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Urea mediated synthesis of $Ni(OH)_2$ nanowires and their conversion into NiO nanostructure for hydrogen gas-sensing application

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ABSTRACT

Synthesis of low cost, scalable, robust, and multifunctional nanomaterials for gas sensing application has been received increasing interest in recent years. Herein, we introduce the urea mediated synthesis of $Ni(OH)_2$ nanowires and their conversion into NiO nanostructure for hydrogen gas-sensing application. The $Ni(OH)_2$ nanowires were synthesized by a facile hydrothermal method, where the urea concentration was adjusted to get desired nanowire morphology. Conversion of $Ni(OH)_2$ nanowires into single phase NiO was done by heat treatment of the as-synthesized product at temperature of 500 °C for 2 h. The morphology, crystal structure, crystallinity, and phase transformation of the products were examined by SEM, HRTEM, XRD, and XPS. Gas sensing properties were measured to various H_2 concentrations ranging from 50 ppm to 1000 ppm at different temperatures. Results show that the synthesized NiO nanostructure can be used to detect H_2 gas at the low concentrations ranging from 50 ppm to 1000 ppm. The sensor also showed good selectivity to CO, NH_3 and CO_2 gases.

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Introduction

Hydrogen gas, a lightest molecule with a tasteless, odorless and colorless characteristics has been recently marked as the highest potential risk of fire and explosion because it is a self-ignited gas and has a wide explosive limit, ranging from approximately 4%–75% volume in air [1–3]. Nevertheless, hydrogen gas has been increasingly used as a green and renewable fuel in the modern societies [1,2]. In addition, hydrogen can also be generated in nuclear power plants, or being used as fuel in space stations, thus leakage of this gas becomes more seriously because when burning, its flame cannot see by human eye. Hydrogen leakage can spread quite quickly while people cannot recognize, thus increasing the possibility of terrible explosive disasters [3]. For instance, Hindenburg disaster (1937), and Fukushima nuclear explosion (2011) were deemed as a result of leaking hydrogen gas.

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Therefore, development of low cost, high sensitivity and low detection limit gas sensor [4] to early warning, detecting and/or controlling H_2 is one of the most important issues in safety use, storage and transportation of hydrogen gas [5–8]. Thus, in the past few years [9,10] great deal of research efforts had directed toward the development of gas sensors for detection of different gases involving H_2 [11–14]. Recently, the p-type semiconducting metal oxides [15] have been received extensive attentions [16] from researchers worldwide because of their advantages over n-type counterparts such as the better catalytic activities [17,18], and possibility to sense some oxidizing [19] and reducing gases [20–22] with enhanced responsibility by doping or catalytic sensitization [23]. The sensing mechanism of the p-type metal oxide semiconductors based on the formation and/or expansion/reduction of hole-accumulation layers upon exposure to analytic gases, and thus variation in sensor conductivity [15]. Of interested p-type semiconducting metal oxides, NiO, one of the most important transition metal oxides, reported as a p-type semiconductor due to the excess of oxygen (or vacancy of metal) in the crystal has received massive attention [24]. This p-type semiconductor (NiO) has a wide direct band gap of about 3.7 eV, and is one of the most interested materials used in electrochemistry [25], magnetism [26], photocatalyst [27], supercapacitor [28], heavy metal adsorbent [29], and gas sensors [22]. Thus, recent researches have been focusing on the fabrication of NiO of different morphologies [23,30], including of nanoparticles [31], nanosheets [19], nanofibers [27], nanowires [22], microspheres [32], urchin flower [33], and etc [34]. For the gas-sensing application, small crystalline size and large specific surface area materials are desired for better gas sensor performance because according to the gas sensing mechanism, the gas adsorption/desorption processes mainly take place on the surface of sensing layer [18]. The nanowire structure of NiO was reported to be better for the detection of reducing gas (ethanol) with a higher response under the operating temperature of 350 °C [34]. Indeed, NiO nanowires of hexagonal structure were synthesized by sol-gel method [35]. Polycrystalline NiO nanowires of an average diameter of about 100 nm were fabricated by magnetic assisted chemical reduction synthesis of Ni nanowires and then followed by thermal oxidation in air to oxidize the metallic Ni into NiO [22]. Other ways to fabricate large-scale production of nanostructured NiO are solution synthetic routes like hydrothermal [34], or electrospinning methods [27]. Hydrothermal method has the advantages of facile synthesis, high uniformity in morphology and large yield products [34]. However, almost recent reports about hydrothermal synthesis of NiO nanowires required complex agents including of nickel source, reducing agent, and/or surfactants, leading to difficulty in fabrication as well as expensive products [36,37]. In addition, hydrothermal synthesis of NiO generally requires two steps, one is the hydrothermal synthesis of nickel metal stable phases, and then their decomposition in to NiO phase [38]. This decomposition process can influence on the morphology, and characteristics of the obtained NiO nanostructure [32]. Therefore, a systematically investigation on the conversion of $Ni(OH)_2$ nanowires into NiO nanostructure is of important to study. Furthermore, the synthesis of NiO nanocrystals with controlled particle size and/or shape for hydrogen sensing application still remains a challenge.

