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Sequential Microwave-Assisted ultra-fast ZnO Nanorod growth on optimized Sol-Gel Seedlayers

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Abstract—recently, the studies on ZnO nanostructure characteristics and their fabrication methods have been increased significantly in order to be able to use its magnificent properties by reaching the desired optimum features. Very high growth rate and high quality growth have always been the center of attention. In this study, a sequential microwave-assisted ZnO nanorod (NR) growth as an ultra-fast, highly efficient method for vertical growth of high quality ZnO NRs on different substrates is proposed. A NRs’ growth rate of 4.2 \( \mu \text{m/h} \) was obtained which led to uniformly distributed, high aspect ratio and vertically-aligned NRs. Furthermore, the influences of spinner’s speed as an important parameter on the quality of ZnO seed and annealing treatment have been studied. The sol-gel casting approach is optimized for the layer. Different seed layers were used to grow the nanostructures and the optimum spinner speed is found to be 2000 rpm. In order to diminish the dependency of the uniformity of ZnO NRs on the quality of seed layers, a remedy is suggested.
I. INTRODUCTION

Zinc Oxide (ZnO) have been vastly studied because of their unique electrical characteristics such as being a wide direct bandgap semiconductor (3.37 eV) with a huge excitonic binding energy of 60 meV at room temperature [1]. Recently, the studies on ZnO nanostructure characteristics and their fabrication methods have been increased significantly in order to obtain optimum features [2–4]. Their potential applications in electronic, optoelectronic and electrochemical devices makes ZnO NRs more and more attractive[5,6].

In order to reach an efficient, simple and cost-effective ZnO nanostructure growth process, solution-based methods have been developed[7]. Hydrothermal ZnO NR growth is one of the most general methods among them in which an aqueous solution containing zinc and oxygen ions as the precursors are used and the growth procedure occurs in rather low temperatures (lower than 100˚C).

The simplicity and effectiveness of this method as well as the possibility of its large-scale production have attracted a lot of attention; yet, one of the obstacles in this method is its very slow rate of nanostructures[8].

One obstacle which limits the growth of the ZnO NRs arises from a high rate of homogeneous nucleation of ZnO nanostructures in the growth solution (formation of floating ZnO nanostructures). Consumption of the precursors and their depletion in the growth solution due to homogeneous nucleation can be high enough to deplete the solution from precursor sources and limit the growth of ZnO NRs on the substrate (heterogeneous nucleation). Xu et al have suggested a method named preferential growth in order to selectively decrease homogeneous nucleation.[8] In this method, ammonium hydroxide and
polyethylenimine (PEI) are added to the growth solution which successfully reduces the homogeneous nucleation and increases the growth of ZnO NRs on the seeded substrates.

Microwave assisted systems have exploited the microwave irradiations to transfer power to the precursors and increase the growth rate. Microwave assisted hydrothermal is one of the most recent methods used in ZnO growth. This method has gained so much attention among other growth approaches due to its high growth rates and high quality of nanostructures as well as being low in cost and safe [17]. Using microwave irradiations, the growth procedure benefits from ultra-fast and uniform solution warm up to the crystallization temperatures.

There are two most common routes used to obtain a ZnO seed layer: sol-gel method and radio frequency (RF) sputtering process [5]. The latter is employed to gain high quality seed layer, nevertheless, it can’t be performed without requisite conditions such as very low pressure. These requirements limit the usage of this technique. In contrast, sol-gel as a versatile, low temperature and low cost method can be used to provide a dense ZnO seed layer.

Pre-growth preparations i.e., the seed layer deposition quality, plays a key role as the cornerstone in the ZnO growth. Many studies have shown that the quality and properties of seed layers can directly affect the growth process. This effect can simply be observed as it determines the quality, size and alignment of ZnO NRs[11,12]. Among seed layer deposition methods, sol-gel is a low cost and fast way to deposit a ZnO seed layer. Although some studies have reported the seed layer dependency of the ZnO NR growth, the efforts to increase the seed layers’ optimum quality as a challenge have yet remained. [11–13].

