LIST OF PUBLICATIONS, CONFERENCES, AND PATENTS

1. List of publications required for PhD

1.1) **Meechai Thepnurat**, Torranin Chairuangsri, Niyom Hongsith, Pipat Ruankham, and Supab Choopun, "Realization of Interlinked ZnO Tetrapod Networks for UV Sensor and Room-Temperature Gas Sensor," ACS Applied Materials & Interfaces, 7 (2015) 24177-24184. (**IF = 6.723**).

1.2) **Meechai Thepnurat**, Pipat Ruankham, Surachet Phadunghitidhada, Atcharawon Gardchareon, Duangmanee Wongratanaphisan, and Supab Choopun, "Efficient Charge-Transport UV Sensor Based on Interlinked ZnO Tetrapod Networks," Surface&Coatings Technology, In Press. (**IF= 1.998**).

2. List of other publications

2.1) Karaket Kaewyai, Supab Choopun, **Meechai Thepnurat**, Atcharawon Gardchareon, Surachet Phadunghitidhada, and Duangmanee Wongratanaphisan, "Preparation and Characterization of Copper Oxide Nanofibers by Microwave-Assisted Thermal Oxidation," Journal of Nanoelectronics and Optoelectronics, 8 (2013) 472-476. (**IF= 0.369**)

2.2) Witawat Ponhan, **Meechai Thepnurat**, Surachet Phadungdhitidhada, Duangmanee Wongratanaphisan, Supab Choopun, "Electrical properties of field-effect transistor with interlinked ZnO tetrapod network as an active layer," Surface&Coatings Technology, In Press. (**IF= 1.998**).

3. List of Conferences

3.1) **Meechai Thepnurat**, Surachet Phadunghitidhada, Atcharawon Gardchareon, and Duangmanee Wongratanaphisan, and Supab Choopun "Microwave-Assisted Synthesis and Characteristics of ZnO Tetrapod-networks for Gas Sensor Application", **10th Asian Conference on Chemical Sensors (ACCS 2013)** Chemical sensor for the sustainable society. November 11-14, 2013, at The Empress hotel, Chiang Mai, Thailand. (Oral presentation)

3.2) **Meechai Thepnurat**, Surachet Phadunghitidhada, Pipat Ruankham, Atcharawon Gardchareon, Duangmanee Wongratanaphisan, and Supab Choopun "UV Sensor Based on Inter-linked ZnO Tetrapod Networks Prepared by Microwave-assisted Thermal Oxidation Technique", **The 19th International Conference on Surface Modification of Materials by Ion Beams (SMMIB-19)** 22nd (Sun.) to 27th (Fri.) November, 2015 at The Empress hotel, Chiang Mai, Thailand. (Oral presentation)

4. List of Patents

4.1) **มีชัย เทพนุรัตน์**, การะเกด แก้วใหญ่, อัจฉราวรรณ กาศเจริญ, สุภาพ ชูพันธ์, ดวงมณี ว่องรัตนะไพศาล, สุรเชษฐ์ ผดุงธิติธาดา, "กระบวนการเตรียมโลหะและโลหะออกไซด์ระดับนาโนเมตรด้วยวิธีเทอร์มอร์ออกซิเดชันร่วมกับคลื่นแม่เหล็กไฟฟ้า," เลขที่คำขอ 1401001279, 10 มีนาคม 2557

APPENDIX A

PUBLICATIONS REQUIRED FOR PhD

1. **Meechai Thepnurat**, Torranin Chairuangsri, Niyom Hongsith, Pipat Ruankham, and Supab Choopun, "Realization of Interlinked ZnO Tetrapod Networks for UV Sensor and Room-Temperature Gas Sensor," ACS Applied Materials & Interfaces, 7 (2015) 24177-24184. (IF = 6.723).

ACS APPLIED MATERIALS & INTERFACES

Research Article

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Realization of Interlinked ZnO Tetrapod Networks for UV Sensor and Room-Temperature Gas Sensor

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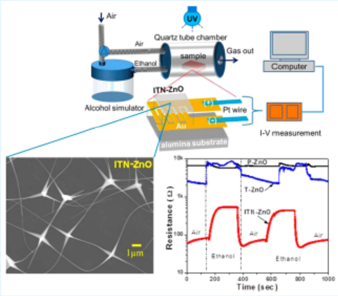
ABSTRACT: Here, interlinked ZnO tetrapod networks (ITN-ZnO) have been realized by using microwave-assisted thermal oxidation. With this simple and fast process, a nanostructured ZnO morphology having tetrapodlike features with leg-to-leg linking is obtained. The electrical and ethanol-sensing properties related to the morphology of ITN-ZnO compared with those of other ZnO morphologies have also been investigated. It has been found that ITN-ZnO unexpectedly exhibits superior electrical and gas-sensing properties in terms of providing pathways for electron transport to the electrode. A UV sensor and a room-temperature gas sensor with improved performance are achieved. Therefore, ITN-ZnO is an attractive morphology of ZnO that is applicable for many new applications because of its novel properties. The novel properties of ITN-ZnO are beneficial for electronic, photonic, optoelectronic, and sensing applications. ITN-ZnO may provide a means to improve the devices based on ITN-ZnO.



KEYWORDS: *ZnO, tetrapod, nanostructure, thermal oxidation, gas sensor*

1. INTRODUCTION

Nanotechnology has been considered a breakthrough technology that is expected to have a great impact on the scientific community and industrial revolution.[1−3] This is mainly due to the novel properties of nanomaterials resulting from reduction in size that could open for new generations of nanodevices[4] a wide range of applications.[5,6] In addition, nanostructures or shapes of nanomaterials also play an important role for this emerging development.[7]

ZnO has caught attention because of its wide variety of structures or morphologies such as nanowires,[8,9] nanobelts,[10] nanorings,[11,12] nanoparticles,[13] and tetrapods[14,15] and may be the richest family among all materials in both structures and properties. For the past decade, nanoengineering[16] of ZnO by controlling its size and morphology using various synthesis methods has been performed, and a great variety of nanostructured ZnO morphologies have been accomplished.[12,17,18] The morphological diversity of nanostructured ZnO leads to some interesting properties including surface-related[19] and optoelectrical properties.[20−22] Thus, controlling the growth kinetics of the synthesis process via nanoengineering is an important issue for utilizing ZnO nanostructures.[18,23,24]

Here we have controlled the growth kinetics of ZnO nanostructures by using microwave-assisted thermal oxidation (MWTO). With only Zn powder and a household microwave oven, we have realized a nanostructured ZnO morphology having tetrapodlike features with leg-to-leg linking, a so-called "interlinked tetrapod network of ZnO" or ITN-ZnO. Moreover, this ITN-ZnO has unexpectedly exhibited superior electrical and gas-sensing properties in comparison with those of other morphologies of ZnO. Characteristics of UV sensors and room-temperature gas sensors, as examples for potential applications, have also been reported.

2. EXPERIMENTAL SECTION

2.1. Preparation of ITN-ZnO. Zinc (Zn) powder (2 g; 99.99%, less than 50 μm particle size) as a precursor loaded on a quartz substrate was placed in a quartz tube with diameter of 2.8 cm and length of 10 cm in a household microwave oven (SHARP model), as shown in Figure 1. The Zn powder was then heated with microwave power of 700 W at a frequency of 2.45 GHz for 60 s under atmospheric conditions. Finally, after the system cooled down, the wool-like ZnO structures were observed in the quartz tube and collected for further investigation.

2.2. Fabrication of Sensors. ITN-ZnO, ZnO tetrapods (T-ZnO), and zinc oxide powder (P-ZnO; 99.9%, Sigma-Aldrich, less than 1 μm in particle size) were mixed in ethanol and screened on alumina substrates with gold interdigital electrodes as shown in Figure 2. Pt wires were then used to connect them with the Au electrodes, and the samples were put in the gas chamber under UV radiation.

