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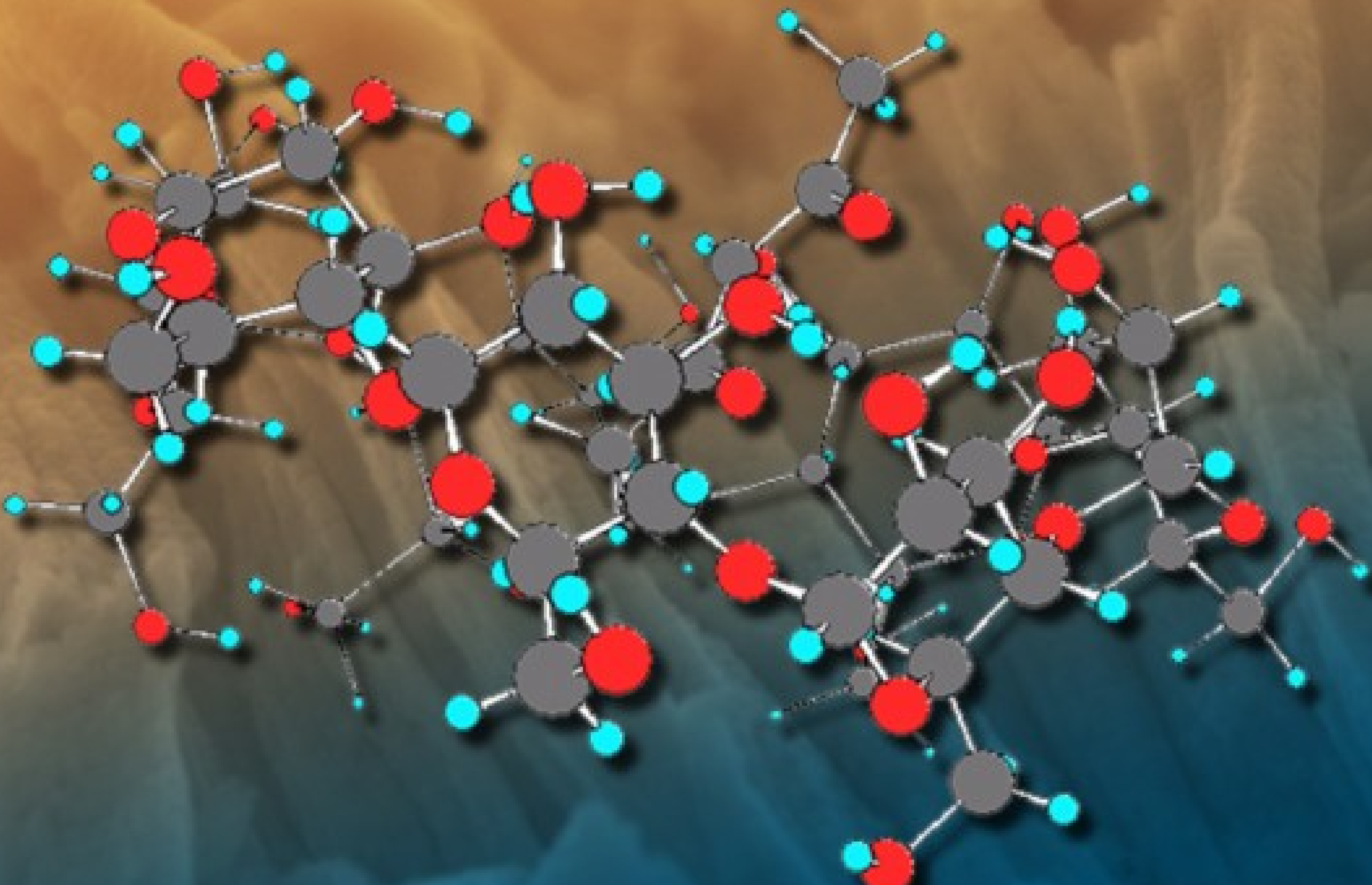

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Synthesis and Characterization of ZnO Nanoparticles as Prepared by Gel-combustion and ZnO Nanomorphologies by Sol-gel

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Abstract: The synthesis of ZnO nanoparticles by a modified gel-combustion method and the preparation of ZnO nanomorphologies using a combination of sol-gel and membrane template method are reported. Optimization of the synthesis parameters and characterization of the materials by X-Ray Diffraction (XRD), Field-Emission Scanning Electron Microscopy (FESEM) and High-Resolution Transmission Electron Microscopy (HRTEM) were performed to evaluate the synthesized materials for potential application in optoelectronic devices and for gas sensors. *Copyright © 2012 IFSA.*

Keywords: Zinc oxide, Nanoparticles, Gel-combustion, Nanotubes, Nano-morphologies, Sol-gel.

