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Morphological and Crystalline Phase Study of ZnO and CuO

Javier E. Morales-Mendoza, Francisco Paraguay-Delgado and G. M. Herrera-Perez
Centro de Investigación en Materiales Avanzados SC (CIMAV), Chihuahua, México

Semiconductor oxides are widely studied materials due to their unique properties, which are used in different applications and engineering. The correlation that exists between microstructure and physical property has always been paramount and important for materials science [1]. Zinc oxide (ZnO) is commonly used either pure or doped with different anionic, cationic, or rare-earth dopant [2]. It is used in applications such as gas sensing, photovoltaic devices, optoelectronics and photocatalysis. Copper oxide (CuO) is a compound with a narrow band-gap and high solar absorption that has been studied attracts great attention in many applications such as anode material, photocatalytic degradation, gas sensor, and others. The change in the microstructure and morphology of these materials allows them to be used in specific applications with adequate physical properties. The surface area of the ZnO and CuO can be improved changing morphologies for example 2D like flake or ribbon shapes. CuO and ZnO are synthesized by the hydrothermal method [3]. Bright-field micrographs and selected area electron diffraction (SAED) patterns were acquired by TEM using HT7700 microscope. The crystalline phase and the microstructure were analyzed by X-ray diffraction (XRD)-Rietveld method. XRD patterns were acquired (30°-70°) using PANalytical X'pertPRO diffractometer. Fullprof suite software [4] was used and single-crystal refinement, it was performed with Thompson-Cox-Hastings pseudo-Voigt profile axial divergence asymmetry function to obtain the apparent average size (D). G-Fourier software was used to visualize crystallite shape using the spherical harmonic method.

XRD-Rietveld analysis and 3D crystallite visualization are shown in Figure 1. ZnO and CuO show hexagonal (P63mc) and monoclinic (C2/c) crystalline phases, respectively. The Chi square (χ^2) value for each profile was carried out until close fit between observed and calculated patterns. ZnO has a smaller average crystallite size than CuO, all refinement information is summarized in Table I.

Table I. Rietveld refinement parameters and apparent average size (D).

Sample	χ^2	R-factors			Hexagonal		Monoclinic			Volume cell (Å^3)	D (nm)
		Rp	Rwp	Rexp	a=b (Å)	c (Å)	a (Å)	b (Å)	c (Å)		
ZnO	1.8	9.7	12.4	9.1	3.251	5.208				47.7	70
CuO	1.7	11.6	7.7	5.9			4.683	3.424	5.130	81.1	208

Figure. 2 shows the bright-field micrographs and SAED patterns indexed using CrysTBox ringGUI software [5]. ZnO shows flake-like morphology (a-b) and hexagonal phase (c). Similarly, CuO shows ribbon or sheet-like morphology (d-e) with monoclinic phase (f). Both phases are agree with the XRD section, present polycrystalline character (c-f) and they have 30-50 nm of thickness. However, the CuO morphology has more thin and elongated shapes.

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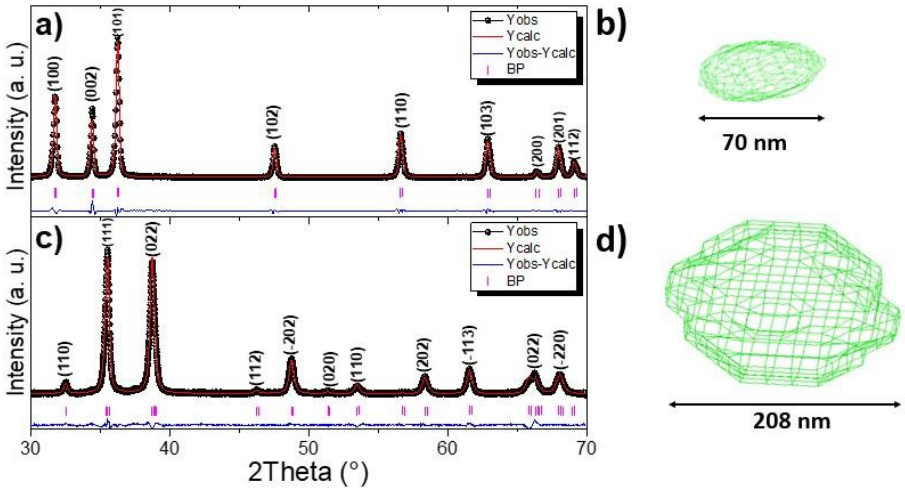

Figure. 1. XRD-Rietveld analysis and 3D crystallite visualization using GFourier software for (a and b) ZnO and, (c-d) for CuO.

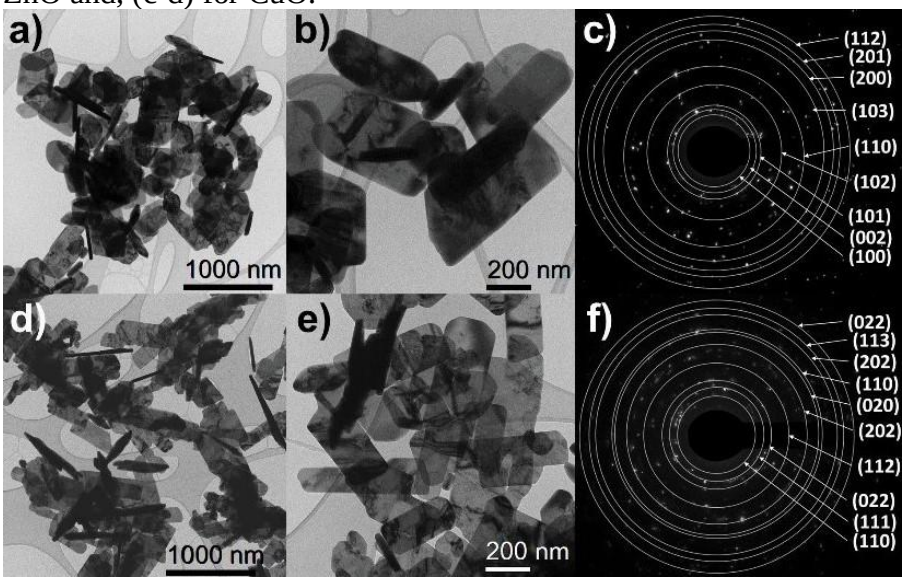

Figure. 2 Bright-field micrographs to monitor the morphology and SAED patterns. (a and b) Flake-like morphology and (c) hexagonal phase for ZnO. (d and e) Sheet-like morphology and (f) monoclinic phase (f) for CuO.

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