2.3. Characterization. Crystallinity and morphology of wool-like ZnO products were characterized via an X-ray powder diffractometer (Siemens D-500) with Cu Kα radiation, field emission electron microscope (FE-SEM; Hitachi S-450 SEM), and transmission electron microscope (TEM; JEOL 2010 FEG STEM/TEM). To investigate the

Received: August 13, 2015
Accepted: October 12, 2015
Published: October 12, 2015

DOI: 10.1021/acsami.5b07491
ACS Appl. Mater. Interfaces 2015, 7, 24177−24184

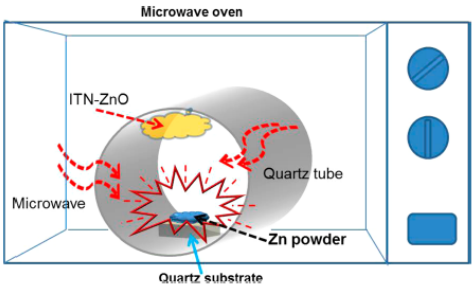


Figure 1. Schematic illustration of the preparation system of ITN-ZnO. This system uses a household microwave oven (SHARP model) with power of 700 W and frequency of 2.45 GHz (λ= 12 cm). Zinc powder (2 g) on a quartz substrate was placed in a quartz tube and heated for 60 s. The wool-like products obtained in the quartz tube were collected for further investigation.

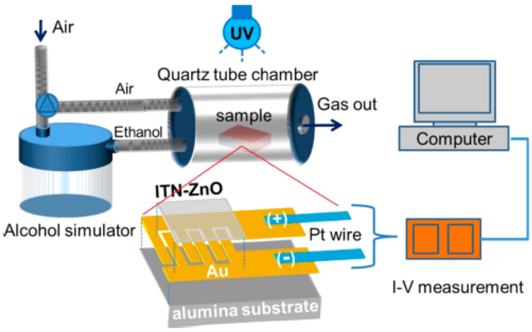


Figure 2. Schematic illustration of the sensor and the measurement system. ITN-ZnO, T-ZnO, and P-ZnO coated on alumina substrates with gold interdigital electrodes were put in the quartz tube chamber with UV lamps (λ = 365 nm; UVA), and $I-V$ characteristics were measured with computer-controlled dc voltage and current sources. Ethanol vapor with a concentration of 1000 ppm was injected into the chamber at operating temperatures varying from room temperature to 500 °C.

UV-sensing properties, a UV light source (365 nm; UVA) with intensity of 2.80 mW/cm^2 was used. Current−voltage ($I-V$) characteristics of the sensors irradiated with UVA were measured in air or under nitrogen conditions using a dc voltage and current sources that were interfaced and controlled by a computer. To characterize the ethanol-sensing properties, the sensors were exposed to ethanol vapor at a concentration of 1000 ppm at an operating temperature varying from room temperature to 500 °C under UV illumination.

3. RESULTS AND DISCUSSION

3.1. Morphologies of Interlinked Tetrapod Network of ZnO. The morphologies of ZnO products obtained from the MWTO are shown in Figure 3. The ZnO products can be distinguished into two regions with two different morphologies as shown in Figure 3a. In the bottom region, tetrapodlike ZnO structures (T-ZnO) about 1 μm in diameter at the middle of their legs and 10−30 μm in length is observed as seen in Figure 3b.

In contrast, in the upper region, the tetrapodlike structure with leg-to-leg linking (ITN-ZnO) is observed as seen in Figure 3c,d. It can be seen that the legs of ZnO tetrapods are interlinked with the legs of neighbor tetrapods about at the end of the legs and look similar to the shape of a neural network. The diameter at the middle of each leg is in the order of 50 nm, which is much smaller than that of T-ZnO in the bottom region. This ITN-ZnO is morphologically different from T-ZnO in the bottom region.

The XRD patterns of ITN-ZnO, T-ZnO, and P-ZnO have been obtained and are shown in Figure 4. It can be seen that the XRD peaks of all samples are consistent with that of Powder Diffraction File No.79-2205 (International Centre for Diffraction Data, 1979). This indicates that the as-prepared ITN-ZnO and T-ZnO are wurtzite crystal structures. Moreover, there is no characteristic peak of Zn metal observed in the XRD spectra. This can be interpreted as meaning that the metallic Zn precursors completely transform into ZnO nanostructures during MWTO.

To understand the ITN-ZnO formation mechanisms, TEM observation has been used for further investigation. Figure 5a shows a bright-field TEM (BF-TEM) image of the two interlinked ZnO tetrapods, and Figure 5b shows a high-magnification BF-TEM image of the leg marked I of tetrapod A connecting with the leg marked II of tetrapod B, together with a corresponding selected-area electron diffraction pattern (SADP) monitored at the connected region of these legs. From trace analysis, it is possible that these legs grow along <0001> c-axis direction, which is usual for the growth direction of ZnO tetrapods.[25] Hence, it can be suggested that at the connected region these legs grow in the opposite but equivalent <0001> c-axis directions.

Figure 5c presents a high-resolution transmission electron microscopy (HRTEM) image of the linking between legs I and II. The spacing of the lattice fringes is about 0.26 nm, which is equal to $c/2$, where c is the c-axis lattice parameter in the ZnO hexagonal structure (0.52 nm). It can be seen that at the boundary between the legs the lattice fringes are continuous suggesting epitaxial linking. However, the lattice fringes of both legs are not fully matched, and a small mismatch displacement of about $c/8$ can be clearly observed.

To explain the structural observation as mentioned above, the schematic plan view of the hexagonal ZnO structure along zone axes is sketched in Figure 5d. It is composed of alternating Zn and O ion planes stacking along the c axis. In the wurtzite structure of ZnO, an oxygen ion plane locates at $c/8$, which is far from the nearest-neighbor oxygen ion plane of $c/2$ along the c axis. Similarly, the distance between the basal zinc plane and the nearest-neighbor one is $c/2$. As seen in TEM images, legs I and II are connected with opposite growth directions and can be bound together via some electrostatic force or ionic bond. Thus, it is likely that the connection between two legs is performed by chemically bonding between positively charged zinc ions with negatively charged oxygen ions because of columbic charge interaction. Hence, the small mismatch of about $c/8$ at connection boundary is required for this chemical bonding as observed in Figure 5c.

Moreover, the residual charges on the surface of ZnO are prominent for the case of single-crystalline materials, as seen in previous reports.[10,12] For our ITN-ZnO, it can be seen from TEM results (Figure 5c) that it exhibits single-crystalline properties. Thus, the residual charges on the surface of ZnO can be taken as resulting in columbic interaction.

On the basis of the collected results and above discussion, it can be noted that the growth kinetics of ITN-ZnO is composed of two main steps: tetrapod growth and connecting and linking growth. The detailed study is our ongoing research. The growth

DOI: 10.1021/acsami.5b07491
ACS Appl. Mater. Interfaces 2015, 7, 24177−24184

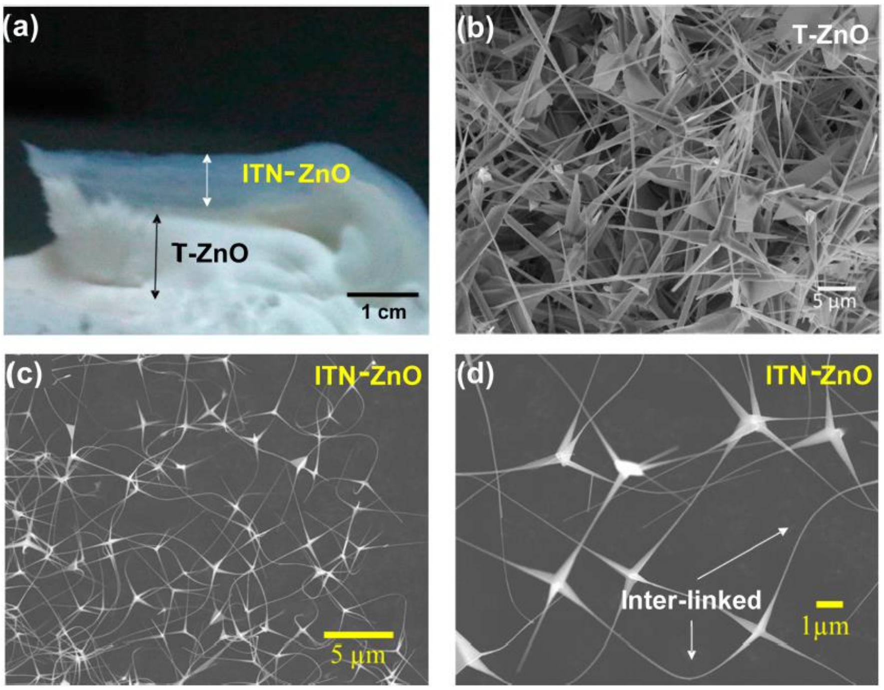


Figure 3. Morphologies of ZnO products obtained from microwave-assisted thermal oxidation: (a) optical image showing two distinguishable regions of ZnO products, (b) FE-SEM image of tetrapodlike ZnO (T-ZnO) obtained at the bottom region, (c) FE-SEM image of ITN-ZnO obtained at upper region, and (d) high-magnification FE-SEM image of ITN-ZnO showing the interlink between tetrapods.

