Fast Lithium Transport in PbTe for Lithium-Ion Battery Anodes

Sean M. Wood, Kyle C. Klavetter, Adam Heller and C. Buddie Mullins*

Departments of Chemical Engineering and Chemistry, Center for Electrochemistry, Texas Materials Institute, University of Texas at Austin, 1 University Station, C0400 Austin, TX 78712-0231, United States

Electronic Supplemental Information

PbO Synthesis

To prepare massicot PbO nanoparticles, a first solution was created by adding 15.9 g of Pb(NO$_3$)$_2$ to 40 mL of distilled H$_2$O, stirring, and heating to 90 °C. A second solution was created by adding 30.4 g of NaOH to 40 mL of water (19 M) with stirring. Both of these solutions must be prepared in quartz glassware. If borosilicate glass is used, a small amount of SiO$_2$ must be added. If Teflon beakers are used, the litharge form of PbO will be synthesized. The first solution was quickly poured into the second solution and allowed to stir for about 30 seconds. Stirring was stopped and the solution sat for another 60 seconds to allow the particles to settle. The solution was decanted and the particles were rinsed with ice cold distilled water. The particles were then centrifuged and washed three times with distilled water and absolute ethanol. The particles were dried in a vacuum oven at 70 °C overnight.

PbO Electrochemical Characterization

PbO electrodes were prepared by forming a slurry comprised of as-prepared PbO nanoparticles (40 wt %), polyacrylonitrile (20 wt %) binder, and Super P Li conductive carbon (Timcal, 40 wt %), using dimethylformamide as the solvent. This formulation was selected because the particles did not create a functioning slurry with the carboxymethyl cellulose/water preparation. The use of polyacrylonitrile as a binder does not significantly affect the cycling results. The slurry was cast onto copper foil and dried in a vacuum oven at 120 °C for at least 6 h. This film was punched into disks that formed the working electrodes of CR 2032 coin-type cells. Each electrode had a mass of approximately 0.2 mg cm$^{-2}$. Coin cell construction was the same as for PbTe.
Supplemental Figures

Figure S1. (a) XRD pattern of the as-synthesized PbO nanoparticles. (b) PbO reference spectrum (JCPDS # 01-076-1796). (c) SEM image of the PbO nanoparticles.
Figure S2. Cycling voltammograms of PbTe at a scan rate of 0.1 mV s\(^{-1}\) in the voltage range (a) 0.01 – 2.5 V and (b) 0.01-1.8 V.
Figure S3. Coulombic efficiency versus cycle index in two voltage ranges at (a) C/5 and (b) varying C-rates.
Figure S4. Discharge capacity versus cycle index for PbO in two voltage ranges at varying C-rates.

Figure S5. Differential capacity profiles for PbTe at varying C-rates over the voltage range 0.01-2.5 V.