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Laser Patterning of Carbon-Nanotubes Thin Films and Their Applications

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1. Introduction

In 1960 T.H. Maiman successful fabricated the first laser and projected the first laser light. Fifty years later, lasers have found extensive applications in fields including energy, materials, communications, biotechnology and mechanical engineering. In the past several decades, continuous developments have improved the performance of lasers. From an initial wavelength of 694.3 nm, new lasers have been developed achieving wavelengths in the infrared (10.6 μ m) and ultraviolet (157 nm) spectra. Laser light has the several features which distinguish it from traditional light sources: directionality, brightness, monochromatity and coherence. These four characteristics have resulted in lasers having a profound influence on industrial development. In the following, the use of laser for material transfer will be briefly introduced.

1.1 Laser transfer techniques

Based on the arrangement of laser, donor thin film and acceptor substrate, laser-induced material transfer can be classified into two groups: laser-induced forward transfer (LIFT) and the laser-induced backward transfer (LIBT), schematically illustrated in Fig. 1(a) and (b), respectively. To date, LIFT is the mainstream technique and has been reported in more applications than LIBT. In LIBT, the accepter substrate needs to be transparent to the laser light, whereas LIFT is not subject to this limitation and can use non-transparent accepter substrates.

1.2 History of the LIFT technique

Bohandy et al.¹ demonstrated the LIFT technique 26 years after the first development of laser light. Using an excimer laser, he successfully deposited copper metal deposition from a supported metal film. The following year Blonder et al.² used LIFT to pattern Al for integrated circuit applications, the first application of LIFT in the field of the microelectronics. He showed that LIFT could not only be used for the deposition of metal on a substrate, but could also be applied in many other areas. In the following years, LIFT was successfully used for the deposition of W³, Cr⁴, Au/Sn⁵ and Al^{6,7}.

1.3 Application of the LIFT technique

The LIFT technique quickly showed its potential in the display, biomedical, energy and microelectronic industries. Display researchers successful deposited a layer of emitting

polymer⁸ or bilayer emitting polymer/metal⁹⁻¹¹ material on a target substrate, demonstrating that organic light emitting display (OLED) pixels can be manufactured by LIFT. In carbon nanotube field emission displays (CNT-FED) and the plasma display panels (PDP), the carbon nanotube cathodes¹² and the phosphor layers¹³ were printed using the LIFT process.

The biomedical industry had long faced problems transferring biological materials. Unlike the deposition of metals, transferring biological materials by laser beam lead to irreversible molecular decomposition. One possible solution was to transfer biological materials in liquid. Using the LIFT technique, researchers successfully transferred DNA¹⁴⁻¹⁵ and several other kinds of biomaterial¹⁶⁻¹⁸. In the energy industry, the LIFT technique was used to deposit TiO₂ thin film¹⁹ to fabricate the dye solar cell devices with a power conversion efficiency up to $4.3\%^{20}$.



Fig. 1. Laser transfer techniques: (a) laser-induced forward transfer (LIFT) (b) laser-induced backward transfer (LIBT).

In microelectronics Ag²¹, silver²² and DNNSA-PANI doped with single-walled carbon nanotubes (SWNT)²³ were applied in the deposition of source/drain transistor electrodes, and a line pattern at the sub-10µm level could be achieved over several cm². Furthermore, multilayer transfer was successfully used to manufacture organic thin film transistors (OTFT)²⁴. The electrodes Ag and the semiconductor layer were simultaneously transferred to the target substrate. The SWNT thin film was also used as the transferring material for the semiconductor layer between the source/drain electrodes to fabricate P- and N-type field effect transistors²⁵. In addition, the LIFT technique also could be used to encapsulated organic electronic devices²⁶, and could be used to repair internal circuit damage.



Fig. 2. (a) (b) LIFT technique used to transfer Al⁷ and Ag electrodes of the OTFT²¹, (c) (d) LIBT technique used to transfer gold nanodroplets²⁹ and microarrays of the cDNA³⁰.

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1.4 Application of the LIBT technique

The LIBT technique has been used to transfer Cr²⁷ on quartz and phahalocyanine solids on PMMA²⁸. However, LIBT is used less frequently than LIFT technique due to its need for a transparent acceptor substrate. However, LIBT can be used to transfer spherical gold droplets²⁹ up to 300 nm to the substrate. Using LIBT technique, the solution material being transferred could have a lower impact force on the acceptor substrate than would be the case using LIFT, thus allowing for the transfer of a smaller amount of solution material to the substrate. cDNA³⁰ had also been printed with both LIBT and LIFT. Figure 2 shows LIFT transfer of Al film⁷ and Ag electrodes to OTFT²¹, and LIBT transfer of gold nanodroplets²⁹ and microarrays to cDNA³⁰.

1.5 Limitations of laser transfer techniques

Laser transfer techniques had several limitations in material deposition³¹. (1) **Cost**: Transferring metal film requires the metal film to be deposited on the substrate in a vacuum chamber. This process is expensive and increases the cost of the fabrication. (2) **Size**: To date, the minimum reported size is a 300 nm droplet²⁹, it could reach several micrometers for metal thin film²². The laser transfer method has a limitation on the size of line patterns and does not work well when the line width is below 75 nm. The suitable range for this method is from the several hundred micrometers to several hundred nanometers. (3) **Oxidation**: In atmospheric conditions, the metal pattern is easily oxidized, which will affect its conductivity and reduce device performance. (4) **Irreversible phase transition**: Some materials are not easily transferred, and materials undergoing vaporization will be damaged, leading to irreversible phase change in biomaterials, in particular.

1.6 Laser transfer mechanism

In 1970, Levene's group³² reported their work of material transfer and described the mechanism for the laser transferring of the material as follows: (1) the material to be transferred was heated by a laser beam; (2) the material was superheated and vaporized at the interface; (3) the vaporized material closest to the interface pushes the non-vaporized material away from the substrate to the acceptor substrate; (4) the transfer material is collected on the accepted substrate. Figure 3 shows the mechanism of the laser transfer technique.

