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Phase Composition and Morphology of ZnO Prepared by the Mild Solution Synthesis Process

------Electronic Supplementary Information (ESI) for "Mild Solution Synthesis of Zinc Oxide Films with Superhydrophobicity and Superhydrophilicity"

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Experimental:

$Zn(NO_3)_2$•$6H_2O$ and hexamethylenetetramine (HMT, $C_6H_{12}N_4$) were used as starting materials. In a typical synthesis, Zn^{2+} and HMT were kept at the same 1:1 molar ratio with 0.1 mM-1.0M [Zn^{2+}]. Commercial borosilicate glass slides were used as substrates. The solution together with a glass plate were sealed in a glass bottle and heated at 95^{o}C for 3 – 192 h. A thin film of ZnO could be produced directly on the substrates. After washing with distilled water three times, the products were vacuum dried at 80^{o}C for 1 h. The phase constitution of the film was determined by X-ray diffraction analysis (XRD, Shimadzu XD-D1) using graphite-monochromized CuKα radiation. Microstructures of films and powders were observed by a scanning electron microscope (SEM,Hitachi S-4100L) and a transmission electron micrograph(TEM, JEOL JEM-2010)

Results:

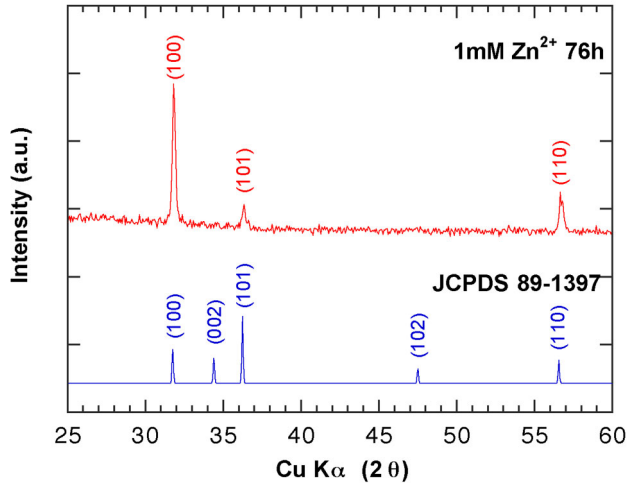

Fig.ESI-1 XRD patterns of ZnO film prepared by heat treatment of 1mM of Zn^{2+}-HMT aqueous solution at 95°C for 76 h, together with that of standard hexagonal ZnO (Joint Committee on Powder Diffraction Standard (JCPDS) card No. 89-1397).

Fig. ESI-1 shows the XRD pattern of ZnO film prepared by the mild solution synthesis process. ZnO film with nanoscrews morphology could be confirmed as hexagonal crystal structure.

Almost no (002) peak of hexagonal structure of nano-screws could be observed, indicating large part of nanoscrews with large aspect ratio laid down on the surface of substrate. This result agreed with that of SEM photographs also (see Fig ESI-2-(c)).

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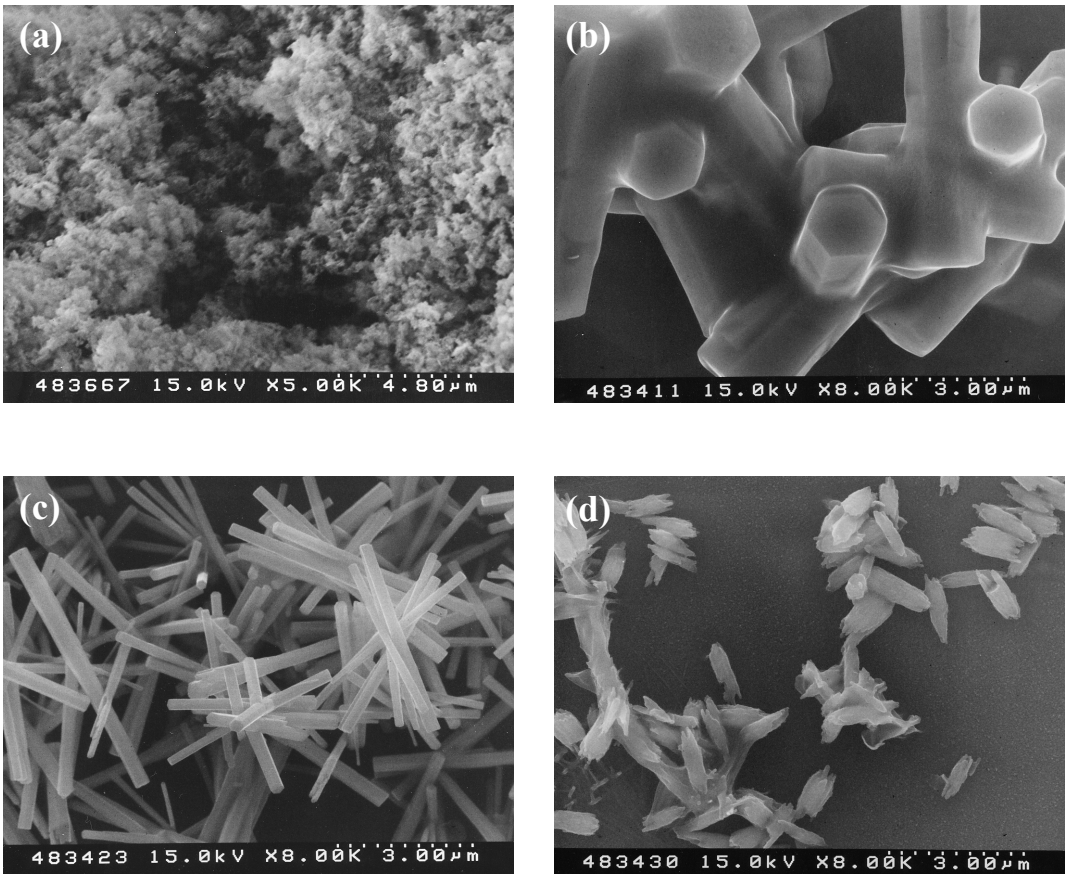

