Electronic Supplementary Information (ESI) for

Highly active Fe$_3$BO$_6$ as an anode material for sodium-ion batteries

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Experimental details

Preparation of Fe$_3$BO$_6$

The Fe$_3$BO$_6$ nanoparticles were synthesized via a facile solid state method. All reagents were purchased from Sinopharm Chemical Reagent Co., Ltd (Shanghai) without further treatment. In a typical experiment, FeC$_2$O$_4$·2H$_2$O and H$_3$BO$_3$ were mixed by ball milling in a molar ratio of 3:1.2 with a proper amount of deionized water as a dispersant. After the grinding and mixing, the obtained rheological phase was transferred into an autoclave and kept at 80 °C for 12 h. The precursor was then calcined at 800 °C for 5 h with a heating rate of 3 °C /min in air. After cooling down to ambient temperature, the product was washed 3 times using boiling water to remove the unreacted boron oxide. In addition, the Fe$_3$BO$_6$@C sample was prepared by mixing Fe$_3$BO$_6$ with oleic acid and then heating it at 500 °C in N$_2$.

Characterization of the materials

The crystal structure of the samples was characterized by an X-ray diffractometer (XRD, Rigaku RINT 2200). XRD data were gained with Cu K$_{α1}$ radiation ($λ = 0.15406$ nm) in the $2θ$ range of 20–80° with a step size of 0.02° and a scan rate of 2° per minute. The morphologies and structure of the Fe$_3$BO$_6$ nanoparticles were investigated by scanning electron microscopy (SEM, Hitachi S-3500N) and transmission electron microscopy (TEM, JEM-2100F, JEOL, Japan). X-ray photoelectron spectroscopy (XPS) tests were carried out on a Kratos Axis UltraDLD spectrometer (Kratos Analytical - A Shimadzu Group company) with monochromatic Al Kα radiation ($hν = 1486.6$ eV).

Electrochemical measurements

The Fe$_3$BO$_6$ powders were mixed with sodium carboxymethyl cellulose (CMC, WALOCEL™ CRT 2000 PPA 12, Dow Wolff Cellulosic) and acetylene black to form a slurry with a weight ratio of 80:10:10. The working electrode was manufactured by casting the slurry on copper foil substrate, which was dried at 80°C overnight. Disks with an area of 1.54 cm$^2$ were punched out of the foil, and the
average mass loading of active material on each disk was about 1.0 mg. In CR2016 coin cells, the Fe$_3$BO$_6$ samples and metallic sodium were using as working electrode and counter electrode, respectively. 1 M NaClO$_4$ (98% Sigma Aldrich) in ethylene carbonate (EC) and diethyl carbonate (DEC) (1:1 by volume) was used as the electrolyte. The cells were assembled in an argon-filled glove box (Mikrouna-China Super 1220/750). The electrochemical properties of the cells were tested using a battery tester (LAND CT2001A Wuhan, China) in the voltage range of 0.01-3.0 V under different current densities. The cyclic voltammetry (CV) tests were carried out on an electrochemical workstation (Autolab PGSTAT 302N) at a scan rate of 0.01 mV s$^{-1}$ in the voltage range of 0.01-3.0 V. The electrochemical impedance spectroscopy (EIS) analysis was conducted on an Autolab PGSTAT 302N electrochemical workstation from 100 kHz to 0.1 Hz with potentiostatic signal amplitude of 5 mV in the fully charged state. The working potential for EIS tests is stable at open circuit potential (about 1.7 V) after charging to 3.0 V (vs. Na$^+$/Na) and resting for 6 h.
Fig. S1 (a) Low-magnification SEM image of as-prepared Fe$_3$BO$_6$; (b) SEM image of the FeC$_2$O$_4$·2H$_2$O precursor.
Fig. S2 XPS spectra of the Fe$_3$BO$_6$ sample: (a) survey spectrum, (b) Fe 2p, (c) B 1s, and (d) O 1s.
Fig. S3 Initial three charge-discharge curves of Fe$_3$BO$_6$ electrode at 100 mA g$^{-1}$. 
Table S1

Table S1: Solution resistance ($R_s$), charge transfer resistance ($R_{ct}$), and constant phase angle element ($CPE$) derived from the equivalent circuit model of EIS curves.

<table>
<thead>
<tr>
<th>Cycles</th>
<th>$R_s$/Ω</th>
<th>$R_{ct}$/Ω</th>
<th>$Y_0$/μF</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>1&lt;sup&gt;st&lt;/sup&gt;</td>
<td>1.98</td>
<td>100</td>
<td>225</td>
<td>0.60</td>
</tr>
<tr>
<td>2&lt;sup&gt;nd&lt;/sup&gt;</td>
<td>2.41</td>
<td>94.4</td>
<td>213</td>
<td>0.63</td>
</tr>
<tr>
<td>30&lt;sup&gt;th&lt;/sup&gt;</td>
<td>2.67</td>
<td>102</td>
<td>192</td>
<td>0.63</td>
</tr>
<tr>
<td>80&lt;sup&gt;th&lt;/sup&gt;</td>
<td>3.24</td>
<td>125</td>
<td>190</td>
<td>0.61</td>
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