

УДК 538.958.

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## Люмінесценція порошків ZnO, отриманих під дією ультразвуку

## Luminescence of ZnO powders grown in ultrasonic fields

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*Нанорозмірні порошки оксиду цинку синтезовано гідротермальним та сонохімічним методами, використовуючи реакцію ацетату цинку та гідрооксиду натрію в середовищі ізопропілового спирту. Досліджено зміну інтегральної інтенсивності фотолюмінесценції (ФЛ) та напівширини піку у видимому діапазоні, як функції від часу проведення синтезу. Відмічено покращення люмінесцентних властивостей ZnO, отриманого сонохімічним методом в порівнянні із гідротермальним синтезом, при малих тривалостях реакції. Відмічено різний характер зміни напівширини піку ФЛ для різних методів синтезу. При сонохімічному синтезі спостерігається об'єднання окремих наночасток у мікроскопічні агломерати.*

*Ключові слова: оксид цинку, сонохімічний синтез, інтегральна інтенсивність, фотолюмінесценція.*

*Nanosized ZnO powders were synthesis by hydrothermal and sonochemical reaction. Reaction were held in 2-propanol environ using zinc acetate and sodium hydroxide as precursors. All synthesized samples demonstrate strong photoluminescence (PL) in visible region by excitation of UV light. Study of PL shows variations of the integrated (PL) intensity and full width at half maximum of the emission band were found to depend on the reaction time. It is noted that the optical properties of ZnO can be improved by employing sonochemical reactions. Changes of the PL full width at half maximum have different character for both synthesis methods. A possible mechanism is proposed to explain the changes in optical properties. The morphology and structure of ZnO have been characterized using atomic force microscopy (AFM). Single nanoparticles obtained by sonochemical synthesis are grouped into microscopic aggregates.*

*Key Words: zinc oxide, sonohimichnyy synthesis, integrated intensity, photoluminescence.*

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### Introduction

Zinc oxide (ZnO) is an important semiconductor material with a band gap of 3.37 eV and exciton binding energy of 60 meV. High thermal and chemical stability, simple tunability of the optical and electrical properties are widely applicable in opto-electric devices [1–3]. Properties of ZnO strongly depend on the morphology, size and crystal structure [4,5]. Due to these features, zinc oxide is widely used in various areas, such as photocatalysts, chemical, gas and biosensors, solar cells, opto- and microelectronics [6–13]. Furthermore, due to its piezoelectric properties, ZnO is an important

acoustoelectronic material utilizing bulk and surface acoustic waves (filters, resonators, delay lines) [14].

Since the morphology and defect content determine opto-electrical properties of ZnO [5] various methods of producing zinc oxide has been developed. In recent years, nano- and micro-sized ZnO materials were obtained using various methods. For example, sputter deposition was used to get ZnO nanorods placed on the Cu/Ti/Si and having an average diameter of 40 nm [15], thermal evaporation produced needle-like ZnO rods, nanoribbons [16], sol-gel reaction yields nanocrystalline ZnO powders as small as about 25 nm in diameter [17], using different solvents, flower-, snowflake-, prism-,

pricklysphere- and rodlike samples were grown from solutions with different pH [18]. These samples typically exhibit emission of light in the spectral range from 385 to 510 nm. Furthermore, different precursors, such as  $\text{Zn}(\text{OH})_4^{2-}$  [19],  $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  [20], were successfully used in hydrothermal synthesis to produce nanorods with different ratios of length to diameter.

Despite these methods have several properties that are advantageous for growth of ZnO materials, there has been a number of impediments for their widespread adoption. These are high temperatures and pressures, toxic reagents, long reaction times, the need to add stabilizers, etc. Today, the synthesis of nanoscale materials under microwave irradiation and during sonochemical reactions become particularly attractive [19–25].

Sonochemical reaction results from acoustic cavitation, which is accompanied by the formation, growth and collapse of cavitation bubbles. Due to the collapse, extreme physical and chemical conditions are formed inside the bubble. Therefore, chemical cavitation effects strongly depend on the content of cavitating bubbles and, hence, the choice of a proper solvent. For example, to minimize the participation of the solvent vapour and to maximize the vapor temperature in the collapsing bubble, hydrocarbon solvents with high boiling point are usually used [25–27].

