

# Acetone sensing properties of CuO nanowalls synthesized via oxidation of Cu foil in aqueous $\text{NH}_4\text{OH}$

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## ABSTRACT

High-density and uniform CuO nanostructures are synthesized via the facile oxidation of Cu foil in aqueous  $\text{NH}_4\text{OH}$  below  $80^\circ\text{C}$ . The  $\text{CH}_3\text{COCH}_3$  sensing properties of the synthesized materials are investigated at the operating temperature of  $200\text{--}360^\circ\text{C}$ . A good sample is further tested with  $\text{C}_2\text{H}_5\text{OH}$  and  $\text{NH}_3$  for demonstrating the gas selectivity at the optimal operating temperature. The selectivity between acetone and ethanol of the CuO nanowalls synthesized using this method is considered and compared with the results obtained by other studies.

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## 1. Introduction

Copper is one of the most abundant metals on earth. Its oxide (CuO) is an intrinsically p-type metal-oxide semiconductor with a bandgap ( $E_g$ ) of  $1.2\text{--}2.0\text{ eV}$  [1]. With the development of nanoscience and nanotechnology, copper oxide nanomaterials are considered as promising materials for a wide range of applications e.g., lithium ion batteries, dye-sensitized solar cells, photocatalytic hydrogen production, photodetectors, and biogas sensors [2–7]. Various micro- and nano-structures of CuO such as microflowers, nanoparticles, nanorods, nanowires, and nanoplates/nanowalls/nanosheets have been synthesized using various physical and chemical approaches [8–14]. The first such approach was proposed by D. W. Shoesmith et al. [15] wherein Cu metal was oxidized, forming CuO nanorods in an alkaline solution of  $\text{Li}(\text{OH})_2$ . Recently, high-density and uniform CuO nanostructures have been grown on Cu foils in alkaline solutions [3,16–25]. In 2011, T. Soejima et al. proposed a facile process for the oxidation synthesis of CuO

nanobelt arrays using  $\text{NH}_3\text{--H}_2\text{O}_2$  aqueous solution [26]. In this method, humid  $\text{NH}_3$  vapor was believed to be the main reactant toward Cu foil, thus forming  $\text{Cu}(\text{OH})_2$  and subsequently CuO nanostructures. Nevertheless, this synthesis process and a later study by T. Soejima et al. ignored the sinking of the Cu foil area in the aqueous  $\text{NH}_3$  solution [27]. In 2017, G. Kaur et al. synthesized CuO nanostructures by treating Cu foils in  $\text{NH}_4\text{OH}$  at room temperature for different treatment times [28]. The surface treatment of Cu in alkaline aqueous solutions is a potential method for the mass fabrication of CuO nanostructures with high uniformity and density. It is interesting to compare the gas sensing properties among CuO nanomaterials synthesized by this approach and by others. Nevertheless, none of above studies investigated the gas sensing properties of as-synthesized CuO nanomaterials.

In this study, CuO nanowalls versus nanoparticles were synthesized via the oxidation process of Cu foil in  $\text{NH}_4\text{OH}$  solution at  $50\text{--}70^\circ\text{C}$ . The gas sensing properties of the as-prepared CuO nanoplates were examined with  $\text{C}_2\text{H}_5\text{OH}$ ,  $\text{CH}_3\text{COCH}_3$ , and  $\text{NH}_3$  at  $200\text{--}360^\circ\text{C}$ . The sensing data were compared with the gas sensing properties of CuO nanostructures synthesized using other methods.

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## 2. Experimental process

The steps of the synthesis process are illustrated in Fig. 1. In this process, commercially pure copper foil (thickness of 0.1 mm, purity of 99.99%, Aldrich) was cut into  $2 \times 2 \text{ cm}^2$  plates (Fig. 1a). The Cu plates were folded at the corners to form table-like plates (Fig. 1b). The folded plates were immersed in acetic acid for 10 min to remove the oxide layer on the foil surfaces. Subsequently, the plates were ultrasonically cleaned in a bath sonicator with acetone, ethanol, and distilled water for 5 min each. After drying the folded plates under a flow of  $\text{N}_2$ , the plates were placed in 20 mL of  $\text{NH}_4\text{OH}$  solution contained in Duran laboratory bottles (capacity of 100 mL). The bottles were placed in a larger bottle (total volume 400 mL) filled with 150 mL of distilled water. After carefully covering the cap with aluminum foil, the 400-mL-capacity bottle was heated on a hot plate; the reaction temperature was controlled using a proportional–integral–derivative (PID) temperature controller (K-type thermocouple was inserted into the reaction vessel and dipped into water). The treatment temperature was varied in the range 50–80 °C (samples 1, 2, 3, and 4 were treated at 50 °C, 60 °C, 70 °C, and 80 °C, respectively) at a heating rate of 1 °C/min for 48 h. After the heat treatment, the samples were dried in an oven at 60 °C for 12 h.