1. Introduction

The nanocrystalline semiconductor ZnO deserves a considerable interest since it is a very useful material for a valuable range of applications: LEDs, transparent transistors, lasers, gas sensors, luminescent devices, solar cells, exhibiting also quantum confinement because of its tuned bandgap [1-4]. It is important to control the parameters affecting the synthesis since electrical, optical, optoelectronic, photochemical and catalytic properties, among others, change considerably with the preparation method [5-8] and the morphology and nanocrystalline structure, being the checking of all these factors useful to develop reliable devices. Otherwise, because of the technological relevance of this material, the nanotechnology has exerted an increasing control over the morphologies and characteristics of nanostructures [9]. An intensive research in ZnO tubular nanostructures has been performed to enhance the photon-to-electron efficiency, gas sensing and photonic performance related to the surface area increase and the quantum confinement effect [10]. In consequence, ZnO nanostructures have attracted much attention during the last decades and ZnO nanowires, nanobelts, nanorods and nanotubes have been prepared by different techniques [11-15]. However, controlled synthesis of large scale ZnO elongated nanomorphologies (nanotubes or nanowires) requires further investigation since a rational design and repeated fabrication of nanostructures is still needed. Resistivity and photoconductivity of nanocrystalline ZnO are actually studied at CINSO to be applied in sensors. Some authors of this paper have already proved for resistivity type gas sensors built with SnO_2 that replacing the conventional microcrystalline material with nanocrystalline SnO_2, leads to amazing results since sensors sensitivity increased in 30-37 % and the operation temperature decreased from 350-450 ºC to 180 to 22 ºC [16, 17]. Consequently, the aim of the first part of this work was to synthesize the nanocrystalline ZnO powder by an adapted gel-combustion method used before by the authors [18-20] characterizing the material by XRD and HRTEM and evaluating the quality of prepared ZnO. In order to grow the ZnO surface to enhance the gas absorption in sensors, nanomorphologies were studied and the aim of second part of this work was to synthesize by modified sol-gel template process [21, 22] ZnO nanomorphologies and to characterize them by XRD, FESEM and HRTEM. The evaluation of the parameters affecting the synthesis was considered to apply the material not only for gas sensors but also for optoelectronic devices.

2. Experimental

2.1. ZnO

ZnO exhibits a direct wide band-gap: 3.37 eV [23] and a large exciton binding energy: 60 MeV [24]. ZnO crystallizes in the wurtzite (B4 type) structure at ambient pressure and temperature, enabling to obtain several anisotropic nanomorphologies [25]. It exhibits a hexagonal lattice, belonging to the space group $P6_3mc$, which is characterized by two interconnecting sublattices of Zn^{2+} and O^{2-}, in such a way that each Zn^{2+} is surrounded by a tetrahedra composed of O^{2-} and vice-versa giving rise this tetrahedral coordination to polar symmetry along the hexagonal axis. This polarity is responsible for several ZnO properties, piezoelectricity among them. The spontaneous polarization results also a very important factor in crystal growth, etching and defect generation. The lattice parameters of the hexagonal unit cell are: a = 3.2495 Å and c = 5.2069 Å, being its density: 5.605 $g.cm^{-3}$ [26].

2.2. Synthesis by a Gel-combustion Method and Characterization of Nanocrystalline ZnO

The gel-combustion process to prepare ZnO nanoparticles (used before by the authors to prepare different nanoceramics particles [18-20]) consisted in the dissolution of the raw material (p/a Merck microcrystalline ZnO) in HNO_3 and water (ratio 1:2) in volume solution. The fuel, glycine, was added to the solution and the preparation was heated to form the gel till producing its complete combustion. The resulting material was calcinated to remove organic residues and ashes appearing in case of fuel excess. The oxide was identified and characterized by XRD with a Philips PW 3710 diffractometer using the Cu $K\alpha_1$ radiation. The crystallite size was determined by the broadening of peaks applying the Scherrer equation [27]. HRTEM was performed with a JEM-2100-JEOL microscope enabling to observe the morphology and size distribution of crystallites.

2.3. Synthesis and Characterization of ZnO Nanomorphologies Prepared by the Combination of Sol-gel and Membrane Template Method

ZnO nanomorphologies were synthesized by a combined sol-gel [21, 22]. A solution in methanol with 15 wt% of precursor Zn $(CH_3COO)_2$. $2H_2O$ was prepared under continuous stirring. The concentration of solution resulted ~0.5 M (another diluted concentration: 0.25 M was also essayed). Aging was produced by heating at 60 ºC for 4 h. Templates (polycarbonate membranes from Millipore with pores diameter 0.2 μm or 0.8 μm) were used. Membranes were impregnated with the solution and a pressure difference was applied (by vacuum or overpressure). The resulting material was dried at 50 ºC (using IR lamp for ~ 1 h) and it was calcinated by a slow heating in a temperature range from 550ºC to 750 ºC. At first, the alcoxide (methoxide) was produced, being hydrolized to form the sol and condensed after drying to produce the gel.