In this study, a sequential microwave-assisted ZnO NRs growth as an ultra-fast, highly efficient method for vertical growth of ZnO NRs on substrates is proposed. We have also investigated the effect of sol-gel spinner speed on the quality of ZnO seed layers and on
morphological parameters as well as uniformity and size of the NRs. Furthermore, some treatments are suggested in order to improve the quality of the sol-gel seed layer.

II. MATERIALS AND METHODS

Here, we report a fast and novel microwave-assisted hydrothermal method for the growth of vertically aligned ZnO NRs. In our system, the rate of heating is being controlled carefully by turning a microwave oven on and off sequentially. For this purpose, a digital controller circuit was designed to manage the heating rate. Using this method, we achieved a lower crystallization temperature and homogeneous nucleation compared to other hydrothermal systems.

Glass slides were used as the substrates for growing ZnO NRs. The substrates were initially cleaned with acetone and DI water to remove any impurity particles. In order to deposit four different ZnO seed layers on the substrates, 5mM solution of Zinc acetate (Zn(CH₃COO)₂.2H₂O) in ethanol (C₂H₆O) was spun and coated on the substrates using different spinner speeds of 1000, 2000, 3000 and 4000 rpm for 30 sec. The coating process was repeated 5 times to reach a highly uniform seed layer. Between each coating step, the sample was left at room temperature for 2 min. Then, samples were heated at 350°C for 20 min in air to remove the extra organic compounds.

To grow an array of vertically-aligned ZnO NRs, a solution containing 25mM solution of zinc nitrate hexahydrate (Zn(NO₃)₂.6H₂O), 13mM hexamethylenetetramine ((CH₉N)₄), 5mM polyethylenimine (PEI) ((C₂H₃N)ₙ) and 8mM ammonium hydroxide (NH₄OH) was prepared in DI water.

After the seed layer preparation mentioned earlier, the substrates were placed inside a sealed Teflon vessel for the growth process. In order to have a uniform molarity of the growth solution during the whole process and to prevent solvent evaporation, the sealed Teflon vessel compatible with high pressure processes was designed and fabricated. The
vessel was made of Teflon to avoid any reaction with the solutions and also to prevent absorption or reflection of the microwave irradiations during the growth process in the microwave oven. Maximum bearable pressure of the vessel was about 7 bar.

The substrates were immersed into the growth solution in the middle of the vessel with the seed layers facing downwards to prevent attachment of suspended ZnO nanoparticles on the NRs during the growth process. One of the advantages of using microwave irradiation is to be able to transfer power to the precursors rapidly. So in this method, microwave power was fixed on 350 watts (maximum available power of our oven) and the process was managed by sequentially turning the microwave power on and off in order to control the solution’s temperature, prevent overheating and vaporization of the solution. After the growth process, the vessel was left at room temperature for 30min to let the solution cool down. The samples were then rinsed by DI water and dried by air flow.

Microwave irradiation was then applied using a commercial microwave oven (Ufesa HM6684) to heat the solution and transfer power. A field emission scanning electron microscope (Hitachi-4289) was used to check the morphological view and extract the growth rate of the NRs. Investigations on the quality of the seed layers were performed using an atomic force microscope (NT-MDT, SOLVER NEXT). XRD analysis also was performed to evaluate the crystalline quality of ZnO NRs.

III. RESULTS AND DISCUSSIONS

In order to control the solution’s temperature and control the growth process, different heating programs were designed and tested. Table I shows the different growth programs used to investigate the effect of the heating rate during the ZnO NR growth. In some of the programs, an initial heating sequence was set to increase the solution’s temperature prior to
the ZnO NR growth. Following the first heating, the microwave irradiation was turned off for a period of time to allow the solution’s temperature to stabilize and let the precursors to start nucleating and forming ZnO NRs on the substrate. It must be mentioned here that the temperature during the process was monitored at real time by an infrared laser thermometer (Model: Terminator-TIR8861). Due to the loss of heat from the solution to the ambient when irradiation is off, the next heating sequence was performed to maintain the solution’s temperature at a specified value and control the ZnO NR growth rate. These sequential heating and cooling periods were performed continuously to utilize all of the precursors to form and grow ZnO NRs uniformly on the substrate.

