ZnO nanorod arrays prepared by chemical bath deposition combined with rapid thermal annealing: structural, photoluminescence and field emission characteristics

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Abstract

ZnO nanorod arrays were prepared by low temperature chemical bath deposition (CBD) combined with rapid thermal annealing (RTA) under different ambient conditions. The structure and morphology of the synthesized ZnO have been characterized by field-emission scanning electron microscopy (FESEM) and x-ray diffraction (XRD). The obtained ZnO samples are highly crystalline with a hexagonal wurtzite phase and also display well-aligned array structure. A pronounced effect on increased nanorod length was found for the RTA-treated ZnO as compared to the as-grown ZnO. Analysis of XRD indicates that the (0 0 2) feature peak of the as-grown ZnO was shifted towards a lower angle as compared to the peaks of RTA-treated ZnO samples due to the reduction of tensile strain along the c-axis by RTA. Photoluminescence (PL) studies reveal that the ZnO nanorod arrays receiving RTA in an O₂ environment have the sharpest UV emission band and greatest intensity ratio of near band-edge emission (NBE) to deep level emission (DLE). Additionally, the effects of RTA on the field emission properties were evaluated. The results demonstrate that RTA an O₂ environment can lower the turn-on field and improve the field enhancement factor. The stability of the field emission current was also tested for 4 h.

Keywords: ZnO nanorod, chemical bath deposition, rapid thermal annealing, field emission, tensile strain

(Some figures may appear in colour only in the online journal)
emitters. Lately, intense research efforts have focused on the preparation of high conductivity, high crystallinity, well-aligned, and metal-ion doped ZnO nanostructures for the improvement of the field emission characteristics. Al-doped ZnO (AZO) nanowires with a controllable length of ~1 μm and a diameter of ~100 nm have been synthesized using the hydrothermal method. The resulting turn-on field \((E_{\text{to}})\) and the field enhancement factor \(\beta\) was reported to be 2.17 V μm\(^{-1}\) and 3131, respectively [10]. Large-scale, homogeneous, dense ZnO nanowires were hydrothermally grown on porous anodic aluminum oxide (AAO) templates [11], and the well-aligned ZnO nanowires with \(L = 1600\) nm and \(D = 30\) nm demonstrated a \(\beta\) value of 6100 and low \(E_{\text{to}}\) value of 1.48 V μm\(^{-1}\). Fang et al [12] synthesized ZnO : Al nanostructures as emitters by thermal evaporation at 650 °C, resulting in an \(E_{\text{to}}\) of 3.8 V μm\(^{-1}\) and a \(\beta\) value that can reach ~6320. Ga-doped ZnO nanowires have been synthesized by thermal evaporation, and an \(E_{\text{to}}\) value of 3.4 V μm\(^{-1}\) and large \(\beta\) value of ~5900 were reported [13]. Lian et al [14] have synthesized tapered ZnO nanostructures onto vertically aligned carbon nanotubes (CNT) using thermal CVD. Their results showed that ZnO-coated CNTs have enhanced field emission and emission current stability. However, the experimental values of \(E_{\text{to}}\) and \(\beta\) were not given in their study. Ordered CNT-coated ZnO heterojunction arrays have been prepared by the vapor-phase transport (VPT) method resulting in an \(E_{\text{to}}\) (~2.8 V μm\(^{-1}\)) and \(\beta\) (~5482) values that were significantly improved as compared to the values of the randomly ordered ZnO nanowires [15]. Zhao et al [16] fabricated three kinds of ZnO nanorod arrays with different tip morphologies by vapor phase growth, finding that the nanoneedle arrays have the highest \(\beta\) value (1464) owing to the small radius and the smallest screening effect, but the stability test has not been carried out [15, 16]. Hsiao et al [17] summarized the \(E_{\text{to}}\) and \(\beta\) of ZnO and doped-ZnO nanostructures with various morphologies, in which \(E_{\text{to}}\) and \(\beta\) values ranged from 3.5 to 11.2 and from 870 to 9050, respectively. We note that the ZnO nanostructures prepared by high-temperature processes usually result in inevitable deep level emission (DLE) associated with the presence of structural defects [18], which somehow affect the performance of the field emission devices.

