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Fabrication of tungsten oxide nanostructure by Sol-Gel method

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Abstract

In this work, we present the effects of mixing sequence of sodium tungstate and nitric acid, as well as the aging duration on the formation of tungsten oxide by sol-gel method. Increase of the aging duration allows the formation of uniform platelet structure where no structure could be detected at 1 aging day while uniform platelet structure could be detected at 30 days. Hydrated tungsten oxide platelets of 1-1.θ ȝm could be formed by mixing sodium tungstate into nitric acid (sample labeled as W-A) which could be reduced significantly in size to 200-600 nm by changing the mixing sequence to drop nitric acid into sodium tungstate (sample labeled as A-W). Crystalline hydrated tungsten oxides were obtained for both samples where W-A sample shows better degree of crystallinity. Room temperature hydrogen gas sensing property was successfully detected by both samples where A-W sample demonstrates ~2.5 times higher sensor response to hydrogen gas compared to W-A sample.

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Keywords: tungsten oxide; sol-gel; gas sensor

Nomenclature

WO_3	tungsten oxide	h	hour

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W-A	sample prepared by dropping Na_2WO_4 into HNO_3	min	minute
A-W	sample prepared by dropping HNO_3 into Na_2WO_4	sccm	standard cubic centimeter per minute
		ppm	parts per million

1. Introduction

The sol-gel process is a facile fabrication technique where colloidal or gel can be prepared by mixing of liquid reactants which is first attempt in 1845 for the synthesis of hydrous silica[1]. To the best of our knowledge, sol-gel fabrication technique was only first demonstrated on the formation of tungsten oxide (WO_3) for its electrochromic property by Chemseddin on 1983[2]. Since then, intensive studies have been conducted for the fabrication of tungsten oxide by sol-gel method. There are three approaches to fabricate WO_3 by sol-gel method, which are acidification of sodium tungstate[3], dissolving tungsten powders in hydrogen peroxide[4] and reaction between tungsten oxy-chloride and isopropanol[5].

With only requirement of chemical reactants, sol-gel technique offers very simple and low synthesis cost to fabricate WO_3, which could also be synthesized in nanocrystalline structure by fine-tuning sol-gel parameters. Existing in a gel form or precipitation, sol-gel derived WO_3 can be coated on the large substrate by dip-coating[6], spray-coating[7] and screen-print[8] methods, where this is a huge advantage to manufacture a large and low cost electrochromic device which still remains as a challenge for commercialize purpose. Owing to this, most of the researches on the sol-gel synthesized tungsten oxide were done to improve the electrochromic properties of tungsten oxide. For the case of acidification of sodium tungstate, additive like H_2O_2 or oxalic acid[9] are usually added into the solution to stabilize the sol for better electrochromic properties. On the other side, relatively little study has been done to understand the fundamental details of the effects of sol-gel parameters (mixing and aging conditions such as flow rate of mixing, pH, time, and temperature) on the properties of tungsten oxide and other applications.

In this work, we demonstrate that the mixing sequences and aging durations of the reactants could significantly affect the properties of the as-grown WO_3 and also its gas sensor application.

2. Experimental procedure

2.1. Materials preparation

40 ml, 0.15 M Na_2WO_4 (BDH chemical Ltd Poole England) was added drop by drop from 25 ml burette into 15 ml 3 M nitric acid (HNO_3) under vigorous stirring. The sample was labeled as W-A. For the other experiment, all the experiment parameters were repeated and remained the same except that 3 M HNO_3 was added into 0.15 M Na_2WO_4. The sample was labeled as A-W. Samples were left aging at 25 °C for 30 days.

2.2. Materials and gas sensing characterization

Samples were characterized by field emission scanning electron microscope (FESEM, FEI Nova NanoSEM 450) and X-ray diffraction (HR-XRD, PANalytical X'Pert PRO εRD PWγ040) to study their morphological and structural properties. To examine the hydrogen sensing properties, the samples were screen-printed on 0.25x0.25 inches interdigitated gold electrodes from Synkera. Prior to the measurement, the devices were annealed at 400 °C for 2 h in ambient air. Hydrogen sensing measurements were conducted at room temperature in a conventional gas flow system with 2000 sccm flow rate of 1000 ppm concentration of hydrogen. Keithley 2400 source meter unit was employed for the current-voltage measurement.