and c-Bi₂O₃ were investigated as well. Fig. 3(a) shows several selected cycles of the charge/discharge voltage profiles of p-Bi₂O₃/Ni at a current density of 100 mA/g. As indicated, the initial discharge/charge capacities are 1923 mAh/g and 1211 mAh/g, respectively, with the Coulombic efficiency of 63 %. Complete reaction of Bi₂O₃ with Li should involve 12 Li ions according to the following reaction:

The corresponding theoretical capacity can be calculated as 690 mAh/g. Bi_2S_3 also went through a similar reaction.¹⁶ In the second cycle, a discharge capacity of 1346 mAh/g indicated that 30 % of the initial discharge capacity is irreversible. This can be attributed to decomposition of the electrolyte and formation of solid electrolyte interface (SEI) on the electrode surface.^{1,4,6} The discharge capacities of the 5th and 10th cycles remain large, 1201 and 1034 mAh/g respectively, corresponding to retention of 89 % and 77 % as compared to the second discharge capacity (1346 mAh/g), and indicating a much better cycle performance than other Bi involved compounds, such as Bi_2S_3 ,^{16–19} Cu_3BiS_3 ,³² and Bi_2Te_3 .²⁰ The corresponding voltage profiles of p- Bi_2O_3 and c- Bi_2O_3 are given in Fig. S5. Fig. 3(b) further illustrates the variation of capacity and demonstrates that p- $\text{Bi}_2\text{O}_3/\text{Ni}$ still retains a capacity of 782 mAh/g after 40 cycles at a current density of 100 mA/g. A possible contribution of NiO formed on p- $\text{Bi}_2\text{O}_3/\text{Ni}$ is very limited because of its negligible weight and low specific capacity (Fig. S1). In comparison, the p- Bi_2O_3 anode has a much lower capacity, which also decreases rapidly after several cycles. The performance of c- Bi_2O_3 powder is even more inferior. A relatively better performance of p- Bi_2O_3 compared to c- Bi_2O_3 may be due to the smaller particle size in p- Bi_2O_3 and better interconnection between Bi_2O_3 nanoparticles in a network-like structure through thin carbon layers, as indicated above by their XRD and TEM analyses. We believe that there are at least three reasons for p- $\text{Bi}_2\text{O}_3/\text{Ni}$ delivering a significantly enhanced electrochemical performance. Firstly, this binder-free anode formulation eliminates the need for a polymer binder and carbon black and bypasses the conventional preparation process via intermediate slurries. Secondly, direct growth of Bi_2O_3 on Ni foam allows more uniform distribution of the active material on the current collector, which leads to a larger surface area per unit volume of the electrode and a higher volumetric utilization efficiency. It can effectively alleviate the huge volume change during the charge/discharge process.^{33,34} Thirdly, the good intrinsic connection between the active material and the current collector enhances the mass transport and shortens the lithium ion diffusion path.³⁵ These three superiorities together with the relatively smaller particle size can be responsible to the fact that the capacity delivered by p- $\text{Bi}_2\text{O}_3/\text{Ni}$ is higher than the theoretical capacity of Bi_2O_3 . This phenomenon has been observed on other anode materials as well.^{36–39} The advantages of this binder free electrode can again be confirmed by the SEM images (Fig. S6) of all these three samples after 40 cycles at a current density of 100 mA/g. The Bi_2O_3 particles are still covered uniformly on the surface of the nickel foam with some cracks, while all the Bi_2O_3 particles aggregated together in p- Bi_2O_3 and c- Bi_2O_3 , showing an inferior volumetric utilization efficiency and worse stability.

The rate performance of p- $\text{Bi}_2\text{O}_3/\text{Ni}$, shown in Fig. 3(c), illustrates that its high capacities are observed even at high current densities, namely, 1130, 910, 775, and 668 mAh/g for 200, 400, 600, and 800 mA/g, respectively. When the current density decreased back to 100 mA/g, the specific capacity rebounded to 906 mAh/g, *i.e.*, showed a 68 % retention rate.

The AC impedance measurements can provide additional insight on the anode performance. Fig. 3(d) compares the Nyquist plots of p- $\text{Bi}_2\text{O}_3/\text{Ni}$, p- Bi_2O_3 , and c- Bi_2O_3 electrodes. Each plot consists of a semicircle at high-medium frequency region representing the charge-transfer resistance and the charging capacitor, accompanied by a straight line at lower frequencies, corresponding to the Warburg impedance due to diffusion.^{1,40,41} The p- $\text{Bi}_2\text{O}_3/\text{Ni}$ electrode shows a smaller charge transfer resistance, indicating improved kinetic transport for the electrode reactions and good electrical contact.⁴¹ Larger charge transfer resistances of p- Bi_2O_3 and c- Bi_2O_3 may be responsible for their lower capacity.

