ISSRNS 2010: Extended abstracts / Synchrotron Radiation in Natural Science Vol. 9, No. 1 – 2 (2010)

EXAFS AND XRD STRUCTURE ANALYSIS OF ZnO NANOCLUSTERS FORMED VIA SELECTIVE OXYGENATION OF ORGANOZINC COMPOUNDS

B. Mierzwa [1], K. Zelga [2], W. Bury [2], K. Sokołowski [2], J. Lewiński [1,2], R. Nietubyć [3], and Z. Kaszkur [1*]

[1] *Institute of Physical Chemistry Polish Academy of Sciences, 44/52, Kasprzaka, 01-224 Warszawa, Poland*
[2] *Faculty of Chemistry Warsaw University of Technology, 3, Noakowskiego, 00-664 Warszawa, Poland*
[3] *Andrzej Soltan Institute for Nuclear Studies, 05-400 Świerk/Otwock, Poland*

Keywords: nanocrystals, ZnO, powder diffraction, EXAFS

**) e-mail:* zbig@ichf.edu.pl

Sample of nanocrystalline ZnO was obtained from crystalline organo-zinc compound of chemical formula $C_{34}H_{62}N_2O_{12}Zn_3$ crystalizing in monoclinic crystal structure in $P1_21/c1$ space group and lattice parameters a = 15.652 Å, b = 13.973 Å, c = 21.833 Å and angle β = 114.031°. The cell view is presented in fig.1. The crystals were ground in nitrogen atmosphere what led to decomposition of crystal structure and formation of ZnO wurtzite nanocrystals of size estimated via Scherrer formula as ~2.5 nm. ZnO nanocrystals in literature were observed in the form of nanorods, nanowires, nanoribbons and ~50nm size hollow nanospheres [1,2].

The sample was studied by powder X-ray Diffraction and EXAFS techniques. The results do not fit with regular ZnO crystallite model of certain shape but rather point to models with reduced number of neighbour atoms for small value of neighbour distance (see model 1 and 2 and Figs. 2 and 3 for coordination numbers) as for hollow crystallites.

The EXAFS analysis proved to be capable to distinguish between two models by correct estimation of coor-dination number (Fig. 3) falling down for the model 2 (hollow) to 50-60% of that of the regular crystallite. The models considered in analysis comprised stack of hexagonal wurtzite structure (space group $P6_3mc$) grown in [001] direction of about 2.5nm size (model 1) and the same size crystallite hollowed out (with empty interior) (model 2). The models for illustrative purpose cut by half are presented below (Fig. 4).

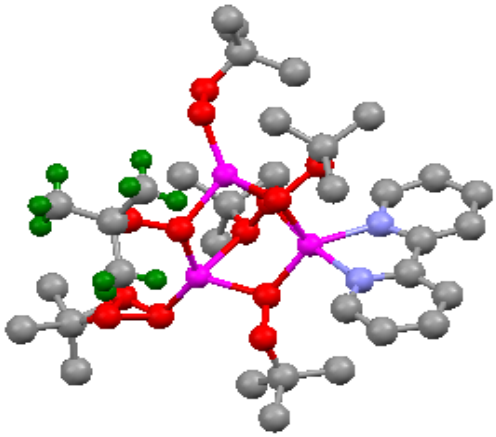


Figure 1. Unit cell view of the crystal serving as a precursor to the studied nanocrystalline sample. Atom colors: Zn atoms- magenta, O in red, C in grey, N in light blue and part of H atoms in green.

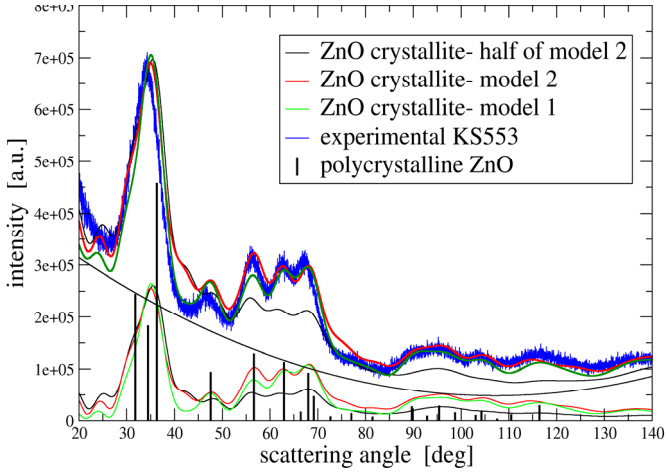


Figure 2. XRD pattern of the nanocrystalline ZnO sample compared to diffractograms of models

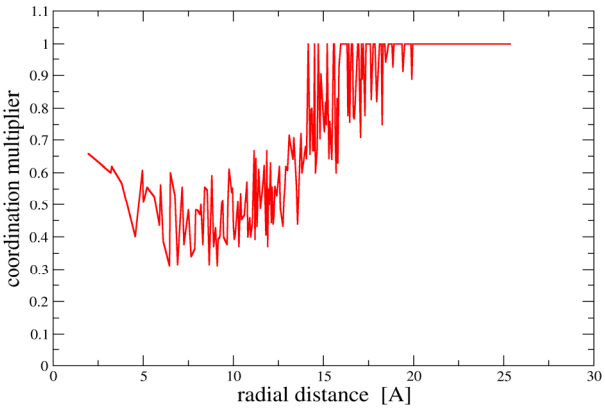
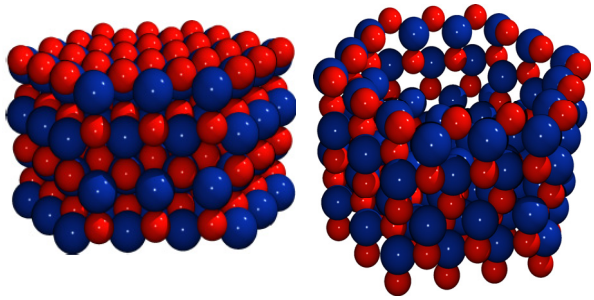


Figure 3. Ratio of Radial Distribution Function (RDF) of model 2 to that of model 1.

The number of neighbours at a distance comparable to the crystallite size is equal for both models. At smaller distances the number of neighbour atoms is for model 2 significantly reduced in comparison to model 1.

Although concluding about the nanocrystal typical morphology can be difficult on the basis of XRD alone, complementing it with EXAFS data may strengthen the conclusions combining arguments based on different physical phenomena.



Half of the model 1: a regular fragment of ZnO lattice. Zn- blue, O- red.

Half of the model 2: a crystallite with empty interior.

Figure 4.

References

[1] Z. Chen, L. Gao, "A new route toward zno hollow spheres by a base-erosion mechanism", *Cryst. Growth Design* **8** (2008) 460–464.

[2] Z. Fan, J.G. Lu, "Zinc oxide nanostructures: synthesis and properties", *J. Nanosci. Nanotechnol.*;**5** (2005) 1561–1673. .