Nanomorphologies were characterized by XRD under the same conditions described in 2.2. Morphology of the nanostructured materials was examined with a Philips 515 SEM microscope and, at higher magnification, with a FESEM, Supra 35 VP, Carl Zeiss). HRTEM was used for structural characterization being the analysis performed with a JEM-2100 (JEOL, Tokyo, Japan).

3. Results and Discussion

3.1. Nanocrystalline ZnO

Results of nanocrystalline ZnO specimens are reported in Table 1: oxidant/fuel ratio and crystallite size (nm). All the samples analyzed with XRD exhibited the wurtzite (B4 type) structure, with hexagonal lattice, belonging to the space group $P6_3mc$.

Table 1. Calcination temperature: 600 ºC.

Specimen Batch	[Oxidant/fuel] Ratio (wt %)	Crystallite Size (nm)
1.	1.628	50 ± 4.2
2.	2.170	24 ± 2.0
3.	2.440	25.6 ± 2.2
4.	2.790	33 ± 2.75
5.	3.255	37 ± 3.10
6.	6.510	117 ± 9.75

Fig. 1 shows the XRD spectrum of nanoparticles appearing the peaks (100), (002), (101), (102), (110), (103), (200), (112 and (201) corresponding to the ZnO wurtzite structure (Card Number 36-1451) with a = 3.2495 Å and c = 5.2069 Å. It is a well known phenomenon that the spectrum peaks broaden as the particle size decreases [27] and peaks broadening was used to determine the crystallite size (s) by the Scherrer equation:

$$s = k\,\lambda / \beta \cos\theta,$$

where k is the constant (usually taken as 0.89), λ is the wavelength of the X-ray beam, β is the full width at half maximum height (FWHM) of a given peak (after removal of the instrumental broadening) and θ is the diffracted angle of the peak. Scherrer equation represents the simplest treatment of peak broadening and it can be extended to include the effect of strain broadening of the peaks [28]. Evidently, this method will only yield an average particle size (usually depending on the synthesis conditions) and it will not provide information on the dispersion of size or the quantity of grains agglomeration. Crystallite size for different [oxidant/fuel] ratio is reported in Table 1.

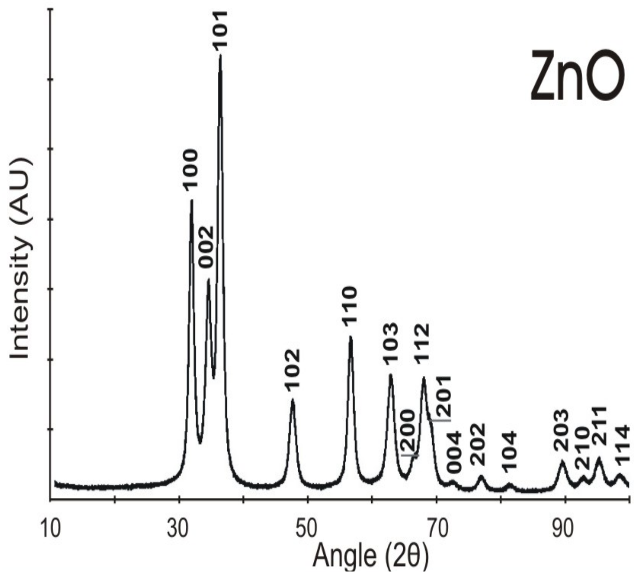

Fig. 1. XRD pattern of nanocrystalline ZnO.

The crystallite size variation with the [oxidant/fuel] ratio is shown in Fig. 2 and a remarkable minimum for crystallite sizes around 25 nm is observed for a low and critical [oxidant/fuel] ratio range between 2.17 and 2.44 wt%.

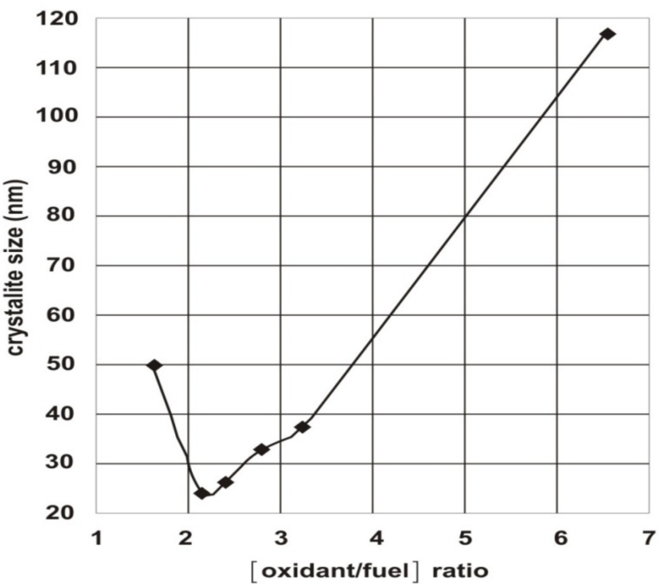

Fig. 2. Crystallites size (nm) vs. [oxidant/fuel] ratio (wt.%).

