Effects of Supercritical Fluids Activation on Carbon Nanotube Field Emitters

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***Abstract*—This paper proposes a novel method to enhance the emission characteristics of carbon nanotubes (CNTs). It is extremely possible for CNTs to adsorb moisture and other contaminants during the fabrication processes, leading to the degraded field emission characteristics. In this work, CNT emitters are activated with commonly used heating process and supercritical carbon dioxide ($SCCO_2$) fluids technology for removing adsorbed residue moisture. Experimental results have demonstrated that the electrical stability and field emission enhancement of CNT emitters are effectively achieved by the $SCCO_2$ fluids treatment compared to the heating process, due to the minimization of residuary moisture in CNTs.**

***Index Terms*—Carbon nanotubes (CNTs), field emission, supercritical carbon dioxide ($SCCO_2$ fluids.**

I. Introduction

IN RECENT years, carbon nanotubes (CNTs) have been employed in electron field emitters and extraordinarily suitable materials for field emission display (FED) applications because of their high mechanical strength and chemical stability coupled with very high aspect ratios leading to extremely strong local fields [1]–[4]. For achieving better emission characteristic, the CNTs need to be produced free of contamination and with minimized moisture adsorption. During the fabrication process of CNTs, it is extremely possible for CNTs to adsorb moisture and other contaminants, leading to degrade field emission characteristics and electrical instability. Experimental work that really focused on the study of moisture adsorption effect has been very scanty so far, and few methods have been reported for minimizing residual moisture and other contaminants in the nanostructure CNTs. Therefore, in this work the influence of residual moisture on the field emission characteristics of CNTs will be investigated first. In addition, the application of supercritical carbon dioxide ($SCCO_2$) fluids will be proposed to activate CNT emitters, minimizing residual moisture and contaminants uptake. The CO_2-based process is attractive because of its environmental compatibility, nontoxic, nonflammable, and unreactive under most conditions [5]. The $SCCO_2$ fluids are similar to nonpolar liquid CO_2, since it could dissolve nonpolar solvent and has good transport property to remove contaminants. Besides, it possesses gas-like properties of diffusivity and viscosity that allow it to carry solvents through the narrow spaces between micro- and nanostructure surfaces, without damage to the structure [6]. Also, the extremely low surface tension of $SCCO_2$ fluids can account for its negligible effect (i.e., extremely low damage) on the morphology and microstructures [7]–[9]. It has been reported in many papers that the use of the $SCCO_2$ fluids technology mixed with the cosolvent, such as C1-C6 alcohols, can effectively remove moisture [10], [11]. In this work, the $SCCO_2$ fluids technology will be applied for the first time to activate CNT emitters. Additionally, the field emission characteristics, material analysis, and electrical stability of CNT emitters will be discussed to exhibit the activation efficiency of the supercritical CO_2 fluids technology.

II. Experimental Procedures

CNTs were generally fabricated by several processing techniques, including arc-discharge, laser ablation, and chemical vapor deposition (CVD). In this study, the raw material of multiwalled carbon nanotubes was synthesized by arc-discharge. The normal CNT-FED diodes were fabricated by screen printing processes [12], [13]. First of all, a cathode pattern coated on a glass substrate by screen-printing a conductive slurry containing silver (Ag) through a patterned screen was carried out. Thereafter, a CNT layer was attached thereon by screen-printing a CNT paste through a mesh pattern screen to form CNT emitters. The CNT paste consisted of organic bonding agent, resin, silver powder, and carbon nanotubes. After that the substrate was soft-baked in an oven at 150 °C to remove volatile organic solvent. A higher temperature sintering process at 400 °C for 20 min was then carried out to solidify the CNTs and well electric coupled with the Ag cathode pattern.

For various CNT applications, it is extremely possible that CNTs suffered from a wet liquid environment during different manufacture processes. In order to investigate the influence of moisture uptake on the electron field emission characteristics of CNTs, the screen-printed CNT emitters were then rinsed with deionized (DI) water for 10 min at 25 °C. A hot-baking step at 100 °C for 1 h was first implemented for the initial drying of the DI-water-rinsed CNTs. Subsequently, we employed two

