Field Emission Properties of SiO₂-wrapped CNT Field Emitter

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Abstract

Carbon Nanotubes (CNTs) exhibit unstable field emission (FE) behaviour with low reliability due to uneven heights of as-grown CNTs. It has been reported that mechanically polished SiO₂-wrapped CNT field emitter gives consistent FE performance due to its uniform CNT heights. However, there are still lacked of studies on the comparison between the FE properties of freestanding and SiO₂-wrapped CNTs. In this study, we have performed a comparative study on the FE properties of freestanding and SiO₂-wrapped CNT field emitters. From the FE measurements, freestanding CNT field emitter requires lower applied voltage of 5.5 V/µm to
achieve FE current density of 22 mA/cm$^2$; whereas SiO$_2$-wrapped field emitter requires 8.5 V/µm to achieve the same current density. This can be attributed to the lower CNT tip electric field of CNTs embedded in SiO$_2$, as obtained from the electric field simulation. Nevertheless, SiO$_2$-wrapped CNTs show higher consistency in FE current than freestanding CNTs. Under repeated FE measurement, SiO$_2$-wrapped CNT field emitter achieves consistent FE behaviour from the 1$^{st}$ voltage sweep, whereas freestanding field emitter only achieved consistent FE performance after 3$^{rd}$ voltage sweep. At the same time, SiO$_2$-wrapped CNTs exhibit better emission stability than freestanding CNTs over 4000s continuous emission.

Introduction

The electronics era begins with the invention of first vacuum tube at the beginning of 20$^{th}$ century as a pioneering prototype for today’s solid-state modern transistor. With the appearance of vacuum tubes, wireless communication, computing and many other electronic functional devices became feasible [1]. As the electronics technology progresses, electronic devices have been downsizing in orders of magnitude over the past decades, reaching above $10^{10}$ transistors per die in today’s era [2]. However, it is believed now that the downsizing will reach its limit in several years due to the reliability and functionality issues as the devices scaled down to nanometre size [3]. To resolve this possible limitation, it has been proposed by NASA that vacuum electronic devices can be a possible better alternative to replace the conventional solid-state transistors [4], due to its high electron mobility and operating frequency range [5–7]. Among various vacuum electronic devices, Traveling-Wave Tube Amplifiers (TWTAs) gained much attention due to its high operation frequency and wide bandwidth [8,9]. To achieve compatibility with solid-state modern transistors, miniaturization of vacuum devices is crucial [4,10]. In the effort of miniaturizing TWTAs, a Planar-Helix Slow-wave Structure (PH-SWS)
has been proposed and patented by our research group, which can greatly reduce the size of the SWS to sub-millimetre scale [11–13]. However, to utilize this PH-SEC, a nanostructure-based, miniaturized field emitter is needed for the operation of miniaturized TWTAs.

It has been reported that Carbon Nanotubes (CNTs) show excellent field emission (FE) properties, attributed to its unique atomic structure and exceptionally high aspect ratio [14,15]. The achievement of high FE current density [16] with relatively low turn-on voltage [17] of CNTs give promising potentials in various vacuum microwave tubes [18,19], including the abovementioned miniaturized TWTAs. To realize these applications, CNT field emitters with consistent FE performance are desired. However, it has been reported that CNTs show unstable FE properties with low reliability [20]. From the reported studies, several factors can lead to unstable field emission, including heat-induced damages, presence of residual gases, and uneven field distribution. Heat induced damages can be attributed to resistive heating from poor CNT-substrate adhesion [21], or high FE current flow in CNTs [22]. The *in-situ* heating of CNTs during FE can cause structural degradation in CNTs which leads to unstable emission [23]. At the same time, the presence of residual gas molecules in the FE chamber may be ionized by the emitted electrons [24], resulting on CNTs which will cause irreversible damages to the CNT emitter [25].