HRTEM observations enabled to conclude that the increasing calcinations temperature produced the change of crystallites size and shape, thus producing the faceting from spherical to hexagonal shape. Fig. 3a is a HRTEM micrograph of a cluster of ZnO particles showing a mixture of spherical and faceted hexagonal ZnO nanocrystallites. Twins are observed in larger crystallites (shown by an arrow). Size of grains is between 25 and 67 nm in agreement with XRD measurements (Scherrer´s method). Fig. 3b is the electron diffraction corresponding Fig. 3a, showing that particles are well crystallized and in the electron diffraction pattern, the (100), (002), (101), (102) and (110) planes can be indexed as wurtzite structure of ZnO in agreement with XRD spectrum of Fig. 1. The unit cell parameters were calculated [29] by the diffraction data minimizing the sum of the squares of 2θ residuals and the calculated values were: a = 3.3125 Å and c = 5.226 Å with an acceptable agreement with parameters values of the Card Number 36-1451.

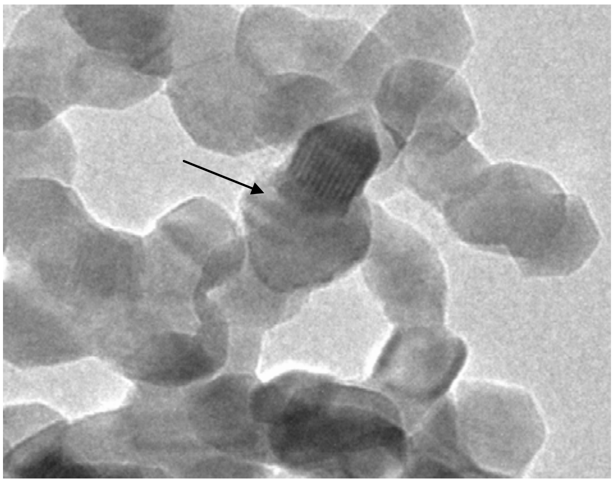

Fig. 3a. HRTEM micrograph of a cluster of ZnO particles showing a mixture of nearly spherical and faceted hexagonal ZnO nanocrystallites. Twins are observed in larger particles (shown by arrow).

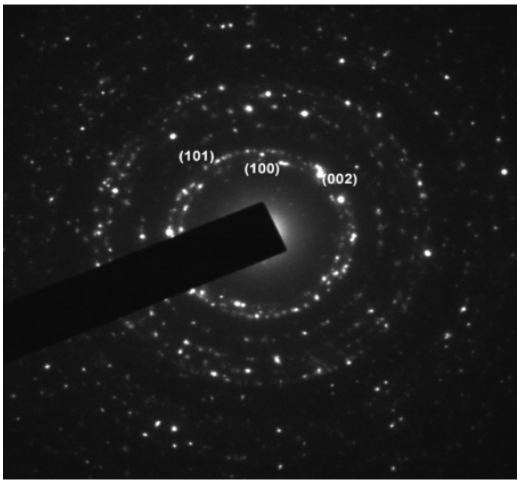

Fig. 3b. Electron diffraction corresponding to the same zone of Fig. 3a.

3.2. ZnO Nanomorphologies

Specimens have been prepared under the following conditions, concentration solution: ~0.25 M or 0.5 M; aging: by heating at 60 ºC for 4 h; pores diameter of the membrane template: 0.2 μm or 0.8 μm; drying: at 50 ºC for ~1 h and calcination at 600 ºC. Optimal results to grow nanotubes were particularly obtained with a solution concentration: 0.25 M and using membranes with the smaller pores size (0.2 μm). Crystallized, faceted, nearly transparent nanotubes appeared exhibiting polyhedric form with a hollow core (Fig. 4a). XRD spectra were in agreement with that of Fig. 1, corresponding to the ZnO wurtzite structure (Card Number 36-1451). SEM observations enabled to prove the appearance of nanotubes with hexagonal cross section. In some areas of the same specimens, also tubes with square, rectangular or rounded cross sections appeared. Faceted nanotubes arranged in bundles were observed too. Tubes dimensions were measured in more than a hundred micrographs finding: mean diameter of tubes resulted from 85 nm to 170 nm, the mean thickness of nanotubes wall was from 8 nm to 20 nm and tubes were from ~800 nm to 2100 nm long. The ZnO nanotubular structure was tightly related to the hexagonal crystallography of this oxide as it is sketched in Fig. 4b. It has been observed a faceted morphology revealing strong evidence that nanotubes grow along the [0001] axis and, consequently, the open extremes of tubes are mostly hexagonal. Several nanotubes have been broken normally or along to the [0001] axis, exhibiting their parts a sheath aspect formed by breakage on the cleavage plane as shown on Fig. 4b. ZnO usually cleaves along the non polar prismatic planes of {1010} though cleavage is also possible along polar surfaces like (0001) or $(000\bar{1})$ [29]. In few areas of the same specimen, the parallel arranged nanotubes presented rectangular or rounded hollow cores, other tubes appeared twisted, open in one extreme or changing their growth direction though maintaining straight sections (Fig. 5) or thinner nanotubes ending in tips appeared agglomerated in bundles (Fig. 6). FESEM enabled to observe the nanomorphologies with higher magnification and it was proved that they were formed by spherical or nearly hexagonal particles.

