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Synthesis of Tungsten Oxide Nanowires onto ITO Glass Using T-CVD

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Abstract

Tungsten oxide is an n-type semiconductor with interesting physical and chemical properties that make it suitable for various technological applications. Tungsten oxide nanowires were synthesized not only at low temperature but also without the use of any catalysts. The tungsten oxide nanowires were synthesized at 550 °C with tungsten layers onto the ITO glass using thermal chemical vapor deposition (T-CVD). The SEM image shows that the tungsten oxide nanowires are effectively grown with the 200 nm tungsten film. The Raman spectra shoulder at ~690 cm^{-1} proves the synthesized of tungsten oxide nanowires.

Abstrak

Sintesis Kawat Nano Tungsten Oksida pada Kaca ITO Menggunakan T-CVD. Tungsten oksida adalah semikonduktor tipe-N yang cocok digunakan dalam berbagai penerapan teknologi karena sifat fisik dan kimiawinya yang menarik. Kawat nano tungsten oksida bukan hanya disintesiskan pada suhu rendah, melainkan juga disintesiskan tanpa menggunakan katalisator. Sintesis antara kawat nano dan lapisan-lapisan tungsten dilakukan pada kaca ITO dalam suhu 550 °C dengan menggunakan pengendapan uap kimia termal (T-CVD). Citraan SEM menunjukkan bahwa pengembangan kawat nano tungsten oksida yang efektif adalah dengan menggunakan film tungsten berketebalan 200 nm. Sintesis kawat nano tungsten oksida dibuktikan dengan pengukuran spektroskopi raman yang mencapai ±690 cm^{-1}.

Keywords: ITO glass, thermal chemical vapor deposition, Tungsten oxide nanowires

1. Introduction

Tungsten oxide is an n-type semiconductor with interesting physical and chemical properties that make it suitable for various technological applications such as catalysts [1], gas sensors [2], or electrochromic devices applications including large area displays [3], rearview mirrors [4], and smart windows [5-7]. Moreover, One-dimensional (1D) nanostructures with high surface-to-volume ratio and small grain size have attracted extensive research interests. Nanostructure could enhance the performances of tungsten oxide thin film [8-10]. Recently, the growth method of tungsten oxide nanowires has been reported by many research groups [11-17].

In this paper, we opted for thermal oxidization method to prepare tungsten oxide nanowires since it was technically simple. The tungsten oxide nanowires grown onto ITO glass not only at low temperature but also without the use of any catalysts. The tungsten oxide nanowires were investigated by X-ray diffraction (XRD), Raman spectroscopy and scanning electron microscopy (SEM).

2. Experiment

Preparation of tungsten oxide nanowires. Initially, tungsten films were deposited onto indium tin oxide (ITO) glass substrates by reactive RF magnetron sputtering system. The sputtering system used pure tungsten target (purity 99.99%) and pure argon (purity 99.99%) as the sputtering gas with the base chamber pressure of ~3×10^{-5} Torr. The substrates were then carried out under argon flow rate of 25 sccm (standard cubic centimeter per minute) and RF power of 150 W at various times from 2.5 min to 10 min.

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The as-prepared tungsten films were subsequently oxidized at 550 ^{o}C by thermal chemical vapor deposition (T-CVD). The tungsten films as a self-catalytic would become tungsten oxide nanowires by T-CVD process. The substrates were first loaded into a quartz boat and placed in the middle of the quartz tube. The quartz tube was evacuated to ~30 mTorr with the continuous flow feed gas composition. The feed gas composition was varied using Ar/O_2 flow rate ratio of 10:1. In the quartz tube, the substrates were ramped up to 550 ^{o}C in pressure of 50 Torr for 2 hours and followed by cooling down to room temperature in 4 hours.

Characterization. The surface morphology of the tungsten oxide nanowires was visualized by JEOL JSM-6500F field emission scanning electron microscopy (FESEM) operated at 15 kV. The crystallinity of the samples was characterized by X-ray diffraction (XRD). The XRD pattern was recorded on a 2θ configuration using Bruker D8 Discovery X-ray diffractrometer with non-monochromated Cu Kα X-ray radiation (λ= 1.54056 Å). The 2θ investigation region was in the range 20^{o}-70^{o} with scanning speed of 2 degree/s. Raman spectrum were obtained by a Raman spectroscopy with the wavelength of 532 nm, laser power of 5 mW and beam size of 5 µm.