Besides the abovementioned factors, a possible failure mechanism of CNT field emitter is the uneven distribution of electric field on CNTs [26], due to the non-uniform heights of as-grown CNTs. From a reported simulation study, the non-uniformity of CNT heights in a CNT field emitter will lead to uneven distribution in localized electric field on the CNTs, causing overloaded CNTs to burn off [27]. Even in a well-controlled Chemical Vapor Deposition CNT
growth, the CNT heights follow Gaussian distribution with standard deviations of up to 6.3%, which may cause CNTs to overload and burn off [28]. To achieve uniform CNT height, Sun et al. demonstrated the fabrication of CNT field emitter with coaxial gate. In the reported study, uniform CNTs are achieved by conformal deposition of SiO$_2$ layer on as-grown CNTs, followed by SiO$_2$ polishing and dry etching to outcrop the protruding SiO$_2$-wrapped CNT tips. The obtained CNT field emitter shows highly consistent FE properties, attributed to the uniform CNT height [29]. However, to fully utilize the SiO$_2$-wrapped CNTs in FE application, comprehensive understanding on the effect of SiO$_2$ deposition on the FE properties of CNTs is needed. But, despite promising FE properties shown by SiO$_2$-wrapped CNTs reported by Sun et al., the comparison between the FE properties of SiO$_2$-wrapped CNTs and freestanding CNTs is not fully explored. Therefore, comparative studies on the FE properties of freestanding and SiO$_2$-wrapped CNTs are called for.

In this study, we have performed a comparative study between the FE properties of SiO$_2$-wrapped CNT and freestanding CNT field emitters. To fabricate SiO$_2$-wrapped CNTs, SiO$_2$ is conformally deposited onto the pre-grown freestanding CNTs, followed by mechanical polishing to form SiO$_2$-wrapped CNTs with even heights. After that, FE measurements are carried out repeatedly on both freestanding and SiO$_2$-wrapped CNTs. In addition to the fabrication and measurement, we have performed electric field simulation on freestanding and SiO$_2$-wrapped CNT models to investigate the electric field distributions on both CNTs,

**Experimental Method**
The fabrication steps for SiO$_2$-wrapped CNTs are designed based on the process reported by Sun et al. [29]. Figure 1 shows the fabrication process of the SiO$_2$-wrapped CNT arrays. First, 30 nm Ti and 50 nm TiN layers are sputtered sequentially on the highly n-doped silicon substrate (thickness: 300 µm; resistivity: 0.018 Ω.cm) (Figure 1(a)). After that, 20 nm Ni catalyst dots are patterned on the TiN layer by means of photolithography (Figure 1(b)). CNTs are then grown on the pre-patterned catalyst dots by Plasma-enhanced Chemical Vapor Deposition (PECVD) technique. By tuning the PECVD growth parameters (growth temperature, growth duration, plasma power etc.), CNTs of 2~3 µm height are obtained (Figure 1(c); Figure 6(a) and (b)). 2 µm SiO$_2$ is then conformally deposited onto the CNTs by means of PECVD technique, forming protruding SiO$_2$-wrapped CNTs (Figure 1(d); Figure 7(a) and (b)). After that, sequential sputtering of 50 nm Ti adhesive layer followed by 200 nm Au layer is carried out (Figure 1(e)). The Ti/Au layers serve as the protective layer for SiO$_2$ in the polishing step; and shadow mask in the dry etching step. Then, the protruding SiO$_2$-wrapped CNTs are removed by mechanical polishing technique (Figure 1(f)). The mechanical polishing
is carried out using sandpaper of 1000-, 6000-, and 8000-grit, sequentially. By using the Ti/Au layers as the shadow mask, the revealed SiO$_2$ layer is dry etched for ~200 nm (CF$_4$/CHF$_3$ etchant, 170 W power under 150 mTorr pressure, etch rate ~30 nm/min) to reveal the CNT tips (Figure 1(g); Figure 7(c) and (d)).

The morphologies of the CNTs are characterized by Scanning Electron Microscope (SEM, JEOL JSM-IT100) and Transmission Electron Microscope (TEM, Phillips Tecnai 20). The field emission characteristics of the CNT field emitters are measured using a planar-anode measurement system under high vacuum condition (10$^{-6}$ Torr). The measurement is carried out using 20 Hz pulsed voltage source with duty cycle of ~50%. The cathode-anode distance is estimated to be 100 µm, and the effective emission area is estimated to be rectangular with dimension of 1.25 x 0.6 mm$^2$.

