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PECULIARITY OF SEED-LAYER SYNTHESIS AND MORPHOMETRIC CHARACTERISTICS OF ZNO NANORODS

V. Ulianova
Postgraduate student*
E-mail: v.ulianova@gmail.com
A. Orlov
PhD, Associate Professor*
E-mail: a.orlov@kpi.ua
*Department of Microelectronics***
G. Pashkevich
PhD, Senior Research Fellow**
E-mail: pgena@inbox.ru
O. Bogdan
Deputy director**
E-mail: bogdan@ee.ntu-kpi.kiev.ua
Scientific and Research Institute of Applied Electronics*
***National Technical University of Ukraine "Kyiv Polytechnic Institute"
16, Polytechnichna Str., Kyiv, Ukraine, 03056

Представлено оригінальні результати синтезу нанострижнів ZnO за допомогою низькотемпературних методів на підкладках ніобату літію. Вперше продемонстровано «орієнтовані канали», які виникають після відпалу зародкового шару при температурі 400°C і свідчать про початок процесу кристалізації. Проаналізовано вплив властивостей зародкового шару ZnO, сформованого золь-гель методом, на морфометричні характеристики синтезованих гідротермальним методом нанострижнів

Ключові слова: зародковий шар, золь-гель, гідротермальний метод, нанострижні ZnO

Представлены оригинальные результаты синтеза наностержней ZnO с помощью низкотемпературных методов на подложках ниобата лития. Впервые продемонстрированы «ориентированные каналы», которые возникают после отжига зародышевого слоя при температуре 400°C и свидетельствуют о начале процесса кристаллизации. Проанализировано влияние свойств зародышевого слоя ZnO, сформированного золь-гель методом, на морфометрические характеристики синтезированных гидротермальным методом наностержней

Ключевые слова: зародышевый слой, золь-гель, гидротермальный метод, наностержни ZnO

1. Introduction

Zinc oxide (ZnO) has been commonly used in its bulk polycrystalline form for over a hundred years in a wide range of applications: ointments, catalysts, paint pigmentation, piezoelectric transducers and varistors [1].

Nowadays unique ability to form a variety of nanostructures such as nanowires, nanoribbons/nanobelts, nanocombs, nanorings, nanocages, nanocastle, nanofibers etc. have attracted considerable attention to this material for application in a wide range of nanoscale devices. Nanostructured ZnO is a suitable material for electronics, photonics and sensing due to having mechanical, piezoelectric, semiconductor, optical and electrical properties, biocompatibility, nontoxicity, chemical and photochemical stability, high specific surface area, optical transparency, electrochemical activities; it provides strong bonding sites and have large surface-to-volume ratio [2].

2. The overview of recent publications

Among complicated and expensive traditional fabrication techniques such as VLS [3] and MOCVD [4], low-temperature synthesis methods of single-crystalline ZnO nanostructures become more accepted. While electrochemical low-temperature growth [5] allows obtaining well-aligned ZnO nanorods on the conductive films the combined with sol-gel technique hydrothermal method is suitable for nanostructures synthesis almost on any substrates. The hydrothermal method has following advantages: low-cost, ease handling, scalability, opportunity to form various structures subject to process parameters. Sol-gel technique of the seed-layer synthesis is a low-cost process as against ALD [6]. Moreover it is attractive due to ability to conveniently synthesize the films with required properties for a given application. The length, diameter, tilting and crystallinity of the nanorods are highly dependent on the crystalline properties (i.e., grain size) and regularity of the seed-layer.

Existing studies present the structural analysis of nanostructures using scanning electron microscopy method and X-ray analysis [7, 8]. Most of the works are devoted to the analysis of the influence of the hydrothermal synthesis process parameters on the structural properties of the synthesized nanostructures [1, 9, 10], but different authors presented obtaining of unequal nanostructures at the same process parameters. Therefore, it seemed interesting to use others methods such as optical microscopy to analyze the structural features of the seed-layer surface microares which have strong effect on nanosrtuctures formation and find fundamental peculiarity of the synthesis process.

