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The effect of surface treatments on the field emission characteristics of patterned carbon nanotubes on KOVAR substrate

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Abstract

The field emission characteristics of patterned carbon nanotubes (CNTs) the average diameter of which is 16 nm cathodes on substrates with different surface treatments were investigated. The surface treatments of the substrate were performed by nickel electroless plating and palladium coating, which is an activation procedure of electroless plating. CNTs were patterned on the surface treated substrate with radius of 200 µm through conventional photolithography process. Two deposition methods, electrophoresis deposition and spray deposition, were used to investigate the effects of deposition methods on field emission characteristics of the cathodes. It was revealed that the two deposition methods showed similar turn-on field trends, which means that the different surface morphologies of the substrates have more influence on the field emission characteristics than the different deposition methods performed in this study. Through the surface treatments, the roughness of the surface increased and cathodes with a high roughness factor showed better field emission characteristics compared to non-treated ones.



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RESEARCH PAPER

The effect of surface treatments on the field emission characteristics of patterned carbon nanotubes on KOVAR substrate

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Abstract The field emission characteristics of patterned carbon nanotubes (CNTs) the average diameter of which is 16 nm cathodes on substrates with different surface treatments were investigated. The surface treatments of the substrate were performed by nickel electroless plating and palladium coating, which is an activation procedure of electroless plating. CNTs were patterned on the surface-treated substrate with radius of 200 µm through conventional photolithography process. Two deposition methods, electrophoresis deposition and spray deposition, were used to investigate the effects of deposition methods on field emission characteristics of the cathodes. It was revealed that the two deposition methods showed similar turn-on field trends, which means that the different surface morphologies of the substrates have

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Institute of Advanced Composite Materials, Korea Institute of Science and Technology, 864-9 Dunsan-ri, Bongdong-eup, Wanju-gun, Jeollabuk-do 565-902, Republic of Korea more influence on the field emission characteristics than the different deposition methods performed in this study. Through the surface treatments, the roughness of the surface increased and cathodes with a high roughness factor showed better field emission characteristics compared to non-treated ones.

Keywords Multi-walled carbon nanotubes · Field emission characteristics · KOVAR · Surface treatment · Roughness factor

Introduction

Carbon nanotubes (CNTs) are one of the most attractive materials around due to their fascinating properties such as good chemical sensitivity, electrical conductivity, physical and chemical stability, and high aspect ratio. CNTs could be applied to various research fields of electrical engineering such as chemical sensors (Kong et al. 2000; Li et al. 2003), transparent electrodes (Wu et al. 2004), LCD back light units (Kim and Yoo 2005), field emission displays (De Heer et al. 1995; Choi et al. 1999), and X-ray sources (Zhang et al. 2005; Sugie et al. 2001). In particular, it has been proposed that CNTs could be utilized as an electron emission source, because they have both a remarkable high aspect ratio and electrical conductivity properties essential for field emission devices. It was experimentally proven that CNTs used

as field emitters can emit a high current at a low turnon voltage and have a high field enhancement factor. A number of researches have been performed to enhance the field emission characteristics by various methods including plasma treatment (Zhi et al. 2002), doping (Sharma et al. 2006) and laser activation (Chen et al. 2008) on deposited CNT films. However, very few researches have reported on how field emission characteristics would be affected by the surface treatment of the substrates. According to the double barrier model for the field emission mechanism (Zhang et al. 2006), contact between the substrate and the CNT film is also an important factor, not just the surface morphology of emission sites.

In this paper, the field emission characteristics of CNTs on different surface-treated substrates have been reported. KOVAR, an alloy of Fe (53 %), Ni (28 %), and Co (18 %), is chosen as a substrate for application of an X-ray source. Owing to its low thermal expansion coefficient, KOVAR alloy can provide exceptional hermetic seals in X-ray tubes. The KOVAR substrates were surface-treated by Ni-electroless plating (Mallory and Hajdu 1990) and Pdcoating (Charbonnier et al. 1998) was generally used to form the seed layer for electroless plating before the CNTs were deposited. During these processes, the metal coatings on the KOVAR substrates modify the surface morphology of the substrates, especially their roughness. Electrophoretic deposition (EPD) (Gao et al. 2001) and spray deposition (Jeong et al. 2006), are used to investigate how CNT deposition influences the field emission characteristics of the emitter as the roughness of the substrate changes. Furthermore, the CNTs are patterned onto the substrate by a conventional photolithography process, so it could be applied to a triode field emission structure. As a result, the field emission characteristics of an emitter were improved by a simple immersing process.