**Results and Discussion**

**A. Electric Field Simulation of Freestanding and SiO$_2$-wrapped CNTs**

To investigate the electric field distribution on freestanding and SiO$_2$-wrapped CNTs, we have simulated the electric field on single freestanding CNT and CNT embedded in SiO$_2$ using CST STUDIO SUITE®. We have built two simplified models to compare the electric field distribution on CNT tip in freestanding and SiO$_2$-wrapped condition, as shown in Figure 2(a) and (b). In the simplified models, the CNT height and diameter are fixed to be 2 µm and 100 nm, as obtained from the SEM and TEM images in Figure 6. External electric field of 10 V/µm is applied on both models (Figure 2(a) and (b)), and the resulted electric field distribution is shown in Figure 2(c) and (d).
To determine the influence of SiO₂ wrapping on the localized electric field on CNT tips, we have plotted the localized electric field on the CNT tips along the radial direction (x-direction as described in Figure 2), as shown in Figure 3. Generally, freestanding CNT shows higher localized electric field on CNT tips as compared to CNT embedded in SiO₂. The possible causes for the higher electric field on freestanding CNTs include space charge effect [30,31] and the higher permittivity of SiO₂ [32]. Since SiO₂ has higher relative permittivity (εᵣ = 4) than vacuum (εᵣ = 1) [33], the space charge effect between the SiO₂-CNT interface results in lower potential distribution [34]. As a result, the localized electric field on free-standing CNT tip is higher than CNT embedded in SiO₂. Besides the lower electric field by SiO₂ surroundings, we have observed that the higher electric field of freestanding CNT over SiO₂-wrapped CNT is more prominent at the edges of the CNT tips than the body of the tips. Preliminary, it can be inferred that the SiO₂ wrapping gives relatively lower influence on the electric field distributed on the body of the CNT tip as compared to the edges.
Figure 2: Simplified model of (a) freestanding single CNT, and (b) single CNT embedded in SiO$_2$; electric field distribution of (c) freestanding CNT, and (d) CNT embedded in SiO$_2$.

Figure 3: Localized electric field on freestanding and SiO$_2$-wrapped CNT tips

To further investigate the effect of SiO$_2$ wrapping on the CNTs, two models with 5×5 arrays of freestanding CNTs and CNTs embedded in SiO$_2$ are created, as shown in Figure 4. The CNT
height and diameter are remained to be 2 µm and 100 nm as indicated in Figure 2, and the separation distance between adjacent CNTs is fixed at 100 nm. Similar to the single CNT models shown in Figure 2, external electric field of 10 V/µm is applied on both models (Figure 4(a) and (b)). The resulted electric field distribution is shown in Figure 5(a) and (b). The localized electric field on the CNT tips along the x-direction is plotted as shown in Figure 5(c).

![Figure 4: Simplified model of 5×5 (a) freestanding, and (b) SiO2-wrapped CNT array](image)

From Figure 5(c), it is found that the freestanding CNT array shows much higher electric field at the edges as compared to SiO2-wrapped CNT array. On the other hand, both SiO2-wrapped and freestanding CNT arrays show similar electric field on the tips of the CNTs located at the middle of the 5×5 array. This aligns with the simulated electric field as discussed in Figure 3, where SiO2 wrapping gives more significant influence on the electric field on the edges of the tips than the bodies. For freestanding CNTs, the higher electric field on the CNT array edges can be attributed to the electric field concentration on the edges of a conductor, as reported by...
Sajanlal et al [35]. This agrees well with Liu et al., where the CNTs at the edge of a CNT film have maximum localized electric field while the inner CNTs have reduced electric field due to field screening [36]. However, for CNTs embedded in SiO$_2$ layer, the electric field concentration reduces at the edges of the array, possibly due to the higher relative permittivity of SiO$_2$, as discussed earlier. Nevertheless, from the obtained simulation results, it can be postulated that SiO$_2$-wrapped CNTs exhibit lower FE current density than free-standing CNTs due to the lower field enhancement [37]. To justify this postulation, we have performed FE characterizations on the fabricated freestanding and SiO$_2$-wrapped CNT field emitters, which will be discussed in the next section.