During the process of laser transfer, the material is evaporated or melted. This process affects the properties of the transfer material as described above and the researchers have developed several methods³³ to regulate the state of the transfer material: (1) **Use a laser-absorbing layer or a dynamic release layer.** A sacrificial layer is added between the substrate and the transferring material to absorb heat from the laser and repel the transfer material. This layer keeps the transfer material from superheating and will evaporate. (2) **Use composite or matrix-based materials.** This technique was used to transfer various powder mixed with an organic or polymer binder. The binder is photo-sensitive³⁴ and can decompose at lower laser intensities. When the laser irradiates the binder, it decomposes and pushes the powder to the acceptor substrate. It can transfer thicker films with lower substrate temperatures. (3) **Use liquid buffer layer**: This technique transfers liquid phase material as gel ink or biomaterials. The role of the liquid is to absorb laser intensity and vaporize. The biomaterial is then ejected from the substrate to the acceptor substrate. If the liquid can not adequately absorb the laser intensity, the biomaterial will be damaged.



Fig. 3. The laser transfer technique mechanism. (a) The transfer material is heated by a laser beam. (b) The transfer material is superheated and vaporized at the interface. The vaporized material closest to the interface pushes the non-vaporized material away from the substrate to the acceptor substrate. (c) The transfer material is collected on the accepted substrate.

2. Development of carbon nanotubes

In 1959 Richard P. Feynman said, "If we can control miniature bodies in order, we can get a great quantity characteristic out of the ordinary". This was a surprising insight into the nanotechnology which would be developed in the 21st century. The term "Nanotechnology" was coined by N. Taniguchi in 1974, and the preface "nano" has already entered into popular usage. The development of nanotechnology is already starting to be felt in of our daily lives and, as advances in biological and information technology defined the 20th century, nanotechnology is shaping up to define the 21st century.

The 1991 discovery of carbon nanotubes (CNT) by Dr. Sumio Iijima at NEC Corporation opened the possibility of nanotechnology to almost an unlimited extent. CNTs possess many special characteristics including a high aspect ratio, high thermal conductivity and high electrical conductivity. In addition their mechanical strength is one hundred times greater than that of steel. These characteristics present potentially revolutionary applications in fields as diverse as energy, biomedicine, information, electronics, and optoelectronics.

Generally, graphite is composed of planes of carbon atoms in a hexagonal arrangement. In 1985, Sir Harold W. Kroto in the UK, and Robert F. Curl and Richard E. Smalley in the US used lasers to irradiate, and thus completely change the structure of, graphite. After laser irradiation, the carbon atoms in the planar layer in graphite formed three-dimensional hollow spheres composed of pentagonal and hexagonal arrangements of carbon atoms. These hollow spheres were called "buckyballs" (C_{60}), and the discovery merited the 1996 Nobel Prize in Chemistry. In 1991, Iijima was fabricating buckyballs when he discovered a new hollow tube structure of multi-walled carbon nanotubes (MWNTs). In 1993, he also successfully fabricated another type of carbon nanotube - single-walled carbon nanotubes (SWNTs). As their name implies, carbon nanotubes are hollow tubes enclosed by a half buckyball at either end. The tube is laterally composed of planar carbon atoms in pentagonal or hexagonal arrangements. A SWNT can be conceptualized by wrapping a layer of graphite, called graphene, into a seamless cylinder while a MWNT consists of multiple rolled layers (concentric tubes) of graphite. The diameter of a SWNT is about 1.4 nm while the diameter of MWNTs ranges from 20 to 40 nm. The interlayer interactions for MWNTs

occur through van der Waals' forces and CNTs are usually less than 100 μ m in length. SWNTs are generally believed to have greater applications than MWNTs due to their superior performance in a wide range of material properties. More recently, double-walled carbon nanotubes have also been successfully fabricated though the properties of these new structures are still largely unknown.

Several methods have been reported for fabricating CNTs including (a) the arc charging method, (b) the laser vaporization/ablation method, and (c) the chemical vapor deposition (CVD) method. In the arc charging method, an inert gas is infused into a vacuum chamber with two graphite electrodes. When a high voltage bias is applied between the two electrodes, the charge of the generated arc can induce the carbon to form a tube structure around the negative electrode. The laser vaporization/ablation method uses a high energy laser beam to vaporize the target graphite, and carbon tubes can then be formed from high-temperature quartz tubes. Generating CNTs using the CVD method typically involves placing substrates pre-coated with suitable catalysts in a high-temperature chamber. Hydrocarbon gas is pumped into the chamber where it decomposes and forms CNTs on the catalysts. The catalyst is usually made of alloys of Fe, Co and Ni.

Due to their extraordinary properties, CNTs can be potentially applied in areas including displays, CPUs, sensors, batteries, electrical and thermal conductors, coelectromagnetic wave shielding, aircraft, bullet proof vests, space elevators, fireproof materials, etc. It is believed that many of their potential applications will be commercialized in the future. On the other hand, like other nanomaterials, CNTs are potentially hazardous. Recent findings have demonstrated that animal DNA can absorb nanoparticles and CNTs, resulting in modifications to cell functions and influencing growth patterns. Nanoparticles in groundwater can interfere with the development of plant roots. Thus, prior to widespread use, the impact of CNTs on the environment, ecological systems and human health, must be systematically evaluated.

2.1 CNT thin film

Many applications of CNTs are in the form of thin film. For example, the transparent CNT film is seen as having great potential to replace the indium tin oxide (ITO) film as transparent electrodes in the future because the price of indium is keeping increase. Thus, fabrication of CNT thin film draws much research attention.

There are several methods for fabricating a CNT thin film: (1) **CVD**: This method³⁵⁻³⁹ is suitable for the fabrication large area CNT films. A catalyst of alloys of Fe, Co and Ni is coated on the substrate which is then placed into a high-temperature chamber, to which hydrocarbon gas is added. Figure 4(a) shows the SEM image of the CNT film created by CVD. (2) **Spray coating:** The CNT solution is sprayed on the substrate with an airbrush and heated to dry the CNT droplets. This simple, low-cost method⁴⁰⁻⁴¹ is also suitable for the fabrication large area CNT films. Figure 4(b) shows the schematic of the spray coating method and the SEM image of the CNT film spray coating on the substrate. (3) **Dip-coating:** The substrate is immersed into a uniform CNT solution. When removed from the substrate, the CNT solution is adhered to the substrate. As the adsorption CNT solution dries, the CNT network is formed on the substrate. This method⁴²⁻⁴⁴ has advantages in the fabrication of large area CNT films, but high conductivity requires the substrate to be dipped several times and the film thickness does not correlate linearly with the dipping times. Figure 4(c) is the schematic of the dip-coating method and the SEM image of the CNT film dip-coating on the substrate. (4) **Filtration:** The uniform CNT solution is prepared and passed through a