Experimental

Multi-walled CNTs with an average diameter of 16 nm were synthesized through chemical vapor deposition. They were then used to fabricate the electron emission source. In order to carry out EPD, sodium dodecyl sulfate (SDS) was used to disperse the nanotubes and insure that the surface of CNTs was negatively charged. 50 mg of CNTs, 150 mg of SDS

and 100 mL of deionized water were ultrasonicated for 2 h to disperse the CNTs. The solution formed a suspension and it was ultracentrifuged for 10 min at 5,000 rpm to improve the dispersion quality. Owing to the hydrophobic characteristics of CNTs, the hydrophobic site of SDS is adsorbed on the surface of CNTs, thus insuring uniform dispersion.

The radius and thickness of the KOVAR substrate was 22 and 0.3 mm, respectively. Prepared substrates were mechanically polished and surface-treated through Ni-electroless plating (Mallory and Hajdu 1990) and Pd-coating (Charbonnier et al. 1998). Ni-film was coated onto the KOVAR substrate using electroless plating, where the plating solution mainly consisted of sodium hypophosphite (NaH₂PO₂·H₂O), Ni sulfate (NiSO₄·6H₂O), and other materials for adjusting the pH of the solution. The temperature of the plating bath was maintained in the range of 85 ± 2 °C with a plating time of 40 min.

Another KOVAR substrate was surface-treated by Pd-coating. A solution was used for activation of the electroless plating, consisting of Pd-chloride (PdCl₂) and tin-chloride (SnCl₂) in hydrochloric acid. The substrate was immersed into the solution for 1 min at room temperature and rinsed with deionized water. The Pd ions (Pd^{2+}) in the solution were reduced to Pdparticles (Charbonnier et al. 1998), which were then coated onto the surface of the KOVAR substrate. In this manner, substrates that are surface-treated by two different methods will have different surface roughness. The surface morphology of the substrates was examined using the field emission scanning electron microscope (SEM), atomic force microscope (AFM) images, and X-ray photoelectron spectroscopy (XPS) analysis.

CNTs were patterned on different surface-treated substrates by photolithography, as illustrated in Fig. 1a. The diameter of each hole is 200 μ m and they form a hexagonal shape in the center of the substrate. After the development process, the prepared CNT solution was deposited on hole-patterned substrates by spray deposition and EPD. As shown in Fig. 1b, a stainless steel plate was used as a working and counter electrode. The gap between the KOVAR substrate and the electrode was 0.5 cm and it was maintained by a spacer. A constant voltage of 10 V DC was applied for 2 min. The negatively charged CNT–SDS was deposited onto the patterned site of the KOVAR substrate. Also, the prepared CNT solution



Fig. 1 a A schematic of CNT deposition on different surface-treated substrates by b electrophoretic deposition and c spray deposition

was deposited onto the substrate by spray deposition for comparison with EPD, as shown in Fig. 2c. After CNT deposition, they were dried at room temperature for a day and a lift-off process was carried out using acetone.

Annealing of the fabricated cathodes was performed at 380 °C for 1 h in a nitrogen atmosphere to remove impurities. Subsequently, cathodes were activated by adhesive taping. The deposited CNTs were now vertically aligned and the relatively weakly bonded CNTs were removed by taping.

The field emission characteristics of patterned CNTs were measured in a vacuum chamber at 5×10^{-6} Torr. A stainless steel rod was used for the anode, and the gap between the anode and cathode was 1 mm. A total of six cathodes were measured with different surface treatments and CNT deposition methods. The surface of CNT patterns were analyzed by SEM. The fluorescence image of the cathodes was captured using a digital camera.