The as-prepared samples were characterized using a field-emission scanning electron microscope (FE-SEM; JEOL JSM-7610F) operated at an acceleration voltage of 2 kV. The structures of the samples were identified using X-ray diffraction (XRD; XPERT-PRO) with Cu  $K\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ). The gas sensing properties of the as-synthesized materials were characterized using a static gas testing system. For this measurement, the black material covering the treated Cu foil was extracted and dispersed in ethanol solution by scratching and sonicating in a bath sonicator. The received solution was coated on interdigitated electrodes using a spin coater at the coating speed of 3000 rpm for 5 min. The designs of the electrode and gas testing system are shown in Figs. S1 and S2 (supplemental documents), respectively. In this measurement, the gas response is defined as  $S = R_{\text{gas}}/R_{\text{air}}$ , where  $R_{\text{gas}}$  and  $R_{\text{air}}$  represent the sensor resistances in the target gas and air, respectively.

## 3. Results and discussion

The XRD patterns of the Cu foils treated in  $\text{NH}_4\text{OH}$  at 50–80 °C are shown in Fig. 2. In sample 1 (Fig. 2a), the three strong peaks at  $2\theta$  values of 35.5°, 38.7°, and 53.4° can be attributed to the diffraction from the (11 $\bar{1}$ ), (111), and (020) planes of CuO,

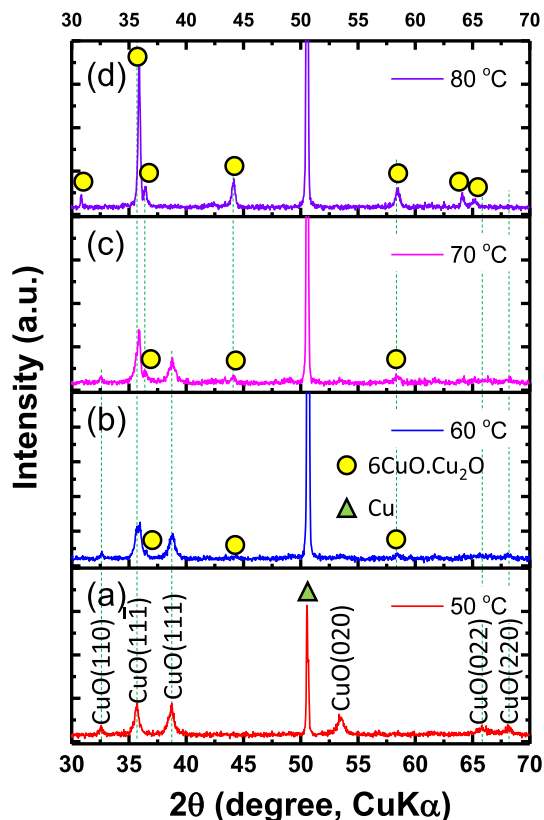


Fig. 2. XRD patterns of Cu foil treated in aqueous  $\text{NH}_4\text{OH}$  at (a) 50 °C, (b) 60 °C, (c) 70 °C and (d) 80 °C.

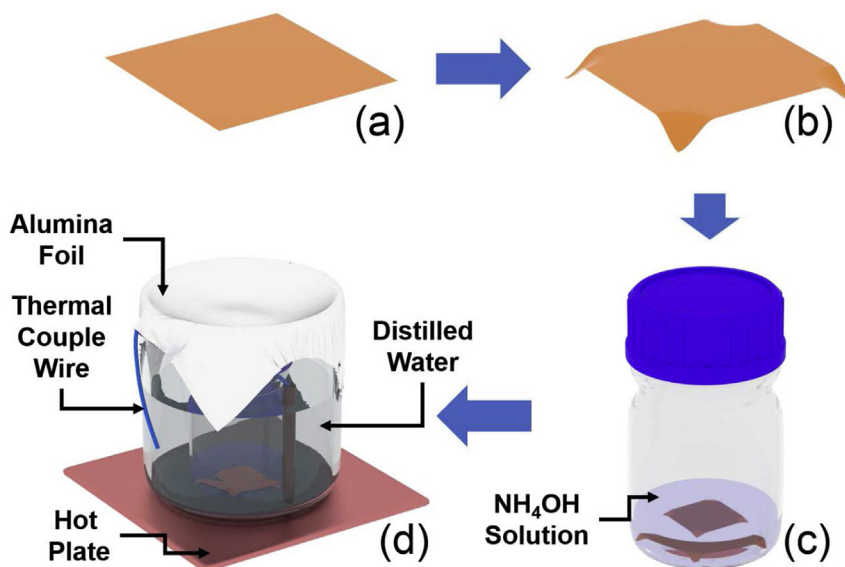


Fig. 1. Experimental setup for the oxidation of Cu in aqueous  $\text{NH}_4\text{OH}$ : (a) Copper foil, (b) Foiled Cu foil, (c) Foiled Cu foil and 25 mL  $\text{NH}_4\text{OH}$  in Duran bottle, (d) Duran bottle heated with a hot plate.

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