Supporting Information for

Facile Insertion of Lithium into nanocrystalline AlNbO₄ at Room Temperature

Experimental:

To demonstrate the crystallite size effects, we prepared micrometer and nanometer-sized AlNbO₄. Micrometer-sized samples of AlNbO₄ are prepared by the conventional solid state reaction (SSR) starting from the stoichiometric amounts of Al₂O₃ and Nb₂O₅ (Alfa, 99.9 %). The reactants are ground well and are calcined at 900 °C for 24h in air. The resulting powder is reground and finally calcined at 1200 °C for 24h and the sample is allowed to cool to RT. Nanometer-sized samples are prepared by polymerizable complex (PC) method. The starting precursors used for this method are Al(NO₃)₃.9H₂O (Fulka, 98 %) and Nb₂O₅.xH₂O. The Nb₂O₅.xH₂O precursor is prepared according to the procedure reported elsewhere.²⁰ The value of x is estimated to be 4.25 from thermo gravimetric analysis. Requisite amount of citric acid (CA) (Spectrochem, 99.7 %) is dissolved in distilled water and the resulting solution is kept at 60 °C. Nb₂O₅.xH₂O is dissolved in the above solution. After complete dissolution of Nb₂O₅.xH₂O precursor, Al(NO₃)₃.9H₂O is added and the solution is maintained at 60 °C for a few hours to ensure metal-complex formation. Ethylene glycol (EG) is now added and the temperature is raised to 90 °C to facilitate the polyesterfication step. The metal: CA: EG mole ratio is maintained as 1:4:16. The obtained gel is dried at 120 °C and is decomposed by heating at 300 °C for 4h. The resulting powder is calcined at 800 °C for 4h to obtain nanocrystalline AlNbO₄.

The phase purity of the synthesized samples was checked using Rigaku mini flex XRD (Japan) using Cu Kα radiation. Lattice parameters were obtained using Autoexe program. A scanning electron microscope (SEM) Philips Field Effect Gun (FEG) XL-30 and transmission electron microscopy (TEM) JEOL 3010 was used to study the sample morphology. Electrodes were fabricated by mixing active material, acetylene black (Denka Singapore Pvt. Ltd.) and poly vinylidene fluoride (PVDF) in the weight ratios 70:20:10. Slurry containing the above mixture was prepared by using N-methyl-2-pyrrolidinone and was spread on a stainless steel foil and dried in an oven at 100 °C for 12h. Swagelok cells were used for electrochemical studies. The cells were fabricated in an argon filled glove box (mBraun, Germany) with Li foil as anode, Teklon (Anatek, USA) as separator and 1 M LiPF₆ in 1:1 ethylene carbonate plus dimethyl carbonate as the electrolyte (Chiel Industries Ltd., Korea). Charge discharge cycling of the cells was carried out in galvanostatic mode at RT by using Arbin battery cycling unit (BT2000, USA).

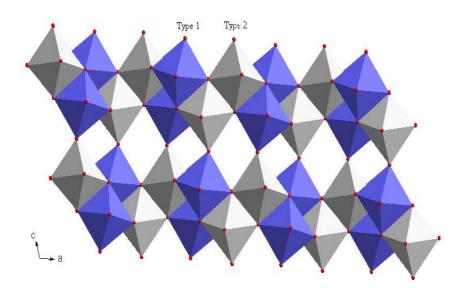


Figure S1. Crystal structure view of AlNbO₄ in ac plane

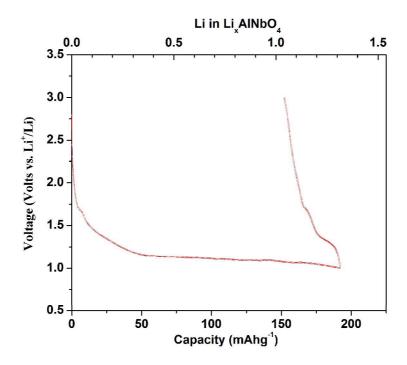


Figure S2 Voltage-composition profile of micrometer-sized AlNbO $_4$ obtained at C/100 rate (~1.4 mA g $^{\text{-1}}$)