SNRU Journal of Science and Technology

Journal home page: snrujst.snru.ac.th

Fabrication of Au Nanoparticles Decorated WO_3 Nanorods by Magnetron Sputtering

Chaiyan Oros[1,*], Mati Horprathum[2]

[1]Faculty of Liberal-arts, Rajamangala University of Technology Rattanakosin, Nakornpathom, 73170 Thailand

[2]Optical Thin-Film Laboratory, National Electronics and Computer Technology Center, Pathumthani, 12120 Thailand

[*]**Corresponding Author**: Chaiyan.Oro@rmutr.ac.th

Received: 30 June 2017; **Revised:** 24 January 2018; **Accepted:** 30 January 2018; **Available online:** 1 May 2018

Paper selected from The 3rd International Conference on Applied Physics and Material Applications 2017 (ICAPMA 2017)

Abstract

The gold nanoparticles (Au NPs) decorated tungsten oxide nanorods (WO_3 NR) arrays have been fabricated by physical vapor deposition method. Firstly, the WO_3 NR arrays were fabricated by dc reactive magnetron sputtering with glancing angle deposition technique. The Au NPs of different sizes and morphology were uniformly sputtered on WO_3 NR arrays via normal dc magnetron sputtering. The influence of Au deposition operated pressure on the structure and morphology of the resulting Au NPs on WO_3 NR arrays were systematically studied. Crystal structure, morphologies and element analysis of as prepared samples were characterized by X-ray diffraction, field-emission scanning electron microscopy, and energy dispersive X-ray spectroscopy. The result indicated that the Au nanoparticles prepared at low operated pressure were mostly located near the top of the WO_3 nanorods.

Keywords: Au decoreated WO_3; glancing angle deposition; sputtering; morphology

1. Introduction

Metal Oxide in forms of nanostructures, such as nanowires, nanobelts, nanotubes and nanorods have been studied for gas-sensing applications due to their large surface-to-volume ratio. The nanostructures of metal oxide semiconductor gas-sensing materials, such as SnO_2, WO_3 and ZnO have showed higher response with improved sensitivity at low gas concentration compared with thin-film materials [1 – 3]. Moreover, the gas sensing performance can be achieved by doped with novel metal such as Au nanoparticle and Pt nanoparticle, which were used as catalyst materials [4 – 6]. Among various metal oxide semiconductor nanostructure materials, Tungsten trioxide (WO_3) is an n-type semiconductor with a band gap of 2.60 − 3.60 eV. Gas sensors based on WO_3 nanostructures have demonstrated high sensitivity and fast response due to their large surface-to-volume ratio [7, 8]. Several fabrication methods have been developed to glow WO_3 nanostructure including chemical vapor deposition (CVD) [9], physical vapor deposition (PVD) [10, 11] and hydrothermal. These methods mostly produce random WO_3 nanostructures, which are not desirable for commercial applications.

Glancing angle deposition (GLAD) deposition technique, in which a high melting point material flux is incident onto the substrate from a glancing angle, α, in which substrate surface is rotated and tilted to an angle of greater than 80° with respect to the normal of substrate surface or less than 10° with respect to the direction of vapor flux. Low surface mobilities of adatoms lead to kinetic limitations such as geometrical confinements and atomic shadowing, resulting in the formation of a variety of porous columnar microstructures [12 – 14]. A physical vapor deposition (PVD) with glancing-angle deposition (GLAD) technique is a method to grow well ordered metal oxide nanostructures. In this work, WO_3 nanorods are prepared by glancing angle deposition (GLAD) technique using reactive DC magnetron sputtering process and the result of operate pressure Au Nanoparticles Decorated WO_3 Nanorods formation is studied.

2. Materials and methods

Tungsten trioxide (WO_3) nanorods were fabricated by dc reactive magnetron sputtering system using GLAD technique. The distance from the target to substrate center and the substrate rotation speed were fixed at 5 rpm. The substrate normal was positioned at an angle of 85° (α = 85°) with the respect to the vapor incident flux. A 3-in. metallic tungsten disc with 99.99% purity was used as sputtering target. Pure argon (99.999%) and oxygen (99.999%) were used as a sputtering and reactive gas, respectively. The flow rate of Ar and O_2 was kept at constant value of 8 sccm and 24 sccm, respectively. The silicon wafers (100) substrates were prepared by ultrasonic washer with acetone, before loaded into the deposition chamber. The deposition time was fixed at 60 min. Then, the gold nanoparticles (Au NPs) were deposited on WO_3 nanorods (WO_3 NR) arrays as illustrated in Fig. 1. The influence of Au Nanoparticles Decorated WO_3 Nanorods was generated with varing operated pressure from 10 mTorr to 30 mTorr were studied. The film crystalline structures were examined by a RIGAKU, TTRAX III X-ray diffractometer operating with Cu-$K_{\alpha 1}$ source. The measurements were conducted from 20 – 60° incident angles. The morphology of the films was examined by using Field Emission Scanning Electron Microscopy (FE-SEM), Hitachi model S-4700 and Transmission Electron Microscopy (TEM).