<table>
<thead>
<tr>
<th>Program no.</th>
<th>Sequences</th>
<th>No. of repeats</th>
<th>Average growth rate(µm/h)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>“On” duration(sec)</td>
<td>“Off” duration(min)</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>30</td>
<td>5</td>
<td>10</td>
</tr>
<tr>
<td>2</td>
<td>60</td>
<td>5</td>
<td>6</td>
</tr>
</tbody>
</table>

Table I. Heating programs without initial heating, used to determine optimum growth conditions

Using thermodynamic theory, the heat equation (Eq. 1) was applied in which “E” is the given energy to the system, “m” is the solution’s total mass which in this study is 100 mgr, “C” is the specific heat constant of the solution and “θ” is the temperature change; the change in the temperature is possible to be controlled by the amount of energy given to or taken from a system. As the microwave power is set to 350W, considering a rough approximation that the microwave absorption of the system is fixed and equal to the applied power by the oven, the amount of absorbed energy equals to the product of microwave power “P” by the duration of microwave irradiation denoted as “t_{Heating}” in (Eq. 2). Eq. 2
expresses the temperature variation in terms of irradiation time, by which the temperature
increase ($\theta_{\text{on-sequence}}$) can be controlled by the time of exposure to microwave radiations.

$$E = m^2 C^2 \theta$$  \hspace{1cm} (1)

$$\theta_{\text{on-sequence}} = \frac{P \cdot t_{\text{Heating}}}{m^2 C}$$  \hspace{1cm} (2)

Temperature loss due to the heat flow from the solution to surrounding environment
can be written as Eq. 3 where “$t_{\text{Cooling}}$” is the off time duration of the irradiation and “K” is
the thermal conductivity. This parameter depends on the properties of the Teflon vessel.

When the solution’s temperature ($\theta_{\text{Solution}}$) was about 85°C, it was determined empirically to
be 0.018 Kmin$^{-1}$. In this experiment ambient temperature ($\theta_{\text{Ambient}}$) was 25°C so that the
temperature loss when the solution is cooling down can be calculated using Eq. 3.

$$\theta_{\text{off-sequence}} = K \cdot (\theta_{\text{Solution}} - \theta_{\text{Ambient}}) \cdot t_{\text{Cooling}}$$  \hspace{1cm} (3)

Using Eq. 2 and Eq. 3 it is absolutely possible to control the solution’s temperature by
sequential monitoring of the microwave applied power. Table I and II show 5 different
heating programs that were tested to find optimum conditions to be used for ZnO NR growth.

<table>
<thead>
<tr>
<th>Program no.</th>
<th>Initial heating(sec)</th>
<th>Sequences</th>
<th>No. of repeats</th>
<th>Average growth rate(µm/h)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>“Off” duration(min)</td>
<td>“On” duration(sec)</td>
<td></td>
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<td>3</td>
<td>90</td>
<td>10</td>
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<td>60</td>
<td>10</td>
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<td>6, 8, 10</td>
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<tr>
<td>5</td>
<td>75</td>
<td>5</td>
<td>15</td>
<td>12</td>
</tr>
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</table>

Table II. Heating programs having initial heating
In programs 1 and 2, although there is no initial heating applied to the solution, the on-off sequences were programmed to gradually warm up the solution, starting from the room temperature (25°C). As the temperature raised, the nucleation rate was increased and when the temperature reached to an appropriate value (at nearly 90°C [7,8]) the ZnO NRs growth mechanisms became faster. Two main problems exist in these cases: Firstly, since the growth rate at low temperature is insignificant, during the time which the solution was heating up no ZnO NR would grow. However, in program 2, heating rates were increased by choosing longer “On” sequences compared to case 1 (see Fig. 1), but an interval was still needed to warm up the solution to the growth temperature. This in turn, led to slow ZnO NR growth.

