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Facile synthesis of $MTaO_4$ (M = Al, Cr and Fe) metal oxides and their application as anodes for lithium-ion batteries



Fanjun Kong^{a,b}, Shi Tao^b, Bin Qian^{a,b,*}, Lei Gao^{a,*}

^a College of Physics, Optoelectronics and Energy of Soochow University, Collaborative Innovation Center of Suzhou Nano Science and Technology, Soochow University, Suzhou 215006, China

^b Department of Physics and Electronic Engineering, Changshu Institute of Technology, Changshu 215500, China

ARTICLE INFO	A B S T R A C T				
Keywords:	- Ternary metal oxides have attracted much attention in energy storage fields. Herein, tantalum-based oxides				
Tantalates	MTaO ₄ ($M = A$), Cr and Fe) are synthesized by a facile co-precipitation method, and their performance as				
Chemical properties Electrodes Batteries	lithium-ion battery anodes are evaluated. Among them, the FeTaO ₄ electrode presents superior electrochemical performance compared to the $MTaO_4$ ($M = Al$ and Cr) and a reversible capacity of more than 200 mAh g ⁻¹ can be maintained after 100 cycles, while a capacity of 53.6 and 128.9 mAh g ⁻¹ can be obtained at the same condition for AlTaO ₄ and CrTaO ₄ , respectively. The explanation that FeTaO ₄ exhibits the excellent electrochemical performance in $MTaO_4$ ($M = Al$, Cr and Fe) are further discussed.				

1. Introduction

Lithium-ion batteries (LIBs) with outstanding advantages of high energy density, long-cycling life, safety and environmental friendliness are to meet the demand of portable devices, electrical appliances and even large scale equipments [1–4]. The graphite as an anode material in commercial LIBs has some disadvantages such as the lithium consumption during the formation of solid electrolyte interphase and safety hidden trouble related to the lithium dendrites at high rate, which limit its development [5–8]. Thus the potential alternative anodes are largely investigated for the higher power density and safety.

Recently, niobium-based oxides are with special structure broadly studied due to excellent electrochemical properties. Binary Nb-based nano-oxides, mainly Nb₂O₅, are applied into LIBs, supercapacitors solar cells and photocatalytic fields [9-14]. Ternary niobium-based oxides with different molecular formula and structure have attracted extensive attention owing to multi redox couples and high redox potentials. $TiNb_2O_7$ and $Ti_2Nb_{10}O_{29}$ with monoclinic structure have Ti^{4+}/Ti^{3+} , Nb^{5+}/Nb^{4+} and Nb^{4+}/Nb^{3+} redox couples, and deliver considerable capacity [15-17]. The electrochemical lithium insertion of MNb₂O₆ (M = Cr, Mn, Fe, Co, Ni, Cu, Zn, Cd) were reported [18,19]. Reddy and Varadaraju prepared nanometer-sized sample AlNbO₄ with a reversible of 130 mAh g⁻¹ and its lithium insertion mechanism can be described and proved by our group as follows: AlNb $O_4 + xLi^+ + xe^- \rightarrow Li_xAlNbO_4$ [20,21]. Shim et al. synthesized orthorhombic FeNbO4 via hydrothermal method and deliver a specific capacity of over 200 mAh g⁻¹ after 20 cycles [22]. The reversible lithium insertion of FeNbO₄ was reported and facilitated either by the Nb⁵⁺/Nb⁴⁺ redox couple or by Nb⁵⁺/Nb⁴⁺ and Nb⁴⁺/Nb³⁺ redox couples [23]:

 $\text{FeNb}^{V}\text{O}_{4} + x\text{Li}^{+} + xe^{-} \rightarrow \text{Li}_{x}\text{FeNb}_{1-x}^{V}\text{Nb}_{x}^{IV}\text{O}_{4}$

 $\text{Li}_x \text{FeNb}_{1-x}^V \text{Nb}_x^{\text{IV}} \text{O}_4 + y \text{Li}^+ + y \text{e}^- \rightarrow \text{Li}_{x+y} \text{FeNb}_{1-x}^V \text{Nb}_{x-y}^{\text{IV}} \text{Nb}_y^{\text{III}} \text{O}_4$.