Results and discussion

Figure 2 shows the AFM and SEM images of different surface-treated KOVAR substrates. Several nanoscaled scratches on the non-treated (just polished) substrate can be seen in Figs. 2a, d, 3b, e, c, f show the surface morphology of the Ni-electroless plated and Pd-coated substrate, respectively. AFM images of Fig. 2 show several micro-sized Ni-grains for which the height is <100 nm on the Ni-plated substrate, and Pd-particles for which the height is <200 nm on the surface of the Pd-coated substrate.

Figure 3 shows the SEM images of CNT patterns with diameters of 200 μ m and the morphologies of CNTs deposited by EPD on different surface-treated substrates. Figure 3a, b present the patterns of CNTs deposited by EPD and the spray method, respectively. Figure 3c–e show the morphology of deposited CNTs on the non-treated, Ni-plated, and Pd-coated substrates. The grain boundary of the Ni-plated substrate below the deposited CNTs is shown in Fig. 3d, and coated Pd can be seen under the deposited CNTs in Fig. 3e, but in case of the non-treated substrate, there are only deposited CNTs, as can be seen in Fig. 3c.

The XPS spectra of the CNTs deposited substrates are presented in Fig. 4. Figure 4a shows the spectrum of the Ni 2*p* range of the substrates. This result is in agreement with the spectrum of Ni-oxide thin film (Sasi and Gopchandran 2007). In case of the nontreated and Ni-plated substrate, both Ni 2*p*_{1/2} and Ni 2*p*_{3/2} peaks were obtained, whereas no peaks were found in the Pd-coated substrate. Peaks were obtained at the binding energies of 856.55 (Ni 2*p*_{3/2}) and 874.6 eV (Ni 2*p*_{1/2}) and their satellite peaks (Kim and Winograd 1974) were also found to the left of each for the Ni-plated substrate. In case of the non-treated



Fig. 2 The AFM and SEM images of different surface-treated substrates. AFM images were scanned over 10 μ m by 10 μ m. Only nano-sized scratches are found for (a), d the non-treated substrate. The height of the protruding sites is <10 nm. b and e showing the surface of the Ni-plated substrate. Several micro-

substrate, the binding energies at each peak are almost identical to those of the Ni-plated substrate (856.35 for Ni $2p_{3/2}$ and 874.5 ev for Ni $2p_{1/2}$). However, a stronger Ni-peak intensity was obtained after Ni-electroless plating. Figure 4b shows the Pd 3d range of the substrates. A Pd-peak was found at 342.5 (Pd $3d_{3/2}$) and 337 eV (Pd $3d_{5/2}$) for the Pd-coated substrate only. This spectrum is coincident with that of Pd-oxide (Marcell 1985).

In order to confirm the effects of surface morphology on the field emission characteristics, EPD and the spray method were used to deposit CNTs on the substrates and the results were compared. Figure 5 and Table 1 show the current–voltage characteristics and Fowler–Nordheim plot (FN-plot) of the emitter with CNTs deposited by EPD (Fig. 5a, b) and the spray method (Fig. 5c, d) and their respective fluorescence images. The current–voltage characteristics were measured using a stainless steel anode where the distance of the anode and cathode was maintained at 1 mm.

In the case of EPD, the turn-on fields, which are defined as the electric field when the current density

sized Ni-grains can be seen and the height of the grains is <100 nm. **c** and **f** are the surface morphology of the Pd-coated substrate. The protruding sites of the Pd-coated surface have a height <200 nm higher than that of the Ni-plated surface

excess charge is 100 μ A/cm² are 3.6, 3.3, and 2.4 V/ µm for the non-treated, Ni-plated and Pd-coated substrate, respectively. Above all, the contact between surface-treated substrate and deposited CNTs should be discussed to analyze the turn-on field difference. Several researches were performed with contact between metals and CNT (Lim et al. 2009; Zhang et al. 2000). The contact between metals and CNT varies by kind of metals. For transition metals, like Pd and Ni, as d orbital vacancies increase, generally the wettability of metals gets better. The metals such as Ti, Cr, and Fe which have good wettability to CNT show low contact resistance than have poor wettability owing to large contact area between them and CNTs. On the other hand, contact resistance of the metals which have poor wettability is affected to the work function difference between metal and CNTs (Lim et al. 2009). However, the non-treated substrate mainly including Fe shows highest turn-on field in this study. This is because remaining oxygen gas reacted with the surface of the substrate even though in environment of flowing of nitrogen gas during annealing process. As a result, the thin oxide layer



