A close correlation between nucleation sites, growth and final properties of ZnO nanorod arrays: Sol-gel assisted chemical bath deposition process

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Different ZnO seed layers are synthesized by changing sol-gel parameters, including precursor concentration, type of solvent, and type of additive in order to systematically investigate the importance of seed layer properties on the nucleation, growth, and final properties of zinc oxide (ZnO) nanorod (NR) arrays. The X-ray diffraction (XRD) and field emission scanning electron microscope (FESEM) show the importance of the seed layer on ZnO NRs properties. Results verify that the relative intensity (RI factor) of (002) polar planes in the XRD patterns of ZnO nanoparticles (NPs) together with the aspect ratio, density, and alignment of ZnO NRs control the structural characteristics of those arrays. For instance, the RI factor of ZnO NPs and NRs follow the same trend when changing precursor concentration in sol preparation step. However, the importance of other parameters, including aspect ratio, density, and alignment of NRs is confirmed by changing solvent and additive. In addition, FESEM images show that the density of ZnO NRs is proportional to NPs density and inversely proportional to the size of ZnO NPs in the seed layers. Besides, the significant role of wrinkled inter-layer on NRs properties, together with the formation mechanism of that inter-layer are profoundly investigated.

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1. Introduction

Selecting appropriate materials that show a variety of morphologies and therefore different properties is inevitable in order to adapt a material for a wide range of applications such as solar cells [1–3], water purification [4,5], transistors [6,7], and light emitting diodes (LEDs) [8,9]. As a result of having diverse morphologies such as nanorods (NRs), nanobelts, nanorings, nanocomb, and so forth, zinc oxide (ZnO) is undoubtedly the richest material, both in structure and properties [10]. ZnO is one of the most crucial inorganic semiconductors with wide direct bandgap of approximately 3.37 eV, large exciton binding energy (60 meV) [11], high electron-hole recombination rate, and high electron mobility of 100 cm² V⁻¹ S⁻¹ [12]. Furthermore, it has hexagonal wurtzite structure which belongs to the space group of C₆₃ and its non-centrosymmetric structure leads to piezoelectric properties [5,13–15].

Owing to its simplicity, being cost efficient, and capability to form different morphologies by changing the parameters [16], chemical bath deposition (CBD) has been opted to synthesize one-dimensional ZnO nanostructures among a variety of methods such as pulsed laser deposition [17], laser ablation-catalytic growth [18], chemical vapor deposition, and physical vapor deposition [19]. There are many parameters in CBD process such as precursor and additive materials, temperature and time of CBD, and type of substrate [20,21], which lead to distinct features, and hence different morphological, crystallographic, and optoelectronic properties. The characteristic of a substrate is of paramount importance to synthesize ZnO NRs both in nucleation and growth processes and final properties of ZnO NR arrays [22,23]. The seed layer can decrease the required activation energy for ZnO NRs to grow. Thus, it is feasible to synthesize one-dimensional ZnO nanostructures in a lower level of supersaturation.

Several vital items such as structure, crystallography, and morphology should be considered to choose a suitable seed layer deposition technique. It is possible to modify the seed layer properties to achieve different ZnO NRs. In order to enhance the driving force of ZnO NRs growth, sol-gel dip coating method is extensively applied to deposit ZnO thin films on a bare glass substrate [24]. Precursor concentration, type of solvent, and type of additive are some of the sol-gel parameters, which determine

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the final properties of seed layers; and hence, can adjust ZnO NRs to different applications by changing its properties.

Lockett et al. [23] have deposited seed layers by means of four different methods in order to investigate the effect of deposition method on the seed layer and their corresponding NRs properties. In addition, Son et al. [25] have prepared three different seed layers and investigated the effect of those seed layers on the properties of ZnO NRs in the perovskite solar cells. Until very recently, there is no research, which provides the close correlation between sol-gel synthesized seed layers and CBD grown ZnO NR arrays. Thus, the main purpose of this paper is to systematically investigate the significant role of sol-gel synthesized seed layer properties on the structural and morphological properties of ZnO NR arrays. In addition, the nucleation and growth mechanism is precisely figured out and portrayed in different seed layers.