3. Goal and tasks of the research

The main goal of this study was analysis of peculiarities of ZnO seed-layer synthesis by optical microscopy and scanning electron microscopy methods and definition of influence of seed-layer structural features on morphometric characteristics of ZnO nanorods such as length, diameter, tilting, aspect ratio and crystallinity. The tasks of the research included formation of ZnO seed-layer on the lithium niobate (128-XY $LiNbO_3$) substrate by sol-gel method, its characterization by optical microscopy and scanning electron microscopy methods, ZnO nanorods synthesis on as-prepared substrates by hydrothermal methods, its characterization by microscopy methods and X-ray analysis.

4. Materials and methods

Two main steps in ZnO nanostructures growing by hydrothermal method were used. There were (1) preparation of a ZnO seed-layer by sol-gel method for providing crystallization centers and (2) nanostructures array growth in salt solution.

Initially the 128-XY $LiNbO_3$ substrate was cleaned with hydrogen peroxide at 30°C for 30 min. For the first step zinc acetate dihydrate (ZnAc) $Zn(COOCH_3)_2{\cdot}2H_2O$ was used as the starting salt material to prepare ZnO thin films by sol-gel method. The ZnAc was dissolved in isopropanol ($(CH_3)_2CHOH$). Then monoethanolamine (MEA) $HOCH_2CH_2NH_2$ solution was added at room temperature. The concentration of ZnAc was 0,3 mol/l and molar ratio of MEA to ZnAc was kept to 1,0. The mixture was stirred by the magnetic agitator at 65 °C until the clear and homogeneous solution was formed. Prepared sol-gel was cooled to room temperature and filtered with 0,22 μm membrane filter. Film deposition was carried out in air at room temperature. The precursor solution was spin coated at 3000 rpm for 30 s on the substrate. After each coating the obtained film was dried at 100 °C for 30 min at the sintering furnace. The preheat-treatment temperature at 100 °C is required for the complete evaporation of organics and the initiation of formation and crystallization of the ZnO film. After the deposition of the fifth layer, the resulting thin films were annealed at 400 °C in air for 1 h to obtain the stable film with crystallization centers.

Second step was carried out in zinc nitrate based solution. Analytically pure zinc nitrate ($Zn(NO_3)_2{\cdot}6H_2O$) and hexamethylenetetramine ($C_6H_{12}N_4$) were used in equimolar concentration. The concentration of zinc nitrate was 0,05 mol/l. The chemicals were solved in deionized water, resulting in a transparent solution under magnet stirring for 5 min at room temperature. The pH of the growth medium significantly effect on the final structures grown on the substrates [8]. The water solution of sodium hydroxide (NaOH) was added dropwise for obtaining pH=7. The as-pretreated $LiNbO_3$ substrate with formed seed-layer was immersed and suspended in the mixed solution and the growth of ZnO was carried out by heating the reaction solution from room temperature to 95-98 °C and then stayed for 2 h without any stirring at the sintering furnace. The as-grown pattern was rinsed with deionized water for several times and dried in air at 60 °C before characterization.

Optical microscopy (Leitz Ergolux Trinocular Microscope) was used to examine the morphology of the ZnO seed layer. Scanning electron microscopy (Hitachi S4800) was used to define the seeding structure and morphometric characteristics of nanostructures as well. The structural properties of the as-prepared ZnO nanorods were characterized by X-ray diffractometry (Rigaku ULTIMA IV) in asymmetric mode.

5. Characterization of seed-layer and ZnO nanorods

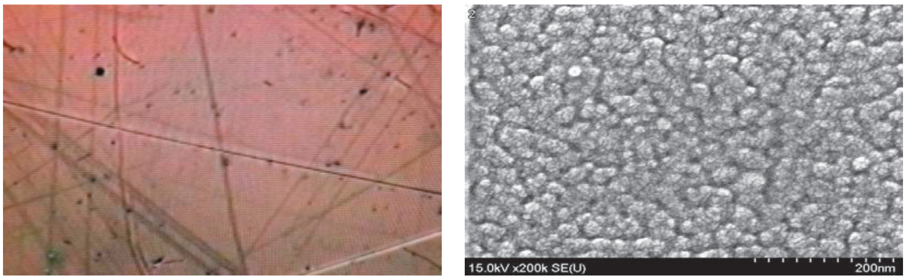
Process parameters of chemical methods materials synthesis, substrate preconditioning and seed-layer growth significantly affect the quality and the shape of the obtained structures.

