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Large scale synthesis of ultrathin cupric oxide nanosheets via a rapid microwave-assisted and template-free route

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ABSTRACT

A facile one-step microwave-assisted synthetic route was developed to prepare ultrathin and defect free CuO nanosheets without any surfactant. During the synthetic process, the 100 mL of water and ethanol mixed solution (1/1, v/v) containing 0.86 g of $CuCl_2{\cdot}2H_2O$ and 2 g of sodium hydroxide was heated by microwave irradiation at a power of 750 W for 20 min. Most of these CuO sheets possessed a length of 80–110 nm, 40–60 nm in width, and only 11 nm in thickness. The microstructure and morphologies of the CuO product were investigated in details by XRD, SEM, HRTEM, FT-IR, and XPS techniques, and it was found that the shapes of CuO were dependent on the concentrations of sodium hydroxide. The band gap of such CuO nanosheets was estimated to be 2.17 eV according to the UV–vis absorption curve. This effective microwave-assisted approach may be a promising route to prepare other transition metal oxides.

1. Introduction

Cupric oxide (CuO) exhibits a narrow band gap of 1.2 eV and has been recognized as an industrially important material for a variety of practical applications such as gas sensors, catalysts, lithium ion batteries, and water treatment. Nanoscaled CuO may possess unique properties including unusual optical, electrical, and catalytic performance that can be significantly different from those of its bulk counterpart [1–5]. For example, gas sensors constructed by CuO nanoflowers, nanorods, hierarchical CuO microspheres, and porous CuO nanosheets displayed an excellent gas response to various target gases (ethanol, acetone, methanol, toluene, xylene, H_2S, etc) in the working temperature ranging from 200 to 400 °C with the advantages of good sensitivity, short response time, and low detection limit [6–9]. Yang *et al* reported that CuO nanodendrites were active toward the thermal decomposition of ammonium perchlorate, and promoted the photo-degradation of rhodamine B and methyl orange [10]. Due to these applications, shape-controlled synthesis and well-defined morphologies for the formation of nanostructured CuO are technologically interesting because of the physical and chemical properties of this material depend on its morphologies, structure, size, and size distribution.

To date, a number of methods such as hydrothermal/solvothermal, thermal oxidation, sol-gel, and sonochemical approaches have been employed to prepare CuO nanostructures with different shapes [5]. Typically, Flower-like CuO nanostructures and hollow CuO microspheres could be prepared with the assistance of hexamethylenetetramine (HMTA) [11,12]. Wang et al. reported that CuO nanoribbons with widths of 10–80 nm, thicknesses of 5–20 nm, and lengths ranging from several hundred nanometers to several micrometers and CuO nanorings with diameters of 100–300 nm were synthesized in the presence of sodium dodecyl benzenesulfonate (SDBS) [13]. CuO nanosheets with thickness of about 50 nm were hydrothermally prepared at 140 °C for 24 h using PEG20000 as a surfactant [14]. However, most of these methods suffered from shortcomings of time-consuming fabrication, tedious manipulation, and poor size distribution. Therefore it is desirable to develop a facile and rapid method to synthesize nanoscaled CuO without using any hazardous organic templates. In recent years, the microwave-assisted synthesis has emerged as a promising method for producing many nanomaterials because of its unique effects such as fast volumetric heating, increased reaction rate, shortened reaction time, as well as the enhanced reaction selectivity and energy savings.

In this work, we demonstrated a one-step microwave method to prepare CuO nanosheets with thin thickness. Compared with the previous reports about synthesis of CuO nanostructures, our method is rapid, inexpensive, template-free, and nontoxic. As synthesized CuO nanosheets were characterized for their morphological, structural, and optical characteristics using different analytical techniques.

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2. Experimental procedure

2.1. Materials and method

All chemicals were of analytical grade and used as received without further purification. In a typical synthesis, 0.86 g of $CuCl_2 \cdot 2H_2O$ was dissolved into 50 mL of ethanol in a flask, and 50 mL of sodium hydroxide aqueous solution (1 M) was dropwise added with magnetic stirring for 20 min. The mixture was then put into a microwave oven connected with a condenser, and the solution was heated by microwave irradiation at a power of 750 W for 20 min. After reaction, the precipitate was collected by centrifugation, washed with DI water and absolution ethanol several times, and finally dried at 60 °C in air.