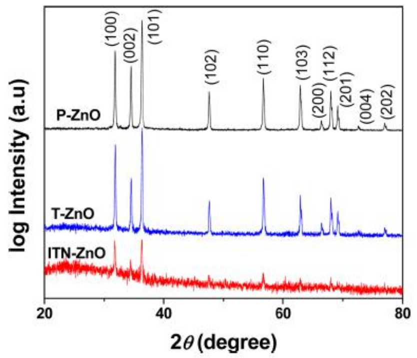


Figure 4. XRD patterns of ITN-ZnO, T-ZnO, and P-ZnO characterized by X-ray powder diffractometer (Siemens D-500) with Cu Kα radiation. It can be seen that all samples have a similar XRD pattern and that the diffraction peaks can be indexed as the wurtzite structure ZnO with $a = b = 0.3253$ nm and $c = 0.5213$ nm, which is in good agreement with PDF No.79-2205, ICDD, 1979.

kinetics of ZnO tetrapods can be explained in terms of a high supersatuation ratio, which results in an increase in the nucleation probability as given by

$$P = B\exp\left(\frac{-\pi\sigma^2}{{k_B}^2T^2\ln(\alpha)}\right) \quad (1)$$

where B is a parameter constant, σ is the surface energy of the solid tetrapod, k_B is the Boltzmann's constant, T is the absolute temperature, and α is the supersaturation ratio between the actual vapor pressure and the equilibrium vapor pressure corresponding to the temperature T (usually $\alpha > 1$).

Unlike to other synthesis methods, MWTO is able to generate a high vapor pressure in very short time, resulting in an ultrahigh supersaturation condition inside the quartz tube (within 60 s in this case). Then, the ITN-ZnO can be formed by condensation of ZnO vapor at supersaturation conditions with fast growth along the c axis with leg-to-leg linking and slow growth in the other directions.[25−27] This can be achieved at the upper region because the highest vapor pressure can be likely obtained at the top of quartz tube because of the vapor accumulation, leading to the condensation of ZnO super-saturation vapor forming on the upper crust.

3.2. Electrical Properties of ITN-ZnO under UV-Light Illumination. Sensors based on the nanostructured ZnO (ITN-ZnO and T-ZnO) and commercially available zinc oxide powder (P-ZnO) were fabricated using alumina substrates with gold interdigital electrodes as shown in Figure 2. The electrical properties of ITN-ZnO under dark and UV illumination in either air or nitrogen ambient are investigated and are shown as $I-V$ characteristics with a linear scale in Figure 6a and a semilogarithmic scale in Figure 6b. It can be seen that the current of ITN-ZnO sensor exhibits the highest value (lowest resistance) under the condition of UV illumination in nitrogen ambient. Comparing with the value collected under the dark condition, the lower resistance under UV illumination can be explained by the increment of photogenerated electrons caused

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ACS Appl. Mater. Interfaces 2015, 7, 24177−24184

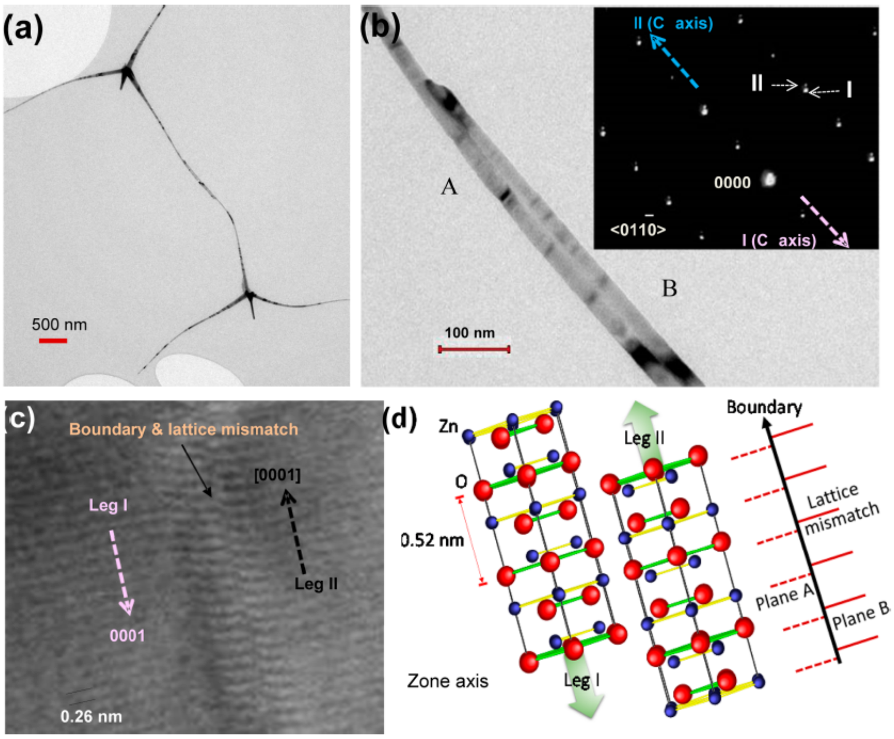

Figure 5. TEM images of two interlinked ZnO tetrapods: (a) BF-TEM image of two interlinked ZnO tetrapod network (A and B). (b) High-magnification BF-TEM image of the leg I of the tetrapod A connecting with the leg marked II of the tetrapod B, together with a corresponding selected-area electron diffraction pattern (SADP) from the connected region of these legs. (c) HRTEM image of the boundary between legs I and II. (d) Schematic plan view of the hexagonal ZnO structure along <10$\bar{1}$0> zone axes shown in order to explain the small mismatch displacement of about $c/8$ at the boundary.

by the light absorption of ZnO nanostructures. In addition, the further decrease in resistance measured under UV illumination in nitrogen ambient can be explained by the increase of conduction-band photoelectrons resulting from the decrease of oxygen adsorption at the surface. Thus, UV light and oxygen atmosphere play an important role for electrical properties of ITN-ZnO.

Figure 6c shows the reversible switching curves of electrical current through the devices for different ZnO morphologies tested under UV illumination in air. When the UV light is switched on/off every 200 s at a constant bias voltage of 5 V, the on/off current ratio of ITN ZnO exhibits the highest value of about 7400 compared to about 2.6 and 0.3 for T-ZnO and P-ZnO sensors. This suggests that the ITN-ZnO sensor exhibits superior UV-detecting properties and clearly differs from the T-ZnO and P-ZnO sensors. This may be attributed to a better transport pathway of ITN-ZnO for electrons that required reaching the Au electrodes. Comparing with T-ZnO and P-ZnO sensors, the ITN-ZnO sensor provides negligible effects of grain boundaries that limit electron transport because the legs are connected with the columbic interaction as described above. Interestingly, by comparing this characteristic with the best results of previously reported literature as summarized in Table 1, the photo−dark current ratio (I_{UV}/I_{dark}) of our ITN-ZnO device is a higher value (7400) in comparison with that of the device based on nanotetrapod network of ZnO (4500).[28] These results suggest that the ITN-ZnO has great potential for UV sensor application.

3.3. Ethanol-Sensing Properties of Different ZnO Morphologies. The ethanol-sensing properties of devices based on different ZnO morphologies are investigated. Figure 7a shows two cycles of resistance change under ethanol ambient for sensors based on different ZnO morphologies at concentration of 1000 ppm and operating temperature of 450 °C. It can be seen that the sensor resistance rapidly decreases when ethanol vapor is injected into the chamber, and the value returns back to the original resistance when ethanol vapor is removed. This indicates that the nanostructured ZnO morphologies are applicable for ethanol sensors.