Herein, we describe a facile hydrothermal method to synthesize $Ni(OH)_2$ nanowires by using nickel chloride and urea as precursor materials, and their conversion in to NiO nanostructure for hydrogen gas sensor application. The nanowire structure of the $Ni(OH)_2$ was obtained without using any surfactants and/or structure-directing agents. Thermal conversion of $Ni(OH)_2$ nanowires into NiO nanostructure was systematically studied by some advanced techniques such as SEM, HRTEM, XRD, TGA-DTA, and XPS. In addition the gas sensing properties of the synthesized NiO were studied for the detection of H_2 at different temperatures. Results demonstrate that the synthesized NiO nanostructure is potential for sensitive and selective detection of low concentration H_2 gas.

Experimental

Materials synthesis

The hydrothermal was carried out to synthesize $Ni(OH)_2$ nanowires by using nickel chloride hexahydrate and urea as starting precursors without using any further surfactants or template-directing agents. In a typical synthesis, 2.38 g (0.01 mol) nickel chloride hexahydrate, and 0.5 g urea were dissolved in 50 ml distilled water at temperature of approximately to 30 °C to obtain a clear solution. Thereafter, this solution was transferred into a Teflon-lined autoclave of 100 mL in volume for hydrothermal process. The autoclave was sealed, inserted into an oven and maintained at 180 °C for 16 h to perform the hydrothermal reaction. After naturally cooling down to approximately to 30 °C, the light green precipitate was collected by centrifuging at 4000 RPM and rinsed five times with distilled water and subsequently two times with ethanol before being dried at 45 °C. This process is very important to ensure the total removal of the unreacted agents and the fast drying of the products. By varying the amount of urea, we could synthesize different morphologies of the $Ni(OH)_2$ materials such as nanowires or nanoparticles (see Figs. S1 and S2, ESI). Conversion of $Ni(OH)_2$ into NiO was performed by heat treatment of the hydrothermal product at temperature of 500 °C for 2 h in air (see Figs. S3, ESI). Herein, we selected the annealing temperature of 500 °C to ensure the total decomposition of $Ni(OH)_2$ into NiO [39]. This value is also higher the working temperature of semiconductor based hydrogen gas sensor to avoid the distortion of stability [14].

Materials characterization

Phase transformation of $Ni(OH)_2$ over heat treatment in air was studied by thermal gravimetric analysis (TGA), and differential thermal analysis (DTA). The structural morphology of synthesized materials was investigated by field emission scanning electron microscopy (SEM, JEOL model 7600) and high resolution transmission electron microscopy (HRTEM) model JEM-2100F. Crystal structure of samples was studied by X-ray diffraction (XRD). The XRD measurements were carried out for powder samples using $Cu_{K\alpha}$-X-radiation with

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