Electronic Supplementary Material (ESI) for Chemical Communications

Supporting Information

Does $Li_4Ti_5O_{12}$ need carbon in lithium ion batteries? : Carbon-free electrode with exceptionally high electrode capacity

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Experimental

$Li_4Ti_5O_{12}$ was synthesized by ball-milling and subsequent heat-treatment. Stoichiometric amounts of Li_2CO_3 (Aldrich) and anatase TiO_2 were ball-milled for 1 hour by using a vibrant-type mill (SPEX8000 mixer/mill) and the mixture was heat-treated at 850 ^oC for 5 hours in air.

The structure and phase-purity of $Li_4Ti_5O_{12}$ were analyzed by X-ray diffraction (XRD, Philips X'Pert MPD, Cu Kα radiation) and its morphology was observed by scanning electron microscopy (SEM, Hitachi S-4700). Their results were illustrated in supporting Fig. S1 and S2.

X-ray photoelectron spectroscopy (XPS) analyses were performed with Φ PHYSICAL ELECTRONICS (QUANTUM 2000 SCANNING ESCA MICROPROBE) spectrometer using a focused monochromatized Al Kα radiation (1486.6 eV). To avoid any external contamination, all samples were transferred under inert gas from the glove box to the spectrometer using a transfer chamber.

$Li_4Ti_5O_{12}$ electrodes were prepared by slurry casting and laminating process. The electrode compositions are $Li_4Ti_5O_{12}$: carbon black (SuperP) : poly vinylidene fluoride (PVdF) = 98:0:2 and 90:5:5 in weight. 2032 coin-type cells for electrochemical characterization were assembled by stacking a microporous polyethylene separator containing liquid electrolyte (1.3 M $LiPF_6$ in EC/DEC/DMC (ethylcarbonate/diethylcarbonate/dimethylcarbonate) 3/5/2 in volume) between lithium foil (LectroMax 100) and $Li_4Ti_5O_{12}$ electrode. The cells were charged and discharged galvanostatically at various C-rates between 2.5 and 1.0 V (*vs*. Li/Li^+) using a battery tester (Toyo, Toscat-3000U). Typically constant-current/contant-voltage mode (CC/CV) was applied during charge (lithium insertion) and constant-current mode (CC) during discharge (lithium de-insertion). However, the reverse condition was used to test only charge rate-capability. In CC/CV mode, CV was fixed to 1/20C.

Figure S1. XRD pattern of $Li_4Ti_5O_{12}$ and its reference position (code: 01-072-0426 in X'Pert HighScore Plus).

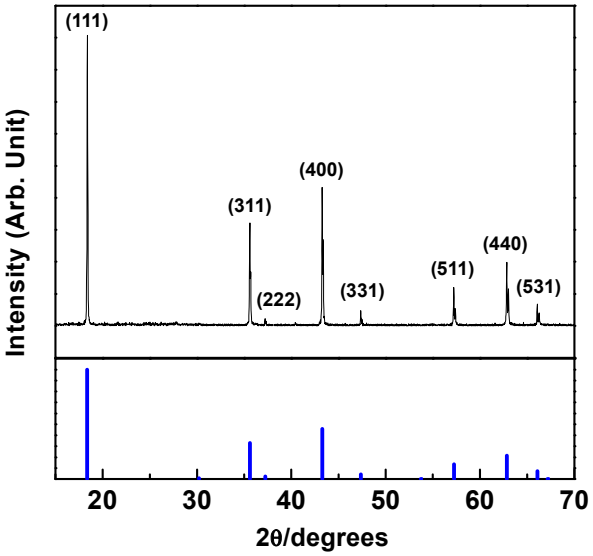

Figure S2. SEM image of $Li_4Ti_5O_{12}$. $Li_4Ti_5O_{12}$ particles with uniform and spherical morphology are observed.