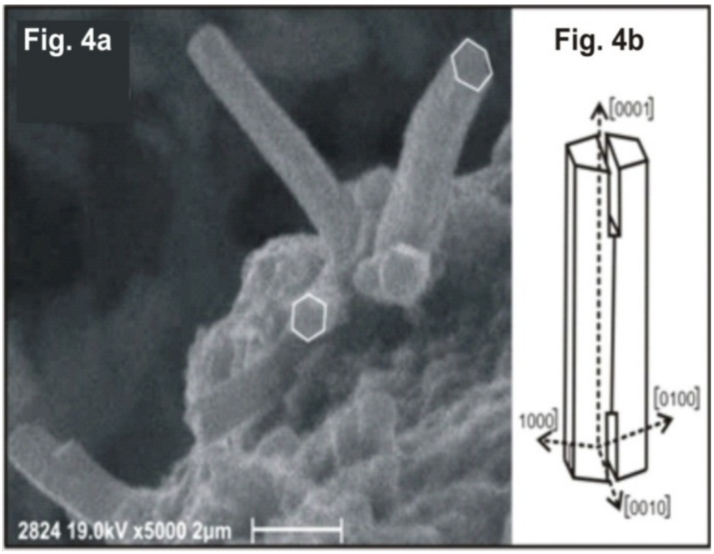

Fig. 4. (a) SEM image showing the nanotubes related to the ZnO hexagonal structures sketched in **Fig. 4b.**

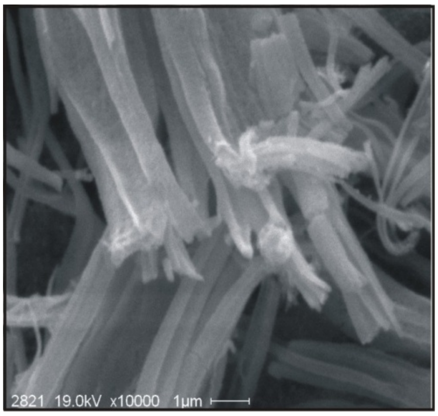

Fig. 5. Parallel ordering of faceted nanotubes (SEM).

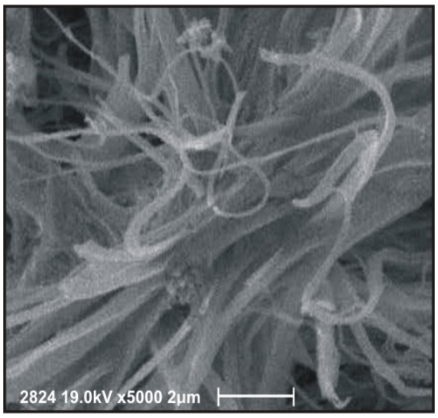

Fig. 6. Bundles of nanotubes and nanobelts (SEM).

Fig. 7 shows with higher magnification (FESEM) flat tubes with hexagonal or rectangular section, arranged similarly to those of Figs. 5 and 6. Growth conditions were the same for nanotubes of Fig. 7 with the exception of the solution concentration (0.5 M). The nearly parallel array of flat tubes (with square or rectangular cross sections) was observed, which surface looked soft and free of agglomerated particles, being probably this aspect due to the smaller diameter size of particles forming the nanomorphologies. Isolated nanomorphologies, tubes or rods ending in a tip on one extreme and formed by particles, were also found and two of them have been chosen for comparison in Figs. 8 and Fig. 9. Both micrographs were obtained with the same magnification on specimen grown from 0.25M and 0.5M solution concentrations, respectively but with same pore diameter of polycarbonate membrane: 0.8 μm. Both, the increasing solution concentration and the major pores size of membrane, usually contribute to increase the crystallites diameter with mean values ranging from ~70 to 100 nm (Fig. 8) to ~90-200 nm (Fig. 9). However, the isolated formation of Figs. 8 and 9 are representative of the structure in which nanotubes exhibit a closed tip on one extreme and on the other the tube is open. It is also interesting to point out that these formations appeared superimposed with sheaths (probably formed as before explained) as shown in Figs. 10 and 11.