The CrNbO₄ nanoparticles synthesized by co-precipitation maintain 210 mAh g⁻¹ after 50 cycles higher than the capacity of microparticles (89 mAh g⁻¹), indicating that the particle size can affect the electrochemical properties owing to nanoparticles can shorten diffusion path lengths and mitigate strain related to insertion reactions [24].

Although tantalum-based oxides have similar structure to niobiumbased oxides, there is little report on the use in LIBs, especially ternary tantalum-based oxides. In this work, the AlTaO₄ with monoclinic structure and MTaO₄ (M = Cr and Fe) with tetragonal structure were firstly prepared via a simple co-precipitation method. The electrochemical performance of MTaO₄ (M = Al, Cr and Fe) are reported and compared for the first time. We also explain that FeTaO₄ exhibits the excellent electrochemical performance in MTaO₄ (M = Al, Cr and Fe) from the physical and chemical aspects.

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^{*} Corresponding authors.

E-mail addresses: njqb@cslg.edu.cn (B. Qian), leigao@suda.edu.cn (L. Gao).

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2. Experimental

2.1. Synthesis

MTaO₄ (M = Al, Cr and Fe) were prepared using co-precipitation method. Stoichiometric amounts of TaCl₅ and M(NO₃)₃·9H₂O (M = Al, Cr and Fe) were respectively dispersed into the absolute ethyl alcohol and stirred for 30 min. The pH value of solutions were adjusted at ~ 7.0 for AlTaO₄ and 9.0 for MTaO₄ (M = Cr and Fe) using ammonia hydroxide. The resulting precipitates were centrifugated, washed with ethanol and deionized water several times, and dried at 80 °C in a vacuum oven for overnight. The obtained powders were grounded and annealed at different temperature for 6 h in air and labeled as MTO (M= A, C and F).

2.2. Characterizations

The phase and structure of $MTaO_4$ (M = Al, Cr and Fe) were identified using powder X-ray diffraction (XRD) (Rigaku Dmax-2200 X-ray diffractometer) with Cu K α radiation. Morphology analysis was characterized by field emission scanning electron microscopy (FESEM, SIGMA, 20 kV) coupled with an EDX spectrometer, transmission electron microscopy (TEM, JEOL-2000CX, 200 kV) and high-resolution TEM (HRTEM, JEOL-2010F, 200 kV). The BET specific surface areas were tested from the N2 adsorption and desorption isotherms using Micromeritics tristar II Plus instrument (Micromeritics, USA).

2.3. Electrochemical measurements

The slurries were produced through mixing active material, Super P carbon and polyvinylidene difluoride (PVdF) in a weight ratio of 8:1:1 in N-methyl pyrrolidinone (NMP) and coated on a copper (Cu) foil and dried at 120 °C overnight in a vacuum oven. Coin cells (CR2016) were assembled in the Ar-filled glovebox with lithium foil as counter electrode and Celgard 2500 membrane as the separator. A solution of $1 \text{ mol L}^{-1} \text{ LiPF}_6$ dissolved in ethylene carbonate, dimethyl carbonate and ethyl-methyl carbonate with a volume ratio of 1:1:1 as the

Table 1 Refined structural parameters and BET specific surface areas for MTO (M = A, C and F).

samples	structure	lattice parameters/Å			weighted factors/%		$\frac{\text{BET}}{\text{m}^2\text{ g}^{-1}}$
		a	b	c	R _{wp}	R _p	
ATO CTO FTO	monoclinic tetragonal tetragonal	12.2014 4.6550 4.6937	3.7596 4.6550 4.6937	6.5001 3.0276 3.0576	8.69 5.46 7.24	6.18 4.22 5.87	1.99 3.13 4.36



Fig. 2. Crystal structure view of (a, c) ATO and (b, d) MTO (M = C and F).

electrolyte. Galvanostatic discharge-charge tests were executed on a battery test system (LAND CT2001A, China) at various current densities. Cycle voltammetry (CV) were performed on the VSP



Fig. 1. Refined XRD patterns of (a, a') ATO, (b, b') CTO and (c, c') FTO.

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