nanoporous filter, leaving a uniform CNT film which can then be transferred to the desired substrate. This method⁴⁵⁻⁴⁹ has the advantages in the homogeneity of the resulting CNT film but the size of the CNT film has limited by the size of the filter. Figure 4(d) shows the schematic of the filtration method and the SEM image of CNT film filtration on the nanofilter. (5) Imprint: This method involves printing CNT film with a standard office laser printer⁵⁰, inkjet printer⁵¹⁻⁵² or contact printer⁵³⁻⁵⁵. In a laser printer, CNT powder replaces the toner. The CNTs could be patterned on polymer film. Figure 4(e) shows the schematic of the laser-printer method and the CNT film laser-printed on the substrate. In inkjet printing, the CNT solution replaces the ink. Figure 4(f) shows the CNT film inkjet on the substrate. This method has an advantage in that the patterns are easily edited by a graphic or wordprocessing software on a computer. The contact printing method is popular for transferring CNT film. The growth or network CNT film is laid on the target substrate, and the transfer takes place during the contact printing process. Figure 4(g) shows the schematic of the contact printing method and the SEM image of the CNT film contact printing on the substrate. (6) Electrophoretic deposition: A uniform CNT solution is prepared in the sink. Two metals are connected to direct current (DC) and are used as the anode and the cathode. The CNT film is then deposited on the anode surface. This method⁵⁶⁻⁵⁸ is well-suited to the fabrication of large-size CNT films, and produces films with high homogeneity and proper surface roughness. However, the substrate needs a conductive anode and must be immersed in the solution. Figure 4(h) shows the schematic of the electrophoretic deposition method and the SEM image of CNT film electrophoretic deposition on the substrate. (7) Laser transfer: This method can produce large CNTs films⁵⁹ and transfer the film to a selected position 12,25,60-62. A uniform CNTs solution is placed on a transparent substrate. After the CNTs self-assemble on the transparent substrate, the mask is placed in front of the substrate. High intensity irradiation forms a thick CNT film. The mask is removed and we apply a low laser intensity to irradiate the thick pattern of CNTs. The transparent substrate has a thin residual CNTs film. Figure 4(i) shows the schematic of the laser transfer method and an image of the CNT film deposition on the substrate.

3. Patterning of CNT thin films by laser transfer and their applications

3.1 Carbon nanotube field emission display (CNT-FED)

3.1.1 Development of the field emission display (FED)

The development of the cathode ray tube (CRT) in 1879 led to the invention of the CRTbased television. Continuous improvements, including the development of color and new techniques for mass production led to this device becoming an indispensable home appliance up until the early 1990s. Although high quality CRT displays can be manufactured at low cost, conventional CRT displays cannot be scaled up in size without incurring a proportional increase in depth and weight.

Field emission displays involve an electron hitting a screen and thus inducing a light spot to appear on the screen. This principle is used extensively in displays. Generally, metal surfaces cannot easily be induced to emit electrons and doing so requires overcoming the energy barrier of the metal. The work functions for metals ranging from 2 to 5 eV. Raising the temperature of the metal provides the electron with enough energy to be emitted from the metal, a process known as heated cathode emission. On the other hand, by applying an external electric field with a magnitude large enough to overcome the work function, the electron can be emitted from the metal. This approach does not require heat and is therefore referred to as cold cathode emission.



Fig. 4. Schematics for and CNT films resulting from (a) CVD³⁷ (b) spray coating⁴⁰ (c) dipcoating⁴⁰ (d) filtration⁴⁷ (e) laser-printer⁵⁰ (f) ink-jet⁵¹ (g) contact printing⁵⁵ (h) electrophoretic deposition⁵⁷ (i) laser transfer⁵⁹.

In 1968, Spindt et al.⁶³ at the Stanford Research Institute used a semiconductor manufacturing process to fabricate a Mo tips array as seen in Fig .5(a). The field emission effect is generated by applying a high voltage bias to draw the electrons from the Mo tips to impact the phosphor where it generates a spot of light. Figure 5(b) shows the schematic of the field emission effect. The Mo tips array has a diameter of about several hundred nanometers. However, Mo-based FEDs are still not widely used because the fabrication process is still complicated and the Mo tips are easily damaged.



Fig. 5. Mo field emission display: (a) SEM image for Mo tip array. (b) Schematic of the field emission effect.

Development of CNT-FED began after Iijima's invention of CNTs in 1991. Due to their excellent emission properties, CNTs had been seen as a very promising candidate as an emitter for Flat Panel Displays (FPD). Traditionally, emitters made of Si, W and Mo suffer quick performance degradation, especially under high-voltage conditions, and researchers were searching for a substitute which could maintain a high emission current density even at low operation voltages.

Table 1 shows the electron field emission characteristics of typical emissive materials⁶⁴. The nano-diamond and the carbon nanotubes have had lowest turned on voltages in the range of $2\sim5$ V/µm. The threshold emission voltages for Si and Mo emitters are within 50 and 100 V/µm. For diamonds grown by CVD this value can range from 30 to 120 V/µm. Reports show that nano-diamond can have turn-on voltages as low as those for CNTs (i.e., $3\sim5$ V/µm). However, processing nano-diamonds as cathode emitters requires higher temperatures and is rather more complicated than fabricating CNT emitters. Fabricating CNT-FED devices involves coating and patterning CNT thin films upon an ITO glass, applying a bias voltage between the ITO glass and a positive pole with a pre-coated phosphor. The voltage bias draws electrons from the CNT tips to the phosphor plate to generate a light spot.