Figure 5: Electric field distribution of (a) freestanding CNTs, and (b) CNTs embedded in SiO$_2$; (c) Localized electric field on freestanding and SiO$_2$-wrapped CNT tips
B. Fabrication and FE Characterizations of Freestanding and SiO\textsubscript{2}-wrapped CNT Field Emitters

Figure 6: (a, b) SEM and (c, d) TEM images of freestanding CNTs

Figure 6 shows the SEM and TEM images of freestanding CNTs. As shown in Figure 6(a), CNT array of 2–3 µm CNT heights are grown using the PECVD technique. At higher magnification, it can be observed that CNTs are sparsely-grown, as observed in Figure 6(b). Visible gaps can be seen between CNTs, forming an empty space within the CNT bundle. This can be attributed to the sparse nature of PECVD-grown CNTs, agreeing well with the outcomes reported by Patra et al. [38]. From Figure 6(b), the diameters of the CNTs are measured as 100–150 nm. For further investigation on the morphological properties of the CNTs, we have
performed TEM analysis on the freestanding CNTs. As shown in Figure 6(c), multi-walled, tubular structure of freestanding CNTs with outer diameter of ~100 nm can be seen. The obtained diameter in TEM images aligned with the measurements obtained in Figure 6(b). Metal cluster found on the closed CNT tips shown in Figure 6(d) indicating the tip-growth mechanism of CNTs [39], as illustrated in Figure 1(c). The obtained CNT morphology is similar to the TEM images reported by Baro et al. [40], which further justifies the formation of CNTs by PECVD process.

Figure 7: SEM images of SiO$_2$-wrapped CNTs (a, b) before, and (c) after mechanical polishing; (d –f): Magnified view of SiO$_2$ windows
Figure 7 shows the SEM images of SiO$_2$-wrapped CNTs before (Figure 1(d)) and after (Figure 1(g)) mechanical polishing step. As shown in Figure 7(a) and (b), SiO$_2$ is deposited conformally onto freestanding CNTs, forming protruding SiO$_2$-wrapped CNTs. The protruding SiO$_2$-wrapped CNTs are then mechanically polished and dry-etched, forming SiO$_2$ windows as described in Figure 1(f) and (g). The randomized shapes of SiO$_2$ “wrapping” results in the formation of irregular shape SiO$_2$ windows, as shown Figure 7(c). The magnified view of SiO$_2$ windows can be seen in Figure 7(d - f). From the SEM images of the windows, it can be seen that CNT tips are revealed from the SiO$_2$ wrapping after SiO$_2$ dry etching, which is crucial for FE measurement. It is observed that the centre of the CNT bundles is empty. This can be caused by the sparse nature of PECVD-grown CNTs, as presented in Figure 6(b). Besides, it is also possible that the SiO$_2$ layer cracks during the mechanical polishing step, leaving an empty space in the CNT bundle. Nevertheless, it is found that the diameters of the revealed tips are estimated to range from 150 ~ 450 nm, as obtained from Figure 7(d – f). The estimated diameters are much larger than the as-grown CNTs, as presented in Figure 6(b), (c) and (d). The larger diameter of CNTs after SiO$_2$ deposition can be attributed to the conformal deposition (or “wrapping”) of SiO$_2$ layer onto CNTs, which agrees well with the literature studies [41,42].
To investigate the FE properties of freestanding and SiO₂-wrapped CNT field emitters, we have performed repeated FE measurements on both emitters. The FE measurements are repeated by sweeping the voltage from 0 to 900 V for 5 – 6 times until a stable, consistent FE data is obtained. The FE data for both freestanding and SiO₂-wrapped CNT field emitters is shown in Figure 8: FE measurements of (a) SiO₂-wrapped, and (b) freestanding CNT field emitters; (c) FN plots of free-standing and SiO₂-wrapped CNTs (obtained from Sweep 5)
Figure 8. For freestanding CNTs, the FE current shows consistency only after Sweep 3; whereas SiO$_2$-wrapped CNTs show highly consistent FE current values from Sweep 1. This can be attributed to the possible variation in CNT heights in freestanding CNT field emitter, leading to uneven distribution in electric field. As a result, CNTs with high localized electric field will be burnt off before achieving consistent FE current [22,27,43]. On the other hand, SiO$_2$-wrapped CNTs possess better uniformity in CNT heights due to the mechanical polishing (Figure 1(f)) and dry etching (Figure 1(g)) steps. Therefore, SiO$_2$-wrapped CNT field emitter shows consistent FE properties from Sweep 1 onwards.