Fig. 3 SEM images of patterned CNTs with a pattern size diameter of 200 μ m on substrates by **a** EPD and **b** the spray method. **c**, **d** and **e** showing deposited CNTs for the non-treated, Ni-plated, and Pd-coated surface, respectively. CNTs are deposited and form random networks on the substrate on all



Fig. 4 The XPS spectra of CNTs deposited substrates. The range of the Ni 2p and Pd 3d spectrum is shown in (**a**) and (**b**). The intensity of the Ni-plated surface is higher than that of the non-treated surface. The Pd-coated surface does not have any

was produced between substrate and CNTs in all samples. Therefore, it should be explained by work function difference rather than wettability. The work occasions. The non-treated surface shows only the deposited CNTs and for the Ni-plated surface, the Ni-grains are found under deposited CNTs. No grains are observed on the Pd-coated surface, and only coated Pd protruding sites are found under deposited CNTs



peaks in the Ni 2p range. In the Pd 3d range, no peak was found for the non-treated and Ni-plated surface, whereas a peak was obtained for the Pd-coated surface

function of Fe, Ni, Pd, and metallic CNT are 4.5, 5.15, 5.25, and approximately 4.6–5.1 eV, respectively (Lim et al. 2009). The low work function metals

	Non-treated	Ni-plated	Pd-coated
RMS roughness value (nm)	4.59	43.74	90.55
Electrophoretic deposition			
Current (mA)	7.4 (@5.5 V/µm)	10.4 (@5.2 V/µm)	18.2 (@4.5 V/µm)
Turn-on field (V/µm)	3.6	3.3	2.4
Field enhancement factor (β) (low field region)	1,248	1,591	2,828
Field enhancement factor (β) (high field region)	5,698	5,874	8,004
Spray deposition			
Current (mA)	7.5 (@5.5 V/µm)	9.4 (@5.2 V/µm)	15.6 (@4.5 V/µm)
Turn-on field (V/µm)	3.7	3.3	2.7
Field enhancement factor (β) (low field region)	1,104	1,499	2,021
Field enhancement factor (β) (high field region)	2,520	4,959	6,583

Table 1 The field emission characteristics of the each of the cathode

For both EPD and spray deposition, a similar trend was observed as the RMS roughness value increases. The turn-on field and field enhancement factor are enhanced

suffer from higher resistance between metal and CNTs compared to high work function metal because higher voltage need to be applied to inject electrons into the CNTs (Ngo et al. 2004). The field emitter which uses Pd and Ni showed better characteristics than non-treated (KOVAR) one in our study. This is because the work function of Fe which is main component of the KOVAR is lowest among Fe, Ni, and Pd. The work function difference between Ni and Pd is 0.1 eV. In spite of the small work function difference, the emitter which uses Pd showed much better field emission characteristics than Ni. Thus, turn-on field difference of the emitters should be analyzed to the other directions.

As shown in the AFM images of Fig. 2a-c, the surface morphologies of the three substrates have different surface roughness. The root mean square (RMS) roughness values of the surfaces are calculated over 10 µm by 10 µm scan areas, as shown in Table 1 (inset). The RMS values of the substrate were 4.59, 43.74, and 90.55 nm for the non-treated, Ni-plated and Pd-coated substrate, respectively. When the electric field is applied between the anode and cathode, the electrons are transported from the KOVAR substrate to the CNT network directly for the non-treated substrate. However, in the case of the Ni-plated and Pd-coated substrates, the electrons are transported via the Ni-grains and coated Pd-particles. The applied electric field is concentrated on these protruding Ni-grains and coated Pd-particles. As shown in the AFM images of Fig. 2a–c, the coated Pd-particles are higher and narrower than the Ni-grains, and these differences affect the turn-on voltage of each cathode. It can be stated that an increase in the roughness factor of the substrate will lower the turn-on field of the emitter.

