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Structural and physical properties of undoped and Ag-doped $NaTaO_3$ synthesized at low temperature

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ABSTRACT

Undoped and Ag-doped $NaTaO_3$ materials were synthesized via a sol–gel method using sodium hydroxide, tantalum (V) ethoxide, and silver nitrate as precursors. Both samples were thermally treated at 600 °C, for 6 h. Powder X-ray diffraction (XRD), ultraviolet (UV) diffuse spectra, scanning electron microscopy (SEM/EDX), BET (Brunauer, Teller) surface area, and electrical measurements are used to characterize various properties of the obtained materials. The average crystallite size was 50.2 nm for undoped $NaTaO_3$ and 37.2 nm for Ag-doped $NaTaO_3$. Electrical measurements have confirmed a typical semiconductor behavior of the obtained materials, with electrical band gap about 0.82 eV for undoped sample and 1.04 eV for Ag-doped sample.

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1. Introduction

Transition metal oxides with perovskite structure (such as niobium, tantalum-based semiconductor materials) have attracted considerable attention in recent decades due to their interesting properties and potential applications [1]. Within different materials, the metallic oxides with perovskite structure presents diverse physicochemical characteristics, such as ferroelectric properties ($BaTiO_3$, $LiTaO_3$ and $LiNbO_3$), ferromagnetic properties ($SrRuO_3$) or weak ferromagnetic properties ($LaFeO_3$), superconductors ($YBa_2–Cu_3O_7$), large thermal conductivity due to excitation transport ($LaCoO_3$), insulator-to-metallic transitions of interest for thermistor application ($LaCoO_3$), transport properties of interest for high-temperature thermoelectric power (La_2CuO_4) and in the last 10 years, $NaTaO_3$ have been widely characterized as photocatalyst for hydrogen production via water splitting reaction [2–6].

The conventional technique for preparing $NaTaO_3$ powder is mainly based on the solid state reaction, which requires long time at high temperatures (typically 1000–1300 °C) [7]. The solid-state synthesis method was widely used to prepare the vast majority of the tantalates acting as photocatalysts [8,9]. A few studies developed other synthetic routes for the tantalates. The solid-state method requires high-temperature and long-duration calcinations, leading to grain growth (thus small surface area) and loss of stoichiometry of the products [10]. The sol–gel method has been used in syntheses of nanosized materials with considerably larger surface area [11].

Recently, many efforts have been made to synthesize $NaTaO_3$. For example, He and Zhu have successfully synthesized the perovskite-type $NaTaO_3$ powder with a cubic morphology, via the solvothermal method (utilizing $Ta(OC_4H_9)_5$ and NaOH as the starting materials and the ethanol as the solvent) [12]. Nelson and Wagner have

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obtained $NaTaO_3$ nanorods from $TaCl_5$ by the alkalide reduction method [13].

The present paper reports a study regarding the influence of silver as dopant on some physico-chemical characteristics of sodium tantalite, for possible application as thermoelectric materials. The dopant influence and heat treatment on phase composition, particle size, morphology and electric properties of the obtained materials have been investigated by X-ray diffraction (XRD), ultraviolet (UV) diffuse spectra, scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), BET, and electrical measurements.

2. Experimental method

Analytically pure chemicals (Aldrich) were used for sol–gel synthesis of the materials. Undoped $NaTaO_3$ (denoted by Nat) were prepared as follows: 1 M solution of NaOH in ethylic alcohol and distilled water (1:1) was prepared. In this solution, 1 mL of tantalum (V) ethoxide was added, at room temperature, under continues stirring on a magnetic stirrer. The resulted mixture it was a white suspension which was, further, heated for 2 h at 90 °C. The obtained gel was dried in a furnace at 110 °C for 4 h.

For the synthesis of Ag-doped $NaTaO_3$ material (denoted by AgNat), same reaction conditions and precursors amounts were used, but in obtained $NaTaO_3$ suspension, 0.04 g of $AgNO_3$ was added, under continues stirring, resulting a gray suspension. The resulted suspension was processed in same conditions as undoped $NaTaO_3$ material.

For crystallization, both precipitates (Nat and AgNat) were thermally treated for 6 h at 600 °C, at a heating rate of 5 °C/min.