The sensor response (ratio of the resistance measured in air to that in ethanol) is plotted in Figure 7b as a function of operating temperatures. The sensor response depends on ZnO morphology, and the highest sensor response is observed in the T-ZnO device. This suggests that, at high operating temperature, the ITN-ZnO sensor exhibits different ethanol-sensing properties to the other ZnO morphologies.

From the above discussion, in order to get further insights into the electrical and gas sensing properties related to the morphology of nanostructured ZnO, the combined effect of UV illumination and ethanol ambient on the device characteristics have been investigated. Thus, ethanol-sensing properties under UV illumination are investigated at room temperature

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ACS Appl. Mater. Interfaces 2015, 7, 24177−24184

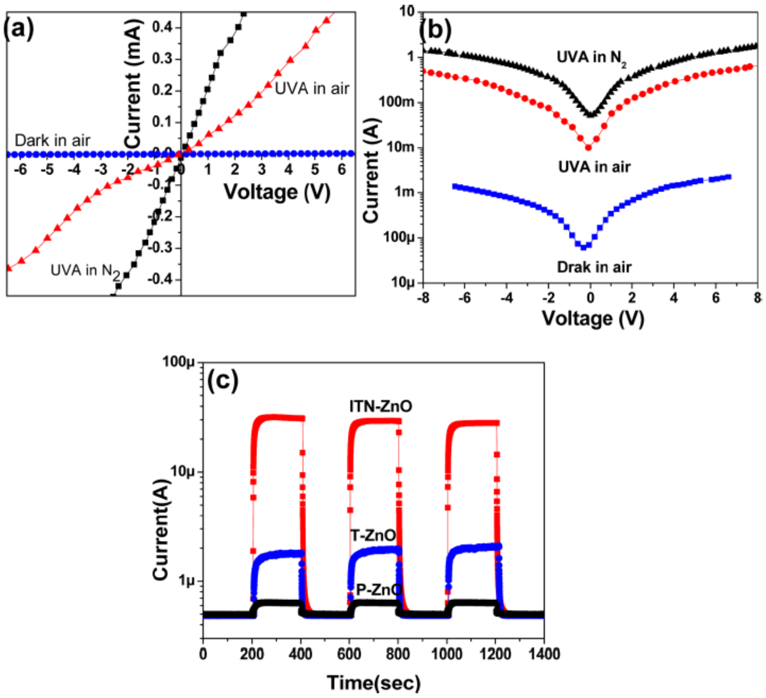

Figure 6. *I*−*V* characteristics of ITN-ZnO sensor monitored under dark and UV illumination in either air or nitrogen ambient plotted (a) in a linear scale and (b) in a semilogarithmic scale. (c) Reversible switching curves of electrical current for devices based on different ZnO morphologies.

Table 1. Summary of the Previous Reports on UV Sensors/DetectorsBased on ZnO Nanostructures

type of structure	duration of growth and method	UV intensity at 365 nm (mW/cm^2)	bias voltage (V)	I_{UV}/I_{dark}	rise time	decay time	ref
nanorodnetwork	3−5 h, HT	0.3	4	1.8	2 s		30
nanoneedle network	4 h, C-FTS	15−20	0.3	312	22 s	7−12 s	28
nanotetrapod network	<5 s, B-FTS	15−20	2.4	4500	67 ms	30 ms	28
powder	Sigma-Aldrich	2.8	5	0.3	4.38 s	4.76 s	this work
T-ZnO	60 s, MWTO[a]	2.8	5	2.6	4.73 s	2.00 s	this work
ITN-ZNO	60 s, MWTO[a]	2.8	5	7400	3.52 s	0.67 s	this work

[a]MWTO: microwave-assisted thermal oxidation.

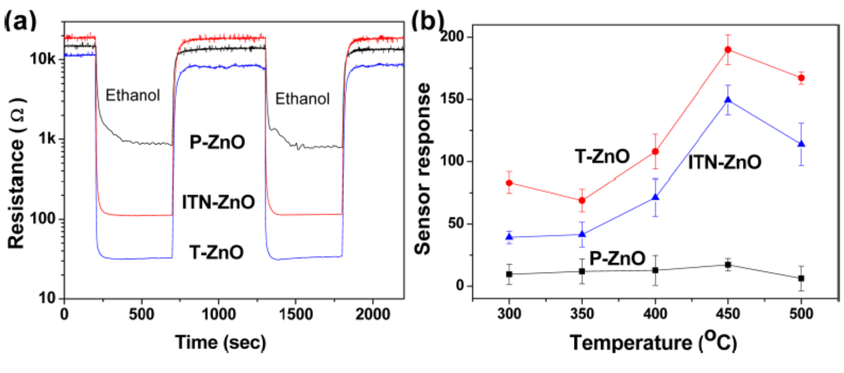

Figure 7. (a) Two cycles of resistance change under ethanol ambient for a sensor based on different ZnO morphologies at an ethanol concentration of 1000 ppm and an operating temperature of 450 °C. (b) Plot of sensor response (ratio of resistance measured in air relative to that in ethanol) at various operating temperatures.

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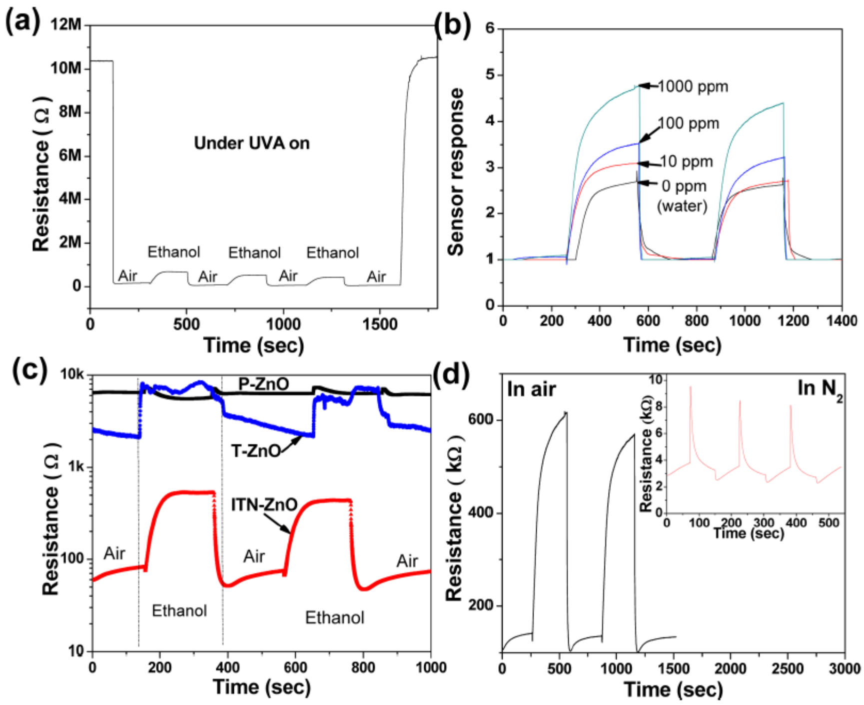

Figure 8. (a) Resistance change of ITN-ZnO sensor tested under UV illumination in air and ethanol ambient as a function of time at room temperature and ethanol concentration of 1000 ppm. (b) Sensor response at room temperature and ethanol concentration of 10−1000 ppm. (c) Resistance change of ITN-ZnO sensor in comparison with P-ZnO and T-ZnO sensors tested under UV illumination at room temperature and ethanol concentration of 1000 ppm. (d) Resistance change of ITN-ZnO sensor under UV illumination at room temperature and ethanol concentration of 1000 ppm, tested in air, along with an inset showing the resistance tested in nitrogen ambient.

and different operating temperatures. It is worth noting that thermal energy is not required because UV radiation is used instead for conduction band electron excitation. The resistance of ITN-ZnO sensor under UV illumination in air and ethanol ambient at room temperature is plotted as a function of time as shown in Figure 8a. The resistance (conductance) of the ITN-ZnO device rapidly decreases (increase) because of the increase of photoelectrons by photon absorption and electron excitation from valence band to conduction band. After the UVA irradiation is cut down, the sensor resistance returns to the original value. However, when the ethanol vapor is injected in the chamber, the resistance of the ITN-ZnO sensor surprisingly increases, which is opposite to the case at high operating temperature. (Typically, the resistance increase under ethanol vapor indicates p-type behavior of semiconductor.) Also, the increase in resistance and sensor response depends on the ethanol concentration, as shown in Figure 8b from 10 to 1000 ppm. This suggests the possibility for new application as a gas sensor operating at room temperature.

