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WO_3 nanorods prepared by low-temperature seeded growth hydrothermal reaction

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ABSTRACT

This work describes the first tungsten oxide (WO_3) nanorods hydrothermally grown on W foil. WO_3 nanorods were successfully grown at low hydrothermal temperature of 80 °C by seeded growth hydrothermal reaction. The seed layer was prepared by thermally oxidized the W foil at 400 °C for 0.5 h. This work discusses the effect of hydrothermal reaction and annealing period on the morphological, structural, and electrochromic properties of WO_3 nanorods. Various hydrothermal reaction periods (8–24 h) were studied. Monoclinic WO_3 nanorods with 5–10 nm diameter were obtained after hydrothermal reaction for 24 h. These 24 h WO_3 nanorods were also annealed at 400 °C with varying dwelling periods (0.5–4 h). Electrochromic properties of WO_3 nanorods in an acidic electrolyte were analyzed using cyclic voltammetry and UV–vis spectrophotometry. WO_3 nanorods annealed at 400 °C for 1 h showed the highest charge capacity and the largest optical contrast among the 24 h WO_3 films. The sample also showed good cycling stability without significant degradation. Based on the results, the reaction mechanism of WO_3 nanorod formation on W foil was proposed.

1. Introduction

Tungsten oxide (WO_3) has elicited much attention due to its potential applications in electrochromics, photochromics, photocatalysis, sensors, and lithium batteries [1,2]. Its electrochromic property is the most interesting because WO_3 film has the ability to undergo optical coloration when voltage is applied. WO_3 can change from colorless to blue when electrons and ions such as H^+, Li^+, or Na^+ are added or intercalated in its structure. WO_3 film reverts to colorless when electrons and ions are extracted or deintercalated from the structure. The electrochromism of WO_3 has generated interest in energy-saving smart windows, displays, and antiglare mirrors [3]. Compared with polymer-based electrochromic devices, WO_3-based devices show better thermal and mechanical stabilities, as well as longer lifetime [4]. In addition, WO_3 has high cycling stability and high coloration efficiency compared with other transition metal oxides. WO_3 also exhibits high contrast ratio, good memory effect, and low power consumption [5].

WO_3 nanostructures are prepared typically by various methods, such as hydrothermal [6], sol–gel [7], electrodeposition [8], electrospinning [9], sputtering [10], and chemical vapor deposition [11]. Among these methods, hydrothermal is the most promising method for synthesizing WO_3 nanostructures owing to its simple set-up, low cost, flexible substrate selection, possibility in controlling the morphologies, and large-scale production, which make it beneficial for industrial production. There are two types of the hydrothermal process to produce WO_3. First type involves the centrifugation of hydrothermal solution to obtain WO_3 powders and followed by dispersion of the WO_3 powders on a substrate to form WO_3 structures [12]. Second type involves the immersion of substrate in the hydrothermal solution and directly grows WO_3 structures on the substrate during the hydrothermal reaction [13]. The substrates in this second type of hydrothermal process could be seeded with WO_3 film before the hydrothermal reaction. Seeded growth hydrothermal reaction has many advantages as the seeded substrate can lower the thermodynamic barrier by providing nucleation sites in the hydrothermal system. The seeded substrate also provides preferential growth of WO_3 structures during the hydrothermal process.

WO_3 and hydrated WO_3 structures have been grown directly on various substrates (seeded and none seeded substrates), such as conductive glass (ITO and FTO) [13–29], microscope glass [30], soda–lime glass [31], alumina plate [32], and W foil [32–41]. Various morphologies of WO_3 and hydrated WO_3 structures have been grown, including nanorods [13,16,17,26–28,31], nanowires [19,29], nanotrees [32–35], and nanoflakes/nanosheets/nanobricks/platelets [18,20–25,36–41]. All WO_3 nanorods have been

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grown on glass, but no report on WO_3 nanorods grown on W foil has been published. A range of WO_3 nanorod sizes were obtained on the glass. Ding et al. have grown WO_3 nanorods with 200 nm diameter on bare soda–lime glass at 120 °C [31]. Zheng et al. have grown WO_3 nanorods with 55 ± 22–70 ± 28 nm and 50 ± 30 nm diameter on bare ITO glass at 170 and 180 °C, respectively [16,17]. Ma et al. have grown WO_3 nanorods with 20–120 nm diameter on WO_3 seeded FTO glass at 180 °C [26]. Most works have