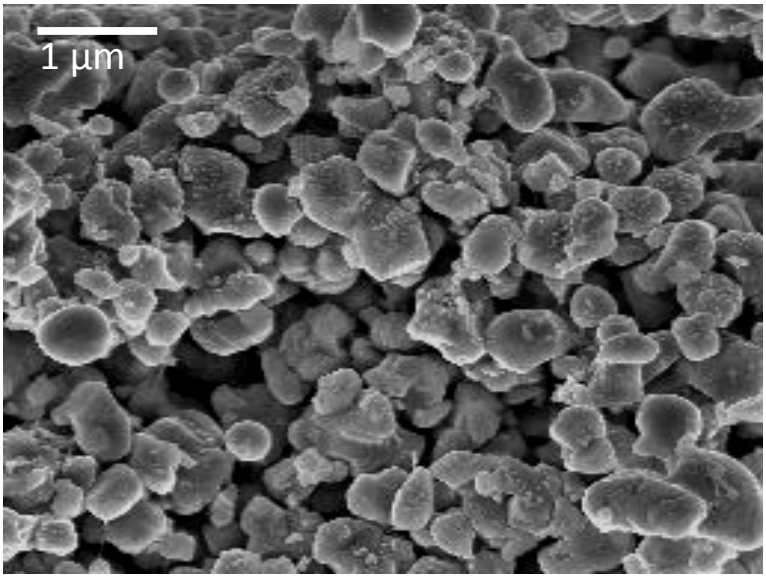

Figure S3.(A) Comparison between cyclic voltammograms (CVs) of $Li_4Ti_5O_{12}$ electrodes with and without carbon. CVs were recorded between 2.5 and 1.0 V at a scan rate of 0.1 mV s^{-1}. The results are in good agreement with the charge/discharge profiles shown in Fig. 1.

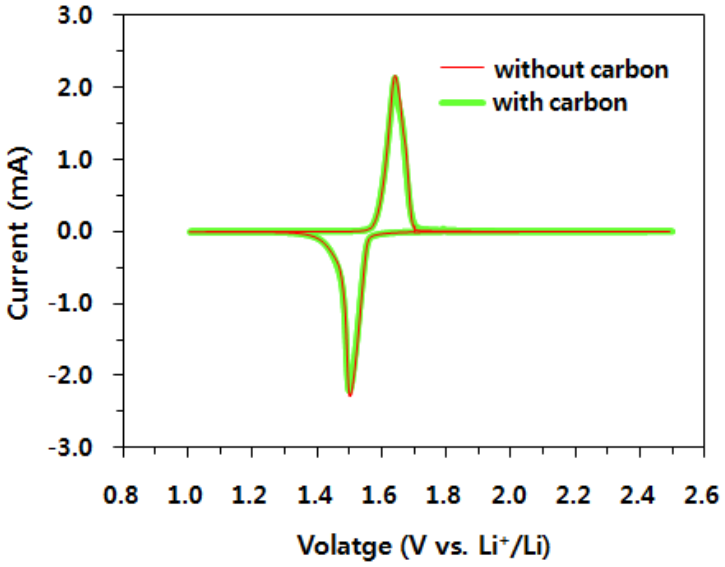

Figure S4. Charge and discharge voltage curves during the rate capability test (see Fig. 3A).

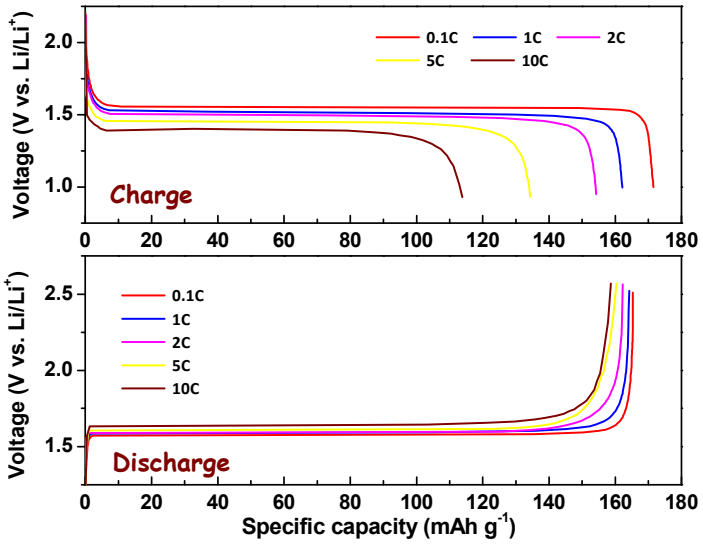

Figure S5. Discharge rate-capability of nano-$Li_4Ti_5O_{12}$ (Carbon-free electrode with a composition 98 $Li_4Ti_5O_{12}$: 2 PVdF wt.%) at room temperature. Commercially available nano-$Li_4Ti_5O_{12}$ (nGimat Co.) was used to verify the carbon-free electrode concept. It is the same powder characterized in elsewhere (K.-S. Park et al. *J. Am. Chem. Soc.* **2008**, *130*, 14930–14931).