Compared with the sensors constructed from T-ZnO and P-ZnO, the ITN-ZnO sensor exhibits superior ethanol-sensing properties under UV illumination at room temperature as seen in Figure 8c. To understand the effects of oxygen molecules in air on the ethanol-sensing characteristics, the sensor resistance of a ITN-ZnO device monitored in air is compared with that measured in nitrogen ambient as shown in Figure 8d. It can be seen that the increased resistance in nitrogen is much smaller than that of in air. This suggests that oxygen molecules play an important role in the ethanol-sensing mechanism at room temperature.

Thus, the resistance increase can be explained by using the ethanol-sensing mechanism at room temperature as sketched in Figure 9. The proposed sensing mechanism is composed of four stages. First, under UV illumination, photoelectrons are generated from photoexcited electrons in the valence band moving to the conduction band via UV absorption, resulting in the decrease in sensor resistance. Second, oxygen molecules in the air trap the electrons, forming superoxide radicals (O_2^-). At

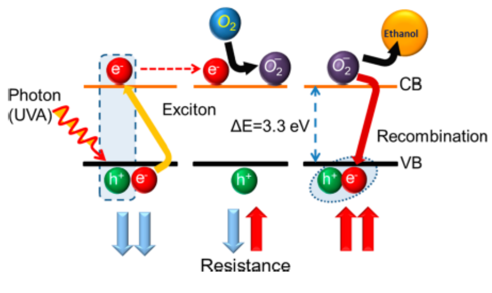

Figure 9. Schematic diagram for ethanol-sensing mechanisms that can be used to explain the resistance increase under ethanol ambient at room temperature. There are four stages for sensing mechanism: photoelectron generation, oxygen adsorption, oxygen-ethanol reaction, and free electrons injection back to valence band.

DOI: 10.1021/acsami.5b07491
ACS Appl. Mater. Interfaces 2015, 7, 24177−24184

this stage, sensor resistance increases because of loss of photoelectrons in the conduction band and results in moderate resistance compared with that in the first stage. This moderate resistance can be confirmed by the lower resistance of the sensor measured in air (130 kΩ) in comparison with that in nitrogen atmosphere (2.8 kΩ). The third process is related to the ethanol oxidation by superoxide radicals, producing free electrons. Typically, at high temperature, the free electrons inject back to the conduction band because there are still a lot of the available conduction band states, resulting in the decrease in resistance (as seen in Figure 7a). However, at room temperature, there are much fewer available conduction band states compared with the number at high temperature (low probability according to a simple Boltzmann distribution). Therefore, we propose that in the fourth stage the free electrons inject back to the valence band with high probability by recombining with holes in the valence band resulting in the increase in resistance. The resistance increase can be mainly explained by the lack of photoelectrons that return back to the conduction band.

It is worth noting that at high operating temperature the ethanol response is similar to that of the case of with and without UV illumination because the thermally excited electrons dominate in this case. Therefore, ITN-ZnO is an exciting morphology of ZnO that can lead to many new applications because of its novel properties. Because ITN-ZnO can be obtained by the simple and fast synthesis process of MWTO, scaling up to mass production of ITN-ZnO would be easily performed and also increase feasibility for device fabrication at low cost.

The novel properties of ITN-ZnO are beneficial for electronic, photonic, optoelectronic, and sensing applications. ITN-ZnO may provide a means to improve the devices based on ZnO. Here, a UV sensor and a room-temperature gas sensor with improved performance are demonstrated. The UV sensor could be applied in space-based applications because of the superior properties of radiation-damage resistance of ZnO.[29] The gas sensor can raise feasibility for room-temperature gas sensor applications, which is one major limitation in using gas sensors based on metal-oxide semiconductors. In addition, this room-temperature gas sensor could be applied for sensing various gases including hydrogen, which would require a low operating temperature for safety reasons.

4. CONCLUSIONS

ITN-ZnO has been successfully synthesized by using a microwave-assisted thermal oxidation method. With this simple and fast process, ITN-ZnO has been obtained. Moreover, this ITN-ZnO also unexpectedly exhibits superior electrical and gas sensing properties when compared with T-ZnO and P-ZnO. With the advantage of its better transport partway for electron transport to the electrode, a UV sensor and a room-temperature gas sensor with enhanced performance are obtained. Therefore, ITN-ZnO is an attractive morphology of ZnO that can lead to many new applications because of its novel properties.

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Author Contributions

M.T. performed most of the experiments and data analysis. T.C. performed TEM characterization and SADP analysis. Explanation model in Figure 9 was initiated and proposed by N.H. and S.C. All authors discussed the results. M.T. wrote the manuscript with assistance from T.C., N.H., and P.R. Finally, S.C. supervised and revised the writing.

Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

This work was partially supported by Thailand Research Fund (TRF), and Thailand Center of Excellence in Physics (ThEP center). M.T. acknowledges the financial support via scholarship from the Graduate School, Chiang Mai University, Thailand.

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2. **Meechai Thepnurat**, Pipat Ruankham, Surachet Phadunghitidhada, Atcharawon Gardchareon, Duangmanee Wongratanaphisan, and Supab Choopun, "Efficient Charge-Transport UV Sensor Based on Interlinked ZnO Tetrapod Networks, Surface&Coatings Technology," In Press. (IF= 1.998).

Surface & Coatings Technology xxx (2015) xxx–xxx

Contents lists available at ScienceDirect

Surface & Coatings Technology

journal homepage: www.elsevier.com/locate/surfcoat

Efficient charge-transport UV sensor based on interlinked ZnO tetrapod networks

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ARTICLE INFO

Article history:
Received 24 December 2015
Revised 14 March 2016
Accepted in revised form 4 April 2016
Available online xxxx

Keywords:
UV sensor
Zinc oxide (ZnO)
Interlinked ZnO tetrapod networks (ITN-ZnO)
Charge transport
Microwave-assisted thermal oxidation

ABSTRACT

UV sensors based on inter-linked ZnO tetrapod networks (ITN-ZnO) were fabricated, characterized and compared with ones based on ZnO tetrapods (T-ZnO) and ZnO powders (P-ZnO) in terms of morphology-related charge dynamics. Photoluminescence measurement showed that the ITN-ZnO had the highest order of crystallinity among these nanostructures. Moreover, impedance spectroscopy analysis revealed that potential barrier at the grain boundary of ITN-ZnO was relatively lower that of T-ZnO and P-ZnO. This was because the coulombic leg-to-leg linking of ITN-ZnO as confirmed by electron microscope observation. The lower potential barriers significantly promoted transport of UV-generated charge carriers that were required to reach Au electrodes, leading to lower sensor resistance. Based on the achieved results, charge transport mechanisms were also proposed. These sensor characteristics suggested that the ITN-ZnO was greatly applicable for UV sensors and other opto-electronic devices.

1. Introduction

Novel properties of nanomaterials have allowed enormously advantageous utilizations of nanodevices [1–3]. Nanostructured zinc oxide (ZnO) inorganic semiconductors, one of the most promising materials for optoelectronic devices, have provided various beneficial properties such as wide band gap (3.37), large exciton binding energy (60 meV) at room temperature, high electron mobility, ease of synthesis, and morphological diversity [4]. Among various morphologies of ZnO nanostructures, tetrapod-like ZnO (T-ZnO) is one of the interesting structures since it exhibits 3D geometry with four rod-shaped legs connected together at the core center with tetrahedral angles [5,6]. This morphology is applicable for various semiconducting devices. Ultraviolet (UV) sensors, one of the potential applications for environmental and military sensing, based on T-ZnO are able to provide high UV sensitivity with short response time and recovery time. This is because T-ZnO has high surface-to-volume ratio and high light scattering on the surface [7–9].