2.2. Characterizations

The phase purity of CuO was investigated by X-ray diffraction (XRD, Bruker D8 diffractometer) with Cu Kα radiation. FESEM images were recorded on a JEOL JEM-6700F microscope. TEM image, SAED pattern, and HRTEM image were obtained on a JEOL JEM2100F microscope with accelerating voltage of 200 kV. The composition and optical properties of the CuO nanosheets were analyzed by using FTIR spectroscopy (Nicolet iS 10 FT-IR spectrometer) and UV–vis-NIR spectroscopy (SHIMADZU UV-3600), respectively. XPS was conducted on an ESCA 2000 spectrometer using an Al Kα X-ray as excitation source.

3. Results and discussion

A typical XRD pattern of the obtained sample was shown in Fig. 1a. All the diffraction peaks could be indexed to the monoclinic phase CuO (JCPDS No. 48-1548), and the high intensity of these peaks clearly confirmed that the product was well crystalline. No signals of impurities were detected, indicating that pure CuO nanostructures were prepared via current synthetic route. Fig. 1b depicted the EDS spectrum, which confirmed the stoichiometry

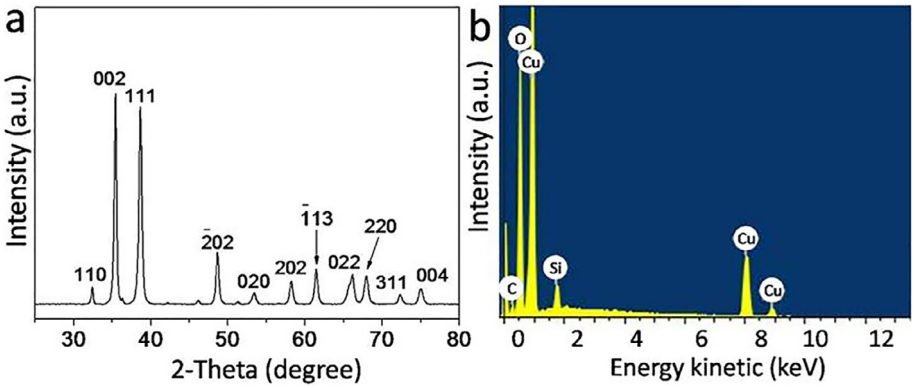

Fig. 1. (a) XRD pattern and (b) EDS spectrum of CuO sample obtained in the typical synthesis.

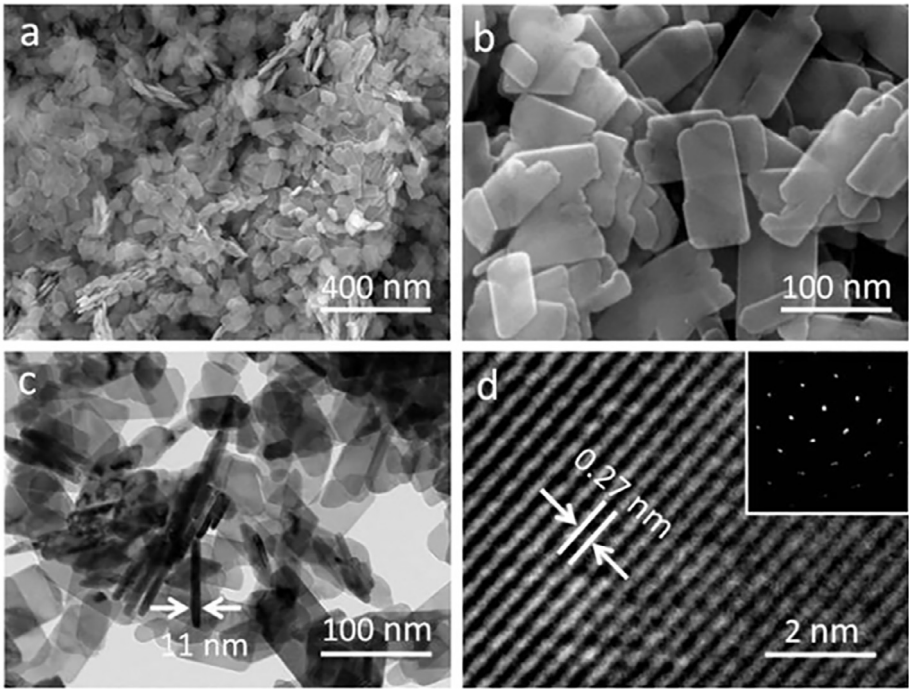

Fig. 2. (a, b) SEM images, (c) TEM image, and (d) HRTEM image of CuO nanosheets.