



Synthesis and field electron emission properties of waste cooking palm oil-based carbon nanotubes coated on different zinc oxide nanostructures



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ABSTRACT

Carbon nanotubes (CNTs) from waste cooking palm oil were synthesised on different zinc oxide (ZnO) nanostructures including nanorods (ZNRs), nanoflowers (ZNFs) and nanorods-nanoflakes (Al:ZNRs-FLs) via thermal chemical vapour deposition method. The ZnO nanostructures were controllably synthesised by sonicated sol–gel immersion method. The morphologies and crystal structures of the nanostructures were observed using field emission scanning electron microscopy, photoluminescence spectroscopy, X-ray diffractometer and micro-Raman spectroscopy. The diameter and density of CNTs were affected by the presence of ZnO nanostructures. Moreover, the morphology of ZnO nanostructures was modified during the synthesis of CNTs. The presence of ZnO reduced the barrier layer between CNTs and substrate, thus enhanced the field electron emission (FEE) properties of CNTs. Among the ZnO nanostructured prepared, the growth of CNTs on ZNFs gave the best FEE performance with the lowest turn-on field (0.8 V/μm at 1 μA/cm²). The ZNFs trapped more iron (Fe) elements and promote the tip and body-emission processes in the sample. Moreover, the presence of Fe elements also reduced the work function of ZNFs/CNTs nanocomposite and acted as additional electrons. Therefore, the significant FEE performance enhancement was observed.

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1. Introduction

In the recent years, studies on the electrical properties of carbon nanotubes (CNTs) have attracted much attention in view of their application in field electron emission (FEE) devices due to their high conductivity, high aspect ratio, high chemical and mechanical stabilities [1–7]. The shape-modification of CNTs with a low threshold electric field of 3.75 V/μm, high field emission current density of 1.6×10^{-5} A/cm² and good emission stability has been reported [8]. Extremely low turn-on and threshold fields of 0.33

and 0.48 V/μm respectively were obtained by fabricating CNTs yarn [9]. Nevertheless, low resistance at oxygen ambience [10] and weak bonding between CNTs and substrate [11] imposed the commercially available electron field emitter based on CNTs. Many attempts were conducted in order to improve the FEE performance of CNTs such as substrate and tip modifications [12], nitrogen plasma treatment [13] and deposition temperature controlling [14]. However, the presented methods are less effective in terms of cost and procedures since the sophisticated equipment and complex procedures are required.

Recently, the researchers have focussed their attention on the synthesis of carbon nanocomposites due to their improvement of excellent physical, chemical and mechanical properties [15–19]. Combination of metallic CNTs and semiconducting zinc oxide (ZnO)

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nanostructures led to the semiconducting/metallic heterojunction which was very useful in nanodevice applications. ZnO itself has become one of the intensive studied materials because of its special properties such as wide band gap (~3.37 eV), high conductivity, good electron mobility and large exciton binding energy (60 meV) [20]. As known that the carbon based nanostructure acted as an electron acceptor while the ZnO nanostructure served as electron donor, the combination of CNTs and ZnO resulted in adorable advantages [21]. Moreover, one dimensional ZnO nanorods (ZNRs) with high aspect ratio were also suitable as the direct template for the growth of tubular nanostructures [22]. Yan et al. [23] reported a notable effect in utilizing CNTs/ZnO nanocomposite as an emitter compared to those on pristine CNTs and ZnO. In their study, the turn-on field of CNTs/ZnO nanocomposite was 1.80 V/ μm , much lower than pristine CNTs (2.80 V/ μm) and ZnO (5.30 V/ μm). Pan et al. also reported a lower turn-on field of CNTs (1.17 V/ μm) after composited with ZnO [24]. The improvement of FEE performance with lower turn-on field and higher current emission were resulted due to a better ohmic contact between CNTs and ZnO [24,25]. Moreover, the presence of ZnO avoided the spot welding of CNTs at the nanometre contact area due to the local joule heating [23] as well as enhanced the adhesion between CNTs and the substrate [11,26]. The additional of CNTs into ZnO nanostructures was also reported to successfully enhance its FEE properties as well as facilitated the growth of ZnO nanoflowers (ZNFs) on ZNRs [27]. Table 1 presents the previous related works on the FEE study of nanocomposited ZnO and CNTs.

Furthermore, as the morphology of nanostructure influenced its FEE performance, the controlling of its morphology became a crucial aspect [31]. Diverse morphologies of ZnO nanostructures have been synthesised through gas and aqueous thermal deposition [32–37]. Among the presented methods, the use of aqueous-based synthesis process was simple, greener, relatively low temperature and pressure as well as less expensive. Moreover, through this approach, the morphology of ZnO nanostructures was easily tailored by controlling the deposition parameters such as temperature, pH, deposition time and molarity [38–40]. Comparison studies on FEE properties of different ZnO nanostructures, including ZnO nanowires, nanocones and microspheres [31] and balls, nunchakus and belts [41] have been reported and they showed significant distinction on FEE characteristics. However, direct comparison studies on the effect of different ZnO nanostructures/CNTs nanocomposites on their FEE properties are rarely reported. Previously, the growth of nanostructured ZnO/CNTs nanocomposites showed different FEE characteristics [42–44]. Therefore, the investigation on the deposition of CNTs grown on different ZnO nanostructures is still a challenge.