Under repeated FE measurement, it is found that both freestanding and SiO$_2$-wrapped field emitter show stabilized FE current after Sweep 4. At stabilized FE current, freestanding CNT field emitter shows lower turn-on field of 2.5 V/µm to achieve 0.1 mA/cm$^2$ current density, as compared to 3.5 V/µm by SiO$_2$-wrapped CNT field emitter. At the same time, freestanding CNTs require lower applied electric field of 5.5 V/µm to achieve ~22 mA/cm$^2$ current density, as compared to 8.5 V/µm by SiO$_2$-wrapped CNTs. From the FN plots shown in Figure 8(b), the field enhancement factors ($\beta$) of the CNT emitters can be calculated using the well-established FN theory [44]. The $\beta$ values of freestanding and SiO$_2$-wrapped CNT field emitters are calculated as 1824 and 991, respectively. It is possible that the higher $\beta$ value of freestanding CNTs is attributed to relatively taller freestanding CNTs (2~3 µm) as compared to SiO$_2$-wrapped CNTs (< 2 µm), resulting in higher CNT aspect ratio [38]. From the dry etching step illustrated in Figure 1(g), SiO$_2$-wrapped CNTs can be expected to have shorter height than free-standing CNTs after ~200nm etching. This higher aspect ratio of CNTs can result in lower FE current density, due to lower field enhancement on the CNT tips [45,46]. From the simulation outcomes presented in Figure 3 and Figure 5, it shows that CNTs embedded in SiO$_2$ layer exhibits lower electric field around the edges of the CNT tips. This
agrees well with the obtained FE measurements of freestanding and SiO$_2$-wrapped CNT field emitter, where CNTs embedded in SiO$_2$ shows lower FE current and $\beta$ values due to the lower localized electric field on the tips.

To further investigate the emission stability of SiO$_2$-wrapped CNTs, emission stability tests are carried out on free-standing and SiO$_2$ wrapped CNTs, as shown in Figure 9. The stability test is carried out over a duration of 4000s, similar to the reported studies [22,47]. At the beginning of this emission stability test, the applied electric field on free-standing and SiO$_2$-wrapped CNTs is tuned to 5.5 and 8.5 V/µm, respectively, to obtain similar $\sim$22 mA/cm$^2$ current density for both CNTs as obtained in the earlier FE measurements (Figure 8). It is found that the current density emitter by free-standing CNTs fluctuate from 20~25 mA/cm$^2$ current density from 0s to 100s. This fluctuation at the beginning of emission is similar to the outcomes reported by Thong et al., where a fraction of CNTs are damaged before achieving stable emission current [48]. This damage on CNTs has been suggested to be caused by migration of adsorbed gas molecules and ion bombardment on the emitter surface [49,50]. After the fluctuation, free-standing CNTs experienced stable emission at $\sim$ 22 mA/cm$^2$, in line with the FE measurement presented in Figure 8. From 1000 to 4000s, free-standing CNTs experienced gradual reduction in FE current density, similar to the reported studies [14,23,51]. At 4000s, free-standing CNTs emit lower current density of $\sim$16 mA/cm$^2$ as compared to $\sim$20 mA/cm$^2$ by SiO$_2$-wrapped CNTs. This reduction in current density can be due to the degradation of CNTs during emission [25]. This degradation in CNTs can be attributed to several reasons, including heat-induced damages on the CNTs during emission [51], foreign ionic adsorbates damaging [25,52], which requires further investigation.
On the other hand, SiO2-wrapped CNTs show relatively stable FE current density over 4000s continuous emission, as compared to free-standing CNTs. From Figure 9, it can be seen that SiO2-wrapped CNTs emit lower current density than free-standing CNTs after 4000s continuous emission. As discussed earlier, a possible cause for the emission current fluctuation in CNTs is the migration of adsorbates and ion bombardment on CNTs. In the case of SiO2-wrapped CNTs, it can be speculated that the SiO2 wrapping provides a protection layer to CNT emitters, which makes them less susceptible to ion bombardment. Besides, another possible role of the SiO2 wrapping is to improve the CNTs-substrate adhesion by “anchoring” the CNTs onto the substrate, which can possibly improve the emission stability of CNTs [53,54]. As suggested by Scott et al., CNTs anchored on substrate exhibit better electrical contact and less Joule heating effect [55], resulting in high emission stability. This aligns with our finding presented in Figure 9, where SiO2-wrapped CNTs exhibit better emission stability than free-standing CNTs.
For the sake of comparison, we have compiled the FE properties obtained in the reported literatures with the FE measurements obtained in this study, as shown in Table 1. The CNT field emitter reported in current study has the largest emission area among the investigated literature data. As the miniaturized TWTA design reported by our research group requires CNT field emitter with substantial emission area, larger effective emission area of ~0.75 mm² is selected to optimize the functionality of the TWTA [56,57]. Using similar SiO₂ wrapping technique, Sun et al. reported achievement of 550 µA FE current under 160 V/µm applied electric field; whereas, the achievement of higher FE current by Sun et al. than this present study can be attributed to its smaller CNT diameter and the growth of individual CNT in each SiO₂ window, which further enhances the β value of the CNT emitter. However, as TWTA requires high stability, reliable field emitter, growing multiple CNTs in a single SiO₂ window possibly yields better reliability as CNT emitters are generally fragile and more susceptible to large field-induced failure [58,59].