The field emission characteristics of the cathodes fabricated by the spray method are depicted in Fig. 5c, d. The turn-on voltages of the cathodes on which CNTs were spray deposited are $3.7, 3.3, and 2.7 V/\mu m$ for the non-treated, Ni-plated, and Pd-coated substrate, respectively. As shown in the results, the Pd-coated substrate shows the lowest turn-on field, followed by the Ni-plated and non-treated substrate. A similar trend was observed in the case of EPD. It has been reported that the adhesion property between the substrate and CNTs deposited by EPD is better than the spray method owing to the metal hydroxides at the surface that are produced during EPD. These metal hydroxides assist formation of hydrogen bonds on the surface of CNTs (Thomas and Boccacciniw 2005). However, there is no direct relationship between the turn-on field and the method of deposition. Therefore, owing to the similar trends observed in both EPD and the spray method, it can be concluded that the surface morphology of the substrate affects the field emission characteristics more than the type of deposition method used.

The FN-plot is depicted in Fig. 5b, d. The field enhancement factor (β) is calculated from the slope of the FN-plot $B\varphi^{3/2}d/\beta$, where *B* is a constant of



Fig. 5 The current–voltage characteristics of emitters with CNTs deposited by \mathbf{a} EPD and \mathbf{c} the spray method, and \mathbf{b} and \mathbf{d} the Fowler–Nordheim plot. The Pd-coated surface that has the highest roughness factor shows the best field emission characteristics, followed by the Ni-plated and non-treated

cathode CNTs deposited by both EPD and the spray method. The fluorescence images of the Pd-coated emitter CNTs deposited by \mathbf{e} EPD and \mathbf{f} the spray method. The cathode deposited by EPD shows better emission uniformity than the spray method

 $6.83 \times 10^9 \text{ eV}^{-3/2}$ /m, φ is the work function, and d is the distance between the anode and cathode. The work function of CNTs is assumed to be 5 eV, as presented in Table 1 (inset) (Bonard et al. 2002). In general, β is a geometric factor that is related to the height and radius of the emitter. However, the slope of the plot exhibits non-linearity for all emitters, as shown in Fig. 5b, d. The decrease in slope in the high field region has frequently been observed in field emission researches, and can be explained through the spacecharge effect and variation of effective work function (Collins and Zettl 1997; Chen et al. 2009). It is found that the slope of the plot increases again in the very high field region, and this region is circled with a green line in Fig. 5b, d. A similar phenomenon was also reported in (Kim et al. 2010) where if the spread in the value of beta is very narrow; a number of individual CNTs will be easily activated, resulting in an increase in total current at the very high electric field region. The information on the spread of β has not been confirmed in our study, but given that the adhesion property of CNTs deposited by EPD is better than the spray method (Thomas and Boccacciniw 2005), and thus results in better uniformity; it can be inferred that the EPD CNT substrate reaches the very high field region relatively earlier, as shown in the FN-plot. It is considered that through adhesive taping, the number of CNTs deposited by the spray method that had been removed is more than that of EPD. As depicted in Fig. 5e, f, CNTs deposited by EPD show better emission uniformity than that of the spray method, which means that CNTs activated in the high field region have a longer life-time compared to CNTs of the spray method.

It is revealed that the β value increases as the roughness factor of the substrate increases in both EPD and the spray method cases, as shown in Table 1. Although the surface roughness enhances the β value of the emitter, β is not directly proportional to the increase in the roughness factor. This is because the RMS roughness factor only provides information on the height and depth of the protruding sites, but not the width, which influences the β value of the emitter. CNT deposited by EPD shows a larger β value than that of the spray method. Owing to the difference in the adhesion property, it is assumed that more CNTs with high β values remained in case of EPD during adhesive taping and field emission.

Conclusion

It was reported that the field emission characteristics of cathodes can be improved by surface treatment. The surface-treated substrates showed different RMS roughness factors, which influenced the turn-on field of the emitters. Although the RMS factor is not an absolutely decisive factor for the turn-on field, due to the absence of the width factor of protruding sites, nonetheless, it can be a reference factor for the field emission characteristics of an emitter. Also, the emission properties were investigated by comparing different deposition methods; EPD and the spray method, which are costcompetitive compared to the direct growth method. It is revealed that the roughness of the surface has a more significant effect on the field emission characteristics than the deposition method. The CNT-patterned KOVAR substrate that was used in this study shows great promise for X-ray applications in the future.

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