Cathode material	Threshold field (V/μm) for a current density of 10 mA/cm ²	
Mo tips	50-100	
Si tips	50-100	
p-type diamond	160	
Defective CVD diamond	30-120	
Amorphous diamond	20-40	
Cesium-coated diamond	20-30	
Graphite powders	10-20	
Nano-diamond	3-5 (unstable >30 mA/cm ²)	
Carbon nanotubes	2-5 (stable >1 μ A/tube)	

Table 1. Electron Field Emission Characteristics of Typical Emissive Materials⁶⁴

3.1.2 Development of CNT field emission displays

CNT-FED has many advantages over other currently available monitor products. They can be used as light sources for various purposes including street lights, table lamps, back lights for liquid crystal panels and x-ray generators. In 1995, Heer et al.65 first measured the fieldemission current density of CNTs and demonstrated them as a source for generating electrons. Many methods have been proposed to use patterned CNT thin films as cathode emitters. The most commonly-used methods include CVD, electrophoresis deposition and screen printing, and each method has its advantages and disadvantages. CNT films produced by CVD^{35-38,66} and electrophoresis deposition⁶⁷⁻⁶⁹ have been described previously in Section 3. The screen printing method has CNTs mixed with paste and squeezed onto metal meshes to pattern on the substrate⁷⁰⁻⁷⁴. The paste can firmly absorb the CNTs on the substrate. However, a weakness of the screen printing method is that the CNT tips are covered by the paste, which will affect the turn on voltage and the emission current density of the CNT film. Several post-production methods can remove the paste from the CNT tip, including the use of adhesive tape75, mechanical crushing, high speed airflow cleaning76, atmospheric pressure plasma77, laser surface treatment78 and polydimethylsiloxane elastomer⁷⁹. Figure 6(a) shows SEM images of a microcathode produced by CVD and Fig. 6(b) shows the emission image of the CNTs deposited by electrophoresis. Figure 6(c) shows the emission of fully-sealed SWNT-FEDs in color mode with red, green, and blue phosphor columns (left) and the SEM image of a screen-printed CNT cathode (right).



(a)

(b)

(c)

Fig. 6. (a) SEM images of a microcathode by the CVD; (b) emission image of CNTs deposited by electrophoresis (c) emission image of fully sealed SWNT-FED in color mode with red, green, and blue phosphor columns (left) and SEM image of screen printed CNT cathode (right)

3.1.3 Fabrication of carbon nanotube field emission cathodes by LIFT

Screen printing of large scale CNT displays is made difficult by insufficient CNT outcrops and non-uniform surface emission densities. The LIFT technique is proposed to address this problem. By this method CNT film can be printed on various substrates at room temperature with a CNT outcrop silver paste surface. It also has the advantage of sticking the CNTs firmly in the paste, thus preventing the CNTs from escaping when the substrate temperature is raised during illumination. Chang-Jian et al.,¹² demonstrated the feasibility of using the LIFT technique to print CNTs on silver paste with the steps as follows: (1) the CNT solution is dropped on transparent glass and allowed to self-assemble as in Fig. 7(a); (2) the silver paste is dissolved in alcohol and spun on the ITO substrate; (3) a uniform Nd-YAG pulse laser (1064 nm) is used to irradiate the MWNT film, with alcohol applied to the transparent side; (4) the laser light passes through the mask to the pre-coated CNT film which absorbs the laser energy, evaporates and is deposited on the silver paste as in Fig. 7(b); (5) the desired pattern is obtained on the silver paste which is heated in an oven for 1.5 hour at 100°C as in Fig. 7(c). Figure 7(d) shows the lighting image captured by CCD camera, emitting from a 12 × 12 dot array of MWNTs emitters, deposited in a 3 mm by 3 mm area on the ITO surface. Each dot MWNTs emitter has a diameter of 100 μ m. Fig. 7(e) shows a partial SEM image of a MWNTs dot array deposited on an ITO glass by laser transfer. Figure 7(f) shows the enlarged SEM image from Fig. 7(e). With a higher magnifying power, the inset shows that the feature size of the patterned emitter as small as 12 μ m.



Fig. 7. (a)-(c) CNTs film transfer on the substrate by LIFT¹²; (d) The lighting image emitting from 12×12 dot arrayed MWNTs emitters that were deposited within a square, with the dimensions of 3 mm by 3 mm, and on an ITO glass substrate. (e) SEM images showing the patterned CNT spots on ITO glass; (f) corresponding enlarged surface image of (e) with the inset SEM image enlarged from (f).

3.2 CNT field effect transistors (CNT-FETs)

3.2.1 Development of CNT-FETs

The structure of SWNTs reveals both metal and semiconductor-like characteristics that enable their application to CNT field-effect transistors. The diameter of a SWNT is about 1.4 nm, which allows for the scale of electronic devices to be greatly reduced. SWNTs have high heat conductivity that aids in the cooling in electronics, and more research is ongoing into finding applications of SWNT properties in electronic devices.

Two studies on SWNT field-effect transistors found that SWNTs can transfer both types of carriers, electrons and holes, at room temperature and that they have excellent semiconducting characteristics⁸⁰⁻⁸¹. The researchers deposited a catalyst at the source and drain of the electrodes to grow a single SWNT that spanned the two electrodes by CVD with the aim to replace the traditional semiconductor layer with a single SWNT which not only functioned as a traditional transistor, but also permitted a higher mobility and on/off ratio. However, synthesizing a 100% semiconducting SWNT is still a challenge, and current techniques usually max out at 70%.

In 2000, a researcher analyzed the relationship among metal and metal characteristic SWNTs, semiconductor and semiconductor characteristic SWNTs. The only the connection found was that metal and semiconductor characteristic SWNTs. The only the connection found was that metal and semiconductor characteristic SWNTs would transfer the hole and electron at room temperature⁸², a finding which prompted research into CNT-FETs using non-individual SWNTs as a semiconductor. The dropped⁸³ and printing⁸⁴⁻⁸⁵ methods were used to place networked SWNTs between the two electrodes. The SWNTs reveal *p*-type characteristics when they are exposed to air and absorb oxygen. Given that logic circuits are composed of both types of transistors, *n*-type CNT-FETs should be developed as well, and there are several methods to do this using SWNTs including deposition of potassium⁸⁶, desorbing oxygen by vacuum annealing⁸⁷, covering with polymer⁸⁸⁻⁸⁹, or contacting with metals⁹⁰⁻⁹¹.

3.2.2 Fabrication of CNT-FETs

There are several methods for fabricating CNT-FETs. The more common methods include CVD^{80,82, 86-88, 89-91}, spin coating⁸¹, dropping⁸³ and printing^{84-85,93,94}, each with its own advantages and disadvantages. In the CVD method the catalyst is deposited on the source/drain electrode. The SWNT is grown from the catalyst in the high temperature chamber with gas input, with the SWNT bridging the source and drain electrodes. In spin coating the SWNT solution is spread on a substrate with predefined source/drain electrodes and the SWNT network is placed between them. In the dropping method the SWNT solution dries, the SWNT network is placed between the electrodes. In the printing method the CVD growth SWNT pattern contacts the PDMS (dimethylsioxane). The SWNT pattern is removed from the substrate and then the PDMS contacts the receiving substrate. The SWNT pattern is then transferred to the predefined source/drain electrodes and the SWNT network is placed between them. Figure 8(a) shows the schematic of the CNT-FETs. Figures 8(b)-(e) show the SWNT positioned between the source and drain in the CVD, spin coating, dropped, and printing methods.