The Fig. 1, a was obtained by optical microscopy and showed ZnO seed-layer with observed area of 200x270 μm. The “boundaries” or “oriented channels” had appeared on the seed-layer surface after annealing. It reminded “breakdown channels”, which were described in [11] for ZnP_2 and CdP_2 monocrystals after crystallographically oriented electric discharge. Occurrence of such channels could be evidence of crystalline structure formation of the layer. Crystallinity of the seed-layer is a necessary criterion for single-crystal nanorods growing. We carried out the deposition of the ZnO seed-layer on isotropic material substrate (quartz glass) to eliminate the effect of the piezoelectric substrate and obtained the same result. Therefore, formation of “breakdown channels” was caused by the seed-layer structure, not by the process on the interface between the layer and the substrate.

The scanning electron microscope (SEM) images (Fig. 1, b) illustrated high quality regular ZnO seed-layer synthesized by sol-gel method on $LiNbO_3$ substrate. The diameter of the single seeds is about 50 nm.

The characterization of ZnO nanorods was carried out by scanning electron microscopy (Hitachi S4800) and presented on Fig. 2.

The top-view and cross-sectional SEM images of the ZnO nanorods on $LiNbO_3$ substrate were obtained. From observations of the Fig. 2, ZnO nanorods formed on the seed-layer showed hexagonal structure almost along the full length, the diameter was 35-65 nm and the length was about 0,5 μm.

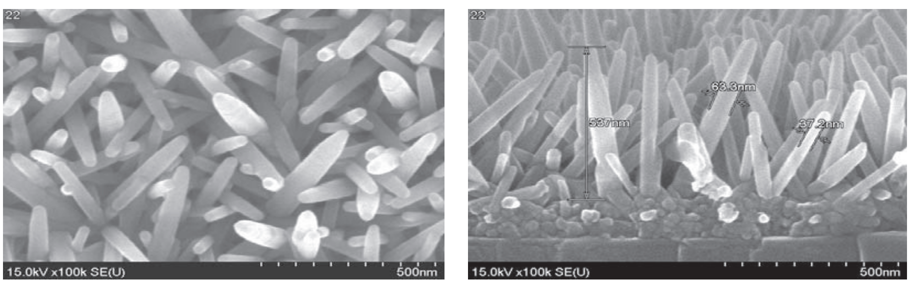


a b

Fig. 1. ZnO seed-layer images observed under: a — optical microscope (observed area of 200x270 μm); b — scanning electron microscope

There were problems in the determination of the distance between adjacent rods due to their tilting, but the space between the bottom parts of the nanorods was 50 nm. Average aspect ratio was equal to 9.3. The ZnO rod structures were regularly situated all over the substrate surface.

From the cross-sectional SEM images (Fig. 2, b) it could be mentioned that seed-layer wasn't smooth. The roughness of the seed-layer could cause tilting of the ZnO nanorods due to the formation of nanorods along the c-axis but not in vertical orientation.



a b

Fig. 2. SEM images of ZnO nanorods grown on $LiNbO_3$ substrate: a — top-view; b — cross-sectional

In comparison with electrodeposition technic, which is described in [5], it should be noticed that deposition of seed-layer has allowed formation of separated single ZnO nanorods but tilted due to the roughness of seed-layer, when in the case of electrodeposition on metal-coated $LiNbO_3$ substrates overgrowth at the roots of ZnO nanorods is occurred. It is expected that vertically oriented ZnO structures can be obtained on smooth seed-layer, formed at adjusted process parameters such as sol-gel concentration and annealing temperature.

The crystalline phase of the nanorods was assessed by conducting XDR measurements (Fig. 3).