2. Experimental

2.1. Materials

Zinc acetate dihydrate (Zn(CH3O2).2H2O/ZAD) as a precursor, methanol (CH3OH/MeOH), ethanol (C2H5OH/EtOH), 1-propanol (C3H7OH/1-PrOH), 2-propanol (C3H7OH/2-PrOH), and 1-butanol (C4H9OH/1-BuOH) as alcoholic solvents, and amino-additives including triethylamine (N(CH2CH3)3/TeA), monoethanolamine (MEA), diethanolamine (DEA) as alcholic solvents, and amino-additives including triethylenediamine (N(CH2CH2)3/TeA), monoethanolamine ((CH2CH2OH)NH2/MEA), diethanolamine ((CH2CH2OH)2/NH/DEA), and triethylenediamine ((CH2CH2OH)3N/TEA) as stabilizing agents were purchased from Merck KGaA and utilized without further purification. Hexamethylenetetramine (C6H12N4/HMT) as a complexing agent and pH buffer, and deionized water (DI) were used to synthesize ZnO NRs.

2.2. Sol preparation and seed layer deposition

According to our previous work [26], a variety of different sols were prepared by dissolving a zinc precursor and an amino-additive in an alcoholic solvent. Briefly, the initial sol contains 0.2 M ZAD as a precursor, the same molarity of TeA as an additive, and 1-PrOH as an alcoholic solvent. The sol was prepared by mixing these materials for 30 min at room temperature. Then, dip coating procedure was repeated 10 times. Finally, the calcination process was carried out at 500 °C for 60 min Table 1 listed various seed layers and their special features. For each sample, while one parameter is changed, other parameters were held constant.

<table>
<thead>
<tr>
<th>Sample no.</th>
<th>ZAD conc. (M)</th>
<th>Type of solvent</th>
<th>Type of additive</th>
<th>NPs size (nm)</th>
<th>NPs density (%)</th>
<th>NPs RI</th>
<th>NRs length (nm)</th>
<th>NRs diameter (nm)</th>
<th>NRs aspect ratio</th>
<th>NRs density (%)</th>
<th>NPs RI factor</th>
<th>NRs RI factor</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.1</td>
<td>1-PrOH</td>
<td>TeA</td>
<td>30</td>
<td>91</td>
<td></td>
<td>950</td>
<td>110</td>
<td>8.6</td>
<td>85</td>
<td>0.55</td>
<td>0.96</td>
</tr>
<tr>
<td>2</td>
<td>0.2</td>
<td>1-PrOH</td>
<td>TeA</td>
<td>40</td>
<td>90</td>
<td></td>
<td>1000</td>
<td>120</td>
<td>8.3</td>
<td>80</td>
<td>0.36</td>
<td>0.81</td>
</tr>
<tr>
<td>3</td>
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<td>1-PrOH</td>
<td>TeA</td>
<td>50</td>
<td>85</td>
<td></td>
<td>1300</td>
<td>125</td>
<td>10.4</td>
<td>75</td>
<td>0.95</td>
<td>0.99</td>
</tr>
<tr>
<td>4</td>
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<td>MeOH</td>
<td>TeA</td>
<td>60</td>
<td>80</td>
<td></td>
<td>950</td>
<td>120</td>
<td>7.9</td>
<td>73</td>
<td>0.85</td>
<td>0.92</td>
</tr>
<tr>
<td>5</td>
<td>0.2</td>
<td>EtOH</td>
<td>TeA</td>
<td>55</td>
<td>82</td>
<td></td>
<td>950</td>
<td>80</td>
<td>11.8</td>
<td>75</td>
<td>0.93</td>
<td>0.93</td>
</tr>
<tr>
<td>6</td>
<td>0.2</td>
<td>2-PrOH</td>
<td>TeA</td>
<td>65</td>
<td>78</td>
<td></td>
<td>1000</td>
<td>105</td>
<td>9.5</td>
<td>70</td>
<td>0.88</td>
<td>0.90</td>
</tr>
<tr>
<td>7</td>
<td>0.2</td>
<td>1-BuOH</td>
<td>TeA</td>
<td>50</td>
<td>85</td>
<td></td>
<td>400</td>
<td>100</td>
<td>4.0</td>
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<td>0.83</td>
</tr>
<tr>
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<td>1-PrOH</td>
<td>MEA</td>
<td>30</td>
<td>92</td>
<td></td>
<td>1100</td>
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<td>1200</td>
<td>90</td>
<td>13.3</td>
<td>81</td>
<td>0.43</td>
<td>0.93</td>
</tr>
</tbody>
</table>