Fig. 8. (a) CNT FETs schematic; (b)-(e) SEM images of the SWNT between the source and drain by CVD⁹⁰, spin coating⁸¹, dropped⁸³, and printing methods⁹³.

3.2.3 Fabrication of CNT-FETs by LIFT

Figures 9(a)-(d) show the fabrication processes of *p*-type CNT FETs devices with MWNT electrodes. First, a uniform MWNT film is formed on a transparent glass support. The substrate is a Si wafer pre-coated with a 100 nm thick SiO₂ dielectric layer. The gate electrode is gold. As shown in Fig. 9(a), a metal mask in the desired pattern with through holes is placed between the transparent glass support and the Si substrate. The glass support, mask and the substrate are then clamped together. A pulsed Nd:YAG laser ($\lambda = 1064$ nm) irradiates the transparent glass support to transfer the MWNTs through the hole patterns in the mask and deposit the transferred MWNTs onto the SiO₂ surface. After removing the mask, the patterned MWNT electrodes are printed on the SiO₂ layer, as in Fig. 9(b).

For the *p*-type CNT FETs, the SWNTs are used as the semiconducting layer, requiring the fabrication of another transparent glass support coated with SWNT networks following the same procedures as those for the MWNT glass support. However, an additional laser irradiation step is required to regulate the amount of SWNTs on the substrate, which can be accomplished through direct laser illumination on the uniform SWNT film. Laser illumination evaporates most SWNTs on the glass leaving only a small portion on the substrate. Another mask is prepared and placed on the reverse side of the glass support. Here, however, a microscope is required for the mask alignment, as shown in Fig. 9(c).

The same procedures as those for the laser transfer of MWNTs as electrodes are used to transfer the networked SWNTs as the semi-conducting layer. Through the 2^{nd} mask, the SWNT networks can be printed on the SiO₂ layer and between two MWNT electrodes, as shown in Fig. 9(d), thus producing *p*-type CNT FETs. Figure 9(e) shows an SEM image of the MWNT electrodes on the SiO₂ dielectric layer⁶². The patterns assume square and "L" shapes. The distance between the two "L" shapes is 200 µm. The inset shows a close-up of the circled section where the individual MWNT can be easily identified. We find that the transferred MWNTs are compactly interconnected, resulting in excellent electrical conductivity for use as electrodes.

Figure 9(f) shows the I-V characteristics of the fabricated *p*-type CNT FETs after laser transfer of the SWNTs between the MWNT electrodes. The I_{SD}-V_{SD} curves are V_{SD} sweeping from 0 to 7 V as V_G varied from -15 to +15 V at an incremental step of 15 V. The inset shows I_{SD}-V_G curve as V_G sweeping from -15 to 15 V under a constant S/D bias at V_{SD} = 10 V. The current of I_{SD} decreases with the increase of V_G, revealing the characteristics of *p*-type CNT FETs, and mobility is 0.019 cm²/V s. Figure 9(g) presents an SEM image showing the formed

SWNT networks following laser transfer printing. It shows that the printed SWNTs were well interconnected between the Au source and drain electrodes²⁵. The inset is a CNT FET with the SWNT positioned between the Au source and drain electrodes. Figure 9(g) is a close-up of the circled section.

Figure 9(h) shows the transfer characteristics of the fabricated *p*-CNT FETs. The reported device had an $R_{SD} = 58 \text{ k}\Omega$. The I_{SD} - V_G curve were obtained as V_G first sweeping from -15 V to 15 V and then back to -15 V at a sweeping rate of 1 V/s and under a constant S/D bias of $V_{SD} = 0.5$ V. The arrows indicate the sweeping direction of the V_G . The inset depicts a family of I_{SD} - V_{SD} curves with V_G varying from 15 V to - 15 V at an incremental step of -7.5 V. The I-V characteristics of the device also exhibited an obvious hysteresis. These I-V characteristics reveal that the fabricated device was a *p*-type CNT-FET, and confirms the efficiency of the laser transfer printing method for fabricating networked and interconnected CNT-FETs.

3.3 CNT buckypaper

3.3.1 Development of CNT buckypaper

Buckypaper is a free-standing CNT film composed of several billion individual CNTs yet retaining the fundamental property of individual CNTs. It is lightweight, stronger than steel, thinner than paper, highly electrically and thermally conductive. Many researchers hope that buckypaper can find applications in aerospace, communications, textiles and displays. Buckypaper has been demonstrated to be 500 times stronger than the steel at only one-tenth the weight and is thus expected to contribute to stronger⁹⁵⁻⁹⁶ and lighter aircraft which would also be immune to lighting strikes. The relative lightness of such an aircraft would not only save on fuel consumption, but would also have reduced pollution output.

In textiles CNTs can be mixed with other fibers to make clothing resistant to electromagnetism or bullets. A buckypaper bulletproof vest would be much lighter and tougher than a traditional one, and the high thermal conductivity of the CNTs would provide additional comfort to the wearer⁹⁷.

The popularity of mobile phones is somewhat offset by concerns about the effect of electromagnetic waves on human health. A buckypaper covering for mobile phones could effectively shield callers from electromagnetic energy emanating from the phone⁹⁸, and this application could be extended to equipment which requires electromagnetic shielding.

Buckypaper can also be employed in the field emission displays⁹⁹, and can be used to easily create for large-sized displays at low cost. Buckypaper with two-faced CNT tips could be used to fabricate a "double-emission" display, simultaneously showing different images on a single piece of buckypaper, thus displaying messages more economically and effectively.

3.3.2 Fabricating CNT buckypaper

Commonly used methods for fabricating buckypaper included CVD^{95,100-103}, electrophoretic deposition⁹⁶ and filtration^{98-99,97,104-107}, with each method subject to advantages and disadvantages. CVD and electrophoretic deposition of CNT film had been described above in section 3.1. In the filtration method, the CNTs are suspended in a solution and deposited on the surface of a filter. After the CNTs are dry, the film is removed from the supporting filter membrane. This low cost method is fast and convenient, and can cover a large area. However, the most significant drawback is that buckypaper breaks easily while being separated from the membrane, especially in large and thin sheets. Therefore, the yield rate is low.

