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Contributed Paper

Preparation of MoO_3 Nanowhiskers

Meechai Tapnurat [a], [b], Supab Choopun [a], Pongsri Mangkorntong [a] and Nikorn Mangkorntong* [a]

[a] Department of Physics, Faculty of Science, Chiang Mai University, Chiang Mai, 50200, Thailand

[b] Institute for Science and Technology Research and Development, Chiang Mai University, Chiang Mai, 50200, Thailand

*Author for correspondence, E-mail: nikornm@gmail.com

ABSTRACT

Molybdenum oxide (MoO_3) is a wide bandgap semiconductor which can be used as a gas sensor. In this work, molybdenum oxide nanowhiskers were prepared by vapor transport method. First, molybdenum oxide powder were mixed with tin oxide (SnO_2) powder of the ratio of 0%, 20%, 50%, 80% and 100 % by weight. The mixed powder was grounded and pressed into the form of cylindrical tube. Then, the tube was sintered in air at 550°C for 6 h. From Field Emission Scanning Electron Microscopy (FE-SEM) results, nanowhiskers of molybdenum oxide were observed both inside and outside the tube for 50% SnO_2 mixed sample. A typical nanowhisker size is about 100–300 nm wide, 5–20 μm long and 80-150 nm thick. For 20% and 80% SnO_2 mixed sample, nanowhiskers were observed only inside the tube but none of nanowhiskers were observed for pure MoO_3 and pure SnO_2 sample.

Keywords: molybdenum oxide, MoO_3, vapor transport method, whisker, nanowhisker.

1. INTRODUCTION

MoO_3, a wide bandgap semiconductor (energy gap = 3.2 eV), is considered to be an alternative oxide semiconductor for gas sensor applications. The commonly used semiconductors for gas sensor such as ZnO and SnO_2 have some disadvantages due to their lack of selectivity [1-3]. The gas sensors made from MoO_3 thin films have been intensively studied and the gas sensing properties of these gas sensors toward various gases such as NO_2, CO, NO [1-6] have been investigated in order to search for better performance gas sensors. However, MoO_3 in the form of whiskers or nanobelts is expected to have better performance due to its high surface-to-volume-ratio [1-2,6-8]. Thus, preparation of MoO_3 whiskers or nanobelts is of great interest.

The fabrication of MoO_3 whiskers and MoO_3 nanobelts can be prepared using several techniques. For example, H. C. Zeng and co-workers have prepared molybdenum trioxide platelet-crystals (whiskers) from MoO_3 powder by vapor phase growth [9]. S. Choopun and co-workers have grown MoO_3 whiskers by vapor transport method [1] and X.– L. Li and co-workers have grown MoO_3 nanobelts by solution method. [10].

In this work, we report the preparation of MoO_3 nanowhiskers by a simple vapor transport method.

2. MATERIALS AND METHODS

MoO_3 (99.9% purity Fluka chemical) powder was mixed with 0(pure MoO_3), 20, 50, 80 and 100(pure SnO_2) wt% of SnO_2 (99.9% purity Fluka chemical) powder. It was grounded and pressed into a form of cylindrical tube (radius: r_{inside} = 0.56 mm, $r_{outside}$ = 0.75 mm and length = 3 mm) using a load

of about 2.5 tons. Then, the tube was sintered in air at 600 °C for 6 h. The morphology and composition of the tubes were investigated by Field Emission Scanning Electron Microscopy (FE-SEM).

3. RESULTS AND DISCUSSION

The FE-SEM images of the inside tube of mixed sample with the MoO_3 and SnO_2 ratio of 0-100% are shown in Figure 1. The nanowhiskers can be clearly observed in the samples. Composing of 20, 50 and 80 wt% SnO_2. For pure MoO_3, plate-like microstructures with the plate size of 1-2 micrometer have been observed. For pure SnO_2, none of nanowhiskers has been observed. The obtained nanowhiskers lie both along the surface and out of the surface. It looks like the number of nanowhiskers depends on the content of MoO_3. By increasing the amount of MoO_3, the number of nanowhiskers increased. This indicates that the quantity of