2.3. Synthesis of ZnO NRs

ZAD and HMT were separately dissolved in DI and then mixed together [21]. In this research, 0.02 M of ZAD and the same concentration of HMT were dissolved in DI. Next, with the help of the Teflon holder, the prepared ZnO seed layers were placed vertically into this solution and put into the bath at 90 °C for 4 h under constant stirring. After the CBD process, all of the substrates were rinsed with DI water and dried at 90 °C for 10 min. Finally, all the films were annealed at 500 °C for an hour.

2.4. Characterization techniques

X-ray diffraction method (XRD, Rigaku Ultima IV diffractometer with Cu Kα radiation, λ = 1.5418 Å) was used to investigate the structural properties of ZnO seed layers and ZnO NRs. Field emission scanning electron microscope (FESEM, ZEISS- SIGMA VP) was utilized to find out the relationship between morphological properties of ZnO nanoparticles (NPs) and ZnO NRs; hence, this analysis can make it possible to expound the nucleation sites and growth mechanism. The density of ZnO NPs and NRs was measured by a couple of methods; counting the number of ZnO NPs and NRs in a randomly selected area and measuring porosity between NPs and NRs. Both methods result in the same value of density.

3. Results and discussion

As a result of existing high structural coherency between ZnO NPs and NRs, ZnO thin film is the most appropriate choice to opt as a seed layer. This characteristic can diminish the entailed activation energy of nucleation and growth. In addition, (002) polar surfaces have the highest surface energy among the other facets of the wurtzite structure. Thus, this is the most suitable plane for ZnO NRs to nucleate and grow.

3.1. Precursor concentration

Various ZnO seed layers have been synthesized by changing ZAD concentration as a precursor in the sol preparation step. Therefore, ZnO NRs with different structural and morphological properties were obtained. Fig. 1 shows the XRD patterns of ZnO seed layers and their corresponding ZnO NRs. Both ZnO NPs and NRs have hexagonal wurtzite structure according to JCPDS card no. 36-1451. As can be seen, the intensity of wurtzite planes in ZnO seed layers and their corresponding ZnO NRs. Both ZnO NPs and NRs are measured by a couple of methods; counting the number of ZnO NPs and NRs in a randomly selected area and measuring porosity between NPs and NRs. Both methods result in the same value of density.
Eq. (1): explained by means of the Landau–Levich model as expressed in Eq. (1):

$$h = 0.94 \left( \frac{\eta}{\gamma} \right)^{2/3} \left( \frac{\rho g}{u} \right)^{1/2}$$

where $\gamma$, $\eta$, $\rho$, $g$ and $u$ are the surface tension, the viscosity, the density of the fluid, the acceleration due to gravity, and the withdrawal speed, respectively [27]. It is self-evident that increasing ZAD concentration results in increasing sol viscosity ($\eta$), and based on this model, the thickness of the ZnO thin films increases and consequently the intensity of wurtzite facets enhances.

To analyze the XRD patterns of ZnO NRs, the first point is the discrepancy in peak intensities of ZnO NPs and NRs, which is depicted in all samples. As it is shown by scale bars, peaks in ZnO NRs are more intensive than those in ZnO NPs, which mainly depends on the NRs growth along c-axis. In addition, the XRD patterns of ZnO NRs contain shoulder-like peak which is due to the difference between crystallinity of ZnO NPs and NRs.