Buckypaper is now commercially available, but it is relatively expensive, with a disc-shaped piece of filtration fabricated buckypaper 125 mm in diameter and 0.1 mm thick selling for

\$1000. Until this price falls, buckypaper can not be used in everyday applications. Figures 10(a)-(c) show buckypaper fabricated by CVD, electrophoretic deposition and filtration.



Fig. 9. (a)-(d) schematic of CNT FET fabrication by laser transfer; (e) SEM image showing the transferred MWNT electrodes on the SiO₂ layer⁶², the inset is close-up of the circled section; (f) *p*-type CNT FETs characteristics with MWNT electrodes; (g) SEM image of SWNT networks – the inset is a CNT FET in which the SWNTs lie between the Au source and drain electrodes and the circled section is a close-up of (g); (h) p-CNT FETs characteristics of (g) inset²⁵.



Fig. 10. (a)-(c) buckypaper fabricated by CVD¹⁰⁰, electrophoretic deposition⁹⁶, and filtration¹⁰⁵.

3.3.3 Fabricating CNT buckypaper by laser irradiation

Recently, we propose a new laser transfer technique to manufacture buckypaper. The proposed technique has many advantages. It can avoid film rupture when separating from a thin and large MWNT film, thus resulting in a higher yield rate for the fabrication of thinner and larger buckypaper than can be obtained through compression. This method is also less expensive than CVD which requires fabrication in a vacuum.

Figures 11(a)-(c) illustrate the processes of fabricating MWNT buckypaper by pulsed laser separation: 1) a pipette is used to drop the MWNT solution on a transparent glass support until the whole surface is covered with a thin liquid film. The alcohol evaporated after about 20 minutes, leaving a thin uniform MWNT film on the front side of the glass, as shown in Fig. 11(a); 2) a patterned mask is placed on the glass support and a uniform laser pulse (Nd:YAG: 1064 nm, pulse width 7 ns) with a 123 mJ/cm² laser intensity irradiated the glass from the mask side, as show in Fig. 11(b); 3) scanning the entire MWNT file with a pulsed laser produces a free-standing buckypaper in the designated pattern, as shown in Fig. 11(c). Depending on the degree of transparency set in the mask pattern, a special contrast in the

height difference between the patterned and unpatterned regions was generated on the surface of the resulting buckypaper. Here a commercial PC (polycarbonate) slide was employed as the mask. The designated pattern was printed on the mask by a commercial black-and-white laser printer using HP carbon cartridges while the degrees of transparency for the mask pattern were arranged by tuning the pattern's grey levels in Microsoft Word, as shown in Fig. 11(d). Figure 11(e) shows the resulting patterned buckypaper deposited by the proposed laser-separation method. The "CCU ME" is clearly patterned on the buckypaper. The film thickness was about 40 μ m and was placed on a transparent glass slide. Figure 11(f) shows the glass support which separates the pattern from the buckypaper, as shown in Fig. 11(e). Figure 11(f) presents a photographic image showing that the MWNTs have remained on the glass support and the "CCU ME" is patterned on the glass support.

Buckypaper is able to emit electrons from both sides, which is physically appropriate for a double-sided field emitter and, thus, has potentially applications for a double-sided flat panel display or lighting source. Figure 11(g) shows the results of the field-emission characterizations obtained in a vacuum chamber at 2×10^{-5} torr base pressure. The results show the turn-on field was 1.4 V/µm at a current density of 2 µA/cm². The corresponding Fowler-Nordheim plots are shown in the inset, presenting the linear relationship between $\ln(J/V^2)$ and 1/V.



Fig. 11. (a)-(c) fabrication of CNT buckypaper by pulsed laser separation; (d) PC mask; (e) buckypaper patterned by laser-separation; (f) glass support separating the pattern from the buckypaper; (g) field-emission characterizations of double-sided buckypaper; (h) buckypaper ship on water; and (i) MWNT net on paper

The proposed approach also allows for the straightforward fabrication of buckypaper with a curved surface. Figure 11(h) shows a buckypaper presented in the shape of a ship floating on water. This was accomplished by dropping the MWNT solution on the inside of a PMMA spoon, allowing the MWNTs to coat the inside surface of the spoon. We irradiated the MWNT film from the outer surface of the spoon, after which a convex MWNT film separated from the spoon's inside surface. Due to the lightweight and hydrophobic nature of the MWNTs, this buckypaper ship floated very well on water. Thus, shaped buckypaper can be fabricated directly from curved MWNT film, rather than being folded from flat buckypaper, but the process requires a transparent mold in the desired shape as a support.

Figure 11(i) shows a fabricated MWNT net placed on a dollar bill. The net is composed of square grids of different sizes. The inset shows a magnified SEM image demonstrating the line interconnection of a grid point. The line width was about 189 μ m. To achieve this effect, we dropped the MWNTs solution on the transparent glass support and waited about twenty minutes for the alcohol to evaporate, leaving the transparent glass support coated with the MWNT film. We then wet the MWNT film with alcohol and placed a mask in the shape of square grids on top of the transparent glass support. A high fluence laser (187.5 mJ/cm²) passed through the square grid mask and transparent glass support. The sections of the MWNT film unprotected by the mask were separated from the transparent glass support, leaving only the protected square grid shape. We removed the mask and used a low fluence laser (123 mJ/cm²) to scan the transparent glass support. The MWNT film separated from transparent glass support and forming a square grid free-standing MWNT net about 100~200 μ m wide. An MWNT net fabricated by laser separation must be line-limited. If the mask's line pattern is too narrow, it will be broken by the irradiation energy of the laser.

3.4 CNT thin films

3.4.1 Development and fabrication of CNT thin film

In section 3.1 we described several methods for fabricating CNT thin film. At present, CNT thin film could be fabricated in the random networks, vertically alignment and horizontal alignment. Research interest into transparent CNTs thin films has increased with the price of indium tin oxide (ITO) film. ITO film has been used as a universal electrode in various optoelectronic devices such as organic light emitting diodes, solar cells and liquid-crystal displays because of their high transmittance in the visible region and good electrical conductivity. However, not only are ITO films expensive, but their electric conductivity changes when they bend^{43,48,50}. Transparent carbon nanotube thin film is highly conductive, it does not change when bent and its low cost make it very suitable for replacing ITO film in next generation products. SWNT/PET sheet resistance was ~80 Ω /sq, with ~80% transparency. It has been extensively used in the optical applications⁴⁵, organic light-emitting diodes⁴⁷, transistors⁵², gas sensors⁴⁶, solar cells⁴¹ and field emission devices.^{51,59}.