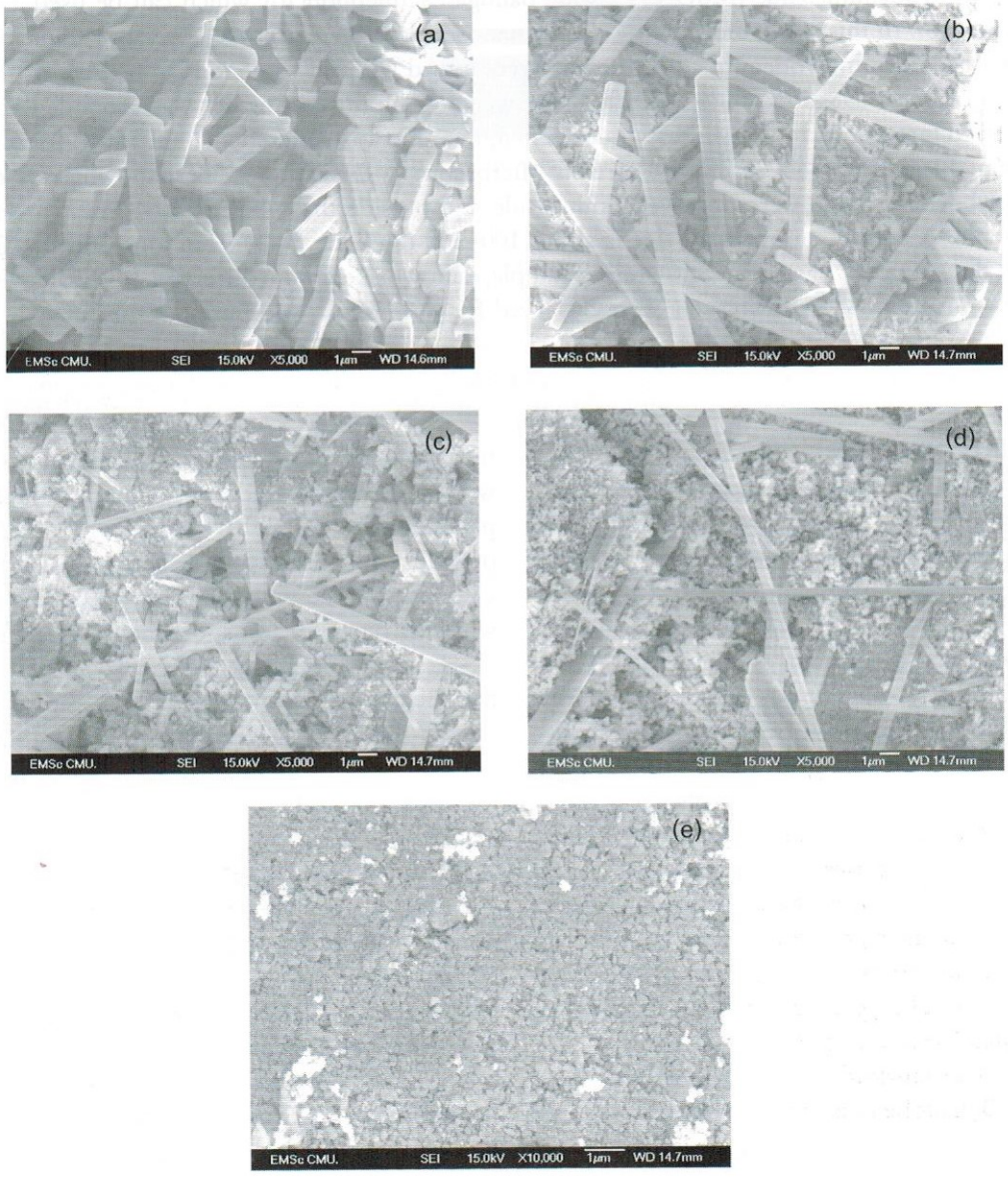

Figure 1. FE-SEM images of the inside tube of mixed sample with the MoO_3 and SnO_2 ratio of (a) pure MoO_3, (b) 20%, (c) 50%, (d) 80%, and (e) pure SnO_2.

MoO_3 plays an important role for the growth of nanowhiskers. However, the reason for this effect is still unclear and it is an ongoing research.

An FE-SEM image of 50 wt% SnO_2 sample at magnification of 30000x is shown in Figure 2 (a). It can be clearly seen that the nanowhiskers have a belt-like structure. Figure 2 (b) shows an FE-SEM image of the same sample at the edge with magnification of 2500x. The obtained nanowhiskers are straight and have some particles on the surface.