Several research studies have been conducted to prepare the high-conductivity ZnO nanostructures with suppressed defect-related emission in the visible region by hydrothermal growth [10], post-deposition heat treatment [19], or post-deposition annealing [20, 21]. In reference [20], the effects of RTA temperature and annealing, in Ar, N\(_2\), O\(_2\) and air, on the ZnO thin films by pulsed laser deposition (PLD) are presented.

Figure 1. FESEM images of the as-grown ZnO nanorod arrays: (a) surface morphology and (b) cross-section. The distribution histograms for the diameter (c) and the length (d) were obtained by averaging 50 nanorods in SEM images.
The analyses of XRD and photoluminescence (PL) revealed that the ZnO that received RTA at 700 °C in Ar demonstrated strong UV to visible emission; however, at a lower annealing temperature (200–400 °C) or annealing in air at a temperature higher than 600 °C yielded no annealing effects. Xu et al [21] investigated the effect of RTA in ambient conditions on the PL properties of ZnO films, and reported that the ZnO films that received RTA (800 °C, Ar) exhibited enhanced near band-edge emission (NBE) accompanied with pronounced deep-level emission (DLE) in the visible region. It was also found that RTA under O2 ambient conditions effectively suppressed the DLE intensity by reducing the oxygen vacancies, but UV emission was significantly degraded, which may be attributed to the native nature of the sputtered-ZnO films [21]. Wang et al [10] studied the temperature effect (300–500 °C) of oxygen annealing for 1 h on thermally-grown Al-doped ZnO nanowires and reported that high temperature annealing could improve the largest UV to visible emission ratio. Optimum field emission characteristics with a low $E_{\text{th}}$ value of 1.70 V μm$^{-1}$ and large $\beta$ value of 4547 were obtained.

In this work, we synthesized high quality and well-aligned ZnO nanorod arrays by a simple and low-temperature chemical bath deposition (CBD) followed by RTA at 400 °C under different ambient gases. The effects of RTA on the crystallinity, strain relaxation, and defect properties were investigated on the basis of SEM, XRD, and PL analysis. Furthermore, we show that the ZnO nanorod arrays which received RTA at O2 ambient exhibit the lowest turn-on field and largest field enhancement factor. The enhanced field emission characteristics are discussed in conjunction with ZnO crystalline and defect properties.

2. Experimental

2.1. Preparation of ZnO nanorod arrays

1D ZnO nanorods were grown by a CBD on indium tin oxide (ITO) coated glass (AUO Co., Ltd) with a sheet resistance of 7 Ω/□. As precursors, 400 ml aqueous solutions containing zinc acetate-2-hydrate [Zn(CH$_3$COO)$_2$·2H$_2$O] (99% purity, Showa)
mixed with Hexamethylenetetramine (C₆H₁₂N₄, HMTA, 99.5% purity, Riedel-de Haen) to obtain a precursor concentration of 0.05 M were prepared, where HMTA was used to serve as a pH buffer by releasing OH⁻ ions [17, 22]. In the first stage, the ITO-glass was dipped in the precursor sol (kept at 25 °C) for 10 s, and then dried in an oven at 100 °C for 10 min to obtain ~200 nm ZnO seeding layer. In the second stage (CBD process), the seeded substrates were vertically positioned in the continuously stirred zinc acetate precursor while heating up to, and maintained at 95 °C, for 1.5 h. Through the CBD reaction, the pH of the solution was adjusted to 7.5 by adding NH₃OH (28 wt%) into the reaction bath. In the third stage, the CBD samples were calcined at 400 °C for 1 h using an electric furnace to obtain as-grown ZnO. Alternatively, the above CBD samples were subjected to rapid thermal annealing (RTA) by an Infrared Lamp Heating System (MILA-5000, ULVAC-RICO Inc.) to obtain the RTA-treated ZnO. The RTA chamber was pumped down to 1 × 10⁻² Torr and then fed with air or O₂ to a final pressure of 6 × 10⁻² Torr. The temperature was then raised to the operating temperature in 30 s and kept fixed at 400 °C for 1 min, finally cooling down to room temperature in 5–6 min using a recirculating bath held at 15 °C.