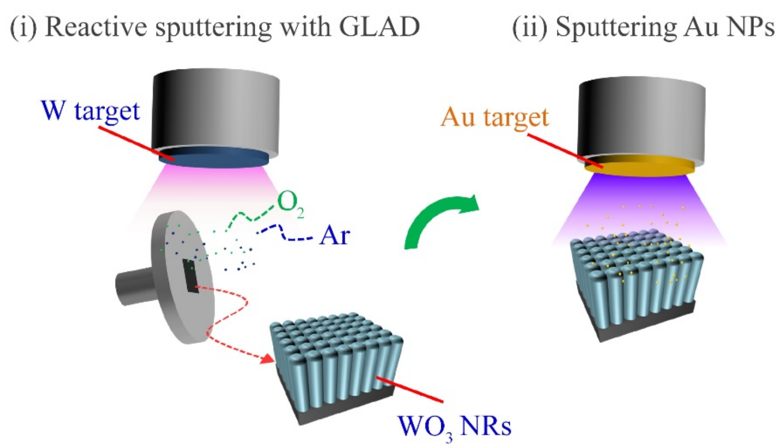

Fig. 1 Schematic of GLAD with sputtering process

3. Results and Discussion

The crystal structure of WO_3 nanorod thin films with ad-deposit (ADS) and Au NPs decorate pressure of 10 to 30 mTorr are revealed in X-ray diffractograms as shown in Fig. 2. It can be seen that the crystal structure of all WO_3 nanorod films exhibits amorphous structure due to the low energy and the low mobility. In addition, there are no diffraction peak corresponding to the Au phase of the decorated nanoparticles. The results indicated that the Au NPs should be very thin so that its diffraction signals was too low to be detected by the XRD. In addition, it can be noticed that the Au NPs deposited on WO_3 NRs were not clearly observed by the necked eyes due to the small particles size. The EDS, FE-SEM and TEM will be used to confirm the presences of the Au nanoparticles.

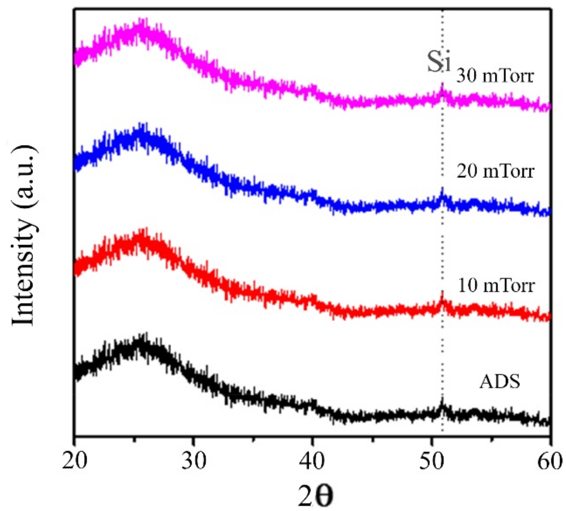

Fig. 2 Typical X-ray diffraction patterns of WO_3 nanorods with as-deposited (ADS) and Au NPs decorate pressure of 10 to 30 mTorr.

Typical SEM images of WO_3 thin films with ad-deposit (ADS) and Au NPs decorate pressure of 10 to 30 mTorr are shown in Fig. 3. It can be seen that the WO_3 thin films exhibits columnar nanorods structures formed by atomic self-shadowing due to glancing angle deposition. From the SEM images, the average diameter of WO_3 nanorods are estimated to be 50 nm and everage length of WO_3 nanorods about 167 nm. After decorated Au NPs with operate pressure 10 to 30 mTorr, a number of small spots Au decoration were observed to be distributed rather uniformly over the WO_3 nanorods and the everage size of Au NPs are estimated to be 5 – 10 nm . In addition, the Au nanoparticle on the WO_3 nanorods film decreases from 2.10 to 0.90 wt % (1 to 0.40 Atomic %) as Au decoration pressure increases from 10 to 30 mTorr due to the Au NPs were lower scatter at high pressure, which low plasma energy to sputter Au target. The Weight (%) and Atomic (%) of Au NPs Vs. Au decorate pressure 10 to 30 mTorr as shown in Fig. 4.