At outside tube, we can observe the nanowhiskers for only the 50% SnO_2 sample as shown in Figure 3 (a). The typical morphology of other mixed samples is shown in Figure 3 (b) which indicates that there is no nanowhisker formed.

The sizes of nanowhiskers observed at inside and outside of the tube were summarized in Table 1.The dash means no nanowhisker has been observed. Clearly, the size of nanowhiskers has some relation with the ratio of MoO_3. For 50% SnO_2 sample, the obtained nanowhiskers have the smallest size (100–330 nm wide, 80-150 nm thick and 5–14 μ m long). It is still under investigation to find out why this sample has smallest nanowhiskers. Comparing with other reports, the obtained nanowhiskers in this work have the size in between the size of typical whiskers and nanobelts.

4. CONCLUSIONS

In conclusion, the MoO_3 nanowhiskers can be prepared by simple method of vapor

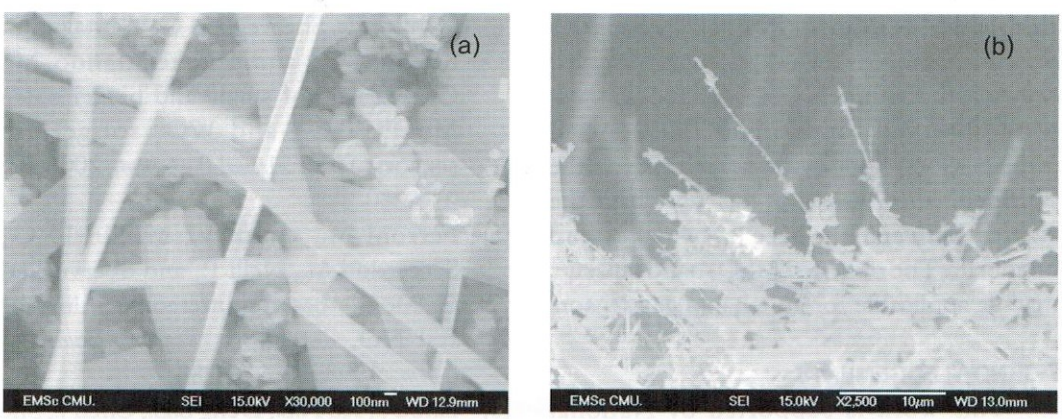

Figure 2. FE-SEM images of the outside tube of mixed sample with the MoO_3 and SnO_2 ratio of 50:50 (a) 30000x (b) 2500x.

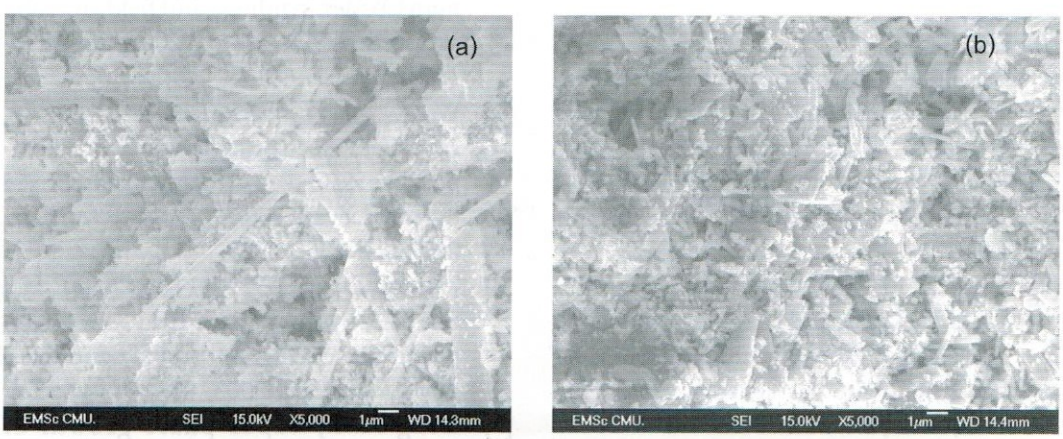

Figure 3. FE-SEM images of the outside tube of mixed sample with the MoO_3 and SnO_2 content of (a) 50% and (b) 20%.