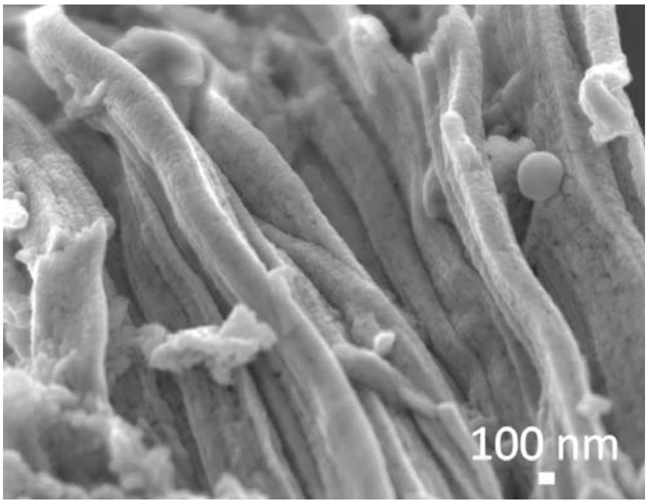

Fig. 7. FESEM micrographs of flat nanotubes arrays. ZnO nanomorphologies were prepared from 0.25 M solution concentration.

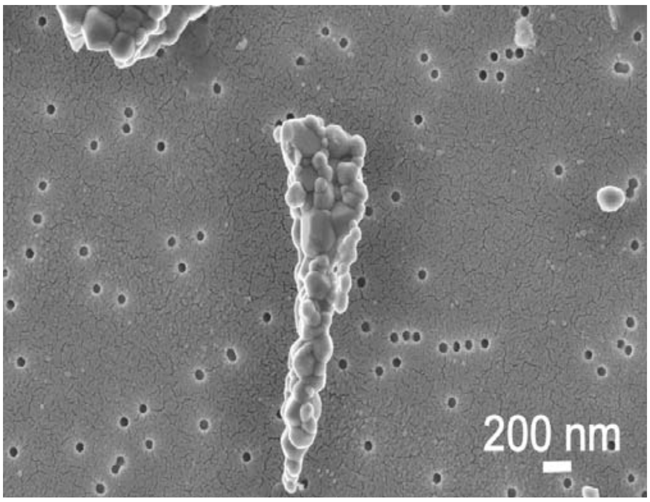

Fig. 8. ZnO nanotube ending in a tip and formed by clusters of nanoparticles (FESEM). Solution concentration: 0.25 M and membrane pores size: 0.8 μm.

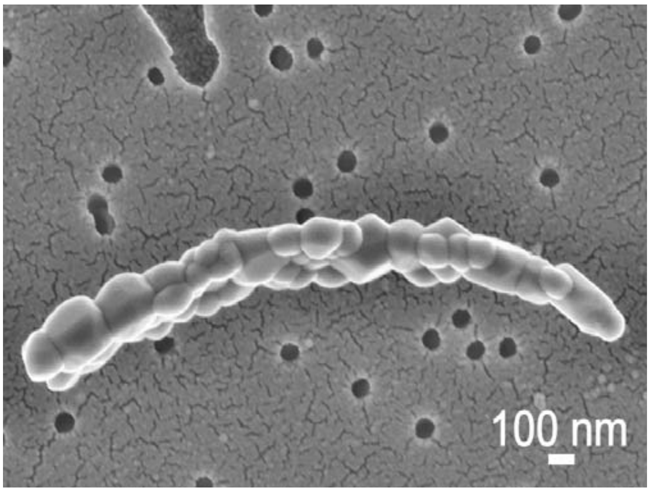

Fig. 9. ZnO nanotube ending in a tip and formed by clusters of nanoparticles (FESEM).
Solution concentration: 0.5 M and membrane pores size: 0.8 µm.

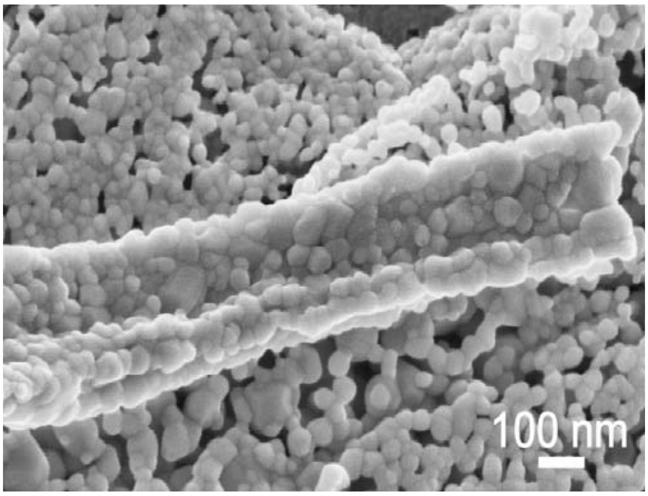

Fig. 10. ZnO sheath formed by clusters of nanoparticles (FESEM).
Solution concentration: 0.25 M, membrane pores size: 0.8 µm.