Table 1: Compilation of the FE current densities obtained in this study with the reported studies

<table>
<thead>
<tr>
<th>CNT Field Emitter</th>
<th>CNT Height</th>
<th>Applied Electric Field @ Current</th>
<th>Cathode Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>This work</td>
<td>~1.8 µm</td>
<td>8.5 V/µm @ 160 µA</td>
<td>0.75 mm²</td>
</tr>
<tr>
<td>Sun et al. [29]</td>
<td>~1.75 µm</td>
<td>160 V/µm @ 550 µA</td>
<td>0.2 mm²</td>
</tr>
<tr>
<td>Scott et al. [55]</td>
<td>10 µm</td>
<td>1.5 V/µm @ 20 µA</td>
<td>0.08 mm²</td>
</tr>
<tr>
<td>Lin et al. [60]</td>
<td>0.8 µm</td>
<td>3.2 V/µm @ 8 µA</td>
<td>0.03 mm²</td>
</tr>
<tr>
<td>Niemann et al. [61]</td>
<td>10 µm</td>
<td>35 V/µm @ 1 µA</td>
<td>0.002 mm²</td>
</tr>
</tbody>
</table>
Conclusion

A comparative study on the field emission (FE) properties of SiO$_2$-wrapped and freestanding CNT field emitter is reported. SiO$_2$-wrapped CNT field emitter is fabricated by conformally depositing SiO$_2$ onto freestanding CNTs, followed by mechanical polishing to remove the protruding SiO$_2$-wrapped CNTs to reveal the CNT tips. From the FE measurements, freestanding CNT field emitter requires lower applied voltage of 5.5 V/µm to achieve FE current density of 22 mA/cm$^2$; whereas SiO$_2$-wrapped field emitter requires 8.5 V/µm to achieve the same current density. At the same time, SiO$_2$-wrapped CNT field emitter exhibits higher field enhancement factor of 1824 than freestanding CNTs of 991. This can be explained by the electric field simulation, where SiO$_2$-wrapped CNT shows lower localized electric field on the CNT tip than freestanding CNT. From the FE measurements, it is found that freestanding CNTs show consistent FE properties after Sweep 3; whereas SiO$_2$-wrapped CNTs show consistent FE properties right from Sweep 1 due to the uniform CNT heights. At the same time, SiO$_2$-wrapped CNTs exhibit better emission stability than freestanding CNTs over 4000s continuous emission. These results provide an insightful view on the FE properties of SiO$_2$-wrapped CNTs, which gives an effective approach to improve the consistency in FE performance of CNTs for its application in vacuum electronic devices.

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