3. Results and Discussion

The SEM images of tungsten thin films thickness prepared on the ITO glass substrates are shown in Figure 1. The tungsten thin films were deposited at three different thicknesses: 70 nm, 100 nm, and 200 nm, respectively. In this study, the function of tungsten thin film is a self-catalytic layer to grow tungsten oxide nanowires. It is also noted that the thickness of tungsten thin film has important role for growing nanowires.

Furthermore, Figure 2 shows the SEM image of tungsten oxide nanowires (TONWs) and the tungsten oxide films (TOF), with various tungsten thin film thickness (70 nm, 100 nm, and 200 nm) on ITO glasses at 550 ^{o}C. The TOF and nano particles were grown on the tungsten thin film thickness of 70 nm are shown in the Figure 2(a). Figure 2(b)-(c) show TONWs during the tungsten thin film thickness increase. It display that the TONWs on the tungsten film thickness of 200 nm are longer and bigger than thickness of 100 nm. The TONWs grown on ITO glasses were like nanowires bundles which were also reported by Ha *et al.* [18].

The Raman spectra in the range of 250-1100 cm^{-1} of the samples investigated in this paper are showed in Figure 3. It was found that there were two ranges presented in Raman spectra. The two ranges located in the range of 250-400 cm^{-1} region and 600-900 cm^{-1} region associated to phonon activity since the effects of the growth temperature on the oxidation states tungsten oxide nanowires [19]. As shown in Figure 3, the Raman spectra increased the intensity at ~297, 694, 789, and 807 cm^{-1}. The Raman peaks at 297 cm^{-1} were assigned to O-W-O bending modes of binding oxygen, and the

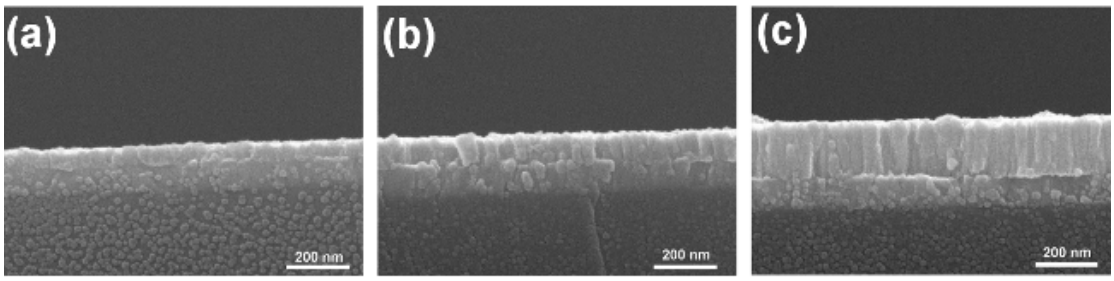

Figure 1. SEM Images of the Tungsten Thin Film Thickness: (a) 70 nm; (b) 100 nm; (c) 200 nm

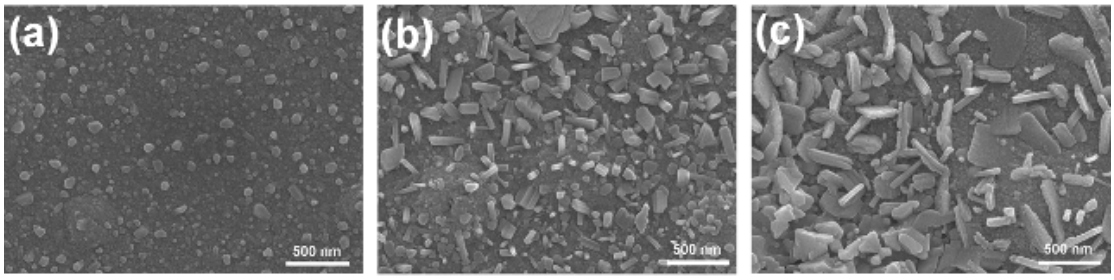

Figure 2. SEM Image of the Tungsten Oxide Nanowires Grown on Different Tungsten Thin Film Thickness: (a) 70 nm; (b) 100 nm; (c) 200 nm.