In the operation processes, the UV-generated charge carries are required to reach the electrodes through the physically-touching legs of T-ZnO network. The charge transfer across the grain boundaries of T-ZnO is usually limited by the potential barrier formed at the interface of nanojunction between legs of T-ZnO [10]. In order to achieve a higher UV responsibility, this potential barrier should be reduced.

In our previous report [11], we have introduced an interlinked tetrapod network of ZnO or ITN-ZnO, which is synthesized by a simple and fast microwave-assisted thermal oxidation (MWTO). The ITN-ZnO has tetrapod-like geometry with leg-to-leg linked together using coulombic interaction. The UV sensors constructed from ITN-ZnO have shown superior electrical property in comparison with that constructed from T-ZnO and ZnO powder (P-ZnO).

In this work, we constructed a UV sensor based on ITN-ZnO and characterized it using impedance spectroscopy to get further insight into the charge transfer mechanisms of ITN-ZnO sensor. The obtained results showed impressive charge transfer activities with lower potential barrier at the grain boundary in comparison with the devices based on T-ZnO and P-ZnO. The sensing properties of these devices were also reported.

2. Experiment

2.1. Preparation and characterization of ITN-ZnO

ITN-ZnO and T-ZnO were prepared by microwave-assisted thermal oxidation (MWTO) as previously reported in the literature [11]. A

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http://dx.doi.org/10.1016/j.surfcoat.2016.04.005
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Please cite this article as: M. Thepnurat, et al., Surf. Coat. Technol. (2015), http://dx.doi.org/10.1016/j.surfcoat.2016.04.005

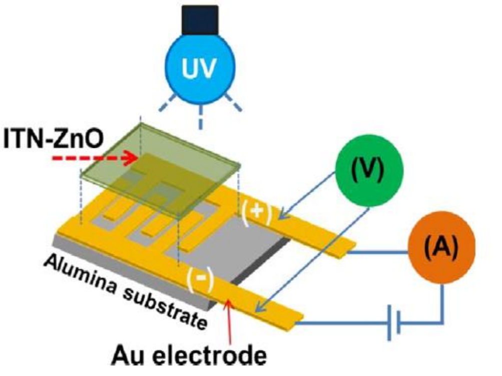

Fig. 1. Schematic illustration of measurement system used for UV-sensing characterization.

house hold microwave oven (700 W, 2.45 GHz) was used as a microwave generator. Zinc (Zn) powder (Asia Pacific Specialty Chemicals Limited, 99.9%) with diameter of <50 μm as a precursor for ITN-ZnO and T-ZnO was loaded in a quartz tube and irradiated with microwave for 30, 60, and 90 s. Aftercooling down to room temperature, the wool-like ZnO products were obtained.

A scanning electron microscope (SEM, Hitachi S-450, 200 keV) and a transmission electron microscope (TEM, JEOL 2010 FEG STEM/TEM) were used to observe morphology of the products. X-ray diffraction (Siemens D-500 with Cu Kα radiation) and Photoluminescence (PL) measurements (RF-5301 PC fluorescence spectrophotometer equipped with a 150 W xenon lamp) were carried out to investigate crystal structure of the products.

2.2. Device fabrication and gas sensing properties

Sensors devices constructed from different morphologies of ZnO (ITN-ZnO, T-ZnO and ZnO powder (P-ZnO; Sigma-Aldrich, average size < 1 μm, purity 99.9%)) were fabricated and compared. Gold interdigital electrodes coated alumina plates were used as substrates for the sensors. Mixtures of various ZnO structure materials (ITN-ZnO, T-ZnO and ZnO powder) with ethanol were screen-printed on the alumina substrates. Measurement of UV sensing properties was performed by biasing the sensors with DC voltage at 1 V and detecting output DC current and resistance. The measurement was operated under UVA ($\lambda = 365$ nm) irradiation from light source with different intensity of 1, 2, 3, 4 and 5 mW/cm^2 at room temperature. A schematic illustration of the measurement is shown in Fig. 1. Impedance spectroscopy was performed to investigate charge dynamics of the devices by using sinusoidal potential amplitude of 20 mV in a frequency range from 1 Hz to 10,000 Hz under UV irradiation.

3. Results and discussion

3.1. Morphology and crystallinity

Fig. 2a and b show optical images of plasma generated during the microwave arcing and the wool-like ZnO products collected after the arcing. It can be seen clearly that the as-synthesized ZnO products are nanomaterials with two different nano-scale morphologies as reported in our previous work [11]. Coarse white wool-like product in the bottom region is T-ZnO and the upper translucent wool-like is the ITN-ZnO where their legs are connected using coulombic interaction, as seen in Fig. 2d and e. The selected area diffraction pattern (SADP) of the ITN-ZnO in Fig. 2(f) confirmed that these legs grow along <0001> *c*-axis direction.

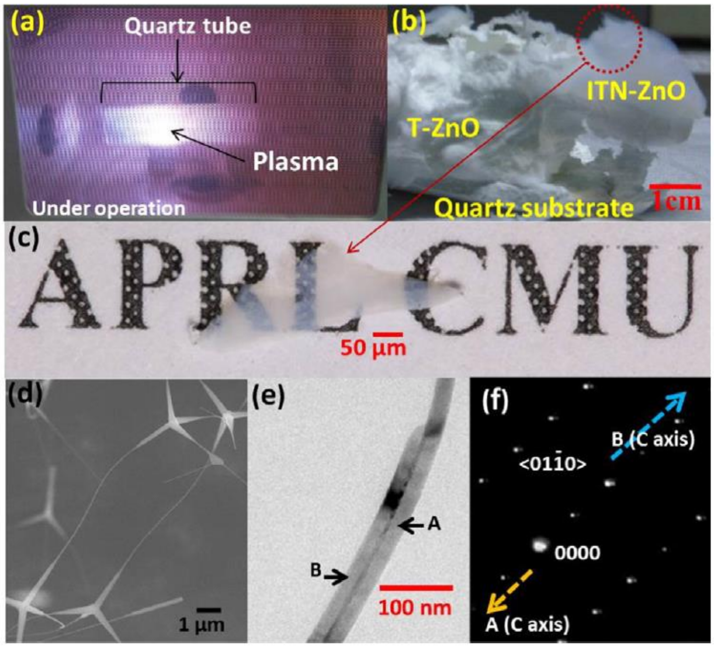

Fig. 2. Optical image of (a) the samples during irradiating by a household microwave oven and (b) the products obtained after MWTO, (c) translucent upper region of ITN-ZnO, (d) SEM image of ITN-ZnO, and (e) bright field TEM image showing the connection between two neighbor legs (A and B) of ITN-ZnO and (f) SADP observed at the connected region of ITN-ZnO.

Please cite this article as: M. Thepnurat, et al., Surf. Coat. Technol. (2015), http://dx.doi.org/10.1016/j.surfcoat.2016.04.005

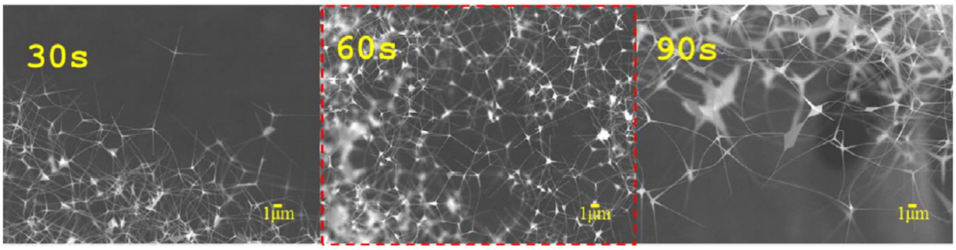

Fig. 3. Morphologies of ZnO products obtained from microwave-assisted thermal oxidation: FE-SEM image of ITN-ZnO was synthesized under microwave oven at 30–90s.

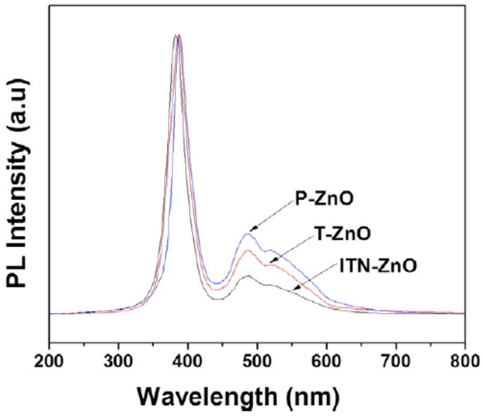

Fig. 4. PL spectra of ITN-ZnO compared with T-ZnO and P-ZnO.