3.4.2 CNT thin film fabrication by laser irradiation

Recently, we proposed a practical method for using laser separation to form an MWNT thin film on a flexible transparent substrate⁵⁹. The fabricated MWNT thin film can be sparsely networked and, given sufficient outcrop tube tips, the film surface demonstrates good field-emission characteristics. The laser-based method is less vulnerable to the problem of insufficient outcrops found in the filtration method. By contrast, CVD fabrication must be

executed in a high vacuum and at high temperatures, which raises the cost and is not suitable for flexible substrates. In addition to its ability to form MWNT thin films directly on a curved surface, our proposed method can also be used to fabricate a MWNT thin film with precision patterns and with varying spatial concentrations.

Figures 12(a)-(d) show the processes of fabricating a patterned MWNT thin film with extrusive and vertically aligned surface MWNTs by laser separation. (1) The designated amount of MWNTs is dispersed uniformly in alcohol. (2) A pipette is used to drop the MWNT solution on a polycarbonate (PC) substrate until the whole surface is covered with a thin liquid film which, once the alcohol evaporates, leaves a thin uniform MWNT film on the flexible substrate. (3) A mask with the desired pattern is placed in contact with the back surface of the substrate, i.e., without the pre-coated MWNT film. (4) The pre-coated MWNTs film is wetted with alcohol and then irradiated by a high fluence laser as shown in Fig. 12(a). (5) A small portion of the laser energy is dissipated by reflection by the mask and absorption by the substrate. The penetrating energy is mainly absorbed by the MWNTs around the substrate and the MWNT film interface, causing the MWNTs to evaporate and providing a thrust to separate the unprotected MWNTs from the substrate. Following laser irradiation, the protected part of the MWNT film remains on the substrate, as shown in Fig. 12(b). (6) The mask is removed, and a second stage of laser irradiation is executed with a low fluence laser scanning the substrate from the back, as shown in Fig. 12(c). (7) The second stage of laser irradiation leaves a thinner, more transparent patterned MWNT film on the substrate. At the same time, a free-standing CNT sheet (buckypaper) is formed with the same pattern, as shown in Fig. 12(d). This second-stage laser irradiation reduces the thickness of the patterned CNT film and reinforces the CNTs' vertical alignment. The light source was a pulsed Nd:YAG laser with a wavelength of 1064 nm, a repetition rate of 10 Hz, a pulse width around 7 ns, a maximum output energy of 28 mJ, and a raw beam diameter of 2.75 mm. The fluences for the first-stage and second-stage laser irradiations were set at 187.5 and 123 mJ/cm², respectively.

Figure. 12 (e) shows a bent PC substrate used to fabricate the MWNT film with line, grid and square patterns. The substrate was placed on a captioned paper and the caption can be clearly identified through the fabricated transparent MWNT film. Figure 12(f) shows a MWNT thin film on a curved surface, demonstrating the feasibility of direct film fabrication on a curved surface. Figure 12(g) and its inset show a side view SEM image of a fabricated MWNT film surface, showing numerous tube tips protruding from the film's surface, sufficiently dense for field-emitter applications. Figure 12(h) shows the current density as a function of the electric field for a fabricated MWNT thin film on a PC substrate. The turn-on voltage is around 1 V/µm and the inset shows the Fowler-Nordheim plot of the same data.

Widely-used methods for fabricating CNT field emitters include screen-printing, *in situ* growth of CNT tips, and melt mixing of CNT composites. The reported turn-on voltage was in the range 1 to $6 \text{ V}/\mu\text{m}$. Without any parameter optimization in the fabrication process and thin film configuration, the present approach produces excellent field-emission properties even on a plastic substrate. The low turn-on voltage was due to the CNT playing the roles of both the electrode conductor and the field emitter. In terms of supporting electron conduction, this type of configuration can avoid possible non-Ohmic contacts or interface barriers caused by different work functions between the conductor electrode (e.g., silver paste or indium tin oxide) and the CNT emitter. CNTs with good vertical alignment have been demonstrated to exhibit better field-emission characteristics which facilitate electron transport and reduce the turn-on voltage.



Fig. 12. (a)-(d) Fabrication of a patterned MWNT thin film by laser separation;⁵⁹. (e) MWNT pattern on the PC substrate; (f) MWNT film on the curved surface; (g) side view of the SEM image showing numerous protruding tube tips on the film surface; And (h) characteristics of MWNT field emitter on PC substrate.

3.4.3 Pattern DWNT thin films by laser ablation

A transparent DWNT flexible thin film was used to fabricate a flexible matrix touch panel by laser ablation, achieving several advantages: the transparent DWNT thin film is considerably less expensive than the ITO film it replaced, and the manufacturing process is less environmentally damaging. In addition, the process is faster while produces larger outputs and higher yield rates than other methods.

Figures 13(a)(b) show the steps for fabricating patterns on transparent DWNT flexible thin film. First, we placed a metal mask on a polyester (PET) substrate which had been preloaded with a transparent DWNT flexible thin film (XinNano materials, Inc). The DWNT thin film had the thickness of 20–40 nm, with a sheet resistance of 10^{2} - $10^{3} \Omega/sq$ and visible light transmittance of 75-88%. The metal mask with line patterns is laid in contact with the PET substrate on the side without the DWNT thin film. Laser light then passes through the mask openings to irradiate the DWNT thin film below. By simultaneously moving the mask and the PET substrate, the designated area on the PET substrate can completely be exposed to laser light, as shown in Fig. 1(a). The laser used is a pulsed Nd:YAG laser (Continuum) which operates at a wavelength of 1064 nm with a pulse duration between 5 and 7 ns. The size of the laser spot is about 6 mm in diameter and it has a maximum output power of 485 mJ which corresponds to 1716 mJ/cm² in intensity.

As the laser light passes through the mask openings and the transparent PET film, its energy is mainly absorbed by the DWNT thin film on the other side of the PET, causing the DWNTs to evaporate from the PET substrate. Finally, the non-irradiated portion of the DWNT thin film, protected by the mask, remains on the PET substrate to complete the DWNT thin film through laser ablation. Figure 13(b) shows the patterned DWNT thin film on the PET substrate after the mask is removed. Figure 13(c) presents a photographic image of a substrate with a patterned DWNT thin film. The patterned area is 7 cm \times 7 cm and contains 100 DWNT line patterns. The caption printed on the paper behind the PET with the patterned DWNT thin film is clearly visible.