**Figure 3.** The distribution histograms for the diameter (a), (c), (e) and the length (b), (d), (f) of the RTA-treated ZnO corresponding to the samples shown in figure 2.
2.2. Characterization of ZnO and electrical measurement

The morphology and crystalline structure of the resulting samples were characterized by field emission scanning electron microscopy (FESEM, JEOL JSM-6700F) and x-ray diffractometer (XRD, Bruker D2 Phaser) using CuKα radiation, respectively. The defect and optical properties were investigated using PL spectroscopy (miniPL system, Pneum Japan) at room temperature. The field emission properties of the samples were detected using high-voltage units (Keithley 237), where the ZnO side was set as a cathode emitter and the ITO-glass was set as an anode. The distance between the cathode and anode was ~60 μm and the active emission area was 0.5 × 0.5 cm². The field emission measurement was performed in a vacuum chamber at a pressure of 1 × 10⁻³ Torr at room temperature.

3. Results and discussion

3.1. Characterization of ZnO nanostructure

Figures 1(a), (c) and 2 show the surface and cross-section SEM images of the ZnO nanorod arrays calcinated at 400 °C (as-grown ZnO) and the RTA-treated ZnO under different ambient gas concentrations, respectively. These solid ZnO nanorods exhibit a uniform, clear hexagonal structure, and also show non-polar planes formed by the coalescence mechanism [23]. The nanorods have an average diameter of 71–74 nm; however, the average length of the nanorods increased from ~0.78 μm to 1.18 μm after RTA treatment. The distribution histograms for the diameter and the length of all ZnO nanorods are presented in figures 1(b), (d) and 3. The highest aspect ratio value of ~16.4 was achieved after RTA in O₂ ambient gas concentration. It appears that the ZnO nanorods are still vertically grown on the substrate during RTA, but this argument should be verified with evidence of XRD analysis. Figure 4 shows the XRD patterns of the as-grown and RTA-treated ZnO nanorod arrays on the ITO substrate. The appearance of the (1 0 0), (0 0 2), (1 0 1), (1 0 2), (1 1 0), (1 0 3) and (1 1 2) feature peaks, which is in good agreement with standard ZnO (JCPDS Data Card No: 36–1451) [24, 25], and indicates that our ZnO samples have excellent hexagonal structure. It is noted that the feature peak (1 0 1) becomes sharper for RTA treated in air and O₂ ambient concentrations, indicating that the ZnO wurtzite structure and crystal quality are improved. Meanwhile, RTA treatment can enhance the vertical alignment on the ITO substrate by the observation of a higher (002) peak intensity, also evidenced by the cross-section SEM images (figures 1(c) and 2(b), (d), (f)). Figure 4(b) shows the magnified XRD spectra in the range of 30–40°. It is clear that the (002) feature peak of the as-grown ZnO is shifted toward a lower angle as compared with the peak of the RTA samples, implying that uniform tensile strain is applied to the nanostructure grains [26].

It is well known that the crystalline size and lattice strain due to dislocation can be determined from the XRD spectra. Here the grain size of the ZnO nanorods was evaluated by the broadening of x-ray diffraction peaks using the Scherrer equation [27]:

\[
d = \frac{k \lambda}{B \cos \theta}
\]

where \(d\) is the grain size in the nanostructure, \(k\) is the Scherrer constant (the shape factor) = 0.9, \(\lambda (=1.54056 \text{ Å})\) is the wavelength of the incident CuKα radiation, \(B\) denotes full-width at half maximum (FWHM) of the respective peak, and \(\theta\) is the Bragg diffraction angle. The lattice constants, \(a\) and \(c\), of our ZnO nanorods can be calculated from the (002) peak position using Bragg’s Law. The strain-tensile along the \(c\)-axis denoted by \(\varepsilon_c\) can be calculated by the following equation [28, 29]:

\[
\varepsilon_c = \frac{c - c_0}{c_0} \times 100
\]