![Fig. 1 Temperature variation of the solution in programs 1 to 5. The insets show ZnO NRs grown a) during 12 times 15 sec “on” 5 min “off” b) during 8 times 30 sec “on” 10 min “off”. Scale bars are 500 nm.](image)

Secondly, as shown in Fig. 1, overheating the solution (more than 100°C) could be responsible for an increase in the homogeneous nucleation rate of ZnO particles which consequently reduced the heterogeneous nucleation rate of ZnO NRs. Thus, the solution wouldn’t have enough precursor materials for the growth [10,14]. Furthermore, this overheating evaporates the solution to some extent and as a result, it could change the
parameters of the study (molarity and pressure inside the vessel). In programs 1 and 2 after few on-off sequences were passed, the solution reached the suitable temperatures for ZnO growth and then an ultra-fast growth process began. By continuing the process, the overheating—as explained before—would occur.

The explained problems in programs 1 and 2 led to a slow ZnO NR growth and very short NRs. In order to tackle the problems and increase the growth rate an initial heating was added to the programs (see programs 3 to 5 in Table II). In fact, the idea was to increase the solution’s temperature to a suitable value, and then maintain its temperature by applying proper on-off sequences, transfer microwave energy to the precursors and let the NRs grow.

In program 3, the initial heating time was too long so that not only the solution’s temperature became too high leading to overheating, but also due to the high thermal shock, the grown NRs together with the seed layer were peeled off from the substrate (This process was repeated 5 times on glass and also different substrates).

Program 4 started with a moderate (60 seconds) initial heating followed by a different number of sequences. The final optimum goal was to achieve a process which completely consumed the solution’s precursors to form ZnO NRs on the substrate. Using cross-sectional SEM images, it was observed that usually after the 6th sequence, the density of precursors in the solution would reduce enough so that the length of ZnO NRs wouldn’t change noticeably if the growth procedure was continued up to the 10th sequence.

By trying different initial heating times and the resultant ZnO NRs, the optimised program was determined (see program 5 in Table II) where the initial heating time was 75 seconds. By choosing 30 seconds “on” and 10 minutes “off” sequentially, the solution’s temperature would alter ±5°C around 85°C (see Fig. 1) and the grown ZnO nanostructures would tend to
be like nanoneedles rather than hexagonal NRs as shown in insets of Fig. 1. Therefore, the optimized ZnO NR growth procedure is program 5 in Table II.

Fig. 2 shows the XRD data of ZnO NRs grown on a glass substrate using the optimized program. The strong peak at 34.5° that corresponds to the [0002] peak in wurtzite ZnO crystals indicates that using this method, it is possible to form highly crystalline ZnO NRs vertically along the c-axis. The accompanying SAED pattern (see the inset in Fig. 2) also confirms that the ZnO NR has a hexagonal wurtzite structure. The grown nanostructures had the average diameter of about 100 nm. This method has reached ultra high growth rates close to 70 nm/min which is about 7 times faster than conventional hydrothermal methods [8].

One of the major advantages of our sequential method over other reported microwave-assisted methods is the total amount of energy consumed to form similar ZnO nanostructures. Other approaches usually apply a fixed microwave power to the solution continuously for a long time that results in a high amount of energy consumption[9,14,15]. As reported previously in a study, 850W microwave irradiations were applied for about 10 minutes to 200ml of similar growth solution which equals to about 708 Joules/Liter [10].

