



Parametric study of self-forming ZnO Nanowall network with honeycomb structure by Pulsed Laser Deposition

B. El Zein^a, S. Boulfrad^b, G.E. Jabbour^{b,c,*}, E. Dogheche^{a,**}

^a Institute of Electronics, Microelectronics and Nanotechnology – UMR 8520 CNRS, University Lille Nord de France, Avenue Poincaré, 59652 Villeneuve d'Ascq, Cedex, France

^b Solar and Photovoltaic Engineering Research Center, King Abdullah University of Sciences and Technology (KAUST), Thuwal, Saudi Arabia

^c Renewable Energy Center, University of Nevada, Reno, USA

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ABSTRACT

The successful synthesis of catalyst free zinc oxide (ZnO) Nanowall networks with honeycomb like structure by Pulsed Laser Deposition (PLD) is demonstrated in this paper. The synthesis was conducted directly on Silicon (Si) (1 0 0) and Glass-ITO substrates without the intermediate of metal catalyst, template or chemical etching. Kinetic of growth and effects of gas pressure and substrate temperature were studied by depositing ZnO films on P type Si (1 0 0) substrates with different deposition parameters. The optimized growth parameters were found as: 10 mTorr oxygen pressure, 600 °C substrate temperature, and deposition duration equal or higher than 10 min. X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Photoluminescence (PL) measurements were used to investigate structural, microstructural and optical properties of ZnO Nanowall networks produced. They exhibit a non-uniform size high quality honeycomb structure with low deep level defects.

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1. Introduction

ZnO is a very attractive material for applications in optical devices such as blue-violet and ultraviolet light emitting diodes (LED) laser diodes and solar cells, owing to its direct and wide band gap (~3.3 eV), and large exciton binding energy (~60 meV) [1,2]. Recently, attention has been given to low dimensional ZnO nanostructures like 0 Dimensional such as Quantum Dots (QD) [3], and 1 Dimensional such as nanotubes [4], nanowires [5] and nanobelts [6]. Compared with 1D nanostructure, nanowall is novel 2D nanostructures that hold promise in photo catalysts, biosensors, solar cells, and other fields demanding higher surface to volume ratio, as well as templates for growth of other types of nano materials like nanowires [7,8]. Very recently ZnO Nanowall networks have been employed as a seed layer to initiate and guide the orientation growth of nanowires at defined regions on lattice matching silicon substrate without using any other metal catalyst [9]. It is expected that this new honeycomb design will reduce the amount needed

of semiconductor to be produced and boost efficiency in solar cells [10], gas sensors [11] and photonic devices. Up to now the fabrication methods used were based on Vapor Liquid Solid (VLS) [12] and Metal Organic Chemical Vapor Deposition (MOCVD) [13] with metal catalyst which might be a source of contamination affecting their properties. Pulsed Laser Deposition (PLD) [14] and soft solution [15] were also employed. PLD is considered as a powerful and simple deposition technique, which provides many advantages for materials deposition with interfacial epitaxy, owing to its ability to transfer the stoichiometry from the target to the film. Furthermore, due to the high energetic content of the ejected species, PLD allows low temperature deposition processes [16]. ZnO films have been largely deposited using this technique. In this paper, we present and discuss the process and the optimization of PLD parameters for the growth of catalyst-free ZnO Nanowalls Network (NwAN). The structural, microstructural and optical properties of the produced 2D nanostructures were investigated using XRD, TEM, SEM and PL.

2. Experimental

A Krypton fluoride (KrF) laser system ($\lambda = 248$ nm, 15 ns, 10 Hz and 500 mJ per pulse) was used in this experiment. The laser beam was focused by focal lens on to a ZnO target for around 8 J mm^{-2} . The target was prepared by pressing and sintering ZnO powders

* Corresponding author at: Solar and Photovoltaic Engineering Research Center, King Abdullah University of Sciences and Technology (KAUST), Thuwal, Saudi Arabia. Tel.: +966 775 784 1603

** Corresponding author. Tel.: +33 6 31 14 30 87.

E-mail addresses: Ghassan.jabbour@unr.edu (G.E. Jabbour),

Elhadj.dogheche@univ-valenciennes.fr, elhadj.dogheche@gmail.com (E. Dogheche).

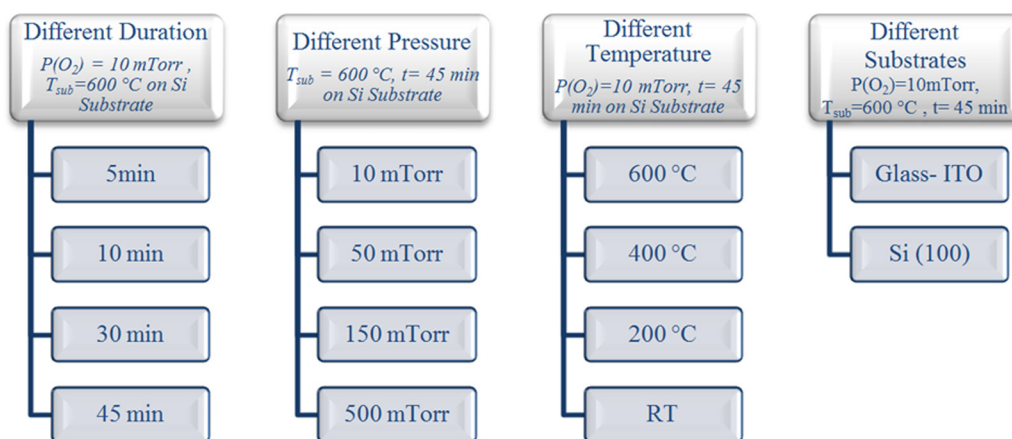


Fig. 1. Samples presented in the paper with their growth parameters.