where \(c\) and \(c_0\) are the lattice constants of our ZnO samples and standard lattice constants of unstrained ZnO (JCPDS 36–1451: \(a = 3.2495 \text{ Å}\) and \(c = 5.2069 \text{ Å}\) [25]), respectively.
The negative $\varepsilon_c$ value indicates compressive strain in the nanostructure, while the positive $\varepsilon_c$ indicates tensile strain [29]. The strain and other structural properties of the ZnO nanorod arrays are summarized in table 1. It is observed that the grain size is increased by RTA as compared to that of the as-grown ZnO, indicating the homogenization and stabilization of the RTA-treated ZnO nanorods [19]. The decrease in lattice constant is due to the fact that the (0 0 2) peak of the RTA-treated ZnO nanostructures is shifted towards higher angles as illustrated in figure 4(b), which means that the ZnO becomes more condensed by the decrease in the distance between planes. Also it is noteworthy that the lattice parameters, $a$ and $c$, of the as-grown ZnO are slightly greater than those of ZnO (JCPDS 36–1451), which indicates that little compressive strain along the $c$-axis exists within the ZnO structure. On the contrary, the lattice constants of the RTA-treated ZnO are well-matched with ZnO (JCPDS 36–1451), and the tensile strain along the $c$-axis can be relaxed by RTA to confirm the high quality of the ZnO nanostructure. As a result, the expansion of the lattice and the possible structural or intrinsic defects as usually seen in ZnO prepared in high-temperature can be avoided [27].

### Table 1. The structure parameters of as-grown and RTA-treated ZnO nanorod arrays.

| Thermal treatment       | Grain size (nm) | Lattice parameter (nm) $c/a$ | Tensile strain |
|-------------------------|-----------------|-------------------------------|---------------|
| As-grown                | 36.4            | $c = 0.5242$ $a = 0.3253$     | 1.611 0.6741  |
| RTA in vacuum           | 37.2            | $c = 0.5217$ $a = 0.3238$     | 1.611 0.1939  |
| RTA in air              | 39.5            | $c = 0.5219$ $a = 0.3238$     | 1.611 0.2324  |
| RTA in O$_2$            | 40.5            | $c = 0.5217$ $a = 0.3239$     | 1.610 0.1939  |

3.2. RTA effect on the PL properties

PL spectroscopy was utilized to investigate the effects of an RTA atmosphere on the optical and defect properties of the ZnO nanostructures. Figure 5 shows the room-temperature PL spectra of the as-grown ZnO and RTA-treated ZnO samples. In general, ZnO nanocrystals exhibit near band-edge
UV emission at a wavelength of 380 nm originating from recombination between free excitons and visible emissions ranged from 500–700 nm due to structural or intrinsic defects. These defects, including oxygen and zinc vacancies or interstitials, would result in a complex PL spectrum, responsible for blue, green, yellow, and red emissions as discussed in [30–32]. It can be seen that the PL spectra for vacuum, air, and O2 are similar and show excellent UV to visible emission. The UV emission (~378 nm) becomes stronger for ZnO nanorods processed by RTA, whereas the sample that received RTA in O2 exhibits the strongest UV emission. In other words, the decrease in the intensity of the UV near-band-edge emission by the as-grown ZnO reflects a decrease in the crystal quality by an increase in the nonradiative recombination from a large number of defect states [33]. The redshift effect as seen in the ZnO branched nanorods [34] is indistinctive for our well-aligned ZnO nanorod arrays. Figure 5(b) further reveals the effect of an RTA atmosphere on the intensity ratio of NBE to DLE peak (\(R = I_{\text{NBE}}/I_{\text{DLE}}\)) and FWHM of NBE peaks corresponding to the PL results from figure 5(a). The R value of as-grown ZnO is around 7.7, which can be improved after RTA treatment and can increase as high as \(R \approx 34.5\) for RTA in O2 atmosphere. This high R value is greater than the R value (~19) of the hydrothermally synthesized Al-doped ZnO under oxygen annealing at 500 °C for 1 h. Meanwhile, RTA enhanced the UV emission by reducing the FWHM of the NBE peak to ~11.8 (in an O2 atmosphere). The PL results suggest that the RTA in an O2 atmosphere effectively improves the structural defects and ZnO crystallinity [10]. The effect of oxygen partial pressure on the PL spectra was not further studied since the direct growth and structural relaxation were insensitive to oxygen partial pressure [20]. It is noted that the R value and the FWHM of the RTA-treated ZnO (in air) is smaller than those of the ZnO received RTA in a vacuum and O2 due to the larger tensile strain (see table 1) as also mentioned in the RTA-treated ZnO thin films [20].