In this work, we propose to use zinc acetate and sodium hydroxide as precursors and isopropyl alcohol (2-propanol) as a solvent in sonochemically producing ZnO and show that the resulting ZnO powder exhibits competitive luminescence performance.

### Experimental

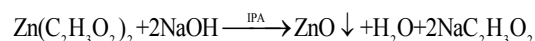
Two routes of chemical reactions were used here, allowing to produce ZnO powders by hydrothermal and sonochemical synthesis. Isopropyl alcohol (2-propanol) was used as a solvent because it has fairly high boiling temperature of 82.4°C. The reaction was achieved using 2-propanol (99.99%), zinc acetate and sodium hydroxide. We first prepared a solution of ZnAc in 2-propanol (solution 1) and sodium hydroxide in 2-propanol (solution 2), which was cooled down to 0°C.

At hydrothermal synthesis, these solutions (1 and 2) were mixed and heated to 65°C. They were then stored at this temperature for more or less prolonged stirring. At time instants (*t*) of 1, 30, 60 and 120 minutes, the samples grown in chemical reactions were taken. The resulting powders (white in color) were

dried and washed in distilled water and methanol. They were then again dissolved in 2-propanol.

For sonochemical synthesis, the above solutions (1 and 2) were mixed at room temperature and placed in sonochemical reactor. The details of our sonochemical bath were given elsewhere [28]. During the sonoactivation of the solution, the temperature raised to about 80°C, which was due to absorption of the ultrasonic power in the solution.

In both chemical and sonochemical reactions, the synthesis of ZnO was achieved in the chemical reaction of the following type:



where,  $\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2$  is the zinc acetate, NaOH is the sodium hydroxide, IPA is the 2-propanol and  $\text{NaC}_2\text{H}_3\text{O}_2$  is the sodium acetate.

Photoluminescence (PL) spectra were excited using an  $\text{N}_2$  laser with the wavelength  $\lambda=337.2$  nm and repetition frequency 100 Hz. PL was dispersed by a DMR-4 monochromator and sensed by a FEU-79 photomultiplier tube.

The powder size and the roughness of particle's surface were observed by Atomic force microscopy (AFM) FemtoScan Online. Scanning was conducted in the contact mode in ambient air using the fpN10S cantilever (radius of curvature of the tip was  $\approx 10$  nm).

### Results and Discussion

In contrast to the chemical route, which results in a micron-sized ZnO powder, sonochemical reactions produce a mixture of  $\mu\text{m}$ - and  $\text{nm}$ -sized grains. The size and shape distributions of the sonochemically synthesized particles are exemplified in Fig. 1. It is found that the particles have nearly spherical shape, and smaller ones are collected into larger agglomerates. Single particles are not more than about 500 nm in diameter. Importantly, the particles exhibit good reproducibility in size,

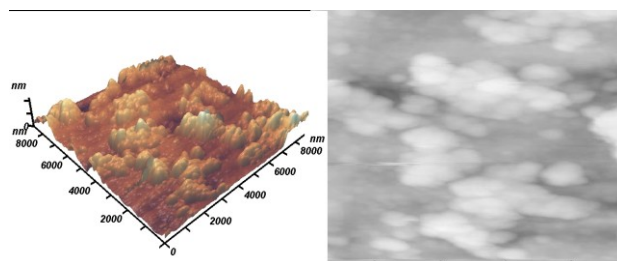


Fig. 1. AFM images (3D-, left, and 2D-, right, pictures) of the sonochemically synthesized nm-sized ZnO powder.

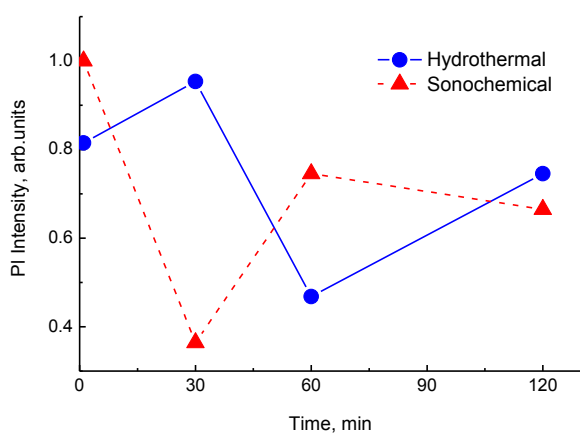


Fig. 2. Integrated intensity of the yellow emission band vs stirring time for hydrothermal (circles, solid lines) and sonochemical (triangles, dashed lines) synthesis.

dependent on the ultrasonic amplitude and irradiation time.