Here we report a comparative investigation on FEE properties of CNTs grown on ZnO nanostructures. For the first time, CNTs synthesised from waste cooking palm oil (WCPO) were grown on different morphology of ZnO which are ZnO nanorods (ZNRs), nanoflowers (ZNFs) and nanorods-nanoflakes (Al:ZNRs-FLs). Compared to the previous studies on the synthesis of nanocomposited ZnO and CNTs, this study offers a low cost and simpler

technique to fabricate and enhance the field emission of ZnO/CNTs nanocomposites. The ZnO nanostructures were synthesised via sonicated sol–gel immersion method under several controlled modifications. Meanwhile, natural WCPO precursor was used for the synthesis of CNTs using thermal chemical vapour deposition (TCVD) method. The structural and optical properties of the samples were characterised and the emission properties were also investigated.

2. Experimental

The synthesis of ZnO/CNTs nanocomposites were conducted in multi-step deposition process involving deposition of seeded catalyst, growth of ZnO nanostructures via sonicated sol–gel immersion method and synthesising CNTs from WCPO by TCVD method. The overall deposition process is summarized in the flow chart as presented in Fig. 1.

2.1. Synthesis of ZNRs and ZNFs on magnesium zinc oxide seeded catalyst layer

Detail preparation for the growth of ZNRs and ZNFs on magnesium zinc oxide (MgZnO) seeded catalyst layer has been reported elsewhere [45]. The sol–gel was prepared by dissolving zinc acetate dihydrate ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$), magnesium nitrate hexahydrate ($\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) and mono-ethanolamine ($\text{C}_2\text{H}_7\text{NO}$) into 2-methoxyethanol. The precursors were sonicated in ultrasonic water bath (Memmert, operate at 230 V, 50–60 Hz and 2000 W) for 30 min at 50 °C and stirred at room temperature for 2 h. MgZnO seeded catalyst was obtained via depositing the MgZnO solution on silicon (Si) substrate using spin coating method operated at 3000 rpm for 60 s. The ZNRs and ZNFs were then grown on MgZnO seeded catalyst layer. The ZnO solution was prepared by dissolving zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) and hexamethylenetetramine ($\text{C}_6\text{H}_{12}\text{N}_4$) (HMT) into deionized water. The ZnO precursor was also sonicated and stirred similar with the previous MgZnO solution. MgZnO-coated substrates were then immersed into ZnO solution by placing at the top, facing downward, to produce ZNRs and at the bottom of the solution, facing upward, to grow ZNFs. The synthesis was carried out for 4 h at 95 °C in water bath. The annealing process was then conducted at 500 °C for an hour.

2.2. Synthesis of Al:ZNRs-FLs on aluminium zinc oxide seeded catalyst layer

The preparation of Al:ZNRs-FLs on aluminium zinc oxide (AlZnO) seeded catalyst layer was similar as described elsewhere [46,47]. The aluminium nitrate nonahydrate ($\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$) was added in sol–gel ZnO prepared from zinc acetate dihydrate, mono-ethanolamine, and 2-methoxyethanol. They were initially sonicated for 30 min at 50 °C, stirred and aged for 3 h at room temperature. The aluminium nitrate nonahydrate was also added as a dopant in the ZnO solution. The AlZnO-coated Si substrate was then immersed in the ZnO solution by placing at the bottom of the

Table 1
Previous related works on the FEE performances of nanocomposited ZnO and CNTs.

No.	Turn on field	Max. current density	References
1.	2.2 V/ μm at 10 $\mu\text{A}/\text{cm}^2$	~340 $\mu\text{A}/\text{cm}^2$	Carbon/ZnO nanocomposites by CO_2 laser ablation by Kaushik et al. [21].
2.	2.8 V/ μm at 10 $\mu\text{A}/\text{cm}^2$	~8 mA/cm^2	CNTs/ZnO heterojunction arrays by Yan et al. [23].
3.	2.8 V/ μm at 1 mA/cm^2	~1 mA/cm^2	Surface fluorinated ZnO/CNTs by Wang et al. [25].
4.	3.7 V/ μm at 10 $\mu\text{A}/\text{cm}^2$	~300 $\mu\text{A}/\text{cm}^2$	Highly stable field emission of ZnO/CNTs by Patra et al. [28].
5.	1.85 V/ μm at 10 $\mu\text{A}/\text{cm}^2$	218 $\mu\text{A}/\text{cm}^2$	ZnO/CNTs composites via DC electrophoresis by Min et al. [29].
6.	0.31 V/ μm at 10 $\mu\text{A}/\text{cm}^2$	N/A	ZnO/CNTs composites via microwave plasma jet CVD by Su et al. [30].
7.	4.6 V/ μm at 1 $\mu\text{A}/\text{cm}^2$	~110 $\mu\text{A}/\text{cm}^2$	Enhanced field emission of ZnO grown on CNTs by Suriani et al. [27].

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