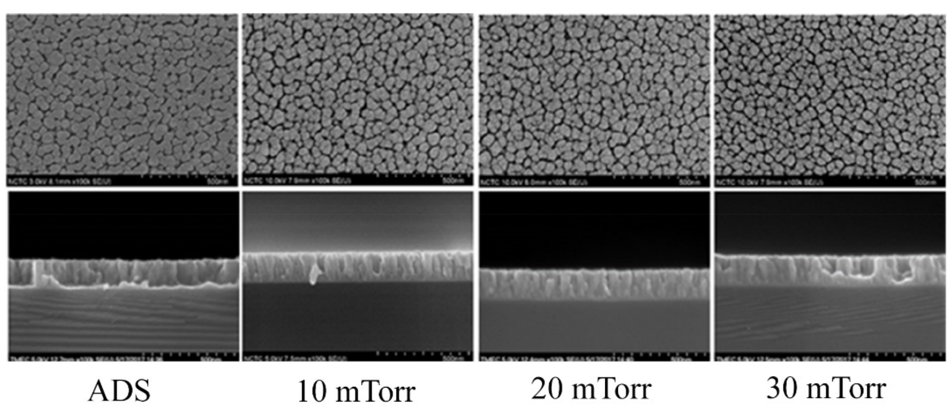

Fig. 3 Typical FE-SEM image of WO_3 nanorods with as-deposited and Au NPs decorate pressure of 10 to 30 mTorr

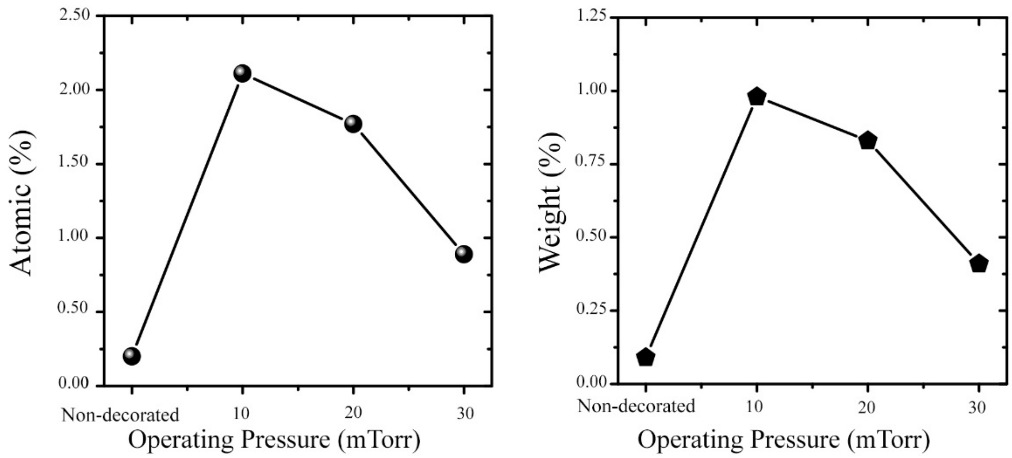

Fig. 4 Weight (%) and Atomic (%) of Au NPs Vs. Au decorate pressure 10 to 30 mTorr

The detailed structure and morphologies of the prepared nanostructure were further investigated by TEM. In Fig. 5 shows a typical Au NPs decorated on WO_3 NRs deposited at 10 mTorr-operated pressure. It can be confirmed that the nanoparticles are mostly located near the top of nanorods and the Au particle diameter was estimated be in the range of ~5 nm which correspond to the SEM from the previous result.

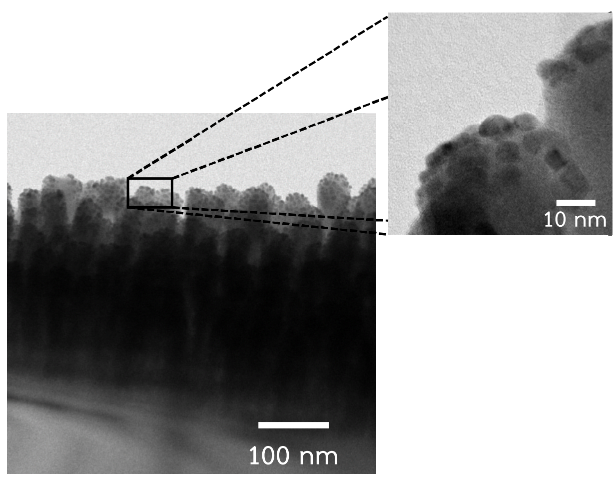

Fig. 5 TEM images of the Au nanoparticles decorated on WO_3 nanorods

4. Conclusion

In conclusion, the WO_3 nanorods were deposited by dc magnetron sputtering with GLAD technique. The structure and morphology of Au NPs decorated on WO_3 nanorods with varies operate pressure 10 to 30 mTorr were studied. It was found that the crystal structure of all WO_3 nanostructure thin films exhibits amorphous due to the low energy and the low mobility. A number of Au nanoparticle decoration are observed to be distributed over the WO_3 nanorods and decreases with decoration pressure increases. Future this work will be determined the effect of decorated Au nanoparticles for WO_3 nanorods gas sensing.