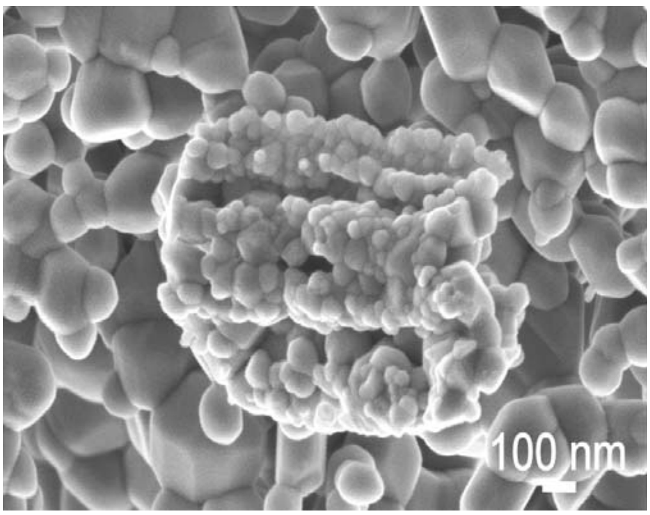

Fig. 11. Solution concentration: 0.5 M, membrane pores size: 0.8 µm (FESEM).

The nanomorphologies resulted irregular, formed by larger particles, showing rough surfaces and thicker walls. In spite of being the formations longer and wider, it was very difficult, even impossible, to measure their length and width because of the interaction of the different found nanomorphologies. In case of specimen grown from a more concentrated solution (0.5 M) and using a pore sized (0.8 μm) membrane, nanomorphologies were scarcely parallel arranged and often forming nanostar structures (Fig. 12). The nanostars were formed by nanorods produced by the stacking of round or hexagonal particles.

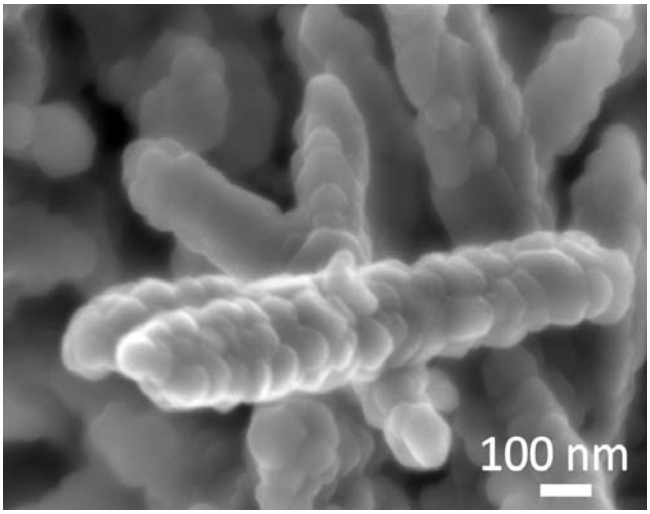

Fig. 12. ZnO nanostar formed by nanorods produced by the stacking of particles. Precursor solution concentration: 0.5 M (FE-SEM), membrane pore size: 0.8 μm.

Fig. 13a: is the HRTEM image of a typical nanotube, formed by stacked particles ending in one side in a tip and broken on the other end, shown in the upper part of figure, exhibiting tubular structure with nearly uniform wall thickness. The walls exhibit a darker contrast because of the thickness of tubes which is homogeneous in this case, though usually the wall thickness looked non homogeneous.

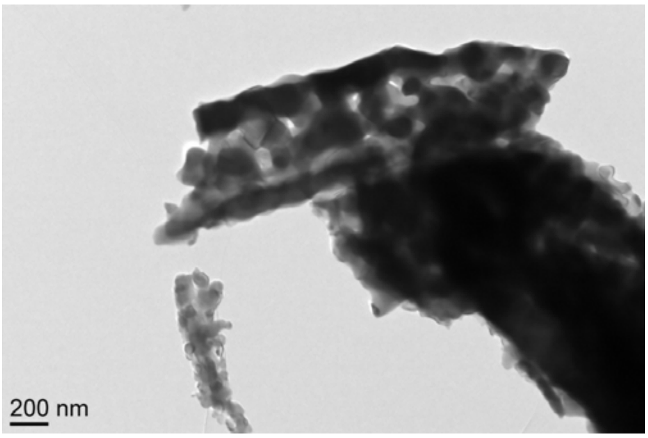

Fig. 13a. HRTEM micrograph of a polycrystalline straight nanotube, ending in a tip on one extreme (upper right side) and being broken on the other extreme.

Nanotubes were generally straight and polycrystalline and they exhibited smooth surfaces. Fig. 13b: is the electron diffraction pattern of the same ZnO nanotube (Fig. 13a) showing the diffraction pattern:

(100), (002), (101), (102), (003) and (100) which corresponds to the ZnO wurtzite structure. On the image of the tube of Fig. 14a is shown the selected area electron diffraction (SAED) which inset is shown in Fig. 14b. The SAED pattern agrees with XRD pattern, being the only difference between both analyses, the relative intensities between the diffraction peaks.