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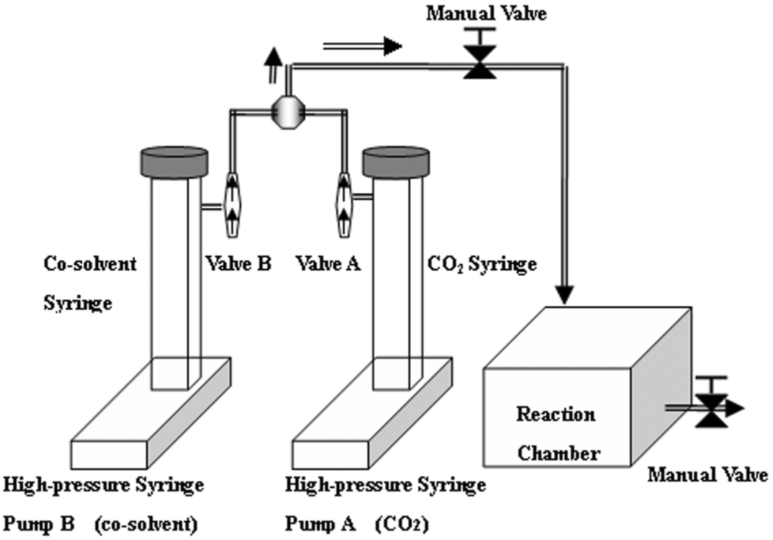

Fig. 1. Schematic showing the configuration of an $SCCO_2$ system.

kinds of activation methods to improve the field emission characteristics of CNTs. One was a typically used thermal annealing process in a furnace at 200 °C for 15 min under N_2 atmosphere. The second proposed technology was that processing the DI-water-rinsed CNTs at a $SCCO_2$ system for only 5 min to minimize residual moisture. Moreover, a chemical additive, propyl alcohol, was studied to incorporate into $SCCO_2$ fluids for enhancing the CNT activation efficiency.

Fig. 1 shows basic components of an $SCCO_2$ system. Both $SCCO_2$ fluids and cosolvent were compressed in syringes by a high-pressure syringe pump until achieving the goal pressure values, and then were injected into the reaction chamber. In this experiment, the $SCCO_2$ fluid mixed with 0–7 vol.% propyl alcohol was compressed under 1100–3000 psi, and heated in a reaction chamber at 50 °C. The reaction chamber included a stirrer for mixing the $SCCO_2$ fluids and cosolvent uniformly. After different activating processes, the material analysis was characterized with thermal desorption spectroscopy (TDS). The field emission characteristic and stability of CNT were measured in a vacuum chamber with 5×10^{-6} torr at room temperature. The measured field emission area was 1 cm^2 with the parallel-plate geometry, and emission current was characterized with Keithley 237 measurement instrument in the range of 0–1100 V by a dc mode.

III. Results and Discussion

Fig. 2 shows the field emission current of CNT emitters as a function of the applied electric field under different activation treatments. From the results, it is found among various activation treatments the DI-water-rinsed CNTs after only an initial 100 °C-baking step exhibits the poorest field emission performance (applied at ~ 5 V/μm, the current density reaching 50 μA/cm^2). The inferior field emission is inferentially due to residual moisture in the CNT emitters. Moisture adsorption in CNTs would form a blanket of H_2O capping layer. The moisture passivation layer speculatively results in a field emission barrier, thereby causing the degraded filed emission of the CNT emitters. The further improvement in the field emission current (applied at ~ 4.6 V/μm, the current density reaching 50 μA/cm^2) is observed for the CNT emitters activated by a furnace annealing step at a higher temperature of 200 °C. This suggests that the activation of DI-water-rinsed CNTs can be enhanced with increasing activation temperatures from 100 °C to 200 °C, due to the further elimination of residual moisture. The supercritically activated CNT emitters treated with pure $SCCO_2$ fluids, in contrast, are not exhibiting expectedly good field emission properties. In contrast, the enhancement in the field emission current and the decrease in the onset field emission voltage (~4 V/μm, defined by emission current reaching 50 μA/cm^2) can be achieved by incorporating a trace of cosolvent propyl alcohol into the $SCCO_2$ fluids.

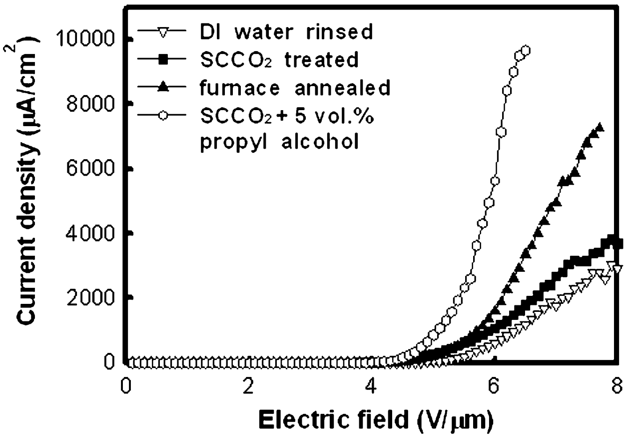

Fig. 2. Field emission current of DI-water-rinsed CNT emitters activated by different treatments, including initial 100 °C-hot-baking (denoted by DI water rinsed), 200 °C-furnace-annealing (denoted by furnace annealed), pure $SCCO_2$ fluid, and $SCCO_2$ with 5 vol.% propyl alcohol.