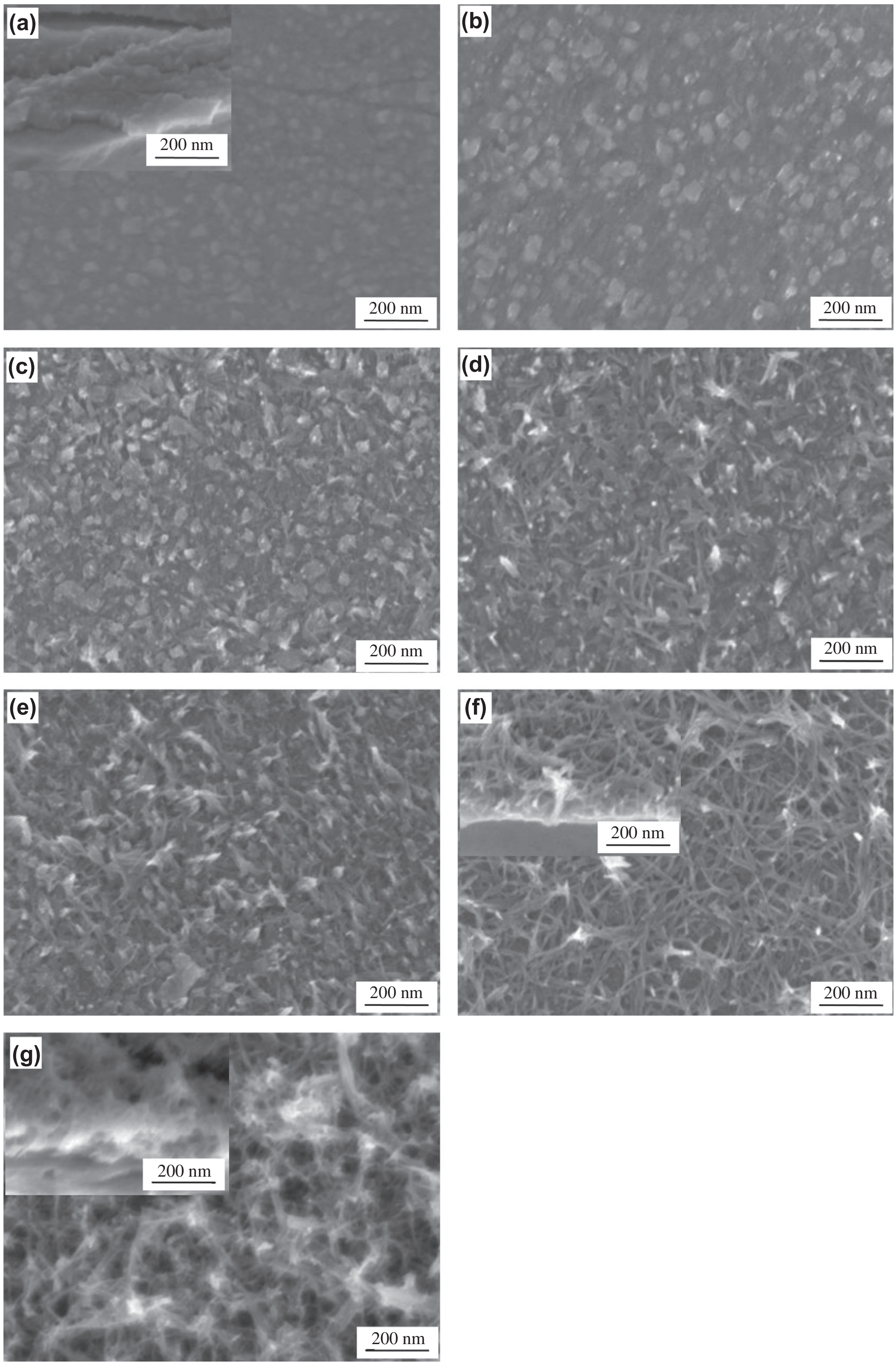

Fig. 1. FESEM images of (a) WO_3 seed layer and WO_3 films synthesized at various hydrothermal reaction periods: (b) 8 h, (c) 12 h, (d) 16 h, (e) 20 h, (f) 24 h, and (g) 24 h without CTAB. Insets are cross-sectional views of the samples.