The cyclic voltammetry (CV) of p- $\text{Bi}_2\text{O}_3/\text{Ni}$, p- Bi_2O_3 , and c- Bi_2O_3 evaluated in the potential range of 0.05–2.5 V (vs. Li^+/Li) at a scan rate of 0.2 mV/s, are shown in Fig. 4. The multi-peak features of the CV curves indicate that the reaction of Bi_2O_3 with Li proceeds in a multi-step fashion. For p- $\text{Bi}_2\text{O}_3/\text{Ni}$, there are three obvious cathodic peaks in

the first scan at potentials ~1.4, ~1.2, and ~0.45 V. In accordance with a current consensus,^{24,42,43} we assign the 1.2 V peak, appearing in the CVs of all samples, to reduction of Bi₂O₃ to Bi (see reaction 2 below). The peak around 1.4 V may result from the reduction of tetragonal Bi₂O_{2.33} (reaction 3), which only appears in p-Bi₂O₃/Ni and p-Bi₂O₃ but not in c-Bi₂O₃. The cathodic peak at 0.45 V is associated with both the formation of SEI and the reaction of Bi with Li to form Li₃Bi. The peak is replaced by two small peaks around 0.75 and 0.7 V, indicating the lithiation of Bi divided into two steps (formation of LiBi and Li₃Bi separately) in the subsequent cycles.^{15,16,24} The obvious anodic peak at around 0.9 V is attributed to the de-alloying process^{15,16} and the peaks at 1.75 and 2.25 V are associate with the oxidation of Bi to Bi₂O₃. These two peaks are less pronounced in p-Bi₂O₃ and c-Bi₂O₃ and do not survive in the subsequent cycles, which indicates only partial reversibility of conversion between Bi₂O₃ and Bi, similar to the previously reported performance of SnO₂.^{10,11,44} For p-Bi₂O₃/Ni, the reversibility of the redox reaction between Bi₂O₃ and Bi is significantly improved. Thus, the reaction mechanism of charging and discharging Bi₂O₃ can be represented as following:

Discharging:

Charging:

Good replication of the CV shapes in the subsequent cycles of p-Bi₂O₃/Ni demonstrates excellent reversibility and structural stability of this construction. Weaker redox peaks for c-Bi₂O₃ and p-Bi₂O₃ further suffer gradual decline with increasing the cycle number, indicating poor reversibility during the lithiation and delithiation processes.

Conclusions

In summary, we report on the first successful application of Bi₂O₃ as an anode material for LIBs. Facile polymer-assisted synthesis of Bi₂O₃ on Ni foam (p-Bi₂O₃/Ni) shows superior electrochemical properties in comparison with the standard slurry casting produced using either polymer-assisted (p-Bi₂O₃) or commercial (c-Bi₂O₃) powders on Ni foam. It has been demonstrated that the capacity of p-Bi₂O₃/Ni is as high as 782 mAh/g after 40 cycles at the current density of 100 mA/g and 668 mAh/g at the current density of 800 mA/g after 20 cycles. This one-step, binder-free strategy for p-Bi₂O₃/Ni electrode construction shows the advantages of a high volumetric utilization efficiency and short lithium ion diffusion path, which greatly enhances the electrochemical performance.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

Acknowledgments

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Fig. 1. XRD patterns of p-Bi₂O₃/Ni (a), p-Bi₂O₃ powder (b), and c-Bi₂O₃ powder (c).

Fig. 2. (a) Low-, (b) medium-, and (c) high magnification SEM images of p-Bi₂O₃/Ni with the inserts showing SEM images of blank Ni foam, (d) TEM and (e) HRTEM images of p-Bi₂O₃ powder; and (f) TEM image of c-Bi₂O₃ powder.

Fig. 3.

(a) Voltage profile of p-Bi₂O₃/Ni at a current density of 100 mA/g; (b) Cycle performance of p-Bi₂O₃/Ni, p-Bi₂O₃ powder, and c-Bi₂O₃ powder; (c) Rate performance of p-Bi₂O₃/Ni; (d) Nyquist plots of p-Bi₂O₃/Ni, p-Bi₂O₃ powder, and c-Bi₂O₃ powder.

Fig. 4. Cyclic voltammetry profiles of p-Bi₂O₃/Ni (a), p-Bi₂O₃ (b), and c-Bi₂O₃ (c) of the first five cycles at a scan rate of 0.2 mV/s; (d) the First cycle of cyclic voltammetry profiles for all three samples.

Scheme 1.
Schematic diagram of growth mechanism of p-Bi₂O₃/Ni.