Fig.ESI-2 SEM photographs of ZnO powders prepared by heat treatment of various concentration of Zn^{2+}-HMT aqueous solution at 95°C for 3 h. (a)[Zn^{2+}]=1M; (b)[Zn^{2+}]=0.1M; (c)[Zn^{2+}]=1mM; (d)[Zn^{2+}]=0.1mM.

Fig.ESI-2 shows the SEM photographs of ZnO powders prepared by heat treatment under different concentration of Zn^{2+}-HMT solution. It is clear that rod like structure could not be produced at such high concentration as 1M because of the high nucleus number during treatment. Only agglomerated powders could be obtained. On the other hand, non-complete hexagonal structure could be formed at such low concentration as 0.1mM because of the insufficient zinc ion

supplement. Nanorods structure was obtained in the concentration range of 1mM-0.1M only.

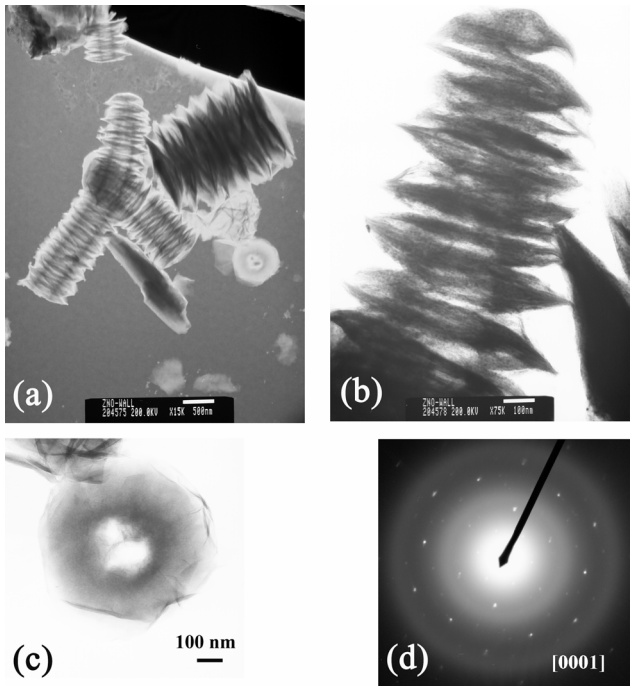

Fig.ESI-3 TEM photographs of the superstructure of nanoscrews.

(a)dark-image of nanoscrew; (b) bright-image of nanoscrew; (c) bright-image of disk; (d) ED pattern of nanodisk of (c).

Fig.ESI-3 shows the TEM photographs of the nanoscrew and nano-disk, together with the ED pattern of the nanodisk. It is obvious that the nano-screw structure can be confirmed. The nano-disk separated from the nanoscrew also showed good crystallinity, although the yield of the nano-disk was not so high at the present stage.

Conclusion

Based on the results of ESI, it might be concluded that the nanorods, nanoscrews and nanodisks possessed well-crystallized structures.