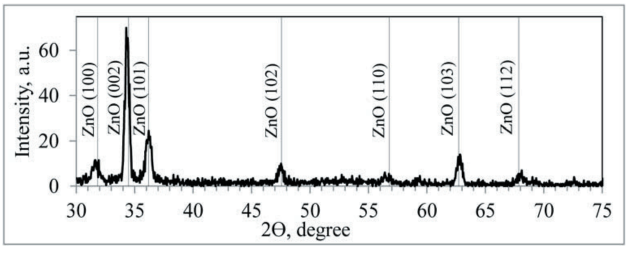


Fig. 3. Obtained in asymmetric mode XRD patterns of ZnO nanorods grown on $LiNbO_3$ substrate

It was carried out in asymmetric mode, which allows obtaining information about the crystalline phase of the surface layer, substrate in this measurement mode makes a minimum contribution to the resulting X-ray spectrum.

It could be seen that synthesized nanostructured film demonstrated diffraction peaks corresponding to the (100), (002), (101), (102), and (112) planes of hexagonal ZnO. Mostly vertical orientation of the nanostructures along the c-axis was shown on the diffraction pattern by the most intensive peaks corresponding to the plane (002). This implied that atoms were arrangement in c-axis which was perpendicular with the substrate and the nanorods had single-crystal structure.

6. Conclusion

The results of separated single zinc oxide nanorods synthesis on the lithium niobate substrate by hydrothermal method were shown.

Optical microscopy and scanning electron microscopy were used to ascertain the peculiarities of seed-layer synthesis and to examine the morphology of the formed structures.

The "oriented channels" caused by particular properties of ZnO after annealing was detected for deposited by sol-gel method ZnO seed-layers for the first time.

The effects of structure and morphology of ZnO seed-layer formed by sol-gel method on the morphometric and structural characteristics of ZnO nanorods was analyzed.

The formation of seed-layer allowed synthesis of separated single ZnO nanorods with average aspect ratio of 9.3, but tilted due to the roughness of seed-layer. It is expected that vertically oriented ZnO structures can be obtained on smooth seed-layer, formed at adjusted process parameters such as sol-gel concentration and annealing temperature.

The obtained new results could be applied for the design of nanoscale devices and its functional or structural units such as sensing element of surface acoustic wave sensors and energy storage cells for energy harvesting. Application of ZnO nanorods will promote quality and accuracy enhancement of sensing devices, manufacturing simplification, and improvement of simulation techniques due to mathematical representation simplicity of the regular-shaped structures.

The proposed method will ensure high repeatability and consistency of production when all process parameters are satisfied.

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ТРАСФІЛДЕРИ ПОТОКІВ ВЕКТОРІВ ІНДУКЦІЇ ЕЛЕКТРОМАГНІТНОГО ПОЛЯ РЕАКТИВНИХ КОМПОНЕНТІВ

В. Г. Кудря
Кандидат технічних наук, доцент
Кафедра інформаційних систем і мереж
Одеська національна академія харчових технологій
вул. Дворянська, 1/3, Одеса, Україна, 65082
E-mail: tasir.onaft@gmail.com

Аналізуються принципи розробки реактивних компонентів електричних кіл на рівні рівнянь Максвелла та встановлюються загальні принципи способів їх конструктивної реалізації. Отримані результати відкривають шлях до створення індуктивностей, ємностей та трансформаторів на засадах використання не лише струму провідності, але і струму зсуву, як похідної потоку вектора електричної індукції

Ключові слова: реактивні компоненти, конденсатор, котушка індуктивності, потік вектора індукції, ємнісний трансформатор

Анализируются принципы разработки реактивных компонентов электрических цепей на уровне уравнений Максвелла и устанавливаются общие принципы способов их конструктивной реализации. Полученные результаты открывают путь к созданию индуктивностей, емкостей и трансформаторов на основе использования не только тока проводимости, но и тока смещения, как производной потока вектора электрической индукции

Ключевые слова: реактивные компоненты, конденсатор, катушка индуктивности, поток вектора индукции, емкостной трансформатор

1. Вступ

В роботі розглядаються засадничі принципи розробки компонентів радіо телевізійних та цифрових електронних засобів. Зокрема йдеться про такі пасивні реактивні компоненти як індуктивність, ємність та трансформатор. Незважаючи на чималу кількість способів реалізації проблеми їх мікромініатюризації