The purity and crystallinity of as-prepared samples were analyzed with a PANalytical X'PertPRO MPD Diffractometer with Cu tube. The light absorption properties of the materials were studied by UV–vis diffuse reflectance spectroscopy (DRUV-VIS) using a Lambda 950 Perkin Elmer device. The morphology of the powders was investigated by SEM using a FEI Inspect S scanning electron microscope and elemental analysis and mapping was performed by Energy Dispersive X-ray Analysis (EDX). For characterization by scanning electron microscopy the samples were prepared as follows: a small amount of undoped or doped sodium tantalite was dispersed in few milliliters of ethylic alcohol, and the solution was ultrasonically assisted in ultrasonic bath for 20 min. After sonication, the obtained suspensions were deposited on glass plates. For quantitative analysis of the materials, after alcohol evaporation, a thin substrate of carbon (~100 nm) was deposited on the samples.

Surface area (BET (Brunauer, Emmett, Teller)) and pore size distribution (BJH Barrett–Joyner–Halenda) measurements were achieved by using Quantachrome Nova 1200e device in nitrogen at 77 K temperature.

For measurements of the electrical resistance, R, of samples at different temperatures, T, over the range (30–150) °C, a laboratory made experimental setup has been used. Each sample was inserted into a glass tube with a diameter $D=3.8$ mm and length $L=11$ mm, in contact with two metal electrodes, connected to an ohmmeter. The glass tube containing the sample was inserted into an electric furnace and the temperature of the sample was measured with a thermocouple.

3. Results and discussions

3.1. XRD analysis

The samples were characterized using CuKα radiation with $\lambda=1.54060$ Å, operating at 45 kV and 30 mA. The measurements were taken in the range of 20° to 80° (2θ) with a step size of 0.0130° (2θ). Fig. 1 displays the XRD spectra of the undoped sodium tantalite (pattern a) and of Ag-doped $NaTaO_3$ (pattern b). The diffraction peaks were all indexed to a perovskite structure, and no impurities were observed. One can see that in both cases the materials are well crystallized corresponding to pure phase $NaTaO_3$; characteristic reflections can be indexed to (110),(200), (201), (220), (221), (311), (400), (312) and (331) planes, according to JCPDS No. 01-073-0881. The crystal structure of compound Nat belongs to tetragonal symmetry, space group P4/m b m (127), with $a=b=5.508(1)$, $c=3.882(2)$ Å, $V=117.767$ Å^3. As regards AgNat, its crystal is of tetragonal symmetry, space group P 4/m b m (127), with $a=b=5.4421(2)$, $c=3.8292(1)$ Å,$V=113.407$ Å^3.

Crystallite sizes can be estimated from the XRD patterns through Scherrer equation $d_{hkl}=0.9\ \lambda/(\beta\cos\ \theta_B)$, where d is the average crystallite size, λ is the wavelength of the incident X-ray, β is the full width at half-maximum, and θ_B is Bragg angle [14]. Thus, the crystallite sizes estimated by the peaks corresponding to (110), (200) and (220) are 50.2 nm for Nat and 37.2 nm AgNat, respectively.

The successful doping of Ag is also evidenced by a stretching and rightward shift of XRD peaks in AgNat, which may be due because the atom radius of Ag (1.60 Å) smaller than Na (1.80 Å) and larger than Ta (1.46 Å) (Fig. 1 (inset)).

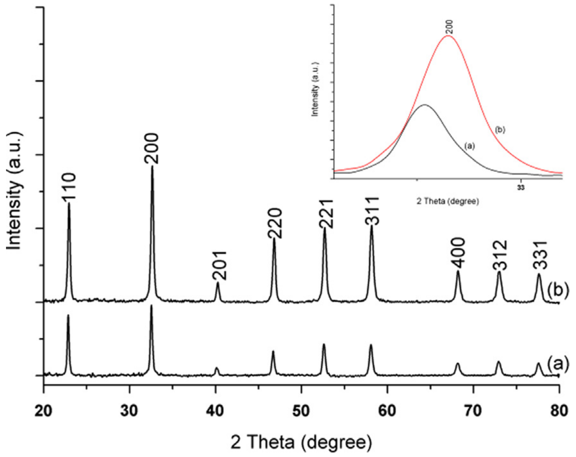

Fig. 1. XRD patterns for Nat (a) and AgNat (b) annealed at 600 °C; diffraction peak indexed to (200) crystalographic plane(inset).

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