**Fig. 2** XRD analysis of the grown ZnO NRs. The inset is a selected-area electronic diffraction pattern (SAED).
In our optimized program (program 5) not only the total amount of energy given to the solution is about 0.25 Joules/Liter which is significantly lower in consumption compared to the latter case, but also led to similar results. This implies that in our method, by proper control over the applied power using a sequential program and using the vessel, we were able to minimize the energy consumption, reduce the thermal budget and propose a highly efficient and ultra-fast ZnO NR growth method.

As mentioned earlier, pre-growth preparations e.g., the seed layer deposition quality, plays an important role in the ZnO NR quality and uniformity. This phenomenon can simply be observed by monitoring the quality, size and alignment of the grown ZnO NRs. Fig. 3 shows the FESEM images from the top view of the NRs grown on different seed layers prepared with different spinning speeds of the sol-gel on the glass substrates.

![FESEM images of ZnO NRs grown on different seed layers](image)

**Fig. 3** FESEM images of ZnO NRs grown on different seed layers (scale bars are 3 µm). The seed layers are made at the spinning speeds of (a) 1000 rpm, (b) 2000 rpm, (c) 3000 rpm, (d) 4000 rpm.

In this study, the only altering parameter that affects the quality of seed layers is the rotation speed of the spinner at which the seed layers are coated, thus the uniformity of the ZnO NRs
can only be attributed to the spinner’s speed. In order to verify these results, the similar experiment was conducted four times and still the results were the same. The substrate seeded with a sol-gel containing 5mM solution of Zinc acetate (Zn(CH\textsubscript{3}COO)\textsubscript{2}.2H\textsubscript{2}O) in ethanol (C\textsubscript{2}H\textsubscript{6}O) at rotation speed of nearly 2000rpm has a uniform NRs density of \(6 \times 10^6 \text{cm}^{-2}\) (Fig. 3b). However, at other rotation speeds (Fig. 3a, c and d), plenty of ZnO islands with completely different NR densities are observed.

The Formation of ZnO islands at the spinner speed of 1000rpm (see Fig. 3a) can be interpreted as the high viscosity of the solution that doesn’t let it get coated uniformly. At the speed of nearly 2000rpm which is the optimum point, the seed solution gets enough energy to cover the substrate. Increasing the spinning speed will create many islands close to the edges and separate the uniform seed layer into many sub-seed layers sitting beside each other. Non-uniformity in the seed layer coating leads to the formation of ZnO islands and consequently the occurrence of non-uniformity in ZnO NRs.

The average density of ZnO NRs can’t be easily measured on the ZnO islands, but they obviously have higher density compared to the areas around them (see the inset of Fig. 3d). This phenomenon can be referred to as the pattern dependency of the growth. The pattern dependency is partly due to the variation of the exposed area for deposition in cases such as what we are faced with here for the speeds below/over 2000 rpm. Higher density of the available sites on the ZnO islands compared to other areas surrounding them causes a non-uniform consumption of the inlet precursors. This will in turn decrease the growth rate on the islands on one hand and affect the growth rates in areas close to the islands on the other hand.

As the precursors were consuming to form ZnO NRs, their density in the solution and also around the NRs decreased. This created a local density gradient which acted as a sink that forced the precursors to diffuse continuously towards the ZnO NRs. As a result, the consumption of precursors on the ZnO islands became faster than the areas around them and
the low growth rate at the border of ZnO islands resulted in a low density of NRs (see the inset of Fig. 3d). At the speed of nearly 2000rpm the lowest numbers of ZnO islands have formed on the substrates. This implies that this simple and cheap method is still promising for growing uniform ZnO NRs on glass substrates.

The length and diameter of ZnO NRs along the c-axis are measured using cross-sectional SEM images (Fig. 4a-d). Fig. 4e shows the average aspect ratio (length/diameter) of ZnO NRs grown at microwave irradiations. The seed layers spun and coated at the speed of 2000 rpm have the highest average ZnO NRs aspect ratio among other samples. This can be elucidated by the fact that using other spinning speeds didn’t provide a uniform platform for the growth. Therefore, the aspect ratio of NRs decreases either by increasing or decreasing the speed of the spinner. It has also been reported previously that the existence of small ZnO crystal grains in the seed layers results in higher available surface for growth and thus an increase in the density of the NRs. This can consequently decrease the diameter of the ZnO NRs [12].
Fig. 4 (a-d) cross-sectional SEM images (scale bars are 1um) and e) the average aspect ratios of ZnO NRs grown on the seeds with different spinner’s speed.