The field emission current of CNTs was determined by the Fowler–Nordheim (F-N) equation

$$I = aV^2 \exp(-b\phi^{3/2}/\beta V) \qquad (1)$$

where I, V, ϕ, and β are the emission current, applied voltage, work function, and field enhancement factor. The larger β expresses the larger field concentration, and the lower effective

TABLE I
The Slope of $\ln(J/E^2)$ Versus $1/E$; the Efficient Emission Is Obtained by Reducing the Moisture in Carbon Nanotubes

	DI water rinsed	$SCCO_2$ treated	Furnace annealed	$SCCO_2$ + 5 vol.% propyl alcohol
Slope	-59.785	-55.127	-48.12	-44.879

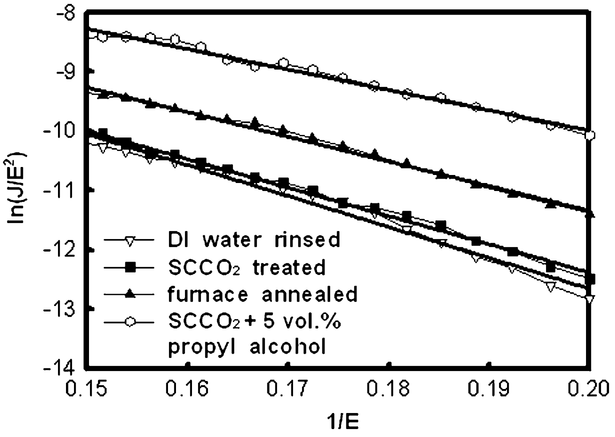

Fig. 3. The $\ln(J/E^2)$ vs $1/E$ plots of the CNT emitters, where J (A/cm^2) is the current density and E (V/μm) is the applied electric field.

threshold voltage for emission. Therefore, the lower work function and larger field enhancement factor are required for obtaining higher field emission current [14]. The F-N plot and the slope of $\ln(J/E^2)$ versus $1/E$ plot of the CNT emitters with different activating process are shown in Fig. 3 and Table I, respectively. It can be observed that field emission current corresponds to the F-N tunneling characteristic. A superior emission characteristic will exhibit the larger β and lower ϕ, expectedly showing a relatively gradual slope in the plot of $\ln(J/E^2)$ versus $1/E$ by (1). From Table I, we can find that the CNT emitter has the best emission characteristic after activation treatment in $SCCO_2$ with 5 vol.% of propyl alcohol, while the DI-water-rinsed CNTs after only an initial 100 °C baking shows the poorest emission characteristic. This also indicates that the absorbed moisture will seriously degrade the field enhancement factor and cause a barrier to the field emission, leading to an inferior field emission characteristic.

To reasonably verify the inference that the field emission enhancement correlates with the decrease of residual moisture content, it was monitored the evolution of moisture desorption in CNTs after different activation treatments. TDS, as shown in Fig. 4, was carried out upon heating these activated CNTs from 80 °C to 200 °C at a heating rate of 10 °C/s in vacuum (10^{-5} Pa). In the TDS analysis, M/e (mass-to-charge ratio) = 18 peak that is attributed to H_2O was monitored to evaluate the residual moisture of the CNTs. The desorption distribution at 100–200 °C is attributed to moisture adsorbed on the CNT emitters surface [15]. From Fig. 4, the highest residual moisture content is observed in the initial 100 °C-hot-baked CNTs, which showed inferior electron field emission previously. The residual moisture content is decreased further at a raised annealing temperature (200 °C). Furthermore, the minimized residual moisture content is detected in the CO_2 supercritically

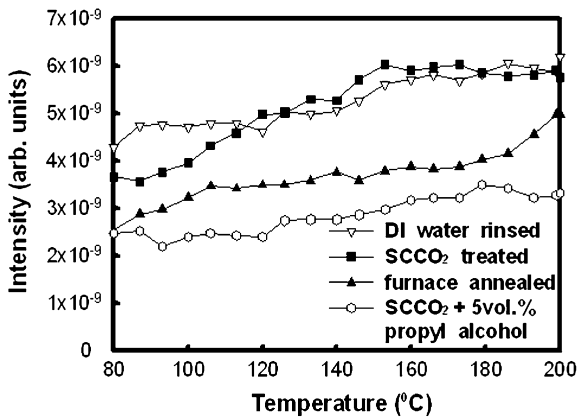

Fig. 4. TDS of DI-water-rinsed CNT emitters with different activation treatments, including initial 100 °C-hot-baking (denoted by DI water rinsed), 200 °C-furnace-annealing (denoted by furnace annealed), pure $SCCO_2$ fluid and $SCCO_2$ with 5 vol.% propyl alcohol.