### 3.3. Field emission characteristics and current stability

The field emission properties of all ZnO nanorod arrays have been studied by the Fowler–Nordheim (F–N) theory. Consequently, the relationship between the current density \(J\) and the applied field strength \(E\) can be depicted as:

\[
J = A\beta^2 E^2/\phi \exp(-B\phi^{3/2}/\beta E)
\]

where \(A = 1.56 \times 10^{-10} (\text{A eV V}^{-2})\), \(B = 6.83 \times 10^3 (\text{V (\mu m eV}^{3/2})^{-1})\), \(\beta\) is the field enhancement factor, \(E (=V/d)\) is the applied field, and \(\phi\) is the work function of the ZnO nanostucture. It is well accepted that the turn-on field \(E_{\text{to}}\) and threshold field are defined as the electric field required for establishing a current density of 1 \(\mu\text{A cm}^{-2}\) and 1 \(\text{mA cm}^{-2}\), respectively. The field emission from the ZnO nanorod arrays is shown in figure 6 as a function of applied electric field versus emission current density. From figure 6(a), the \(E_{\text{to}}\) was found to be 5.27, 2.76, 1.47, and 0.47 \(\mu\text{A cm}^{-2}\) for as-grown ZnO and RTA-treated ZnO (in vacuum, air and O2), respectively. Figure 6(b) shows the F–N plot, i.e. \(\ln(J/E^2)\) versus \(1/E\), derived from the \(J–E\) curves in figure 6(a). The values of \(\beta\) estimated from the slope of the F–N plot are 1266, 4096, 2222, and 5853, respectively. The ZnO nanorod arrays, which received RTA in O2 exhibits low \(E_{\text{to}}\) and large \(\beta\), which are superior to those of the Al-doped ZnO nanowires [10] and comparable to the complex CNT-coated ZnO heterojunction arrays [15]. The field emission characteristics of our ZnO nanorod arrays and other ZnO nanostructures [10, 12, 14–16, 32, 35, 36] are summarized in table 2. Figure 7 shows the typical emission current stability of the as-grown and RTA-treated ZnO (in O2) measured at a preset current density of 1.0 mA cm\(^{-2}\). After stability testing for 4h, the drift rate of the as-grown ZnO is around 0.67 \(\mu\text{A cm}^{-2}\) min\(^{-1}\), which is relatively higher than that (0.54 \(\mu\text{A cm}^{-2}\) min\(^{-1}\)) of the RTA-treated ZnO (in O2). In addition, as seen in figure 8, the as-grown ZnO has larger fluctuations in the emission current compared with the RTA-treated ZnO (in O2) during the stability test. The stability improvement can be attributed to a more thermally stable,
rigid structure [15] and less tensile strain after RTA. In summary, ZnO nanorod arrays by RTA in O2 demonstrated excellent field emission and stability due to the enhanced vertical alignment, less tensile strain, and fewer oxygen defects, indicating the potential use in display technology for high-brightness applications.

4. Conclusion

Vertically, well-aligned ZnO nanorod arrays have been grown on ITO substrate by low temperature CBD combined with RTA treatment at 400 °C for 1 min in different atmospheres. It was observed that all ZnO samples demonstrated hexagonal wurtzite structure from FESEM images and XRD feature peaks. The average length \( L \sim 1.18 \mu m \) of the RTA-treated ZnO was larger than that of the as-grown ZnO \( L \sim 0.78 \mu m \), meanwhile the XRD (002) peak shifting and calculated strain confirmed that the tensile strain along the c-axis can be effectively reduced by RTA under either vacuum, air, or O2 atmospheres. PL spectra revealed that the ZnO nanorod arrays, which received RTA in an O2 atmosphere have the highest intensity ratio of near-band emission to deep-level emission due to the inhibition of oxygen defects. The pronounced UV emission and sharpest FWHM\(_{NBE}\) confirmed the improvement of ZnO crystallinity. Furthermore, the ZnO that received RTA in an O2 atmosphere exhibits low turn-on field \( E_{to} \) of 0.47 V \( \mu m \) \(^{-1} \) and large \( \beta \) value of 5853, and also shows greater stability of the emission current with a duration of 4h. The results demonstrate that the CBD-derived ZnO nanorod arrays followed by RTA in O2 possess excellent potential for use in display technology for high-brightness applications.

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