(99.99% purity from Sigma Aldrich). ZnO films were produced in vacuum chamber pumped down to a base pressure of 10^{-6} Torr. The substrates are (100) Si p-type wafers pre-diced into 1.5 cm^2 pieces and positioned around 9 cm from the target surface. The substrate surface is placed parallel to the target surface. Before starting deposition, the target surface is ablated for a few minutes to clean its surface from any possible environment contaminants. After deposition, the substrate are left to cool down to RT, and then taken out of the PLD chamber, it is noticed that the surface of the substrate is colorful reflecting the thickness of the deposited film. Fig. 1 summarizes the different parameters set during the experiments.

The crystalline structures of the grown NWaNs were characterized using Bruker D8 Discover high resolution XRD with $\text{CuK}\alpha$ and $\lambda = 1.5406\text{ \AA}$ and transmission electron microscopy–FEI–TEM Tecnai. The photoluminescence properties were studied using Raman Lab. The samples were excited using HeCd laser at 325 nm.

3. Results

The growth of ZnO NWaNs with honeycomb structure is affected by different growth parameters such as deposition duration, oxygen pressure and substrate temperature. Results are presented as morphological, structural and optical properties.

3.1. PLD deposition parameters – morphological properties

ZnO NWaNs were deposited at different period of times of 5 min, 7 min, 10 min, 15 min, and 45 min. The morphology of the deposited films is illustrated in Fig. 2.

After 5 min deposition time, ZnO nanoparticles are deposited on the surface of the silicon substrates. The distribution is random and the size varies between 40 nm and 390 nm. It is noticed that at 7 min of deposition, coalescence occurs between the ZnO islands. Starting from 10 min, two-dimensional ZnO NWaNs were grown vertically on the substrates. The pores size ranges from 50 to 140 nm and the walls between the honeycomb exhibits a thickness of ~ 50 nm. The size of the nanowalls is remarkably uniform over the whole substrate. The density of the nanowalls increased with the deposition time. Fig. 3 reveals the cross section tilted view of the ZnO NWaNs, confirming the honeycomb like structure.

ZnO films were deposited at different oxygen pressures of 10 mTorr, 50 mTorr, 150 mTorr and 500 mTorr. The morphology of the deposited films is illustrated in Fig. 4.

It is noticed that the morphology of films obtained at low oxygen pressure is quite different from that obtained at high oxygen pressure Fig. 4b–d. At low pressure 10 mTorr, the film is composed of

ZnO NWaNs with honeycomb structure with non-uniform cell size (Fig. 4a).

At 50 mTorr oxygen pressure, smaller honeycomb can be identified in Fig. 4b morphology. ZnO grains are randomly deposited on the substrate surface of 30–43 nm sizes. On the other hand, at 150 mTorr, small nanopillars with aggregated grains (131–160 nm size) are observed in Fig. 4c. While at higher pressure (500 mTorr), a macro-porous film is grown, and composed of nanopost with pencil shape morphology of 80 nm diameters and spaced by 73 nm.

ZnO NWaNs were deposited at different substrate temperatures of 600 °C, 400 °C, 200 °C and RT. The morphology of the deposited films is illustrated in Fig. 5.

The interlinked ZnO NWaNs were obtained at 600 °C (Fig. 5a) and 400 °C (Fig. 5b). By decreasing the temperature to 200 °C (Fig. 5c) and then to RT (Fig. 5d), dense films were deposited with low surface roughness. By increasing the substrate temperature, the surface roughness increases. Fig. 5b reveals the SEM image of the deposited ZnO NWaNs grown at 400 °C, showing honeycomb structure of 91–136 nm size.

As shown in Fig. 6, the growth of ZnO NWaNs on Glass–ITO substrates was successfully demonstrated at 10 mTorr oxygen pressure, for duration of 45 min and at substrate temperature of 600 °C.

3.2. PLD deposition parameters – structural properties

The XRD spectra of the deposited ZnO films at different deposition times; pressures and substrate temperatures are presented in Fig. 7. Two diffraction peaks were identified in Fig. 7a, in addition to the substrate and substrate holder's peaks. A high intense peak at 34.5° corresponds to the ZnO (002) Bragg Reflection, and a low intensity peak at 66.4° corresponds to the ZnO (200) plane. This suggests c-axis preferred orientation of the NWaNs. Thus, increasing the deposition time leads to thicker films with improved crystallinity.

Fig. 7b reveals the XRD spectra at different oxygen pressure. Many planes are identified (002), (101), (102), (103), (200) for the deposition at high oxygen pressure of 500 mTorr. Hence the crystalline quality has degraded. However some peaks vanish or become weaker when oxygen pressure decreases, the film quality is improved and crystallites become more aligned.

Furthermore, Fig. 7c shows XRD pattern of the ZnO film grown at different substrate temperatures. The strong (002) Peak shows that the ZnO nanostructures were preferentially oriented along the c-axis. Thus, the as grown nanostructures have good epitaxial orientation with the Si Substrates at different temperatures. However, the epitaxial interface might not be critical as the similar results were obtained on Glass/ITO substrates.

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