5. Acknowledgement

This work was supported by Rajamangala University of Technology Rattanakosin and Optical Thin-Film Laboratory, National Electronics and Computer Technology Center, National Science and Technology Development Agency (NSTDA), Thailand.

6. References

[1] B. Wang, L.F. Zhu, Y.H. Yang, N.S. Xu, G. W. Yang, Fabrication of a SnO_2 Nanowire Gas Sensor and Sensor Performance for Hydrogen, J. Phys. Chem. C. 112(17) (2008) 6643 – 6647.

[2] D. Deniz, D. J. Frankel, R. J. Lad, Nanostructured tungsten and tungsten trioxide films prepared by glancing angle deposition, Thin Solid Films. 518 (2010) 4095 – 4099.

[3] J. M. LaForge, M. T. Taschuk, M. J. Brett, Glancing angle deposition of crystalline zinc oxide nanorods, Thin Solid Films. 519 (2011) 3530 – 3537.

[4] S. Kim, S.H. Park, S.Y. Park, C. Lee, Acetone sensing of Au and Pd-decorated WO_3 nanorod sensors, Sens. Actuators B. 209 (2015) 180 – 185.

[5] K. Li, W. Wang, D. Cao, Metal (Pd, Pt)-decorated carbon nanotubes for CO and NO sensing, Sens. Actuators B. 159 (2011) 171 – 177.

[6] Q.N. Abdullah, F.K. Yama, Z. Hassan, M. Bououdina, Pt-decorated GaN nanowires with significant improvement in H_2 gas-sensing performance at room temperature, J. Colloid Interface Sci. 460 (2015) 135 – 145.

[7] M. Horprathum, K. Limwichean, A. Wisitsoraat, P. Eiamchai, K. Aiempanakit, P. Limnonthakul, N. Nuntawong, V. Pattantsetakul, A. Tuantranont, P. Chindaudom, NO_2-Sensing Properties of WO_3 Nanorods Prepared by Glancing Angle DC Magnetron Sputtering, Sens. Actuators B. 176 (2013) 685 − 691.

[8] C. Wongchoosuk, A. Wisitsoraat, D. Phokharatkul, M. Horprathum, A. Tuantranont, T. Kerdcharoen, Carbon Doped Tungsten Oxide Nanorods NO_2 Sensor Prepared by Glancing Angle RF Sputtering, Sens. Actuators B. 181 (2013) 388 − 394.

[9] C.M. White, T. Dane, E. Whitney, S.H. Lee, A.C. Dillon, Flexible Electrochromic Devices Based on Crystalline WO_3 Nanostructures Produced with Hot-Wire Chemical Vapor Deposition, Thin Solid Films. 517 (2009) 3596 − 3599.

[10] Y.B. Li, Y. Bando, D. Golberg, K. Kurashima, WO_3 Nanorods/Nanobelts Synthesized via Physical Vapor DepositionProcess, Chem. Phys. Lett. 367 (2003) 214 − 218.

[11] Y. Shen, T. Yamazaki, Z. Liu, D. Meng, T. Kikuta, N. Nakatani, Influence of Effective Surface Area on Gas Sensing Properties of WO_3 Sputtered Thin Films, Thin Solid Films. 517 (2009) 2069 − 2072.

[12] C. Oros, M. Horprathum, A. Wisitsoraat, T. Srichaiyaperk, B. Samransuksamer, S. Limwichean, P. Eiamchai, D. Phokharatkul, N. Nuntawong, C. Chananonnawathorn, V. Patthanasettakul, A. Klamchuen, J. Kaewkhao, A. Tuantranont, P. Chindaudom, Ultra-sensitive NO_2 sensor based on vertically aligned SnO_2 nanorods deposited by DC reactive magnetron sputtering with glancing angle deposition technique, Sens. Actuators B. 223 (2016) 936 – 945.

[13] D. Deniz, D.J. Frankel, R.J. Lad, Nanostructured tungsten and tungsten trioxide films prepared by glancing angle deposition, Thin Solid Films. 518 (2010) 4095 – 4099.

[14] H.J. Gwon, H.G. Moon, H.W. Jang, S.J. Yoon, K.S. Yoo, Sensitivity enhancement of nanostructured SnO_2 gas sensors fabricated using the glancing angle deposition method, J. Nanosci. Nanotechnol. 13 (4) (2013) 2740 – 2744.