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A vapor-phase carbon-deposition route to efficient inorganic nanosheet-based electrodes

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ABSTRACT

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1. Introduction

Recently 2D nanosheets of inorganic solids attract a great deal of research interest because of their unique physicochemical properties and valuable functionalities [1]. In comparison with graphene, these inorganic nanosheets boast much greater diversity in chemical compositions, crystal structures, and applicabilities [2]. Among them, 2D nanosheets of high-valent transition metal oxides such as titanoniobate are promising candidates for electrode applications for lithium ion batteries (LIB) and supercapacitors, since they can accommodate large amount of Li^+ ions via reduction process of Ti^{4+} and Nb^{5+} ions [3]. Of prime importance is that highly anisotropic 2D nanosheet morphology with very thin thickness is fairly advantageous for repeated lithiationdelithiation process because of the provision of many surface reaction sites, short Li⁺ diffusion paths, and high electrochemical stability [4]. However, the poor electrical conductivity of this material severely limits its electrode performance especially under high current density condition. To circumvent this drawback of titanoniobate material, it is demanded to explore an efficient carbon deposition method for metal oxide 2D nanosheet without a significant frustration of 2D morphology. Taking into account excellent diffusivity and high reactivity of gaseous C_2H_2 reactant [5], a reaction with C_2H_2 vapor is supposed to be effective in depositing carbon species on the surface of inorganic 2D nanosheets. To the best of our knowledge, there is no other work about the carbondeposition of inorganic 2D nanosheet via C2H2 treatment and its

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fective route to high-performance inorganic nanosheet-based electrode materials.

Efficient inorganic nanosheet-based electrode materials can be synthesized by the calcination of ex-

foliated Ti_5NbO_{14} nanosheets under C_2H_2 flow. While the calcination in Ar atmosphere causes a phase

transformation from layered Ti₅NbO₁₄ to TiO₂ and Nb₂O₅, employing C₂H₂ atmosphere leads to the

maintenance of the original layered structure of Ti₅NbO₁₄ upon the heat-treatment, which is attributable

to the stabilization of layered lattice by surface passivation by deposited carbon layer. The resulting

carbon@titanoniobate materials show mesoporous house-of-cards-type stacking structure of 2D na-

nosheets. This carbon@titanoniobate material delivers large discharge capacity of \sim 320 mA h g⁻¹ with

excellent cyclability and rate performance, which is much superior to that of carbon-free homologue. The

present study clearly demonstrates that the heat-treatment under C₂H₂ flow provides a simple and ef-

In the present study, efficient electrode materials of carbon@titanoniobate nanosheets is synthesized by the heat-treatment of restacked precursor nanosheet under C_2H_2 flow, as illustrated in the left panel of Fig. 1. The carbon@titanoniobate nanosheets are applied as LIB electrodes to probe the usefulness of vapor-phase carbon-deposition method for exploring efficient inorganic nanosheet-based electrode materials.

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2. Experimental

The exfoliated titanoniobate 2D nanosheet was prepared by the protonation of K₃Ti₅NbO₁₄ and the following intercalation of tetrabutylammonium cations [6]. The exfoliated titanoniobate nanosheet was restored by the electrostatically-derived restacking with proton. The restored titanoniobate nanosheet was heated at 400 °C under 5% C₂H₂ gas diluted with Ar gas for 2, 5, and 10 h to synthesize carbondeposited titanoniobate nanosheets (These materials are denoted as carbon@titanoniobate-2, carbon@titanoniobate-5, and carbon@titanoniobate-10, respectively). For comparison, the calcination in Ar atmosphere was also done at 400 °C for 5 h (This carbon-free material is denoted as titanoniobate-5). Powder X-ray diffraction (XRD) and transmission electron microscopy (TEM)/field emission-scanning electron microscopy (FE-SEM) were carried out to examine the crystal structures and morphologies of the present materials, respectively. The chemical compositions, bonding natures, and pore structures of these materials were determined with energy dispersive spectrometry (EDS) - elemental mapping analysis, thermogravimetric analysis









Fig. 1. (Left) Schematic illustration of the synthesis of carbon@titanoniobate nanosheet. (Right) Powder XRD patterns of (a) restored titanoniobate, (b) carbon@titanoniobate-2, (c) carbon@titanoniobate-5, (d) carbon@titanoniobate-10, (e) titanoniobate-5, (f) anatase TiO_2 , (g) Nb_2O_5 , and (h) layered $K_3Ti_5NbO_{14}$ (PDF 72-0908).

(TGA), micro-Raman spectroscopy, and N_2 adsorption-desorption isotherm measurement, respectively. The electrode functionalities of

the obtained nanocomposites were characterized with galvanostatic discharge–charge process with the 2016 coin-type cell of Li/1 M $LiPF_6$



Fig. 2. (Top) TEM and (bottom) FE-SEM data of (a) restored titanoniobate, (b) carbon@titanoniobate-2, (c) carbon@titanoniobate-5, (d) carbon@titanoniobate-10, and (e) titanoniobate-5.

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