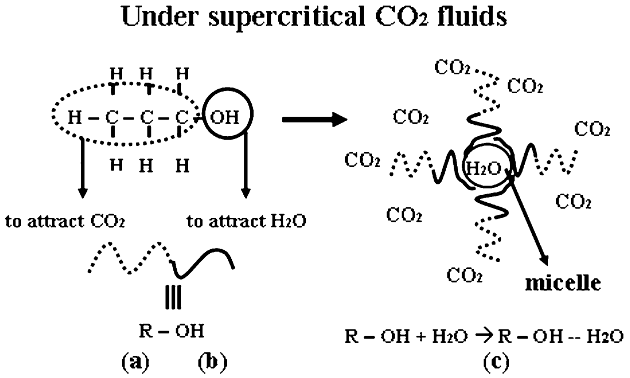

Fig. 5. The formation process of water nanodroplet in a supercritical carbon dioxide solution with the surfactant of propyl alcohol. (a) Hydrophobic hydrocarbon group. (b) Hydrophilic hydroxyl group. (c) Micelle.

activated CNT with 5 vol.% propyl alcohol. This indicates the addition of a trace of cosolvent into the $SCCO_2$ fluid can effectively minimize residual moisture adsorption in the nanostructure CNTs, increasing activation efficiency. The role of cosolvent propyl alcohol can be deduced further as sketched in Fig. 5.

In this work propyl alcohol acts as a surfactant between hydrophobic supercritical CO_2 fluids and polar H_2O molecules, capable of enhancing the polarization of $SCCO_2$ fluids. The propyl alcohol has a hydrophilic hydroxyl group, easily attracting with H_2O molecules and associates in $SCCO_2$ solution with hydrophobic hydrocarbon group to form a micelle. At certain temperatures, pressures, and cosolvent concentrations, such micelles spontaneously self-assemble in $SCCO_2$ solution. Water will be incorporated into the core of the micelle [16], generating a nanodroplet of water in the carbon dioxide solution, and be easily carried away from CNTs by the enhanced polarization $SCCO_2$ fluids. It is thereby critical to maximize

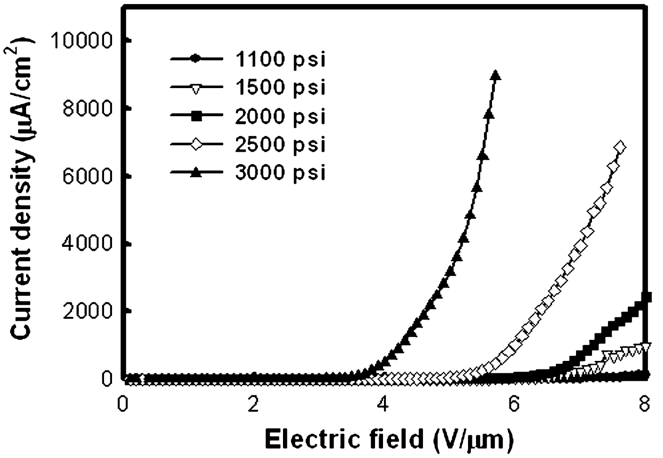

Fig. 6. Field emission characteristics of CNT emitters activated by $SCCO_2$ fluids under different pressure, mixed with 5 vol.% propyl alcohol and heating at 50 °C.

the solubility of propyl alcohol in $SCCO_2$ fluids in order to enhance the polarization of $SCCO_2$ fluids for minimizing residual moisture in CNTs, by micelles self-assembled in the $SCCO_2$ fluids. Experimental results show the solubility of cosolvent in the $SCCO_2$ fluids increases with increasing $SCCO_2$ fluid pressure [17], [18]. In this work, the solubility of propyl alcohol in $SCCO_2$ fluids is ranging from 3 vol.% to 20 vol.% under 1100 psi–3000 psi. Fig. 6 shows the field emission current of CNT emitters which were activated in $SCCO_2$ fluids mixed with 5 vol.% propyl alcohol at 50 °C under different pressures. The CNTs emission characteristic is obviously improved with adding 5 vol.% propyl alcohol to the $SCCO_2$ fluids under 3000 psi, compared to the case under 1100–2000 psi. This finding indicates that water nanodroplets could effectively be formed and picked off from CNTs under higher pressure, as a result of the propyl alcohol and micelles sufficiently dissolved in $SCCO_2$ fluids. Therefore, the sufficient solubility of cosolvent in supercritical fluid is indeed critical for a successful $SCCO_2$ activation process.