All synthesized samples demonstrate strong PL spanning the region from 450 to 620 nm, which is typical for ZnO [29–33]. The peak position of the dominating yellow emission band observed at about 600–610 nm appears to be nearly unaffected by ultrasonic activations of the reacting mixture. In contrast, the integrated intensity and the full width at half maximum (FWHM) of the PL band are affected by ultrasound.

The integrated intensity of the yellow emission band as a function of the stirring time is displayed in Fig. 2 and appropriate change in the FWHM is shown in Fig. 3. The integrated PL intensity was normalized to its value observed in the sonochemically synthesized sample prepared at  $t=1$  min. It is seen in Figs. 2 and 3 that sonochemical synthesis offers advantageous luminescence performance at stirring times smaller than  $\approx 30$  min for FWHM (triangles in Fig. 3) of the PL band and  $\approx 1$  min for integrated intensity (triangles in Fig. 2) in comparison with the hydrothermal synthesis of ZnO. The integrated intensity exhibits  $\approx 20\%$  increase and the emission band displays  $\approx 30\%$  narrowing in the ultrasonic field.

At  $t \approx 30$  min, a decreased PL intensity and increased FWHM of the emission band are observed in the sonochemically prepared sample. On the other hand, hydrothermal sample prepared at this time demonstrates a greater integrated intensity. Finally, at  $t \geq 60$  min, a slightly increased PL intensity is observed in the sonochemically prepared sample, whereas the FWHM remains almost identical in both fabrication methods.

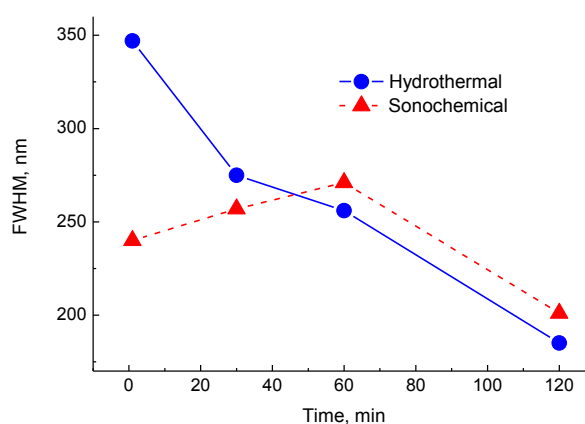


Fig. 3. FWHM of the yellow emission band vs stirring time for hydrothermal (circles, solid lines) and sonochemical (triangles, dashed lines) synthesis.

The observed PL enhancement and FWHM narrowing in ultrasonic fields can tentatively be attributed to higher probability of the radiative transitions and narrower donor band due to sonication of the reacting mixture. The narrower band is thought to arise from improved donor distribution inside the powder grains obtained with the ultrasonic action. The shortened reaction time in the ultrasonic field is explained by an extremely high temperature at the interface between a collapsing bubble and the bulk solution, which is presumed to hasten the hydrolysis and condensation of the zinc acetate. It is therefore most likely that improvements in the ultrasonic-induced mass transport are actually responsible for the luminescence improvements at small stirring times. Prolonged sonication times turn out to increase the density of defects in the grown grain thus deteriorating the luminescence performance.

## Conclusions

In summary, ultrasonic treatment of the solution of zinc acetate and sodium hydroxide with high-intensity ultrasound can bring improvements in luminescence properties of the resulting ZnO powders. Sonochemical synthesis of luminescent ZnO particles at small stirring times is found to be superior to traditional hydrothermal method in the integrated intensity and emission linewidth. One of the reasons for this behavior is the ultrasonic-induced mass transport in the reacting mixture, which improves the donor distribution in the resulting powder. This is due to the fact that acoustic cavitation, leading to the formation, growth, and

implosive collapse of bubbles in the mixture, creates extreme conditions inside the collapsing bubble and serves as the origin of the observed improvements in

the powder grown from the sonicated liquid-solid slurry.

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Надійшла до редколегії 26.06.15