In order to obtain the optimal condition for ITN-ZnO preparation, irradiation with microwave for 30, 60, and 90 s was performed. The FE-SEM images of the ITN-ZnO products are shown in Fig. 3. It is seen that 60 s of microwave irradiation is the optimal condition for obtaining large volume of the ITN-ZnO with homogenous morphology. Irradiating microwave for only 30 s provides smaller amount of ITN-ZnO. Longer irradiation up to 90 s gives inhomogeneous morphology. Therefore, the sample irradiated with microwave for 60 s was further investigated and selected for UV sensor fabrication.

In addition, the normalized PL spectra of all samples (Fig. 4) show two luminescence bands: (1) a narrow band centered at 386 nm corresponding to near band edge emission of ZnO (energy gap of 3.22 eV), and (2) a broad band in the visible region (440–640 nm) referring to the defects in the ZnO crystal including interstitial and vacancy [12–13]. The intensity of the PL peaks in inter visible region is relatively lower than that of the near band edge peaks. Especially, the ITN-ZnO exhibits the lowest luminescence intensity in the visible region, indicating that it has highest order of crystallinity among these samples.

3.2. Electrical properties and UV sensing responses

Impedance measurement was used to characterize the kinetics of electron transfer of sensors constructed from ITN-ZnO, T-ZnO, and P-ZnO. The Nyquist plots of recorded impedance spectra were shown in Figs. 5. The experimental data was fitted using an equivalent circuit model as shown in the inset of Fig. 5 and the extracted parameters were summarized in Table. 1.

Normally, impedance spectra of sensors exhibit three semicircles at high, medium and low frequency regions. However, in this work, only one semicircle at medium frequency was observed. As seen in Fig. 5, the diameters of the arcs are definitely different. The largest one was obtained from the P-ZnO sensor. For the extracted parameter, the R_1 resistance in the equivalent circuit is related to charge transport resistance of the materials. The charge transfer resistance (R_{CT}) and space-charge layer capacitance (C) refer to the charge transfer at the ZnO/ZnO boundary and the ZnO/Au interface. It is seen from Table 1 that the R_1 of all samples are comparable but the R_{CT} of ITN-ZnO is significantly lower than that of the T-ZnO and P-ZnO devices. Especially, under UV irradiation, the R_{CT} decreases since more charge carriers are generated, increasing conductivity of the materials. The lowest R_{CT} and C of ITN-ZnO sensor is probably attributed to the coulombic connection between legs of ITN-ZnO, which is able to reduce potential barrier at the boundary. In comparison with the case of P-ZnO and T-ZnO devices, charge transfer of the ITN-ZnO device is more efficient.

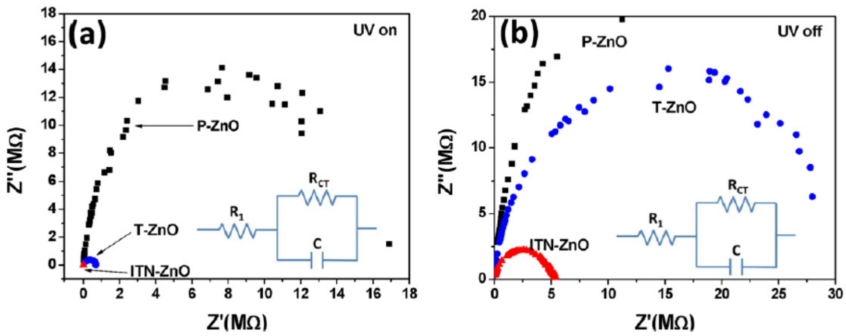

Fig. 5. Nyquist plots of the UV sensors fabricated from P-ZnO, T-ZnO and ITN-ZnO under (a) UV irradiation and (b) dark condition.

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Table 1
Electrical properties of UV sensors with different types of structure.

Type of structure	R_1(kΩ) UV off (on)	R_{CT}(kΩ) UV off (on)	C (pf) UV off (on)
P-ZnO	29(21)	56,000(18,000)	84(60)
T-ZnO	34(16)	30,000(600)	72(58)
ITN-ZnO	54(2.8)	4900(3)	61(21)

The reversible switching resistance of electrical current through the devices for different ZnO morphologies is shown in Fig. 6(a). When the UV radiation is switched on/off every 200 s at a constant bias voltage of 5 V, ITN ZnO exhibits the on/off resistance ratio with the highest value of about 7400 compared with ZnO tetrapod and ZnO powder for about 2.6 and 0.3, respectively. In order to investigate the sensing properties of the UV sensors, typical responses were collected under various UV intensities (1–5 W/cm^2) and the results are shown in Fig. 6(b). It is seen that all sensors showed higher resistance ratio at higher UV intensity. The UV sensitivity of samples increased as a function of UV intensity, as seen in Fig. 6(b). The ITN-ZnO sensor has different sensor properties with high and rapid response of UV. This represents that ITN-ZnO show superior UV detecting properties clearly different from ZnO tetrapod and powder. The photo-dark resistance ratio of ITN-ZnO also exhibits higher value of 7400 than that of the previous report (4500) [10]. These results suggest that the ITN-ZnO has a potential for UV sensor application.

To understand the distinguishable optical and electrical properties of ITN-ZnO from P-ZnO or even a free standing ZnO tetrapod, schematic drawings presented in Fig. 7(a) are used to explain the current increase under UV irradiation for UV sensing mechanisms. The response variation for sensors based on ZnO to UV radiation at room temperature can be explained by the energy-band theory of semiconductor, photoelectric effect, and superoxide. Since ZnO has energy gap of about 3.3 eV, an electron (e^-) in the valence band (VB) can be excited by UVA light (3.10–3.94 eV) to the conduction band (CB), leaving a hole (h^+) in the VB. Then, the UVA generated electrons transport across the grain boundaries of nanostructures in order to reach the Au electrode for charge collection. The increase in conduction band electrons causes the reduction of resistance for ZnO-irradiated with UVA light in the air as confirmed by the results showing in Fig. 6a.

Regarding to the transduction/conduction mechanisms in these devices, it is seen that the photo-dark resistance ratio of the ITN-ZnO sensor is significantly higher than that of the T-ZnO and P-ZnO devices. This is caused by the reduction of potential barrier at the grain boundary of ITN-ZnO as confirmed by impedance analysis. The UV-generated charge carriers are able to transfer across the grain boundaries with fewer potential barriers and are able to reach the Au electrode, showing the dramatic drop in sensor resistivity under UV irradiation in comparison with the other devices. Therefore, the leg-to-leg linking with coulombic interaction of ITN-ZnO significantly reduces the potential barrier at the grain boundary and provides the efficient charge transport of the device.

4. Conclusion

UV-sensors based on nanostructures were constructed and characterized. The ITN-ZnO sensor demonstrated the lowest sensor resistance in comparison with the T-ZnO and P-ZnO ones. The main reason behind the dramatic decrease in sensor resistance was the efficient charge

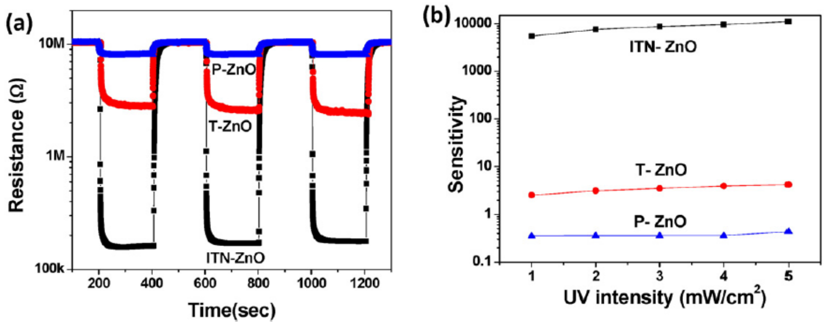

Fig. 6. Resistance characteristics of the sensor; (a) reversible switching of electrical resistance at 5 V biasing voltage with 200 s periodic illumination of UV light of 3 mW/cm^2 for ITN-ZnO, T-ZnO and P-ZnO devices; and (b) the photo-dark resistance ratio (Sensitivity: (R_{uv} − R_{dark})/R_{dark}) × 100) of ITN-ZnO, T-ZnO and P-ZnO sensors under illumination by a 365 nm of UV intensity at a power of 1, 2, 3, 4, and 5 W/cm^2.