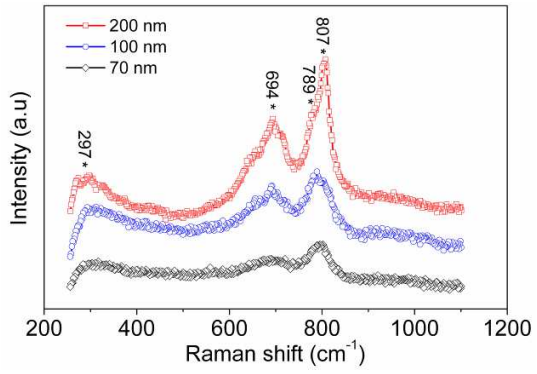

Figure 3. Raman Spectra of Tungsten Oxide Nanowires

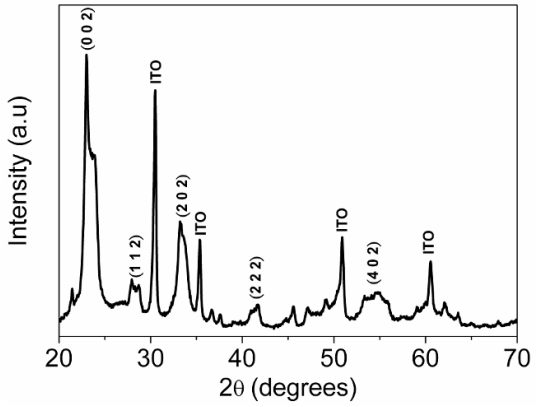

Figure 4. XRD Pattern of Tungsten Oxide Nanowires

peaks at 694, 789, and 807 cm^{-1} were assigned to the corresponding stretching modes [18-19]. Furthermore, the Raman spectra shoulder at ~690 cm^{-1} proved the synthesized of the tungsten oxide nanowires [20].

The lowest intensity of Raman spectra occurred when the thickness tungsten film was 70 nm. It was also confirmed by Figure 2(a) that there were a little nano particle at thickness of 70 nm. When the thickness tungsten film was 200 nm the Raman spectra had highest intensity and it was also confirmed by Figure 2(c) that there were nanowires at 200 nm thickness. The Raman spectra verified that there was a blue shift from 789 to 807 cm^{-1} with the 70 nm to 200 nm tungsten oxide thin film thickness. The blue shifts on Raman spectra indicated that the particle size of tungsten oxide became nano due to quantum size effect [21].

The XRD pattern of tungsten oxide nanowires grown onto ITO glass substrate is shown in Figure 4. The observed XRD pattern which was compared with standard JCPDS card showed that there were three diffraction peaks at $2\theta^o$ values 23^o, 28^o and 33.2^o. The diffraction peaks could be well indexed to an Orthorhombic WO_3 Phase (cell constants: a= 7.341, b= 7.57, c= 7.754; JCPDS 71-0131).

4. Conclusions

The tungsten oxide nanowires (WO_{3-x}) grown on the ITO glass substrates were synthesized not only at low temperature but also without the use of any catalysts. In this study, we have successfully synthesis tungsten oxide nanowires. The tungsten oxide nanowires were synthesized at 550 ^oC with tungsten layers onto the ITO glasses using thermal chemical vapor deposition (T-CVD). The tungsten film as a self-catalytic layer enhances the tungsten oxide nanowires growth. The SEM image shows that the tungsten oxide nanowires are effectively grown with the 200 nm tungsten film. The Raman spectra shoulder at ~690 cm^{-1} proved the synthesized of tungsten oxide nanowires. The XRD pattern of tungsten oxide nanowires could be well indexed to an Orthorhombic WO_3 Phase (cell constants: a= 7.341, b= 7.57, c= 7.754; JCPDS 71-0131).

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