The second point is the relative intensity (RI) of (002) polar planes, which is defined as follows to compare the XRD patterns of ZnO NPs and ZnO NRs, and to figure out the correlation between seed layers and NRs:

$$R(002) = \frac{l(002)}{l(100)+l(002)+l(101)}$$

The RI factor of 0.1, 0.2, 0.4 M ZAD seed layers are 0.55, 0.36, and 0.95, respectively. Although the intensity of (002) polar surfaces does not significantly change by increasing ZAD concentration from 0.1 M to 0.2 M, the intensity of (100) and (101) lateral planes raises, which leads to the reduced RI factor and therefore the reduction of the suitable nucleation sites for ZnO NRs. However, the intensity of (002) polar facets greatly enhances by increasing ZAD concentration to 0.4 M. The RI factor elevates and a number of suitable nucleation sites are available in 0.4 M sample. Accordingly, it can be deduced that the amount of suitable nucleation sites in ZnO seed layer is proportional to the RI factor of that layer.

The amount of additive also increases by increasing the ZAD concentration to keep $[\text{ZAD}] / \text{[TeA]}$ ratio constant. In fact, there is a competition between the amount of ZnO which results in more preferred orientation along c-axis and the presence of organic materials which causes a reduction of (002) intensity in XRD pattern of ZnO NPs. Forasmuch as almost all organic materials is evaporated in the drying step, the effect of the organic residue on preferential growth is less than the effect of ZnO content. Thus, the presence of a considerable amount of ZnO in 0.4 M ZAD sample and the small amount of organic material in 0.1 M ZAD sample result in larger NPs RI factor in comparison with 0.2 M sample.

As shown in the XRD patterns of ZnO NRs, the intensity of (002) polar facets reduces by increasing ZAD concentration from 0.1 M to 0.2 M, whereas the peak intensity of (002) enhances by increasing ZAD concentration to 0.4 M. These changes chiefly depend on the seed layer properties especially NPs RI factor or the amount of (002) polar planes. It should be noted that the influence of other parameters on the ZnO NRs peaks intensities should be also considered.

Fig. 1 shows FESEM images of ZnO seed layers followed by ZnO NRs with different seed layer sol concentration. In addition, low magnification FESEM images are provided in order to confirm the uniform distribution of ZnO NRs (see the supplementary section, Fig. S1). The NPs size, NRs aspect ratio, and density for all samples are listed in Table 1. As shown in the FESEM images and Table 1, the size of ZnO NPs enhances by increasing ZAD concentration in seed layers. Moreover, ZnO NPs density decreases as a result of NPs growth. By analyzing the FESEM images of ZnO NRs, it can be deduced that the density of ZnO NRs reduces with increasing ZAD concentration in seed layers. Therefore, density of ZnO NRs is found to be inversely proportional to the size of the ZnO NPs and proportional to NPs density. This may also explain why the intensity of (002) polar planes decreases by increasing ZAD concentration from 0.1 M to 0.2 M. Another vital conclusion which is derived from this part is that the intensity of (002) peak in XRD patterns of ZnO NRs is affected by RI factor of the seed layer more than the density of ZnO NRs due to the fact that the density of ZnO NRs decreases by increasing ZAD concentration to 0.4 M, but the peak intensity of (002) dramatically enhances.

While the aspect ratio of ZnO NRs enhances by increasing ZAD concentration from 0.2 M to 0.4 M, it diminishes when ZAD concentration increases from 0.1 M to 0.2 M (Table 1). Thus, it can be another explanation, which confirms the RI factor changes in the XRD patterns of ZnO NRs.

As it is illustrated in Fig. 3, some changes in NRs dimensions occur. There are several factors which control NRs dimensions. The first factor is precursor type and its concentration. The second item is the capping agent and its concentration. The last one is seed layer RI factor and NPs size on which ZnO NRs should be grown. The more amount of precursor may result in larger NRs...
Fig. 2. FESEM images of ZnO seed layer and their corresponding ZnO NRs; (a, b) 0.1 M, (c, d) 0.2 M, (e, f) 0.4 M ZAD. Insets show the cross sectional view of NRs.
either along a- or c-axes because there are more materials available to take part in NRs growth. Furthermore, different capping agents have distinct behavior toward ZnO NRs. They can chelate to the polar or non-polar facets of wurtzite structure based on additives molecular properties and enhance or retard the growth along c-axis. In this research, HMT is utilized as a capping agent to prevent growth along a-axis in order to obtain ZnO NRs. HMT is a non-ionic and heterocyclic compound and plays three important roles. It is the pH buffer and complexing agent. Furthermore, HMT is capped to non-polar facets of wurtzite structure and inhibits its lateral growth along a-axis. Thus, one may change the [HMT]/[ZAD] ratio to alter the diameter of NRs.