Figure 13(d) is an SEM image showing the DWNT line patterns on the PET substrate. The brighter regions are the DWNTs which remain on the PET substrate, while the darker areas are the PET substrate where the pre-coated DWNTs have been ablated by laser. The width of the DWNT lines is 200 μ m and the distance between the lines is 150 μ m. The inset is an SEM image enlarged from the marked square part of Fig. 13(d), showing that the DWNTs tangle together and the tube diameter ranges from 10 to 20 nm. With the laser ablation technology, we can easily make more complicated patterns. Figure 13(e) is an SEM image showing curved DWNT patterns with a line width of 223 μ m. Again, the darker region is the PET surface where the DWNT thin film was ablated.

Figures 13(f)(g) demonstrate the feasibility of fabricating a transparent matrix touch panel by laser patterning of the DWNT thin film. Figure 13(f) schematically shows the exploded diagrams of this matrix touch panel. A spacer is placed between two PET substrates where patterned DWNT thin films are formed by the proposed laser ablation method. The spacer is a commercially-available overhead transparency with a thickness of 100 μ m. We use a CO₂ laser to fabricate nine through holes. Each hole is a 1.5 cm ×1.5 cm square, and the distance between any two holes is 2 mm. On each of the PET substrates three pieces of 60 μ m-thick aluminum foil are placed in contact and aligned in parallel with the patterned DWNT lines, as shown in Fig. 13(f).

The aluminum foil serves as an electrode. The PET substrates, spacer, and aluminum foil pieces are clamped together using metal clips. The two PET substrates are arranged in such way that their patterned DWNT lines are perpendicular to each other. Figure 13(g) demonstrates the function of this transparent matrix touch panel under bending. The curved

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surface of the panel is created by sticking it to the convex surface of a cylindrical glass cup using adhesive tape. The radius of the cup cylinder is 6.5 cm. The top-right corner of Fig. 13(g) presents a schematic showing the top view of the glass cup with the matrix touch panel above a schematic showing the arrangement of the testing circuit. We used a 9 V battery to power a blue LED. When the location on the panel surface is pressed, the patterned DWNT lines on the two separated PET substrates can be well connected and the LED is activated successfully.



Fig. 13. (a)(b) DWNT film patterning process; (c) DWNT pattern; (d) SEM image of (c), with the inset showing the enlarged SEM image; (e) SEM image of the DWNT pattern; (f) combined schematic of the matrix touch panel; (g) function of the flexible transparent matrix touch panel. The blue color LED is driven by a 9V battery.

4. Summary

We report on the employment of the Nd:YAG laser (1064 nm) to pattern the CNT thin films and fabricate the CNT-based devices in this article. We demonstrate fabrications of carbon nanotubes field emission display (CNT-FED), carbon nanotubes field transistor (CNT-FETs), CNT buckypaper, CNT thin film and transparent DWNT flexible matrix touch panel.

In the CNT-FED, we patterned the CNT field emission cathodes on the substrate by the LIFT technique. The emission test revealed favorable emission characteristics, such as high emission current density, low threshold electrical field, good emission stability and good emission focusing. This method can also afford high pattern resolution, with a feature size down to $10 \,\mu$ m, high feasibility of using various substrates, good MWNTs adhesion and fast deposition rate. Furthermore, all steps can be executed in an ambient environment and at low temperature, consequently offering the benefit of potential low cost fabrication of precision pattern deposition.

As for the CNT-FETs, we report the SWNT printing between the Au electrode and reveal the p-type CNTFETs by LIFT technique. We also successfully demonstrated patterning MWNTs electrodes on SiO_2 substrate and SWNTs between the MWNTs electrode by the LIFT technique. It revealed the p-type CNTFETs characteristics. By introducing a X-Y stage, this method can easily be extended to fabricate large area CNT circuits.

In regard to the CNT buckypaper, we simply accomplished it through employing a pulsed laser to irradiate a transparent support and separate the precoated MWNTs film. Based on this approach, buckypapers with surface patterns and with three-dimensional surfaces were fabricated and demonstrated. The film thickness of the resulting buckypaper could be regulated through adjusting the film thickness precoated on the support. The flexibility of the formation of complicated surface patterns and feasibility in fabrication of thin papers makes the proposed approach a prospective method forbuckypaper fabrication.

Regarding the transparent CNT thin film, we report a new low-temperature method for fabricating thin film MWNTs on a polycarbonate substrate based on the laser peeling method. This method can fabricate a film with varying MWNTs concentrations straightforwardly. The fabricated sparsely networked MWNTs thin film exhibits the feature of sufficient outcrop tube tips on the film surface. It is a favorable arrangement for the field-emission application. The emission test reveals that a low turn-on of 1 V/ μ m is obtained without any optimization in process and device configuration. Furthermore, all steps can be executed in an ambient environment and at low temperature, consequently offering the benefit of potential low cost fabrication for patterned MWNTs films on flexible substrates.

Regarding the transparent DWNT flexible matrix touch panel, we again use the laser to pattern the DWNT thin film on a plastic substrate and successfully fabricated a flexible matrix touch panel. By increasing the laser energy, the ablation depth of transparent DWNT thin film is increased but sheet resistance is decreased. When laser energy intensity reaches 117 mJ/cm², the DWNT can be ablated completely from transparent DWNT flexible thin film. The method is rapid, simple, applicable to large area processing and very suitable for mass production.

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Carbon nanotubes (CNTs), discovered in 1991, have been a subject of intensive research for a wide range of applications. In the past decades, although carbon nanotubes have undergone massive research, considering the success of silicon, it has, nonetheless, been difficult to appreciate the potential influence of carbon nanotubes in current technology. The main objective of this book is therefore to give a wide variety of possible applications of carbon nanotubes in many industries related to electron device technology. This should allow the user to better appreciate the potential of these innovating nanometer sized materials. Readers of this book should have a good background on electron devices and semiconductor device physics as this book presents excellent results on possible device applications of carbon nanotubes. This book begins with an analysis on fabrication techniques, followed by a study on current models, and it presents a significant amount of work on different devices and applications available to current technology.

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