So far, these data reveal that the preparation of seed layer plays an important role on the quality of ZnO NRs grown by this method. The suggested optimum rotation speed of the spinner for seeding process is about 2000 rpm which leads to the growth of uniform and vertically-aligned ZnO NRs with high aspect ratio.

In order to eliminate the dependency of seed layers’ quality on the spinner speed, the samples spun with 1000 rpm were annealed in a furnace at 350°C for 24 and 48 hours and the effects of these thermal treatments were investigated using atomic force microscope images.

Fig.5a shows the AFM image of a sample prepared at 1000 rpm and then heated at 350°C for 20 minutes i.e., normal treatment for all seed layers. Looking at the AFM images here and comparing it with Fig. 3a, one can conclude that the non-uniformity of the NRs is almost
exactly matched with the morphology of the seed layer. Fig.5 implies that, as it can be expected from Fig. 3a, the size and distribution of the ZnO islands are clearly random and non-uniform in the substrates without any long annealing process.

**Fig.5** AFM images of the substrates annealed at 350°C for (a) 20 min, (b) 24 and (c) 48 hours. The RMS roughness of the seedayers are (a) 52.7, (b) 32.5 and (c) 28.8 nm.

Fig.5b and c show the AFM image of the latter sample annealed at 350°C for 24 hours and 48 hours, respectively. Fig.5b shows that by an annealing process for 24 hours the ZnO seeds and islands tend to merge and become bigger in size but more uniformly distributed. Another heating treatment as shown in Fig.5c gives ZnO islands enough energy to migrate to the neighboring ZnO islands and become very large in size and little in density.
Previous explanations for Fig. 5b are also applicable for Fig. 5c except that in Fig. 5c by continuing the annealing process for 48 hours, the seed layer has significantly changed and the size and distribution of the ZnO seeds has become more uniform. It is worth mentioning here that the surface RMS roughness of the seed layers have been reduced from 52.7 nm in Fig. 5a to 28.8 nm in Fig. 5c.

The latter explanations in association with results from Fig. 3 support the idea that increasing the annealing time can lead to more uniform seed layers. After reaching the optimum thermal budget, the size of the ZnO grains increases by the thermal annealing. The more uniform the seed layers become, the more uniformly ZnO NRs can be grown. As a result, it is possible to decrease the dependency of the ZnO seed layers to the spinner speed in the sol-gel method.

IV. CONCLUSIONS

In summary, a sequential microwave-assisted growth controller and a vessel were designed and used for ultra-fast producing of uniform ZnO NRs. The microwave oven power programs were planned in a way to attain the maximum NR growth by using an initial heating step. The highest growth rate was 4.2 μm/hour. The spinner’s speed as an effective parameter on the quality of ZnO seed layer in the sol-gel method was explored and it was observed that the optimum spinner speed for this method is 2000 rpm. In order to diminish the dependency of the uniformity of ZnO NRs on the quality of seed layers, a 48 hours thermal annealing at 350°C as a remedy was suggested as well. A microwave-assisted hydrothermal system for ZnO NRs growth capable of growing uniformly distributed, high aspect ratio and vertical aligned NRs was designed and investigated. Very high growth rates of 70nm/min were obtained which is well above the previous reported values.
V. REFERENCES


• A novel method for ultrafast growth of ZnO nanorods on substrates has been developed.
• The microwave-assisted grown ZnO nanorods are crystalline and vertical along c-axis.
• A Sol-Gel seed layer is proposed and optimized for ZnO growth.
• Seed layer and heating method strongly affect the quality of ZnO nanorods.