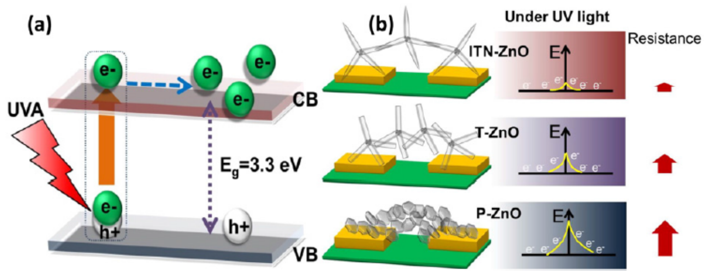

Fig. 7. Mechanism interpretation for the UV sensor; (a) schematic diagram explaining the current increase under UV irradiation; and (b) transduction/conduction mechanism in three ZnO nanostructures (ITN-ZnO, T-ZnO and P-ZnO).

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transfer with low potential barriers at the grain boundary. These results suggested that the ITN-ZnO with coulombic leg-to-leg linking is applicable for efficient UV sensor and other optoelectronic devices.

Acknowledgments

Meechai Thepnurat would like to acknowledge the financial support via scholarship from Graduate School, Chiang Mai University.

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Please cite this article as: M. Thepnurat, et al., Surf. Coat. Technol. (2015), http://dx.doi.org/10.1016/j.surfcoat.2016.04.005

LIST OF PATENTS

1. **มีชัย เทพนุรัตน์**, การะเกด แก้วใหญ่, อัจฉราวรรณ กาศเจริญ, สุภาพ ชูพันธ์, ดวงมณี ว่องรัตนะไพศาล, สุรเชษฐ์ ผดุงธิติธาดา, “กระบวนการเตรียมโลหะและโลหะออกไซด์ระดับนาโนเมตรด้วยวิธีเทอร์มอร์ออกซิเดชันร่วมกับคลื่นแม่เหล็กไฟฟ้า,” เลขที่คำขอ 1401001279, 10 มีนาคม 2557

สำเนา

แบบสป/สผ/อสป/001-ก

คำขอรับสิทธิบัตร/อนุสิทธิบัตร ☑ การประดิษฐ์ ☐ การออกแบบผลิตภัณฑ์ ☐ อนุสิทธิบัตร ข้าพเจ้าผู้ลงลายมือชื่อในคำขอรับสิทธิบัตร/อนุสิทธิบัตรนี้ ขอรับสิทธิบัตร/~~อนุสิทธิบัตร~~ ตามพระราชบัญญัติสิทธิบัตร พ.ศ 2522 แก้ไขเพิ่มเติมโดยพระราชบัญญัติสิทธิบัตร (ฉบับที่ 2) พ.ศ 2535 และพระราชบัญญัติสิทธิบัตร (ฉบับที่ 3) พ.ศ 2542	**สำหรับเจ้าหน้าที่** วันรับคำขอ 10 มี.ค. 2557 — เลขที่คำขอ 1401001279 วันยื่นคำขอ 28 ก.พ 2557 สัญลักษณ์จำแนกการประดิษฐ์ระหว่างประเทศ ใช้กับแบบผลิตภัณฑ์ ประเภทผลิตภัณฑ์ วันประกาศโฆษณา — เลขที่ประกาศโฆษณา วันออกสิทธิบัตร/อนุสิทธิบัตร — เลขที่สิทธิบัตร/อนุสิทธิบัตร ลายมือชื่อเจ้าหน้าที่

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3.ผู้ขอรับสิทธิบัตร/~~อนุสิทธิบัตร~~ และที่อยู่ (เลขที่ ถนน ประเทศ) มหาวิทยาลัยเชียงใหม่ ตั้งอยู่เลขที่ 239 ถนนห้วยแก้ว ตำบลสุเทพ อำเภอเมือง จังหวัดเชียงใหม่ 50200 และ สำนักงานกองทุนสนับสนุนการวิจัย ตั้งอยู่เลขที่ 979/17-21 อาคารเอสเอ็มทาวเวอร์ ชั้น 14 ถนนพหลโยธิน แขวงสามเสนใน เขตพญาไท กรุงเทพฯ 10400	3.1 สัญชาติ 3.2 โทรศัพท์ 053-210731-2 3.3 โทรสาร 053-210733 3.4 อีเมล์ -

4.สิทธิในการขอรับสิทธิบัตร/~~อนุสิทธิบัตร~~

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3. นายสุรเชษฐ์ ผดุงธิติธาดา อยู่บ้านเลขที่ 21 หมู่ที่ 9 ตำบลสันทะ อำเภอนาน้อย น่าน 55150
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Publication: Applied Materials

Publisher: American Chemical Society

Date: Nov 1, 2015

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Author: Meechai Thepnurat,Pipat Ruankham,Surachet Phadunghitidhada,Atcharawon Gardchareon,Duangmanee Wongratanaphisan,Supab Choopun

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Billing Address	Meechai Thepnurat 113/222 Sunkampang Chiangmai - - Muang, Thailand 50200 Attn: Meechai Thepnurat
Total	0.00 USD
Terms and Conditions	

CURRICULUM VITAE

Author's Name Mr. Meechai Thepnurat

Date/Year of Birth 9 July 1980

Place of Birth: Lamphun Province, Thailand.

Education

	2012 - 2015	Ph. D. (Applied Physics), Chiang Mai University
	2007 - 2009	M. Sc. (Applied Physics), Chiang Mai University
	1999 - 2003	B.Sc. (Physics), Thaksin University
	1996 - 1999	Rajaprajanugroh 26 School, Lamphun, Thailand.

Training

1. Quality Management System ISO 9001: 2008, (2 week) 2008, Thai Industrial Standards Institute, Bangkok, Thailand.
2. Electron Microscopy Laboratory Course, (5 month) 2008, Department of Physics & Materials Science, Chiang Mai University, Chiang Mai, Thailand.
3. DualBeam Basic Course Quanta 3D, (2 week) 2007, The FEI Application Laboratory, Brno, Czech Republic.
4. FEI Quanta 3D Basic Operating Course, (2 week) 2006, Chiang Mai University, Chiang Mai, Thailand.
5. Nanotechnology Basic Course, Nanotechnology electronic and Bio-Nanotechnology (1 week) 2005, National Nanotechnology Center, Bangkok, Thailand.

Experience

1. Department of Geological Sciences, Faculty of Science, Chiang Mai University, Chiang Mai, Scientists (2009-2011) 2 year.

2. Nanosience and Nanotechnology Center Project, faculty of Science,Chiang Mai University. Chiang Mai, Scientists and Master's Degree (2006-2009), 3 year.

3. Applied Physics laboratory, Department of Physics & Materials, Faculty of Science, Chiang Mai University, Chiang Mai, Assistant Researchers (2004-2006), 2 year.

4. National Metal and Materials Technology Center, National Science and Technology Development Agency, Pathumthani, apprentice student (2002) 3 month.

Patents

•The patent of screening test alcohol meter from breath, Patent number 0501000067, Thailand, December 27, 2004.

Publications

1. Sakultanchareonchai, S., Chomsaeng, N., Thepnarat, M., Kurata, H., Isoda, S., Chairuangsri, T., Nisaratanaporn, E., "The role of boron on grain refinement in sterling silver alloy," Chiang Mai Journal of Science, Volume 39, Issue 2, 2012, Pages 264-275

2. Nikorn Mangkorntong and Meechai Tapnurat, "FIB-เครื่องกลึงสำหรับงานระดับนาโนเมตร," Thai Physical Society journal 2007; 24(3): 15-16.

3. <u>Meechai Tapnurat</u>, Supab Choopun, Atcharawon Gardchareon, Pisith Singjai, Pongsri Mangkorntong, Nikorn Mangkorntong. “Thick Film of Carbon Nanotube Composite for Ethanol Sensor,” CMU. Journal Special Issue on Nanotechnology (2005) Vol. 4(1) 21.