More importantly, ZnO seed layer plays a decisive role in determining NRs dimensions. NPs RI factor and their size are both key parameters, which control NRs length and diameter. More material is attracted to the seed layer with more suitable nucleation sites or higher RI factor. In addition, NPs density is proportional to NRs density. Then, the growth of ZnO NRs is limited by their adjacent NRs, which results in NRs with lower diameter. Since materials type and their concentrations were held constant in this experiment, NPs RI factor together with NPs size control NRs dimensions.

The last parameter which may affect the structural properties of ZnO NRs is their alignment. As shown in Fig. 2, alignment of ZnO NRs retards by increasing ZAD concentration from 0.1 to 0.2 M and enhances by increasing the concentration to 0.4 M. This observation is hard evidence, which proves XRD patterns of ZnO NRs and the importance of alignment to analyze the NRs structural properties.

### 3.2. Solvent type

The XRD patterns of ZnO thin films and corresponding NRs of different solvents are shown in Fig. 4 to depict the role of the solvent type in sol preparation step on structural properties of ZnO seed layers and ZnO NRs. As can be seen, XRD patterns of ZnO thin films changes by changing solvent type in sol preparation step. These changes depend mainly on solvents properties including viscosity, boiling point, and their corresponding reactions in each sol (see the supplementary section, Table S1). As it is confirmed by Landau–Levich model, the thickness of ZnO thin films increases by increasing sol viscosity. Therefore, it is expected to obtain thicker ZnO thin films by using a solvent with longer chain length, which results in more intense (002) polar planes in XRD patterns of ZnO NPs. However, the boiling point of alcoholic solvents increases by increasing the chain length and more stress occurs in solvent evaporation process during drying step when using solvents with a higher boiling point. Consequently, (002) polar planes can grow more easily when using solvents with a lower boiling point. It can be concluded that the competition between these two parameters is the main factor, which determines the (002) polar planes intensity in the XRD patterns of ZnO thin films.

Fig. 5 illustrates the RI factor of different seed layers and NRs. The same trend occurs in RI factors of ZnO NPs and NRs. As mentioned before, it is expected that the intensity of (002) polar planes in ZnO NRs enhances by increasing the RI factor of ZnO NPs. Thus, it is sensible to have highly oriented peaks along c-axis when using MeOH and EtOH as solvents to prepare seed layers. In addition, short peaks can be achieved when using 1-PrOH as a...
Fig. 6. FESEM images of ZnO seed layer and ZnO NRs for different solvents: (a, b) MeOH, (c, d) EtOH, (e, f) 2-PrOH, (g, h) 1-BuOH.
solvent. However, NRs peaks intensities are not so high in case of 2-ProOH and 1-BuOH solvents. Although the RI factors of ZnO NPs are high, the RI factors of ZnO NRs are lower than expected values.

Fig. 6 illustrates the FESEM images of ZnO seed layers followed by ZnO NRs for different solvents (see the supplementary section, Fig. S2). The alignment of NRs grown on 2-ProOH seed layer is overly low, which is attributed to the large NPs size (Table 1). This observation may explain short peaks in its XRD pattern. Furthermore, the aspect ratio of the NRs is too low (Table 1) when using 1-BuOH as a seed layer and therefore causes the reduction of (002) polar planes in XRD patterns. Therefore, even though the RI factor is relatively high, the aspect ratio and alignment of ZnO NRs are not consequently high. These observations confirm the claim that the RI factors of ZnO NRs are affected not only by NPs RI factor but also by NRs morphological properties.

Moreover, the correlation between NPs size and NRs density is confirmed by analyzing these FESEM images. For instance, 2-ProOH seed layer has the largest NPs (Table 1) and the least density of ZnO NRs (Fig. 6).