Table 1. Size of nanowhiskers observed at inside and outside the tube of mixed sample with the MoO_3 and SnO_2 ratio of 0-100%.

Material	Inside	Outside	Wide (nm)	Thick (nm)	Long (μm)
MoO_3 – 20%SnO_2	nanowhisker	-	370-1388	259-314	6-12
MoO_3 – 50%SnO_2	nanowhisker	nanowhisker	100-330	80-150	5-14
MoO_3 – 80%SnO_2	nanowhisker	-	250-370	100-148	5-20
MoO_3	-	-	760-2530	250-630	3-7
SnO_2	-	-	-	-	-
MoO_3 whisker[1]	-	-	$(1\text{-}1.5)\times10^5$	$(3\text{-}15)\times10^3$	$(2\text{-}50)\times10^3$
MoO_3 nanobelts[11]	-	-	50-300	10-60	2-4

transport method. The nanowhiskers of MoO_3 were observed both inside and outside the tube for 50% mixed sample with the smallest size (100–330 nm wide, 80-150 nm thick and 5–14 μ m long). For 20% and 80% mixed sample, nanowhiskers were observed only inside the tube. None of nanowhiskers were observed for 0% and 100% mixed sample. Comparing with other reports, the obtained nanowhiskers in this work have the size in between the size of whisker and nanobelts. The obtained MoO_3 nanowhiskers may be applicable for an ethanol gas sensor.

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REFERENCES

[1] Choopun S., Mangkorntong P., Subjareon P., Mangkorntong N., Tabata H., and Kawai T., Orthorhombic molybdenum trioxide whiskers by vapor transport method, *Jpn. J. Appl. Phys.*, 2004; **43**: L91-L93.

[2] Wan Q., Li Q.H., Chen Y.J., Wang T.H., He X.L., Li J.P., and Lin C.L., Fabrication and ethanol sensing characteristics of ZnO nanowire gas sensors, *Appl. Phys. Lett.*, 2004; **84**: 3654-3656.

[3] Law M., Kinal H., Messer B., Kim F., and Yang P., Photochemical sensing of NO_2 with SnO_2 nanoribbon nanosensors at room temperature, *Angew. Chem.*, 2002; **41**: 2405-2407.

[4] Galatsis K., Li Y.X., Wiodarski W., Comini E., Sberveglieri G., Cantalini C., Santucci S., and Passacantando M., Comparison of single and binary oxide MoO_3, TiO_2, and WO_3 sol-gel gas sensors, *Sensor Actuat B - Chem.*, 2002; **83**: 276-280.

[5] Ferroni M., Guidi V., Martinelli G., Saccerdoti M., Nelli P., and Sberveglieri G., MoO_3 –based sputtered thin films for fast NO_2 detection., *Sensor Actuat B - Chem.*, 1998; **48**: 285-288.

[6] Zhou J., Xu N.S., Deng S.Z., Chen J., She J.C., and wang Z.L., Large-area nanowire Arrays of Molybdenum and Molybdenum Oxides: synthesis and field emission properties, *Adv. Mater.*, 2003; **15**: 1835-1839.

[7] Wan Q., Yu K., Wang T.H., and Lin C.L., Low-field electron emission from tetrapod-like ZnO nanostructures synthesized by rapid evaporation, *Appl. Phys. Lett.*, 2003; **83**: 2253-2255.

[8] Zhou J., Deng S.Z., Xu N.S., Chen J., and She J.C., Synthesis and field – emission properties of aligned MoO_3 nanowires, *Appl. Phys. Lett.*, 2003; **83**: 2653-2655.

[9] Zeng H.C., Vapor phase growth of orthorhombic molybdenum trioxide platelet crystals at normal pressure of

purified air, *J. Cryst. Growth.*, 1998; **186**: 393 - 402.

[10] Li X.L., Liu J.F., and Li Y., Low-temperature synthesis of large –scale single – crystal molybdenum trioxide (MoO_3) nanobelts, *Appl. Phys. Lett.*, 2002; **81**: 4832 - 4834.

[11] Li Y.B., Bando Y., Golbery D., and Kurashima K., Field emission from MoO_3 nanobelts, *Appl. Phys. Lett.*, 2002; **81**: 5048-5050.