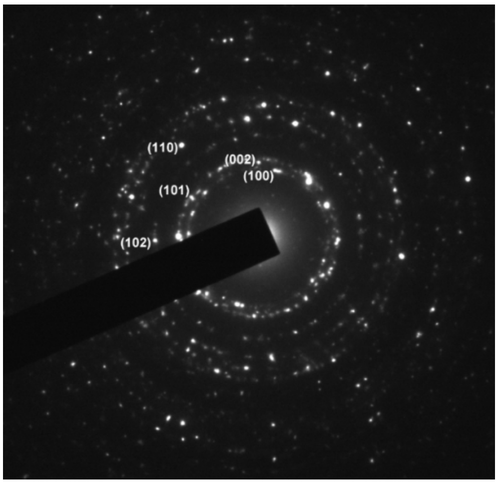

Fig. 13b. Electron diffraction pattern of the ZnO nanotube of Fig. 13a, corresponding to the ZnO wurtzite structure.

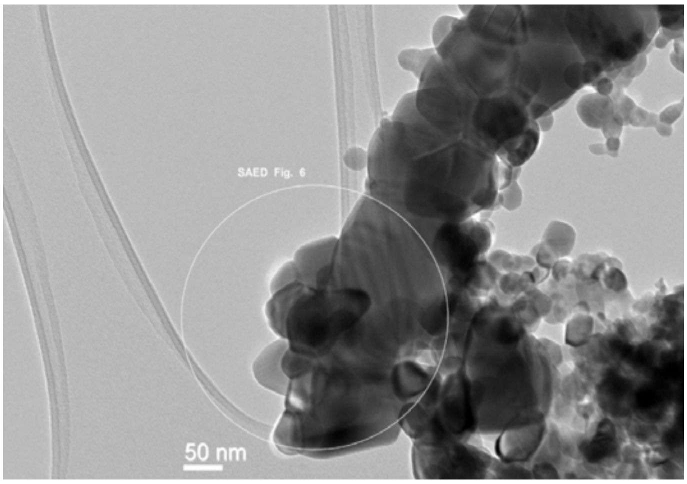

Fig. 14a. HRTEM micrograph of a polycrystalline nanotube.

4. Conclusions

A simple modified gel-combustion method is proposed to prepare ZnO nanoparticles controlling the [oxidant/fuel] ratio to grow crystallites with different diameters. The powder was initially formed by spherical particles evolving to faceted hexagonal platelets by thermal treatment. Characterization was performed by XRD and HRTEM being coincident both analysis indicating the ZnO hexagonal wurtzite structure. A simple modified sol-gel template method was proposed to grow ZnO nanomorphologies, evaluating the parameters affecting the synthesis and characterizing the material with XRD, FESEM and HRTEM it was possible to determine the strict required conditions to grow (nearly aligned) nanotubes with hexagonal cross section. The effect of the concentration solution, aging process, pores diameter of the template membrane, drying process and duration and temperature of calcinations were carefully evaluated, establishing the optimal conditions to the mostly growing of ZnO nanotubes.

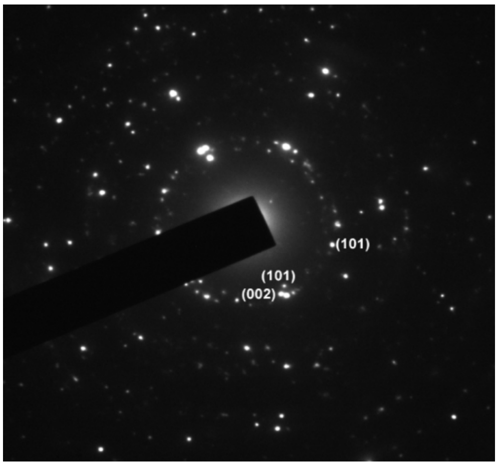

Fig. 14b. SAED pattern of the zone shown in Fig. 14a.

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Guide for Contributors

Aims and Scope

Sensors & Transducers Journal (ISSN 1726-5479) provides an advanced forum for the science and technology of physical, chemical sensors and biosensors. It publishes state-of-the-art reviews, regular research and application specific papers, short notes, letters to Editor and sensors related books reviews as well as academic, practical and commercial information of interest to its readership. Because of it is a peer reviewed international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per year by International Frequency Sensor Association (IFSA). In additional, some special sponsored and conference issues published annually. *Sensors & Transducers Journal* is indexed and abstracted very quickly by Chemical Abstracts, IndexCopernicus Journals Master List, Open J-Gate, Google Scholar, etc. Since 2011 the journal is covered and indexed (including a Scopus, Embase, Engineering Village and Reaxys) in Elsevier products.

Topics Covered

Contributions are invited on all aspects of research, development and application of the science and technology of sensors, transducers and sensor instrumentations. Topics include, but are not restricted to:

- Physical, chemical and biosensors;
- Digital, frequency, period, duty-cycle, time interval, PWM, pulse number output sensors and transducers;
- Theory, principles, effects, design, standardization and modeling;
- Smart sensors and systems;
- Sensor instrumentation;
- Virtual instruments;
- Sensors interfaces, buses and networks;
- Signal processing;
- Frequency (period, duty-cycle)-to-digital converters, ADC;
- Technologies and materials;
- Nanosensors;
- Microsystems;
- Applications.

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