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Original Research Paper

Characterization and synthesis of $KTa_{0.1}Nb_{0.9}O_3$ particles via high temperature mixing method under hydrothermal conditions

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ABSTRACT

$KTa_{0.1}Nb_{0.9}O_3$ (KTN) particles with an orthorhombic perovskite structure have been synthesized via a high temperature mixing method (HTMM) under hydrothermal conditions. The obtained samples were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), transmission electron microscopy (TEM), selected area electron diffraction (SAED), and high-resolution transmission electron microcopy (HRTEM). The influence of alkaline concentration and solvent composition on the phase structure and morphology of the obtained powders was investigated. The results show that the well-crystallized KTN powders with sizes of 200–500 nm are successfully prepared at temperatures as low as 240 °C when the KOH concentration is 2.0 M and the isopropanol/water (I/W) volume ratio equals to 100/0.

1. Introduction

Potassium tantalate niobate (KTN) with an orthorhombic structure is a well-known ferroelectric material that has found wide applications in electromechanical, nonlinear optical, and other technological fields [1], and it has, therefore, attracted considerable interests among material scientists. In general, KTN powders are mainly prepared via a conventional solid state reaction (SSR) or sol–gel method at high temperature [2–4]. However, it is rather difficult for the SSR route to control the crystallinity and particle size of the final product, often resulting in localized segregation of components and stoichiometry because of the high temperature used in the method. On the other hand, hydrothermal synthesis, a common conventional wet chemical process, is promising in the direct synthesis of advanced nanoparticles. Such method reportedly yields highly crystalline products with great purity, narrow size distribution, and low aggregation [5–8]. Moreover, the morphology and crystal form of the final product can be controlled by adjusting the hydrothermal reaction conditions. $KNbO_3$, $NaNbO_3$, and $KTaO_3$ powders are among those successfully synthesized via the hydrothermal method [9–13]. Additionally, the method has been employed to prepare KTN nanorods and nanoplates over the past decades [14].

Orthorhombic KTN powders can only be synthesized via the hydrothermal method when the alkaline concentration is higher than 16.0 M [15]. Such high alkalinity condition usually causes serious corrosion in the reaction vessel, preventing the powders from large scale industrial manufacturing [16]. Meanwhile, it is known that when the temperature and pressure of a solvent are above its critical point, dissolving ability similar to that of a regular liquid is observed but with better transport properties, and, therefore, chemical reactions are accelerated when carried out in supercritical solvents. Since the critical temperature T_c of isopropanol is 235.03 °C, if its temperature is 240 °C, the isopropanol is in the state of supercritical fluid [17,18], and any chemical reactions carried out at this temperature in isopropanol should observe acceleration. The required alkaline concentration for glycothermal synthesis of $KNbO_3$ was reportedly reduced to 0.5 M using supercritical isopropanol as solvent [19].

As a modified hydrothermal process, a high temperature mixing method (HTMM) under hydrothermal conditions is used in this work. During HTMM, the starting solutions and raw materials are heated to a preset reaction temperature separately in a double-chambered autoclave before they are mixed to start the hydrothermal reactions. In our previous work, Cd-Hap [20], Pb–Sr Hap [21] and (K, Na) NbO_3 [22,23] powders were successfully synthesized via HTMM.

In this work, the KTN powders are successfully synthesized via HTMM within the solvent of isopropanol. The optimum reaction parameters, microstructure, and morphology of the resulting samples are investigated.

2. Experimental

Reagent grade KOH, Nb_2O_5, Ta_2O_5, and isopropanol were used as raw materials and purchased from Sinopharm Chemical Reagent Co., Ltd., China. Pre-determined amounts of KOH were dissolved

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