### 3.3. Additive type

Fig. 7 illustrates the XRD patterns of ZnO seed layers and NRs to show the influence of different amino-additives on the structural properties of ZnO thin films and NRs. Additive properties such as molecular structure and polarity explain why the peaks intensities change by changing the additive [26]. MEA produces the smallest nano-colloids in comparison with other additives due to its molecular structure. Thus, the evaporation of this additive in drying step is more than other additives. Consequently, the (002) polar surfaces grow more easily when using MEA as an additive in seed preparation step. In addition, the polarity of additives decreases by using DEA, TEA, and TeA which are type II and III amino-additives compared to MEA. Accordingly, the size of nanocolloids increases and the evaporation of additives in drying step retards, which results in (002) peak with lower intensities.

According to the NPs RI factor, it is sensible to have high and low NRs RI factor when using MEA and TeA as an additive in the sol preparation step, respectively. Furthermore, although the NPs RI factors in DEA and TEA samples are not high, the largest RI factor occurs when using DEA and TeA as an additive in the sol preparation step. The high NRs aspect ratio (Fig. 8 and Table 1) is a compelling reason, which confirms the importance of NRs dimensions to control the XRD patterns of NRs (see the supplementary section, Fig. S3). In fact, NRs RI factor enhances not only by NPs RI factor but also with NRs aspect ratio.

### 3.4. Nucleation and growth mechanism

A short time CBD was performed in order to systematically investigate the nucleation and growth mechanism in the early stages of ZnO NRs synthesis. Fig. 9 schematically shows the nucleation and growth processes of ZnO NRs and FESEM images are used to confirm these graphics (see the supplementary section, Fig. S4). First, ZnO seed layers are deposited on bare glass substrate. In the CBD process which is the second stage of ZnO NRs synthesis, the wrinkled inter-layer is formed on ZnO NPs. Finally, ZnO NRs are grown on the interface layer. In addition, a low concentration CBD process is carried out so as to verify the presence of wrinkled inter-layers (see the supplementary section, Fig. S5). Randomly distribution of the (002) polar planes which are the suitable nucleation sites on the ZnO seed layers is the proposed mechanism explaining why these wrinkled inter-layers are formed on the ZnO seed layer before the NRs growth in the chemical bath. Consequently, some particles are more suitable for ZnO nuclei to deposit. Then, it is sensible to have such a wrinkled inter-layer before NRs growth in the CBD process. All things considered, it is predicted that these wrinkled inter-layers form on every kind of ZnO seed layers before NRs growth. It is worth saying that insufficient activation energy for NRs growth should be provided in order to detect the presence of this phenomenon. The growth of ZnO NRs can be inhibited by decreasing the CBD time, the CBD temperature, and the precursor concentration. In the final analysis, the structural and morphological properties of ZnO seed layers can modify the wrinkled inter-layer and hence NRs properties.

### 4. Conclusions

In order to investigate the influence of the seed layer on the structural and morphological properties of ZnO NRs, different ZnO thin films have been synthesized by means of changing sol-gel parameters including, the precursor concentration, type of solvent, and type of additive. The importance of different structural and morphological characteristics of ZnO seed layers on the NRs properties is discussed. Results show that four main parameters control the structural characteristics of ZnO NRs. The RI factor of the seed layer, aspect ratio, density, and alignment of ZnO NRs are all proportional to the NRs RI factor. Besides, seed layer morphological properties include NPs size and density control the dimension, alignment, and density of the NRs. Further analysis of NRs morphological properties confirms that it is possible to change the aspect ratio of ZnO NRs just by changing seed layers. The longest and the shortest NRs were grown on the seed layers.
Fig. 8. FESEM images of ZnO seed layers and NRs for different additives; (a, b) MEA, (c, d) DEA, (e, f) TEA.
synthesized by the sols containing DEA and 1-BuOH, respectively. Finally, a proposed mechanism is posited about the presence of wrinkled inter-layer and confirmed by short time and low precursor concentration CBD and it is predicted that this interface is affected by the seed layer and hence control ZnO NRs properties.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.ceramint.2016.06.098.

References


Fig. 9. Nucleation and growth processes of ZnO NRs together with FESEM images of (a, b) ZnO seed layer, (c, d) Wrinkled inter-layer, (e, f) ZnO NRs.