The stability of emission performance was evaluated by monitoring the emission current after a high electric field stress. Fig. 7 shows the field emission current of CNT emitters before and after the high electric field stress of 11 V/μm for 30 min. In this result, the emission performance of CNTs obviously degrades after the bias stress, resulting in threshold voltage shifts for the DI-water-rinsed and the pure $SCCO_2$ -treated CNTs especially. The electrical instability of bias-stressed CNTs can be attributed to the damage from joule heating and a trace of ion-bombardment damage. As a result of local joule heating along the carbon tube, the emission current may burn the CNTs [19], [20]. Besides, the adsorbed containments could outgas from the CNTs by joule heating, and sequentially be ionized under the stress of high electrical field. These resultant ions easily bombard the carbon nanotubes and cause to the field emission degradation [14]. Therefore, emission current of CNTs decays and higher threshold voltage is required, after the electric field stress for the moisture-absorbed CNT emitters. From Fig. 7, the emission performance of CNTs activated in $SCCO_2$ fluids with 5 vol.% propyl alcohol is almost intact because of minimizing residue absorbates. Also, it represents the superior electrical stability of CNT emitters, while residuary moisture in CNTs is minimized.

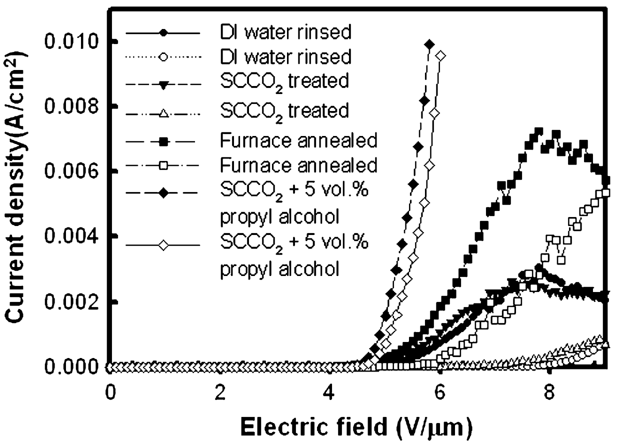

Fig. 7. The field emission characteristics of different activated CNT emitters before (full dots)/after (empty dots) high electric field (11 V/μm) stressing for 30 min.

IV. CONCLUSION

In this paper, we have experimentally disclosed that the field emission characteristics of CNT emitters will suffer from residue moisture adsorption. Also, the residue moisture leads to conspicuous electrical degradation of CNT emitters after stressed by high voltage bias. This indicates the removal of residual moisture is really critical to the field emission performance and reliability of CNT emitters. The $SCCO_2$ fluid has high mass transfer rates, zero surface tension, and is applicable to remove contaminants in microstructures with no additional damage. In this work the optimization activation technology for CNT emitters has been obtained by the addition of cosolvent propyl alcohol into $SCCO_2$ fluids. Compared to the typical furnace heating process, TDS analysis also has demonstrated $SCCO_2$ fluids with propyl alcohol can minimize residual moisture in CNTs. The $SCCO_2$-activated CNT emitters, thereby, possess superior electron field emission characteristics and electrical stability. Additionally, it is observed that the solubility of propyl alcohol in $SCCO_2$ fluids plays a critical role in the activation efficiency. Under high pressure $SCCO_2$ ambient, efficacious improvement of field emission current can be attained, due to high solubility of propyl alcohol in $SCCO_2$ fluids, leading water micelles to be picked off from CNTs. These experimental results suggest the modified $SCCO_2$ fluids will be promising for the activation of CNT emitters to minimize moisture residual. The supercritical CO_2 fluid technology is thereby expected to be a serviceable drying method for application in the nanotechnology field.

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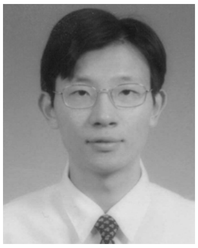

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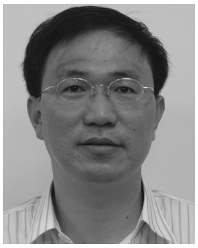

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